STATE OF MICHIGAN

IN THE SUPREME COURT

ATTORNEY GENERAL FOR THE STATE OF MICHIGAN *ex rel.* MICHIGAN DEPARTMENT OF ENVIRONMENT, GREAT LAKES AND ENERGY,

Plaintiffs-Appellees,

and

THE CITY OF ANN ARBOR, WASHTENAW COUNTY, THE WASHTENAW COUNTY HEALTH DEPARTMENT, WASHTENAW COUNTY HEALTH OFFICER JIMENA LOVELUCK, THE HURON RIVER WATERSHED COUNCIL, AND SCIO TOWNSHIP

Intervenors-Appellees,

v

GELMAN SCIENCES, INC., a Michigan Corporation,

Defendant-Appellant.

Supreme Court Docket No. _______

Court of Appeals Docket No. 357598

Washtenaw County Circuit
Court Case No. 88-034734-CE

DEFENDANT-APPELLANT'S APPENDIX

VOLUME II

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DEFENDANT-APPELLANT'S APPENDIX.

TABLE OF CONTENTS

Volume II

STATE OF MICHIGAN

IN THE CIRCUIT COURT FOR THE COUNTY OF WASHTENAW

ATTORNEY GENERAL FOR THE STATE OF MICHIGAN *ex rel*. MICHIGAN DEPARTMENT OF NATURAL RESOURCES AND ENVIRONMENT,

Plaintiff,

and

THE CITY OF ANN ARBOR,

Intervenor,

and

WASHTENAW COUNTY,

Intervenor,

and

THE WASHTENAW COUNTY HEALTH DEPARTMENT,

Intervenor,

and

WASHTENAW COUNTY HEALTH OFFICER, JIMENA LOVELUCK,

Intervenor,

and

THE HURON RIVER WATERSHED COUNCIL,

Intervenor,

and

SCIO TOWNSHIP,

Intervenor,

v

GELMAN SCIENCES, INC., a Michigan Corporation,

Defendant.

_______________________________________/

Case No. 88-34734-CE

Hon. Timothy P. Connors

GELMAN SCIENCES, INC.'S REPLY BRIEF IN SUPPORT OF MOTION FOR RECONSIDERATION

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DEFENDANT GELMAN SCIENCES, INC.'S REPLY BRIEF IN SUPPORT OF MOTION FOR RECONSIDERATION

INTRODUCTION

The Intervenors ask this Court to ignore the record established in decades of prior proceedings and in binding case law, all of which demonstrate that the envisioned remedy hearing, and the Scheduling Order setting that hearing, are in clear error. In an attempt to distract from the valid concerns Gelman has expressed in its motion for reconsideration—concerns which the Court itself recognized as substantial and meritorious during the February 4, 2021 hearing—the Intervenors paint a selective picture of the history of this decades-old enforcement action and the Court's role in enforcing the terms of the multiple Consent Judgments agreed to by the parties to this matter—the State and Gelman. As set forth below, none of the Intervenors' grounds for urging the Court to proceed with the procedurally and substantively improper remedy hearing have merit.

For the reasons set forth herein, Gelman respectfully reiterates its request that this Court reconsider and vacate its Fourth Amended Scheduling Order, and either dismiss the interventions without prejudice (which would allow Gelman and the State to enter a bilateral agreement), or order the Intervenors to file their complaints so that the merits of their claims can be litigated without further delay.

I. Intervenors misstate the limited circumstances under which a court can modify a consent judgment and misrepresent this Court's past enforcement of the existing terms of the Consent Judgment.

Intervenors spend nearly half of their opposition asserting that the Court's inherent and equitable powers enable it to "enforce its own directives" via a modification to the existing Consent Judgment—without the consent of the parties to that action and without any allegation of a violation of the existing terms. This is wrong, and in any event, does not provide any legitimate basis for allowing the envisioned remedy hearing to proceed.

A. The Consent Judgment cannot be modified to address the long-anticipated change in the cleanup criteria without the consent of the parties to that agreement.

The Intervenors argue that EGLE's adoption of more stringent cleanup criteria is a "changed circumstance" justifying modification of the Consent Judgment. This argument is entirely without merit. First, EGLE and Gelman anticipated precisely this circumstance—the adoption of new cleanup criteria—and fully addressed how the parties would respond to the changed criteria in the Consent Judgment. Second, neither EGLE nor Gelman—the only parties to the Consent Judgment—is seeking relief from the existing judgment. Accordingly, this anticipated regulatory change does not meet the strict standard for judicial modifications to a consent judgment—let alone court-ordered changes made *sua sponte*.

"[C]ourts have traditionally considered the modification of a consent decree to be serious, leading to 'perhaps irreparable' consequences." *US v State of Michigan*, 940 F2d 143, 150 (CA 6 1991) (quoting from *Carson v American Brands, Inc.*, 450 US 79, 84; 101 S Ct 993, 996; 67 LEd2d 59 (1981)). Thus, "[t]he standard for justifying the modification of a consent decree is a *strict* one." *US v Michigan*, 940 F2d at 150 (emphasis in original). Gelman does not dispute that there are limited circumstances, including mistake, fraud and changed circumstances, under which a court may modify a consent judgment, but such a modification must be at the request of a **party** to that agreement, and must be made under court rules providing for relief from a judgment. *Vanguards of Cleveland v City of Cleveland*, 23 F3d 1013, 1018 (CA 6 1994), citing *Rufo v Inmates of Suffolk County Jail*, 502 US 367, 383, 112 S Ct 748, 760 (1992) (decided under Fed R Civ P 60(b)).[1] These limited circumstances manifestly do not apply to the present situation.

First, when a party to an agreement—which the Intervenors are not—seeks to modify a consent decree based on "events that actually were anticipated at the time it entered into a decree," the modification should be denied. *Rufo*, 502 US at 385 ("[M]odification should not be granted where a party relied upon events that actually were anticipated at the time it entered into a decree"); *see also Vanguards*, 23 F3d at 1018. Here, EGLE and Gelman anticipated exactly the "changed circumstances" at issue—the adoption of new cleanup criteria—and fully addressed such

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[1] The Michigan court rule analogue to Fed R Civ P 60(b) is MCR 2.612.

circumstances within the terms of the existing Consent Judgment. Such anticipated changes therefore do not provide a basis for unilateral judicial modifications to a consent judgment.

The Third Amendment to Consent Judgment was entered in March 2011. In negotiating that agreement, EGLE and Gelman were fully aware that the USEPA had revaluated the toxicity of 1,4-dioxane and issued its final Toxicological Review in August, 2010 (Exhibit 1). EGLE made clear that it anticipated adopting more restrictive criteria for 1,4-dioxane based on USEPA's reevaluation, and to that end, insisted on including new language in the Third Amendment addressing this anticipated circumstance in the Consent Judgment's "reopener" provisions. *See* Exhibit 2, Consent Judgment, Section XVIII "Plaintiffs' Covenant Not To Sue And Reservation of Rights" at pp 29-31.

Subsections XVIII A-D describe the scope of the State's covenant not to sue Gelman with respect to the "Covered Matters," a promise given in exchange for Gelman's commitment to undertake the response activities identified in the Consent Judgment. Subsection XVIII.E. then identifies the only circumstances in which the State is not bound by that covenant and can initiate litigation against Gelman. Specifically, Section XVIII.E. states:

> Notwithstanding any other provision in this Consent Judgment: (1) <u>Plaintiffs reserve the right to institute proceedings in this action or in a new action seeking to require Defendant to perform any additional response activity at the Site</u>; and (2) Plaintiffs reserve the right to institute proceedings in this action or in a new action seeking to reimburse Plaintiffs for response costs incurred by the State of Michigan relating to the Site. <u>Plaintiffs' rights in E.1. and E.2. apply if the following conditions are met</u>:
>
> 1. For proceedings prior to Plaintiffs' certification of completion of the Remedial Action concerning the Site,
>
> > a. (i) conditions at the Site, previously unknown to the Plaintiffs, are discovered after entry of this Consent Judgment, (ii) new information previously unknown to Plaintiffs is received after entry of the Consent Judgment, <u>or (iii) [EGLE] adopts one or more new, more restrictive cleanup criteria for 1,4-dioxane pursuant to Part 201 of the Natural Resources and Environmental Protection Act (NREPA), MCL 324.20101 et seq., after entry of the Consent Judgment</u>; and

> b. these previously unknown conditions, new information, and/or change in criteria <u>indicate that the Remedial Action is not protective of the public health, safety, welfare, and the environment</u>;

Exhibit 2, p 30, Section XVIII.E. (emphases added).

Gelman and EGLE added the underlined language in Subsection XVIII.E.1.a.(iii) to address the precise and sole circumstance that Intervenors now claim empowers this Court to modify the Consent Judgment on its own volition and in disregard of these specific terms. The precedents are uniform that anticipated changes which were already explicitly addressed in the parties' agreement cannot support a Court-directed Consent Judgment modification. *Rufo*, *supra; Vanguards*, *supra*. In fact, far from supporting the Court's proposed remedy hearing, the existing Consent Judgment makes clear that, absent an agreement of the parties to the contrary, such a changed circumstance would require the reopening of *litigation*, necessitating that Gelman's liability and responsibility be factually and legally established before further remedial proceedings could occur. Thus, Intervenors' key argument that "changed circumstances" authorize this Court to hold the proposed remedy hearing as a precursor to unilaterally modifying the Consent Judgment to impose additional remedial obligations on Gelman plainly fails. Such a ruling would not only be inconsistent with the terms of the existing Consent Judgment, but it would also be beyond the Court's authority.

Second, and in any event, neither of the parties to the Consent Judgment is seeking relief from the existing judgment. Even in a situation where the parties did not anticipate or address the "changed circumstances" at issue—a situation not present here—a court may not modify a consent judgment on a *sua sponte* basis as Intervenors suggest without a request from a party to do so. Rather, a court's discretion to modify a consent judgment only arises in response to a motion for relief from a judgment, i.e., MCR 2.612(C) or Fed R Civ P 60(b). *See Rufo*, 502 US at 383.

Notably, Intervenors fail to identify any precedent that would authorize a court to take such unilateral action. Rather, all of the cases that allow for a modification of a consent judgment due to unanticipated changed circumstances arise out of at least one *party's* motion seeking a change on this basis. Further, in the context of a consent judgment, the Court is not entitled to impose its preferred remedy where the parties to the consent judgment have agreed on another or different remedy. *See United States v State of Michigan*, 940 F2d 143, 159 (CA 6 1991) (District court abused its discretion in overly intruding upon sovereignty of Michigan and the terms and conditions of a consent decree by materially extending and modifying the consent decree).

For all the reasons discussed above, a *sua sponte* ruling determining the appropriate remedy in this case or otherwise modifying the existing Consent Judgment would require the Court to ignore the plain terms of that document. And even if the parties had not anticipated and addressed the possibility that the State would adopt more restrictive cleanup standards—which they did—the Court's inherent power to *enforce* its judgments (discussed below) is limited and still does not include the power to unilaterally modify a consent judgment as Intervenors suggest. In short, there is no legal support for Intervenors' requested course of action.

B. The Court's inherent authority to *enforce* its judgments is not so broad as to allow it to unilaterally *modify* the Consent Judgment as Intervenors suggest, nor has the Court done so in the past.

Intervenors also erroneously suggest that the Court's prior enforcement of the previous Consent Judgment somehow gives the Court authority to unilaterally modify the Consent Judgment today. They further argue, incorrectly, that Gelman is judicially estopped from opposing the Court's proposed remedy hearing, which would result in a modification of that agreement. Far from providing the Court with a valid basis to proceed with the envisioned remedy hearings, these arguments simply reflect that the Intervenors misunderstand both the prior proceedings and the law of judicial estoppel.

Intervenors' incomplete summary of the enforcement history of this case demonstrates, if nothing else, that the State has actively enforced the terms of the Consent Judgment for decades. What the enforcement history does not support is Intervenors' claims that the Court has repeatedly and unilaterally modified the Consent Judgment or taken any action with respect to the Consent Judgment on a *sua sponte* basis. Rather, the Court has in the past: (i) responded to requests by one of the parties to enforce the existing terms of the Consent Judgment; (ii) resolved petitions for dispute resolution as required by the Consent Judgment regarding the proper interpretation of existing terms of the Consent Judgment and what they required; or (iii) received (but never addressed) a request for relief from the existing terms of the Consent Judgment based on the well-recognized bases for seeking such relief summarized above—mistake of fact or an *unanticipated* change in circumstances. Neither the Court's previous actions nor Gelman's related legal arguments provide any support for Intervenors' baseless suggestion that this Court can simply issue a "supplemental remediation order" (Intervenor Opp'n, I.B., pp. 6-9)—which does not even purport to enforce the current Consent Judgment and especially here, where neither EGLE nor Intervenors allege that Gelman is in violation of the existing Consent Judgment.

The remediation Gelman is required to carry out is not a "cleanup regime" that can be unilaterally modified by the Court as Intervenors suggest: "The remedial action is to be performed according to *the specific terms of the Consent judgment* and plans approved by [EGLE] *under the Consent Judgment.*" (Exhibit 3, Plaintiffs' Motion to Enforce Consent Judgment dated 2/14/2000, ¶ 1 (e*mphases added*)). Intervenors' reliance on the Court's references in its past orders to its "inherent and equitable powers to enforce its judgment" or its "inherent powers to enforce its own directives" (i*d.*, p. 5-6) is misplaced. In each case, those statements were made in the context of enforcing the *existing* Consent Judgment requirements. And as set forth below, each of those enforcement proceedings and related orders upon which Intervenors rely involved the Court's enforcement or interpretation of the existing terms of the Consent Judgment *at the request of one of the parties*, not a *sua sponte* modification of the Consent Judgment. Moreover, even if one or more of the Court's previous orders could be construed as analogous to the type of unilateral remediation order Intervenors seek—and as explained below, they cannot—the fact that Gelman did not appeal those historical orders in the past does not somehow bar Gelman today from seeking appellate review with respect to a new order for a proposed *sua sponte* remedy hearing, or otherwise serve as precedent authorizing this Court to take action that is contrary to the terms of the Consent Judgment and applicable law.

1. The 2000 Remediation and Enforcement Order arose from the State's motion to enforce the terms of the existing Consent Judgment.

Intervenors argue, without basis, that entry of the July 2000 Remediation and Enforcement Order ("REO") supports their position that the Court can proceed with the proposed remedy hearing and issue a supplemental remediation order imposing additional remedial responsibilities on Gelman. (Intervenor Opp'n, p. 4). In reality, the REO resolved the State's Motion to Enforce Consent Judgment, which alleged several violations of the terms of the existing Consent Judgment and related EGLE-approved work plans. EGLE's motion sought significant stipulated penalties and asked the Court to order Gelman to increase its treatment capacity and to undertake other response activities in order to achieve compliance with the existing Consent Judgment requirements (Exhibit 3, ¶¶ 44-47, p. 10). For its part, Gelman vigorously refuted EGLE's allegations and explained that many of the alleged violations and delays arose from EGLE's failure to provide required approvals of work plans, the City's refusal to provide access for required pipelines, all of which were required to allow Gelman to ensure continued compliance with the Consent Judgment requirements. Gelman also pointed out that its ability to increase its treatment capacity as demanded by the State was being held up by the challenges to Gelman's requested NPDES discharge permit amendment filed by the City and a local citizens' group (the predecessor for the current CARD group that opposed the now-rejected settlement). (Exhibit 4, Gelman's Opp'n to Plaintiffs' Motion to Enforce Consent Judgment, pp 6, 12-15, 21-23).

The Court's REO resolved the parties' disputes regarding alleged violations of the existing Consent Judgment requirements, as presented to the Court via the State's enforcement motion, by essentially ordering Gelman to implement the steps it told the Court that it needed to take in order to implement the existing Consent Judgment requirements, thus doing away with the need for the tardy State approval. (REO, Exhibit 5, p 4-5, ¶¶ 2-7).[2] The Court's entry of the REO—upon motion by a party and to enforce the terms of an existing agreement—does not support Intervenors' request that this Court issue a unilateral supplemental remediation order or a modification of the Consent Judgment. Here, there are no alleged violations of the existing settlement agreement, and no motion by a party to the Consent Judgment seeking either enforcement or interpretation of its terms, but rather a request for relief by entities who have not even filed actual Complaints in this lawsuit.

2. The Unit E Order/Prohibition Zone Order arose from EGLE's assertion that Gelman was required to address the Unit E Plume under the Consent Judgment.

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[2] Intervenors will point out that the Court also added the goal of completing the mutually desired aquifer restoration within five years (Exhibit 5, p 4). Gelman chose not to appeal that aspect of the REO, but rather took on the challenge of trying to meet the goal set by the Court. Gelman's decision to pursue the shared goal of aquifer restoration rather than appealing that aspect of the REO in no way supports the Court's issuance of a unilateral remediation order now in the absence of any alleged Consent Judgment violations. It should be noted that, although aquifer restoration proved infeasible due to a number of geologic factors and the limits of pump and treat technology not understood by the parties at the time, the Court recognized Gelman's efforts and the progress made in its subsequent Order regarding the Unit E plume, concluding that "[Gelman] has complied with the terms of [the REO]." (Exhibit 6, p 3).

Intervenors next argue, again without basis, that the Unit E Order/Prohibition Zone Order supports their demands that the Court enter a supplemental remediation order. (Intervenors Opp'n, pp 5-6). These Orders arose from the parties' dispute over whether the Consent Judgment required Gelman to address the newly discovered Unit E plume and, if so, what steps Gelman should be required to take in order to comply. (Unit E Order, Exhibit 6, pp. 3-4). While reserving the right to contest the applicability of the Consent Judgment to the Unit E plume, Gelman presented its proposed cleanup plan to the Court, as did the State. As noted by Intervenors, the Court rejected Gelman's argument that the Unit E plume was not part of the Consent Judgment's Western System requirements (Intervenor Opp'n, p. 5; Unit E Order, p. 4). Having concluded that the Consent Judgment required Gelman to remediate the Unit E plume, the Court made clear that its Unit E Order/Prohibition Zone Orders arose from the Court's "enforcement" of the existing Consent Judgment: "The Court has the inherent and equitable powers *to enforce its judgment* with all appropriate measures and sanctions as to Unit E contamination." (Unit E Order, p. 4) (emphasis added). Because the Court's Unit E Order adopted most aspects of Gelman's proposed cleanup plan for Unit E, there was no reason for Gelman to appeal the Court's conclusion that the Unit E plume fell within the Consent Judgment.

The Unit E resolution was entirely different than the current situation, where there is no allegation that Gelman has failed to address any area of contamination that it is required to address under the existing Consent Judgment. Gelman has fully complied with the existing Consent Judgment and, as a result, neither party to the agreement has sought to enforce its terms or petitioned the Court to resolve a dispute arising from the requirements therein. Therefore, there is no basis for the Court to issue an order "enforcing" the current Consent Judgment, let alone grounds to undertake a remedy hearing to impose judicially selected terms beyond the agreement of the parties.

3. In 2007 Gelman properly sought relief from certain Consent Judgment requirements because of unanticipated changed circumstances and mutual mistake.

Intervenors also argue that Gelman's 2007 Motion to Amend Consent Judgment ("Motion to Amend") supports issuance of a unilateral remediation order and now prevents Gelman from arguing otherwise. (Intervenor Opp'n, p. 7). In fact, Gelman's filing was entirely consistent with the legal precedent described above that precludes the Court from unilaterally modifying the Consent Judgment based on the results of the Intervenors' proposed remedy hearing.

Gelman's filing arose from a long-running technical dispute regarding Gelman's compliance with the existing Consent Judgment's requirement that Gelman capture the leading edge of the D2 Plume in the Evergreen Area. The State claimed Gelman violated this requirement and sought to impose stipulated penalties for the alleged violation. Gelman argued that the alleged violation occurred because the Evergreen extraction wells were unexpectedly pulling 1,4-dioxane from the Unit E plume, previously believed to be hydrogeologically separate from the D2 plume. Of course, neither Gelman nor EGLE was aware of the Unit E plume when they established the Evergreen D2 capture cleanup objective in 1992. To address this unanticipated development and to challenge the State's attempt to assess stipulated penalties for an alleged noncompliance, Gelman contemporaneously filed both: (i) a Petition for Dispute Resolution challenging the State's interpretation of the Consent Judgment requirements in light of this new data and attempt to assess stipulated penalties (Exhibit 7); and (ii) its Motion to Amend, which sought relief from and amendment of the Consent Judgment based upon mutual mistake of fact and an unanticipated change in circumstances. (Brief in Support of Motion to Amend Consent Judgment, Exhibit 8, pp 8-10 (citing, *Vanguards*, *supra*; *Rufo*, *supra*)). In fact, the Court never amended the Consent

Judgment in response to Gelman's motion; instead, the parties ultimately agreed to incorporate the changes Gelman sought into the Third Amended Consent Judgment, entered by stipulation in 2011.

Thus, far from supporting the Intervenors' position here, Gelman's 2007 request for relief from the Consent Judgment due to unanticipated changed circumstances and mistake of fact is entirely consistent with the law governing modification of consent agreements. Furthermore, it is on all fours with Gelman's current opposition to any effort by the Court to unilaterally modify or supplement the Consent Judgment requirements to address changed circumstances already anticipated in the Consent Judgment and at the behest of non-parties to the Consent Judgment.[3]

4. The 2011 Order was in fact part of a consensual amendment to the Consent Judgment.

Finally, Intervenors mysteriously assert that the 2011 "*Stipulated* Order Amending Previous Remediation Orders" was not "entered as a consensual amendment to the Consent Judgment" and that its entry would now support their request for a similar non-consensual modification of the Consent Judgment via the envisioned remedy hearing. (Intervenor Opp'n, p. 4). As the word "Stipulated" would suggest, this Order was in fact entirely by consent. Moreover, it was submitted and subsequently entered contemporaneously with the agreed upon Third Amendment to Consent Judgment, which set forth extensive—and *agreed-upon*—modifications to the previous version of the Consent Judgment. (*See* 2011 Stipulated Order, Exhibit 9, ¶ D, p 1)

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[3] Intervenors' judicial estoppel argument thus falls flat. The Court is barred from unilaterally modifying the Consent Judgment because EGLE and Gelman anticipated and addressed in the Consent Judgment the very "changed circumstances" Intervenors now claim justify such a modification. *Vanguards*, 23 F3d at 1018; *Rufo*, 502 US at 385. This argument is entirely consistent with Gelman's 2007 argument that the Court did have jurisdiction to modify the Consent Judgment at that time, in response to Gelman's properly supported motion based on *unanticipated* changed circumstances and mistake of fact. The Court should therefore reject Intervenors' judicial estoppel argument, which would require Intervenors to show that Gelman took "wholly inconsistent" positions, a high burden unsupported by the facts at bar. *Spohn v Van Dyke Public Schools*, 296 Mich App 470, 480 (2012); *Pashke v Retool Industries*, 445 Mich 502, 510 (1994).

("By their signatures on the Third Amendment, the Parties stipulate and agree to its entry by the Court"). In fact, the 2011 Order provides that to the extent the Court's previous remedial orders are inconsistent with the Third Amendment, the agreed-upon Third Amendment "shall govern." (*Id.* ¶ 1, p 2). The 2011 Stipulated Order thus provides no support for Intervenors' request that this Court unilaterally modify or supplement the Consent Judgment.

II. Gelman's due process argument is properly raised as part of its Reconsideration Motion.

Intervenors have no basis or standing to demand that the Court unilaterally modify the Consent Judgment where Intervenors have not filed complaints or had those complaints tested through motion practice.[4] Their right to participate in litigation over the proper Consent Judgment requirements has not been established (and in fact, allowing them to do so is contrary to the Court's own intervention orders). Indeed, it is fundamentally unfair to Gelman to require it to defend against a remedy hearing before its liability to Intervenors has been established. Intervenors argue that this due process argument is premature. Gelman disagrees.

The term "due process of law" when used in relation to judicial proceedings is not nearly so limited as Intervenors would suggest—rather, it is "intended to secure to the citizen the right to a trial according to the forms of law of the questions of his liability and responsibility, before his person or his property shall be condemned." *Chrysler Corp v Appeal Board of Michigan Unemployment Compensation Commission*, 301 Mich 351, 357-58; 3 NW 302 (1942) (quotation marks and citation omitted). Due process is a flexible concept applied to any adjudication of important rights. *Thomas v Pogats*, 249 Mich App 718, 724, 644 NW2d 59 (2002). The procedural protections it affords, including fundamental fairness, are based on what the individual situation

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[4] For this reason, Intervenors' choice to style their opposition as "Intervening *Plaintiffs'* Brief in Opposition" is particularly misleading. No fewer than three times, Intervenors refer to their filings as "draft complaints."

demands. *Id.* "Due process requires that there be an opportunity to present every available defense," *Lindsey v Normet*, 405 US 56, 66 (1972) (quotations omitted. Here, due process certainly requires more than notifying Gelman when the hearing will take place as Intervenors contend; it requires that Gelman have the opportunity to contest the very claims that would, if they were to survive, frame Intervenors' preferred remedy.

In an attempt to avoid the obvious due process flaws their argument creates, Intervenors have the temerity to suggest that this Court need not even address their claims in order to modify the Consent Judgment as they request, and that there is no need for them to even file the complaints that purport to establish the basis for their supposed right to injunctive relief. Intervenors claim the Court can address their claims for relief—including injunctive relief—in subsequent proceedings, after the Court's proposed remedy hearing (Intervenor Opp'n, p. 11). But the Intervenors' asserted basis for their intervention in this proceeding was to obtain injunctive relief *with respect to the remediation*. If the Intervenors' claims are not relevant to determining the appropriate remedy, then Intervenors had no right or basis to seek intervention in the first place. As such, if Intervenors are to remain in this proceeding at all, the Court's Intervention Orders require them to file and defend their complaints, just like any other litigant must. Ordering a remedy hearing before Intervenors' rights to any such legal remedy have been established violates Gelman's due process rights to first be heard, and to defend itself, on the issue of liability. Gelman's due process arguments are thus not only timely, but essential.[5]

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[5] This Court should also not entertain Intervenors' spurious suggestion that Gelman did not timely file its reconsideration motion. (Intervenors' Opp'n, p 3 (claiming Gelman filed its reconsideration motion 49 days after the Court's original (and soon thereafter vacated) Scheduling Order). Each of the Court's three amended scheduling orders vacated the previous orders. Gelman filed its reconsideration motion within 21 days of the then-effective Third Amended Scheduling order (dated 12/17/2020). Gelman then refiled its Amended Motion for

III. If USEPA takes control over the Site, as Intervenors request, this Court will no longer have jurisdiction over EGLE's enforcement action, which will be dismissed.

Intervenors devote a large portion of their brief to disputing Gelman's claim that their demands that USEPA take control over the Site would ultimately divest this Court of jurisdiction over this matter. Intervenors' arguments appear to completely misunderstand Gelman's point: If USEPA were to take jurisdiction and control over the Site, EGLE's enforcement action would be dismissed and replaced by a federal enforcement action filed in federal court and any associated local control will be lost. Federal courts have exclusive jurisdiction over all controversies arising under CERCLA—the federal law by which Intervenors wish to have this remediation governed—and any consent decree or litigation between Gelman and USEPA must be entered or pursued in Federal Court. 42 USC § 9613(b). Indeed, USEPA's recently published "FAQs" regarding the Gelman site, which Intervenors attach to their brief, confirm that if USEPA takes the enforcement lead, it will negotiate "a federal consent decree" that by law must be entered in federal court. (FAQ No. 4, p 1, Exhibit 10).

Gelman never claimed that EGLE's enforcement action would be dismissed before USEPA decided whether to list the Site, as Intervenors appear to suggest. Nor did Gelman assert that other claims relating to the Site arising under state law could not, in the appropriate circumstances, be filed in state court. *See Atl Richfield Co v Christian*, 140 S Ct 1335, 1349, 206 L. Ed. 2d 516 (2020) (finding that CERCLA "deprives state courts of jurisdiction over claims brought under the Act," "[b]ut it does not displace state court jurisdiction over claims brought under other sources of law"). But if USEPA agrees to take the enforcement lead, as Intervenors have requested, the federal enforcement action will be pursued in federal court, not pursuant to the Consent Judgment

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Reconsideration on January 28, 2021, just days after entry of the Fourth Amended Scheduling Order (dated 1/25/21) vacated the previous scheduling order.

or in this Court—and any remedy will be dictated by, and subject to the sole approval of, the USEPA. *Id.* at 1355 (noting that landowners must "first obtain EPA approval for the remedial work they seek to carry out" to the extent it is inconsistent with the remedy ordered by USEPA). EGLE may have some limited role acting at USEPA's direction, but its enforcement action in this Court will be displaced.

Gelman's focus in raising this jurisdictional issue was considerations of judicial economy. It makes little sense for any party, or this Court, to continue to expend public, private, and judicial resources litigating the terms of a state court remedy when that remedy will become irrelevant if USEPA takes the enforcement lead. This course of action is particularly wasteful because EGLE and Gelman are willing to enter a fully protective bilateral Consent Judgment now—one that can remain in place while USEPA considers the site, and which would preserve Intervenors' rights to seek additional response activities if USEPA declines to take the Site and the case remains in this Court.

III. As the Court recognized, proceeding with the hearings contemplated by the Scheduling Order would risk putting the Court in an untenable position.

Intervenors inappropriately dismiss Gelman's concern that a court that will act as a trier of fact cannot involve itself in substantive settlement discussions that would compromise its ability to subsequently rule. (Intervenor Opp'n, pp 17-19). Gelman's concerns were based on the Court's own comments during the November status conference regarding wanting to understand the bases positions taken by the negotiating parties, suggesting that it might use the occasion of the hearing to attempt to refine the previously reached, but then rejected, compromises so that an acceptable solution might be achieved. Exhibit 11, Transcript of November 19, 2020 status conference, pp 38-39,45-49. Indeed, at the hearing on Gelman's Motion for Stay, the Court itself recognized that if it ruled on a remedy based on new cleanup criteria and the Court of Appeals subsequently ruled that the Court had no authority to do so, the trial court could not then go back to adjudicate the Intervenors' right to participate:

> You raise a very good point, Mr. Caldwell, that if I try to impose a remedy at this point without the due process of litigation of what right, if any, intervenors have in that, that would be error. If it came back, we'd have to get a whole []other judge.

Exhibit 12, Motion for Stay Transcript, at p 5, lines 20-24. As discussed at length in Gelman's motion, the Court was correct when it expressed this concern. *See, e.g.*, *Crampton v Michigan Dept of State*, 395 Mich 347, 351, 353-355; 235 NW2d 352 (1975); *Okrie v State of Michigan*, 306 Mich App 445, 471-472; 857 NW2d 234 (2014); *Bayati v Bayati*, 264 Mich App 595, 603; 691 NW2d 812 (2004); MCR 2.003(C)(1)(b), (c). Gelman stands by its position that, by proceeding with the hearing as contemplated in the Scheduling Order, the Court risks putting itself in an untenable position with respect to any future merits proceeding.

CONCLUSION

The Intervenors—no fewer than seventeen times—refer to the "cleanup regime" and concede that the "hearing set by the Scheduling Order was . . . to potential[ly modify] the existing cleanup regime." (Intervenor Opp'n at 3). This is a creative euphemism, as here, the "cleanup" is governed by a Consent Judgment. The Intervenors fabricated this euphemism because even they recognize that they cannot admit that the hearing set by the Scheduling Order was precisely so that this Court—unilaterally and *sua sponte*, with no motion from any signatory to the Consent Judgment—could alter the terms of that existing, consensual agreement at the behest of entities who have not yet filed a complaint, whose intervention was by its terms limited to participating in negotiations, and whose very right to a legal remedy has not yet been adjudicated.

For the reasons outlined herein and in Gelman's motion for reconsideration, it would be palpable error for this Court to proceed with the proposed remedy hearing and unilaterally modify the Consent Judgment (or similarly issue a remediation order without consent of the parties) to incorporate a preferred remedy addressing the new cleanup criteria, particularly where, as here, the parties to that agreement have not sought any such relief and the entities pursuing modification are not parties to the litigation. Gelman respectfully asks this Court to reconsider and vacate its December 17, 2020 Scheduling Order and the subsequent amendments thereto, and either dismiss the interventions without prejudice and enter a bilateral agreement reached between Gelman and EGLE, or order Intervenors to file their complaints so that their claims can be litigated.

Respectfully Submitted:

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Dated: March 15, 2021

PROOF OF SERVICE

The undersigned certifies that a copy of the foregoing instrument was served upon all parties to the above cause to each of the attorneys of record herein at their respective addresses as directed on the pleadings on March 15, 2021 by:

☒ E-FILE ☐ US MAIL ☐ HAND DELIVERY ☐ UPS
☐ FEDERAL EXPRESS ☐ OTHER

/s/Brenda Ann Smith
Brenda Ann Smith

STATE OF MICHIGAN
IN THE CIRCUIT COURT FOR THE COUNTY OF WASHTENAW

STATE OF MICHIGAN, et al,
Plaintiff, Case No. 88-34734-CE
Hon. Timothy Connors
vs.

GELMAN SCIENCES
Defendants.
_________________________/

VIDEOTAPED PROCEEDINGS

Motion for Reconsideration
BEFORE THE HONORABLE TIMOTHY P. CONNORS
Ann Arbor, Michigan - March 22, 2021

TRANSCRIBED BY: Kimberly H. Callahan-Golden, CSR - 7995
Core Litigation Support, LLC

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Also present, Raymond Ludwiszewski

Monday, March 22, 2021
Ann Arbor, Michigan
9:01 a.m.

* * *

THE CLERK: Now on record. Frank Kelly vs. Gelman Sciences, Case No. 88-34734-CE.

THE COURT: Good morning. This is Judge Connors. I wanted to tell you procedurally how we're going to proceed this morning. I have a child welfare case also set, and I'm going to bring them in and tell them to -- let me say this: I have read the briefs. I've gone over them. I'm familiar with it. So I really think your oral argument on the motion for reconsideration should really hit the high points.

I was thinking if I tell the NA case to come back by 10:30 -- because I will make a decision on the motion for reconsideration -- do you think that's a realistic time or should I tell them to come back later? What I don't want to do is have all those people waiting around.

MR. CALDWELL: That sounds very realistic to me, your Honor. And again, this is Mike Caldwell on behalf of Gelman Sciences.

MR. POSTEMA: Yes, Judge, I think that's

reasonable. Stephen Postema, City of Ann Arbor.

THE COURT: Okay. All right. So you all can go get a cup of coffee real fast while we kick you out and bring you back in just a second, okay? I'll talk to them.

MR. CALDWELL: Thank you, Judge.

THE COURT: You're welcome.

(Whereupon there was a recess from 9:03 a.m. to 9:16 a.m.)

THE CLERK: Now on the record. Frank Kelly vs. Gelman Sciences, Case No. 88-34734-CE.

THE COURT: Good morning. This is Judge Connors. Thank you for your patience as we worked on some logistical matters with the other case. If we could have appearances of the attorneys, please.

MR. CALDWELL: Your Honor, this is Mike Caldwell on behalf of Gelman Sciences. With me is Ray Ludwiszewski as well.

MR. POSTEMA: Your Honor, Stephen Postema on behalf of the City of Ann Arbor. I have with me today Abigail Elias. And from outside counsel, I have Fred Dindoffer and Nathan Dupes who will present the argument on that portion of the argument that Ann Arbor is presenting today. Good

morning.

MR. NEGELE: Good morning, your Honor. Brian Negele, assistant attorney general representing the Michigan Department of Environment, Great Lakes, and Energy.

MR. DAVIS: Good morning, your Honor. Robert Davis on behalf of the County intervenors.

MS. METTE: Good morning, your Honor. Erin Mette on behalf of the Huron River Watershed Council.

THE COURT: Mr. Stapleton? You're muted, sir. You're muted, Mr. Stapleton.

MR. STAPLETON: I'm sorry, Judge. William Stapleton for Scio Township.

THE COURT: Thank you.

Mr. Caldwell, this is your motion for reconsideration. As I said, I've read the briefs and given the intervenors an opportunity to respond to your motion, as well as set it for oral argument. So go right ahead, sir.

MR. CALDWELL: Thank you, your Honor. I will try to just hit the highlights because I know the Court has read these briefs thoroughly.

Your Honor, quite simply, we don't believe that there's any procedural legal basis for

the proposed remedy hearing. As we discussed in our briefs, the Court cannot modify a consent judgment without the consent of the parties absent a mutual mistake of fact or an unanticipated change of circumstances. And that change of circumstances would be to render the compliance with the consent judgment, but significantly more difficult. And here there is no mutual mistake of fact or an unanticipated change of circumstance.

And importantly, neither party to the consent judgment has sought relief from this court. Gelman and EGLE fully anticipated the change of circumstances that intervenors claim justifies the proposed remedy hearing, and that's the change in the cleanup criteria. Not only did we fully anticipate that change, we accommodated that change to the consent judgment itself when we stipulated to the third amended consent judgment.

And, you know, this was not a surprise, your Honor. As we point out in our reply, the EPA modified its toxicological review of dioxane in 2000 -- the filed version was published in August of 2010. The draft version was published in March of 2009. So throughout the negotiations of the third amended consent judgment, EGLE insisted, and

understandably so, that the reopener provision of the CJ be modified to include the adoption of more restrictive cleanup criteria, and we agreed to that. And so this is not some anticipated -- unanticipated change of circumstance. This is something that was fully accommodated in the terms of the consent judgment. And under the specific terms of the consent judgment, it can only be modified by consent of the parties or this circumstance, if the State were to seek to reopen the consent judgment. And that -- and that, then, opens up litigation, not actually a modification of the CJ itself.

So there really is no basis for the proposed remedy hearing, which is intended to develop a new -- from the intervenors' point of view, a new cleanup regime, and they have alleged that we are somehow judicially stopped from arguing otherwise. But in 2007, the brief that they point to, we were actually seeking relief from the existing requirements of the CJ, because a completely unanticipated change of circumstance had rendered our compliance with the existing terms of the consent judge impossible. And that was brought to the Court by motion of Gelman, and the two parties, you know, litigated that issue. Ultimately, the Court didn't issue an order in response to that. The parties eventually agreed to modify the consent judgment and to accommodate -- to address that changed circumstance.

And the intervenors argue that this and other orders issued by the court somehow justify the proposed remedy hearing either to unilaterally modify the consent judgment on a sua sponte basis or in order to issue some supplemental remedial order. And that's just not the case, your Honor. None of the orders that -- and the ones we're talking about were issued by Judge Shelton. None of those constitutes a nonconsensual modification of the consent judgment. Rather, those orders all rose from proceedings initiated by one or both of the parties to resolve disputes about what the existing terms of the consent judgment said and/or what the existing terms of the consent judgment required.

These were not modifications. These were interpretations and applications, and to use the word that they repeatedly -- the intervenors repeatedly use, enforcement of the existing consent judgment. And I can get into the details; I will

spare you that. Unless the Court has any questions, I'm happy to ask -- answer any questions you have about the past proceedings because, you know, I was there and the intervenors were not, and that may explain why they so dramatically misrepresented what happened back then.

But I think the intervenors know that they're wrong on this, because rather than asking the Court to modify the consent judgment using that phrase, they say the Court should go ahead with the remedy hearing so that it can modify the existing cleanup regime. Your Honor, there is no cleanup regime separate or independent from the terms of the consent judgment.

And if I could just quote something that the State said in the 2000 motion to enforce, which is just completely accurate, consistent with the law of consent judgment, in this case the remedial action is to be performed according to the specific terms of the consent judgment and plans approved under the consent judgment. And based on the law that we've cited and basic principles of law, those terms can't be modified by the Court absent the consent of the parties under the terms of the CJ itself or in response to a motion by one or both of

the parties for relief from the requirements of the existing terms of the consent judgment in response to an unanticipated change of circumstance, which is not present here.

And then very briefly, your Honor, we've argued in our motion for reconsideration and did not get any -- did not get any persuasive response in response to -- from the intervenors. But due process -- I mean, the point of this hearing is to allow the intervenors to weigh in on what relief they think is appropriate based on what they've sought in their complaints, which have not been filed.

And, you know, just as a matter of basic due process, Gelman is entitled to defend those claims, present its defense, which, as we outlined in our initial brief, are substantive. This is not a stalling tactic, or -- we have legitimate defenses to the claims of the intervenors, particularly the claims for injunctive relief that justify their involvement in this case in the first place. Now, intervenors, again, I think know this as well, and --

THE COURT: Mr. Caldwell?

MR. CALDWELL: Yep.

THE COURT: I'm sorry to interrupt you. I don't know who the individual is, but this is a Zoom hearing; somebody is chatting comments to everybody. Please don't do that. This is part of my court record, and if this were to happen in the courtroom, I'd have to ask you to leave. So please, people, I don't care what you say when it's off the record on the hearing, you're free to do that, but please don't be doing that during the hearing.

Go ahead, Mr. Caldwell.

MR. CALDWELL: Thank you, your Honor.

I think the intervenors know that due process -- this proposed hearing is not consistent with basic principles of due process. And again, the hearing is set by a scheduling order, but that scheduling order has to be read in context with the Court's statements in the November 2020 status conference. The purpose is clearly to establish a new remedy. And so intervenors are really forced to take the -- kind of an incredible position that the proposed remedy hearing in determining what modifications to the consent judgment, or in their phrase, the existing cleanup regime, should be made to go forward before -- not only before their complaints are even filed, but before we even get to issues about what relief they might be entitled to. Well, your Honor, it's those claims set forth in their draft complaints that they provided to the Court with their motions to intervene, particularly their claims for injunctive relief, that form the basis for why they're entitled to intervene in this case in the first place.

So if intervenors don't think that their claims need to be adjudicated before a remedy is determined, then they should be dismissed from this case. Their intervention should be dismissed, and they should pursue those claims independent, as the City of Ann Arbor did back in 2004. They have no right whatsoever to participate in the determination of a remedy until, and if, their claims are filed and this Court finds that Gelman is somehow liable to the intervenors.

And so just as a matter of basic due process, we're entitled to that sequence of events and the remedy hearing in this situation would be in gross violation of those basic due process rights. And then, very briefly, your Honor, the issue with regard to EPA, our point in pointing out that -- I mean, first of all, there's no question

that this Court will lose jurisdiction over the case if EPA takes jurisdiction over the cleanup. Everything the EPA does is done in federal court; federal courts have exclusive jurisdiction over CERCLA. CERCLA is the statute that the community seems to think will provide a better remedy. So that's all going to happen in federal court. There's maybe some misunderstanding about when this Court will lose jurisdiction. But once the EPA decides to take this case over, this Court will lose jurisdiction and that will move to federal court.

And our point in -- and there is some time in which the state court consent judgment will stay in effect, you know, while they're deciding whether they should list the site, for instance. But it makes -- and our point in making this argument is it makes no sense to us why anybody, including the intervenors, would want to continue to litigate what remedy might be available under state law, while they're pursuing a federal remedy, because everything we do may be rendered irrelevant. And from Gelman's point of view, if this Court were to go through the proper steps and find that intervenors are entitled to some relief,

that relief -- under state law, that relief might be different, inconsistent with what EPA determines, and we could be in a position where we're implementing one cleanup that gets overruled by the federal authorities.

And so what we think makes the most sense is -- because we've been ready, willing, and trying our darndest to enter into a protective consent judgment with the State of Michigan. We think that we could quickly come to a third agreement with the State of Michigan if we're allowed to enter a bilateral CJ modification, and then dismiss or even stay the intervention while the EPA deliberates on whether they're going to take the site.

Otherwise, we're just wasting a tremendous amount of judicial, public, and private resources litigating over a remedy, that's ultimately, if they do take the case, is going to become irrelevant. So that was our point in making that argument, your Honor.

And then finally, as the Court recognized in our last hearing, if this Court were to proceed with the remedy hearing, determine what remedy, what relief the intervenors might be entitled to before they file their complaints or engage in any

back-and-forth mediation efforts, and this went up to the court of appeals and the court of appeals remanded it with instructions to go through what we believe are the proper procedural steps in terms of filing their complaint, adjudicating our defenses, and making a determination of liability, if that were to happen, we don't see how the trial court could continue to serve as the judge, and I think the Court recognized that.

I mean, you know, every judicial summary conference I've ever been in, you know, the first question of the judge is: Is this a jury trial? Because if it's a jury trial, the jury is going to be the finder of fact and then the judge can get involved in the settlement discussion. Conversely, if you have a case where the claims are equitable in nature and the judge is going to be the finder of fact, there's not going to be a jury, then that settlement conference gets spun off to another judge.

I just recently had that with Judge Kuhnke, and I have an equitable claim in that case, and we were sent to Judge Collins judicial attorney, Ms. Roberts, for our settlement conference. I mean, that's basic judge rule of stuff, I believe, your Honor. And we think proceeding with the remedy hearing as proposed would really put this Court in a tough situation if the court -- if the case came back and the Court had to make those due process decisions that we've talked about.

So I mean, that's basically our argument, your Honor. And we fully responded to the intervenors' brief in opposition in our reply brief. If there's any questions you have, your Honor, I'm happy to answer them.

THE COURT: Mr. Caldwell.

On behalf of the State.

MR. NEGELE: Your Honor, we're, you know, consistent with our last filing. We're not taking a position on either intervenors' points or on Gelman's pleadings.

THE COURT: Thank you, sir.

Intervenors.

MR. POSTEMA: Yes, Judge. As I've indicated -- Stephen Postema -- we've divvied up the argument so as to not be repetitive. And if we may, we'll -- the intervenors' attorneys have agreed to an order and I'll let them start in the order that they've agreed to on the argument to

make it as concise as possible for you. Thank you.

MR. DUPES: Thank you, Stephen.

Nathan Dupes on behalf of the City of Ann Arbor, Judge. As Mr. Postema just mentioned, there's several issues here and the intervenors have kind of divvied up responsibility to present the oral presentation today. So there's five issues, your Honor. There's this Court's authority to modify the current cleanup orders. There's due process. I'll be handling those first two arguments, your Honor. There's the EPA listing issue, and judicial disqualification. Mr. Stapleton will be addressing those two issues. And finally, there's the statutory and public health code arguments, and Mr. Davis will speak to those.

So first, your Honor, I wanted to make a few overarching points that really should permeate all of the arguments you hear today. And No. 1 is in 2016 there was a watershed moment in this case when the State of Michigan issued a finding of emergency, finding that releases of 1,4-dioxane posed a threat to public health, safety and welfare, and that the current cleanup criteria for 1,4-dioxane were not protective of public health.

I just heard Mr. Caldwell talk at length about how the parties, somehow back in 2011, had fully anticipated changes in cleanup criteria, yet in 2016 you had the State of Michigan take the drastic step of issuing that finding of emergency, and reducing the cleanup criteria that had been in place for years by more than an order of magnitude, Judge, going from 85 parts per billion down to 7.2.

The second overarching point, your Honor, is I don't think any of us have seen this much briefing, argument, or handwringing over a scheduling order for a hearing at which the party who is opposing the process will have every opportunity to make whatever arguments it sees fit, to respond to the arguments of the opposing side, to be able to bring in its experts, submit expert reports, and have multiple days of hearing before the Court enters any substantive ruling.

And thirdly, Judge, if Gelman is so confident that what it may present at some future date between itself and the State is fully protective of public health and environment, then it begs the question why it's so vigorously resisting a hearing at which it'll have the opportunity to explain to your Honor why that's the

case and why the proposed modifications and enhancements that the intervenors would like to offer are not supported by the law or the science. So Judge, we're going to mostly address Gelman's reply brief because, of course, we fully briefed in opposition the arguments in the motion for reconsideration.

But Gelman's reply was remarkable because it significantly walked back or changed the arguments it had made in its motion for reconsideration. Now, for example, as Mr. Stapleton will explain in a moment, Gelman has all but given up the argument that EPA process is somehow an impediment to this court proceeding. And Gelman offered zero argument in the way of addressing the statutory mandate that the Washtenaw County entities have to protect public health. Gelman offered no response for that, despite filing an almost 20-page reply brief.

But moving on to the main -- one of the main issues here, your Honor, which is the authority of this Court to modify the cleanup orders. And at first, your Honor, the reason that the intervenors use the phrase, "cleanup regime," which Mr. Caldwell seems to want to belittle, is because this case is not simply a contract about a contract between two parties that the Court is being asked to enforce. This case is a wide-ranging, decades-long, public health cleanup case, and the cleanup regime is actually several judgments and orders. It's not simply a two-party consent judgment that was entered with the consent of both parties.

We pointed out to the Court that there's numerous orders this Court has entered to supplement. And not only to supplement, to drastically change the affect, the requirements, the obligations set out in the consent judgment. And those changes that the Court implemented, again, were not the product of EGLE and Gelman signing on the dotted line at the bottom of an amendment to the consent judgment. They were the product of the same type of hearing, your Honor anticipated, before this motion for reconsideration was filed.

There were briefs filed. There was oral argument. There were witnesses in fact were brought in. There was expert testimony. And the Court heard all of that and said based on the current objectives, as set out in the consent

judgment and based on what the parties are telling me, certain things need to be changed. So as Mr. Caldwell will tell it, it's simply the Court stepping in to enforce the plain language of the consent judgment. Again, going back to his argument that this is simply a contract between two parties. That's simply not the case.

This Court's remediation and enforcement order ordered Gelman to bring everything within compliance in less than five years. The Court ordered Gelman to install additional extraction wells. The Court ordered Gelman to install additional monitoring wells. None of that was required by the then-existing consent judgment.

So after citing these examples from the actual case history, your Honor, Gelman essentially gave up that argument in its reply brief. So now we hear Mr. Caldwell arguing that well, Judge, yes, the Court does have the authority to make those changes and to -- has all the inherent equitable powers to enforce its own directives, which, of course, you have, your Honor. Now, the argument is well, in those situations, it was either Gelman or a party that was the asking for the Court's assistance, and here the intervenors have become

involved, so somehow that's different. Well, your Honor, again, Gelman is off base. In the examples that we've given you, the remediation enforcement order, the Unit E order, being the primary ones, the Court then simply enforced the existing terms or awarded stipulated penalties because EGLE thought that Gelman had violated the existing terms.

The Court fundamentally changed the nature of the obligations that Gelman had to meet. And in one of those orders, your Honor, over the objection of the State, this Court issued the Prohibition Zone order, which was a huge departure from -- and again, in our words -- the cleanup regime at the time, and has now become a central feature of the Gelman site. That was not -- the Prohibition Zone was not entered because the parties submitted a modification to consent judgment.

The Prohibition Zone order was entered because the parties had discovered that the contamination had migrated to a different aquifer and something had to be done. There were changed circumstances. So Mr. Caldwell says, well, Judge, that's not an appropriate example here because

there are no changed circumstances. But we would submit that the 2016 finding of emergency and the Court's -- excuse me, the State's emergency change of the rules from the cleanup criteria that existed to more than an order of magnitude lower, are the exact kinds of changed circumstances that warrant action today.

And, in fact, Gelman and the State agreed. There's no dispute in this case. And even now in oral argument you haven't heard from Mr. Caldwell argue that the current set of cleanup orders and judgments can remain, and the status quo can remain. In fact, he's telling you that the State and Gelman are prepared to submit something to the Court to do just that, to reflect the current cleanup criteria.

So if we all agree that something needs to be changed in the Court's existing orders and directives, then all the more reason for the Court to hold the hearing that is scheduled, so that each of the parties -- Gelman, EGLE, the intervenors -- can present the law and the science that support what those changes should be. That's exactly the hearing that your Honor's order envisioned. And just getting back, your Honor, real quickly to the standard and what the Court -- the powers that the Court has, you'll recall in our brief we pointed out that Gelman itself has recognized that a consent judgment or decree may be modified where, quote, enforcement of the decree without modification would be detrimental to the public interest. And that's the Vanguards of Cleveland case that Gelman cited previously in briefing before this Court.

So enforcement of the current cleanup regime, in light of the drastic change in cleanup criteria, would be contrary to public interest, your Honor. And as both EGLE and Gelman have argued previously to this Court, this Court has all the inherent equitable powers to enter any order proper necessary to effectuate it's existing orders and objectives.

And as for Gelman's arguments about, you know, why the intervenors are even involved in the first place, your Honor heard all of those arguments, you recall, four years ago. Gelman strenuously objected to this Court allowing the intervenors any seat at the table or any part in the process. Your Honor heard those arguments. Your Honor properly rejected those arguments.

Gelman took those arguments to the Court of Appeals, took those arguments to the Michigan Supreme Court; he wasn't able to convince either of those courts that they merited interlocutory review. And now we're back with the same types of arguments. And your Honor made the very prudent decision that having the intervenors in this case for the purpose of providing input on how to address the State's 2016 emergency finding was the appropriate course.

As your Honor recall, up to that point, the State and Gelman had been negotiating an amendment to the consent judgment with very little public insight into the process, no participation from the intervenors. And that's the whole point of our motion for intervention in the first place, was it's appropriate to have the public values and public interest group get a seat at the table because they represent many constituents.

This is not simply a two-party contract. This is series of orders and judgments that have a significant impact on the public and certainly appropriate for those public entities to have a say in the process. So, your Honor, we're at the same place in this case as we were when you allowed us

to intervene. And the same concerns, which really animated your decision allowing intervention, should continue to direct how this Court handles the intervenors. And finally, your Honor, the last issue I'm going to address briefly is due process, and then I'll turn it over to Mr. Stapleton.

So, your Honor, these same considerations I just discussed really undermine Gelman's due process argument. Everyone agrees that the existing set of orders and judgments need to be changed to reflect the current cleanup criteria. And if that's the case, then really this issue of intervenors' claims or Gelman's defenses to intervenors' claims is totally irrelevant.

I mean, this Court, at the hearing as scheduled, gave no indication that it intended to order final relief to the intervenors, which relief includes things as varied as response activity costs and damages for common law tort claims. None of that is going to be litigated or needs to be litigated at the hearing that your Honor envisioned. The purpose of this hearing was limited, okay? It was to address the procedure for the modifications to the existing set of orders and regimes. And as for the process, in terms of what

process is appropriate for Gelman to have before any modifications ordered, your Honor already went above and beyond what the due process clause requires. You provided Galvin with notice, an opportunity to submit briefs, Nexus reports responding to the intervenors' arguments, and multiple days of hearings.

And finally, your Honor, and perhaps most importantly, Gelman's arguments aren't right for review, okay? The due process clause requires process before a deprivation of life, liberty or property. What we're talking about today is a scheduling order so the parties can present argument. The Court has made no substantive ruling, so Gelman's due process arguments, if they have any merit at all, are premature.

And with that, unless your Honor has questions for -- on my issues, I'll turn it over to Mr. Stapleton.

THE COURT: Thank you very much.

Mr. Stapleton.

MR. STAPLETON: Thank you, your Honor. Your Honor, I just want to make one thing really clear up front. The EPA petitions that haven been filed by the intervenors and discussions with EPA that are currently going on, have no impact whatsoever on the proceedings in this Court. And we know this because last Thursday there was a public meeting with EPA officials and EGLE officials, and everyone agreed on this point at that meeting, Judge. The EPA officials said repeatedly that its lengthy evaluation process would have no effect on the Court proceedings, and that this evaluation process and the Court case could proceed on parallel tracks. And Judge, this is exactly what the intervenors are doing.

I mean, we understand that the EPA process is a very lengthy process. We also understand that it's very uncertain. We don't know if the EPA is going to take this case. And that's why it is so critical that this Court enter a cleanup order that incorporates the new cleanup standards and that is protective -- as protective as possible of the environment.

During the Thursday meeting, Judge, the EPA officials made it pretty clear that even if they decided to take the site, it could take up to ten years to reach a record of decision, and which would be the remediable action plan for the site. So as I said, a very lengthy process. And so what

Gelman's argument boils down to is because there is a chance, you know, ten years from now, that a federal consent decree might be entered, that this Court should not hold a hearing and let intervenors provide the Court with its ideas on what should be in the cleanup, but this Court should not hold that hearing because -- simply because of the possibility of EPA involvement in this case.

You know, Judge, obviously this argument doesn't make any sense. And, you know, it's really just another attempt by Gelman to prevent intervenors from providing this Court with the law and the science and the basis for what intervenors believe needs to be in this cleanup order.

And, Judge, I want to make just a couple of points about the possible entry of a federal consent decree. You know, let's be clear about something. The federal government, if they decide to take this case years from now, they aren't going to come into this site, where remediation activities have been going on for 30 years and throw everything out. There's an extensive monitoring network. There are many extraction wells. And these will all remain in place.

If the federal government were to get

involved, what is likely to happen is that it would incorporate the terms of this Court's order in a federal consent decree and likely supplement it with additional monitoring perhaps, additional extraction wells. So, you know, Gelman's argument that this is a -- this process is a waste of judicial resources is simply not true. I mean, this -- the proceeding that the Court has contemplated will result in a cleanup order that's going to be in effect and control the remedial activities at this site, your Honor, I would say for at least ten years and maybe longer.

You know, the second point about the federal -- about the potential federal consent decree is, you know, even if EPA were to become the lead enforcement agency, this Court does not lose jurisdiction over the intervenor claims or the EGLE claims. And this was -- you know, we cited the recent Supreme Court decision in our brief, Judge, and, you know, it was the Atlantic Richfield case. And the point of that case is that federal courts don't have exclusive jurisdiction over actions that may relate to superfund sites that arise under state law. It's clear -- it's clear law. It's recently been held by the United States Supreme

Court. So, you know, this Court -- the point, Judge, is this Court does not lose or get stripped of its jurisdiction, even if a federal consent decree were to be in place. So, you know, Judge, the bottom line on the EPA issue is, you know, it's really a nonissue. There is nothing going on right now with the EPA that has any effect at all on this Court's process and this Court's jurisdiction to enter an effective cleanup order for this site.

Judge, just briefly on the potential disqualification issue. I guess intervenors don't really understand the issue here. What we understood was the judge was contemplating a hearing where you would be trier of fact, you would listen to the law and science from the parties, and you would make a decision about -- about what needs to be in a cleanup order.

So, you know, even if that were to go up to the Court of Appeals and come back, I guess intervenors don't understand, you know, the potential disqualification issue. And that's why, you know, we cited some law in our brief that, you know, judges -- you know judges can get involved in settlement discussions if it's in the course of the court's normal judicial duties, without -- you know, without becoming disqualified. You know, certainly there have been lengthy settlement negotiations in this case and there's the potential that, you know, that -- you know, some of that would come out in a hearing that the Court would hold. And I guess our point is even if that were to occur, Judge, we don't think that's a basis for disqualification. Our understanding is, you know, that the Court is not proposing to the parties that it serve as a mediator. It's simply proposing a hearing in which it would serve as the trier of fact.

And so unless your Honor has any questions with that, I would turn it over to Mr. Davis, who would like to address the special role of Washtenaw County.

THE COURT: Thank you, Mr. Stapleton.

Mr. Davis.

MR. DAVIS: Good morning, your Honor, and thank you for the opportunity. Your Honor, I -- generally when I put my appearances on before you, I represent myself as the attorney for the County. And I am the attorney for the County and the County as a whole endorsed as our brief -- participated in the brief, and wholly endorses the arguments that

we've made this morning. But I want to put on my hat as the attorney for the County Health Department and the County Health Officer, Judge, which I did argue before you at the time we were arguing for intervention. The jurisdiction of the health department starts with the state Enabling Law and then the law at MCL 333.2428 creates the role of the health officer as a separate legal entity. So I am two entities, Judge, that are before you by statute.

And the public health code, which operates as the Enabling Act, creates a statutory duty to protect the people of Washtenaw County, as a whole, against health hazards. And all of this I laid out for you, Judge, is confirmed by the Michigan Supreme Court in the McNeil vs. Charlevoix County decision, 484 Mich. 69 in 2009. So when we delve into the statutory duty of my two entities with the 2016 emergency declarations by the State of Michigan, the State of Michigan declared in 2016 new, quote, cleanup criteria for 1,4-dioxane, Judge.

They didn't set cleanup or arbitrary levels, they didn't fail to define what the levels were supposed to be, Judge. They set them as

cleanup criteria, and they declared a threat to the public health and they declared that the existing criteria was no longer protective of public health. So the county health department and its director, its health officer, intervened, petitioned for intervention before you, Judge, not necessarily as an intervening local unit of government, but two entities that have separate and unique statutory duties.

They are entities that are compelled by state law and the rulings of the Michigan Supreme Court to be involved here. They're compelled by a statute, Judge, to be involved in the declared state MDEQ cleanup emergency. My two entities must ensure that the implementation of the new cleanup criteria is implemented in a fair and reasonable way to protect the public health of all persons in Washtenaw County.

We can't ignore that statutory enabling rule, Judge, and it can't be overlooked as we to head towards getting rulings from your Honor. All of the mandates imposed upon the health department and the public health officer are by shall standards, and we are well aware of what shall means in statutory construction.

So Judge, I am adopting in full the arguments made on behalf of the county as a whole. But I want to point out to you the unique role that my health department plays and the unique role that my health officer plays, I have to be involved in the implementation of this new criteria. The public health code even envisions that I will work cooperatively with other agencies, such EGLE, but I have to be at the table for the implementation to address this public health concern.

And with that, Judge, I know you're glad that I'm done. And I thank you for your time.

THE COURT: Mr. Davis, I always enjoy you in front of me.

MR. DAVIS: Thank you, judge.

MR. POSTEMA: Judge, I think that's all from the intervenors at this point.

THE COURT: All right. Mr. Caldwell, this is your motion and you are entitled to a rebuttal agreement.

MR. CALDWELL: Very briefly, your Honor, because I think we addressed all of those arguments in the -- in our reply. Other than the County's argument, which I -- frankly I understand to be their argument as to why they were allowed to intervene, and I -- but I don't understand what that -- those arguments have to do with the propriety of the proposed remedy hearing before any of those claims set forth in the County's brief have been adjudicated before our defenses to those claims have been reviewed and adjudicated. And I would also point out that for all of Mr. Davis's reliance on mandatory language, this case was over 30 years old before the County sought to intervene in the first place.

But with regard to this emergency order that after four years they decided to bring up again, that emergency order -- excuse me. The emergency rule was as we explained back when this -- and I'll remind the Court when we were briefing the interventions, that was an administrative tool that the State used in order to get more restrictive cleanup criteria in place by the end of the year 2016, which the then-Governor Snyder had promised the mayor of Ann Arbor he would do.

Prior to that, the larger administrative rules package failed to get past the legislature, and so suddenly the State was left with two options. One, they could pass a specific rule for Dioxane, which is what they ultimately did. And

which, ironically, I was discussing with Mr. Negele at the time of whether -- basically expressing to him that if he thought that that was a viable method to get the more restrictive criteria in place, we would discuss that with our client, who would very likely stipulate to the extent we had to, or not oppose that effort. And so -- and I would explain this in the context of the assertion that we somehow didn't anticipate the change in cleanup criteria because the State issued this now-rescinded emergency rule.

We were -- Mr. Negele and I were talking about adopting -- the possibility of a Dioxane-specific rule being adopted because we were so far along in negotiating the consent judgment, we were faced with the possibility that we would have a consent judgment incorporating the more restrictive criteria, ready to be put in place and presented to this Court for entry before the State changed the rule and changed the cleanup standards.

So we certainly fully anticipated the change in cleanup standards, and this emergency rule, which, again, was not a -- was nothing more than an administrative tool to get to an end that we didn't really oppose. With regard to the

assertion that the previous orders dramatically changed the CJ is simply not true. The remediation enforcement order entered in 2000, Mr. Dupes -- the intervenors claim that that changed what the CJ required. In fact, the State of Michigan argued that those things were required, that we had to up our extraction to 800 parts -- 800 gallons a minute. Those things were required by the existing terms of the CJ and the quote that I read earlier from the State's motion attest to that.

With regard to the Unit E and the 2004 decision, I would like to read -- it's ironic that the intervenors put such weight on the Unit E decision, the Unit E order, when the order -- if I can -- on page 14 of the Unit E order, and I would love everybody to take a look at this.

Judge Shelton says, quote, it is not the role of this Court to devise or fashion remedies for the spread of -- spread of pollution in this Unit E Aquifer. It is the role of this Court to enforce the consent judgment and make sure whatever remedy is implemented conforms to that judgment and to the pollution statutes of the city.

So by its very terms, the Unit E order was not to modify, not to impose a -- a judicially

crafted remedy on the parties. It was to enforce the existing terms of the consent judgment, which is -- and in this case, there is no allegation that we were in violation of the consent judgment, as there was in the previous two examples I just discussed. There's no -- there's no debate that the existing consent judgment requires us to do something that we're not doing.

And there is no unanticipated change that has occurred that would give this Court authority to modify the consent judgment in response to a motion filed by one of the parties. Those past situations simply do not serve as precedence for a unilateral sua sponte judicial modification of the consent judgment.

And, you know, your Honor, even if those orders could be construed as modifying the terms of the existing consent judgment and we didn't appeal that, so what? I mean, the Unit E order, we had a debate about what the -- you know, whether the western system requirements applied to the Unit E, whether -- what remedy would be appropriate under those terms, if they did. And the judge, in large part, accepted our interpretation of those requirements. And so there was no reason for us to appeal that, even if it could be construed as a modification. And as Judge Shelton pointed out, I mean, I think we all agree that Judge Shelton was not hesitant to express the outer boundaries of his authority, and he recognizes it. It's not the role of the Court to fashion or devise remedy; it's to enforce the terms of the consent judgment. And so I don't think that those are good examples of good support for what the intervenors are seeking here.

And then finally, I would point out with regard to the -- you know, whether this Court would lose the ability to adjudicate and preside over future merits, determinations, and proceedings, we cite the Crampton decision. And you know, as Mr. Stapleton pointed out, the purpose of this remedy hearing is for this Court to act as a fact finder and make a determination of what the appropriate remedy is. And we agree with that. That does seem to be the purpose of the proposed remedy hearing.

But if that went up to the Court of Appeals after the Court had served as fact finder, if the court of appeals says, you know, Gelman was right, there needs to be -- due process needed to be followed for whatever reason, we don't think

that you could possibly serve as -- that it is possible you would not be in a position to act as the judge for the intervenors' claims. And one of the situations where disqualification is appropriate is where a judge might have prejudged the case because of prior participation as an accuser, investigator, fact finder, or initial decision maker.

And I just don't see how -- and I think the Court recognizes this and expressed in the previous hearing, how you can make -- you could be that fact finder and initial decision maker with regard to the relief that the intervenors are seeking, and then if we had to go back and adjudicate whether their -- Gelman is in fact liable to the intervenors and whether they are entitled to any relief, I just don't know how that would work. So I think the Court's concern in that regard were well-founded.

And I think with that, your Honor, we've beaten this to death.

THE COURT: Thank you very much. This matter is before the court this morning on Gelman's motion for reconsideration. And after allowing briefing and response and oral argument, I find as

follows: First, I do appreciate Mr. Caldwell going back and reminding how long this litigation has been going. I'm the third judge on the case. I remember when it was being litigated in the motion days, sitting in motion practice in front of then-Judge Patrick Conlin and the litigation taking up all morning, the rest of us never even got to get heard on anything. So I'm very familiar with the extensive background of the litigation.

I'm also very, very sensitive to the huge cost that kind of litigation takes for everybody involved, for taxpayers, for the public. And when we have issues that continue to be litigated, going up and down to the Court of Appeals and coming back, frequently that results in not much progress on the underlying issue.

In terms of who can participate, I did allow discretionary intervention of the health department. I appreciate Mr. Davis's reminder that his really is of a statutory duty as opposed to simply a local government unit. I also thought it prudent to have the local government units of the City of Ann Arbor and of Scio Township because they're the most directly affected in terms of where the initial pollution took place. And I also

thought it prudent to have at least one environmental group, and I was very appreciative that they came in on the Watershed Council. I'm going back to this last point that Mr. Caldwell made about, you know, due process and ultimately maybe having to litigate, is somehow Gelman liable to these other intervenors? I'll simply say the issue is pollution of water. That's the issue. I mean, where that water, where that pollution spreads is really the issue. So it's not as of it's some independent tortuous act on these other parties. It's you put it into the water and it needs to be cleaned up.

So in terms of who can participate, I did do that. I'm glad I did. I feel very fortunate that we've had this time spent of these legal minds, scientific evidence, exchange of experts. I think it's been wonderful.

In terms of disqualification, I really don't care. And the reason I don't care is I'm the third judge on the case and I won't be the last. And in terms of those discussions, settlement discussions, I was very purposeful, simply to create a space and a place where there would be dialogue. You went off in separate rooms. I would meet with you in court in circle just to see how things are going. Is there anything I need to do? You need anything from me? At no time did I ever have specific discussions about that. And the attorneys were very careful about that as well. And I appreciated that because I knew at some point I might have to make those decisions.

I do want to say that I thought the attorneys worked hard during that time period, and I was disappointed when all of the -- all of the groups came in and had a proposed judgment. The decision makers that they represent decided they were not happy with that, and I was disappointed, and I'm very concerned about the litigation cost going forward. But the point is we don't have that; and therefore, who makes the decision? I have to.

This argument about due proses, we're already in the remedial stage. We're past all that. We're decades beyond litigation of whether or not Gelman polluted the water. And the issue, the significant issue, I still think the change, which requires the judicial oversight to make the decision when the parties cannot reach agreement, is the fact in 2016 the cleanup measure was not

protective of public health at 85 parts per billion, but instead, was recommended down to 7.2. That's an over 90-percent reduction in acceptable levels. So I do think we are in the stage where the only thing really to determine is exactly what I was proposing; that we set up a time, hearing, under oath, witnesses, legal argument, and I will make the finding of fact given this change of acceptable levels of what the cleanup program will be. And then that can go up for appellate review and whatever the Court of Appeals decides, including if they say, get a different judge, I can live with it. But we need to move forward. We need to have some decision made, and it's my responsibility to make that decision.

We'll set the new dates. Let me suggest to you, since I am -- will be your fact finder, that the most effective way for me is for each of you to approach -- and we all know when we brief -- you've done wonderful, extensive briefs. But we also know the written briefs are for multiple audiences. Obviously, one of those audiences is the fact finder. But a second audience, of course, is the Court of Appeals. You already have that for those arguments that go up. A third audience, of

course, is your client. A fourth audience is opposing counsel, and a fifth audience is the public. So I'm going to suggest to you I recognize you have to file those written briefs to satisfy multiple audiences. But in your presentation with me, three rhetorical questions at the beginning: What is it you want me to do specifically? I know the facts of the case.

Second, how can I do it legally? Court rule, statute, case law, and specific to the point, not string-on citations. And then third, why should I do it? And I'll hear the evidence, and at the end of it, I'll come back to you and say how does this evidence support what you want me to do, both legally and why I should? And that's how we'll conduct it. And then I'll make the call, the chips will fall where they may, you know, and I'll stay with the case as long as I'm entitled to stay with the case.

So I think we should work on a briefing schedule. I think we're ready to go. I think we got -- I was hopeful we could start this today. Do you need any more time, or can we just set the dates and we'll start trying it?

MR. CALDWELL: Your Honor, I think the

suspension, and frankly the indication on February 4th that the Court was very likely to reconsider the decision to have the hearing --

THE COURT: I'm sorry. Mr. Caldwell, you're right. The motion is denied. I should've said that. Now, I recognize that with this denial it starts your appellate review of my denial of that, but I'm moving forward. So I recognize you too would be on a parallel path of having to try the case in front of me while asking that it get overturned with the court of appeals. I'm not going to stay proceedings, but I am going to move forward.

So I'd like to try this as soon as possible for several reasons. It's been way too long, one. Two, we still can't get juries in here with the COVID. But once we do, we're so backed up, my time is going to be limited. So I've been doing the child welfare docket, but I'd really like to get to this.

MR. CALDWELL: Your Honor, just so I'm clear, is the purpose of the hearing to modify the existing consent judgment?

THE COURT: The purpose of the hearing is to hear what is the proposal for the cleanup; why;

how I can do it; why; and then I'm going to order it. And it will be an evidentiary hearing.

MR. POSTEMA: And Judge, just for clarification, we've already been down the path of getting our legal briefs and everything before, so from the intervenors standpoint, it did cut it off, but I think that they can speak better that the procedure you had prior -- excuse me, previously set out is already in place, you know, from our end, and we can go back to where we were, and I don't believe that it will take that long for us to finish this, nor do I believe that it will take that long for Gelman to do what you asked before.

THE COURT: I'm thinking if we could do it in April, that would be great.

MR. POSTEMA: Yep, that's fine for the County, Judge.

THE COURT: So Ms. Fire, are you there? I can't even tell. There's so many people on the screen, I can't see them all. Can we get a series of maybe three days, Ms. Fire? Or Lindsay, can you hear us?

THE CLERK: Yes. Just the morning? All day? What are you thinking?

THE COURT: Because I know we aren't going

to have any of those cases we were hoping to go to jury trial, we're not going to be able to go, I know that.

MR. CALDWELL: Your Honor, I think as a practical matter -- and I do have some personal -- some minor surgery issues in the later part of April -- I think early May would probably be the earliest that we could get this all together. I mean, we've -- since February 4th, anyways, we were under the impression that this was not going to go forward and would have to really spin up a lot of work.

THE COURT: No, Mr Caldwell, now wait a minute. What I said is I thought you raised serious concerns and I was willing to hear that first, but I never gave any indication this was a slam dunk.

MR. CALDWELL: I understand that, your Honor. I'm just saying during that time we've been focused on whether there would be a hearing, and I think that, you know, early May might be the most practical time to go forward with this.

THE COURT: What do we have early May, Ms. Ostroski [phonetic]?

THE CLERK: Monday, May 3rd, Tuesday,

May 4th, or Wednesday, May 5th are all open.

THE COURT: Give me those three, 3rd, 4th, and 5th of May, and we'll see where we are after that.

MR. DAVIS: Judge, would you want our briefs in a certain time before that?

THE COURT: Just that weekend before; I'll read them all through the weekend before. But again, the main thing is the focus for me, you know, is come in with opening statement, what specifically you want me to do. How I can legally and why. And then I'll start hearing the evidence.

MR. POSTEMA: Just to clarify, the briefs that would be coming in that are almost done from the intervenors' standpoint would be exactly what you asked before, that is: What is the law? What do you want to do? And what's the science? And we're prepared to do that. We could get that easily to you one or two weeks before those dates.

THE COURT: That's greet. The earlier, the -- the earlier I get it, the better. It gives me more time to read it and to think about it.

MR. POSTEMA: So could we set up a schedule on the briefing that's a little more -- I know nobody wants to dump this on you, you know,

the weekend before. But perhaps if we could move it back and just use the prior outline that we had as far as time.

THE COURT: Which is what, two weeks before?

MR. DAVIS: Yes, two weeks before.

MR. CALDWELL: And Judge, I think we had a staggered briefing schedule, so we had two weeks to respond to the intervenors' briefs.

THE COURT: Yeah, if you get it two weeks before and then get me the response the weekend before, that's fine, you know?

MR. NEGELE: But we had a staggered schedule, you know, the intervenors would file first, Gelman would file second in response, and then there was a third filing date for a reply for intervenors and also for the State to chime in. And, you know, trying to avoid this sort of, you know, circular firing squad that will result if we're all filing at the same time.

THE COURT: You make a very good point. I let all of you as a democracy decide your scheduling order and I signed it. So I don't care. If you want to keep those same dates or that same staggard approach and back it off of that, that's

Page 53

fine with me.

MR. POSTEMA: I think the intervenors, we would be interested in that. And I think that we can work with Gelman to incorporate that prior sequence on this date because we have plenty of time between May 3rd, and, you know, I think that would be appropriate, Judge.

MR. DAVIS: Your Honor, we can circulate a proposed order. We use Bill Stapleton as our typist, so we can have Bill wrangle up a proposed order.

THE COURT: That would be good. And then we can all read it together on May 1st.

MR. STAPLETON: And Judge, just a point of clarification. The Court intends to hear from witnesses during this hearing, from expert witnesses; is that correct?

THE COURT: Whatever -- it's your case. So if you're saying, here's the science and we have this witness available, if you have questions, it's your case how you want to present it. But obviously if you don't have stipulation on the submission of a report, you're going to have to bring in a witness to lay the foundation and move for its admission.

Page 54

MR. STAPLETON: Thank you, your Honor. There was some -- there was a question with the previous proceeding as to whether there were going to be live witness, so that's why I asked. Thank you.

THE COURT: No, I think -- and I'll give it as many days as it takes. But again, I view it as an evidentiary hearing. I'll be the fact finder. I'll make a ruling, and then it'll be off to the court of appeals.

MR. DAVIS: Okay.

THE COURT: If Mr. Caldwell is successful in getting to intervene before that, then they will. But that's their call; I'm not going to say it.

MR. POSTEMA: Judge, we'll do what you said and we will submit something. Bill is not just the typist, he's the scrivener. We consider him the scrivener; it's a little more elevated title.

MR. STAPLETON: Thank you. I appreciate that.

THE COURT: And again, I'm going to repeat this: I really, truly appreciate the professionalism of the attorneys in this case with

Page 55

me, even when I've had to make rulings that you don't like. I have thoroughly appreciated it and I look forward to the same kind of approach going into this evidentiary hearing.

MR. CALDWELL: I appreciate that, you're Honor.

MR. DAVIS: Thank you, Judge.

MR. DINDOFFER: We'll full circle back with the State and with Gelman, your Honor, and come back with an order to you quickly.

THE COURT: You know, counsel, it's good to see you again. Because the last time, COVID just about hit, we tried to sit in a circle to see where it'd go and you did the first elbow bump, and I kind of looked. You knew more about what was coming than the rest of us, I'll tell you that.

MR. DINDOFFER: You weren't sure if it was needed then, were you?

THE COURT: No, it' been a hard, hard year, I'll tell you. I know it's hard for everybody, but I'm not kidding. The families on the child welfare docket that I know you know I do, they have really been hit, and we have to keep that in mind. And as we come out of it, I hope that -- I hope we all learn from this. I hope we really

Page 56

all learn. But it's a pleasure. I'll see you all soon.

(Proceedings concluded at 9:30 a.m.)

* * *

STATE OF MICHIGAN)
COUNTY OF MACOMB)

Certificate of Notary Public

I certify that this transcript, consisting of 57 pages, is a complete, true, and correct record of the audio transcription, to the best of my ability, on the Motion for Reconsideration Hearing held on March 22, 2021.

I further certify that I am not related to any of the parties or their attorneys or agents, nor am I an employee of any of the parties or their attorneys; and that I am not interested directly, indirectly, or financially in the matter of controversy.

Kimberly H. Callahan-Golden, CSR - 7995
Core Litigation Support, LLC
Certified Shorthand Reporter
Notary Public, Macomb County, Michigan
My Commission expires 5/13/2026

DATE: March 25, 2021

STATE OF MICHIGAN

IN THE CIRCUIT COURT FOR THE COUNTY OF WASHTENAW

ATTORNEY GENERAL FOR THE STATE OF MICHIGAN *ex rel*. MICHIGAN DEPARTMENT OF NATURAL RESOURCES AND ENVIRONMENT,

Plaintiff,

and

THE CITY OF ANN ARBOR, WASHTENAW COUNTY, THE WASHTENAW COUNTY HEALTH DEPARTMENT, WASHTENAW COUNTY HEALTH OFFICER JIMENA LOVELUCK, THE HURON RIVER WATERSHED COUNCIL, AND SCIO TOWNSHIP,

Intervenors,

v

GELMAN SCIENCES, INC., a Michigan Corporation,

Defendant.

Washtenaw Circuit
Court N. 88-34734-CE

Hon. Timothy P. Connors

DEFENDANT GELMAN SCIENCES, INC.'S MOTION FOR ENTRY OF PROPOSED ORDER

/

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/

BRIEF IN SUPPORT OF DEFENDANT'S GELMAN SCIENCES, INC'S MOTION FOR ENTRY OF ORDER SETTING BRIEFING/DEPOSITION SCHEDULE AND NEW HEARING DATES

Gelman relies on MCR 2.119 and 2.602(B)(4) in support of its Motion.

Respectfully submitted,

ZAUSMER, P.C.

/s/Michael L. Caldwell

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Dated: March 29, 2021

PROOF OF SERVICE

The undersigned certifies that a copy of the foregoing instrument was served upon all parties to the above cause to each of the attorneys of record herein at their respective addresses as directed on the pleadings on March 30, 2021 by:

☒ E-FILE ☐ US MAIL ☐ HAND DELIVERY ☐ UPS
☐ FEDERAL EXPRESS ☐ OTHER

/s/Brenda Ann Smith
Brenda Ann Smith

STATE OF MICHIGAN

IN THE CIRCUIT COURT FOR THE COUNTY OF WASHTENAW

ATTORNEY GENERAL FOR THE STATE OF
MICHIGAN *ex rel*. MICHIGAN
DEPARTMENT OF NATURAL RESOURCES
AND ENVIRONMENT,

Plaintiff,

and

THE CITY OF ANN ARBOR, WASHTENAW
COUNTY, THE WASHTENAW COUNTY
HEALTH DEPARTMENT, WASHTENAW
COUNTY HEALTH OFFICER JIMENA
LOVELUCK, THE HURON RIVER
WATERSHED COUNCIL, AND SCIO
TOWNSHIP,

Intervenors,

v

GELMAN SCIENCES, INC., a Michigan
Corporation,

Defendant.

Washtenaw Circuit
Court N. 88-34734-CE

Hon. Timothy P. Connors

DEFENDANT GELMAN SCIENCES, INC.'S MOTION FOR ENTRY OF PROPOSED ORDER

______________________________________/

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DEFENDANT'S GELMAN SCIENCES, INC'S MOTION FOR ENTRY OF ORDER SETTING BRIEFING/DEPOSITION SCHEDULE AND NEW HEARING DATES

Defendant, Gelman Sciences, Inc. ("Gelman"), through its attorneys, Zausmer, P.C., asks this Court to enter an Order setting a briefing/deposition schedule and new hearing dates for the hearing on Modification of Consent Agreement set by the Court during the March 22, 2021 hearing on Gelman's Motion for Reconsideration for the following reasons:

1. Contemporaneously with this motion, Gelman has filed its Objections to the Intervenors' proposed Order Regarding Defendant's Motion for Reconsideration under the 7-Day Rule, MCR 2.602(B). Attached as Exhibit A to Gelman's Objections is Gelman's proposed alternative Order submitted under MCR 2.602(B)(c) ("Proposed Order"). Gelman files this motion in further support of entry of its Proposed Order because, in addition to addressing deficiencies in Intervenors' 7-Day Order, it provides a necessary briefing/deposition schedule and new hearing dates in connection with the remedy hearing.

2. On March 22, 2021, this Court heard oral argument regarding Gelman's Motion For Reconsideration And Motion To Stay. In addition to denying those motions, this Court set a

hearing on Modification of the Consent Agreement for May 3 – 5, 2021. The Court further instructed counsel to work out a staggered briefing schedule along the lines of the Court's previous scheduling orders regarding this hearing. The Court also indicated for the first time that the hearing would include live testimony from expert witnesses.

3. Counsel have not reached agreement with respect to either a briefing schedule or on whether counsel will be able to take the depositions of testifying witnesses prior to the hearing.

4. A staggered briefing schedule similar to the one included in this Court's previous scheduling orders and as the Court authorized during the March 22, 2021 hearing is necessary to provide the Court with briefing materials, testimony, and argument that efficiently present the relevant issues, science, and legal authorities.

5. As expressed by the Assistant Attorney General at the March 22, 2021 hearing, it is Gelman's understanding that counsel for the State also believes that a staggered briefing schedule is appropriate to best present the relevant facts and science to the Court.

6. Counsel for Intervenors have repeatedly assured the Court that Intervenors' papers were nearly prepared. Consequently, requiring Intervenors to file and serve their legal and technical briefs far enough in advance to allow Gelman and the State to respond (and for Intervenors to have the opportunity to file a reply) will not burden Intervenors.

7. These same considerations drive the need to take the depositions of testifying witnesses prior to the hearing. The remedy hearing set by the Court will take place before Intervenors have filed their complaints, before the Intervenors claims have been litigated, and before the parties have conducted any discovery with respect to these claims and the relief Intervenors are seeking. At a minimum, Gelman should be allowed to take the depositions of the testifying expert witnesses so that the bases for their opinions can be explored and the nature of

their testimony can be fully understood before the Court hears and potentially relies on their testimony in determining how to modify the existing Consent Judgment.

8. It is Gelman's understanding that counsel for the State also believes that the expert depositions must occur in order for the counsel to efficiently present the relevant facts and science to the Court.

9. There is not sufficient time before the May 3-5, 2021 hearing dates set by the Court for the reasonable staggered briefing schedule that the Court asked counsel to establish and the depositions of witnesses to occur.

10. Therefore, Gelman asks this Court to enter Gelman's Proposed Order, which provides an expedited schedule for the briefing schedule, a week for expert depositions, and new hearing dates of May 24 – 26th that can accommodate these important steps.

11. Gelman does not, by identifying by seeking to address these procedural issues or entry of its Proposed Order waive its objections to the decision to hold the hearing.

Respectfully submitted,

ZAUSMER, P.C.

/s/Michael L. Caldwell

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Dated: March 29, 2021

PROOF OF SERVICE

The undersigned certifies that a copy of the foregoing instrument was served upon all parties to the above cause to each of the attorneys of record herein at their respective addresses as directed on the pleadings on March 30, 2021 by:

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/s/Brenda Ann Smith
Brenda Ann Smith

STATE OF MICHIGAN

IN THE CIRCUIT COURT FOR THE COUNTY OF WASHTENAW

ATTORNEY GENERAL FOR THE STATE OF MICHIGAN *ex rel.* MICHIGAN DEPARTMENT OF NATURAL RESOURCES AND ENVIRONMENT,

Plaintiff,

and

THE CITY OF ANN ARBOR, WASHTENAW COUNTY, THE WASHTENAW COUNTY HEALTH DEPARTMENT, WASHTENAW COUNTY HEALTH OFFICER JIMENA LOVELUCK, THE HURON RIVER WATERSHED COUNCIL, AND SCIO TOWNSHIP,

Intervenors,

v

GELMAN SCIENCES, INC., a Michigan Corporation,

Defendant.

Washtenaw Circuit
Court N. 88-34734-CE

Hon. Timothy P. Connors

DEFENDANT GELMAN SCIENCES, INC.'S OBJECTIONS TO PROPOSED 7-DAY ORDER

____/

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DEFENDANT GELMAN SCIENCE'S OBJECTION TO PROPOSED 7-DAY ORDER DENYING MOTION FOR RECONSIDERATION AND SCHEDULING HEARING DATES

Defendant, Gelman Sciences, Inc. ("Gelman"), through its attorneys, ZAUSMER, P.C., hereby objects to the proposed Order Regarding Defendant's Motion for Reconsideration as submitted on March 29, 2021, under MCR 2.602(B) for the following reasons:

1. The Proposed Order does not reflect that the Court denied Gelman's January 22, 2021 Motion to Stay Order Scheduling Hearing on Modification of Consent Agreement.

2. The Proposed Order does not specify the date or complete title of Gelman's January 28, 2021 Motion for Reconsideration of Order Scheduling Hearing on Modification of Consent Agreement.

3. The hearing scheduled by the Court should be described, as it has in the previous four scheduling orders including the original order prepared by the Court, as "hearing on Modification of Consent Agreement."

4. The Proposed Order does not include a staggered briefing schedule that is necessary to provide the Court with briefing materials, testimony, and argument that efficiently present the

relevant issues, science, and legal authorities to the Court as the previous scheduling orders have and as the Court authorized during the March 22, 2021 hearing. As expressed by the Assistant Attorney General at the March 22, 2021 hearing, it is Gelman's understanding that counsel for the State also believes that a staggered briefing schedule is appropriate to best present the relevant facts and science to the Court. Counsel for Intervenors have repeatedly assured the Court that Intervenors' papers were nearly prepared. Consequently, requiring Intervenors to file and serve their legal and technical briefs far enough in advance to allow Gelman and the State to respond (and for Intervenors to have the opportunity to file a reply) will not burden Intervenors.

5. The Proposed Order fails to indicate whether hearing will include live testimony and, if so: (i) a date by which witnesses must be disclosed; and (ii) an opportunity to take the depositions of the other parties' witnesses prior to the hearing. If the hearing will include live testimony, both of these steps are necessary to provide the Court with the salient facts and legal authorities. It is Gelman's understanding that counsel for the State also believes that the expert depositions must occur in order for the counsel to efficiently present the relevant facts and science to the Court.

6. There is not sufficient time before the May 3-5, 2021 hearing dates set forth in the Proposed Order for the reasonable staggered briefing schedule that the Court asked counsel to establish and the depositions of witnesses to occur.

7. Pursuant to MCR 2.602B(3)(c), Gelman submits the attached Order (**Exhibit A**), which addresses these deficiencies.

8. Gelman does not, by identifying these objections or its presentment of a proposed scheduling order waive its objections to the decision to hold hearing.

Respectfully submitted,

ZAUSMER, P.C.

/s/Michael L. Caldwell

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Dated: March 29, 2021

PROOF OF SERVICE

The undersigned certifies that a copy of the foregoing instrument was served upon all parties to the above cause to each of the attorneys of record herein at their respective addresses as directed on the pleadings on March 30, 2021 by:

☒ E-FILE ☐ US MAIL ☐ HAND DELIVERY ☐ UPS
☐ FEDERAL EXPRESS ☐ OTHER

/s/Brenda Ann Smith
Brenda Ann Smith

EXHIBIT A

RECEIVED by MSC 10/4/2021 5:25:41 PM

STATE OF MICHIGAN

IN THE CIRCUIT COURT FOR THE COUNTY OF WASHTENAW

ATTORNEY GENERAL FOR THE STATE OF MICHIGAN ex rel. MICHIGAN DEPARTMENT OF NATURAL RESOURCES AND ENVIRONMENT,

Plaintiff,

and

THE CITY OF ANN ARBOR, WASHTENAW COUNTY, THE WASHTENAW COUNTY HEALTH DEPARTMENT, WASHTENAW COUNTY HEALTH OFFICER JIMENA LOVELUCK, THE HURON RIVER WATERSHED COUNCIL, AND SCIO TOWNSHIP,

Intervenors,

v

GELMAN SCIENCES, INC., a Michigan Corporation,

Defendant.

Case No. 88-34734-CE

Hon. Timothy P. Connors

ORDER DENYING DEFENDANT GELMAN SCIENCES, INC.'S MOTION FOR RECONSIDERATION AND MOTION TO STAY, AND SCHEDULING HEARING ON MODIFICATION OF CONSENT AGREEMENT

____/

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ORDER DENYING DEFENDANT GELMAN SCIENCES, INC'S MOTION FOR RECONSIDERATION AND MOTION TO STAY, AND SCHEDULING HEARING ON MODIFICATION OF CONSENT AGREEMENT

At a session of said Court held in the
City of Ann Arbor, County of Washtenaw,
State of Michigan on _______________

PRESENT: Hon. Timothy P. Connors

Defendant Gelman Sciences, Inc. ("Gelman") having filed its January 28, 2021 Motion for Reconsideration of Order Scheduling Hearing on Modification of Consent Agreement ("Motion for Reconsideration") and January 22, 2021 Motion to Stay Order Scheduling Hearing on Modification of Consent Agreement ("Motion to Stay"), the parties having filed responses, and the Court having heard oral argument on February 4, 2021 and March 22, 2021;

IT IS ORDERED that Gelman's Motion for Reconsideration and Motion to Stay are each denied for the reasons stated on the record.

IT IS FURTHER ORDERED that there is a hearing on Modification of Consent Agreement set for May 24 – 26, 2021 at 9:00 am ("Hearing"). Before commencement of the Hearing, counsel shall submit Briefs and Expert reports, identify testifying witnesses, and take the depositions of such witnesses, if desired, in accordance with the following schedule:

- Intervenors by 4/9/2021.
- Gelman response by 4/30/21.
- All parties will identify the witnesses they intend to call at the hearing by 4/30/21.
- EGLE response/Intervenors' reply by 5/7/2021.
- Depositions of the identified witnesses may be taken from 5/10/21 until 5/14/21.

This Order does not resolve the last pending claim and does not close the case.

CIRCUIT COURT JUDGE
Timothy P. Connors

STATE OF MICHIGAN

IN THE CIRCUIT COURT FOR THE COUNTY OF WASHTENAW

FRANK J. KELLEY, ATTORNEY GENERAL,
et al,
Plaintiffs,

Case No. 88-034734-CE

-vs-

GELMAN SCIENCES, INC.,
Defendant.
_______________________________________/

VIDEOTAPED PROCEEDINGS

Objections to Proposed 7-Day Order

BEFORE THE HONORABLE TIMOTHY P. CONNORS

Ann Arbor, Michigan - April 1, 2021

TRANSCRIBED BY: Ginger K. Hoffman, CSMR-9234
Core Litigation Support, LLC

APPEARANCES:

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ALSO APPEARING: Mr. Raymond B. Ludwiszewski

Thursday, April 1, 2021
Ann Arbor, Michigan
11:50 a.m.

* * *

THE CLERK: Do we have everybody we need?

MR. CALDWELL: I think so.

THE CLERK: Okay. We are now on record in the Frank Kelley versus Gelman Sciences, Case No. 88-34734-CE. This is defendant's objection to the proposed seven-day order and motion for entry of order setting briefing, deposition dates.

THE COURT: Good morning. This is Judge Connors. Could we -- first of all, I apologize for the delay. My computer crashed and I had to reboot, and technology was not kind to me. But we're back.

Could we have appearances on the record, please.

MR. CALDWELL: Good morning, Your Honor. Mike Caldwell on behalf of Gelman Sciences. Ray Ludwiszewski is also here on behalf of Gelman Sciences.

MR. POSTEMA: Good morning, Your Honor. Stephen Postema for the city of Ann Arbor. With me today is Abigail Elias and outside counsel Fred

Dindoffer.

MR. STAPLETON: Good morning, Your Honor. William Stapleton for Scio Township.

MR. NEGELE: Good morning, Your Honor. Brian Negele, Assistant Attorney General, for the Michigan Department of Environment, Great Lakes, and Energy.

MR. DAVIS: Your Honor, Robert Davis on behalf of the County. But I can't see my face.

THE COURT: Nor can we, sir.

MR. CALDWELL: It's a win-win situation.

MR. DAVIS: That's not a bad thing, but I don't know how to make it show. Oh, hang on.

MR. POSTEMA: OH, well.

THE COURT: Hit start video.

MR. DAVIS: Oh, there we go.

MR. DINDOFFER: Oh, there you are.

THE COURT: And Ms. Mette, I think you were about to put your appearance.

MR. DAVIS: Thank you, Judge.

MS. METTE: Yes. Good morning, Your Honor. Erin Mette on behalf of the Huron River Watershed Council.

THE COURT: Let me tell you that I have had -- and when I'm looking down, it's not because I'm not listening to you, it's because I have your motions on an iPad in front of me. I've read them. Ms. Mette, I saw your response came in yesterday. And my understanding is, though, while -- first of all, thank you. You're the one that drafted it. But that everyone else on the intervenors is signing off on that; is that correct?

MS. METTE: Yes. That's correct.

MR. POSTEMA: Correct, Your Honor.

THE COURT: So will you be arguing the motion this morning, Ms. Mette?

MR. STAPLETON: Yeah. Your Honor, I will be arguing the motion on behalf of intervenors.

THE COURT: Okay. And Mr. Gelman, will you be -- I'm sorry. Mr. Gelman. Oh, Mr. Caldwell, I'm in trouble now.

Mr. Caldwell, will you be arguing the motion?

MR. CALDWELL: Yes, I will, Your Honor. Thank you.

THE COURT: Go right ahead. It's actually your -- it's your objection, I believe.

MR. CALDWELL: Yeah. I just wanted to note at the outset that we filed objections to the proposed order, but we also filed the independent motion that the -- that was read into the record. And I wanted to point that out because the intervenors object to the

scope of our objections, which admittedly go beyond specific objections to their proposed order and address issues that were not specifically addressed by the Court in the hearing. And that is why we filed that independent motion.

As the Court knows, there was no filing by any party that resulted in the scheduling of this hearing, so there was no -- previously no motion to -- that we filed or a motion that we could respond to to address these issues beforehand and, therefore, we filed the standalone motion. It's supported by our brief.

At the outset, just a couple of the technical issues -- and I think, unless Your Honor has a different idea, we should probably just go objection by objection and get the response to each one rather than having me just go through the whole thing, if that makes sense.

THE COURT: No, it doesn't make any sense at all.

You know, I mean, it's there. It's in writing. I get it. You know, I'm not going to just rehash the written motion. If you want to highlight a few points, go right ahead in oral argument.

MR. CALDWELL: Okay.

THE COURT: It's already part of it. I don't want to just be reading in written motions.

MR. CALDWELL: Oh, well, I wasn't going to do that. But, okay.

The first objection, there's no provision for the court having denied our motion for stay --

THE COURT: Mr. Caldwell. Mr. Caldwell. I am interrupting you. I apologize for interrupting you. I can read. I've read it. I don't need you to say the same thing.

MR. CALDWELL: Okay.

THE COURT: If there's something in addition to the written document that you have, now is the time to say it.

MR. CALDWELL: All right. On the title of the motion for reconsideration, want to point out that we filed two motions for reconsideration because the order in which the first motion was filed got vacated by the fourth amended scheduling order, and we just want the record to be clear.

The purpose of the hearing, we propose to use the description that has been used in all four scheduling orders, including the order that the Court filed and drafted itself, the original one. That is hearing on modification of consent agreement. The

intervenors want to refer to the previous -- the remediation orders that were discussed in the reconsideration hearing. The truth is, there is only one active document at this point, and that is the third amended consent judgment.

The 2011 stipulated order that was attached to the intervenors' response to our motion for reconsideration makes that very clear. It says to the extent the third amendment is inconsistent with any of the requirements of the remediation order or the Unit E order, the third amended consent judgment shall govern. And then it also -- so it basically incorporates the provisions that are not inconsistent with the third amendment, makes the third amendment the controlling document. So I don't know why there would need to be any reference to the remediation orders. It's not actually accurate and would just be confusing.

The -- you know, the two big issues I think are the staggered -- the lack of a staggered briefing schedule. We believe that that is necessary. We believe the Court encouraged the parties to come up with that briefing schedule, as Mr. Negele, on behalf of the State and EGLE, noted during the hearing, not having that and having everyone file their motions at the -- or their briefs at the same time is akin to a circular firing squad.

If we don't know what they're going to say, what they're going to propose, we don't know what to respond to. We don't know what we should address in our initial pleading, and it will provide -- a staggered briefing schedule will provide the Court with a much more efficient and thorough examination of the factual issues. And I don't think anybody really disagrees with that.

Our staggered briefing schedule differs in two ways from one that the intervenors attached to their response to our objections. One, it gives us -- gives Gelman three weeks to respond. They only provide for two weeks to respond to their initial filing, which shouldn't be any prejudice for them to file. They've -- counsel for intervenors have indicated several times that it's nearly done, if not completely done at this point.

We ask for three weeks because indications are, from talking to intervenor counsel, that their submission is going to be massive and it will take a significant amount of time to digest. I know the Court has asked us to not start at the far end and list every possible demand, but indications are that that might not be the case. And if that -- if I am wrong, and

perhaps Mr. Dindoffer, as I believe the primary author, perhaps the Court could inquire as to how long their briefs are going to be. And if they're not as long as I'm anticipating, then maybe two weeks would be sufficient.

The second aspect that differs on the staggered briefing schedule is that our proposed order provides for the identification of the witnesses that are going to testify on behalf of each entity, each participant, and the ability to -- it provides time to take their depositions. Short period --

(Cell phone interruption.)

THE COURT: Excuse me.

MR. CALDWELL: We think that's necessary both for due process grounds and for the Court's -- the presentation of the information to the Court.

Due process, obviously, we object on due process grounds to this entire hearing. There's nothing we're going to do here absolve -- you know, taking care of those objections. But intervenors have repeatedly said Gelman will get due process at the hearing, but we can't obtain due process through this hearing if it's trial by surprise. And I think the -- I mean, the witnesses, we need to know what they're going to say, we need to explore what the bases for

those opinions are, and we need to, you know, pressure test their qualifications and the bases for those opinions. I don't think this is an unreasonable request. And I understand that the State, who's actually going to have to implement whatever remedy ultimately gets put in place, feels the same way.

So those are the two basic differences in the staggered briefing schedule that we've proposed.

And the third -- then that triggers the third change, and that is the hearing date. There simply isn't enough time between now and May 3rd, which is under -- admittedly, the date that the Court set for the hearing, to accomplish a weeklong period of depositions and the staggered briefing schedule. We've proposed a date later in May, a couple weeks later in May. Still going to happen on a very expedited basis.

Time considerations are certainly important, Your Honor, but I would point out that we had the May -- or excuse me -- the February 4th hearing on the -- our motion for stay, and the reconsideration issue came up and kind of pushed that back, and we waited over six weeks to have the hearing on the motion for reconsideration. So I don't think -- and maybe there are other time constraints, and I'm certainly not going to suggest that the Court's -- the Court noted

that it's child neglect docket will likely pick up shortly, and I am not suggesting that anything is more important than that. That certainly has to be a priority, but we can work around that.

Gelman has been continuing to implement the response activities that are required by the current version of the consent judgment for the four years since the new cleanup criteria came into effect, Your Honor, and those response activities have successfully and will continue to successfully prevent any unacceptable exposures. There's no imminent issue related to the -- you know, on an environmental basis that can't accommodate a two -- a couple weeks' adjourn -- postponement of the hearing.

So I don't think anything we're asking for is unreasonable, but I'll look forward to intervenors' counsel telling us why it is.

THE COURT: Thank you. And one correction, Mr. Caldwell. The child welfare docket has picked up and has never gone away. I've been trying those cases since last March. My point is, is that I will continue to do that, and I know that we're going to have to jury trials. Now's the time we need to address that. But I'm not -- we've been doing the child welfare docket daily for --

MR. CALDWELL: Good. Good.

THE COURT: Mr. Stapleton?

MR. STAPLETON: Yes. Yes, your Honor.

Your Honor, to the best of my recollection, at the hearing, the Court ordered three things. It -- and all of which are included in our proposed order. The Court denied Gelman's motion for reconsideration. The Court scheduled the hearing regarding the modification of the cleanup order based on the revised cleanup criteria for May 3, 4, and 5. And the Court directed the parties to file briefs and expert reports by April 30. Those are the three things that I recall the Court ordered.

The Court did say that the parties could work out a briefing schedule, if they wanted, but we have not been able to do that. So the intervenors submitted what we are required to do under the court rules. We submitted a seven-day order, which embodied exactly what the Court ordered; nothing more, nothing less. And Gelman now responds with an order with things that it wants in the order but that were not included in what was discussed during the March 22nd hearing or what the Court ordered. I mean, Gelman wants more time, they want to push the hearing date back three weeks, they want depositions. None of this was

ordered by the Court, none of it was discussed, and it shouldn't be considered when we are submitting an order under the seven-day rule, which should simply reflect what the Court ordered.

Judge, regarding the briefing schedule that Mr. Caldwell proposes, I just need to remind the Court: A few hours after the March 22nd hearing, intervenors sent a proposed briefing schedule, which is still entirely workable. We would file our briefs April 9, Gelman would file its response by April 23, two weeks later -- and the way, this two-week response period Gelman has agreed to in three previous scheduling orders -- parties would identify witnesses by April 26, intervenors file their reply, EGLE files its response by April 30, and we have the hearing on May 3, 4, 5. This timeline is very similar to what happened previously embodied in the Court's scheduling orders and agreed to by all the parties. And as I said, Judge, this remains a very workable schedule with keeping the court date of May 3, 4, and 5.

Judge, on the issue of depositions, you know, I guess the intervenors find this somewhat humorous. You know, this is an unusual case, admittedly. We have been essentially conducting discovery in this case for the past three years. I mean, the attorneys and the

expert for all the parties have spent hours and hours and hours in a room together discussing all aspects of this cleanup for this -- for the Gelman site, and the product of this was the fourth amended consent judgment, which has been made public.

Judge, Dr. Larry Lemke has spent -- he's the intervenors' primary expert. He's spent hours explaining to Mr. Caldwell the basis of his opinions. Your Honor, Dr. Lemke has seven videos posted on the City website which explain in detail the basis of his opinions as they relate to every component of this cleanup. It's there for Mr. Caldwell and others to view. It's public knowledge. It's out there. And wants again, Mr. Caldwell has had the benefit of spending many hours with Dr. Lemke addressing his opinions on this -- for this remediation.

You know, I would venture to say, Judge, that Mr. Caldwell has probably never been better prepared for an expert witness in his professional life. He doesn't need the deposition of Dr. Lemke because he knows exactly what Dr. Lemke is going to say.

So from the intervenors' standpoint, this request to push off the hearing for three weeks, requests for depositions, another delay tactic by Gelman, and we just think the Court should order what

we provided under the seven-day rule, which set the hearing for May 3, 4, and 5, and provided that everyone submit their briefs by April 30, and we'll hold the hearing, and we'll take it from there.

Thank you, Your Honor.

THE COURT: I suppose technically, Mr. Caldwell, you get a rebuttal argument on your motion. And if you'd like to do it, go ahead, sir.

But you are muted, so your words have no effect right now.

MR. CALDWELL: So, Your Honor, I would like to respond briefly, but I would defer, in the first instance, to Mr. Negele, who has not had an opportunity to respond.

THE COURT: Fair enough. Mr. Negele, that's true. You previously have taken no position, but I take it you are taking a position.

MR. NEGELE: Yes, I am. Thank you.

As to the question about depositions, yes, you know, we've heard lots of things that Larry -- Dr. Lemke has said and, you know, I understand he's not the only expert we're talking about. But, you know, we don't know what is actually going to be filed until it's filed -- the expert report. And I assume that that's really what we're going to want to be testing and not what has been stated in various videos or what's been stated during, you know, our negotiations.

And the other thing is, too, is, you know, the scheduling order that was originally proposed really cut short by, you know, one week EGLE's response time. You know, the one that Gelman proposes gives us, you know, basically four weeks to respond, which was consistent with the original scheduling order that we had had.

And so those are the two primary things that I wanted to address.

THE COURT: Thank you, sir.

Mr. Caldwell, do you still want the last word?

MR. CALDWELL: Just briefly, Your Honor.

With regard to the sanctity of the previous scheduling orders, those scheduling orders were agreed to before live testimony was on the table, or recall previously the Court had specifically said live testimony would not be included.

And with regard to the availability of Dr. Lemke throughout the negotiation process and the videos, Dr. Lemke in the videos supported the agreement that the intervenors rejected. So there's two possibilities. Either his testimony in this hearing

will support the intervenor -- the version that intervenors rejected, which would be surprising, or there's going to be something else. And we're entitled to find out what that something else is and explore the bases for those opinions after that report is submitted.

And we hope that doctor -- or excuse me -- Mr. Stapleton only spoke with regard to Dr. Lemke. There was another expert. I don't know if he will participate, but he was only at one or two or maybe three of the meetings that Mr. Stapleton refers to you. And there's no record, obviously, of any of those discussions. They're all subject to the confidentiality rule.

So I think there is definitely a basis for taking those depositions and a need for that in order to provide the Court with a full record that it's going to use in order to determine a remedy that's going to affect the good people of this community for many years.

THE COURT: Thank you.

First, Ms. Mette, I want to congratulate you. I think you captured and distilled the essence of my ruling. Mr. Stapleton's point is good. That is my ruling. And so if you can submit that by MiFile. I

know it was part of the pleading, but if you can't separately submit that to Ms. Ostrowski, the clerk, I'll get that signed today.

And then that includes the denial of the motion for reconsideration. So Mr. Caldwell would have been -- hopefully, it'll be signed today and we'll get you confirmation. We'll e-mail it.

Mr. Stapleton, I think you make a very good point on the fact -- certainly, you know, before I took the bench I was a civil litigator, and you know that I've tried civil cases the whole time on the bench. I can't imagine a situation where both sides were more prepared, and I've been ordering that they share information and disclosure over years. I mean, candid discussions.

These are the -- this is the type of thing in the court rule that's contemplated where the defense attorney gets to go in and talk to a treating physician before -- you know, sometimes even without the patient there. So, you know, no, I'm not going to order depositions.

Here's what I need: For -- whether or not I'm correct, I made the ruling that this needs to be addressed, and I'm the judge assigned, and I've not been part of your discussions. I don't know what your

information is, and I have to be the fact-finder. I have to make that determination. I've set aside those three days.

Here's what I need. I'm telling you as a fact-finder here is what I need. We're not playing to a jury. I understand you have other audiences -- clients, Court of Appeals, public, et cetera -- but help me, please. Let me do my job, at least, and then you can go up and I'm happy if the Court of Appeals comes down and says you need to do it differently, you need to do it better. It's okay.

What I need is to hear from the various scientists, given this new cleanup criteria, what can be done? First of all, just what are the possibilities? And then what should be done and why? Because it's -- you know, if it can't be done, it's silly to talk about what should be done. But if it can be done, explain to me what can be done, how it can be done, as an educator. You're educating me as the fact-finder. That's the role where an expert's going to be effective to me.

And similarly, each of the attorneys, you legally then say, Judge, given this science, here's what we believe you can do legally -- court rule, statute, case law -- and, obviously, some will say and why you can't -- and then what you should do, what the law permits. It's really pretty straightforward, folks. And at the end of the day, someone has to make the call. This is the opportunity.

I also said I would take as many days as it takes. So if we get through the third day and we say, Judge, we'd like the opportunity; we want to come back, we want to present you some more evidence, you make that argument just like any other nonjury trial, and I'll give you honest feedback of whether I think that would be helpful in my decision-making or not.

So I'm signing today, Ms. Mette, if you sent that over, please. And I'm leaving -- I'm not ordering any briefing schedule. Get your briefs in by April 30th. You know, we've just got to roll up our sleeves and get at it, okay?

MR. CALDWELL: Your Honor, if I may --

MR. STAPLETON: I thank you, Your Honor.

MR. CALDWELL: -- will the Court issue an order denying our motion for stay?

THE COURT: If you send it to me by MiFile, I'd be happy to do that.

MR. CALDWELL: Thank you.

THE COURT: You prepare it.

MR. CALDWELL: AND THEN counsel will -- well,

I'll send it around and hopefully we can agree on something.

THE COURT: Okay.

MR. DAVIS: Okay.

THE COURT: Yeah, try -- yeah, not to keep coming back on these objections to proposed orders; all right?

MR. CALDWELL: Your Honor.

THE COURT: I'm fitting you in the middle of -- I'm going to make you be a guardian ad litem on my next child welfare docket, Mr. Caldwell. It's coming up at 1:00.

MR. CALDWELL: That's a good idea, Judge. Good idea.

THE COURT: All right.

MR. CALDWELL: Thank you.

THE COURT: All right. Good to see you both.

MR. DAVIS: Same to you, Your Honor.

THE COURT: Take care.

MS. ELIAS: Thank you.

THE COURT: And, Lindsay, do we start back at one?

THE CLERK: 1:00.

THE COURT: 1:00 on the NA?

THE CLERK: Yes.

THE COURT: Okay. Thank you.

(Proceedings concluded at or about 12:16 p.m.)

* * *

STATE OF MICHIGAN)
COUNTY OF OAKLAND)

Certificate of Notary Public

I certify that this transcript, consisting of 24 pages, is a true, correct, and complete record of the above-named proceedings and testimony taken in this case on April 1, 2021, and that I am not a relative, employee, attorney or counsel of any of the parties, nor financially interested in the action.

Ginger K Hoffman

NDTCA Signed Electronic Transcript

Ginger K. Hoffman, CVR, CSMR-9234
Notary Public, Oakland County, Michigan
My Commission expires 12/13/2021

Dated: April 6, 2021

STATE OF MICHIGAN

IN THE CIRCUIT COURT FOR THE COUNTY OF WASHTENAW

ATTORNEY GENERAL FOR THE STATE OF MICHIGAN ex rel. MICHIGAN DEPARTMENT OF NATURAL RESOURCES AND ENVIRONMENT,
Plaintiff,
and
THE CITY OF ANN ARBOR, WASHTENAW COUNTY, THE WASHTENAW COUNTY HEALTH DEPARTMENT, WASHTENAW COUNTY HEALTH OFFICER JIMENA LOVELUCK, THE HURON RIVER WATERSHED COUNCIL, AND SCIO TOWNSHIP,
Intervenors,
v
GELMAN SCIENCES, INC., a Michigan Corporation,
Defendant.

Case No. 88-34734-CE
Hon. Timothy P. Connors

ORDER DENYING DEFENDANT GELMAN SCIENCES, INC.'S MOTION FOR ENTRY OF ORDER SETTING BRIEFING/DEPOSITION SCHEDULE AND NEW HEARING DATES

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ORDER DENYING DEFENDANT GELMAN SCIENCES, INC'S MOTION FOR ENTRY OF ORDER SETTING BRIEFING/DEPOSITION SCHEDULE AND NEW HEARING DATES

At a session of said Court held in the
City of Ann Arbor, County of Washtenaw,
State of Michigan on 4/6/2021

PRESENT: Hon. Timothy P. Connors

Defendant Gelman Sciences, Inc. ("Gelman") having filed its March 30, 2021 Motion For Entry Of Order Setting Briefing/Deposition Schedule And New Hearing Dates, the above-listed Intervenors having filed their response, and the Court being otherwise advised in the premises;

IT IS HEREBY ORDERED that Gelman's Motion For Entry Of Order Setting Briefing/Deposition Schedule And New Hearing Dates is denied.

This Order does not resolve the last pending claim and does not close the case.

/s/ Timothy Connors 4/6/2021

CIRCUIT COURT JUDGE
Timothy P. Connors

APPROVED AS TO FORM

/s/Brian J. Negele	/s/Michael L. Caldwell
BRIAN J. NEGELE (P41846) Attorney for Plaintiffs	MICHAEL L. CALDWELL (P40554) Attorneys for Defendant
/s/Fredrick J. Dindoffer	/s/Robert C. Davis
FREDRICK J. DINDOFFER (P31398) NATHAN D. DUPES (P75454) Attorneys for City of Ann Arbor	ROBERT C. DAVIS (P40155) Attorney for Washtenaw County, Washtenaw County Health Department, and Washtenaw County Health Officer
/s/Erin E. Mette	/s/William J. Stapleton
ERIN E. METTE (P83199) Attorney for Huron River Watershed Council	WILLIAM J STAPLETON (P38339) Attorney for Scio Township

STATE OF MICHIGAN

IN THE CIRCUIT COURT FOR THE COUNTY OF WASHTENAW

ATTORNEY GENERAL FOR THE STATE OF MICHIGAN ex rel. MICHIGAN DEPARTMENT OF NATURAL RESOURCES AND ENVIRONMENT,
Plaintiff,
and
THE CITY OF ANN ARBOR, WASHTENAW COUNTY, THE WASHTENAW COUNTY HEALTH DEPARTMENT, WASHTENAW COUNTY HEALTH OFFICER JIMENA LOVELUCK, THE HURON RIVER WATERSHED COUNCIL, AND SCIO TOWNSHIP,
Intervenors,
v
GELMAN SCIENCES, INC., a Michigan Corporation,
Defendant.

Case No. 88-34734-CE
Hon. Timothy P. Connors

ORDER DENYING DEFENDANT GELMAN SCIENCES, INC.'S MOTION TO STAY

_______________________________________/

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ORDER DENYING DEFENDANT GELMAN SCIENCES, INC'S MOTION TO STAY

At a session of said Court held in the
City of Ann Arbor, County of Washtenaw,
State of Michigan on 4/6/2021

PRESENT: Hon. Timothy P. Connors

Defendant Gelman Sciences, Inc. ("Gelman") having filed its January 22, 2021 Motion to Stay Order Scheduling Hearing on Modification of Consent Agreement ("Motion to Stay"), the parties having filed responses, and the Court being otherwise advised in the premises;

IT IS HEREBY ORDERED that Gelman's Motion to Stay is denied.

This Order does not resolve the last pending claim and does not close the case.

/s/ Timothy Connors 4/6/2021

CIRCUIT COURT JUDGE
Timothy P. Connors

APPROVED AS TO FORM

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/s/Michael L. Caldwell

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/s/William J. Stapleton

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Attorney for Scio Township

STATE OF MICHIGAN

IN THE CIRCUIT COURT FOR THE COUNTY OF WASHTENAW

ATTORNEY GENERAL FOR THE STATE
OF MICHIGAN *ex rel.* MICHIGAN
DEPARTMENT OF NATURAL RESOURCES
AND ENVIRONMENT,

Plaintiffs,

-and-

Case No. 88-34734-CE
Hon. Timothy P. Connors

CITY OF ANN ARBOR; WASHTENAW COUNTY;
WASHTENAW COUNTY HEALTH DEPARTMENT;
WASHTENAW COUNTY HEALTH OFFICER
JIMENA LOVELUCK, in her official capacity;
HURON RIVER WATERSHED COUNCIL; and
SCIO TOWNSHIP,

Intervening Plaintiffs,

vs.

GELMAN SCIENCES, INC., a Michigan corporation,

Defendant.
____/

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ORDER DENYING MOTION FOR RECONSIDERATION AND SCHEDULING HEARING DATES

At a session of said Court held in the Courthouse,
City of Ann Arbor, County of Washtenaw, State of
Michigan on 4/6/2021 __________, [redacted]

PRESENT: Hon. ______________________________
TIMOTHY P. CONNORS
Circuit Court Judge

This matter, having come before the court on Defendant Gelman Sciences, Inc.'s Motion for Reconsideration of the court's previous Order scheduling a hearing on modification of the Consent Judgment and briefs having been filed by the parties and Court having heard oral argument;

IT IS HEREBY ORDERED:

1. Gelman Sciences, Inc.'s Motion for Reconsideration is DENIED for the reasons state on the record.

2. A hearing on implementation of revised cleanup criteria and modification of response activity Orders and Judgments is set for May 3, 4 and 5, 2021 at 9:00 AM.

3. Before commencement of the hearing, counsel for all parties shall submit Briefs and Expert Reports on or before April 30, 2021.

3. This is not a final order and does not close the case.

SO ORDERED.

/s/ Timothy Connors 4/6/2021

Timothy P. Connors
Circuit Court Judge

SEAL OF THE CIRCUIT COURT OF WASHTENAW CO. MICH.

Court of Appeals, State of Michigan

ORDER

Attorney General v Gelman Sciences Inc

Docket No. 356859

LC No. 88-034734-CE

James Robert Redford
Presiding Judge

David H. Sawyer

Douglas B. Shapiro
Judges

The motions for immediate consideration are GRANTED.

The motion for a stay of proceedings pending appeal is DENIED.

The application for leave to appeal is DENIED without prejudice to Defendant-Appellant reasserting its substantive claims after the Washtenaw Circuit Court enters an order or a judgment amending the existing consent judgment. Nothing in this order precludes an interlocutory appeal from any order entered during or subsequent to the evidentiary hearing.

James Robert Redford
Presiding Judge

A true copy entered and certified by Jerome W. Zimmer Jr., Chief Clerk, on

April 29, 2021
Date

Jerome W. Zimmer Jr.
Chief Clerk

STATE OF MICHIGAN

IN THE WASHTNAW COUNTY CIRCUIT COURT

ATTORNEY GENERAL FOR THE
STATE OF MICHIGAN, *ex rel.* MICHIGAN
DEPARTMENT OF NATURAL RESOURCES
AND ENVIRONMENT,

Case No. 88-34734-CE
Hon. Timothy P. Connors

Plaintiff,

and

CITY OF ANN ARBOR, WASHTENAW COUNTY, WASHTENAW COUNTY HEALTH DEPARTMENT, WASHTENAW COUNTY HEALTH OFFICER ELLEN RABINOWITZ, in her official capacity, the HURON RIVER WATERSHED COUNCIL, and SCIO TOWNSHIP,

Intervening Plaintiffs,

-v-

GELMAN SCIENCES, INC., d/b/a PALL LIFE SCIENCES, a Michigan Corporation,

Defendant.

INTERVENING PLAINTIFFS' JOINT BRIEF IN SUPPORT OF AN ADDITIONAL RESPONSE ACTIVITIES ORDER ("2021 ORDER") TO IMPLEMENT REVISED CLEANUP CRITERIA AND TO MODIFY EXISTING RESPONSE ACTIVITY ORDERS AND JUDGMENTS

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INTERVENING PLAINTIFFS' JOINT BRIEF IN SUPPORT OF AN ADDITIONAL RESPONSE ACTIVITIES ORDER ("2021 ORDER") TO IMPLEMENT REVISED CLEANUP CRITERIA AND TO MODIFY EXISTING RESPONSE ACTIVITY ORDERS AND JUDGMENTS

TABLE OF CONTENTS

I. INTRODUCTION

The Intervenors[1] jointly request[2] entry of a new Gelman Response Activities Order ("2021 Order") which would implement revised cleanup criteria set by the State of Michigan and which would modify and largely replace the existing orders and judgments in the case that govern response activities, actions, obligations and duties related to 1,4-dioxane that continues to spread from defendant's ("Gelman") facility located on Wagner Road in Scio Township. The existing orders and judgments are referred to herein, collectively, as the "Current Court Orders."[3] Except as specifically modified by the proposed 2021 Order, the Current Court Orders should remain in full force and effect -- but if there if there is any ambiguity or if there are any conflicts of requirements, the 2021 Order should prevail. As requested, this brief: (i) identifies additions and changes the Intervenors seek; (ii) presents the legal and scientific/technical justification for those changes; and (iii) proposes specific terms to be placed in the 2021 Order.

One of the principal driving forces for replacement of the Current Court Orders was the state's 2016 adoption of new cleanup criteria for 1,4-dioxane, which reduced allowable concentrations by more than an order of magnitude (e.g., from 85 ppb[4] to 7.2 ppb for

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[1] The intervening plaintiffs are the City of Ann Arbor, Washtenaw County, the Washtenaw County Health Department, the Washtenaw County Health Officer, Scio Township, and the Huron River Watershed Council (collectively, "Intervenors").

[2] The City of Ann Arbor does not join in or assert any claims against Gelman in this action that it released in the 2006 "Release of Claims and Settlement Agreement" entered between the City and Gelman. The City asserts in this action only claims reserved to the City in that 2006 settlement.

[3] There is no single, official comprehensive document in the case file that constitutes the Current Court Orders. There have been a series of evidentiary hearings, motions, briefs and arguments, and accordingly as used herein the term "Current Court Orders" means the numerous resulting findings, determinations, orders, judgments and consent judgments that are currently in effect, as modified, which govern response activities related to the Gelman 1,4-dioxane contamination and plumes. For ease of reference purposes the Intervenors have compiled a document which shows changes to the judgments through the Third Amendment to the CJ -- attached as **Ex. A**.

[4] Concentrations expressed as ug/L (micrograms per liter) are equivalent to ppb (parts per billion) in dilute aqueous solutions Therefore, those terms are used interchangeably.

groundwater to be used for residential drinking water; and from 2,800 ppb to 280 ppb for groundwater venting to surface water – at the groundwater to surface water interface "GSI"). This resulted from findings by the State of Michigan that the cleanup criteria for 1,4-dioxane then in effect were **not** protective of public health.

Based on these revised cleanup criteria, the state, Gelman and the Intervenors negotiated to modify and supplement the Current Court Orders. When no more could be achieved through negotiation, the then-proposed document, which was entitled the "Fourth Amended and Restated Consent Judgment" (referred to herein as the "Proposed 4th CJ") was publicly considered and voted upon, but was rejected as insufficient by the Intervenors' governing bodies. The Key Differences between the Current Court Orders and the Proposed 4th CJ are set out in the following Chart:

Chart of Key Differences Between Current Court Orders and Proposed 4th CJ

Issue	**4th CJ Text Location**
New, lower cleanup criterion (85 ppb to 7.2 ppb) incorporated in definition of 1,4-dioxane Groundwater Contamination	III.K (Definitions), p.4
Prohibition Zone (PZ) expanded to cover additional area to account for the reduction in the drinking water cleanup criterion	III.Q (Definitions) and Attachment C (map of new PZ), p.5
Definition of 1,4-dioxane Soil Contamination changed to reflect new, lower cleanup standard (1700 ppb to 500 ppb)	III.W (Definitions), p.6
Removed Maple Road Containment Objective to prevent concentrations of 1,4-dioxane above 2800 ppb (the old GSI criterion) from migrating east of Maple Road, while adding that Gelman must prevent venting of 1,4-dioxane to surface waters in Eastern Area above the new, lower GSI criterion (280 ppb) except in compliance with state law	V.A.1.b (Eastern Area Objectives), p.8
PZ boundary may not be expanded unless clear and convincing evidence that there are compelling reasons expansion is necessary to prevent an unacceptable risk to human health	V.A.2.f (Eastern Area Objectives), p.10

Issue	**4th CJ Text Location**
Requires installation of additional monitoring wells on northern PZ boundary (called Sentinel Wells) and elsewhere on PZ boundary (PZ Boundary Wells) in order to detect and prevent potential breaches of the PZ boundary before they occur; establishes trigger levels which impose additional obligations on Gelman if exceeded (e.g., increased sampling, installation of additional monitoring wells, and provision of municipal water to potentially impacted wells)	V.A.3.a-d (Eastern Area Objectives), pp. 12-13; V.A.4-5 (Eastern Area Objectives), pp.17-22
Requires installation of new Rose and Parklake Wells in order to more than double the rate of groundwater that is pumped and treated. Provides the possibility for treated water from the Parklake Well to be discharged to First Sister Lake	V.A.3.e-f (Eastern Area Objectives), pp. 13-16; V.A.8.g (Eastern Area Objectives), pp. 28-29
Requires installation of three additional monitoring wells/clusters in order to further delineate the migration of 1,4-dioxane downgradient of Maple Road	V.A.5.f (Eastern Area Objectives), p. 22
Creates a PZ boundary review process to occur every five years to determine whether the boundary of the PZ can be contracted	V.A.6 (Eastern Area Objectives), pp. 23-24
Requires Gelman to prevent venting of 1,4-dioxane to surface waters in Western Area above the new, lower GSI criterion (280 ppb), except in compliance with state law	V.B.2 (Western Area Objectives), pp. 31-32
Requires installation of six additional monitoring wells/clusters in order to further delineate the migration of 1,4-dioxane in the Western Area	V.B.3.b (Western Area Objectives), pp. 33-34
Removes the Little Lake Area System objective of non-expansion of the horizontal extent of groundwater contamination; removed because this system would now be included within the Western Area for purposes of the Western Area Objectives	N/A
Creates more robust Western Area compliance well verification process to ensure that Western Area objectives are met	V.B.4 (Western Area Objectives), pp.36-40
Removed the requirement to investigate former spray irrigation area on Gelman property in order to ensure meeting objective of preventing 1,4-dioxane from venting to Third Sister Lake in excess of 2800 ppb; removed because this area is now included within the Western Area for purposes of the Western Area Objectives and the investigation has already occurred	N/A

Issue	**4th CJ Text Location**
Requires installation of three additional extraction wells in Gelman Property source area at a combined purge rate of ~75 gallons per minute (gpm), with the potential to install three additional extraction wells if required by EGLE	VI.C.1 (Gelman Property Response Activities), pp. 46-48
Requires operation of a phytoremediation system in the source area, which involves planting trees in order to remove 1,4-dioxane via biodegradation and transpiration and extract perched [i.e., not connected to an aquifer] groundwater	VI.C.2 (Gelman Property Response Activities), pp.48-49
Requires operation of a heated soil vapor extraction system (HSVE) in the source area in order to reduce the mass of 1,4-dioxane in the soil, and placement of two impervious barriers in order to inhibit water from percolating through the soils (these requirements replace the previous soils system objective and plan); HSVE technology involves heating the soil to cause 1,4-dioxane to better volatilize and then extracting the resulting vapors	VI.C.4 (Gelman Property Response Activities), pp. 49-51

The added requirements in the Proposed 4th CJ are all technically and scientifically necessary and appropriate response activities [See, Intervenors' Expert Opinion Report ("Int Exp Rept"), p.5] that must be required of Gelman under Part 201 of the Michigan Natural Resources and Environmental Protection Act, MCL 324.20101, et seq. (Part 201), but they are technically and scientifically insufficient [Id.], and in some respects, unacceptable. In this brief, the Intervenors identify specific issues and concerns that go beyond the Proposed 4th CJ (including additional delineation, additional plume management, additional Prohibition Zone controls, and additional mass removal efforts) that need to be addressed, with revised and new terms, and provide the legal and scientific support for those additional terms. For convenience of reference, the following table summarizes the scientific and technical topics of concern, the proposed modifications or additions of requirements beyond the Proposed 4th CJ to address those issues, and the expert scientific justification/opinion supporting the requests. There are a few additional legal and process topics, related to the Intervenors' ongoing involvement in the case, which are discussed at the end of the brief.

Summary Table of Intervenor Concerns and Solutions for Inclusion in 2021 Order [Showing Proposed Modifications and Additions to Terms of Proposed 4th CJ]

Intervenor Concern	Proposed New Requirement for 2021 Order	What this would Achieve	Technical/Scientific Justification	Primary Expert
Incomplete delineation of groundwater contamination	**1A.** Semiannual maps showing extent of 1,4-dioxane concentrations at 1, 7.2, and 280 ppb	Provide a basis for assessing efficacy of remedial actions and assessing risk of future impacts to drinking water wells	Up-to-date maps depicting the extent of 1,4-dioxane contamination are essential for assessing attainment of remedial objectives.	Lemke
Perimeter monitoring well gaps	**1B.** Two additional Sentinel wells along northern PZ boundary (AA, BB); and replacement well for MW-63 (CC)	Reduce spacing between monitoring wells in key areas of concern	1,4-dioxane is known to migrate along narrower pathways in this complex aquifer system; these wells will reduce the likelihood that such plumes are not detected	Lemke
Size of prohibition zone expansion	**1C.** More limited PZ expansion to the south	Appropriate buffer to account for uncertainty commensurate with reduction from 85 to 7.2 ppb	Expansion proportional to concentration gradient along southern edge of plume; expansion aligned with expected migration path	Lemke
Northward migration toward Barton Pond	**1D.** Three additional monitoring wells north of PZ boundary (DD, EE, FF)	Determine aquifer quality, hydraulic gradient, and presence/absence of 1,4-dioxane in this area	Reliable information is needed to assess the potential for northward migration and put community concerns to rest	Lemke
Discharge to Allen Creek at concentrations exceeding the GSI criterion	**2A.** Two high resolution transects (T_1-T_1’ and T_2-T_2’)	Identify zones of high 1,4-dioxane concentrations migrating at all depths above bedrock that will guide additional remedial actions	High resolution transects are commonly used to quantify mass flux and design remedial strategies	Lemke
	2B. Two additional downgradient investigation monitoring wells (GG, HH)	Delineation of 280 ppb extent in the downgradient Eastern Area	Determine if 1,4-dioxane is venting to Allen Creek from north or south; detect 1,4-dioxane migration further downgradient in artesian area	Lemke
	2C. Shallow groundwater profiling and monitoring along Allen Creek Drain	Delineate contamination at or above GSI on north and south flanks of Allen Creek Drain	Ensure “Groundwater-Surface Water Interface Objective” is met	Gadway / Lemke
500 ppb extraction well termination criterion is too high	**3A.** Terminate extraction after pumping no longer contributes to	Extend benefits of additional mass removal	Extraction well concentrations may not reflect maximum concentrations in the	Lemke

Intervenor Concern	Proposed New Requirement for 2021 Order	What this would Achieve	Technical/Scientific Justification	Primary Expert
	beneficial reduction in 1,4-dioxane mass		surrounding aquifer.	
Public opposition to Parklake Well discharge into First Sister Lake / NPDES permit risk	**3B.** Pipe treated water to the Gelman Property and discharge under existing NPDES permit	Avoids NDPES permit risk while providing flexibility, and avoids potential adverse environmental impacts.	200 GPM exchanges the volume of First Sister Lake approximately once each month, giving rise to potential adverse environmental impacts.	Lemke
Limited reach of Source Area extraction wells pumping at low rates in low conductivity zones	**3C.** Concurrent pump-and-treat from 6 or more purge well locations on the Gelman property	Accelerating pumping from the shallow aquifer underlying the Source Area maximizes mass removal in the shortest time frame	Given demonstrated aquifer heterogeneity, wells distributed throughout the Source Area make sense, and there is no compelling reason to wait.	Gadway
Performance monitoring criteria have not been specified for the phytoremediation systems – How will we know if they are working?	**3D.** Gelman to develop phytoremediation effectiveness verification plans including monitoring groundwater 1,4-dioxane concentrations, water table elevations, and 1,4-dioxane in plant tissue	Ensure that the phytoremediation systems are achieving groundwater table control and mass removal objectives	This is relatively new technology. Performance monitoring is needed to demonstrate effectiveness of phytoremediation systems and verify that the Western Area GSI Objective is attained.	Gadway
Potential enhancements can be incorporated into the HSVE system design	**3E.** Install permanent cap prior to HSVE operation and cycle HSVE system before termination.	More efficient HSVE system operation and avoidance of premature termination	The HSVE system will operate more effectively with a cap in place. System cycling if exhaust air concentrations become asymptotic will demonstrate HSVE has reached its effective limit.	Gadway
Documented presence of 1,4-dioxane in Allen Creek, Third Sister Lake, unnamed tributary to Honey Creek	**4A.** Annual sampling of surface water bodies and drainage systems	Detection will trigger investigation to determine risk of exceeding the GSI criterion	Changes indicating venting of groundwater with 1,4-dioxane at new locations or rising concentrations will not be detected without regular surface water body testing.	Lemke
Western Area Non-Expansion Cleanup Objective verification threshold is too high	**4B.** Reduce exceedance threshold from 7.2 to 3.5 ppb	Expansion of Western Area groundwater contamination will be detected before it has migrated to the compliance well location	An increase in concentrations to 7.2 ppb at a compliance well is evidence that expansion of the horizontal extent of contamination has already taken place.	Lemke

Intervenor Concern	Proposed New Requirement for 2021 Order	What this would Achieve	Technical/Scientific Justification	Primary Expert
Inconsistent requirements to initiate and subsequently scale back response activities based on threshold exceedances	**4C.** Adopt a consistent three-month-in-a-row requirement to initiate or cease responses at Sentinel, Boundary, and Compliance Wells	A three-in-a-row requirement to both initiate and interrupt remedial activities is more consistent and more protective	Statistical variation is just as likely to result in low concentration measurements as high concentration measurements.	Lemke
1,4-dioxane detections in residential drinking water wells	**4D.** Municipal Water Connection Contingency Plan (MWCCP) for Breezewood Ct; three-in-a-row requirement to stop bottled water supply	Proactive planning for Breezewood Ct residents (same as Elizabeth Rd); More consistent and protective bottled water requirements	1,4-dioxane has been detected in a residential well on Breezewood Ct (just like Elizabeth Rd). The same protections should be afforded there. Three-in-a-row is consistent with response activity threshold frequencies in 4C.	Lemke
	4E. Use of EPA Method 522 to analyze water from residential wells within 1,000 feet of the mapped limit of 1,4-dioxane contamination	Lower analytical method detection limits for residential water well samples near the plume will give a greater sense of confidence to homeowners	Use of EPA Method 522 for the analysis of drinking water from wells in close proximity to the plume is consistent with the requirements imposed on operators of public drinking water supplies.	Gadway
Gaps, inconsistencies, and delays accessing Gelman analytical data	**4F.** Provide universal access to the Gelman database via a cloud-based system for all monitoring well, extraction well, and NPDES treatment and discharge activity information; Release copies of source area environmental and engineering studies	A single database containing all relevant analytical information associated with monitoring, extraction, and permitted discharges will ensure that all parties are viewing and making decisions based on the same information	Accurate and timely access to site data are needed by all stakeholders including Gelman, EGLE, and the general public. Prior environmental and pilot engineering studies are essential for understanding the basis for selected source area remedies.	Lemke

II. FACTUAL AND PROCEDURAL BACKGROUND

When devising a 2021 Order to supplement and modify the response activities required by the Current Court Orders, we are not writing on a clean slate. Not only is this case scientifically challenging, it also has a long, complex procedural and legal history. This lawsuit was initiated in 1988, more than 32 years ago. During the ensuing years, the statutes serving as the basis for the state's claims have been changed, new statutes were enacted, even the new statute (Part 201) has been amended several times, the cleanup criteria for 1,4-dioxane have been changed several times, there have been evidentiary hearings, findings of fact and conclusions of law determined by court action, there have been settlements of certain disputed matters by agreement of the parties, there have been bargained waivers of certain claims or rights by parties in order to achieve other objectives, and there have been determinations and rulings by the Court's opinions and orders when no agreement could be reached on certain issues. Sometimes judgments have been entered by consent of the parties and other times orders or judgments have been entered by the Court's own determinations, regarding matters which were strenuously disputed. All of the foregoing add up to what now constitutes the Current Court Orders in the case.

In creating the 2021 Order, one must recall that some provisions in the Current Court Orders are the product of bargaining, by which a party relinquished or waived certain claims/rights in order to achieve other important goals. What was achieved in those trades should not be extinguished as a 2021 Order is created. The appropriateness of the proposed terms of a 2021 Order should be viewed in light of the long history of the case.

The main components of the Current Court Orders and what led up to each (up through what is titled the "Third Amendment to Consent Judgment") are summarized in the following sub-sections and are incorporated into the meaning of that term as used in this brief.

A. The original action and resulting consent judgment and initial amendments (1988 – 1999).

The State of Michigan brought this action in 1988 to address 1,4-dioxane that Gelman dumped or sprayed into the environment between 1966 and 1986, resulting in widespread contamination of the surrounding soil and groundwater. The contamination has continued to spread from the "Gelman Property" on Wagner Road, and multiple groundwater contaminant plumes now stretch more than four miles under Scio Township and the City of Ann Arbor. Int Exp Rept p. 5. 1,4-dioxane is completely soluble (or miscible) in water and is held together by strong molecular bonds that prevent it from breaking down readily in groundwater.

In 1992, the Court entered the original consent judgment which required Gelman to remove and treat all of the contaminated groundwater. In 1996, the Court entered the First Amendment to Consent Judgment, which revised the cleanup criteria in the consent judgment so that they were consistent with the cleanup criteria developed under Part 201 of Michigan's Natural Resources and Environmental Protection Act (MCL 324.20101 et seq.) ("Part 201"), a statute that had recently been enacted to regulate contaminated sites in Michigan.[5] In 1999, the Court entered the Second Amendment to Consent Judgment, which provided for alternate disposal methods for certain purged groundwater.

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[5] Michigan's Department of Environment, Great Lakes, and Energy ("EGLE") establishes cleanup criteria under Part 201, which are the numerical criteria for hazardous substances that, in EGLE's judgment, are required for response activities to be protective of public health, safety, welfare, and the environment. MCL 324.20120a; Mich Admin R 299.3.

B. The REO – the Court enters a supplemental order to require additional response activities (2000).

In 2000, the Court entered its Opinion and Remediation Enforcement Order ("REO"). **Ex. B**. The REO resulted from EGLE's motion to enforce the Consent Judgment and three days of evidentiary hearings. The Court ruled:

> It is also clear, however, that that purging of 1,4-dioxane has not occurred fast enough to provide the public, or the Court, with assurance that the plume of 1,4-dioxane was contained as early as it should have been or that there is an ongoing approved plan that will lead to the removal of unlawful levels of this pollutant from the area's water supplies.
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> * * *
>
> Based upon the evidence submitted, this Court is going to grant equitable relief in the sense that the Court will use its equitable powers to enforce the consent judgment to insure that 1,4-dioxane levels in these water supplies is brought within acceptable standards as soon as possible. Both sides in this dispute appear to need the intervention of the Court to keep them moving toward this goal.

Id., pp. 2, 3.

The Court required Gelman to, among other things, (1) submit a detailed plan to reduce 1,4-dioxane in all affected water supplies below legally acceptable levels within a maximum period of five years; (2) install additional monitoring and extraction wells; (3) install an additional ultraviolet treatment unit; and (4) increase the pumping rate in existing extraction wells. *Id.*, pp. 4-5. The Court established tight timeframes for each requirement. For example, Gelman's detailed plan was due within 45 days of the order. *Id.*, p. 4. Gelman did not appeal the REO. Instead, it prepared the Five Year Plan as directed in the REO and, on January 10, 2001, by stipulation of the parties, the Court approved the plan, which required Gelman to remediate 1,4-dioxane in the groundwater to concentrations below the cleanup criteria then in effect.

C. The Unit E and Prohibition Zone Orders – the Court establishes the Prohibition Zone after Gelman discovers the plume had migrated in an unanticipated way (2001 – 2005).

In 2001, Gelman discovered that 1,4-dioxane had migrated to a deeper aquifer which the parties called "Unit E." EGLE and Gelman disagreed over how to address the contamination and the parties presented the issue to the Court for decision. The fundamental disagreement between the parties was whether Gelman would be required to comply with the aquifer protection rules and, if not, what conditions Gelman would need to satisfy. The aquifer protection rules impose stringent requirements concerning contamination of groundwater in aquifers:

> (5) The horizontal and vertical extent of hazardous substance concentrations in an aquifer above the higher of either the concentration allowed by section 20120a(1)(a) [i.e., the generic residential cleanup criteria] or (10) [i.e., the target detection limit or background concentration] of the act, as applicable, shall not increase after the initiation of remedial actions to address an aquifer, except as approved by the director as provided in section 20118(5) and (6) of the act.
>
> (6) All remedial actions that address the remediation of an aquifer shall provide for removal of the hazardous substance or substances from the aquifer, either through active remediation or as a result of naturally occurring biological or chemical processes which can be documented to occur at the facility, except as provided in section 20118(5) and (6) of the act.

Mich Admin R 299.3. Simply stated, the aquifer protection rules require "…removal of hazardous substances from the aquifer … through active remediation…" and prohibit expansion of such hazardous substances exceeding residential cleanup criteria after the initiation of cleanup. Section 20118(5) and (6) of Part 201, referenced in the aquifer protection rules, provide that EGLE can waive compliance with the rules in very limited situations. MCL 324.20118.

EGLE concluded in its Decision Document for addressing the Unit E plume that:

> [E]xtracting and treating contaminated groundwater in the vicinity of Wagner Road and Maple Road, coupled with capture of the "leading edge" of contamination is necessary to comply with Part 201, and the Consent Judgment. The performance objectives for the groundwater extraction in the vicinity of Maple Road, the vicinity of Wagner Road, and for the leading edge are that, once initiated, a hydraulic barrier should be created to halt the further migration of concentrations of 1,4-dioxane above 85 ppb [the drinking water cleanup criterion] in the downgradient or easterly direction.

Ex. C, p. 2. Nevertheless, EGLE determined that, if Gelman instead satisfied six conditions, capture of the leading edge of the plume would not be necessary. *Id*. One of those six conditions was "[p]revention of any further migration of 1,4-dioxane contamination beyond Maple Road in excess of 2,800 ppb (the criterion protective of surface water)." *Id*., p. 12.

Gelman's preferred alternative to address Unit E relied on an institutional control to prevent consumption of contaminated groundwater. Gelman argued that the Court had the power to issue such a control based on the Court's inherent authority to enforce its judgments and issue any order to fully execute its judgments. **Ex. D**, Supp. Filing in Support of Remedial Alternative, p. 5-6, citing MCL 600.611, *Cohen v Cohen*, 125 Mich App 206 (1983), and *Spurling v Battista*, 76 Mich App 350 (1977). Gelman also agreed to prevent migration of 1,4-dioxane downgradient of Maple Road in excess of 2,800 ppb. *Id*., p. 8-9.

In 2004, the Court entered its Opinion and Order Regarding Remediation of the Contamination of the "Unit E" Aquifer ("Unit E Order"). **Ex. E**. The Court first addressed the questions the parties had raised "about the applicability of the Consent Judgment to Unit E, the responsibility of the Court to review EGLE actions, and the scope of the Court's role in this process." *Id*., p. 3. The Court found that the Unit E plume was subject to the consent judgment and that the Court "has the inherent and equitable powers to enforce its judgment with all appropriate measures and sanctions as to Unit E contamination." *Id*., p. 4. The Court further determined that it had broad authority to review EGLE actions and broad powers to assure that the cleanup of the 1,4-dioxane was achieved "as soon as possible." *Id.* p. 4-5.

The Court then found that "[t]he goal set by the [EGLE] of total capture of the width of the plume is certainly appropriate – if it can be done….[T]he primary [EGLE] rationale is that controlling groundwater contamination at or near its source is more efficient than trying to capture it later as it spreads through the aquifer. There is ample support for that position." *Id.* p. 7-8. The Court ordered Gelman to perform an investigation and submit a work plan to EGLE which would, "to the maximum extent feasible, prevent further migration of groundwater contamination above 85 ppb of 1,4-dioxane [the drinking water standard at the time] eastward into the Unit E aquifer." *Id.*, p. 9.

The Court then moved to address the contamination that had already spread eastward into the Unit E aquifer. It first observed that although it would not be possible to extract all 1,4-dioxane from the aquifer, "the goal must be to remove as much of the contaminant as possible, as quickly as possible, so that the ultimate dilution will take place with minimal impact on the water resource." *Id.* The Court then addressed the dispute between the parties over the conditions that EGLE required to grant a waiver from the aquifer protection rules. One of those conditions was use of an institutional control to restrict groundwater use. The Court directed the parties to submit an order establishing an area where use of groundwater would be prohibited. The Court later entered such an order in 2005, titled Order Prohibiting Groundwater Use ("Prohibition Zone Order"). **Ex. F**. It was that order that first established the "Prohibition Zone." Finally, the Unit E order required Gelman to submit a work plan to MDEQ within 30 days from the Unite E Order for the treatment and reinjection of Unit E water. **Ex. E**, p. 9-10. The Court directed that the work plan "will be designed to purge enough water so that any water escaping from the purging zone in Unit E will not exceed 2,800 ppb recommended by [EGLE]." *Id.*, p. 10.

D. The Consent Judgment is amended a third time to address new cleanup criteria and increased knowledge of the contamination (2009 – 2011).

In 2009, the Court entered an Order Regarding Potential Remedial Modifications. **Ex. G**. The Court observed:

> The parties have decided to explore possible modification of the cleanup program that incorporates a coherent remedial approach to the groundwater contamination and reflects changes in state environmental law over time, the parties' current knowledge of site conditions, and the previous rulings of the Court. Over the last number of months, the parties have been discussing potential modifications to the cleanup program. The goal of any modifications will be to continue to protect the public while increasing the effectiveness and efficiency of the cleanup.

Id., p. 2. At the time, the parties were still working on the proposed modifications but were considering the following cleanup objectives: (1) prevention of contamination from migrating past the Prohibition Zone boundaries, as established by the Court in 2005; (2) monitoring of the migration of contamination within the Prohibition Zone to ensure that it does not expand beyond the Prohibition Zone or underflow the Huron River; (3) continued mass removal through continued operation of existing extraction wells and installation of at least one additional extraction well; and (4) continued prevention of "groundwater with concentrations exceeding 2,800 ppb (the groundwater/surface water interface criterion) from migrating east of Maple Road in order to insure that levels above the GSI criterion do not reach the Huron River." *Id.*, p. 3-4. The Court established a schedule for considering the proposed modifications.

After additional negotiations between the parties, in 2011 the Court entered the Third Amendment to Consent Judgment. **Ex. H**. The Third Amendment implemented a number of changes to the cleanup regime, including revisions to the cleanup criteria and expansion of the Prohibition Zone. The Third Amendment also divided the cleanup program into two main systems, Western Area and Eastern Area, based on the location of the remedial activities in relation to Wagner Road. The Maple Road containment objective, which the parties previously negotiated as part of the establishment of the original Prohibition Zone, was expressly maintained:

> The current Unit E objective set forth in the Unit E Order of preventing contaminant concentrations above the groundwater-surface water interface criterion of 2,800 ug/l (subject to approval by the Court of the application of a new criteria) from migrating east of Maple Road shall apply to the Eastern Area System, regardless of the aquifer designation, or depth of groundwater or groundwater contamination.

Id, p. 4-5. The Third Amendment also required Gelman to meet the generic GSI criterion at other points (e.g., the Honey Creek Tributary and Third Sister Lake, see, *id*., p. 24-25), even though Part 201 allowed application of mixing-zone based criteria.[6] See, 228 PA 2010.

Although the Prohibition Zone was expanded, the Third Amendment required Gelman to maintain the integrity of the expanded boundary and, in particular, the parties agreed that "any further expansion of the northern boundaries of the Prohibition Zone or Expanded Prohibition Zone…should be avoided, unless there are compelling reasons to do so." **Ex. H**, p. 6.

In connection with entry of the Third Amendment, the Court entered a Stipulated Order Amending Previous Remediation Orders. **Ex. J**. That Order recited that "the Court has also supplemented the Consent Judgment with several cleanup related orders, based on information about the nature and extent of contamination acquired after the Consent Judgment and the Amendments were entered," including the REO, Unit E Order, and the Prohibition Zone Order. *Id*., pp. 2-3. The Order further recited that "[s]ince entry of the REO and the Unit E Order, the parties have further refined their understanding of the nature and extent of contamination at the

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[6] "A mixing zone is an allocated portion of the receiving surface water body where venting groundwater discharge is mixed with surface waters. The mixing zone is used to develop mixing zone-based GSI criteria." **Ex. I**, RRD Policy and Procedure No. 33, p. 2.

Gelman Site, which is reflected in the Third Amendment." *Id.*, p. 3. The Order did not supersede the prior cleanup orders in full; it simply provided that the Third Amendment would control in the event of an inconsistency with the prior cleanup orders. *Id.*

E. The state significantly lowers cleanup criteria and negotiations begin over a fourth amendment to the Consent Judgment (2016).

In October 2016, EGLE released the results of a shallow groundwater investigation, revealing the presence of 1,4-dioxane in two test wells in a residential area just west of downtown Ann Arbor.

Almost immediately after this discovery, EGLE issued a "finding of emergency":

> Releases of 1,4-dioxane … **pose a threat to public health, safety or welfare of its citizens and the environment.** Recent shallow groundwater investigations in the Ann Arbor area have detected 1,4-dioxane in the groundwater in close proximity to residential homes…. The extent of 1,4-dioxane groundwater contamination … is unknown; and 1,4-dioxane contamination is expected to be present beneath many square miles of the City of Ann Arbor occupied by residential dwellings. **[T]he current cleanup criteria … are not protective of public health.** **Ex. K** (emphasis added).

As part of its emergency order, EGLE imposed stricter cleanup criteria. *Id.* Prior to the emergency order, the 1,4-dioxane cleanup criterion for drinking water was 85 ppb. EGLE concluded that standard to be "outdated and not protective of public health," and tightened the criterion, on an emergency basis, to 7.2 ppb. *Id.* EGLE later published rules making the change to 7.2 ppb permanent, and lowering the GSI criterion from 2,800 ppb to 280 ppb.

In light of these events, EGLE and Gelman began negotiating a further amendment to the consent judgment. The Court later granted Intervenors' petitions to intervene and the parties (EGLE, Gelman and the Intervenors) engaged in lengthy settlement negotiations, culminating in the Proposed 4th CJ (attached as **Ex. L**) that the Court made public in an August 31, 2020 Order. Some of the most significant changes to the Current Court Orders included in the Proposed 4th

CJ include: (1) expansion of the Prohibition Zone boundary to account for the reduction in the drinking water standard from 85 ppb to 7.2 ppb; (2) installation of new monitoring wells to further investigate the migration of 1,4-dioxane; (3) establishment of trigger levels to serve as an early warning system and require action to prevent the migration of contamination beyond the Prohibition Zone boundary before it occurs; (4) installation of multiple new extraction wells; and (5) implementation of new remediation techniques on the Gelman property (phytoremediation and heated soil vapor extraction). See Summary of Key Differences Chart at pp. 2-4, supra.

After an extensive public comment period, the governing bodies of the Intervenors voted not to approve the Proposed 4th CJ. There were numerous reasons for the rejection, but primarily the Intervenors wanted more extraction of 1,4-dioxane from the aquifers, believed that extraction and treatment of groundwater from the proposed Parklake extraction well was appropriate but did not believe the treated water should be discharged to First Sister Lake, wanted delineation of the plume to the drinking water standard of 7.2 ppb and wanted more monitoring wells to detect further migration of the plumes. After the votes, the Court held a status conference on November 19, 2020 at which it directed the parties to explain at a hearing before the Court how they believe the existing cleanup regime should be modified, and to provide the legal and technical justifications for their positions.

III. LEGAL FRAMEWORK FOR GELMAN RESPONSE ACTIVITIES

Gelman's obligations in this matter stem from two main sources: (1) the statute itself, Part 201; and (2) the Current Court Orders. The Current Court Orders require Gelman to undertake various response activities to address the 1,4-dioxane that originated from the Gelman site. However, none of the Current Court Orders has been revised to reflect the 2016 revisions to the cleanup criteria. Similarly, Part 201 imposes various obligations on a party to address contamination for which the party is liable.

The Third Amendment to Consent Judgment (which is part of the Current Court Orders) provides various means by which the Court can modify the existing cleanup regime. For example, Article XVI, which was in the original Consent Judgment and has continued through the Third Amendment, provides that the Court is the ultimate arbiter of disputes between EGLE and Gelman. These disputes include substantive modifications to the cleanup regime (e.g., EGLE's choice of alternatives to address the possibility that contamination is going to migrate outside of the Prohibition Zone, see Third Amendment, p. 6). The consent judgment also gives EGLE the right to ask the Court to order additional response activities if, for example, new information comes to light concerning the contamination or EGLE adopts more restrictive cleanup criteria. Third Amendment, p. 30. The Court also has inherent and equitable powers to enforce its judgments and orders. EGLE and Gelman each have invoked these powers at various times over the course of this case when seeking the Court's intervention and the Court has relied on those powers to, for example, enter supplemental remediation orders (i.e., the REO, the Unit E Order, and the Prohibition Zone Order).

Part 201 is the current, primary statutory framework for remediation of contaminated property in Michigan.[7] The remedial obligations under Part 201 are focused in large part on "liable parties." One of the classes of liable parties under Part 201 is "[t]he owner or operator of a facility if the owner or operator is responsible for an activity causing a release or threat of release." MCL 324.20126(1)(a). A "facility" is defined as "any area, place, parcel or parcels of property, or portion of a parcel of property where a hazardous substance in excess of the concentrations that satisfy the cleanup criteria for unrestricted residential use has been released,[8] deposited, disposed of, or otherwise comes to be located." Gelman's property is a "facility" because hazardous substances, including 1,4-dioxane, have been released, deposited, and disposed of in excess of cleanup criteria. Gelman is liable for the contamination on its property because it owns and operated the property and is responsible for an activity causing a release.

A liable party must, among other things, "determine the nature and extent of the release at the facility," "[i]mmediately stop or prevent an ongoing release at the source," and "diligently pursue response activities[9] necessary to achieve the cleanup criteria established under [Part 201]." MCL 324.20114(1)(a), (c), (g). A liable party is jointly and severally liable for response

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[7] The Third Amendment incorporates by reference several provisions of Part 201. See, e.g., Third Amendment, p. 11, referring to the authority of the Court under MCL 324.20135a to grant a liable party access to property in order to conduct response activities.

[8] "Release" broadly includes "any spilling, leaking, pumping, pouring, emitting, emptying, discharging, injecting, escaping, leaching, dumping, or disposing of a hazardous substance into the environment, or the abandonment or discarding of barrels, containers, and other closed receptacles containing a hazardous substance." MCL 324.20101(1)(pp).

[9] "Response activity" includes "evaluation, interim response activity, remedial action, demolition, providing an alternative water supply, or the taking of other actions necessary to protect the public health, safety, or welfare, or the environment or the natural resources. Response activity also includes health assessments or health effect studies carried out under the supervision, or with the approval of, the department of community health and enforcement actions related to any response activity." MCL 324.20101(1)(vv). "Remedial act" includes "cleanup, removal, containment, isolation, destruction, or treatment of a hazardous substance released or threatened to be released into the environment, monitoring, maintenance, or the taking of other actions that may be necessary to prevent, minimize, or mitigate injury to the public health, safety, or welfare, or to the environment." MCL 324.20101(1)(qq).

activity costs incurred by the state or another person and damages to natural resources. MCL 324.20126a.

In selecting or approving a remedial action, the following must be considered:

> (a) The effectiveness of alternatives in protecting the public health, safety, and welfare and the environment.
>
> (b) The long-term uncertainties associated with the proposed remedial action.
>
> (c) The persistence, toxicity, mobility, and propensity to bioaccumulate of the hazardous substances.
>
> (d) The short- and long-term potential for adverse health effects from human exposure.
>
> (e) Costs of remedial action, including long-term maintenance costs. However, the cost of a remedial action shall be a factor only in choosing among alternatives that adequately protect the public health, safety, and welfare and the environment, consistent with the requirements of section 20120a.
>
> (f) Reliability of the alternatives.
>
> (g) The potential for future response activity costs if an alternative fails.
>
> (h) The potential threat to human health, safety, and welfare and the environment associated with excavation, transportation, and redisposal or containment.
>
> (i) The ability to monitor remedial performance.
>
> (j) For remedial actions that require the opportunity for public participation under section 20120d, the public's perspective about the extent to which the proposed remedial action effectively addresses requirements of this part.

MCL 324.20120(1).

Where EGLE determines that "there may be an imminent and substantial endangerment to the public health, safety, or welfare, or to the environment because of an actual or threatened release from a facility," Part 201 authorizes the attorney general to bring an action against a liable party to secure appropriate relief and "the court has jurisdiction to grant such relief as the public interest and the equities of the case may require." MCL 324.20126a(6).

IV. REQUESTED MODIFICATIONS TO GELMAN'S RESPONSE ACTIVITIES FOR THE PROPOSED "2021 ORDER"

The existing cleanup regime under the Current Court Orders does not adequately address the Gelman 1,4-dioxane contamination. The changes in the Proposed 4th CJ are improvements and are necessary, but they still are technically and scientifically insufficient. Int Exp Rept, p. 5. To assure that Gelman's response activities are sufficient, to the extent of current scientific knowledge, the court should issue a "2021 Order" that requires Gelman to perform all aspects of the Proposed 4th CJ, but with the additions and modifications described below. For each requested modification, the Intervenors provide (1) an explanation of the response activity topic at issue and the language in the Proposed 4th CJ that addresses that activity, (2) an explanation of the legal and scientific basis for the modification, and (3) proposed language to add to or modify what is in the Proposed 4th CJ to effectuate the modification. A document incorporating all of the combined language into a proposed 2021 Order is attached as **Ex. M**.

A. Additional Delineation of Gelman's 1,4-Dioxane is Necessary.

1. Gelman's plumes have not been delineated adequately under the Current Court Orders.

As described in earlier sections, Part 201 requires a liable owner/operator of a facility, among other things, to "…determine the nature and extent of the Release at the facility." MCL 324.20114(1)(a). In other words, Gelman must test and determine where (how far, how wide and how deep) the 1,4-dioxane has spread from its property, and where it will go next. This is referred to as "delineation" of the plume(s) of contamination. Knowing the nature and extent of the 1,4-dioxane plumes is necessary to design and implement effective remedial measures to contain and cleanup the contamination. Int Exp Rept, p. 6.

As professor Lemke explained (at Int Exp Rept, p. 6), Gelman's 1,4-dioxane plumes have not been adequately delineated for two principal reasons. First, neither the state nor Gelman publicly has used current monitoring well system data to determine the extent of the plumes, and depict them on groundwater plume maps, under Michigan's new groundwater cleanup criteria (e.g., 7.2 ppb for drinking water and 280 ppb for GSI). Second, even if new plume maps were prepared, the gaps between the existing monitoring wells are too large to assure they are accurate. Simply stated, narrow plumes in this heterogeneous aquifer system easily may have slipped undetected through those gaps.

Over the decades Gelman's 1,4-dioxane plumes have proved to be elusive. Because of the heterogeneous geology below ground, narrow plumes have moved in a variety of unpredictable directions and at varying depths. The Intervenor experts describe the scientific circumstances as follows:

> The glacial aquifer system affected by the Gelman 1,4-dioxane contamination is highly heterogeneous, consisting of a complicated mixture of very permeable sand and gravel units interspersed with less permeable silts and clays making it difficult to determine connected

> groundwater flow pathways. As a consequence, the plumes have moved in a variety of directions and at different depths, making it difficult to predict contaminant movement. Int Exp Rept, p. 5.

Several times, Gelman's experts have discovered that their beliefs, of the extent and migration pathways of the 1,4-dioxane contamination, were incorrect. As a prime example, in 2001 Gelman discovered a previously unknown and unexpected 1,4-dioxane plume, following a pathway deeper under the ground, that had migrated into the Evergreen subdivision. That eventually led to the court's 2004 Unit E Order. **Ex. E**. Even now, nearly two decades later, under the Current Court Orders (see, in particular, the Third Amendment to the Consent Judgment), from a scientific/technical perspective one cannot conclude that the Gelman 1,4-dioxane plumes have been delineated adequately because large gaps are present in the monitoring well system. As noted in the Intervenor experts' opinion, in 2016 EGLE concluded that the extent of Gelman's contamination was unknown and to date that lack of knowledge has not been remedied:

> At the present point in time, the extent of groundwater contamination (i.e., 1,4-dioxane concentrations at 7.2 ppb or more) emanating from the Gelman Site has not been fully defined. When promulgating emergency rules setting the 7.2 parts per billion (ppb) 1,4-dioxane residential drinking water cleanup criterion in 2016, EGLE (then MDEQ) stated: "The extent of 1,4-dioxane groundwater contamination…greater than 7.2 parts per billion is unknown (MDEQ, 2016)."
>
> Since that time, neither Gelman's technical experts nor EGLE's technical experts have publicly presented a map showing 7.2 ppb or 1.0 ppb (the analytical detection limit) concentration lines based on currently available data. Consequently, we have relied upon maps generated by our own technical consultants and the Washtenaw County Health Department. Uncertainty in the present-day distribution of 1,4-dioxane and the location of 1,4-dioxane migration pathways gives rise to four primary Intervenor concerns regarding the proposed Fourth Amended and Restated Consent Judgment (Proposed 4th CJ):
>
> 1A. Contaminant delineation maps
> 1B. Perimeter monitoring well gaps
> 1C. Unwarranted Prohibition Zone expansion

1D. Northward migration toward Barton Pond. Int Exp Rept, p. 6.

Suggested language for implementing proposed additional requirements is provided in section IV.A.5 of this brief, below.

2. New updated plume maps should be required.

The Proposed 4th CJ does not contain any requirement that Gelman prepare and update plume maps on a continuing basis. The Intervenor experts explain why having such updated plume maps are critical to determine remedial measures and to evaluate their effectiveness:

> **Scientific Rationale.** Up-to-date maps depicting the extent of 1,4-dioxane contamination are essential tools needed by all stakeholders including Gelman, EGLE, and the general public. Such maps provide a basis for assessing attainment of remedial objectives, assuring compliance with regulatory standards, evaluating the efficacy of remedial activities, documenting changes in contaminant distributions over time, and evaluating risks of future impacts on drinking water supply wells in the surrounding communities.
>
> Given the frequency with which monitoring wells are sampled across the Gelman Site, semi-annual updates such as those currently provided in Quarterly Reports are appropriate and should be required as part of any court order providing comprehensive requirements that are necessary to address the Gelman dioxane. Int Exp Rept, p. 6.

To correct this deficiency, plume maps should be required, and updated semi-annually, which depict the extent of contamination, at 280 ppb, 7.2 ppb and 1 ppb. Suggested language for implementing such requirements is provided in section IV.A.5 of this brief, below.

3. The new Monitoring Well clusters[10] required in the Proposed 4th CJ are necessary but insufficient.

The Proposed 4th CJ would require several additional monitoring well clusters in the Eastern Area, in the Downgradient Area Within the Prohibition Zone ("PZ"), and in the Western Area, all of which will improve delineation of the nature and extent of the Gelman 1,4-dioxane plumes and help predict where those plumes will migrate in the future. The Proposed 4th CJ provides for 14 new clusters of Monitoring Wells[11] at locations designated as A, B, C (on northern PZ boundary) (p. 12), D, E (near southern PZ boundary[12]) (p. 12), F, G, H (downgradient areas within PZ) (p. 22), I, J, K, L, M, and N (Western Area locations) (p. 34). All of these locations are described and depicted on maps contained in the Intervenors' Expert Report.

Technically and scientifically, these 14 added Monitoring Well clusters are necessary and appropriate, but they are not sufficient, because they leave substantial gaps through which narrow 1,4-dioxane plumes may be moving undetected. As stated in the Intervenors' Expert Report:

> Both the Eastern Area Prohibition Zone Containment Objective and the Western Area Non-Expansion Cleanup Objective stated in the Proposed 4th CJ share the goal of preventing 1,4-dioxane from migrating beyond the (revised) Prohibition Zone area of institutional control (Eastern Area) or present known extent of groundwater contamination (Western Area). Thus, the Proposed 4th CJ includes perimeter monitoring wells intended to serve as sentinel wells, boundary wells, delineation wells, and compliance wells. Those additional monitoring wells are all necessary to help delineate the extent of groundwater contamination, but are insufficient because gaps in the monitoring well network remain along

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[10] Monitoring Well cluster means that at a particular location multiple wells are installed to assess if dioxane concentrations differ at varying depths below the ground (e.g., shallow, intermediate, and deep).

[11] To simplify the discussion these are referred to throughout this brief simply as Monitoring Well locations, but all actually are Monitoring Well clusters.

[12] The Intervenor experts agree that the PZ Boundary Well E is necessary, but asserts that its location should be adjusted, as shown on the map in the Intervenors' Expert Report.

> the northern perimeter of the Eastern Area Prohibition Zone and the southern boundary of the Western Area dioxane plume. Gaps in the Eastern Area are significant because Scio Township residences, which rely on well water, and Barton Pond, which supplies the majority of Ann Arbor's municipal drinking water, are located north of the Prohibition Zone. The Western Area gap arises from the abandonment of MW-63, the southwestern most point in the compliance well network, in 2019. Int Exp Rept, p. 7.

4. Additional monitoring necessary to determine the extent of Gelman's 1,4-dioxane to address the above shortcomings.

i. PZ Perimeter Gap filling requires two additional Sentinel Wells in the Eastern Area and one replacement for former MW-63 in the Western Area.

The Intervenor Expert Report (pp. 7-9) makes clear that three additional Monitoring Well clusters, beyond those in the Proposed 4th CJ, are needed to help delineate the perimeter of the plumes in the PZ.[13] In the Eastern Area two additional Sentinel Well clusters (called AA and BB) are needed and in the Western Area a replacement is needed for the former MW-63 location. The Intervenor experts depict these locations on maps within their report and describe them as follows:

> Additional monitoring well clusters in strategically important areas are needed to ensure early detection of contaminant migration to the north and potential expansion of the Western Plume to the southwest. Monitoring well clusters include nests of wells with screened intervals at different elevations designed to detect 1,4-dioxane migrating through different layers of the glacial aquifer system. Multiple screens are necessary because it is difficult to know with certainty at what level contaminated water will migrate until it arrives at a monitoring well. Locations where additional monitoring well clusters are needed include:
>
> - A Sentinel Well (AA) closing the gap between MW-133 and MW-121
> - A Sentinel Well (BB) near the northeast Prohibition Zone boundary between MW-135 and MW-97
> - A replacement well (CC) in the vicinity of the former MW-63 well cluster. Int Exp Rept, p. 7.

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[13] Of course, the adequacy of these proposed wells is based on the degree of current scientific knowledge. If that changes in the future, additional monitoring could be needed.

The Intervenor experts describe the reasons that these locations are necessary to narrow the open gaps in the monitoring well system, and provide the Scientific Rationale for these additional wells as follows:

> **Scientific Rationale.** The rationale for including additional, more closely-spaced monitoring wells to detect potential migration along the perimeter of the known contamination extent relies on observations of 1,4-dioxane concentrations and migration in areas of densely-spaced monitoring wells. For example, in the area east of Wagner Road, wells MW-71 and MW-108s/d are spaced less than 200 feet from each other, yet display remarkably different concentration histories, despite being screened at the same elevation. Further downgradient, east of Maple Road, dioxane concentrations in MW-86 have been consistently non-detect, despite the fact that MW-86 is located approximately midway between MW-82s and MW-83s, which have seen dioxane concentrations as high as 370 and 645 ppb, respectively. <u>These observations indicate that contaminant transport pathways are narrower and more complex than shown on most site maps, and that bypassing of monitoring wells, either laterally or vertically, is possible</u>. Large gaps between monitoring wells along the plume perimeter should therefore be avoided, particularly in sensitive areas proximal to residences relying on private drinking water wells. Int Exp Rept, p. 9 (emphasis in original).

ii. Gap filling to assess possible northward migration of 1,4-dioxane toward Barton Pond requires new Monitoring Wells.

Barton Pond supplies the majority of Ann Arbor's drinking water. The pond lies directly north of the PZ, but no wells have been installed to determine if 1,4-dioxane is migrating in that direction toward the Pond, or if the subsurface geology is comprised of materials that would permit such a migration. While technical experts believe it is unlikely that the 1,4-dioxane plumes will migrate north to Barton Pond, if a narrow plume did migrate, the consequences would be enormous. The Intervenor experts describe the circumstances and conclude that three new monitoring wells (at locations DD, EE and FF) should be installed and monitored. Int Exp Rept, p 14. They depict these locations on maps in their report and state as follows:

> **Scientific Rationale.** In a recent study prepared for the City of Ann Arbor, environmental consultants at Tetra Tech evaluated potential sentinel monitoring well locations to provide advance warning to protect the City's drinking water supply in the event that the Gelman 1,4-dioxane plume were to migrate towards Barton Pond (Tetra Tech, 2020). Tetra Tech identified four potential sentinel well locations (**Figure 7**) based on their relation to topographic elevations and position opposite the surface water drainage divide. The additional wells proposed by the Intervenors are consistent with Tetra Tech's recommendations. *Id.*

iii. Added delineation needed to evaluate 1,4-dioxane above GSI limits approaching and hitting Allen Creek.

To protect Allen Creek and the Huron River, the Current Court Orders required Gelman to prevent any 1,4-dioxane exceeding the 2,800 ppb groundwater-surface water interface (GSI) cleanup criterion from migrating east of Maple Road. That criterion was reduced in 2016 to 280 ppb. Unfortunately, groundwater with 1,4-dioxane exceeding 280 ppb already is east of Maple Road, and the plumes are hitting Allen Creek. For example, samples taken from 2017 to 2020 have shown rapidly rising concentrations (now up to 49 ppb). As the Intervenor experts explain, that means that the concentrations entering the creek are much higher and may exceed the GSI limit. Int Exp Rept, p. 17. To fully evaluate the issues, as detailed at Int Exp Rept, pp. 17 – 25, the Intervenor experts describe three additional delineation actions that are necessary to determine the extent of the plumes with high concentrations, to allow these concerns to be addressed:

> To address concerns over discharge to Allen Creek at concentrations exceeding the GSI criterion, the Intervenors propose the following additions to activities included in the Proposed 4th CJ:
>
> 2A. High-resolution characterization to identify downgradient migration pathways
> 2B. Additional delineation of 280 ppb extent in the downgradient Eastern Area
> 2C. Shallow groundwater monitoring along the Allen Creek Drain. *Id.*, p 17.

a. Two sets of Transects are necessary to provide the high resolution characterization referenced as 2A, above.

The Intervenor experts depict on maps and describe these high resolution efforts as two North-South lines of temporary bore hole transects: one line along Maple Road between Dexter and Miller Roads; and the other line along Glendale-Grandview-Westwood streets near MW-82s. As the Intervenor experts note, the U.S. Environmental Protection Agency has identified this type of site characterization as a preferred method for evaluating sites. The physical boring efforts are described as follows:

> Temporary boreholes in each transect should be placed at a 200-foot minimum lateral spacing and water samples should be taken at 10-foot vertical increments to establish a concentration profile at each borehole location. Results can be used to position permanent monitoring wells in zones of highest observed concentrations, quantify contaminant mass flux across each transect, and to guide additional downgradient investigation (Sections 2B and 2C). Two north-south profiles (perpendicular to the primary direction of groundwater flow) are needed (**Figure 10**): *Id.*, p 19.

The Intervenor experts' full description of and scientific rationale for these transects is provided at Int Exp Rept, p 19 - 22. In essence, these transects are needed to identify the flow pathways (the position and the depth) of Gelman's 1,4-dioxane plumes headed to Allen Creek, which to date have never been determined.

b. Two additional Monitoring Well clusters are needed to delineate the 280 ppb extent of 1,4-dioxane in downgradient parts of the Eastern Area, to address 2B, above.

As noted earlier, Section V.A.5.f., the Proposed 4th CJ (at p 22) would require three wells at locations F, G and H to help determine the downgradient extent of Gelman's 1,4-dioxane plumes within the PZ. However, the Intervenor experts explain that these wells alone are insufficient to delineate the extent of contamination exceeding the 280 ppb GSI criterion necessary to ensure that the Groundwater Surface Water Objective in the Eastern Area is met. They note that the currently highest downgradient measurements are at MW-82s, but that location likely is not the leading edge or the centerline of the most contaminated groundwater. As stated by the Intervenor experts, it is more likely that "…MW-82s represents lateral dispersion (like MW-76s or MW-91) from one or more unrecognized higher concentration finger(s) of 1,4-dioxane migrating north or south of MW-82…." Int Exp Rept, p 23. Therefore, it is necessary to install additional monitoring wells to locate and depict the true downgradient extent of contamination exceeding 280 ppb. While wells F, G and H are needed, the Intervenor experts demonstrate that two more wells designated as GG and HH also are necessary.

> **Scientific Rationale.** The proposed monitoring well at location GG on the south side of MW-82s will complement the proposed well at location H on the north side of MW-82s (**Figure 14**). Both of these locations can be optimized based on the results of transect T_2-T_2'. Monitoring wells at locations GG and H will determine if higher concentrations of 1,4-dioxane are flanking MW-82s. An additional proposed monitoring well at location HH in the Allen Creek surface drainage way, will investigate the potential for 1,4-dioxane at concentrations above GSI along the expected migration pathway through a loosely defined area of artesian groundwater conditions conducive to additional venting to the Allen Creek Drain or the creation of shallow groundwater conditions at elevations close to residential basements in this area. Together, monitoring wells at proposed locations GG and HH will help to ensure that the Eastern Area "Groundwater Surface Water Interface Objective" in the Proposed 4th CJ is met. Int Exp Rept, p 23.

c. Shallow groundwater profiling is necessary along Allen Creek Drain, as noted in 2C, above.

As succinctly stated by the Intervenor experts:

> In addition to the delineation work (transects and monitoring wells) described above, it is necessary to identify the extent of groundwater contamination greater than 280 ppb entering the Allen Creek Drain upgradient of West Park so that appropriate response activities can be undertaken. Int Exp Rept, p 24.

This would be "a high-resolution profiling survey along the edges of the South Branch of the Allen Creek Drain…" (*Id.*), followed by the installation of three or more "shallow groundwater monitoring nests along each side of the Allen Creek Drain where the presence of groundwater at or above GSI concentrations has been delineated." *Id.*

Groundwater samples along the high-resolution profiles would be taken every five feet in depth, from a series of temporary wells, placed 100 feet apart, along both sides of the drain to a minimum depth 10 feet or more below the drain. These locations are depicted on maps and described at Int Exp Rept, pp. 24-25, along with the following scientific rationale for the efforts:

> **Scientific Rationale.** High-resolution profiles of groundwater concentrations will provide information about the distribution of 1,4-dioxane in excess of 280 ppb near the Allen Creek Drain. Establishing maximum concentrations is part of the requirement for use of the mixing zone criterion for GSI compliance under Part 201, as is estimating the cross-sectional area of the plume perpendicular to the groundwater flow that encompasses the entire portion of the plume exceeding GSI. Both of these requirements will be facilitated by the Drain profiles and the permanent, shallow groundwater monitoring well nests installed after the profiles are completed. Moreover, the wells can serve as alternative monitoring points (in the parlance of the GSI regulations) that will provide continuing information about the distribution of 1,4-dioxane in excess of 280 ppb near the Allen Creek Drain. Int Exp Rept, p 25.

5. Proposed provisions to include in the 2021 Order to properly delineate Gelman's 1,4-dioxane contamination.

To address the delineation issues discussed in sections IV.A.1 through IV.A.4, above, Intervenors propose that the 2021 Order contain the following sections from the Proposed 4th CJ, with the modifications and additions proposed above by the Intervenors typed in red.

(1) The 2021 Order should contain the following language from Section XII (pp. 56-57) of the Proposed 4th CJ, adding a new sub-section B to require plume maps, which would be updated semi-annually [with new language shown in red]:

"XII. PROGRESS REPORTS AND UPDATED PLUME MAPS

A. Defendant shall provide to EGLE written quarterly progress reports that shall: (1) describe the actions which have been taken toward achieving compliance with this Order during the previous three months; (2) describe data collection and activities scheduled for the next three months; and (3) include all results of sampling and tests and other data received by Defendant, its consultants, engineers, or agents during the previous three months relating to Remedial Action performed pursuant to this Order. Defendant shall submit the first quarterly report to EGLE within 120 days after entry of this Order, and by the 30th day of the month following each quarterly period thereafter, as feasible, until termination of this Order as provided in Section XXVI.

B. Gelman shall utilize all available existing monitoring well data to determine by appropriate hydro-geologic techniques the extent of various concentrations of 1,4-dioxane in groundwater in concentration intervals that are approved by EGLE, which shall include down to concentrations of 1 ppb, 7.2 ppb and 280 ppb and Gelman shall graphically depict plume maps showing those extents of 1,4-dioxane on maps that shall be publicly disclosed. Gelman shall re-determine the extent of the 1,4-dioxane at various concentrations in groundwater semi-annually and shall prepare and publicly disclose updated plume maps. "

(2) The 2021 Order should contain the following language from Sections V.A.3. a-d (pp. 12-13) of the Proposed 4th CJ, regarding the Eastern Area Sentinel Well and PZ Boundary Well requirements (showing requested modifications in red):

"3. Monitoring and Extraction Well Installation and Operation. Defendant shall install the following additional wells in the Eastern Area according to a schedule approved by EGLE and subject to access and receipt of any required approvals pursuant to Section VII.D:

"a. Sentinel Well Installation. Defendant shall install the following three monitoring well clusters to monitor movement of 1,4-dioxane south of the northern Prohibition Zone boundary, in addition to MW-120, MW-123, and MW-129 that are already in place (collectively referred to herein as "Sentinel Wells"):

i. Residential area in the general vicinity of Ravenwood and Barber Avenues (Location "A" on map attached as Attachment G);
ii. Residential area in the general vicinity of Sequoia Parkway and Archwood Avenues between Delwood and Center (Location "B" on map attached as Attachment G); and
iii. Residential area in the general vicinity of Maple Road and North Circle Drive (Location "C" on the map attached as Attachment G);
iv. Residential area roughly half way between locations of MW-133 and MW-121 (Location "AA" on the map attached as Attachment G);
v. Residential area, near the northeast PZ boundary, between MW-135 and MW-97 (Location "BB" on the map attached as Attachment G);
vi. Residential area north of Location C (Location "DD" on the map attached as Attachment G);
vii. Residential area north of Location C (Location "EE" on the map attached as Attachment G); and
viii. Residential area north of Location C (Location "FF" on the map attached as Attachment G)."

"b. PZ Boundary Well Installation. Defendant shall install the following two monitoring well clusters to monitor the movement of 1,4-dioxane near the PZ Boundary (collectively referred to herein as "PZ Boundary Wells"):

i. Residential, commercial, and vacant area east of South Wagner Road, north of West Liberty Road, west of Lakeview Avenue, and south of Second Sister Lake (Location "D" on map attached as Attachment G); and

ii. Residential area south/southeast of the MW-112 cluster (Revised Location "E" on map attached as Attachment G)."

(3) The 2021 Order should contain the following language from Section V.A.5.f (p.22) of the Proposed 4th CJ, regarding the Eastern Area Downgradient Investigation within the Prohibition Zone (showing requested modifications in red):

"f. Downgradient Investigation. The Defendant shall continue to implement its Downgradient Investigation Work Plan as approved by EGLE on February 4, 2005, as may be amended, to track the Groundwater Contamination as it migrates to ensure any potential migration of Groundwater Contamination outside of the Prohibition Zone is detected before such migration occurs with sufficient time to allow Defendant to maintain compliance with the Prohibition Zone Containment Objective and to ensure compliance with the Groundwater-Surface Water Interface Objective. Defendant shall, as the next phase of this iterative investigation process investigate the area depicted on the map attached as Attachment G, including the installation of monitoring wells at the following locations subject to access and receipt of any required approvals pursuant to Section VII.D:

i. A monitoring well nest in the residential area in the general vicinity of intersection of Washington and 7th Streets (Location "F" on Attachment G);

ii. A shallow well in the residential area in the general vicinity of current monitoring well nest MW-98 (Location "G" on Attachment G); and

iii. A monitoring well nest in the residential area in the general vicinity of Brierwood and Linwood Streets (Location "H" on Attachment G);

iv. A monitoring well nest in the residential area in the general vicinity south of MW-82s (Location "GG" on Attachment G); and

v. A monitoring well nest in the residential area (Location "HH" on Attachment G);

vi. Install and sample two sets of temporary transect borings set in a roughly N-S line, each boring placed with approximately 200 foot lateral spacing, with groundwater samples to be taken at 10 foot vertical intervals in each boring down to bedrock, with samples to be analyzed for 1,4-dioxane concentrations. The lines of transect borings to be located as follows:

    a. Line 1 – along Maple Road, from Dexter Road to Miller Road (with Location as depicted on Attachment G); and

    b. Line 2 – along Glendale-Grandview-Westwood Streets, near MW-82s (with Location as depicted on Attachment G); and

vii. Install and sample temporary transect borings on both sides of and closely adjacent to the South Branch of the Allen Creek Drain, with each boring placed with approximately 100 foot lateral spacing, with groundwater samples to be taken at 5 foot vertical intervals in each boring down to 10 feet below the base of the drain, with samples to be analyzed for 1,4-dioxane concentrations. (with Location as depicted on Attachment G). Results of the foregoing shallow groundwater profiling will be used to install a minimum of three shallow groundwater monitoring well nests along each side of the Allen Creek Drain where the presence of groundwater at or above GSI concentrations has been delineated. Each monitoring location should include at least two monitoring wells screened at the equivalent depth of the drain and 5 feet deeper so that a vertical hydraulic gradient can be determined.

viii. The data from the above wells will be used to guide additional downgradient investigations as necessary to ensure compliance with the Eastern Area Objectives."

(4) The 2021 Order should contain the following language from Section V.B.3.b. (pp. 33-34) of the Proposed 4th CJ, regarding Western Area Delineation Monitoring Wells (showing requested modification in red):

"b. Western Area Delineation Investigation. Defendant shall install the following additional groundwater monitoring wells pursuant to a schedule approved by EGLE and subject to the accessibility of the locations and obtaining access and any required approvals under Section VII.D at the approximate locations described below and on the map attached as Attachment G to address gaps in the current definition of the Groundwater Contamination and to further define the horizontal extent of Groundwater Contamination in the Western Area:

i. Commercial area north of Jackson Road (across from April Drive) and south of US-Highway I-94, near MW40s&d. (Deep well only) (Location "I" on Attachment G);

ii. Commercial area north of Jackson Road (across from Nancy Drive) and south of US-Highway I-94, east of MW-40s&d and west of the MW-133 cluster (Location "J" on Attachment G);

iii. Residential area west of West Delhi, north of Jackson Road and south of US-Highway I-94 (Location "K" on Attachment G);

iv. Residential area southwest of the MW-141 cluster in the vicinity of Kilkenny and Birkdale (Location "L" on Attachment G);

v. Residential area along Myrtle between Jackson Road and Park Road (Shallow Well only) (Location "M" on Attachment G);

vi. Residential and vacant area within approximately 250 feet of Honey Creek southwest of Dexter Road (Location "N" on Attachment G); and

vii. Location of former MW-63 well cluster (shown on Attachment G). ****"

B. The Prohibition Zone Should Not Be Expanded as Much as Suggested in the Proposed 4th CJ.

As discussed more fully in Section II.C. of this brief (pp. 11-14 supra), Gelman is not automatically entitled to an Institutional Control such as the Prohibition Zone which would allow its contamination to flow away and continue to impact clean aquifers. In 2001, Gelman discovered that 1,4-dioxane had migrated to a deeper aquifer which the parties called "Unit E." EGLE and Gelman disagreed over how to address the contamination and the parties presented the issue to the Court for decision. The fundamental disagreement between the parties was whether Gelman would be required to comply with the aquifer protection rules and, if not, what conditions Gelman would need to satisfy. The aquifer protection rules impose stringent requirements concerning contamination of groundwater in aquifers:

> (5) The horizontal and vertical extent of hazardous substance concentrations in an aquifer above the higher of either the concentration allowed by section 20120a(1)(a) [i.e., the generic residential cleanup criteria] or (10) [i.e., the target detection limit or background concentration] of the act, as applicable, **shall not increase after the initiation of remedial actions to address an aquifer,** except as approved by the directors as provided in section 20118(5) and (6) of the act. Mich Admin R 299.3 (emphasis added).

Ultimately, the state agreed to waive the aquifer protection rules and accept a Prohibition Zone, but only if Gelman agreed to assure that no concentrations of 1,4-dioxane exceeding the GSI cleanup criterion would ever be allowed to migrate east of Maple Road. That Maple Road Containment requirement was an integral piece of a bargain which induced the state to agree with a Prohibition Zone. That bargain and the consideration for it should not be lost or overlooked in determining what changes should be allowed now.

When considering any expansion of the existing Prohibition Zone, it is crucial to keep in mind what the Prohibition Zone is, why it was imposed, and what it does. The Prohibition Zone is a type of "institutional control." As opposed to most engineered controls, such as an extraction well, an institutional control does not remediate contamination. To the contrary, an institutional control is an administrative or legal control used *precisely because* contamination remains in place. See, e.g., MCL 324.20118(d)(ii) (permitting hazardous substances to remain in an aquifer if certain conditions are met, including "enforceable land use restrictions or other institutional controls necessary to prevent unacceptable risk from exposure to the hazardous substances"). Rather than require cleanup, an institutional control typically restricts the use of property in order to control exposure (e.g., a restrictive covenant limiting the use of property to non-residential use). See, e.g., MCL 324.20121. Because of these features, institutional controls are typically used to supplement, not supplant, active remedial measures. See, e.g., MCL 324.20118; 40 CFR 300.430(a)(1)(D) ("EPA expects to use institutional controls such as water use and deed restrictions to supplement engineering controls as appropriate.… The use of institutional controls shall not substitute for active response measures (e.g., treatment and/or containment of source material, restoration of ground waters to their beneficial uses) as the sole remedy unless such active measures are determined not to be practicable, based on the balancing of trade-offs among alternatives that is conducted during the selection of remedy.").[14]

Unlike many institutional controls, the Prohibition Zone not only restricts the use of Gelman's property, it limits the rights of numerous property owners spread over an area of 2,000 acres (or 3.2 square miles). Any expansion of the Prohibition Zone will interfere with groundwater rights of additional property owners. An expansion also would allow more natural resources to become or remain polluted without an obligation to remediate or restore them.

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[14] 40 CFR Part 300 is the National Oil and Hazardous Substances Pollution Contingency Plan, the set of regulations used by the EPA at federal Superfund sites.

The Court initially created the Prohibition Zone not for Gelman's convenience or because it had a right to such an institutional control, but for expediency and to address the exigent public health concerns caused by the discovery of the Unit E aquifer contamination. See, generally, **Ex. E** and **F**. The parties and the Court understood that, while the boundary of the Prohibition Zone could be expanded in the future, any expansion would need to be justified and Gelman could not use the possibility of an expansion to evade its obligations to control the spread of the plume. In the Current Court Orders, Gelman committed to "prevent the plumes of groundwater contamination emanating from the GSI Property [i.e., Gelman's property] from expanding beyond the current boundaries of such plumes, except into and within the Prohibition Zone and Expanded Prohibition Zone." **Ex. H**, p 4. Gelman further committed to implement a verification plan "to ensure that any potential migration of groundwater contamination outside of the Expanded Prohibition is detected before it occurs." *Id.*, p 5. If Gelman determines that groundwater contamination will migrate outside of the Prohibition Zone, it is required to conduct a feasibility study to determine the options available to prevent the migration from occurring. Although Gelman can propose expanding the Prohibition Zone as an option in the feasibility study, Gelman agreed that such an expansion would be a last resort. See, e.g., *id.*, p. 6 ("[t]he parties agree that any further expansion of the northern boundaries of the Prohibition Zone or Expanded Prohibition Zone should be avoided, unless there are compelling reasons to do so."); see also, Section V.A.2.f of the Proposed 4th CJ (subject to narrow exceptions, "the Prohibition Zone boundary may not be expanded unless the moving Party demonstrates by clear and convincing evidence that there are compelling reasons that the proposed expansion is needed to prevent an unacceptable risk to human health.").

With that as a backdrop, Gelman has proposed a large expansion of the Prohibition Zone, ostensibly due to the reduction in the drinking water standard from 85 to 7.2 ppb. Intervenors agree that a limited expansion is appropriate because 1,4-dioxane at concentrations above 7.2 ppb already has migrated beyond the existing boundary. Int Exp Rep, 10. In particular, Intervenors accept the entirety of the proposed expansion in the north. *Id*. But the size of the proposed expansion in the south is not scientifically justified.

As Intervenors' experts explain, a more limited expansion in the south is justified by the fairly steep concentration gradient in that area, meaning that the concentration falls off quickly as one moves toward the edge of the plume. *Id*., 10, fn. 2. This suggests that a buffer of at most 400 feet in the southern area is appropriate to address the reduction of the drinking water standard, as described in Figure 4 of the Intervenors' experts' report.[15]

Gelman may argue that an additional buffer is appropriate because of the uncertainty over the migration of 1,4-dioxane and because a larger buffer is more protective of public health. Such arguments should be rejected. Gelman should not be permitted to use the uncertainty caused by its failure to delineate and properly model the future migration of the plume as a basis for a larger Prohibition Zone. See, MCL 324.20114(1)(a) (requiring a liable party like Gelman to "determine the nature and extent of the release at the facility."). The starting point should be maintenance of the existing Prohibition Zone. Only credible technical data should be sufficient to support an expansion. Gelman has not come forward with credible data for a larger southern expansion. As for protecting public health, one always could argue that a greater Prohibition Zone provides greater protection. That argument has no limiting factor, yet expansion of the

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[15] If the Intervenors' proposal for the southern Prohibition Zone boundary is accepted, then the proposed new boundary well at location E should be adjusted accordingly, as reflected on Figure 4 of the Intervenor Expert Report.

Prohibition Zone comes with a steep price—interference with property rights and the contamination of additional natural resources.

The benefits of an increased Prohibition Zone expansion in reality would flow to Gelman. The larger the Prohibition Zone, the less active remediation Gelman need perform in order to maintain the integrity of the boundary. Gelman also surely knows that it will be very difficult to expand the Prohibition Zone in the future given the high standards in the Current Court Orders and Proposed 4th CJ and wants as much cushion as it can obtain. But the standard Gelman must meet for a future expansion is high for a reason. The Prohibition Zone is not a mechanism that exists for Gelman's convenience and it comes with significant cost. The Court should adopt the Intervenors' proposed expansion as set forth in Figure 4 to their experts' report because any expansion beyond that is unsupported by data and is not necessary to protect public health.

C. The increased *Active* Remedial Measures Proposed in the Proposed 4th CJ Are Necessary.

The Current Court Orders do not have sufficient *active* remedial measures in place to achieve the intended remedial objectives when the new, more stringent cleanup criteria (7.2 ppb drinking water and 280 ppb GSI) are applied. First, the PZ was designed to prohibit wells in areas where the residential drinking water cleanup criterion (then 85 ppb) has been or might be exceeded. But the south boundary of the current PZ already has been breached by 1,4-dioxane exceeding the new residential drinking water cleanup criterion of 7.2 ppb. Similarly, the Maple Road Containment Objective was established to prohibit groundwater exceeding the GSI value (then 2,800 ppb) from migrating past Maple Road. A number of extraction wells were required by the Current Court Orders at and upgradient of Maple Road to achieve that objective. Again, however, that line has been breached by groundwater with 1,4-dioxane exceeding the new GSI value of 280 ppb. Third, private wells in Scio Township are at risk, with measurable 1,4-dioxane nearing the new 7.2 ppb drinking water level.

Scientifically, to achieve the above objectives now, when the new cleanup criteria are applied, requires both adjusted containment lines (e.g., new PZ boundaries and new downgradient GSI limits) and additional active remedial measures at the Gelman Property and at remaining hot spots to lower 1,4-dioxane concentrations moving downgradient. Accordingly, the Proposed 4th CJ requires that Gelman perform the following, necessary additional active remedial measures that are not required by the Current Court Orders: (1) install wells and extract, treat and dispose water from identified hot spot areas at the new "Rose Well", at the new "Parklake Well" and at six or more locations on the Gelman Property; (2) install and operate a Heated Soil Vapor Extraction System ("HSVE System") in the Burn Pit area on the Gelman Property; and (3) implement a Phytoremediation treatment system in two areas of the Gelman Property. Removing additional 1,4-dioxane mass from these hot spots will result in a significant reduction in the downgradient concentrations of 1,4-dioxane as the groundwater migrates past and beyond that area. This reduces the likelihood that, in the future, downgradient wells will exceed allowable limits, that the PZ boundaries will be breached, or that groundwater venting to surface waters will exceed the GSI limits. Int Exp Rept, p 25. Intervenors agree with these provisions and support their inclusion in the proposed 2021 Order, provided certain modifications are made.

D. Treated Water From Parklake Well Should Be Piped To And Discharged From Gelman's Existing Outfall At The Gelman Property.

1. The Proposed 4th CJ plan to discharge treated Parklake water to First Sister Lake is not appropriate.

As noted in prior sections of this brief, sections V.A.3.e – g of the Proposed 4th CJ (pp. 14-16) would require Gelman to install the Parklake Well in an identified hot spot, and extract, treat and dispose of the treated water. That active remedial action would be appropriate, effective and necessary to remove 1,4-dioxane mass and reduce the subsequent downgradient 1,4-dioxane concentrations in groundwater. This then reduces the likelihood that homeowners' wells would be contaminated, reduces the likelihood that the PZ boundaries would be breached and reduces the likelihood that the GSI criterion would be exceeded when the water vents to a surface water such as Allen Creek, Honey Creek or the Huron River.

However, section V.A.3.g. of the Proposed 4th CJ (p. 16) currently would require Gelman to install and operate this important Parklake hot spot treatment system *only if* Gelman is permitted to discharge 200 gallons per minute of the treated water year round into First Sister Lake. That discharge would have negative consequences. As examples, the discharge would raise the water level of First Sister Lake by about 6 – 12 inches (adversely affecting a raingarden recently installed by the City of Ann Arbor); the discharge of groundwater with a year round temperature of 55 degrees F will make the lake cooler in summer and warmer in winter (disturbing the habitat of plants and animals that depend on seasonal changes to water temperatures); the 200 gpm volume of water will turn over the entire lake water volume every 35 days (which could adversely impact fish and amphibious creatures and flora around the lake by changing temperature and chemistry of the water). Int Exp Rept, pp. 27-28. Gelman should not be allowed to discharge its treated water to First Sister Lake when such negative consequences are possible.

Gelman's obligation to implement this necessary and appropriate active Parklake Well extraction and treatment remedial measure should not and need not be conditioned on permitting a discharge to First Sister Lake. A pipeline can be installed in roadways from Parklake to the Gelman Property where this water can be discharged through Gelman's existing permitted outfall. The only downside to Gelman is the cost of the pipeline. That cost should be borne by the liable party. However, Intervenors believe the additional cost to install that pipeline, all of which could be in public rights-of-way, is relatively minimal, looking at the entirety of the remediation costs Gelman is incurring. In addition, the Intervenors are prepared to cooperate with Gelman regarding installation of the pipeline in public rights-of-way.

2. Scientific Rationale for piping treated Parklake water to Gelman Property for discharge.

There would be several environmentally harmful consequences if the treated Parklake well water is discharged into First Sister Lake. In contrast, those adverse consequences would be eliminated (and no significant additional adverse consequences to human health or the environment would result) if, instead, a pipeline is installed into road rights of way and that water is then piped to the Gelman Property where it would be discharged through Gelman's current outfall into the Honey Creek tributary. As stated by the Intervenor experts:

> Although 200 gpm may not sound like a large amount of water, over the course of a week or a month or a year it adds up to a considerable volume, and if the treated water from the Parklake Extraction Well were discharged into First Sister Lake, the impacts on First Sister Lake and the surrounding areas likely would preclude issuance of an NPDES[16] permit. To avoid a likely unsuccessful application for an NPDES permit, other options need to be considered and the effects of those options need to be fully assessed.

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[16] NPDES is an acronym for National Pollutant Discharge Elimination System, a federal permitting program that has been delegated to the State of Michigan. Gelman would be required by federal and state law to obtain a NPDES permit prior to discharging to First Sister Lake.

> Alternatives to direct discharge into First Sister Lake involve questions of engineering and access. Therefore, flexibility is warranted to enable Gelman and the affected communities to devise an acceptable solution while navigating the NPDES permitting process. Int Exp Rept, p 28.

The only downside to this approach would be the cost to Gelman to install the pipeline to transport its contaminated water. An alternative approach should be allowed only if Gelman satisfies EGLE (and this Court if a dispute resolution is required) that any different alternative it proposes will not have an adverse impact on human health or the environment.

3. Proposed Provisions for Parklake treated water discharge in the 2021 Order.

Intervenors propose that Section V. A. 3.g. (p. 16) of the Proposed 4th CJ be deleted in its entirety and replaced with the following provision in the 2021 Order as shown in red:

> g. Transport and discharge/disposal of treated Parklake Well water.
>
> i. Unless EGLE approves an alternative that satisfies g. ii, below, after groundwater extracted by the Parklake Well is properly treated with ozone/hydrogen peroxide technology, Gelman shall transport the treated water through a pipeline to the Gelman Property where it shall be discharged from Gelman's existing permitted discharge outfall, in accordance with the terms of its existing permit (as same may be amended or replaced). Gelman shall obtain any permits or amendments to permits necessary to authorize such discharge. Gelman shall obtain all necessary permits or authorizations from all applicable state or local governmental authorities necessary to install, maintain and operate a pipeline to transport the treated water from the Parklake Well treatment system to the Gelman Property. Gelman shall install, maintain and operate the pipeline at its sole cost.
>
> ii. Subject to EGLE approval, Gelman may propose and implement alternative means or methods to discharge or dispose of water from the Parklake Well (following treatment with ozone/hydrogen peroxide technology), in lieu of the pipeline described in g.i., above. Such an alternative may be approved and permitted by EGLE, only if Gelman demonstrates that the alternative will have no adverse impacts or consequences to human health or the environment.

E. Concurrent Installation and Operation of the 6 Identified Extraction Well Locations on the Gelman Property.

1. The Need for More Extraction Wells in the Source Area.

The highest concentrations of 1,4-dioxane remain on the Gelman property where the chemical was released into the soil and groundwater as part of the company's manufacturing process. The releases of 1,4-dioxane occurred through seepage lagoons, land spray irrigation and direct discharges to the environment. Concentrations of 1,4-dioxane in the source area have been as high as 225,000 ppb and still remain at levels in excess of 10,000 ppb in some areas. Those areas with the highest concentrations are identified in the Proposed 4th CJ as Former Ponds 1 and 2, Former Burn Pit and the Marshy Area. The existing contaminant levels greatly exceed the drinking water standard of 7.2 ppb and the GSI standard of 280 ppb.

As a liable party under Part 201, Gelman has 3 basic legal duties with respect to the releases of 1,4-dioxane at its facility: (i) Determine the nature and extent of the releases at the facility; (ii) Immediately stop and prevent an ongoing release at the source; and (iii) Diligently pursue response activities necessary to achieve the cleanup criteria established under Part 201. MCL 324.20114. While Gelman has certainly removed a considerable amount of 1,4-dioxane from the source area, it has failed to comply with its basic obligations to stop ongoing releases at the source and diligently pursue the response activities necessary to achieve applicable cleanup criteria. Gelman's failure to comply with Part 201 has resulted in a large plume of groundwater contamination migrating through the City of Ann Arbor which continues to be fed by high concentrations in the source area.

The Gelman property is located in the "Western Area" as identified in Section V.B. of the Proposed 4th CJ. There are two primary objectives in the Western Area which Gelman is required to meet. The first objective is that Gelman shall prevent the horizontal extent of groundwater contamination from expanding, regardless of aquifer depth. The second objective is that Gelman shall prevent 1,4-dioxane from venting into surface waters in the Western Area at concentrations above the GSI criterion of 280 ppb (Proposed 4th CJ, pp. 30-32). The nearest surface water of most concern is the Honey Creek Tributary, located just north of the Marshy Area which has groundwater concentrations in excess of 10,000 ppb.

The more 1,4-dioxane that is removed from the source area, the more likely it is that Gelman will meet the objectives for the Western Area. The high concentrations in the source area exist largely in the shallow groundwater. These concentrations seep into the lower aquifers which then migrate off-site and continue to feed the Eastern Area plume. These concentrations also migrate laterally, which has the potential to expand groundwater contamination in the Western Area and vent into Honey Creek, Third Sister Lake and other nearby surface waters in excess of the GSI criterion. Removing as much 1,4-dioxane as possible from the source area is the best way to prevent continued vertical and lateral migration and satisfy the Western Area objectives. Int Exp Rept, p 29-30.

Acknowledging the importance of increased mass removal, Gelman agreed in the Proposed 4th CJ to install 3 additional extraction wells in the source area. Int Exp Rept, p, 29, fig. 18. These 3 proposed wells will collect groundwater at a combined rate of 75 gallons per minute ("gpm") and the 1,4-dioxane will be removed at Gelman's on-site treatment facilities. Based on the performance achieved from these 3 extraction wells, the Proposed 4th CJ provides that Gelman and EGLE will evaluate whether 3 additional extraction wells will be installed in the source area, which are identified in Attachment I to the Proposed 4th CJ. The criteria for evaluation is whether the 3 additional extraction wells "would accelerate mass removal to a degree that meaningfully benefits the remediation." Proposed 4th CJ, pp. 46-47. There is no question that 3 additional extraction wells will accelerate mass removal and therefore Intervenors request that the Proposed 4th CJ be modified to require that Gelman install and operate concurrently all 6 extraction wells identified in Attachment I to the Proposed 4th CJ. Based on the performance of these initial 6 wells, more extraction wells may be required in the Source Area.

2. Scientific Rationale for More Extraction Wells in the Source Area.

The Marshy Area where the highest concentrations of 1,4-dioxane remain consists largely of peat soil, which is typical of wetland areas. The peat absorbs water and therefore the flow of groundwater in this area is more limited than in areas of more sandy soils. As a result of the poor hydraulic conductivity, extraction wells installed in or near the Marshy Area have a smaller radius of influence (i.e., the influence of pumping from these wells is significant over a relatively small area). This means that you need to install more extraction wells to achieve hydraulic control of the contaminated groundwater.

The Proposed 4th CJ requires the installation of 3 extraction wells in the northwestern, central and southwestern sections of the source area. Given the peat matrix and the poor hydraulic conductivity, it is unlikely that only 3 wells will be sufficient to reach all hot spots in the source area. The 3 "optional" extraction wells are designated to be installed in the northern, eastern and southeastern sections of the source area which would provide a more complete recovery system and make it more likely that the highest concentrations of 1,4-dioxane would be removed from the aquifer. Int Exp Rept, p.30.

Mass removal in the source area is directly related to Gelman's obligation under Part 201 to prevent an ongoing release at the source. The installation of 3, rather than 6, extraction wells will likely result once again in Gelman failing to meet its Part 201 obligations. Only 3 extraction wells will leave large sections of the source area without any hydraulic control. This will in turn allow continued migration of 1,4-dioxane down to lower aquifers which flow off-site into the

City of Ann Arbor. The lack of hydraulic control will also allow lateral migration of contaminated groundwater which vents into the Honey Creek Tributary and other surface waters in the area. Lateral migration also presents a risk of contaminating residential wells located near the edge of the plume. Int Exp Rept, p 7.

All parties agree that more mass removal from the source area will benefit the overall remediation effort for the reasons discussed above. It is undisputed that more extraction wells will remove more 1,4-dioxane from the environment. Given that 6 extraction wells are already contemplated in the Proposed 4th CJ, it is a simple and highly effective modification to require the installation of the 3 “optional” wells so that all 6 extraction wells operate concurrently to maximize mass removal from the source area. Based on the performance of these initial 6 wells, more extraction wells may be required in the Source Area.

3. Proposed Provisions for More Extraction Wells to include in a 2021 Order.

Intervenors propose that the 2021 Order contain the following Section VI. C. 1. (pp. 46-48) of the Proposed 4th CJ modified as shown in red:

> 1. Additional Groundwater Extraction. Defendant shall install and operate ~~three~~ six ~~“Phase I”~~ extraction wells (one of which was previously installed) at the general locations depicted in the attached Attachment I to enhance control and mass removal of 1,4-dioxane from this area of shallow groundwater contamination. Defendant shall operate these extraction wells at a combined purge rate of approximately ~~75~~ 150 gpm, subject to aquifer yield. Defendant shall have the discretion to adjust the individual well purge rates in order to optimize mass removal. Subject to Defendant’s ability to adjust individual well purge rates, Defendant shall continue to extract a combined purge rate of approximately ~~75~~ 150 gpm, subject to aquifer yield, . . .
>
> Based on the performance achieved from the above initial six extraction wells, the Parties shall evaluate whether installation and operation of ~~up to three~~ additional extraction wells ~~at the general locations indicated on Attachment I~~ would accelerate mass removal to a-degree that meaningfully benefits the Remediation. If EGLE determines that additional mass removal of 1,4-dioxane from additional wells would be beneficial, Defendant shall, subject to its right to invoke Dispute

Resolution under Section XVI, install and operate these additional wells pursuant to a work plan approved by EGLE.

Groundwater extracted from the extraction wells described in this subparagraph will be conveyed to the Wagner Road Treatment Facility for treatment and disposal pursuant to Defendant's NPDES Permit No. MI-0048453, as amended or re-issued.

F. Termination Criteria For Extraction Wells Should Be Modified To Allow For Continued Operation After 1,4-Dioxane Concentrations are Reduced Below 500 PPB.

1. The Need for Modification of the Termination Criteria for Extraction Wells.

The Proposed 4th CJ includes several extraction wells in different locations which are intended to target the highest concentrations of 1,4-dioxane in the area of the plume. The proposed extraction wells in the source area were discussed in the previous section. There is also an extraction well proposed in the Eastern Area near First Sister Lake called the "Parklake Well." Although the Parklake Well is located downgradient of the Gelman property, its objectives are similar to the extraction wells in the source area. It is located in a hot spot area and its primary purpose is mass removal to reduce the concentrations of 1,4-dioxane which are migrating toward Allen Creek and the Huron River.

Concentrations of 1,4-dioxane in the Parklake Well area substantially exceed the GSI criterion of 280 ppb. Given the rising concentrations in Allen Creek in the West Park area, it appears that contaminated groundwater which exceeds the GSI criterion may be venting into the Creek. Int Exp Rept, p 17. This underscores the importance of the Parklake Well, not only for mass removal but also for compliance with the GSI criterion.

The Proposed 4th CJ includes provisions for terminating the source area and Parklake extraction wells after 1,4-dioxane concentrations are reduced below 500 ppb. (Proposed 4th CJ, pp. 15-16, 41, 47.) This is a completely arbitrary threshold and should be eliminated from the

Proposed 4th CJ. Meaningful mass removal can still occur at lower concentrations and it would be irresponsible to shut these wells down until as much 1,4-dioxane as possible has been removed from the aquifers.

2. Scientific Rationale for Modification of the Termination Criteria for Extraction Wells.

The 500 ppb termination criterion would prevent the maximum removal of 1,4-dioxane from the aquifer and reduce the overall effectiveness of the remediation. Since mass removal can occur at lower concentrations, shutting down an extraction well prematurely simply means that less 1,4-dioxane is being removed from the environment. This makes it more likely that Gelman will not be able to meet the objectives of the Current Court Orders or its obligations under Part 201 of source control and compliance with GSI. In addition to mass removal, other benefits of continuing to operate the extractions wells at lower concentrations include hydraulic capture of contamination and prevention of 1,4-dioxane migration. Int Exp Rept, p 26.

Furthermore, Gelman recognizes the benefits of pumping at lower concentrations because it continues to operate extraction wells with concentrations well below 500 ppb. Int Exp Rept, p. 26, fig. 16. Gelman continues to operate TW-21 and TW-18, where concentrations are currently around 250 ppb. It also continued to operate TW-1, TW-6 and TW-12 until concentrations were in the 20-100 ppb range.

There is no numeric termination criteria in the Proposed 4th CJ for any of the extraction wells currently operated by Gelman. In the Eastern Area, Gelman must operate the wells until it can establish that it has met the Eastern Area Objectives of no contamination migrating beyond the boundaries of the Prohibition Zone and preventing 1,4-dioxane from venting to surface waters in the Eastern Area at concentrations above the GSI criterion in compliance with Part 201 (Proposed 4th CJ, p.8). In the Western Area, Gelman must operate the extraction wells until it can establish that extraction is no longer necessary to prevent expansion of the contamination and to satisfy the GSI criterion (Proposed 4th CJ, pp.30-32).

It would be irresponsible to apply an arbitrary numeric termination criterion for the proposed source area and Parklake extraction wells. It would mean less 1,4-dioxane removed from the environment. It would also be inconsistent with the existing termination criterion that bases termination or reduction in pumping rates on whether objectives in the Current Court Orders have been met.

The termination criterion of 500 ppb is also inappropriate given the nature of how extraction wells operate. Extraction wells are much different from monitoring wells, which passively sample concentrations in the surrounding groundwater. Extraction wells draw water equally in all directions from the groundwater that surrounds them Int Exp Rept, p. 27, fig. 17. Therefore, the concentration measured in an extraction well represents the average concentration of water which the well draws in from every direction. Actual concentrations in parts of the aquifer within the radius of influence of the well can be much greater than what is measured. Int Exp Rept, p 27.

For example, an extraction well placed near the edge of a hot spot could draw concentrations of over 1,000 ppb along with concentrations below 100 ppb. While the concentration in this well could be measured at 500 ppb, much higher concentrations would be present within the radius of influence of the well. This would mean concentrations of over 1,000 ppb would continue to be removed, even though the composite concentration of the 1,4-dioxane measured in the extraction well is lower. There is no scientific basis to terminate an extraction well such as this while it is still removing concentrations of over 1,000 ppb. Since it is virtually impossible to place every extraction well in the exact center of a hot spot, this phenomenon is likely to occur to varying degrees with every extraction well. Int Exp Rept, p. 27.

For these reasons the Intervenors propose a modification to the Proposed 4th CJ with more flexible termination criteria. Similar to the existing criteria, Intervenors request that Gelman be required to operate the Parklake and source area extraction wells until it can be established that a well no longer contributes to the beneficial reduction in 1,4-dioxane mass. This would maximize mass removal and include the accompanying benefits of both source control and more limited migration.

3. Proposed Provisions for Modification of Termination Criteria for Extraction Wells to Be Included in the 2021 Order.

Intervenors propose that the 2021 Order contain the following Section V.A.f.ii of the Proposed 4th CJ (pp. 15-16) modified as shown in red:

> ii. Defendant shall operate the Parklake Well, at a purge rate of approximately 200 gpm, subject to the yield of the aquifer in that area and discharge volume restrictions imposed in connection with the method of water disposal including discharge restrictions during wet weather events, in order to reduce the mass of 1,4-dioxane migrating from that area. Purged groundwater from the Parklake Well shall be treated with ozone/hydrogen peroxide or ultraviolet light and oxidizing agents at the City of Ann Arbor-owned parcel. Defendant shall operate this extraction and treatment system until ~~the 1,4-dioxane concentration in the groundwater extracted from the Parklake Well has been reduced below 500 ug/L. Once concentrations have been reduced below 500 ug/L~~ effluent 1,4-dioxane concentrations indicate continued extraction will no longer contribute to beneficial reduction in 1,4-dioxane mass." Before seeking to terminate or significantly reduce extraction, Defendant shall cycle the Parklake Well off and on for several periods of time approved by EGLE to demonstrate that significant concentration rebound is not occurring. Defendant shall not permanently terminate extraction and treatment of water from the Parklake Well before the second anniversary of the date extraction was commenced. Before significantly reducing or terminating extraction from the Parklake Well (beyond the discharge volume restrictions/variations arising from the approved discharge option/above-described cycling), Defendant shall consult with EGLE and provide a written analysis, together with the data that supports its conclusion that the foregoing conditions have been satisfied. EGLE will review the analysis and data and provide a written response to Defendant within 56 days after receiving Defendant's written analysis and data. If Defendant disagrees with EGLE's

> conclusion, Defendant may initiate dispute resolution under Section XVI of this Consent Judgment. The Defendant shall not significantly reduce or terminate extraction from the Parklake Well during the 56-day review period or while Defendant is disputing EGLE's conclusion.

Intervenors propose that the 2021 Order contain the following Section VI.C.1. of the Proposed 4th CJ (pp. 46-48) be modified as shown in red:

> 1. Additional Groundwater Extraction. . . . Defendant shall continue to extract a combined purge rate of approximately ~~75~~ 150 gpm, subject to aquifer yield, from this system until ~~the 1,4-dioxane concentration in the groundwater extracted from each of these extraction wells has been reduced below 500 ug/L and, once the concentrations in all three of the wells have been reduced below 500 ug/L~~ effluent 1,4-dioxane concentrations from each of these extraction wells indicate continued extraction will no longer contribute to the beneficial reduction in 1,4-dioxane mass and, once this has occurred for all six of the wells, Defendant shall cycle those wells off and on for several periods of time approved by EGLE to demonstrate that significant concentration rebound is not occurring. Before otherwise significantly reducing or terminating extraction from this system, Defendant shall consult with EGLE and provide a written analysis, together with the data that supports its conclusion that ~~the concentration of 1,4-dioxane in the groundwater extracted from each of these wells has been reduced L below 500 ug/~~ continued extraction will no longer contribute to the beneficial reduction in 1,4-dioxane mass, as stated above. EGLE will review the analysis and data and provide a written response to Defendants within 56 days after receiving Defendant's written analysis and data. If Defendant disagrees with EGLE's conclusion, Defendant may initiate dispute resolution under Section XVI of this Consent Judgment. The Defendant shall not significantly reduce or terminate the extraction from this system during the56-day review period or while Defendant is disputing EGLE's conclusion. . . .

G. The Trigger For Response Activities In The Western Area Compliance Wells Should Be Reduced From 7.2PB TO 3.5 PPB.

1. The Need for a Lower Trigger in Western Area Compliance Wells.

The Proposed 4th CJ requires Gelman to install 6 new delineation wells in the Western Area. These are monitoring wells that will be installed as part of Western Area Delineation Investigation and are identified as locations I, J, K, L, M and N on the site map attached as Attachment G to the Proposed 4th CJ. These wells will be part of a network of Compliance Wells which will be sampled quarterly and used to determine whether Gelman is in compliance with the objective of no expansion of groundwater contamination in the Western Area.

The 6 new delineation wells play a vital role in protecting the public from exposure to 1,4-dioxane in their drinking water. The proposed wells are located between the northern edge of the contaminant plume in the Western Area and hundreds of residential drinking water wells located within the potential migration pathway. In fact, regular sampling of residential wells in this area has revealed low levels of 1,4-dioxane already present in wells on Elizabeth Road and Breezewood Court, located just north of the proposed delineation wells. **Ex. N**, Residential Well Sampling.

The Proposed 4th CJ requires Gelman to sample the Compliance Wells on a quarterly basis. If 1,4-dioxane is detected in any Well above 7.2 ppb, this is considered to be a "Verified Compliance Well Exceedance" and Gelman will increase the sampling to monthly. In the event of an Exceedance in two successive monthly samples, then Gelman is required to undertake a series of response activities, including increased residential well sampling, a hydrogeological investigation and a feasibility study of interim measures to control expansion of the plume such as installation of additional extraction wells (Proposed 4th CJ, pp. 36-40).

Given the critical role of the Compliance Wells in protecting public health, Intervenors request that the trigger level for response activities be reduced from 7.2 ppb to 3.5 ppb. The Compliance Wells are the "last line of defense" to prevent the contamination of residential drinking water supplies. Response activities to avoid this disastrous result will be more effective if undertaken before contaminant levels exceed the drinking water standard.

It creates an unnecessary public health risk to "wait" for 1,4-dioxane concentrations in the Compliance Wells to exceed the drinking water standard before taking response activities to protect the drinking water supply. Waiting will only increase the likelihood of widespread residential well contamination due to the failure to implement corrective measures early enough in the process to control migration of the plume.

2. Scientific Rationale for a Lower Trigger in Western Area Compliance Wells.

The Compliance Wells are there to detect any expansion of the contaminant plume in the Western Area. A reading all the way up to 7.2 ppb is not necessary to detect that expansion is occurring. Any increase in concentrations are an indication that levels of 1,4-dioxane at 7.2 ppb and higher are moving outward toward the Compliance Well. It is simply not possible for concentrations in the Compliance Well to rise from non-detect to 7.2 ppb or lower without the position of the 7.2 ppb concentration line moving toward the Compliance Well. Any detection of 1,4-dioxane in a Compliance Well is a *de facto* expansion of the horizontal extent of the groundwater contamination. Int Exp Rept, p. 35-36.

Intervenors request a trigger level of 3.5 ppb, which represents the US EPA Drinking Water Concentration for a cancer risk level of 1 in 100,000. This level is sufficiently above the 1 ppb detection limit so that there should not be any concerns about statistical variability or false positives. Furthermore, in the Proposed 4th CJ Gelman has already agreed to a trigger level of

4.6 ppb for wells on the boundary of the Prohibition Zone. 3.5 ppb is a reasonable and workable trigger level which is more likely to prevent expansion in the Western Area and contamination of residential drinking water supplies.

3. Proposed Provisions for a Lower Trigger for Western Area Compliance Wells to Include in a Supplemental Response Activity Order.

Intervenors propose that the 2021 Order contain the following Section V.B.4.b. of the Proposed 4th CJ (pp. 36-38) modified as shown in red:

> b. Verification Process. Defendant shall conduct the Verification Process as defined in Section III.X for each Compliance Well to verify any exceedance of ~~7.2~~ 3.5 ppb. A verified detection above ~~7.2~~ 3.5 ppb will be considered a "Verified Compliance Well Exceedance." If a second sample does not exceed ~~7.2~~ 3.5 ppb, monitoring of the well will increase to monthly until the pattern of exceedances is broken by two successive sampling events below ~~7.2~~ 3.5 ppb. At that point, a quarterly monitoring frequency will resume.

Intervenors propose that Section V.B.4.c(i) of the Proposed 4th CJ be modified as follows:

> c. Response Activities. In the event of a Verified Compliance Well Exceedance, Defendant shall take the following Response Activities: i. Sample selected nearby private drinking water wells. Defendant shall sample select private drinking water wells unless otherwise the Parties otherwise agree. Prior to sampling the selected wells, Defendant shall submit a list of the wells to be sampled and other sampling details to EGLE for approval. In selecting wells to be sampled, Defendant shall consider data collected from monitoring and private drinking water wells within 1,000 feet of the Compliance Well(s) that exceeded ~~7.2~~ 3.5 ppb, groundwater flow, hydrogeology and well depth. EGLE shall respond within seven days after receipt of Defendant's list of select private drinking water wells and shall either approve the list or propose alternate or additional wells to be sampled.

Intervenors propose that Section V.B.4.c.(ii)(D) of the Proposed 4th CJ be modified as follows:

> ii. If a Verified Compliance Well Exceedance occurs in the same Compliance Well in any two successive monthly sampling events, Defendant shall take the following Response Activities:
>
> > (D) Interim Measures Feasibility Study. During the eight month period after the second consecutive Verified Compliance Well Exceedance, Defendant shall evaluate affirmative measures to control expansion of the Groundwater Contamination as necessary to reduce the concentration of 1,4-dioxane in the relevant Compliance Well to below ~~7.2~~ 3.5 ppb, including adjustments in groundwater extraction rates, the installation of additional groundwater extraction wells or other remedial technologies. 38 Defendant shall submit to EGLE a feasibility study within 240 days of the Verified Compliance Well Exceedance. The feasibility study shall include an evaluation of the feasibility and effectiveness of all applicable measures to control expansion of the Groundwater Contamination as necessary to reduce the concentration of 1,4-dioxane in the relevant Compliance Well to below ~~7.2~~ 3.5 ppb in light of the geology and current understanding of the fate and transport of the Groundwater Contamination.

H. Gelman Should Be Required to Develop a Plan Which Monitors the Effectiveness of the Phytoremediation Systems.

1. The Need for Monitoring the Effectiveness of the Phytoremediation Systems.

Phytoremediation is a remediation technology which uses living plants to remove or immobilize contaminants in soil and groundwater. The root systems can remove contaminants from the groundwater through transpiration and can also dewater a shallow aquifer to prevent contaminants in the soil from migrating to lower aquifers. The Proposed 4th CJ (pp. 48-49) requires Gelman to install two tree systems in the source area to accomplish both of these objectives.

In the former Ponds 1 and 2 Area phytoremediation will consist of poplar and hardwood trees planted primarily to withdraw shallow groundwater and capture precipitation near the ground surface before it infiltrates beyond the root systems. Int Exp Rept, p. 30-31. If the trees are successful in dewatering the shallow aquifer, contaminants in the soil will be less likely to move to lower aquifers and migrate off site. Trees planted in the Ponds I and II Area are also expected to remove some amount of 1,4-dioxane from the shallow soil and groundwater through transpiration and biodegradation. *Id.*

Similarly, in the Marshy Area phytoremediation will consist of willow trees which will capture contaminated groundwater and infiltration water moving through the contaminated soil before it can move vertically and migrate off site. *Id.* p 31. The Marshy Area is located near the Honey Creek Tributary and the trees also serve the purpose of preventing lateral migration of contaminated water and possible venting to the Creek in violation of the GSI criterion. It is also expected that the trees in the Marshy Area will remove 1,4-dioxane from the soil and groundwater through transpiration and biodegradation. *Id.*

Phytoremediation is a promising but experimental technology so it is uncertain how effective it will be in removing and containing 1,4-dioxane in the source area or how best to measure its effectiveness. Yet the Proposed 4th CJ lacks any requirement for Gelman to measure or monitor the effectiveness of the phytoremediation systems. The Proposed 4th CJ vaguely states that Gelman will operate the systems until it determines that phytoremediation is no longer necessary to meet the objective of preventing the migration of 1,4-dioxane from contaminated soils into any aquifer at concentrations which would cause non-compliance with the Western Area Objectives of no expansion and no venting to surface water in violation of the GSI criterion (Proposed 4th CJ, pp. 48-49). But it is not possible to determine whether objectives are being met unless there is a plan to verify the effectiveness of the phytoremediation systems with objective data.

Therefore, the Intervenors request a modification to the Proposed 4th CJ to require Gelman to develop a phytoremediation effectiveness verification plan. Such a plan would generate the following types of data: 1) Measuring concentrations of 1,4-dioxane in groundwater beneath and downgradient from the tree plots; 2) Measuring changes in the groundwater table due to the presence of the trees: 3) Measuring the rate of transpiration; 4) Analysis of tree tissue or leaves for levels of 1,4-dioxane; and 5) Shallow groundwater monitoring points along the Honey Creek Tributary to measure GSI compliance. This data will provide the basis for an objective evaluation of the effectiveness of the phytoremediation systems.

2. Scientific Rationale for Monitoring the Effectiveness of the Phytoremediation Systems.

Trees that are planted as part of the phytoremediation systems likely will not have a significant effect on site hydrogeology and 1,4-dioxane concentrations until 2-3 years after planting. Once the root systems are established, it is expected that dewatering and removal of 1,4-dioxane through biological processes will continue at optimal rates for many years. However, given the uncertainty of the technology and the complex hydrogeology of the source area, it is necessary to collect data to determine the effectiveness of the systems in meeting the non-expansion and GSI objectives in the Western Area. Int Exp Rept, p. 31.

The tree plots will be connected to the groundwater system in two important ways. First, root systems in the Ponds I/II Area will extend to the shallow groundwater unit primarily for the purpose of dewatering and capturing precipitation near the ground surface before it can infiltrate to lower aquifers. Second, the root systems in the Marshy Area extend to a deeper groundwater unit primarily for the purpose of capturing contaminated groundwater before it can migrate laterally to the Honey Creek Tributary or infiltrate a lower aquifer and migrate off site. Int Exp Rept, p. 31.

Because of these organic connections to the groundwater system, the tree plots will play an important role as to whether Gelman can maintain compliance with the Western Area Objectives. Monitoring beneath and adjacent to the tree plots and along the Honey Creek Tributary is essential in determining their effectiveness in preventing the expansion of groundwater contamination and venting into the Tributary. Ideally tree tissue or leaf analysis can be used to identify the location of the highest concentrations of 1,4-dioxane in the northernmost trees which will help determine appropriate locations for groundwater monitoring points adjacent to the tributary to Honey Creek. These points will be used to verify compliance with the GSI criterion.

3. Proposed Provisions for Monitoring the Effectiveness of the Phytoremediation Systems to Include in a Supplemental Response Activity Order.

Intervenors propose that the 2021 Order contain the following Section VI.B.3 (p. 46) in addition to what was in the Proposed 4th CJ:

> 3. Within 180 days of entry of this Order, Gelman shall submit to EGLE for its review and approval a plan to verify the effectiveness of the phytoremediation installations. At a minimum, the plan shall include: (i) procedures to determine or reliably estimate rates of biodegradation and transpiration for 1,4-dioxane in both the Former Pond and Marshy Areas; (ii) measurement of 1,4-dioxane concentrations in groundwater beneath the Former Pond and Marshy Areas; (iii) groundwater logging throughout the tree plots to verify expected dewatering; (iv) verification of the extent to which trees planted in caissons have root systems that penetrate lower aquifers containing high concentrations of 1,4-dioxane; (v) a modeled estimate of the impact of the tree plots on the availability and migration of 1,4-dioxane from the phytoremediation areas; (vi) an evaluation of the 1,4-dioxane content of the trees for categorization purposes once disposal becomes necessary, (vii) monitoring points along the Honey Creek Tributary to determine compliance with the GSI criterion, and (viii) any additional monitoring criteria Gelman and EGLE deem appropriate.

I. The Heated Soil Vapor Extraction System Can Be Enhanced to Maximize Efficiency and Mass Removal.

1. The Need for Enhancement of the Heated Soil Vapor Extraction System.

The Proposed 4th CJ requires Gelman to install a Heated Soil Vapor Extraction System ("HSVE System") in the Burn Pit area on the Gelman property. This is an area with high concentrations of 1,4-dioxane in the soil and the purpose of the HSVE System is to remove contaminants from the unsaturated soil. Removal of contaminants in the soil will reduce the contaminant levels in groundwater as it moves vertically through the soil and migrates off site. This in turn will increase the likelihood of Gelman compliance with its Part 201 obligation to control ongoing releases at the source.

The HSVE System involves blowing heated air into subsurface soils through various injection points in the Burn Pit area. The heated air traverses horizontally and vertically through the subsurface soils and volatilizes the 1,4-dioxane into a vapor which is removed by a system of vacuum extraction wells. These extraction wells create a negative pressure in the unsaturated soils which controls subsurface vapor plume migration. The vapor which is extracted from the subsurface is exhausted into the air and may need to be treated depending on contaminant concentrations.

The Proposed 4th CJ provides that Gelman shall operate the HSVE System until concentrations in the exhaust have been reduced to levels which indicate that continued operation of the System will no longer contribute to meaningful reduction of 1,4-dioxane mass or the soil contamination is reduced below 500 ppb, whichever occurs first. (Proposed 4th CJ p.50). When operation of the HSVE System is terminated, Gelman is required to install an impervious cap over the treatment area to limit the infiltration of water into deeper soils and prevent any remaining 1,4-dioxane in the soil from migrating to groundwater or surface water.

The Intervenors are in agreement that the HSVE System is an appropriate method for removing 1,4-dioxane from soils in the Burn Pit area. However, Intervenors believe that the System can be more effective with two minor modifications. First, the impervious cap should be installed before the HSVE System begins to operate as this will limit infiltration of water and surface air and result in more effective subsurface treatment. Second, once the HSVE System appears to have reached an asymptotic removal rate, the System should be cycled off and on for several periods before shutting it down. This will ensure that the System is not shut down before it has achieved maximum mass removal of 1,4-dioxane from the soils. Int Exp Rept, p. 32-33. The Proposed 4th CJ requires Gelman to cycle the Parklake and source area extraction wells off and on before shutting them down and the same principles apply to the HSVE System.

2. Scientific Rationale for Enhancement of Heated Soil Vapor Extraction System.

The purpose of the HSVE System is to remove vapors with contaminants which have been volatilized and separated from the soil by heated air injected into the subsurface. The removal occurs due to the negative pressure created by the extraction wells which draws the vapors into the well. For the system to work at maximum efficiency, extraction wells should be pulling in only contaminated vapors. However, this negative pressure also results in ambient air near the ground surface being drawn into the HSVE System, which limits its effectiveness. The clean air in the System reduces the area of influence of each extraction well which means there are fewer air pore volume exchanges occurring and less contaminated vapors being removed from the soil. Furthermore, heat in the system is also lost due to infiltration of water and surface air, which reduces the effectiveness of the system.

The simple solution is to install an impervious cap on the surface <u>before</u> starting the HSVE System. This will limit infiltration of water and surface air and result in a more effective subsurface treatment. Gelman has already agreed in the Proposed 4th CJ to install an impervious barrier over the treatment area, but only after the HSVE System is shut down. Making the barrier part of the initial installation is easy to do and will significantly enhance the effectiveness of the HSVE System. Int Exp Rept, p. 34.

Typically, HSVE systems exhibit a diminished rate of contaminant extraction over time and it is expected that mass removal rates of 1,4-dioxane will plateau within several years. The Proposed 4th CJ states that Gelman will operate the HSVE System until levels of 1,4-dioxane in the exhausted discharge air have been reduced to levels such that the continued operation of the System will no longer result in meaningful mass reduction or soil contamination is reduced below 500 ppb, whichever occurs first (Proposed 4th CJ p. 50). At that point, Gelman is required to submit a written request to EGLE to reduce or terminate the operation of the HSVE System. The Intervenors simply request that Gelman be required to cycle the System off and on for several periods once an asymptotic removal rate has been achieved to ensure that a diminished extraction rate of 1,4-dioxane is not a temporary phenomenon. Gelman is already required by the Proposed 4th CJ to cycle the source area and Parklake extraction wells to ensure maximum mass removal, and it would be irresponsible not to impose the same requirement for the HSVE System.

3. Proposed Provisions for Enhancement of Heated Soil Vapor Extraction System to Include in Supplemental Response Activity Order.

Intervenors propose that the 2021 Order should contain the language from Section VI.C.4. of the Proposed 4th CJ (pp. 49-51), modified as follows [with proposed modifications in red]:

> 4. Former Burn Pit Area. Defendant shall undertake the following Response Activities with respect to the former Burn Pit area depicted on Attachments I and J: a. Install, operate, and maintain a Heated Soil Vapor Extraction System ("HSVE System"). The HSVE System shall be designed to reduce the mass of 1,4-dioxane present in the soils in the portion of the former Burn Pit area identified as "Heated Soil Vapor Extraction" on Attachment J. Defendant shall operate the HSVE system until 1,4-dioxane concentrations in the HSVE System's effluent/exhaust has been reduced to levels that indicate that continued operation of the HVSE system will no longer contribute to meaningful reduction of 1,4-dioxane mass in the Former Burn Pit Area Soils or the Soil Contamination in the treatment area is eliminated, whichever occurs first. Defendant shall cycle the HSVE System off and on for several periods of time approved by EGLE to demonstrate that significant concentration rebound is not occurring. Before significantly reducing or terminating operation of the HSVE system, Defendant shall consult with EGLE and provide a written analysis, together with the data that supports its conclusion, that one or both of the above conditions has been satisfied. EGLE will review the analysis and data and provide a written response to Defendant within 56 days after receiving Defendant's written analysis and data. If Defendant disagrees with EGLE's conclusion, Defendant may initiate dispute resolution under Section XVI of this Consent Judgment. The Defendant shall not significantly reduce or terminate operation of the HSVE system during the 56-day review period or while Defendant is disputing EGLE's conclusion.
>
> ~~Following completion of the HSVE treatment~~ As part of the installation of the HSVE System, Defendant shall install an impervious barrier over the HSVE Treatment Area to limit infiltration of surface air and inhibit water from percolating through the soils in the former Burn Pit Area.~~, except with regard to any areas where Defendant can demonstrate to EGLE's satisfaction that Soil Contamination does not exist.~~ Defendant shall maintain the impervious barrier in place until Soil Contamination is no longer present in the underlying soils.
>
> b. Cap the portion of the former Burn Pit area identified as "Capped Area" on Attachment J with an impervious barrier to inhibit water from percolating through the 51 soils in the former Burn Pit area. Defendant shall maintain the impervious barrier in place until Soil Contamination is no longer present in the underlying soils.

5. After completing installation of the Response Activity systems listed in Sections VI.C.2, VI.C.3 and VI.C.4, the Defendant shall submit a separate installation report (i.e., as-built report) for each of the systems. The reports shall describe the systems as installed including, but not limited to, components of a system, location of components within the specific areas, depths of components of a system, and operational specifications of components of a system.

J. Surface Water Bodies and Drainage Systems Should Be Sampled Annually

1. Annual sampling of surface water bodies and drainage systems is necessary to detect changes indicating venting of groundwater with 1,4-dioxane at new locations or rising concentrations.

One of the primary objectives of the Proposed 4th CJ is to prevent 1,4-dioxane from venting into surface waters at concentrations above the Generic GSI Cleanup Criterion, except in compliance with Part 201, in order to ensure public and environmental health and safety. However, the presence of 1,4-dioxane has already been documented in Allen Creek, Third Sister Lake, and at multiple locations along the unnamed tributary to Honey Creek, posing a potential violation of this objective.

In order to ensure early detection of such potential violations of this objective, routine and regular surface water sampling is necessary to detect changes in concentrations that could indicate the venting of groundwater containing 1,4-dioxane at new locations or rising concentrations, and to ensure that appropriate response actions are taken in a timely manner. The Intervenors propose requiring sampling of surface water bodies and drainage systems following protocols developed by EGLE as implemented in 2019 and 2020 sampling.

2. Detection will trigger investigation to determine risk of exceeding the GSI criterion.

1,4-dioxane poses a serious threat to the surface waters of the Huron River watershed because it does not easily biodegrade and thus can remain in the water column at persistent concentrations for a relatively long time. There is currently no measure in place to detect 1,4-dioxane in the surface waters of the Huron River watershed through sampling and analysis, and the current monitoring well scheme alone is insufficient to accurately assess the risk of 1,4-dioxane entering these surface waters through contaminated groundwater. Surface water monitoring is an effective way "to detect discharge of contaminated groundwater and trigger additional subsequent actions required to address whether that discharge represents an exceedance of the GSI criterion." Int Exp Rept, p 35.

3. Proposed provisions for surface water sampling to be included in the 2021 order.

Intervenors propose that the 2021 Order should contain a new section V.A.11 with the following language [new provisions in red]:

> Defendant shall sample annually: Allen Creek, the Allen Creek Drain, and each of its tributaries including the Main, North, South, and Murray Washington branches, as well as the outflow into the Huron River below Argo Dam; First Sister Lake; Second Sister Lake; Third Sister Lake; West Park Pond; Arbor Landing Pond; Smith Ponds; Little Lake; and Honey Creek and its tributaries. This sampling must be conducted under low flow conditions during the months of August, September, or October, following protocols developed by EGLE as implemented in 2019 and 2020 sampling.

Intervenors propose that the 2021 Order should contain a new section V.A.6.g. with the following language [new provisions in red]:

> g. Exceedance of GSI Criteria in Surface Water. Pursuant to V.A.11., if sampling of any of the surface water bodies or drainage systems (with the exception of Third Sister Lake and the South Branch of the Allen Creek Drain downgradient of Maryfield-Wildwood Park) detects the presence of 1,4-dioxane at a concentration greater than 7 ppb, then, within 60 days of receiving such a sampling result, Defendant shall investigate and submit a report to EGLE containing at least the following information: (1) a determination of where and how 1,4-dioxane is likely entering the affected water body, (2) an assessment of the risk that the GSI Cleanup Criterion will be exceeded in the affected water body, (3) proposed Response Activities for preventing 1,4-dioxane from entering the affected water body in a concentration greater than the GSI Cleanup Criterion, and (4) an assessment of the risk that 1,4-dioxane from the affected water body could migrate to groundwater. After receipt and review of Defendant's report, EGLE may require Defendant to undertake additional Response Activities to address the sampling result, including, but not limited to, the installation of additional monitoring wells.

K. Gelman Should Publicly Disclose All Information Related to its Remedial Activities.

1. The need for public disclosure.

Gelman's remedial activities conducted over the course of more than 30 years have necessarily generated a tremendous amount of environmental data and analysis. This would include such information as soil and groundwater sampling, pump tests and capture zone analysis for extraction wells, pilot tests for remedial actions, groundwater flow analysis and plume delineation. Yet it appears that only some of this information has been disclosed to the public. The massive plume of groundwater contamination affects significant populations in Scio Township and Ann Arbor and the public has a right to receive all data and other information related in any way to the remedial actions conducted by Gelman. Furthermore, this information would allow Intervenors and their experts to provide more detailed and constructive input as additional remedial actions are developed at the site.

The Proposed 4th CJ only requires that Gelman submit to EGLE quarterly progress reports which describe activities for the previous 3 months and provide sampling data (Proposed 4th CJ, pp. 56-57). These reports are mostly just raw analytical data from monitoring wells and effluent samples. There are no accompanying reports which analyze and interpret the data and put it into the context of the overall remedial objectives. Furthermore, there is not one common database from which this analytical data can be equally accessed by everyone. Gelman provides the data to EGLE, but it is not in a commonly readable electronic format and so EGLE has to first collate the data and then post it on the EGLE website. This inefficient process has long been a source of frustration for residents impacted by the plume because it has resulted in many discrepancies in the data and delays in disseminating information to the public. The simple solution is for Gelman to establish a single cloud-based database which would include all historical and future analytical data and reports related to the Site.

EGLE has authority under Part 201 (MCL 324.20117) to demand that Gelman provide all information related to the contaminant plume and in fact this statutory authority is already embodied in the Proposed 4th CJ. Section XXII of the Proposed 4th CJ states that, upon request from EGLE, Gelman shall provide copies of all documents and information "relating to activities at the Site or to the implementation of this Consent Judgment" (Proposed 4th CJ, pp. 73-74). Unfortunately, EGLE does not exercise its statutory right on a regular basis and as a result for years the public has been denied access to important information about the nature and extent of the 1,4-dioxane contamination.

A good example of EGLE's failure to request information is the proposed remedial activity in the source area. The Proposed 4th CJ provides that Gelman will install additional extraction wells, install a phytoremediation system and remove soil contamination through heated soil vapor extraction. The Proposed 4th CJ also requires Gelman to provide "as built" installation reports describing the components of each of the source control systems. (Proposed 4th CJ, p. 51). However, selection of these remedial actions would necessarily be based on a considerable amount of data and analysis, none of which has been requested by EGLE or disclosed to the public. For example, these systems are designed to address "hot spots" in the source area. But in order to locate the "hot spots," extensive soil and groundwater sampling and sample analysis in the source area is required. Furthermore, in order to determine placement of extraction wells, typically pump tests are performed to determine the likely capture zones of the wells. Before designing a soil vapor extraction system, it is typically necessary to first run a pilot test to determine the effectiveness of a proposed system. None of this data or analysis has been provided to the public, which is certainly entitled to such basic information as the current levels of soil and groundwater contamination in the source area.

The public should not have to rely on EGLE to exercise its statutory right in order to obtain technical analyses and environmental studies or reports related to the Site. Instead, Intervenors propose a revision to the Proposed 4th CJ which requires Gelman to provide this information to EGLE so that it can be posted on the EGLE website. Furthermore, Intervenors propose that Gelman provide all historical and future monthly analytical data simultaneously to EGLE and the public through a single cloud-based database. These actions would ensure complete transparency and equal access to information, and instill public confidence in the availability and reliability of the data.

2. The legal basis for public disclosure.

Section 20117 of Part 201 provides that EGLE may require a responsible party to provide all information it has related to "the nature or extent of a release or threatened release at or from a facility." MCL 324.20117(1)(b). For purposes of this section, "information" includes, but is not limited to, "documents, materials, records, photographs and videotapes." MCL 324.20117(13). Information obtained by EGLE "shall be available to the public to the extent provided by the freedom of information act [FOIA] . . ." MCL 324.20117910). In providing the information to EGLE, a responsible party may designate certain information which it believes is entitled to protection as trade secrets or if it is of a personal nature under FOIA. Id.

However, certain information is deemed so critical that Section 20117 requires its disclosure to the public regardless of how it may be designated by the responsible party. This information includes the following: 1) The potential routes of human exposure to the hazardous substance at the facility being investigated; 2) The location of disposal of any waste stream released from the facility; 3) Monitoring data or analysis of monitoring data pertaining to disposal activities related to the facility; 4) Hydrogeologic data; 5) Groundwater monitoring data; and 6) The hazards to the public health, safety, or welfare, or the environment posed by the hazardous substance. MCL 324.20117(11).

Section 20117 recognizes the importance of the public having full access to information about hazardous contaminants which potentially impact the health and safety of their communities. Certainly, the plume of 1.4-dioxane contamination which stretches across a city and township has the potential to impact the health and safety of a large population and the public is entitled to access all information in Gelman's possession which relates to its release of 1,4-dioxane into the environment.

3. Proposed provisions for public disclosure of information to include in a 2021 Order.

Intervenors propose that Section XXII of the Proposed 4th CJ (pp. 73-74) be modified as follows [with original provisions in black and new provisions in red]:

> XXII. ACCESS TO INFORMATION
>
> A. Upon request, EGLE and Defendant shall provide to each other copies of or access to all non-privileged documents and information within their possession and/or control or that of their employees, contractors, agents, or representatives, relating to activities at the Site or to the implementation of this Consent Judgment, including, but not limited to, sampling, analysis, chain 74 of custody records, manifests, trucking logs, receipts, reports, sample traffic routing, correspondence, or other documents or information related to the Remedial Action. Upon request, Defendant shall also make available to EGLE, their employees, contractors, agents, or representatives with knowledge or relevant facts concerning the performance of the Remedial Action. The Plaintiffs shall treat as confidential all documents provided to Plaintiffs by the Defendant marked "confidential" or "proprietary."
>
> B. Within 60 days of entry of this Order and to the extent not previously provided, Defendant shall provide EGLE with all technical analyses and environmental or engineering studies or reports related to its Response Activities at the Site. This information shall include, but it is not limited to, the following: a) pump test results and capture zone analysis for all extraction wells; b) analytical results from all soil and groundwater testing at the Site; c) all reports and analysis of groundwater flow and modeling; d) all maps depicting the current Site area,

> delineation of contaminant plume, Prohibition Zone boundary and all monitoring and extraction well locations; e) Defendant's 1,4-dioxane transport model, including underlying assumptions regarding advective movement, retardation (adsorption), degradation, diffusion and dispersion; f) all results of pilot tests for any remedial activity; g) all hydrogeological assessments/investigations or contingency plans created by Defendant; h) all GSI compliance plans; and i) all remedial design data and related assumptions and analyses. The information provided by Defendant shall be promptly posted by EGLE on its Gelman website.
>
> C. Within 60 days of entry of this Order, Defendant shall establish a cloud-based database designed specifically for the storage and validation of data and information associated with all monitoring wells, extraction wells and NPDES treatment and discharge activity. The data shall include identifying information for each well, including address, GPS, X and Y coordinates, top of casing and ground elevations, well logs and lithology, well and screen depths and survey information. This database will be identical to the database maintained by Gelman and will include all historical as well as future information. The information should be available for read-only electronic download in one or more native Excel files (or in a successor program to Excel provided that when the data are migrated to a new program, no data are lost). The database shall be updated by Defendant on a monthly basis. Defendant is required to investigate and remedy any data gaps or discrepancies identified by the Intervenors or members of the public. If information needed to fill data gaps is not available, Defendant will explain why the information is not available.

L. Proposed Provisions to Provide an Ongoing Role and Rights of Intervenors Relative to Implementation and Enforcement of the Court's 2021 Order.

In light of the Intervenors' role in this litigation, their interest on behalf of their residents in the effective implementation of the Court's 2021 Order, and their role and obligations relative to the implementation of the 2021 Order, the Intervenors need to have continuing involvement in connection with the implementation of the 2021 Order, as described below. Intervenors' requests take into consideration EGLE's constitutional and statutory role as the regulatory agency responsible for enforcing the terms of a remediation plan, as embodied in the 2021 Order.

The Proposed 4th CJ was accompanied by a proposed Order that had been negotiated among the parties and provided for Intervenors to have a role and ongoing rights relative to the Proposed 4th CJ. That proposed order, like the Proposed 4th CJ, was rejected by the governing bodies of the Intervenors. Nevertheless, because that proposed Order was necessary for

Intervenors to have a continuing role, and because those provisions now need to be incorporated into this Court's 2021 Order, much of what the Intervenors request in terms of an ongoing role relative to the 2021 Order is based on that proposed Order.

The 2021 Order includes a process for Dispute Resolution and identifies when and how that process would be invoked by Defendant or by EGLE, including the ability to seek court review and resolution if the Dispute Resolution process is not successful. Intervenors request that they be able to participate any time the Dispute Resolution process is invoked, and request that they—individually or collectively—have the right to invoke the Dispute Resolution in those same circumstances, including seeking court review, even if not invoked by Defendant or EGLE.

In situations for which the 2021 Order provides for Defendant or EGLE to proceed directly to court without engaging in the Dispute Resolution process, Intervenors also need to be able to participate in or initiate the court proceedings.

To the extent enforcement responsibilities are EGLE's regulatory responsibility, and/or in situations where the 2021 Order places defined responsibilities on EGLE, Intervenors need a role to be able to ensure EGLE undertakes those responsibilities appropriately. To that end, Intervenors request that they—individually or collectively—be able to petition EGLE if they believe EGLE has not fulfilled its responsibilities appropriately, and to seek court review and action if not satisfied with EGLE's response.

To implement the foregoing, Intervenors request that a section be included in the 2021 Order to provide for and govern their ongoing role and rights relative to implementation and enforcement of the 2021 Order. These proposed provisions are in addition to provisions throughout the 2021 Order that specifically provide certain rights or roles for the Intervenors, e.g., rights regarding Defendant's application to EGLE for an NPDES permit.

1. Intervenors Must Have a Voice and Role Relative to Any Termination, Reduction, or Other Modification of Response Activities or Other Actions Under the 2021 Order.

The Proposed 4th CJ provided procedures and criteria for, and the 2021 Order now requested by Intervenors also provides procedures and criteria for, termination, reduction, or other modifications by Defendant of certain response activities and other actions, including certification of completion and termination. Although the requested rights to be involved vary somewhat according to what the modification is, the requests generally are that:

1. Defendant be required to provide each Intervenor with its analysis supporting its position that the relevant response activity can be terminated, reduced, or otherwise modified under the criteria listed in the applicable section of the 2021 Order, including its Notice of Completion, when Defendant provides that analysis and documentation to EGLE;
2. EGLE be required to consult with Intervenors and consider in good faith their comments and concerns with respect to the proposed termination, reduction, or modification of the response activities;
3. After such consultation, EGLE be required to provide each of the Intervenors with its written response to Defendant's analysis when it provides that response to Defendant; and
4. An Intervenor be entitled to invoke Dispute Resolution under Section XVI of the 2021 Order if it disagrees with EGLE's response, and be entitled to participate fully if Defendant invokes the Dispute Resolution process under Section XVI of the 2021 Order.

With respect to the scientific advisory panel provided for in Section V.C.3, Intervenors also request that:

1. EGLE be required to consult with Intervenors with respect to EGLE's selection of its panel member under Section V.C.3.a;
2. Intervenors be entitled to provide the scientific advisory panel with any submissions requested by the panel under Section V.C.3.b;
3. EGLE be required to provide each of the Intervenors their response to the scientific advisory panel's recommendations when it provides said response to Defendant pursuant to Section V.C.3.c; and
4. An Intervenor be entitled to invoke Dispute Resolution under Section XVI of the 2021 Order if it disagrees with EGLE's position and be entitled to participate fully in any Dispute Resolution process invoked by Defendant under Sections V.C.3.c and XVI of the 2021 Order.

2. Intervenors Must Have a Voice and Role Relative to Any Modification of the Prohibition Zone Boundaries Under the 2021 Order

The Proposed 4th CJ provided procedures and criteria for, and the 2021 Order now requested by Intervenors also provides procedures and criteria for possible modification of the boundaries of the Prohibition Zone that are established by Consent Judgment Section V.A.2. The relevant sections are Sections V.A.2.f (Prohibition Zone Expansion) or V.A.6 (Prohibition Zone Boundary Review). Because those modifications require a motion to and decision by this Court, the rights and role Intervenors request is different than those that may be resolved without a dispute resolution process, and for which the Dispute Resolution process is an option.

If any modification of Prohibition Zone boundaries is proposed, Intervenors ask that:

1. Defendant and EGLE each be required to provide each Intervenor with all court filings filed pursuant to Sections V.A.2.f and/or V.A.6;
2. EGLE be required to consult with Intervenors and consider in good faith their comments and concerns with respect to the proposed modification of the Prohibition Zone boundaries prior to filing any such filings with the Court; and
3. Any Intervenor be entitled to participate fully in the court proceedings, including filing briefs and other documents to inform the Court of their comments and concerns.

3. Modification of the 2021 Order and of Obligations Thereunder by Stipulation Must Be Stipulated to by All Intervenors

Section XXIV of the 2021 Order provides for Defendant and EGLE to stipulate to modifications of the 2021 Order. To avoid possible evasion of the rights of Intervenors to be included in a decision-making process that terminates, reduces, or otherwise modifies a provision of the 2021 Order, or terminates, reduces, or otherwise modifies an obligation of Defendant or EGLE or both under the 2021 Order, Intervenors ask that a provision be included to preclude Defendant and EGLE from making such modifications by stipulation unless each of the Intervenors also stipulates to the modification.

4. Intervenors Need to Have a Role in the Development of Groundwater-Surface Water and Groundwater-Stormwater Systems Work Plans

Because the venting of groundwater to surface water with 1,4-dioxane in concentrations that exceed the Generic GSI Criterion poses a risk to the health of residents as well as the environment, and because the venting or infiltration of groundwater containing 1,4-dioxane into a municipal segregated storm sewer or stormwater system, including a Drain not only poses a similar risk, but also has an immediate impact on the municipality's obligations to prevent inflow of pollutants into the system, and to prevent discharge of pollutants at the system's outlet, Intervenors need to have a role in the development of plans to address such venting of groundwater with 1,4-dioxane. Therefore, to the extent Defendant is required to submit work plan(s) describing Response Activities and/or evaluations to be implemented/undertaken to address any area where groundwater is venting to surface water in concentrations that exceed the Generic GSI Criterion with respect to either the Eastern Area or Western Area, EGLE shall consult with Intervenors and consider in good faith their comments and concerns with respect to the adequacy of the proposed Response Activities and/or evaluations. To the extent Defendant is required to submit work plan(s) describing Response Activities and/or evaluations to be implemented/undertaken to address any area where groundwater is venting to or infiltrating into a municipal segregated storm sewer or stormwater system, including a Drain, EGLE shall consult with Intervenors and consider in good faith their comments and concerns with respect to the adequacy of the proposed Response Activities and/or evaluations.

5. Intervenors Must Have a Voice and Role Relative to Determinations as to the Adequacy of the Financial Assurance Mechanism ("FAM") Requirements Submitted by Defendant

If EGLE is required to make a decision under Section XX.C of the 2021 Order, including but not limited to a determination as to the adequacy of the amount of the FAM submitted by the Defendant, approval of Defendant's periodic calculation of long-term cleanup costs, approval of a conversion of the form of the FAM, or a determination that Defendant is no longer required to maintain a FAM, Intervenors need to be involved to ensure not only that the FAM is adequate, but also to ensure Defendant is basing its calculation of long-term cleanup costs on all the response activities required by this Order, is including and committing to undertaking all required response activities, and has the necessary financial resources to be able to fulfill its obligations. Although Intervenors will get notice of and can be involved relative to most modifications of Defendant's cleanup obligations under the 2021 Order, involvement in these determinations is a safeguard both against modifications of response activities that might be done for purposes of and to reduce the calculation—and then implementation—of long-term cleanup costs, and to be confident in the determination of an adequate FAM.

6. Intervenors Need the 2021 Order to Include a Means to Resolve Disagreements with Defendant Regarding Permits, License, and Other Agreements Required by or Necessary for Defendant to Undertake the Response Actions Required by the 2021 Order

The response activities under the 2021 Order require Defendant to install a number of facilities such as monitoring wells and extraction wells in or on public properties or public rights-of-way. These facilities or types of facilities have been required under the existing Consent Judgment, as amended. However, the affected local governments were not parties to this litigation before they intervened, and the Consent Judgment has not had a provision for resolution of disputes between a local government and Defendant regarding permit or license terms for those facilities. For Defendant to be able to install the facilities it needs to install, and for the local governments to have the protections they need for their properties, rights-of-way, and the public, a dispute resolution process is necessary.[17] Intervenors request that the dispute resolution process under Section XVI of the 2021 Order be available to resolve disputes such as these.

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[17] For example, Defendant has approximately 130 monitoring wells in City of Ann Arbor rights-of-way and on City properties. Because almost all the license agreements for those placements had expired, the City proposed a Master License Agreement to cover all the wells with uniform terms and in a single document that also can be used for the additional monitoring wells the 2021 Order will require be installed. Although the City sent the draft Master License Agreement to Defendant on February 6, 2020, and although Defendant paid current and overdue license fees for all the wells, Defendant has neither executed the agreement nor provided any comments or reasons not to execute the agreement. Without a dispute resolution mechanism or the right to seek assistance from the Court, the situation is untenable.

7. Proposed Provisions to Provide for Intervenors' Ongoing Role and Rights

To incorporate their requests for inclusion going forward, as described and discussed above, Intervenors request that a new section be incorporated into the 2021 Order as follows, probably as Section XXI, which would result in all later sections being renumbered, starting with Record Retention becoming renumbered as XXII. [New provisions in Red].

XXI. ONGOING ROLE AND RIGHTS OF INTERVENORS

A. Termination of Response Activities. Before terminating or significantly reducing the response activities described in Sections V.A.3.f (Evergreen/Parklake), V.A.9 (Wagner Road), V.C.1 (Termination of Groundwater Systems), and VI.C.1–4 (Gelman Property Source Control) of this Order:

1. Defendant shall provide each Intervenor with its analysis supporting its position that the relevant response activity can be terminated or significantly reduced under the criteria listed in those 2021 Order Sections when Defendant provides that analysis to EGLE;
2. EGLE shall consult with the Intervenors and consider in good faith their comments and concerns with respect to the proposed termination/reduction of the response activities;
3. After such consultation, EGLE shall provide each of the Intervenors its written response to Defendant's analysis when it provides that response to Defendant; and
4. Any Intervenor may invoke dispute resolution under Section XVI of this Order if it disagrees with EGLE's response, and may fully participate in any dispute resolution process invoked under Section XVI of this Order.

B. Prohibition Zone Boundary Modification. With regard to modification of the boundaries of the Prohibition Zone established by Consent Judgment Section V.A.2 under either Sections V.A.2.f (Prohibition Zone Expansion) or V.A.6 (Prohibition Zone Boundary Review):

1. The Parties shall provide each Intervenor with all court filings filed pursuant to Sections V.A.2.f and/or V.A.6;
2. EGLE shall consult with Intervenors and consider in good faith their comments and concerns with respect to the proposed modification of the Prohibition Zone boundaries prior to filing any such filings with the Court; and
3. Any Intervenor may (1) participate fully in the court proceedings, including filing briefs and other documents to inform the Court of their comments and concerns.

C. Modification of Termination or Cleanup Criteria. With regard to modification of the termination or cleanup criteria under Section V.C:

1. Defendant shall provide each Intervenor with any proposal prepared pursuant to Section V.C.2.b when it provides the proposal to EGLE, together with all supporting documentation;
2. EGLE shall consult with Intervenors and consider in good faith their comments and concerns with respect to the proposed modification of the termination or cleanup criteria;
3. Following such consultation, EGLE shall provide Intervenors its response to Defendant's proposal when it provides the response to Defendant;
4. Any Intervenor may invoke the dispute resolution procedures described in Section XVI of this Order if it disagrees with EGLE's position. Moreover, any Intervenor may participate fully in any dispute resolution process initiated by Defendant under Section XVI of this Order; and
5. If Defendant invokes the procedures set forth in Section V.C.3:

    a. EGLE shall consult with Intervenors with respect to EGLE's selection of its panel member under Section V.C.3.a;
    b. Intervenors may provide the scientific advisory panel with any submissions requested by the panel under Section V.C.3.b;
    c. EGLE shall provide to Intervenors their response to the scientific advisory panel's recommendations when it provides said response to Defendant pursuant to Section V.C.3.c; and
    d. Any Intervenor may invoke the dispute resolution procedures described in Sections V.C.3.c and XVI of this Order if it disagrees with EGLE's position and may participate fully in any dispute resolution process invoked by Defendant under Sections V.C.3.c and XVI of this Order.

D. Termination of Post-Termination Monitoring. With regard to termination of post-termination monitoring under Section V.D of this Order:

1. Defendant shall provide a copy of any request to terminate post-termination monitoring under V.D to each Intervenor when it submits its request to EGLE;
2. EGLE shall consult with Intervenors and consider in good faith their comments and concerns with respect to the proposed termination of the post-termination monitoring;
3. Following such consultation, EGLE shall provide to Intervenors its written response to any request to terminate post-termination monitoring under V.D when it provides the response to Defendant; and
4. Any Intervenor may invoke dispute resolution under Section XVI of this Order if it disagrees with EGLE's response and may participate fully in any dispute resolution process invoked by Defendant under Section XVI.

E. <u>Groundwater-Surface Water Work Plans</u>. To the extent Defendant is required to submit a work plan(s) describing Response Activities and/or evaluations to be implemented/undertaken to address any area where groundwater is venting to surface water with 1,4-dioxane in concentrations that exceed the Generic GSI Criterion with respect to either the Eastern Area or Western Area, EGLE shall consult with Intervenors and consider in good faith their comments and concerns with respect to the adequacy of the proposed Response Activities and/or evaluations.

F. <u>Groundwater-Stormwater System Work Plans</u>. To the extent Defendant is required to submit a work plan(s) describing Response Activities and/or evaluations to be implemented/undertaken to address any area where groundwater with 1,4-dioxane at a detectible level is venting to or infiltrating into a municipal stormwater or storm sewer system, including drains of the Washtenaw County Water Resources Commission, EGLE shall consult with Intervenors and consider in good faith their comments and concerns with respect to the adequacy of the proposed Response Activities and/or evaluations.

G. <u>Financial Assurance Mechanism ("FAM") Requirements</u>. If EGLE is required to make a decision under Section XX.C of this Order, including but not limited to a determination as to the adequacy of the amount of the FAM submitted by the Defendant, approval of Defendant's periodic calculation of long-term cleanup costs, approval of a conversion of the form of the FAM, or a determination that Defendant is no longer required to maintain a FAM:

1. EGLE shall notify and consult with the Intervenors and consider in good faith their comments and concerns with respect to the determination being made by EGLE; and
2. Any Intervenor may invoke dispute resolution under Section XVI of this Order if it disagrees with EGLE's determination, and may participate fully in any dispute resolution process invoked by Defendant under Section XVI related to a determination by EGLE under Section XX.C.

H. <u>Certification and Termination</u>. When Defendant submits its Notification of Completion and draft final report under Section XXVI.A:

1. Defendant shall provide a copy of its Notification of Completion and draft final report to each Intervenor when it submits these documents to EGLE;
2. EGLE shall consult with Intervenors and consider in good faith their comments and concerns with respect to the Notice of Completion and draft final report;
3. After such consultation, EGLE shall provide its Certificate of Completion to each Intervenor when it provides the Certificate to Defendant;
4. Any Intervenor may invoke dispute resolution under Section XVI of this Order if it disagrees with EGLE's issuance of a Certificate of Completion, and may fully participate in any dispute resolution process invoked by

Defendant under Section XVI related to EGLE's failure to issue such Certificate.

I. Modification of this Order. This Order may not be modified by stipulation unless each of the Intervenors stipulates to the modification.

J. Use of Public Lands and Rights-of-Way by Defendant. Because Defendant must continue to occupy properties and public rights-of-way owned or under the control of one or more of the Intervenors for wells and other facilities, and will be required to place additional wells and other facilities on properties and public rights-of-way owned or under the control of one or more of the Intervenors, and because Intervenors require Defendant to get and comply with the others of permits and/or licenses to occupy those lands for those purposes, Defendant or an Intervenor may invoke dispute resolution under Section XVI of this Order to resolve disputes related to Defendant's use of the Intervenor's properties and/or public rights-of-way for its facilities.

M. Proposed Provisions Regarding Modification of the 2021 Order.

1. The possible need for modifications of the 2021 Order.

The Proposed 4th CJ recognized the possible need and provided for modifications, which might be stipulated by EGLE and Gelman, subject to ongoing rights of the Intervenors (provided in a separate proposed Order). This is true because Response Activities and Remedial Actions required by this Order are not static, but are initial requirements based on the current scientific and technical understanding of 1,4-dioxane that is present at and that has migrated from the Gelman Property. As stated at several places in the Intervenors' Expert Report, future data or knowledge may reveal the need for changes. See, as a few examples:

- Int. Exp. Rept. P.5 [The actions requested by the Intervenors "***represent initial actions needed to respond to the reduced groundwater cleanup standards. Additional remedial activities are likely to be necessary in response to information gained from the initial actions described herein."];
- Int. Exp. Rept. P.9 ["The need to install additional perimeter monitoring wells in strategic positions may become apparent after the results of the new wells proposed here and in the Proposed 4th CJ are analyzed."];
- Int. Exp. Rept. P.14 ["The Intervenors acknowledge that iterative investigations in areas of subsurface uncertainty, such as the region between the northern Prohibition Zone boundary and Barton Pond, are reasonable and

customary. *** In the event that 1,4-dioxane is detected in well DD, EE, or FF, additional investigations may be required to fully understand the hydraulic gradient and contaminant transport pathways in this area."];

- Int. Exp. Rept. P.17, regarding possible future investigations prompted by the results of investigations related to possible discharges to Allen Creek ["These activities are sequential, with each informing and optimizing the next. Information generated by any of these activities could lead to the need for additional investigations."];

- Int. Exp. Rept. P.30, regarding possible addition of extraction wells on Gelman Property ["installation of all proposed wells within a narrow time frame, with a contingency to add additional wells as individual well performance is assessed, will accelerate mass removal and enhance compliance with Western Area GSI objectives."]

2. Proposed provisions for possible need for modifications of the 2021 Order.

To address possible future needs for modifications to the Order, the Intervenors propose that the Section of the Proposed 4th CJ addressing Modifications (pp. 74-74) be amended as follows [with original provisions in black and new provisions in red]:

XXV. MODIFICATION

The Response Activities and Remedial Actions required by this Order are not static, but are initial requirements based on the current scientific and technical understanding of 1,4-dioxane that is present at and that has migrated from the Gelman Property. As more information and data become available in the future, it may be appropriate to modify this Order to increase or decrease the required Response Activities and Remedial Actions. As examples: Data from monitoring wells required herein may show the need for additional monitoring wells or other investigations to define the extent and subsurface transport of 1,4-dioxane; or Data from extraction wells or related to other Remedial Actions may show that added extraction or Remedial Actions may be needed in some areas and less may be appropriate in other areas. Any such modifications to this Order may be proposed by Plaintiff, by Defendant or by one or more of the Intervenors, and shall be subject to the dispute resolution provisions in Section XVI.

This Order may not be modified except by order of this Court. Remedial Plans, work plans, or other submissions made pursuant to this Order may be modified by mutual agreement of the Defendant and EGLE, subject to the ongoing rights of the Intervenors under Section XXI of this Order.

V. CONCLUSION

For the foregoing reasons and for the reasons provided and discussed in the Intervenors' Expert Report, the Intervenors respectfully request that their Proposed "**ORDER IMPLEMENTING REVISED CLEANUP CRITERIA AND MODIFYING EXISTING RESPONSE ACTIVITY ORDERS AND JUDGMENTS ("2021 ORDER")**" (attached hereto as Exhibit M) be entered by the Court.

Respectfully submitted:

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Dated: April 30, 20201.

STATE OF MICHIGAN

IN THE WASHTNAW COUNTY CIRCUIT COURT

ATTORNEY GENERAL FOR THE STATE OF MICHIGAN, *ex rel.* MICHIGAN DEPARTMENT OF NATURAL RESOURCES AND ENVIRONMENT,	Case No. 88-34734-CE Hon. Timothy P. Connors

Plaintiff,

and

CITY OF ANN ARBOR, WASHTENAW COUNTY, WASHTENAW COUNTY HEALTH DEPARTMENT, WASHTENAW COUNTY HEALTH OFFICER ELLEN RABINOWITZ, in her official capacity, the HURON RIVER WATERSHED COUNCIL, and SCIO TOWNSHIP,

Intervening Plaintiffs,

-v-

GELMAN SCIENCES, INC., d/b/a PALL LIFE SCIENCES, a Michigan Corporation,

Defendant.

INDEX OF EXHIBITS

Exhibit A	Complied Consent Judgment Changes
Exhibit B	Remediation and Enforcement Order
Exhibit C	EGLE Decision Document
Exhibit D	Gelman's Supplemental Filing in Support of Remedial Alternative
Exhibit E	Unit E Order
Exhibit F	Prohibition Zone Order
Exhibit G	Order re Potential Remedial Modifications
Exhibit H	Third Amendment to Consent Judgment
Exhibit I	RRD Policy and Procedure No. 33
Exhibit J	Stipulated Order Amending Previous Remediation Orders
Exhibit K	Finding of Emergency
Exhibit L	Proposed 4th Consent Judgment
Exhibit M	2021 Order
Exhibit N	Residential Well Sampling

STATE OF MICHIGAN

IN THE 22nd CIRCUIT COURT (WASHTENAW COUNTY)

ATTORNEY GENERAL FOR THE STATE
OF MICHIGAN ex. rel. MICHIGAN
DEPARTMENT OF NATURAL RESOURCES
AND ENVIRONMENT,
Plaintiff,
And

Case No. 88-34734-CE

THE CITY OF ANN ARBOR,
Intervenor,
And

WASHTENAW COUNTY,
Intervenor,

And

WASHTENAW COUNTY HEALTH
DEPARTMENT,
Intervenor,

And

WASHTENAW COUNTY HEALTH OFFICER
JIMENA LOVELUCK,
Intervenor,
And

THE HURON RIVER WATERSHED COUNCIL,
Intervenor,
And

SCIO TOWNSHIP,
Intervenor,

V.

GELMAN SCIENCES, INC., a Michigan
Corporation,
Defendant.
____./

EVIDENTIARY HEARING HELD VIA ZOOM VIDEOCONFERENCE

BEFORE THE HONORABLE TIMOTHY P. CONNORS

Ann Arbor, Michigan - Monday, May 3, 2021

APPEARANCES:

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TABLE OF CONTENTS

Ann Arbor, Michigan

Monday, May 3, 2021 - 9:09 a.m.

REFEREE SULLIVAN: So Judge, as you can see, we have a lot of people appearing on this case, so if we could keep the, only the people that are parties to the case with their video on and muted when they're not speaking. And I see a whole table full of people that I'm not sure who everybody is, but if everybody could just make sure that all the parties that are supposed to be here are -- are here, that would be really great.

THE COURT: Yes, that would be helpful.

Good morning, everyone. I know we have some observers, and you're welcome to observe. What Referee Sullivan was saying, if you're not a party to the case, if you could just take off your video and keep yourself muted. It's helpful for us to be able to see who's talking.

And then I guess, Mr. Negele, I'll just put it to you. Do we have the attorneys here? I can't -- there's so many people, sir.

MR. CALDWELL: Yes, Your Honor. This is Mike Caldwell on behalf of Gelman Sciences, and we are the group in the room, and I want to assure the Court that we are all wholly vaccinated, and I'm here, again, with Ray Ludwiszewski, Rachel Corley, Bruce Courtade on my far right, and Jim Brody right behind me.

THE COURT: Okay, good. Thank you. Mr. Caldwell, I appreciate that. So I'm going to speaker view because I could hear you but I could not see you. There are so many people on screen. So I think that's the best way for me to go. But do you think we have all the attorneys here?

MR. POSTEMA: Yes, Your Honor.

MR. CALDWELL: All -- all of our attorneys, sir.

THE COURT: Mr. Postema?

MR. POSTEMA: Yes. If you'd like us to introduce ourselves on behalf of the City. Stephen Postema, City attorney. With me, Abby Elias. The attorneys presenting today will be Fred Dindoffer and Nathan Dupes.

THE COURT: Yes.

MR. POSTEMA: So thank you.

THE COURT: Let's --

MR. POSTEMA: For the City of Ann Arbor.

THE COURT: Yeah, let's do this. Lindsay, why don’t you call the case, and then that way we can put all the appearances on the record.

REFEREE SULLIVAN: And just -- just before we get started with that, Your Honor, I know there's members of the press that are present, and if you could just let them know that recording this hearing is inappropriate. There's only one recording and that happens at the -- at the Court, and if they want a copy of it, they have to go through the regular procedure.

THE COURT: All right.

REFEREE SULLIVAN: They're not --

THE COURT: I think that -- I think that's from the Supreme Court. So why don’t we go ahead and have any members of the press, why don’t you unmute yourself, put your video on, introduce yourself, and then make sure we understand that. And now's the time to do it.

MS. GOODING: Hi. My name's Lily Gooding. I'm a student at the University of Michigan and I'm also a reporter for the Michigan Daily covering this case.

THE COURT: And you're welcome to cover, but do you understand --

MS. GOODING: Yes.

THE COURT: -- if you want to record -- okay. Whatever notes you take is fine. And the reason for that is there's been, in the past, litigants who have taken portions of the recording and, as if that's the whole recording. Why don’t tell us about that big screen behind you with that --

MS. GOODING: Oh, it's a --

THE COURT: -- California -- huh?

MS. GOODING: Yeah, it's a poster on my wall.

THE COURT: Oh, all right. Okay. Well, welcome.

Any other members of the press?

(No verbal response).

THE COURT: Okay. So attorneys, if you could, go ahead and put your appearances on the record.

THE CLERK: Judge, I need to call it.

MR. NEGELE: Good morning, Your Honor. Brian Negele, Assistant Attorney General, you know, representing the Department of Environment, Great Lakes, and Energy. I also have with me the author of our expert report, Kevin Lund. I see him onscreen at the moment now, too.

THE COURT: Thank you.

REFEREE SULLIVAN: Ms. Ostrowski has to call the case, Your Honor.

THE COURT: Oh. All right.

THE CLERK: Now on record, Frank J. Kelley versus Gelman Sciences, case number 88-34734-CE. This is set for an evidentiary hearing.

THE COURT: Thank you again. All right, sorry, sir. Go ahead and let's put the appearances on.

MR. NEGELE: I'll jump in again.

THE COURT: All right, thank you.

MR. NEGELE: Good morning, Your Honor. Brian

Negele, Assistant Attorney General representing the Department of Environment, Great Lakes, and Energy. And I have with, Kevin Lund, who is the author of our expert report.

THE COURT: Thank you.

MR. DINDOFFER: Your Honor, Frederick Dindoffer here representing the City of Ann Arbor.

MR. DUPES: Your Honor, Nathan Dupes, also on behalf of the City of Ann Arbor.

MR. POSTEMA: Stephen Postema with Abby Elias here also. Mr. Dindoffer and Mr. Dupes will be presenting testimony, and we have as our expert today for all of the Intervenors, Larry Lemke, who is on the screen, Your Honor.

MR. CALDWELL: Your Honor, once again, Mike Caldwell on behalf of Gelman Sciences. I'm with Ray Ludwiszewski, Rachel Corley, Bruce Courtade, and our expert, Jim Brody.

THE COURT: Thank you.

MS. METTE: Good morning, Your Honor. Erin Mette on behalf of the Huron River Watershed Council.

THE COURT: Good morning.

Mr. Stapleton, I can see you but I can't hear you. I think you're on this case.

MR. DAVIS: Your Honor, while you're tracking down Mr. Stapleton, Robert Davis on behalf of the County Intervenors.

THE COURT: Thank you.

Mr. Stapleton, I can see your mouth moving. I can see you're unmuted, but we can't hear you. I think it's your computer. Can you hear us? Thumbs up if you can. We can't hear you. So I think it's in -- I've had this difficulty before. I think it's probably on your screen, down to the bottom right. The little arrow that goes up, if you hit above that, you see a speaker after that comes up. And you have to increase your volume.

While he's doing that, I so miss the courtroom, folks.

Referee Sullivan, can you help him?

REFEREE SULLIVAN: So he is connected by audio. And did you turn your actual computer volume up? So you can hear us, but we can't hear you. So it has something to do with your microphone.

MR. DAVIS: Maybe you should try logging back in.

REFEREE SULLIVAN: That's always an option.

MR. CALDWELL: Your Honor, while we're awaiting Mr. Stapleton's re-login, I don't know but we, if Keith is on yet, but we have another expert witness that is involved here. I can't see all the pictures.

THE COURT: And what is that person's name? Because Ms. Ostrowski can probably --

MR. CALDWELL: Keith Gadway.

THE COURT: Ms. Ostrowski, is he there?

REFEREE SULLIVAN: Nobody by that name.

THE CLERK: No one's in the waiting room.

THE COURT: So maybe, you know, call him and tell him to log in.

Ms. Ostrowski, why don’t you let me know when Mr. Stapleton comes back in, okay?

THE CLERK: He's coming right now.

THE COURT: Okay.

REFEREE SULLIVAN: Mr. Stapleton, can you unmute?

No, it's not happening. Can you hear us, Mr. Stapleton?

(No verbal response; brief pause to address technical issues).

THE COURT: Referee Sullivan, same problem?

REFEREE SULLIVAN: Yeah. I'm not sure what the -- my only suggestion is he's going to have to try another device.

THE COURT: Yeah, I'm a little reluctant to proceed without all the attorneys here. I mean, I supposed if he can hear us.

REFEREE SULLIVAN: I don't think he can right now. He did before.

THE COURT: All right. Could we possibly have too many people in the room and -- is that what's causing the problem?

REFEREE SULLIVAN: No, Your Honor.

THE COURT: Okay.

REFEREE SULLIVAN: It's on Mr. Stapleton's end. It's something on his end.

THE COURT: Okay. Thank you.

REFEREE SULLIVAN: Yep.

(At 9:20 a.m., brief pause to continue to address technical issues.)

(At 9:21 a.m., proceedings resume.)

REFEREE SULLIVAN: Mr. Stapleton, can you hear us? Raise your hand if you can hear us. All right, excellent. That's part of it. Now, my guess is there's something wrong with your microphone that's in your computer. The only other suggestion I can make is if you have another device at least for the time being, an iPhone or an iPad, you can try that and you can see if we can get you connected that way.

(At 9:22 a.m., continued pause to address technical issues.)

(At 9:23 a.m., proceedings resume.)

THE COURT: Referee Sullivan, is he back? I can't see.

REFEREE SULLIVAN: He's back, but he cannot -- he can hear us. We cannot hear him still. And I gave him the advice of trying another device.

THE COURT: Okay, I think we're going to have to move forward.

So first of all, good morning. Thank you for joining. I've been reflecting a lot on our hearing today, and I've been re-reading a lot about Abraham Lincoln for various reasons, but one of the things that struck me is before he went on his political career, he was a very great trial lawyer. And the reason he was great is he could explain the most complicated things to a jury. And so that's really what I'm asking of the experts and the lawyers today, to explain to me the problem and what can be done, what should be done, how it can be done, and then for the parties to tell me what they think should be appropriate.

So with that, I'm totally open to an opening statement. I know the Court of Appeals weighed in I think on Thursday or Friday saying we should go ahead and proceed as scheduled, and then whatever decision I make will be subject for appellate review. So I guess I don't -- ah, are you in, Bill? No, we still can't hear you. So

I'm just going to have to proceed without you. I'm sorry. You know, try to find a different setup and everything, but there's so many people involved, Mr. Stapleton, I just, I have to -- I have to go ahead.

So with that I think Mr. Caldwell, if you'd like to make an opening statement, that would be fine, and then we'll go from there.

MR. CALDWELL: I appreciate it, Your Honor. I'm going to defer to Mr. Ludwiszewski on this.

MR. LUDWISZEWSKI: Your Honor --

THE COURT: First of all, good morning to you. I know we've got a difficult thing. It's hard to get a smile out of you, but we start with a smile because we're just really trying to find a resolution here, okay?

OPENING STATEMENTS - GELMAN SCIENCES - 9:26 a.m.

MR. LUDWISZEWSKI: Certainly, Your Honor. Well, I'm afraid I'm going to impose upon the Court for a few minutes. I'll try to be as brief as I can because I'm sure you understand I have an obligation to make my record. And then thereafter I have a third component of my opening that will be something that the Court hasn't heard and ruled on before that we would ask the Court think about as we proceed over the next few days.

We have slides. Is it acceptable to the Court if we project?

THE COURT: Absolutely. Lindsay, can you do the share screen?

THE CLERK: It's activated.

THE COURT: Okay, counsel, I can see this.

MR. LUDWISZEWSKI: Thank you, Your Honor.

Your Honor, we have three components to the opening. As I said, I'll be brief, especially with regard to the first two as I'm treading ground that we've all heard before and predominantly doing so to make sure that the record for any future appeal is preserved.

Your Honor, as you know, we believe that the Court lacks substantive power to hold this hearing on this topic. On the materials that were just given us on Friday, the Intervenors offer the Court three sources of authority for today's hearing. Oddly, or it may appear odd, we actually agree with each of the three powers that are identified by the Intervenors; however, they are facially inapplicable to today's situation.

Of course the Court is an arbiter in each suit between EGLE and Gelman. There is no dispute between EGLE and Gelman. We have a proposed consent decree since 2016 (unintelligible) Fourth Amended Consent Decree ready to go between those parties.

The Intervenors say that EGLE has a right to seek additional, go to the Court and ask for additional response activities. Of course that's also true. There's no motion for EGLE to do such a thing.

And then finally, the Intervenors offer the fact that the Court has the ability to enforce its own judgments and orders, and again, we take no issue with that except of course this is not hearing to enforce any order of the Court. It is a hearing to either create a new or modify an existing order. So all of the authorities offered to the Court for this hearing are inapplicable, and thus we believe there's no substantive basis for it.

Now, at times in re-reading the Court's transcripts in preparation, it appears as if the Court is saying that it is not re-opening the Consent Judgment, but instead holding a hearing, taking evidence, and making findings of fact, and then thereafter will simply issue an order, and that will just in this case, coming off of this evidentiary hearing, be a remedy order.

We believe there are two fundamental flaws with that approach, Your Honor. The first is the long procedural history of this case. There was a trial, and that trial did not end up with a liability finding against Gelman. It ended up with a, in the federal practice, which is what I'm more used to, a directed verdict in Gelman's favor on most parts of the case, and some remaining small elements that would have then gone to a defense. At that point, the parties, Gelman and EGLE, decided that a Consent Judgment could be negotiated, and it was, and that Consent Judgment has no liability finding against EGLE. So there's no liability finding which to hook the Court's remedy against EGLE.

And then finally, there's the procedural flaws in the hearing, the absence of standing by the Intervenors who are seeking relief, the absence of complaints, the absence of an opportunity to mount defenses, the failure of the designation of experts, the absence of discovery, all contravene the Michigan Rule of Evidence and rules of court and the Michigan and U.S. Constitution such that findings of fact would not be sustainable. But we've made those arguments, and now I've made my record, and I thank everyone for indulging me on it.

We have, would, however, like to conclude by, with something that the Court hasn't heard before, and we would ask the Court to think about the future precedent that would come out of this hearing as we move forward over the next few days.

The history of this matter so far is clear; Gelman and EGLE saw the change coming in the criterion, the cleanup standard. They met, negotiated, came up with a proposed Fourth Consent Judgment, and were essentially ready to lodge that with the Court for entry when the engineers arrived. EGLE would not agree to that Fourth Amended Consent Judgment if it wasn't fully protective of human health, and if it wasn't compliant with Michigan law, didn't satisfy Michigan law.

When the Intervenors arrived it's, there's no question, the record's clear, Gelman strenuously objected to their participation, but I think the record's also equally clear, and I believe the Court's noted it several times on the record, that Gelman participated in good faith for four years in those negotiations. And I can't get into the back and forth with the negotiations obviously, that would be improper, but the documents, as the next slide shows are -- are the result of the documents, of the negotiations, are public documents now, and perusing through them it's clear that the Intervenors offered meaningful consideration to Gelman to make concessions to go beyond what was required by Michigan law and required by EGLE to protect public health and the environment. They agreed to the PZ orders that had previously been negotiated by EGLE and Gelman. They agreed to dismiss their intervention with any future participation. They agreed to Park Lake discharges into First Sister Lake. And they told the public, they told everyone that those documents should all be viewed together; they're not in isolation. You can't peel them apart. It was a negotiated package.

And from the papers that were filed on Friday evening, it's clear that the engineers now want to revoke all their concessions and take all of Gelman's concessions for free. On top of that, they wish -- they want to add a wish list of additional demands to the agreement. We think that this is fundamentally unfair, but more important, Your Honor, it sets a troubling precedent for settlement negotiations overseen by this Court into the future.

Your Honor's well known as a strong supporter of settlement and for using innovative ways to get to that settlement. This is a very public case. Looking at the way this has proceeded, why would any Defendant in the future negotiate in good faith in settlement contexts under the auspices of this Court, knowing that going forward everything that they gave would be, would be expected, and everything that they thought would be stripped away? And why would any Plaintiff accept the results of those negotiations when they can instead skip the burden of proving their case, sidestep all of the defenses, hold on to every concession they got at the negotiating table, recover all the concessions that they gave at the negotiating table, and come to the Court with a long list of additional desires, wishes, and see how much they're going to be rewarded by the Court for rejecting four years of hard work at the negotiation table that was recommended to them by their lawyers and their experts?

So, we ask the Court to consider that carefully. Consider the history here. Consider the incentives that it's creating as we move to the future. And with that, Your Honor, we're ready to move forward.

THE COURT: I appreciate the -- let's go ahead and take this off the screen if we can.

I appreciate that opening statement. And I will tell all the parties, because I, you know, I'm going in my fourth decade on the bench, and I've always tried to tell attorneys, don't over state your case thinking I'm going to, you know, figure out somewhere in the middle. Give me your absolute -- give me the argument where it hurts, what you can live with, because that's what is persuasive to me, because I know that people have differences of opinion, but the idea behind this was to get as many people at the table and have them talk. So I do appreciate your comments. I also, I'm not offended about your preserving your appellate review of what I do. I get it. I mean, we all trained as lawyers, so I think that's very good.

I'd like to hear if I could -- or did you want me, Mr. Caldwell, I see you looking at somebody. Did you want me to hear from somebody else on opening statement?

MR. CALDWELL: No, Your Honor. I apologize.

REFEREE SULLIVAN: Your Honor?

THE COURT: I'd like to -- yes?

REFEREE SULLIVAN: Your Honor, before we go any further, for the people that are at the table, if when they're speaking, if they could identify who they are, because we have no way of knowing who is speaking because the whole picture lights up.

And for the gentleman that was just speaking, if he could identify himself and spell his name, just to make sure -- there's going to be I'm sure a transcript that has to be produced, and we want to make it as perfect as possible.

THE COURT: Thank you.

Counsel, that's a good point. Counsel, if you don’t mind, if you could just say your name again and spell it for the record so that when the Court of Appeals looks at it, they'll know that you're the one that made the argument.

MR. LUDWISZEWSKI: Certainly. I apologize for not doing so at the beginning. It's the cost of having us all been together for so very long. The attorney that was speaking, myself, was, my name is Raymond Ludwiszewski. The last name, Ludwiszewski, is spelled exactly as it sounds. L-u-d-w-i-s-z, as in zebra, -e-w-s-k-i.

THE COURT: I appreciate your humor.

MR. STAPLETON: Your Honor, William Stapleton. Can the Court hear me now?

THE COURT: Yes, we can. Thank you, Mr. Stapleton. I'm glad you're here.

MR. STAPLETON: Thank you. My --

THE COURT: I'm glad you're able --

MR. STAPLETON: -- was able to fix my --

THE COURT: -- to participate. Okay. I think I'd like to hear from the Attorney General next, please. Assistant Attorney General.

OPENING STATEMENTS - ATTORNEY GENERAL - 9:38 a.m.

MR. NEGELE: Yeah. Again, Brian Negele. If I need to spell it out, last name, N-e-g-e-l-e.

Your Honor, this has been a really unusual experiment in trying to craft revisions to a remedy. It's been evolving to like one degree or another for over 30 years. You know, over the nearly four years of negotiations my optimism, you know, waxed and waned, much like all the parties until shortly before we announced in August settlement, or status conference that we'd reached an agreement on the Fourth Amended and restated Consent

Judgment, and we described our plans to seek public comment on the Fourth CJ.

As we're all too aware, the public's comment on the proposed Fourth CJ were almost uniformly negative, which caused the elected officials of the local governor Intervenors to reject the settlement that their very experienced lawyers and experts had recommended to them.

So my statement is short. So, you know, here we are today with arguments about what more or what less should be in an order, you know, that would take place of that negotiated settlement. You know, again, we'd always prefer a negotiated settlement, but you know, we're here to take part in this process. Thank you.

THE COURT: Thank you.

I'm trying to think maybe from the County next?

MR. POSTEMA: Your Honor, I think the Intervenors have set up a, if I may, I think they have set up an opening statement that has a sequence to it that, that makes sense. If I'm wrong, certainly, and you'd like to hear from the County, that's up to you --

THE COURT: No, no, no.

MR. POSTEMA: -- but I would leave it up to them. I think they've set up a presentation. So if I'm incorrect about that -- otherwise I think the Intervenors have set up a certain methodology here.

THE COURT: You know, Mr. Postema, I appreciate that, and I also appreciate the fact when I did let Intervenors in, the argument that how many and, you know, and that. So I have appreciated that the Intervenors have communicated together and have tried to present their statement together, so I think that's entirely appropriate. So who will be speaking first on behalf of the Intervenors?

We actually heard from the two parties in the case who have indicated for the record they have reached accord. They object to the idea that Intervenors should have any say, and that will be subject to appellate review, although it's gone up and back a couple of times. But I have made that decision that I wanted that voice at the table, and I know that the Intervenors have been speaking, as the parties have indicated, for the last four years and had input into this. So go right ahead.

OPENING STATEMENTS - INTERVENORS - 9:43 a.m.

MR. DUPES: Thank you, Your Honor. This is Nathan Dupes, again, one of the outside attorneys representing the City of Ann Arbor. My last name is spelled D-u-p-e-s.

As Mr. Postema indicated, the Intervenors have prepared a joint opening statement, and if the Court will indulge me, we have a slide deck that we think will help walk the Court through our position and orient ourselves.

THE COURT: I think, Ms. Ostrowski, they still have that ability to share, right?

THE CLERK: That is correct.

MR. DUPES: All right. Can everyone see my screen which the first slide is State versus Gelman Sciences, Inc.?

(No verbal response).

MR. DUPES: All right, I'm seeing Your Honor nod your head, so that's good enough for me.

THE COURT: Yes. Yep. I can see it. Thank you.

MR. DUPES: So, Your Honor, you know, you received a lot of paper from the parties on Friday. I think that's an understatement. So the goal of our joint statement is to really step back, figure out where we are, where we came from, and why we're here today. Before I do that, I just wanted to briefly address some of the points that Mr. Ludwiszewski made on behalf of Gelman, most of which I think are probably properly addressed in a closing argument, but nevertheless, I want to make a couple of comments in response to what he said.

First of all, as to the arguments that he's making for his record on appeal, rather than rehash what we've, what the Intervenors have already argued, we'd rely on the arguments that we made in response to Gelman's motion for reconsideration as well as the other recent filings that Your Honor considered.

In terms of references to the proposed Fourth Consent Judgment that was made public, I just want to make a couple comments. First of all, as should be, as should go without saying, that was the result of years of negotiations. It was not the end all, be all of what the Intervenors wanted, and of course, as a product of negotiations, there was give and take, and I'm not going to go on to the specifics because that wouldn't be appropriate, but there was certainly never any representation I think to the Court, to the public that that Fourth Consent Judgment contained everything that the Intervenors believed was required by law or the science, which of course is what we're here today to talk about.

There was also references to this document that Gelman says that they had negotiated and finalized with EGLE at the time that Your Honor allowed us to intervene. Such a document has never been submitted to the Court for entry, and we object to any reference to negotiations over that document because, again, they were negotiations. Neither EGLE nor Gelman submitted such a document to the Court, and even today on the day of this hearing neither EGLE nor Gelman has tendered a proposed new Consent

Judgment or amendment that they both agree on. And in fact, if you look at even a cursory review of EGLE's brief that they filed on Friday, and Gelman's, today they are asking for very different things, and we'll talk about what those are later on.

And finally, just to orient ourselves, we are not here to talk about the extent of negotiations, what was offered, what was compromised. We're here today to respond to the purpose of this hearing as the Court set out, which is what is, what does the law and the science dictate should happen with this site? And that's -- and with that I'd like to just start by going through the slides. And Your Honor, I'll start our presentation, but one thing we've done, given the number of Intervenors and the number of subject matters, is we've divided our presentation today up among the attorneys based on subject matter, if you'll indulge that, which we think will make for a more efficient presentation.

So, getting back to why we are here, the first slide, Your Honor, is a picture of the Gelman site located on Wagner Road in Scio Township, and this is what we're here to talk about today. We're here because for decades Gelman, which was a manufacturer of filters, disposed of water containing high levels of 1,4-dioxane into the environment. They discharged it into the ponds that you can see on the screen here, which were intentionally unlined so that the wastewater could percolate into the ground in the soils, and enter the groundwater. They were designed for that purpose.

Wastewater was also discharged into a, and burned in this former burn pit. I don't know if you can see my cursor there, but it's right next to Gelman Building 1. And wastewater --

THE COURT: And I can -- Mr. Dupes, I can see it. Thank you. This is --

MR. DUPES: Okay, thank you.

THE COURT: -- very helpful, and I think I've disclosed to all of you I'm quite familiar with the area.

MR. DUPES: Yes. And then, and I'll make this brief, Your Honor, and then finally there's the former spray irrigation field where Gelman would spray the wastewater onto an open area. And again, that would percolate down and eventually reach the groundwater.

In all told, there was hundreds of thousands of pounds of this substance that was discharged into the environment from this property. The Gelman -- one of the things you're going to be hearing a lot about today is the orientation of the site and the geology and the hydrogeology because those are important technical points that drive what we're asking for today. So the Gelman site sits on top of a topographic ridge, meaning water slopes away from the site. And this area that the Gelman property, as well as the surrounding environs, is on a very complex piece of geology. There's sand and gravel aquifers interspersed with clay layers, and I think all the experts who are here today would agree that it's a highly heterogeneous site geologically speaking. And these features have complicated the efforts to remediate --

THE COURT: Mr. Dupes, I will interrupt you and everybody, because when I don't understand what you're saying to me, I ask for clarification.

MR. DUPES: Sure.

THE COURT: So when you're, that term you used about everybody agrees of the geological, you used a term. I don't know what that term means.

MR. DUPES: Heterogeneous, Your Honor?

THE COURT: Yes. I don't know what that means.

MR. DUPES: So I'll certainly defer the better explanation to our experts, but in laymen's terms, rather than have all of the subsurface be comprised of, simply sand or simply clay, right, there's pockets of sand and gravel which allow, you know, basically allow aquifers to form, along with pockets of clay, which are denser and essentially -- and typically prevent groundwater from moving from place to place. So it's kind of like a Swiss cheese or, where it's all interspersed of different glacial deposits so that, the effect of that meaning that groundwater can flow in many different directions.

THE COURT: And is that why, you know, in this area why we have trouble getting septic systems sometimes and why we need to, you know, individual sites is because of the nature of that? That we have these pockets, like if it's clay we can't really have septic systems coming in, you know, individual properties, a well?

MR. DUPES: I think that's right, Your Honor. I can't speak to it in detail, but I believe that's right. it certainly complicates, you know, well drilling and --

THE COURT: Right.

MR. DUPES: -- and also, and again, the placement -- the placement where you would find groundwater is not necessarily where you'd expect.

THE COURT: Okay. Thank you.

MR. DUPES: All right, so that's the Gelman site.

But what pollutant are we here to talk about? We're here to talk about 1,4-dioxane. This is a technical fact sheet publically available from the U.S. EPA. It's an industrial chemical. It's a likely human carcinogen. And importantly I've got some language here highlighted.

It's highly mobile and does not readily biodegrade in the environment. There is no limit to the amount of 1,4-dioxane that can be dissolved in water. It also doesn't readily stick to the soils. And it doesn't biodegrade. So all of those things combined, again, have led to the difficulty in remediating this contamination and also explain why it's been able to spread so far and for so long.

THE COURT: Let me ask you another question if I may. So is this the same chemical that DOW Chemical was using that went into the river and they're still cleaning up on that, or is this different?

MR. DUPES: No, Your Honor, I believe you're referring to dioxin with an "I."

THE COURT: Yeah.

MR. DUPES: Right, so this is 1 -- I'm -- I assume the parties today are probably going to shorten it to dioxane, but this chemical is 1,4-dioxane. A different substance.

THE COURT: And why was it made? I mean, why did they use, when Gelman did this, why, you know, what was the purpose of it?

MR. DUPES: So Gelman made filters, medical filters, and dioxane was used as a solvent in the manufacturing process. So it was used in the production of their filters, and then they were, that process created wastewater containing the dioxane, and then that was discharged into the environment, and from there it went into the ground, groundwater, flowed offsite.

THE COURT: Thank you.

MR. DUPES: You're welcome.

So most of us have probably seen this map before. This is the latest map of the Gelman remediation site and the plumes as prepared by the Washtenaw County Health Department. I'm going to zoom into a couple of these areas to just draw Your Honor's attention to particular spots. This is a zoomed in area of this map showing the Gelman site. You can see it sits right on Wagner Road. You can see a large concentration of monitoring wells. Those are the little circular legend with the black and the white, and then you can see a black and white cross, if you will, and then there's also wells with up arrows which are extraction wells which we'll talk about in a bit.

You can also see at the bottom of the page here, there's two arrows, one heading to the left, designated western area, and one to the right. And that's important because I believe from a Third Amendment to Consent Judgment forward, the remedial activities on the site have been divided into western area west of Wagner Road, and eastern area, east of Wagner Road.

This is an area of the, this is the eastern area essentially, and you'll see, Your Honor, this red hash line around a good portion of the City of Ann Arbor. That is the infamous Prohibition Zone, which we'll talk about a lot today, which essentially prohibits the use of groundwater within the area. And that Prohibition Zone was first entered by this Court in 2005 in a reaction to Gelman discovering that unbeknownst to EGLE or to Gelman, the contamination from the Gelman property had migrated into a deeper aquifer and in fact had spread much farther than any of the parties had anticipated.

This next slide, I put this up here, Your Honor, to orient ourselves a little bit. You know, this is a 1988 case that the State of Michigan brought, and a Consent Judgment was first entered in 1992. And so one might wonder why we're here today with all these filings and argument, and really the main reason is because of what the State of Michigan determined in the fall of 2016, which is despite having, you know, some 20, 30 years of experience with the site and the contaminate concern, the Department of Energy, Great Lakes, and Environment, which we'll just call EGLE today, and at the time was known as the Michigan Department of Environmental Quality, it issued a finding of emergency which found that releases of dioxane had occurred throughout Michigan that posed a threat to public health, safety, or welfare of its citizens and the environment.

The State went on to find that the extent of contamination is less than 85 parts per billion but greater than 7.2 parts per billion is unknown. And most importantly that the current cleanup criterion for 1,4-dioxane initially established in 2002 are outdated and not protective of public health. And by this rule which was signed by the governor October 27, 2016, this, the EGLE on an emergency basis reduced the cleanup criterion for dioxane for drinking water from 85 parts per billion down by more than order of magnitude, 7.2 parts per billion, and those rules were later made final, and as part of that final rule package, the State also lowered the existing cleanup criterion for the pathway that's protective of the interface between groundwater and surface water from 2,800 parts per billion to 280.

And I want to pause for a second, Your Honor, to talk about what cleanup criterion are. So cleanup criterion are established by EGLE and essential under EGLE's statutes, in particular Part 201 of the Michigan Natural Resources and Environmental Protection Act, which we're going to be talking about a lot today, and these are numerical values for a variety of contaminants that reflect EGLE's judgment of what's safe essentially for a variety of pathways. So if there's a pathway for drinking water, all right, what level is safe in the drinking water? I mentioned the groundwater/surface water interface pathway, essentially the part where groundwater vents to surface water bodies, such as the Huron River. And there's other criteria that aren't as relevant today. For example, there's direct contact with soil, et cetera.

And so in our minds, and I hope you'll excuse the pun, this was a watershed moment for this site because the criteria that had been used for over ten years the State of Michigan now determined was woefully inadequate to protect public health. So that's really the reason why we're here today, Your Honor, is to talk about what needs to change as a result of that rather drastic change in cleanup criterion.

Just briefly, Your Honor --

THE COURT: Let me -- if you -- Mr. Dupes, could you go back to that?

MR. DUPES: Yep. Sure, Your Honor.

THE COURT: So this, these emergency rules that were filed with the Secretary of State, that's back in October of 2016?

MR. DUPES: Correct.

THE COURT: And so that was sort of the impetus, am I right, that at least initially Gelman and the State started talking about this? And then when was it that I let the Intervenors in in this process?

MR. DUPES: Your Honor, to answer your first question, yes, the, Gelman and EGLE, I believe they already understood that the rules were going to be changed, so they started negotiating sometime before this, this finding of emergency and the emergency rules, but thereabouts.

And then I believe Your Honor started to allow the Intervenors in shortly after the emergency findings. I don't have the exact date, but it was in, I believe it was that fall.

THE COURT: You think within the same year?

MR. DUPES: I believe so, and somebody else, if -- I see Mr. Postema --

THE COURT: Mr. Postema, do you know, Mr. Postema, because I think you were one of the first Intervenors, or asking if you could step in, I'm just trying to make sure chronologically I understand what this is.

MR. POSTEMA: Yeah, I don't have the exact date, but we went in almost immediately I believe of that fall, and I'm being sent that information I believe right now, and so according to Ms. Elias, Attorney Elias, of course who knows all of these dates, it would be February 2017, Your Honor.

THE COURT: Okay, so within four months?

MR. POSTEMA: Yes. It was immediately recognized, as Mr. Dupes will talk about it, all of the Intervenors recognized the importance of this order of magnitude and getting in before Your Honor, that's correct.

THE COURT: So Mr. Postema, I know you're on the speaker right now, and I know other attorneys probably have some views on what's being said --

MR. POSTEMA: Yeah.

THE COURT: -- and I understand that, but my recollection is that that decision to allow the Intervenors within this short time period as these negotiations, that that issue went up to the Court of Appeals as to whether you had standing at all to be involved, and that ultimately the Supreme Court either didn’t -- I mean, that went up for appellate review; that's right, right?

MR. POSTEMA: I can have Mr. Dupes --

THE COURT: Okay.

MR. POSTEMA: Mr. Dupes can talk about that further, yes.

THE COURT: All right, thank you, Mr. Dupes.

MR. DUPES: Sure. And sorry for not having the exact dates, Your Honor, but I, I believe we moved to intervene that year, and then it may not have been granted until the following year as Ms. Elias pointed out.

But yeah, once Your Honor granted intervention, Gelman applied for leave to the Court of Appeals. That was denied. And they appealed that denial to the Michigan Supreme Court, that was denied. And then we engaged in the settlement negotiations which, you know, Your Honor is familiar about for the ensuing several years until it got to the point of the presenting the proposed Fourth Amended Consent Judgment.

THE COURT: But that, but that legal background is important to me because it was not the case where the Court of Appeals affirmed me; they just denied leave on it. And the same thing with the Supreme Court. So they kind of left it in our hands. Similar, you know, the same kind of thing that happened this week where they said motion for immediate consideration is granted but the appeal is denied, and they will look at whatever we, whatever ruling I do, whatever you all argue, and that anything could happen in the Court of Appeals or the Supreme Court based on what we do here in this hearing. Yes?

MR. DUPES: That's fair, Your Honor.

THE COURT: All right. That's helpful for me to understand. Thank you.

MR. DUPES: All right. So again, just to, back to the topic of where we are, how did we get here, what we talk about in our brief, Your Honor, the current court orders, and that's important because as we've explained in our response to the motion for reconsideration, this is not just a simple story of a bilateral Consent Judgment that's went on its merry way for years without change or without other orders. The Consent Judgment was first entered in 1992. It's been amended three times. This Court entered several separate orders that in our opinion significantly changed the remedial obligations on Gelman, and we have them listed here --

THE COURT: Mr. Dupes, I'm going to --

MR. DUPES: -- and just to --

THE COURT: I'm going to interrupt you again because I want this record clear for the Court of Appeals. When you say "this Court entered," you mean the Washtenaw County Trial Court? I'm the third Judge on this case.

MR. DUPES: That's correct, Your Honor.

THE COURT: Okay.

MR. DUPES: I believe it was your -- I believe it was your predecessor, Judge Shelton, who entered each of the remediation enforcement order, the Unit E order, and the PZ order.

THE COURT: Thank you.

MR. DUPES: So briefly, the remediation and enforcement order required Gelman to submit a plan to reduce dioxane in all affected water levels -- or excuse me -- in all affected water supplies below acceptable levels within five years, as well as install additional monitoring, extraction, treatment, and increase pumping rate, and, for reasons we'll discuss, Gelman didn’t, was not able to achieve that cleanup within the five years that it anticipated.

In 2001, this again was another significant moment in the history of this case, Gelman had discovered that the plume that it had thought that it had a pretty good handle on at the time, had migrated to a deeper aquifer, which the parties have called Unit E, and it was that finding that led Your Honor's predecessor to enter an order first creating the Prohibition Zone, which we're all familiar with, which as I said, is an area, a large area that restricts use of groundwater.

The Prohibition Zone was further expanded already, and once in 2011 with the Third Amendment to Consent Judgment after the plume contamination had migrated in yet another unexpected way.

And we point these things out, Your Honor, because we now hear Gelman explaining to the Court that they have a very good handle on where the plume is, where it's going to go, and one need look no further than the history of this case to show that for right or wrong, you know, the EGLE, Gelman, everybody looking at the case wasn't able to predict exactly where this plume was going to go, and that's for a lot of the reasons we talked about because of the geology of the site, because of the unique characteristics of dioxane which make it difficult to remediate, and so it's a complicated problem, and it's one that still warrants serious attention and additional activities to handle it.

As Your Honor pointed out in a recent hearing, we're talking about the pollution of our water, right, at bottom, and although the Prohibition Zone may be preventing anybody from drinking contaminated water, you know, it effectively condemns a large area of the City of Ann Arbor and its environs and takes away the beneficial use of that groundwater.

Briefly, our legal, the legal framework we're here to talk about today is really governed by two principal sources: Part 201, which deals with remediation of contaminated sites; and of course the existing set of orders and judgment that the, this Court, the Trial Court has entered over the years.

What I have -- what I have here on the screen is probably the most relevant section of Part 201 for today which talks about the duties of a owner or operator of a property that's a facility, and "facility" is a term of art in the statute which means that there are concentrations of one or more hazardous substances at the property in excess of the cleanup criteria for residential use that we talked about. And there's no question that the, Gelman is the owner of the property at issue, and that the property is a facility under that definition.

So under MCL 324.20114, a party -- and another term of art is a "liable party," right, so this describes the obligations of a liable party for contamination at the party's property, and these include determining the nature and extent of the release at the facility. So Your Honor's going to hear a lot about the term delineation; delineate the plume, delineate the contamination. And delineation is essentially a fancy way of saying determining the nature and extent of the contamination.

Moving down to subsection (c), "Immediately stop or prevent an ongoing release at the source." There's some disagreement here among the parties about whether in fact there's an ongoing release, but our contention is that, you know, by, at least by purposely taking contaminated wastewater, putting it into storage lagoons, and letting it seep into the environment, that that certainly is considered a release for purposes of Part 201.

Subsection (d), "Immediately implement measures to address, remove, or contain hazardous substances that are released after June 5th, 1995." You know, Gelman will argue that it's never -- it hasn't used dioxane since the eighties, and our position is that the word "release" in the statute is extremely broad, and includes leeching, omitting, spilling and, and I think the evidence will be clear that the Gelman site continues to be a source of an ongoing release of this hazardous substance.

And finally, subsection (g), "Diligently pursue response activities necessary to achieve the cleanup criteria established under this Part."

There are, EGLE has promulgated rules under Part 201, Your Honor, and there's a couple that we want to draw your attention to. These are from the Michigan Administrative Code, Rule 299.3, and these we call the aquifer protection rules. And you can see down in subsection (5) and (6), essentially what these rules provide, that the horizontal and vertical extent of a plume of contamination in an aquifer shall not increase after remedial actions have been initiated, and that remedial actions to address remediation of an aquifer shall provide for removal of the hazardous substance from the aquifer, either through act of remediation or as a result of naturally occurring biological or chemical processes. And there is a, there are caveats here, except as provided in certain other sections of Part 201 which deal with waivers, waivers from these rules.

This is, this is just the front page of the currently existing last amendment to the Consent Judgment. This is the Third Amendment, which entered in 2011. And this is just one provision of the Third Amendment, but it's probably one of the most important ones for today, and that is what are the objectives that were set out and established in the Consent Judgment? And the main one I think that's the important one for today in the Third Amendment is the systems that Gelman shall install shall be to, quote:

> "Extract the contaminated groundwater from the aquifers at designated locations for treatment (as required) and proper disposal to the extent necessary to prevent the plumes of groundwater contamination emanating from the GSI Property..."

Which is Gelman's property:

> "...from expanding beyond the current boundaries of such plumes, except into and

within the Prohibition Zone and Expanded
Prohibition Zone."

At this point I think it's important to talk about where the parties sit today, because it'd be easy to think that with all the papers that have been submitted and the arguments that you've heard today, that the parties are on different planets, and, and actually, Your Honor, in large part the parties are more aligned than you might think, and I think it's important to start by, before talking about what we disagree with, you know, where are areas that we agree.

THE COURT: Mr. Dupes, let me interrupt you again. You're going exactly what, from my standpoint my, what I feel is my responsibility, I really do want to see where we are in agreement, and then when I hear from the experts, you know, I really -- I don't know what this consent, proposed Consent Judgment was. You know, I don't know the details of it. You just told me you had reached and you were going to go out to the public with it. But when I hear from the experts, I'd like to hear -- let's use that as a starting point, what's good about the proposed Consent Judgment, as you said, what we agree on, and then where are the disagreements and why. And so I really do appreciate you saying, "Let's go back to where we are, where we agree with the proposed Consent Judgment, and then where do we disagree." That helps to, for me, to understand the conversation better. So thank you.

MR. DUPES: Sure, Your Honor. And our experts are prepared to do just that, so.

Just to briefly go over from the attorneys' standpoint where we are, where EGLE is currently arguing for entry of the proposed Fourth Consent Judgment as was made public with some minor modifications, the Intervenors, as you know, agree with what's in the proposed Fourth Consent Judgment, but want some modifications to certain language in there, as well as some additional response activities. And then Gelman's position today is that less than what was in the proposed Fourth Consent Judgment that was made public should be exceptive and protective.

But, as I said, there are significant areas where we're aligned. All parties agree, even Gelman, that the existing regime must be changed in light of the changed cleanup criteria. So every party before you today has submitted or argued for some type of change to the current orders in place. So that's not disputed. We're not here to talk about whether it should be changed; it's just the scope of those changes.

All parties agree what the objectives should be in a new cleanup order. That's -- that's huge. The parties are not disagreeing on fundamental objectives for the site.

And then you'll hear about this in more detail when we get to our experts, Your Honor, but all parties agree to many of these specific response activities; many of them.

And this, this last point, I've got it underlined because I think it's key and I hope Your Honor keeps it in mind throughout today and the rest of the hearings, that what the Intervenors are seeking beyond all of this are matters of degree and not kind. Okay. So for example, the proposed Fourth Consent Judgment would require new monitoring wells. Okay. The Intervenors are simply asking for several additional monitoring wells. The proposed Consent Judgment, Fourth Consent Judgment, and even today the proposed Consent Judgment that Gelman wants Your Honor to order includes additional extraction, additional pump and treat of groundwater. Well, the Intervenors are just asking for a little bit more of that and with some different termination criteria as you'll hear about. So I think that's important to point out is that we are not walking in here before Your Honor and asking for things that are drastically different than what's already on the table.

It's also important to clarify, given what was in some of the briefing that you read, Your Honor, is what the Intervenors are not seeking through this hearing. We're not seeking restoration of the aquifer. Okay. We and our technical experts recognize that given where the plume is today and the complexity of the site, that it would be infeasible to remediate the aquifer down to non-detect or even to the new drinking water standard.

We're also not asking for, that Your Honor will, you know, take out a pen and wipe the Prohibition Zone off the map.

I have this next one in quotes because I pulled it from Gelman's brief, we are not asking for a blanket of additional monitoring wells. Okay. There is some, I believe 140 current monitoring well locations at the site, Your Honor. The proposed Fourth Consent Judgment would require an additional 14 monitoring well locations, and the Intervenors are asking for eight additional locations beyond that in terms of monitoring wells. So we are certainly not asking to blanket the site in wells.

And I'm going to pause there for one second to clarify one point. When I say monitoring well locations, Your Honor, these wells, many of them are nested, which means that they have well screens at multiple depths, so either shallow, close to the ground surface, intermediate, a little bit farther below the ground surface, or deep.

THE COURT: Can you show me, or can you tell me where those proposed eight additional monitoring wells are located, or would be located?

MR. DUPES: Sure, Your Honor. Let me jump ahead briefly.

THE COURT: That's okay. If you're going to get to it --

MR. DUPES: We will get to it, Your Honor.

THE COURT: Yeah, that's fine.

MR. DUPES: In fact, let me do this.

THE COURT: Sure, no that's okay.

MR. DUPES: Okay.

THE COURT: You know, if you're going to address it later, that's fine.

MR. DUPES: Okay. All right, we will definitely address it in just a little bit.

And finally, the current Consent Judgment, Your Honor, has a requirement that Gelman prevents concentrations of 1,4-dioxane from migrating down gradient or east of Maple Road, in excess of the then existing groundwater/surface water interface criterion of 2,800 parts per billion. We are not arguing that that containment objective should be maintained with the new cleanup standard.

So again, I point these things out I hope to impress on the Court and to Gelman and to EGLE that again, what the Intervenors are seeking are not beyond the pale. In our view, they are reasonable, additional asks to address the new cleanup criteria, and are fully supported by the law and science as we'll explain.

All right, so I mentioned that the parties are all in agreement on what the objectives for the system should be going forward, Your Honor, so here are the objective in summary form in the proposed, and I'll just call it for ease of reference, and so with the other attorneys, the proposed Fourth CJ. This is the document that was presented to the public and was voted upon by the Intervenors. So for the eastern area the objective would be Prohibition Zone containment, which is consistent with what, the objective that's already in place through the Third Amendment, meaning Gelman would be required to take actions to prevent 1,4-dioxane from migrating beyond the Prohibition Zone boundary in excess of 7.2 parts per billion, the new cleanup standard.

GSI, again that stands for groundwater/surface water interface, Gelman would need to prevent concentrations of 1,4-dioxane from venting into surface waters above the GSI criterion, or as otherwise allowed by Part 201.

In the western area there, there is no

Prohibition Zone. Right. The Prohibition Zone lies entirely on the eastern area of the site. There the non-expansion objective is for Gelman to prevent the plume from expanding beyond where it currently is. Simply stated. And there are compliance wells in the western area that are used to document whether Gelman is meeting that non-expansion objective.

GSI, essentially the same thing that's required for the eastern area, preventing venting into surface waters above that GSI criterion.

And then the source area, and when we talk about source area, Your Honor, we mean Gelman, the site, the Gelman property. The obligation is to prevent non-compliance with the western area objectives. So essentially take actions that are necessary to prevent the source from triggering non-compliance with that western area, non-expansion and GSI objective.

So all the parties before you today, Your Honor, agree under these objectives.

All right, so now we're going to go into a little bit more detail, Your Honor, on what's in the proposed Fourth Consent Judgment and what are the additional things that the Intervenors are asking for. And here I'm going to deal with --

THE COURT: Mr. Dupes, I'm going to interrupt you for a minute.

MR. DUPES: Sure.

THE COURT: And I apologize. It's just that this is really helpful to me to be able to have this exchange because I'm the one that has to make the decision. And the reason I'm interrupting is the Intervenors are saying, "We are proposing these modifications," but the Intervenors already had signed off on a proposed Consent Judgment. So is it the position you're trying to take in the various clients that Intervenors have? I mean, nobody -- has anybody even signed off on these proposed changes that the attorneys are arguing? Or is it just like, you know, that's -- that's my difficulty because, you know, in good faith I listened to all of you, and then you went to your clients and your clients rejected it. So as I sit here today, how do you determine what you're asking for, and how is that going to, other than the fact you recognize, okay, we're in the court system now, but you're coming back with some proposed changes, and yet I don’t even have -- I don’t even have -- I don’t even know if the parties you represent agreed to that. Do you know what I'm --

MR. DUPES: Yes. That's a very fair question. Let me answer that and also clarify some points that were made earlier.

Your Honor, as to the Fourth, the proposed Fourth CJ that was made public, I think it's an overstatement to say that there was any signing off on it. I think what the Intervenors and the experts said was that in our mind it represented the farthest that the parties could really come through negotiations, and understanding the uncertainties of everything else in the case, that we were at that time prepared to call, you know, an end to negotiations saying we've resolved as much as we can through this process, and that's why we asked Your Honor to make the document public.

Now, of course as Gelman and EGLE have known from the beginning, the Intervenors are public bodies and can't approve a settlement unless they vote on it, and again, there was a, as Your Honor encouraged and we thought was appropriate as well, there was a robust public comment period where a number of concerns, valid concerns were raised by members of the public, and I believe all of our clients heard those, and that informed their vote on that document. And, but again, I don’t, I think it's a mischaracterization to say, which sometimes we hear from the Gelman side, that that was a wholesale rejection of the proposed Fourth Amended Consent Judgment because I think, again, what you'll hear today, Your Honor, is what are the additional things that we think, and when I say "we" I'll explain where we, why we get there, that would make this document acceptable and appropriate and protective of public health, the environment, and meet Gelman's obligations under the law. So again, that proposed Fourth Amended Consent Judgment was a product of settlement. It was not something that we said was all that should be required, but in light of the negotiations and litigation, that's what we were prepared to put before the clients for approval.

So then you asked me also, Your Honor, where do we get these things that we are currently putting before you as what we want now, and again, it's from input from our clients, and input from the comments from the public, and most importantly, input from our experts on what they believe is technically feasible, appropriate, and necessary to address the change in cleanup criteria.

THE COURT: Thank you, I --

MR. DUPES: And so --

THE COURT: You know, I'm asking you some very direct and hard questions, and I appreciate your candor, so you've answered my questions on that. Thank you.

MR. DUPES: Okay. You're welcome, Your Honor. I'm happy to. You're the most important person here, Judge, so don't apologize for interrupting me. Believe me.

THE COURT: Oh, I am not the most important person here. It's whatever three judges you draw from the Court of Appeals and the two of them who agree are the most important here. So we all know what we're doing here. We're just -- this is just establishing a record that will get appellate review. We all know it's going to happen. But thank you.

MR. DUPES: Thank you, Judge.

All right, so I am going to, Your Honor, talk about the Prohibition Zone and delineation, and at that point I will turn it over to some of my colleagues to talk some of the other components of what we're here to talk about.

So, the Prohibition Zone. The proposed Fourth Consent Judgment, Your Honor, included an expansion of the existing boundary of the Prohibition Zone in order to account for the change in cleanup criteria from 85 down to 7.2. The proposed expansion would be an approximately 25 percent increase in the area covered by the zone, and it would be an expansion both on the north side of the existing PZ, as well as the south side. Again, I have this underlined to emphasize that all parties agree that some expansion of the PZ boundary is appropriate to account for the change in the criteria, and again, provided that it's part of a package of additional response activities that Gelman would agree to do. The Intervenors don’t believe that an expansion by itself is appropriate. It needs to be a part of an entire remedial activity package and set of improvements.

So what is, what are the Intervenors proposing?

The Intervenors would be willing to accept the entirety of the northern expansion proposed by Gelman, but argue for a more limited expansion in the south. And so what do I mean by that?

This Your Honor, is a figure from the expert report that the Intervenors submitted to you on Friday, and this shows, among other things, the redline being the current Prohibition Zone boundary, and then -- or excuse me; actually this includes the expanded as well, but you can see in the blue shading the areas where the Intervenors believe is appropriate for an expansion. So you can see in the north the entirety of that proposed expansion is accepted, but in this south this is where we part ways with the other parties. The green area is the larger expansion to the south, and our experts believe that only the smaller blue area on the south is warranted because of the existing data, and in particular something called the concentration gradient, which essentially means that at this area of the plume there's a sharp drop off, so if you picture it going abruptly down a hill, Your

Honor, the space between 85 parts per billion and 7.2 parts per billion, which is the change in criteria, it drops off relatively quickly. So we don't believe that Gelman's argument for a greater buffer zone is technically justified, and we'll explain that.

Delineation. This is another big category, and again as I said this is tied to that Part 201 obligation of determining the nature and extent of the release. So given the change in cleanup criteria, all parties agree that additional monitoring wells need to be installed. So the proposed Fourth Consent Judgment would implement 14 new monitoring well locations, and I'll show you where those are, Your Honor, as you requested. That's coming up I believe in the next slide. The only caveat, and I'll go back a slide quickly, is Monitoring Well E, which you can see on the bottom of this slide, we have an arrow bumping it up. The Intervenors believe that should be moved to optimize its ability to track 1,4-dioxane, and our expert will explain why that's the case. But with that exception, the parties are in agreement on all of these well locations you now see before you.

So this, this map here was an attachment to the proposed Fourth CJ, and you can see Monitoring Wells A through N, so that's the 14 monitoring well locations, the parties are in agreement on installing each of those additional monitoring well locations. And again, as I said, in any one of those, in several of these locations they may be nested, meaning that there's actually multiple screens. So Gelman would be monitoring for dioxane at multiple depths in the aquifer. For simplicity's sake, I'm referring to locations as opposed to potential number of wells which could be greater. So those are the wells, those are the well locations we agree with, except with the exception of that location E moving in a little bit.

So one of the things that the Intervenors request in addition to what was in the proposed Fourth Consent Judgment, well the first thing we're asking for, Your Honor, is for Gelman to produce a map of the extent of contamination, and that's 1.0 parts per billion, 7.2 parts per billion, and 280 parts per billion concentration lines. And let me just quickly bump ahead so I can tell you what that, or show you an example of what that means.

So here is an example of an isoconcentration map that Gelman prepared. This one was from, looks like the quarter ending September 2020 of last year. This is a map that Gelman submitted to EGLE. And you can see there's hash lines here, and you can see here's one that says 85, right, so this is what Gelman believes based on data is the extent of contamination in the plume at 85 parts per billion concentration. You can see a few other concentration lines as you get more in the heart of the plume. Here's 2,000 parts per billion, here's 500. And so what we're talking about when we say a map of the extent of the contaminations, now that the criteria's been lowered, we think it's extremely important to drive, both to have a handle on the nature and extent of the contamination, and to drive response activities for remediation, it's imperative that Gelman publically release a map showing these new concentration values at 7.2 for drinking water, 280 for the groundwater/surface water interface criterion, and also at the detection limit for the method that Gelman uses for monitoring wells, which is at 1.0 parts per billion.

There are some additional monitoring well locations that we believe are appropriate because --

THE COURT: And Mister -- and counsel, this is what I was talking about earlier, so this is answering that question. So there were these additional was it 14 that was agreed to, monitoring wells?

MR. DUPES: Correct.

THE COURT: And so now there's an additional eight that's being requested?

MR. DUPES: Correct.

THE COURT: And that --

MR. DUPES: Well, the --

THE COURT: -- excuse me -- then you're going to show me where the location is of the additional eight, and then I'll hear from the experts why they think that's appropriate, right?

MR. DUPES: Correct.

THE COURT: Okay, go ahead.

MS. CORLEY: Your Honor, with apologies, this is Rachel Corley on behalf of Gelman Sciences. We were kicked out of the Zoom and were hoping that the Court could please let Gelman back in. It should be Zausmer P.C. in the waiting room.

REFEREE SULLIVAN: Erin, I'm --

THE COURT: I'm so sorry.

REFEREE SULLIVAN: I'm going to put you back.

THE COURT: How long have you been out?

(No verbal response).

THE COURT: I'll say it again, I miss the courtroom.

MR. DUPES: I agree, Your Honor.

REFEREE SULLIVAN: I let them in, so they should be in.

THE COURT: Yeah, we should establish for the record how long they've been out, because this is not a good thing.

(At 10:33 a.m., brief pause to address technical issues.)

(At 10:34 a.m., proceedings resume.)

THE COURT: Referee Sullivan?

REFEREE SULLIVAN: Yes, Your Honor.

THE COURT: You can just imagine, once we start trying to do jury trials again what a mess it's going to be.

REFEREE SULLIVAN: Yes, Your Honor.

So I let them in. They're not muted and their video is not available, so I'm not sure what's happening with them.

THE COURT: Okay. If you can hear us, Gelman Science, if you could hear us if you could please unmute yourselves. We want to make sure that you're hearing what's happening.

(No verbal response).

THE COURT: Mr. Stapleton, maybe you can give them a call and give them advice because you had trouble being in the room.

MS. CORLEY: Your Honor, this is Rachel Corley again for Gelman. We have two different computers going right now. The one that I'm speaking from is my laptop.

THE COURT: Okay.

MS. CORLEY: The system we were using we are trying to reboot. Can you hear me on my laptop?

THE COURT: Yes, thank you. We can hear you, but tell me when you were kicked out on the record. Do you know what time that was? Because I've been hearing the presentation from the Intervenors.

MS. CORLEY: It was prior to the start of the present slide, Your Honor.

THE COURT: All right. So you can see on your laptop?

MS. CORLEY: I can but unfortunately the rest of the group cannot, so this is just --

THE COURT: So they're all going to stand over your shoulder?

MS. CORLEY: I'd be happy to accommodate that.

THE COURT: No, I -- I -- so can we go back, if we could, Mr. Dupes. So I want to know, I'd like us to go back over the slide at least. So were you --

MR. DUPES: Here, let me go two back, Your Honor.

THE COURT: Okay, counsel, were you there for the discussion on the delineation?

MS. CORLEY: I don't believe we saw that either.

THE COURT: Okay, go back again.

MR. DUPES: Actually, that's -- this is the first slide for delineation.

THE COURT: But she says she, counsel for Gelman has indicated they don’t think they were there.

MR. DUPES: Okay, did you hear the part about the Prohibition Zone size?

UNIDENTIFIED SPEAKER: Yes.

THE COURT: You heard that? Okay.

MR. DUPES: I'll start here. I'll start here and kind of go over this from the beginning.

THE COURT: Let -- why don’t we do this; why don’t we take a five minute break? I'm going to get another cup of coffee, and let's see if Gelman Science -- it looks like maybe they're back on the screen.

MS. CORLEY: I'm still operating from the laptop at the moment, Your Honor. I think with the Court's indulgence a five minute break would be helpful to try and get that back up.

THE COURT: Let's do ten because it's important that you hear what's being presented so that, you know, you -- well, everybody. So let's take a ten minute break. It's currently -- well, why don’t we come back at a quarter two. That's nine minutes. Okay?

MR. DUPES: All right, Your Honor.

THE COURT: All right. Thank you.

(At 10:36 a.m., off the record.)

(At 10:45 a.m., proceedings resume.)

THE COURT: Okay, I think -- all right, I think you're connected on two different things. Am I right, Referee Sullivan?

MR. CALDWELL: We can hear and see you, Your Honor.

THE COURT: Yeah, but then there's still the one, Referee Sullivan, am I right, there's still another connection up there?

MS. CORLEY: Your Honor, that’s a laptop from which we were projecting our (unintelligible). I'd be happy to leave the Zoom, but if it's acceptable, I'd also be happy to stay in as Gelman Sciences for future projection.

THE COURT: What do you think, Referee?

THE CLERK: I think Referee Sullivan left.

THE COURT: What do you think, Ms. Ostrowski?

THE CLERK: I mean, as long as it's not causing feedback.

THE COURT: Okay.

THE CLERK: Which I don’t hear anything.

THE COURT: Okay, so if we could go back, Intervenors, and if you could make that argument again on this so they can hear from Gelman Sciences.

MR. DUPES: Sure, Your Honor.

MR. POSTEMA: Judge, excuse me --

MR. DUPES: Before I do I think --

THE COURT: Mr. Postema?

MR. POSTEMA: I think we needed Bob Davis back in from the County. He had dropped off I believe.

THE COURT: Thank you, Mr. Postema. I can't keep track of all this, so I appreciate that.

MR. DUPES: I think he's in the waiting room, Judge.

THE COURT: Lindsay, can you let him back in?

THE CLERK: He's in now.

THE COURT: All right. Mr. Davis are you there?

MR. DAVIS: I am.

THE COURT: You're there? Okay. Good. Thank you.

All right, deep breath. Try again.

MR. DAVIS: And Judge, thank you.

MR. DUPES: So, going back just a slide or two to delineation, well I started off, Judge, by saying that, observing that in the proposed Fourth Consent Judgment the parties had agreed on 14 new monitoring well locations, and that continues to be the case even today with the exception of a monitoring well E, which the Intervenors believe should be moved slightly to optimize its performance. And what I'm showing on the next slide are the 14 monitoring well locations. This was an attachment to the proposed Fourth CJ, and this represents the areas where the parties agree monitoring wells should be installed, additional monitoring wells.

So then that brought me to what are the Intervenors seeking for delineation beyond those 14 monitoring wells, and we start off by asking that Gelman be ordered to prepare a map of contamination at different concentrations. and the next slide is an example of such a map. This was prepared by Gelman submitted to EGLE near the end of last year, and these lines that have numbers attached to them are known as isoconcentration lines. So for example, you can see this outermost line with my cursor, it says 85. So this is what Gelman represents is what they believe to be the extent of 85 parts per billion dioxane concentration. There's other lines closer into the plume. Here's one for 2,000. So what this does is show graphically a representation of various concentrations of the plume in this area.

So what we're asking for is now that the standard has been lowered both for drinking water and for the GSI cleanup criteria, that those maps should be done at those new levels as well as the 1.0 part per billion, which is the detection limit for the method, the EPA method that Gelman uses for monitoring wells.

We're also asking for some additional perimeter monitoring wells, which I'll show in the, this slide, Your

Honor. This is what, I think the one you were interested in, that fill gaps in the current state of knowledge about the plume. So you can see here, you can see my cursor, AA is an additional well that the Intervenors propose along the northern boundary, the Prohibition Zone. BB, another well at that northern boundary to fill a gap in the current delineation. CC, which is in the western area, this is meant to take the place of a well that Gelman has taken off, previously took offline, but that we believe could give valuable information about the western area. And then you see three wells north of the expanded Prohibition Zone boundary, DD, EE, FF, and those are meant to, first of all, there's no existing monitoring wells in this area because it's beyond the Prohibition Zone, and these are meant to address the possibility, although admittedly small, that 1,4-dioxane may be migrating toward Barton Pond, which of course as Your Honor is probably aware if off this map to the north, but I think all parties would agree that even though the chances of an issue at Barton Pond may be small, the risk if such a event occurred would be tremendous, because that's where the City of Ann Arbor pulls the water that it uses for its municipal water supply. So our argument is that given the risk and the significant, you know, cost and public health issues that would be occasioned by dioxane reaching that,

that it's worth, you know, the additional incremental expense and work to do those additional monitoring wells. And again, our experts will explain why that's -- why that's scientifically justified.

That takes us up through FF. We have two more wells which are part of the expanded eastern area downgradient investigation. So you can see that Gelman is already agreeing to locations H, G, and F. This is near the West Park area near where dioxane may be venting into the Allen Creek Drain. And the two additional wells are GG and HH, which again are to plug gaps in the current state of information about where the plume is, where it's migrating at what concentrations, and perhaps most importantly to ensure GSI compliance. In other words, making sure that dioxane is not venting to surface water in excess of the new cleanup criterion for GSI, which is 280 parts per billion.

I believe -- let me make sure I went through all of them. Those are the additional monitoring well locations. So then going back a slide --

THE COURT: So Mr. Dupes, let me interrupt again, and I apologize for interrupting, but this is something I've been thinking about. I know the attorneys and the experts sort of had that starting point. We're going to go back and listen to that proposed Consent

Judgment was about, and I'm learning about it for the first time, but tell me, you know, in terms of your lawyers, and you know what the Court of Appeals may or may not do, but tell me if I was, if I were to go and say, "Okay, at least the people who know what we're talking about came to this, and we could move forward where there's agreement on it," so we start to have that, and I continue to have like a yearly review, what would be wrong with that proposal?

MR. DUPES: Your Honor, I don’t -- I think from -- people can chime in, but I think from the Intervenor's perspective I don't think we would have a problem with that type of approach. I mean, I think everybody agrees that this is an iterative process, right; our knowledge of where the plume has gone --

THE COURT: Right.

MR. DUPES: -- and treatment technology and the science, it's evolving, right, and so what was good 30 years ago is not good in 2021, so.

THE COURT: Right.

Lindsay, why don’t you put the lawyers into a breakout room, because that would be one way we could address this, saying, "Can we at least have this in place with a yearly review by this Court?" And my sense is the appellate court will not fight that. And I'm happy, otherwise I'm happy to, you know, have a trial.

Counsel, would that be okay with all of you?

(No verbal response).

THE COURT: I see, you're probably like texting each other.

MR. DAVIS: Your Honor, Bob Davis from the County. Are you talking about the Fourth Amended Consent --

THE COURT: Yeah.

MR. DAVIS: -- do that and then --

THE COURT: So what I -- so one suggestion would be that I at least adopt that right now, take whatever appellate issues you want up, and I'll have a review in year. But at least we're moving forward.

MR. DAVIS: Well, Your Honor, one --

THE COURT: By agreement. By agreement. So we could put that --

MR. DUPES: Well, Your Honor, one -- one issue is that, I guess from --

THE COURT: You just got texted.

MR. DUPES: Well, actually, no, this is something else. As you were talking I thought of Your Honor that the proposed Fourth Consent Judgment I guess is no longer on the table from the Gelman side. I mean, their brief basically -- what they're offering is not the proposed Fourth that was --

THE COURT: I understand that. You're all litigating different things, and so I'm offering something as an alternative and let the -- well, first of all, do you need to talk a little bit first individually and then come back?

MR. CALDWELL: Yeah, Your Honor, I think -- this is Mike Caldwell. I think that it would be helpful if we could talk amongst, you know, our individual --

THE COURT: Right. Right.

MR. CALDWELL: -- and then come back and a later time.

THE COURT: Yeah, I mean I'm happy to try the case, and I'll give you as many days as you need, but as I listen here, that would be one idea.

MR. CALDWELL: And Your Honor, if I may, would your idea because that you would essentially order the negotiated Fourth CJ --

THE COURT: Yes. Yes. With a, and then you can all do whatever appellate review you want, but you always come back to me within a year, let's see what's working, what's not working, what else might be needed. I mean I'm going to have continuing jurisdiction one way or the other. The point is we move forward what we agree to or you agree to, and then see how it's working. Because otherwise, Mr. Caldwell, it's just, you know, back and forth with the Court of Appeals and legal arguments; nothing's working, we're not moving forward.

MR. CALDWELL: Thank you for that clarification.

MR. POSTEMA: Judge, I think it would be helpful to hear from somebody of the other Intervenors' attorneys. One suggestion would be to finish the introduction so that you have the full scope, and you've obviously made a suggestion here, but I think on the timing it might be useful to finish up where we are, and then perhaps discuss anything at a lunch break. But really it's, it's obviously your call.

THE COURT: Mr. Postema, I've always respected you, I understand that, and I'm happy to listen to opening statements, but if you would like to do that, that's fine with me.

MR. CALDWELL: And Your Honor, if I may, if we do go down that route, we would like the opportunity to respond before we have this caucus. We obviously had a different impression of what the opening statements would be.

THE COURT: I know. Yeah, I hear you. I understand that.

So, actually, Mr. Postema, I'm going to go ahead and let you all talk. Why don’t we come back in, well, say 11:30.

MR. CALDWELL: Very well.

THE COURT: And then we'll see where we go from there, okay?

MR. DUPES: Thank you, Your Honor.

THE COURT: All right. I'll see you back at 11:30.

THE CLERK: Can you take down the screen share, please?

MR. DUPES: Yes, I will.

MR. DINDOFFER: Just to be clear, Your Honor, you want us all to sign out, and then sign back in at 11:30?

THE COURT: Yeah, I think that would be great.

MR. DINDOFFER: Thank you, Your Honor

THE COURT: Thank you.

(At 10:59 a.m., off the record.)

(At 11:30 a.m., proceedings resume.)

THE COURT: All right. Let's go back to the -- and if we can all -- Lindsay, if you call the case.

THE CLERK: People are still joining right now.

THE COURT: Okay.

MR. DINDOFFER: Your Honor, Mr. Davis asked us to let you know that he would be coming in under William -- oh, I see him there. Never mind. Hi, Bob.

THE COURT: Do you think we have everyone? Lindsay?

THE CLERK: I think so. We're back on the record --

THE COURT: Okay.

THE CLERK: -- in the matter of Frank Kelley versus Gelman Sciences, case number 88-34734-CE.

THE COURT: And thank you. So I think we're all connected, and I can see that Gelman has got their big table there, and welcome. Good to see you.

So, I had the attorneys just talk about an idea, and if it does not work, I'm happy to try the case. So, let me start with Gelman.

MR. LUDWISZEWSKI: Your Honor, we're attempting to reach our client who's on a transcontinental flight, but as Your Honor will recall from the opening today, the Fourth Amended CJ proposal was actually part of three documents: the Fourth Amended CJ, the settlement agreement, and the stipulated order. And the, much of the consideration that was given to Gelman in exchange for their concessions in the Fourth CJ are contained in those other documents. They were always represented within the negotiations and to the public as a package. One of the major parts of consideration was the assurance that we would be able to continue to work with EGLE and that this matter would not go, would not be referred to EPA. Sadly I believe that's been overcome by events, and I'm attempting to get authority from my client as I said. I believe there's some hope, certainly no guarantee, but some hope I could be persuasive on the Court's proposal if the consideration that we gave was met back with the concessions of the Intervenors' case. We would need not just the Fourth Amended, but also the settlement agreement and the entry of a stipulated order. If that were to happen, we would go and put it back in, this two component of the Fourth Amended CJ that we felt were inappropriate in the documents that you got on Friday, and we would unfortunately have permanently lost the EPA issue, which was very considerable, but I have some hope of talking my client into that. I certainly can't make any promises at this stage.

I don't believe I have any hope of talking my client into an arrangement where all of the consideration that we were given is taken out, so it's just the Fourth Amended CJ and not the other documents that came with it, but we are required to do everything and --

THE COURT: I know.

MR. LUDWISZEWSKI: Okay. Then you understand our position.

THE COURT: Yeah, I do. But, if I ordered this today, would you start doing it?

MR. LUDWISZEWSKI: If the Court is asking if you order -- if you order, entered the Fourth Amended CJ today as a court order --

THE COURT: Right.

MR. LUDWISZEWSKI: -- and did so without the countervailing protections, the countervailing concessions that were given to entice the entry of that order, entice the concession to the basis of the Fourth Amended CJ, would we start to do that work in lieu of an appeal, I don't have a, you know, I can't answer that question.

THE COURT: I understand that, but if the court order said, "You must start that today," and then you take the appellate issues, will you follow that?

MR. LUDWISZEWSKI: I don't believe we would be -- I don't believe my client has ever indicated it would not (unintelligible) as a court order. It might -- it might seek an immediate appeal on the stay of the court order.

THE COURT: That's fine.

MR. LUDWISZEWSKI: But, but again, Your Honor, we believe it's fundamentally unfair to enter just the Fourth Amended CJ. There is -- there is a stipulated order that would be entered not -- not as stipulated.

THE COURT: Okay.

MR. LUDWISZEWSKI: More or less --

THE COURT: But if I did that, and you go up to the Court of Appeals, which has rejected you, and you're ordered to do it, if I did that as an interim step and reviewed this every year, that would, you know, be some progress, would you agree?

MR. LUDWISZEWSKI: Well --

THE COURT: The things you agreed to.

MR. LUDWISZEWSKI: -- Your Honor, we agreed to them in exchange for concessions that are in the other documents, this is my opening, that, you know, and one of those concessions are permanently gone, but we're willing to, in cooperating with the Court, were willing to go, if it were willing to recognize that those events have occurred and we can't un-ring that bell but --

THE COURT: Okay.

MR. LUDWISZEWSKI: -- but the other protections that are in the stipulated order, and we don’t care if that's entered as a stipulated order or just as an order of the Court or --

THE COURT: Yeah.

MR. LUDWISZEWSKI: -- or the protections in the settlement agreement, then -- then it's hard to see how the Fourth Amended Judgment is fair under those circumstances.

THE COURT: Okay. Believe me. I know all that stuff. Okay, thank you so much.

Mr. Negele.

MR. NEGELE: Well, of course I've got to, you know, talk to the client, and that's not a, something that was able to happen really within the half hour or less, however long it was. And, you know, there are multiple levels of approval and review and all that sort of stuff, taking that approach. But, you know, as we've identified in our filing is that, you know, we think, you know, a lot of the stuff in the Fourth CJ is good and would be an improvement, but, you know, Mister, you know, Ray is talking about the, you know, the stipulated order of dismissal basically that contained all these rights that the Intervenors want to, you know, basically be able to have some sort of an oversight view basically of EGLE, you know, a consultation really. You know, maybe I mischaracterize it by calling it oversight, but consultation and all that sort of stuff, and that's where those come from. And, you know, those are things that we would not have normally done but to settle this. It's a, you know, it's really kind of an unprecedented situation that we agreed to there.

The EPA request, I don't know, maybe that can be undone, but the -- I'd also point out too that there's a lot of members of the community that believe that EPA is the only way to go.

THE COURT: I appreciate that. Thank you.

Mr. Postema, I don’t -- I really don’t want to go to the opening statements still; I'd just like to hear about my proposal, and if we don't have agreement -- well, I'd just like to hear from that.

MR. POSTEMA: Yeah, I think Mr. Stapleton was going to go first on this round to hear from the Intervenors, and so we'll -- we'll mix it up a little bit for you to keep everybody engaged around lunchtime, Judge. So, yeah.

MR. STAPLETON: Thank you, Your Honor.

Your Honor, you know, we, none of the Intervenor attorneys has had an opportunity to talk to our clients about this, and as you know, we face the issue of the Open Meetings Act Requirements, and we would, to consider any proposal like this, we would need to follow those procedures and consult with our clients, and we haven't had the ability to do that.

One point I would make, though, is if the Court was inclined to enter an order at this stage, one thing that is critical for the Intervenors, and I think the Court recognizes this, is to stay involved as Intervenors and retain our status as Intervenors. And the Court's suggestion of a one year review, you know, we would, the Intervenors would want to be part of that review as, as Intervenors in the case.

So it's a little bit difficult for us at this juncture, Your Honor, because we haven't had an opportunity to talk to our clients, but, you know, obviously it's up to the Court to decide on this issue, but that, just once again, the Intervenors maintaining status so that we can continue to have input, ongoing input in terms of what happens at this site is absolutely critical. And not -- I'll pass it to Mr. Davis at this point.

MR. DAVIS: Your Honor, Robert Davis on behalf of the County Defendants including the Health Department and the Health Director.

Your Honor, having carried Mr. Stapleton's briefcase throughout the duration of this case, I would agree with what he just said, but I would offer the following, I would be happy to take this suggestion that the Court has made to the County for review. I say that, Your Honor, both from a legal and from a science standpoint. When I look at some of the activities that are proposed in the current proposed Fourth Amended Consent document, some of those activities would likely take a year anyways. In other words, to get some of the things constructed, and we probably wouldn't even have some of the decisions necessary to carry out all the activities within that year. And I think the wisdom of the Court saying, "Let's review it in a year," gives us and our clients some time to see what has happened during the year. Maybe we, along with Dr. Lemke, we learn that there is a better position for one of those additional monitoring wells. I know Dr. Lemke is always looking at data, and I know Gelman is always looking at data, as is Brian, and maybe we all learn in one year there's a better location for something that's going on. But given the fact that some of this may take a year anyways, and as long as the County maintained a seat at the table to be heard next year, I would -- I would welcome the opportunity to present this to the County clients.

You know, Judge, I gave you a short brief on behalf of the Health Department and the Health Director. I know you read it with great intensity. And, you know, the duties that arrive from the Public Health Code with respect to two of my intervening parties are fairly unique, and you know, they are looking for certainty on some of these issues, which I think immediate action could help. They have to make day-to-day decisions, Judge. They have to make day-to-day decisions about drinking water wells, safe water, distances; they have to make decisions when the plume is within 100 feet of the proposed drinking water well. These decisions are critical, and you know, the more knowledge we have, the faster we have it, the better for my Health Department to carry out their statutory duties going forward. So as long as we have a seat at the table, Judge, you know, I would be more than happy to take this conversation to the County.

THE COURT: Thank you.

Ms., is it pronounced Mette?

MS. METTE: Yes, that's correct, Your Honor.

Yes, I agree with what my colleagues have stated, and we're certainly open to considering this proposal. I would of course need to consult with my client. But I also want to reiterate that a key issue for us is maintaining our status as Intervenors, and with that, you know, I would also be happy to present this proposal to my client.

THE COURT: So the idea would be as an interim order, I will order the consent agreement with review, and the Intervenors are still there, and so, you know, we can do all that appellate review, but at least we take one step forward. And I will keep continuing jurisdiction on this case with an annual review. Nobody has to come back. You know, I can do it six months if you want. But I'm comfortable with that.

But I'd like to hear from the people who are in the waiting room, and there's like -- who is Kathy Knol? What do you think? What do you think?

BEGIN PUBLIC COMMENT - 11:44 a.m.

MS. KNOL: I have some concerns and --

THE COURT: First of all, tell us who you are.

MS. KNOL: Okay.

THE COURT: You know, just introduce yourself, why you're here --

MS. KNOL: Kathy Knol.

THE COURT: -- and then --

MS. KNOL: Scio Township Trustee.

THE COURT: Okay.

MS. KNOL: I have been involved for over four years on Gelman issues. I'm a member of CARD. I've gotten communication from CARD members during this hearing this morning. I have major concerns about the fact that the CJ would have to stand alone. It should not be linked with the settlement agreement or the proposed order. And I hear that you are ordering it to stand alone, correct?

THE COURT: I'm doing that now.

MS. KNOL: Okay.

THE COURT: But I can tell you right now, I don't know what the Court of Appeals is going to do.

MS. KNOL: Okay, I just wanted --

THE COURT: But we're going to make progress.

MS. KNOL: -- okay, to verify. I don't know how our Township Board is going to feel about this. I have been in touch with one other Board Member during this hearing. She is very concerned. So there are issues we'll have to discuss. I'm glad we are maintaining Intervenor status; that will be important to all of the Intervenors. And I would want to clarify that any order that you've entered would not impact our petition for EPA involvement.

THE COURT: It would not.

MR. POSTEMA: Would not.

MS. KNOL: Correct.

THE COURT: It would not.

MS. KNOL: Okay. Okay, so I have reservations, and I've got --

THE COURT: Well, I do, too. I live in the plume. I'm in the plume.

MS. KNOL: I know.

THE COURT: I live right in the plume. I'm right across from it. I understand.

All right, Kevin. Can you hear me?

MR. LUND: Good afternoon, Judge. Yeah, I -- I live in a neighborhood there, too. My daughter delivered your paper when they were still delivering papers.

We're, as you said, we've got a lot of process on our end, and the agreements that were made were made, and concessions that were made are a bundle. They get to pluck one thing out of that bundle and use that makes the bundle less supportive, but we can go ahead from that.

And there's many things in the agreement that are, are protective of human health and the environment, the major role of EGLE is protection, and we want to verify. We created a model that we believe is pretty robust to better understand where things are and where they're not, and we'll continue to use that model more publically than we have in the past. I'm hopeful that we can put that on the internet in about a month or two, and I think that might help clarify some of the information that is being misrepresented, and be helpful for people to better understand where it is.

So in concept I think, and I speak for Brian too, but I think, you know, going forward is the best thing, finding a path forward and getting something, some of the work in the ground and start collecting data, and as are common with projects like this, your analysis evolves with more information. And EGLE's analysis has evolved over the years, and what that involvement, analysis has been in the big picture is EGLE and Gelman are reaching more consensus on the data than we have in the past. But again, the biggest part of what we do at EGLE is ensuring that there is no exposures and protection of human health and the environment and doing what we need to do that's allowed under the law.

THE COURT: Thank you.

Okay, I'm not doing a thing with City Council or Commissioners, but the next one on my screen is the one who gives me any funding to do anything. So Ms. Shink, if you don't mind, unmute yourself and just tell me what you think. And I need your help. I'm trying to do peacemaking. I keep telling you that, but go ahead. We can't hear you.

MS. SHINK: Thank you, Your Honor. I appreciate that you're interested in our input.

I'm trying to --

(At 11:50 a.m., connection issues occur.)

THE COURT: We can't hear you. We might have to go to Ms. Griswold.

MR. POSTEMA: And we have two Council members on. Thank you. And again, the Open Meetings Act issues and speaking for the whole Council, but we have two representatives here, so thank you, Judge.

THE COURT: Yeah, we -- Ms. Griswold -- Ms. Shink, I can't hear you, but we're going to go to Ms.

Griswold and she's been involved, and I appreciate that, and we'll come back to you Commissioner, okay?

MS. GRISWOLD: Kathy Griswold from City Council. I'm a member of CARD. I've been a very strong advocate of bringing in the EPA, especially because they have stronger polluter pay laws. I did not want to discredit the good work of EGLE in any way, but EGLE is bound by our state polluter pay laws, and so that's the big distinction.

I really appreciate this hearing. I appreciate your solution-oriented approach. There are, I think that there are two deal breakers that we cannot go back to our constituents about; one is the EPA, and the second one is the discharge into the First Sister Lake. I cannot -- I don't represent all of Council, but as one of the two Council members who has been most involved in this, I can tell you that I would appreciate some type of solution where we can immediately start applying the stricter standards.

So, thank you. I'll answer any questions you have.

THE COURT: No, no. Council person, first of all, are you my Council person?

MS. GRISWOLD: I'm sorry?

THE COURT: Are you in -- are you the one I report to?

MS. GRISWOLD: I -- I represent all of the citizens of Ann Arbor when it comes to water quality and cleanup, but no, you're not in my geographic area.

THE COURT: Okay, so I think I'm in -- I don't know if Commissioner Shink is in my district or not, but, no, I think that's the point; that we would have ongoing input, transparency --

MS. GRISWOLD: Uh-huh.

THE COURT: -- public hearings like this; what's working, what's not working.

MS. GRISWOLD: Uh-huh.

THE COURT: It goes solution driven, you know, so I can guarantee that to you, to the Council, even though you're not mine.

MS. GRISWOLD: Thank you.

THE COURT: So I'd like to go back to my, the head of the Commissioners. We couldn't hear you before.

MS. SHINK: Thank you, Your Honor. I added a hotspot. I hope that works. Can you hear me now?

THE COURT: Yes. Absolutely.

MS. SHINK: Thank you. Thank you for wanting to hear our opinions. I appreciate that. and thank you for trying to take a peacemaking approach in what is a very public situation. This is not a contractual situation between business -- business entities; this is about our community.

For the County, I would, you know, I am Chair of the Board, however I can't make decisions like this alone. I would be very willing to take this back to the Commission to have a conversation about it. The things that were sticking points for us, it was -- it was -- you know, we care about the public involvement, but we also, there were issues within the CJ that were problems kind of no matter what the public thought of them. Being able to go to EPA and have that process begin was important. Continuing to have a seat at the table as this cleanup continues, and the stricture standards, the 7.2, and then the discharge into Sister Lake and the lack of appropriate monitoring going toward Barton Pond, which is the source of water for the City of Ann Arbor, and also could potentially result if, you know, many years from now dioxane contaminating Ann Arbor Township's water.

So, as I said, I'd be very willing to take it back to the Commission. I don't know where that will come out. And there are a few issues that are, that are issues, but I think, you know, as long as it can go to EPA and we continue to have a seat at the table, and Sister Lake is protected, I think those are some of the big points. So thank you.

THE COURT: Yeah. You're welcome. And I don’t, you know, I'm just doing my job. I'm going to make a decision on, you know, I'm trying to give the power back, you know, for the input, but you absolutely will have a seat at the table, and I understand you can't speak for others. I could never speak for my other Judges because we have different views, so, I really appreciate that.

All right, I think we're to Mr. Hayner.

MR. POSTEMA: Yes, Mr. Hayner and Mr. Lemke, too. Yeah, thank you, Judge.

MR. HAYNER: Well, thank you, Your Honor, for recognizing me to speak here. I agree with what my elected colleagues said prior to this to specific things. I guess the most important thing to me is that we start operating under the new criteria, the new State criteria. I mean, I think that's critical, and I think my time on -- my time on CARD has shown me that there's differences in the data. People have different views of different data and it's not fully complete all the time, and I think when we start operating under the new criteria, we're going to see that we are on the edge with what's happening here and with this site.

With all due respect to the laws around with our Health Department and the State, the protective remedies are not the same as cleanups, and we've watched -- well, I mean I got really re-involved in this in '92 when I moved back here to Ann Arbor, '91, '92, and you know, we've watched it spread. I mean I, at the risk of holding something up here and not being seen, I mean this is what protection has got us; a huge spread of this plume from '92 to 2017. And so I feel strongly that protective remedies are not the same, which is one of the reasons I think it's essential that the EPA take a second look at this and, and we got it to be that way, and I know that was a challenge for this process, and I appreciate how challenging that was for our attorneys and everybody involved, but I'm just really concerned that continued protection is going to lead to a place where Ann Arbor is bound, like we are now, paying a huge fortune to filter out our water like we do with PFAS where the polluters aren't held accountable, and so, you know, we're paying a million a year to do that, and what's going to happen when dioxane is there?

And so I'm really concerned that protective remedies are not enough, and so anything that we can do and this Court can do to move that forward to, to change the mindset to more of a cleanup would be appreciated by me, and I'm sure a lot of folks in our city, so. Thank you.

THE COURT: Thank you. And Mr. Stapleton told me, you know, of course if the EPA comes in, I'll follow whatever directive they do. I'm just trying to -- I'm just, like all of you, I just live here. I'm just trying to use whatever I can do to move it forward. But I do appreciate your position. I have not ruled it out. I'm just trying to figure out what's the best step next, okay?

I see you nodding your head. Which I appreciate.

Okay, Michigan Daily, you've got to show your screen there. What have you learned today? What are you going to report out? What are you going to tell the community about what you saw? That's the real question. Because what you write will reach other ears, and that's your responsibility.

MS. GOODING: Yeah, well this, I would just like to say this is my first time sort of covering Gelman plume related issues, and it's an issue that we have tracked continuously, so this was sort of my first real experience listening to this sort of back and forth. But I think the main thing that's come out of this is obviously this proposal to sort of adopt the agenda right now, so I think that's probably going to be the focus. Yeah.

THE COURT: Thank you.

I know Jack Eaton from a while. Come on, Jack. What do you think?

MR. EATON: Well, I don't hold any elected office, and so I'm just speaking as a concerned resident and a member of CARD. I've remained involved with CARD. And I would point out, I appreciate that you want to get something rolling. You know, there are new criteria that need to be applied, but there are a number of things in this agreement that really trouble some of us, such as not allowing the Intervenors to seek EPA intervention. The idea that we might use Sister Lake as a depository for partially cleaned up water, including a 500 part per billion standard in this Consent Judgment, it has no basis in science or law. That -- that number was just pulled out of somebody's hat.

So, I do have some real concerns with applying the Consent Judgment that was rejected because of the problems that are included in that, especially if the polluter is going to insist that we take it as a package deal with the other two documents. So, that's just my thinking, Judge. And thank you for the opportunity to talk.

THE COURT: Are you kidding me? Good to see you again.

Okay, I think we have Kristen. Tell us who you -- or oh, it's actually Beth Collins. No, Kristen. I'm sorry. Kristen, go ahead.

MS. SCHWEIGHOEFER: I'm not sure why my video's not on, but.

THE COURT: Yeah, we don't know.

MS. SCHWEIGHOEFER: It's set to be on. Anyway, it was working earlier. Apologies.

THE COURT: It's okay. It's okay. We all deal with it. I mean, in this hearing we've been struggling. But just tell us who you are and why you care about this and what you think.

MS. SCHWEIGHOEFER: Sure. My name is Kristen Schweighoefer. I'm the Environmental Health Director with the Washtenaw County Health Department. So I've been involved in this --

THE COURT: There you go --

MS. SCHWEIGHOEFER: There I am.

THE COURT: We can it.

MS. SCHWEIGHOEFER: I just had to apparently -- technically.

I've been involved in this since I, well, I've been with the Health Department for 21 years, but I, probably this site for the better part of seven or eight years with the position I'm in now. And I've seen a lot of newer things, and you know, I'm excited about a lot of the progress that's been made. It's been very interesting to be part of this history-making process.

You know, I share a lot of what I've heard today about concerns with others on some of the aspects of Consent Judgments, but everything is a give and take; nothing is going to be perfect.

THE COURT: Yeah.

MS. SCHWEIGHOEFER: So I appreciate all the work that's been done, and I have given this a lot of thought, and I don't know that I have a perfect answer for any of this. You know, I hear from the members of CARD and citizens and our elected officials and, you know, I'm in many of those meetings, and again I think that the decision before everyone is very difficult today, and I don't know that I have a lot of wisdom to answer beyond what you've already heard here. Thank you.

THE COURT: No, that was wisdom. It helped me. There is no perfect answer. Every time I have to make a decision about everything, you know, and there's no good answer. So I think that was great wisdom. Thank you.

Okay, Ms. Collins, I think you're up. That's the next one on my screen.

MS. COLLINS: Hi.

THE COURT: Hi.

MS. COLLINS: Your Honor.

THE COURT: Tell us about yourself and who you are and why you care.

MS. COLLINS: I'm a resident and I'm across the

Sister Lake from you.

THE COURT: Okay.

MS. COLLINS: And, yes, your council members are Ali Ramlawi and Erica Briggs.

THE COURT: Thank you.

MS. COLLINS: Our council, so.

But, no, I got involved and just started reading the old articles. I grew up in Ann Arbor, so it wasn't a new topic to me, but when I moved to this neighborhood, you know, the one that the wells were contaminated, I started reading old district library articles on Gelman, and it's funny because when you search Gelman, you get all the profits and all those years of really doing well, too, in addition to contaminating our wells and our aquifer.

I mean, I just -- so lately I've enjoyed that we've maybe been getting some more justice for the public, and the polluter needs to start realizing that this is 2021, and it's different than it was in the eighties. We can't keep contaminating the environment. The, for Sister Lake discharge of course upset me a lot because it's person here, and I think there have been studies even since the CJ that showed that, you know, it probably would damage the wetlands and do damage. And so most of us residents we're all against this, and there are experts within the public too is something I've learned that --

thank you for listening to us and having all the public comments that we were able to have at all the different public meetings we did. And, so, thank you for listening, and I really enjoyed this. I wish we were in person in court, but.

THE COURT: Yeah, me too. I hate this, I just hate it, and we'll probably meet on the street, but tell me which high school you went to?

MS. COLLINS: Pioneer.

THE COURT: Ah.

MS. COLLINS: I grew up in Georgetown. I was born I Georgetown, and then we moved to Brockman a little later, so.

THE COURT: Yeah, I understand. I was like the opening one with the River Rats --

MS. COLLINS: Oh, good.

THE COURT: -- at Huron High School. Yeah, I know. And so --

MS. COLLINS: Well.

THE COURT: And so you know why we are the River Rats. Do you know why?

MS. COLLINS: Right on the Huron.

THE COURT: No.

MS. COLLINS: But I don't know why the rats.

THE COURT: No. No. Because of you. Because of all of you. I was on the school board and, you know, student council. When we first opened up in 1969, of course it was very controversial back then, and they thought it was the Taj Mahal, and Pioneer was just backed up -- did your parents go to Pioneer?

MS. COLLINS: No, they were both Detroiters --

THE COURT: All right.

MS. COLLINS: -- that met at Eastern --

THE COURT: All right.

MS. COLLINS: -- and said, "We're not moving back to Detroit," so.

THE COURT: All right, so I'm going to give you the background.

MS. COLLINS: Okay.

THE COURT: The school opens up, 1972. They already thought they overspent, they over did, and we're down there, and the principal who had left Pioneer, said, "We're going to be the green and gold," which is what the athletic director said. We said we're okay with that. And the mascots were actually like, I don’t even remember what it was, the Trojans or something, and so we opened up school and you know how hockey is so big in Ann Arbor, right? So, you know, we're kind of pulled apart, but the hockey kids were really competitive, and we beat them, you know, in the first game. And so the Pioneer kids stole all these white rats out of the science labs, threw them on the ice, kind of like, you know, Joe Louis with the octopus, and said, "You're a bunch of skanky rats down by the river." This is the true story.

MS. COLLINS: That's great.

THE COURT: So they came back and we said, "We're going to embrace, we are going to embrace that insult, and we will be the River Rats." And the principal was so offended by that.

So I was part of student council. We went to the school board. The school board said they have the right to vote, every school that opens up. We said, "We're fine with the colors, but we want to be the River Rats," because that's the insult. And it had to go all the way up to the appellate process. So they said, "You've got to vote on it." And so we voted on it, but the principal said, "I'm not putting that name on the ballot." We had a write-in ballot; 99 percent said, "We want to be the River Rats." So. And so we, we used to joke about Pioneer saying, "And you were the ones who gave us an insult? You're the Pioneer Pioneers. You can't come up with anything original." So anyway.

MS. COLLINS: Oh.

THE COURT: Thank you for, you know, I hope to meet you on the street.

MS. COLLINS: Thank you.

THE COURT: All right. Let's go to, I can't even see who else is here.

Oh, Mr. Rayle. On the next screen. Mr. Rayle, go ahead. Tell us about yourself and why you're here.

MR. RAYLE: Well, I really appreciate that story about the River Rats. I've heard that again that there was maybe three options that the administration proposed for mascots.

THE COURT: Right.

MR. RAYLE: And the students said, "No way. We're going to embrace the River Rats with the write-in."

THE COURT: Right.

MR. RAYLE: Which shows the power of the public in a situation like that, which is really not unlike what we're dealing with right now, because I've been involved with the Gelman thing since 1993, so I'm in my twenty-eighth year as a citizen volunteer watching over this site. It wasn't -- I expected to be involved maybe a year or two, and in fact, at the end of the second year I helped negotiate a settlement with the then president of the company, Kim Davis, that everybody agreed to. We had all the, the same stakeholders that are involved in CARD now, and Chuck Gelman took the cleanup back away from Kim Davis and reneged on that agreement.

So, I've been trying to get back to some proper settlement ever since, because I might be the only one on the screen who is on a well in Scio Township real close to the Honey Creek, so this affects me personally. But at the time this happened, I lived in the City where the plume is now. It wasn't there when I lived there. And at the time I got involved, I worked over the western plume in Parkland Plaza on top of what we're still dealing with with the spreading of the western plume. And my whole career I've dealt with information technically systems for local governors and other public organizations for 40 years. So it's like fate tapped me on the shoulder and said, you know, "You're it. We need your resources." And I've taken that to heart, because once I see a problem, I really try to get it solved.

So I the intervening years, as a result of early involvement by other citizens and citizen involvement groups, in 1995 we set up a non-profit so we could get some information out to the public. Scio Residents for Safe Water was formed, and that's still providing a lot of the resources that you see when you go to the CARD meeting and the CARD site, because we have a funding mechanism through that non-profit to provide support for CARD. And since nineteen -- since CARD was more formalized, it was formed originally in 2006 as an output of the

Intergovernmental Partnership Organization by the local governments and citizens, but we formalized it in 2006 as a kind of a loose coalition of local governments and citizens to have regular meetings to discuss the Gelman situation. And then in 2016 it was formalized even more with bylaws and that. The members elected me as Chair of CARD.

And so I'm still Chair of CARD, founding member, founding member and Chair of Scio Residents for Safe Water. And I probably have studied this site more than anybody on the screen, and it's kind of a little upsetting to me when I see misstatements, even today, that are allowed to stand. It's, you know, maybe I'm being nitpicky, but when it comes to water, clean drinking water, we have to be as -- we have to be as persistent as the compound we're trying to get cleaned up. And dioxane is one of those forever compounds that once it gets anaerobic in groundwater, it tends to stay there. The preferred treatment is pump and treat, and there are methods to treat it to non-detect. We actually had that in that 1995 agreement, to treat to less than 3 parts per billion. The company has shown they can do that. Even recently with their ozone treatment, just this last month new data showed that they were able to treat to less than, to zero to two parts per billion, even with their ozone treatment. Of course the ozone creates a new carcinogen, bromate, so that's why we don't like the ozone treatment.

But the fact that there's this information that's out there that's not getting to some of the decision makers, including you, is troubling. You haven't seen a lot -- I haven't seen in any court documents any of the plume displays that SRSW has created and provided at various CARD meetings. We have CARD meetings once a month that last two years, so we're discussing this all the time. The problem is that we go, we revert back to the court situation, and some of that information is not making it to you or to some of the other parties.

I like your idea of moving forward, and there are a lot of problems with the Fourth Consent Judgment proposal. It wasn't made public until September. August 30th I think it was. We had a short period of time to comment on it. We made our elected officials aware of those problems. Now, there are some good things about it, one of which has already been discussed, which is to tighten the cleanup standards, 7.2 parts per billion for discharge, and to 284 groundwater/surface water interface.

So one of the things I've been suggesting is that instead of going, jumping straight to Consent Judgment 4.0, take it as a step like, do a Consent Judgment 3.1, and have those cleanup standards be in effect immediately. Because that's something that we -- it's been more than 10 years since the EPA guidelines suggested that that's going to happen. And we know the company is already prepared for that because there's even been a couple plume maps that they apparently created a 7.2 isocontour for their own use, and then erased those contours except for a couple segments before they made it public. You may not know this; maybe some of the people on the -- you know, the 7.2 is something the company was prepared for because they actually had a map that had some 7.2 lines on it, and they just didn't erase them all before they made it public.

The issue about the deep aquifer, E aquifer being contaminated and only being discovered in 2001. Now, the company knew about that in the late eighties, 1980s, because their own supply well was contaminated at 3 parts per billion, according to their data. Now, of course that data is not in electronic form; that's on paper form.

And then their NW30D, which is in the E aquifer, was the only well east of Wagner Road, and that was contaminated three times what the cleanup standard was in 1993. And then the company was allowed to not sample that well for seven or eight years until dioxane was discovered at other points in the site. They went back and resampled that well, and it was over 60 parts per billion in nineteen -- or 2000 I think it was. And then it went on to be over 1,000 parts per billion, and that's the part that's heading north/northeast or maybe even northwest from the Evergreen area.

So there's a lot we don't know yet about this that has to be taken, like you say, a step at a time. We have to continue to review it because there's always new information.

But my well is closer to the Honey Creek, and about the same depth as the home owner well on Breezewood that was contaminated at 1 part per billion a year or so ago. And that could be happening all along Honey Creek, and we don't know if it's from the plume moving straight there, if it's from leakage from the creek from the discharge up to the 7 parts per billion. But we need to find this out; we need to take action now, take action now to prevent future problems. We can't wait for those problems to happen because as you can see, it's really hard to clean up the aquifer once it's been contaminated. So we have to do a better job of constraining -- of doing this -- we need -- we need due diligence to match the scale of the problem. This is probably the nation's, maybe the world's largest dioxane contamination of its type because they used it pure, most of the dioxane sites were used as a stabilizer and other industrial solvents. But here Gelman used it pure to make their high-tech filters. And we haven't found another site that's like that. This is the largest site of its type, so.

I'm available later to answer any questions you might have, but I just wanted to get that in. I really appreciate you taking comments from the public because this really makes a difference to me.

THE COURT: Thank you. That's great, Mr. Rayle. I want to tell you something that, when you talk about the history of this project, I'm going into my fourth decade on the bench, but before that Judge Conlin, who was the first Judge while this was being litigated, and then it was Judge Shelton and now Judge me, but I was a lawyer, and the litigation used to, on the docket would, I mean it just, nobody else could be heard. And so finally Judge Conlin said, "I'm going to make Connors a special master just to get through the discovery motions and make a recommendation." So we've been around a while together.

And then on Honey Creek, I want to tell you that, it's so interesting, so, to me, probably not to -- well, I think because you will share it, I know all about Honey Creek, and my wife is the, I mean, she's the great supporter of my life, but her grandparents grew right up next to Honey Creek, I mean, and they had the house, and my wife remembers going there, and we show our grandkids and everything, the joy there, so I know everything about Honey Creek.

But the only, the last thing I just want to ask is, tell me a little bit about where you grew up and what your background is? I understand you, you know, you found this tap on the shoulder about Gelman, but your story is bigger than that.

MR. RAYLE: Well, I grew up in Traverse City.

THE COURT: I know Traverse City.

MR. RAYLE: So I grew up on Incochee Farm. Do you know where that is?

THE COURT: I don't know that. I know the tribes up there very, very well.

MR. RAYLE: No, the Incochee was actually a name borrowed from a Georgia tribe.

THE COURT: Okay.

MR. RAYLE: By the early owner of Incochee Farm.

THE COURT: Okay.

MR. RAYLE: It means, oh gosh, good will or something like that. I'd have to look it up again. But it was right on the edge of the city limits, so I had full range of this 160 acre farm.

THE COURT: Right.

MR. RAYLE: But I also was able to go to city schools. In fact, I used to walk through the apple orchard to get to the elementary school. And we could see the bay from the upstairs window of the farmhouse, which is no longer there because it's been developed and the whole property is, you know, the lots start at like 100,000 or something like that.

THE COURT: Right.

MR. RAYLE: But I was basically a son of a former sharecropper.

THE COURT: Okay.

MR. RAYLE: So.

THE COURT: I got it.

MR. RAYLE: I went to Michigan, met my wife, had our kids here. Well, not here. Actually I'm in California right now with our first grandchild. We'll finally be able to see her in person after, I don't know, a whole year, so.

THE COURT: Man.

MR. RAYLE: We just had her birthday celebration yesterday. Anyway, I went to school at Michigan, studied industrial engineering, bachelor's and master's, and got involved in local government systems for all my career, and then as I wound down, I helped with the entrepreneurial community and things like that that I still help with.

But I appreciate your being special master. Just to -- the fact that somebody thinks they know everything about this site, no one could know everything about this site.

THE COURT: I know that.

MR. RAYLE: I learned from you that you were special master. That was news to me. I didn't know there was a special master in those early days.

THE COURT: I'm not even sure, I didn't know that either, so we share that.

MR. RAYLE: Yeah, and we were asking for a special master be appointed, use the fees from the, Gelman got charged fees by the DNR at the time, it became DEQ, assess those fees to Gelman, hire a special master, this is something your predecessor could have done, and help, so the Judge can have somebody to gather all the complex information and present it to them to help with decisions. And you might be under the same boat. You might need to have a special master if you're going to review this every year.

THE COURT: Oh, oh. You're talking about, yeah, early on when I had that. Yeah, early on in my career. Yeah, you're right about that, but it --

MR. RAYLE: Well, you might even need that now --

THE COURT: I know but --

MR. RAYLE: -- because there's so much complexity on the site.

THE COURT: And I do want to acknowledge, we're still on the record, and the Court of Appeals is going to think I'm crazy for listening to all of you, and they could all object, but I'm just going to do it. And so I really thank you for that. And I do love the Traverse City area, and congratulations on your grandchild. My wife and I are blessed. We've got six, but two of them have special needs, and so we're home schooling right now. They got COVID when they went back to school, and then my son-in-law has COVID and he's been in the hospital, so we're all dealing with this.

MR. RAYLE: Jeeze.

THE COURT: But the grandchildren are something special. And I learn more about being a grandparent from them then I ever learned from anybody in school. So good luck on your trip there.

MR. RAYLE: Thank you.

We should talk about where your parents or wife's parents, or whatever, lived on Honey Creek. I'd like to know more about that.

THE COURT: Yeah, we'll talk all about it. I'm, you know, you can reach out to me. I'm happy to talk to you. But I see there's --

MR. RAYLE: Thank you.

THE COURT: -- Mr. McKee? Is that you? Hand is up, wants to be heard. There you go.

MR. MCKEE: Hi --

THE COURT: Hi.

MR. MCKEE: -- Judge Connors. We crossed paths many years ago playing basketball at Mack School.

THE COURT: Yes, we did. I forgot that.

MR. MCKEE: Remember Mike Stemford (phonetic) and (unintelligible) --

THE COURT: Yes, we did.

MR. MCKEE: -- were in that game, too, as a regular Monday night game at Mack School for --

THE COURT: Yes, we did, didn't we?

MR. MCKEE: And I was not one of the better players, but I had a good time.

THE COURT: And I wasn't either, but we did it together.

MR. MCKEE: We had a good time.

THE COURT: Yeah.

MR. MCKEE: I just, I want to, and I appreciate very much your willingness to let the public speak here, and I'll try to be to the point. I wanted to amplify and echo what Jack Eaton and Roger Rayle and Beth Collins said, and I think -- I think I wanted to briefly go through what my view of the history is. I've lived in Ann Arbor since '71. I used to work at the U-Seller Warehouse (phonetic) across Jackson Plaza from Gelman. And I've only been recently involved in following this issue. I always figured there's a lot of people involved, and I had a busy law practice back then.

But now the recent history is troubling, and the, this Consent Judgment was negotiated by the Intervenors and Gelman and EGLE, and was presented in, back in September as a good agreement. And it was the activists, Mr. Rayle, Mr. Bicknell, and many other CARD members that said, "No, this is not a good deal." The activist residents were overwhelmingly opposed to the Fourth Consent Judgment. At the various public hearings there was not one single resident who spoke in favor of that deal; not one. There were at least 100 people that spoke against. And the reasons I think are important.

There are many good things about this Consent Judgment, but it had and has a number of what I'd call poison pills in it. And I think that the appropriate way to understand how it was reached, and again, I wasn't in the room so I, you know, the ins and outs and the details of the negotiation are way beyond what I know, but what my understanding is, is that it came to a point and the City

Council Ann Arbor people said to their lawyers, "Please bring back whatever you can get from Gelman because we need to end this process." And the fact is that the offer that was made by Gelman was simply as far as they would go, which is pretty typical in a negotiation. You know, they weren't going to go any farther than this. And I think to characterize it as a settlement that had been reached is not accurate because the clients, which the Intervenor, elected officials, and public, have never agreed to anything. Their lawyers asked Gelman to put the best deal that they were willing to put on the table, and they did. It was soundly rejected. And to treat it now as kind of a basis, this we should just use it, is really going against pretty much the wishes of the entire public. And I think there, like I said at the beginning, there are a lot of good parts of it, but those poison pill cannot be agreed to, the EPA piece and the -- there's a provision in there that allows Gelman to not run any of the extraction wells if parked, if something, if those wells are brought down to 500 parts per billion. There's no scientific basis for that number as Mr. Eaton said. I think all the experts would, that are on the screen here would agree with that.

So there are definitely pieces that should be done, like immediately going to the lower cleanup criteria and many other things, but the agreement is lacking in its scope, and it also has these poison pills. So I think to just take the Fourth Consent Judgment and enter it on an interim basis would not be appropriate here. There would have to be at a minimum some excision of the parts that are, that are not appropriate and were rejected by the public. And that's a process that can be done, but I think where Mr. Dupes was in his presentation was really sort of showing, "Okay, this is what we need to add." I don't think he started to even get to what we should subtract because the, after you made your proposal obviously he hadn't, you know, really had an opportunity to respond to that part of it. And I think that part is really important to recognize and deal with.

That's what I have. Thank you for letting me speak.

THE COURT: Of course. And I really, you know, first of all, thank you for reminding me that we played basketball together all those years ago.

You know, this is not the end. I've lived in this community forever, and I'm going to stay on this case as long as I'm a Judge, and I hope I have a few more years. I just want to take the next step, but I appreciate that you, you know, you care; you care about this and you speak up. And, you know, we'll just go see what the next step is, but thank you very much. Okay.

MR. MCKEE: Thank you.

THE COURT: All right. Who else has not had a chance to speak? And if you'd like to, please do.

And I need to tell you, Ralph, all these lawyers are furious at me for letting you talk like this on the record, but.

MR. MCKEE: Well, I'm -- I'm retired, so you know.

THE COURT: It doesn't matter. It doesn't matter. They could be objecting like crazy and they're probably --

MR. MCKEE: Mike Caldwell is an old friend of mine. We worked a lot of cases together, so.

THE COURT: All right, you want to talk about basketball then?

MR. DAVIS: Your Honor, Rob Davis --

THE COURT: You know Mike Caldwell? Mike Caldwell used to be a great basketball player, except the only good basketball player on this screen is Bill Stapleton. He was All-City with Magic Johnson in East Lansing. And Bill Stapleton actually drove us to the Hall of Fame in the Macker stuff. So the fact you know Mike Caldwell, and Mike Caldwell can't stand that, can he, Bill?

MR. CALDWELL: Your Honor, that has been something that has been under my skin since I found out about it, considering all the Gus Mackers I played in. And I, but I do agree with your assessment of the relative basketball skills of the people involved; that Stapleton clearly is way out front.

MR. STAPLETON: Your Honor, I concur with everything that's been said about my basketball skills.

MR. DAVIS: Your Honor, Robert Davis. Is it possible Mr. Lemke, or, do you, would you like to speak?

MR. LEMKE: Yes.

THE COURT: Yeah, I think Mr. Lemke was there. I'm sorry, Mr. Lemke. I saw you earlier, but then my screen keeps, you know, changing. So yes, I'd like to hear from Mr. Lemke.

MR. LEMKE: No problem. Thank you, Your Honor. My name is Lawrence Lemke. I'm one of the experts for the Intervenors. I've been familiar with this site since 1997 when I moved to Ann Arbor, and I've used it, along with many of my students, as a case study, and an opportunity to move some of the many things we've learned along the way into more generalizations that can help here and elsewhere as well.

I think that this idea of implementing what's contained within the proposed Fourth Consent Judgment now is a step in the right direction. I think that it's essential that we get some forward progress and some action and the ability to address the new significantly lower cleanup standards that have been adopted by the state. And I think it's entirely likely that it's going to take more than a year to implement all the things, at least to begin all of the things that are contained within the Fourth Consent Judgment. There are some things in there that I'm not particularly fond of, but it is a negotiated agreement, and I think everybody's been upfront about that from the beginning.

But what I'm really excited about, the parts that I think are really most beneficial are the remediation activities in the source area, the additional mass removal from pumping wells, and just the whole idea of adding those additional monitoring wells in key sensitive locations.

There are strong technical arguments I believe for additional activities that are really needed and necessary because what in my opinion is contained within the Fourth Consent Judgment is necessary, important, but not sufficient to completely address everything that needs to be done. But if we have an opportunity to come back in a year after we've learned probably a great deal from the implementation of those wells and have a chance to make technical arguments and review that material, I think that maybe there is additional things that could be done.

And I agree with what Mr. Lund said, that there would be potential changes along the way, but the community's concerns, particularly the concerns over First Sister Lake and discharges into First Sister Lake need to be addressed. I'm not an expert on NPDES permits, but it's probably likely that if a permit were applied for, that process would take more than a year to play out, so there's time to review that as well.

Other than that, I don't have any other comments to make other than it has been a very interesting process to watch this play out. I think that the idea that all of the parties involved have a lot more in common than they differ is true. I think we're differing in the area of degree that we would approach to solve these problems, and I think that moving forward now is helpful because dioxane and groundwater and these plumes, they continue to evolve, they continue to move, they're not static, and any delays make it all the more harder to address the problem. Thank you.

ORDER - 12:35 p.m.

THE COURT: Thank you, sir.

Counsel, what I'm thinking is what I, in the order of Mr. Stapleton, I think they've kind of always told you you have to draft up the opinion or the order from what I say. But I'm thinking that of course it would be the year review, but should I put in either a monthly status conference the first of the month just to see if there's any issues, or quarterly or, you know, whenever you think? And I'd like counsel to weigh in on that because I care about this obviously; I live here. And, so what do you think? What do you think about when you want to see me again? I know I'm probably kind of difficult to deal with, but.

MR. STAPLETON: Yes, Your Honor, William Stapleton. I think -- I think maybe quarterly reviews would be an excellent idea. You know, as everyone has said, this is an iterative process, and I think if we were able to sort of, you know, review and reconvene every quarter, you know, I think that would be sufficient to sort of check in and see how things are going at the site because there's constantly data being gathered at this site from the monitoring wells, from the extraction wells. It's an evolving process and it's important to stay on top of it. So you know, I think just, you know, speaking for Scio, I think Scio would very much support a quarterly review process.

THE COURT: And so counsel, you know, if we just had that on the agenda, I'm open to anybody joining in on that to have this discussion. I just want to make sure it's not like swept under the rug. But you know, if issues come up, we could do it that way.

Mr. McKee, what do you think?

MR. MCKEE: I think --

THE COURT: Is that good?

MR. MCKEE: -- a quarterly review would be fine if you, if we don't get stuck with the provisions in the order that are poison pills.

THE COURT: Well, we're going to start with that, and then we're going to do a quarterly review. I'll listen. But I just want to make sure, it's not like a motion or a, you know, a formal thing; that I'm on it, okay?

And the Court of Appeals is going to do whatever they want to do. Counsel, are you okay with that?

MR. DAVIS: I'm good, Judge. Quarterly sounds reasonable to me. It gives time for things to evolve.

MR. CALDWELL: Your Honor, we'll review whatever the order provides with our client.

THE COURT: Okay. I understand.

MR. POSTEMA: Judge, you're talking about -- excuse me, Judge. You're talking about the proposed interim order that would get started that you would like to take, us to take to our clients, and we've said that we would all take to them, and adding this additional thing about quarterly reviews to actually deal with issues come up. Is that correct?

THE COURT: That's correct. Instead of -- so first of all, I'm going to order Mr. Stapleton, I'm going to order that we put into effect right now the proposed Consent Judgment. I would still like to have quarterly review of where things stand, because I know things go up to the Court of Appeals and then I finally -- I mean, I heard on Thursday or Friday. So I'd like to just be able to let people weigh in, where it stands, so it's more than an annual review. I'm just proposing quarterly review.

MR. STAPLETON: And Your Honor, just for clarification, if the Fourth CJ were to be entered now, would the Intervenors retain Intervenor status so we could --

THE COURT: Absolutely.

MR. POSTEMA: Yes.

THE COURT: Absolutely.

MR. STAPLETON: Thank you.

THE COURT: And so the idea, I'm going to have quarterly review just to see where things are standing, instead of annual review. I want to know what's going on. And then the Court of Appeals, you know, all parties are free to appeal me, and the Court of Appeals is free to tell me what I'm doing is wrong.

MR. POSTEMA: And Judge, did you, you talked about doing an interim order on your own, not a consent order --

THE COURT: No, it's my order. That's right, it's my order.

MR. POSTEMA: And the Fourth CJ, the additional documents that they had talked about, the settlement and the other orders, those are not part of it because --

THE COURT: No.

MR. POSTEMA: -- the EPA is gone --

THE COURT: No.

MR. POSTEMA: -- it doesn't make any sense. Right.

THE COURT: No. I am absolutely just saying I'm ordering the proposed Consent Judgment, and then I want to say on it every quarter, and the Court of Appeals, you know, can decide whether that's appropriate or not. And then I'd like the Court of Appeals to weigh in frankly before I take any additional steps. Are you with me?

MR. STAPLETON: So Your Honor, and just so I'm clear --

THE COURT: Sure.

MR. STAPLETON: -- because it sounds like you would like me to draft something and send it out to counsel.

THE COURT: Yes, I do.

MR. STAPLETON: So three components: entry of the proposed Fourth Amended CJ now.

THE COURT: Yes.

MR. STAPLETON: Quarterly review where the parties and the Intervenors review the progress at the site.

THE COURT: Yes.

MR. STAPLETON: And Intervenors retain their status as Intervenors.

THE COURT: Yes.

MR. STAPLETON: Okay.

MR. DAVIS: Bill Stapleton and Judge, Bob Davis here. Bill, on the wording, wouldn't it be more appropriate if the Judge was simply ordering all of the actions set forth in the proposed Consent, Fourth Amended Consent, not adopting a new consent?

THE COURT: Yeah, that might be -- that might be smart.

MR. STAPLETON: Correct.

MR. DAVIS: All right.

MR. STAPLETON: Correct.

THE COURT: For appellate review, I agree.

MR. DAVIS: I learned -- I learned -- everything

I know, Judge, from Bill Stapleton.

MR. STAPLETON: Now that part I --

THE COURT: All right, Mr. Caldwell, Mr. Negele, are you okay? I mean I know you object to this, but are you okay with at least in terms of form not substance?

MR. CALDWELL: Well, Your Honor, I -- for the order that gets entered should not, in our view, refer to a consent agreement --

THE COURT: Proposed. Proposed. Not consent. Proposed consent agreement.

MR. CALDWELL: As long as it's clear that we're not consenting to it.

THE COURT: I understand that.

MR. CALDWELL: All right.

THE COURT: And you can fight with the Court of Appeals. I understand that. But it'd be proposed. Okay?

MR. CALDWELL: Understood.

THE COURT: With all your table of lawyers there.

Mr. Negele?

MR. NEGELE: Yeah, I think we understand what you're saying, and, you know, we'll move forward as we need to.

THE COURT: Let me just thank everybody. I'm on the case, I'm going to stay with the case.

Commissioner? Commissioner, you've learned something here about peacemaking, and I know you came in the early ones, right?

MR. PRATT: That was --

THE COURT: I mean you were there in the room when we could all get in that courtroom and rub elbows, right?

MR. PRATT: As best as possible, yes.

THE COURT: I know. So Commissioner --

MR. PRATT: Are you open for comment or feedback as well?

THE COURT: Yeah, sure, absolutely, but I, Commissioner, I just, you know, I keep talking to you about an approach, and you were part of it today.

MR. PRATT: Yes, so the action-oriented approach is greatly appreciated. Despite having a fancy title of Water Resources Commissioner, my office has really had no standing in this case until, and I'm not sure we really have standing now, but we've not been directly impacted because my office doesn't have jurisdiction over groundwater.

As you've seen in some of the recent documents, however, because it's now, the dioxane is physically in some of the pipes that my office is responsible for and there's a federal rule about the owner of the pipe is responsible for getting the water that's contaminated out of there, or the contaminant more to the point, I'm in the awkward position of being, you know, yet another entity or person who's impacted by something that didn't previously have an impact. I'm now more involved than I previously was, not by my own choice, and I think that's a story that's come up over and over here, right, whether it's the Breezewood well that Mr. Rayle mentioned, or the Elizabeth Street wells previously, the impacts seem to keep on coming. And so the action-oriented, "Let's try to make more progress," is greatly approached in the context that everyone else has previously raised of course, the two or three showstoppers that had been brought out there by those other folks. So that's appreciated because the impacts continue to keep going.

And I'll just add one more thing, for folks who, it's great that somebody gets bottled water or gets a municipal water supply. When somebody gets annexed from a township to a city, they have a number of costs that are not accounted for and are not paid for by someone else. So when someone gets annexed into the city, one of the first things they have to do is hook up to city water and sewer. Even if those costs are covered, their annual cost of paying for those services are much greater than the cost that they used to have for electricity on a well.

So when we see the word "prevent" in prior Consent Judgments, I think that's the greatest concern that's out there, and quite frankly, you know, one of the reasons there's so many people here is that trust is not there the way it used to be I suppose, if it ever was there before.

I would just say one thing in the context of what's being discussed, these regular meetings, my observation, the one thing that's been missing that would be helpful from a trust standpoint is, whether it's an annual report or every couple of years, there's never a visual. There's never something that you can point to, to my mom -- could my mom understand, "Oh, that's why it's better this year; I see." We never have that visual. There's nothing the public gets to see. And quite honestly, even as someone who sees more of the information, maybe not every document in the legal process, it's difficult for me to look at any visuals and really be able to sort out in my own mind, "Well, this is something I could explain to somebody why things are better than they were." And I think that's the crux of it; just to restore that trust.

So, to switch from that, I want to assure you and Mr. McKee that absent COVID, the Mack basketball game has been active. It should fire up again this fall. To call me an active participant might be a stretch, but I do try to hustle back on defense anyway. So that game is still alive. I've got a senior exiting Huron and a freshman coming into Huron, so we're River Rats as well there. So I -- all a way of saying I care about this as a community member.

And last thing, back to the professional side, on the Sister Lakes, if you were to go to the west side of Wagner Road, there is a pipe that's the end of a county drain, and what you will see is as soon as the water gets across Wagner Road, it can't go anywhere. There's a big swamp that prevents that water from moving, so that's yet another reason why maybe it's not the best idea to be pumping extra water into First Sister Lake. I don't know if that's really in the pleadings or not, but I've taken photographs and shared them with counsel and such. But again, when it's raining it would be a bad idea to have water coming into that area because it actually comes to a hard stop on the other side of Wagner Road.

And with that, I appreciate the opportunity for the comments and your desire to see some form of action going forward. Let's have more progress while, as you say, there's ongoing litigation about are these the right technical details. Thank you very much.

THE COURT: Thank you, Evan.

And I think Commissioner Shink, I think Ms. City Councilperson Griswold has her hand up. So we'll go there next, okay?

MS. GRISWOLD: Thank you. I appreciate the forward movement. I think that we need to very carefully control the message to the public. We have to realize that the public was almost unanimously against the proposed Fourth CJ, and so if we simply announce today that we're going to have a court order for the proposed Fourth CJ, you know, people are going to be out in the streets regardless of COVID. So I would just say let's -- let's make sure it's carefully communicated.

THE COURT: That's a very good point. And I've got Kevin in my own neighborhood probably doing a stuff -- you know, I've got to walk around my neighborhood, and my neighborhood is telling me, trying to, what to do. I get that. That's why I'm trying to be careful to say it's one step.

MS. GRISWOLD: Yes.

THE COURT: It's one step with ongoing review. But at least in the order it's like we're going to, that's the first step, and I'm going to stay on the case.

MS. GRISWOLD: Thank you.

MR. DAVIS: Can the order be --

THE COURT: I hope you all --

MR. DAVIS: So can the order be --

THE COURT: I hope you all keep it up.

MR. DAVIS: Can we title the order "Interim"?

THE COURT: Huh?

MR. DAVIS: Will the title of the order be "Interim order"?

MR. POSTEMA: Yes.

THE COURT: Probably a good idea.

MR. CALDWELL: Your Honor, we lose some -- I can't see who --

THE COURT: I --

MR. CALDWELL: Your Honor, from our position --

THE COURT: Okay, I can see him now.

MR. CALDWELL: -- I don't know what the interim nature of this order is.

THE COURT: No, that's --

MR. CALDWELL: It's an order --

THE COURT: No, Mr. Caldwell --

MR. CALDWELL: -- that is undertaken --

THE COURT: Yeah, Mr. Caldwell is -- I think we have to have it in the language, it's order to implement the proposed judgment with all the things we talked about, Mr. Stapleton --

MR. STAPLETON: Yes.

THE COURT: -- annual review, but I think it could be, maybe we should add some language saying it's an order with review waiting for -- we're wordsmithing here. But something about sensitive to appellate review.

MR. DAVIS: Well, it's not a final order, right, Judge?

MR. POSTEMA: Right.

MR. DAVIS: It’s not a final --

THE COURT: No, not -- what Mr. Caldwell is saying, we need to have a final order.

MR. CALDWELL: Yes, Your Honor.

THE COURT: Language in the final order to say this is, you know, this is the steps we're going to go forward, ongoing jurisdiction, on -- you know, still open to let's see how this works, and still keeping open the idea of arguments, you know, with, as we go forward. Because I understand what Mr. Caldwell is saying; he's got to have a final order --

MR. CALDWELL: Yes, Your Honor.

THE COURT: -- to deal with.

MR. CALDWELL: Yes. And we don’t want to -- as we sample them, if we have to implement activities, I mean, you know, there's nothing interim about that, but, yes, we do need --

THE COURT: Right.

MR. CALDWELL: -- for appellate --

THE COURT: Right, and part, Mr. Caldwell, I mean part of it, all attorneys can sign off on this, but I'm ordering that you start this right now.

MR. CALDWELL: I understand.

THE COURT: And so, so I think he's right. He needs a final order so that if he has to go up to the Court of Appeals and say, "This is," whatever, he's got that ability.

MR. CALDWELL: Thank you.

THE COURT: Are you with me, Bill?

MR. STAPLETON: Yes, I -- absolutely, Your Honor. We will include that language.

THE COURT: Okay.

Mr. Negele, are you okay? Not with the substance, just --

MR. NEGELE: Yes, I -- I am.

THE COURT: Okay. All right. My Board of Commissioners, what do you think?

MS. SHINK: Thank you, Your Honor, for caring what we think, and for working hard to try to find a resolution. I hope that this results in cleanup happening, because really at the end of the day, that's what we need, so I'm hopeful.

THE COURT: Thank you.

Kevin, are you okay? I gotta see you in the

neighborhood.

MR. LUND: Well --

THE COURT: No, you're not. Okay.

MR. LUND: I moved.

THE COURT: Okay.

MR. LUND: I moved from the neighborhood a few years ago --

THE COURT: Oh, okay.

MR. LUND: -- when my daughter graduated from school. But yeah the -- talking to some of my -- my management, I think we're in agreement with every -- with getting started with something and moving forward. If we had started something in 2016 we'd be further along than we are today.

THE COURT: I know.

MR. LUND: We'd have more information to make better decisions. So, yeah. Getting started is great.

THE COURT: All right, Roger? Are you okay with it right now?

MR. RAYLE: I'd rather see -- I'd rather see the parts of the Fourth Consent Judgment that were agreed to be implemented.

THE COURT: I'm sure we can do that.

MR. RAYLE: Because it's -- because pulling back on the parts that are a disagreement is going to be harder than adding them later.

THE COURT: Yeah, I think -- I think that, if we could, if you could just send Roger whatever that is, that would be helpful.

MR. RAYLE: And the public really needs to have more chance to review this. I mean this, this current hearing, the documents were released last Friday, and I think I got 'em maybe Saturday, or maybe it was even yesterday, and I didn't have time to read them all even.

THE COURT: I know. Same for me.

MR. RAYLE: It's similar to what happened back in August, and it's --

THE COURT: Same for me.

MR. DAVIS: Well, Roger, the Court --

MR. RAYLE: So if we're going to have --

THE COURT: I've been reading all weekend, so.

MR. DAVIS: Roger, the Court of Appeals didn't rule --

MR. RAYLE: Yeah, but you can't possibly, you can't possibly absorb it all in that short amount of time to make an important decision like this. So if I were, if I were doing this, I would say let's take the parts of the Fourth Consent Judgment that were agreed to --

THE COURT: That's what we did.

MR. RAYLE: -- and then submit those --

THE COURT: That's what we did.

MR. RAYLE: Well, I -- but I'm not sure that that's spelled out. It's very important that that gets spelled out in detail because there's certain things like, as was said, there's poison pills in there that we cannot have in the order because that, the company will take that and run with it, and we'll be left stuck with that poison pill for the rest of our lives, and our children's and grandchildren's lives. So we have to make the right decision now. And the parts that are agreed to, why not just implement those in the order and take on the rest of that in the quarterly follow ups?

THE COURT: That's what we're doing, sir. That's exact what I ruled.

MR. RAYLE: Okay, I appreciate that.

THE COURT: Okay. All right.

Anybody else? Be safe. Stay safe.

Good to see you again, Jack. I'm talking to Jack Eaton. Good to see you again.

All right, stay safe everybody. And -- oh, Ms. Ostrowski, we should probably set up the quarterly review. It's now May. Why don’t we start with -- why don’t we start like June 1st or something? Would that work?

MR. CALDWELL: That's a month, Your Honor.

THE COURT: Yeah, I know. All right, you want me to go all the way to -- I want to do it like on the calendar year.

UNIDENTIFIED SPEAKER: September 1st.

THE COURT: Would that be good?

MR. CALDWELL: Put it in September, so it'd be on a three month --

THE COURT: Right. What about September 1st? What day is that, Ms. Ostrowski?

UNIDENTIFIED SPEAKER: Wednesday.

THE COURT: Let's do it 9:00 a.m.

THE CLERK: That's a Wednesday.

THE COURT: Yeah. Let's just do it, and then December 1st. Just do it on the calendar.

THE CLERK: Okay.

MR. POSTEMA: So June, September, and December, Judge? Is that it?

THE COURT: No. Mr. Caldwell said, you know, June is a month; he's right.

MR. POSTEMA: Okay.

THE COURT: So we'll do September and then quarterly after that.

MR. CALDWELL: That's the limit of my ability to count.

THE COURT: So Mr. Stapleton, just put that in the order, okay?

MR. STAPLETON: I will do that, Judge.

THE COURT: Quarterly reviews starting September 1st.

MR. STAPLETON: Yep. I will do that, and I will circulate a draft order. Hopefully we'll be able to present it to the Court.

THE COURT: Thank you.

All right, Michigan Daily, come back on. You have a responsibility here. You're talking to the public. have you listened to everything they've said?

MS. GOODING: I think so, yes.

THE COURT: All right. They're going to be looking to see if you report it out accurately, especially Roger.

MS. GOODING: I'll --

THE COURT: Okay?

MS. GOODING: -- do my best, yeah.

THE COURT: All right.

MR. POSTEMA: Yeah, Judge, we also have Mr. Stanton from the MLive here.

THE COURT: Well, Mr. Stanton, you were supposed to identify yourself and announce yourself. Do it.

MR. STANTON: Can you guys see me?

THE COURT: No.

MR. STANTON: Let's see.

THE COURT: Invisible media.

MR. STANTON: Oh, yeah. Sorry, I've got a thing over the --

THE COURT: Yeah, you're supposed to be representing the public. Have you been listening?

MR. STANTON: I have been listening, yes, I --

THE COURT: Have you heard what they had to say?

MR. STANTON: I've heard all sides and I will do what I always do and summarize all sides fairly and report what happened here today.

THE COURT: Well, you strive to do that.

MR. STANTON: That's what I strive to do, yeah.

THE COURT: That's the goal. Just like me as the Judge, trying to be fair.

MR. STANTON: This will -- I've tried to do that on this issue with probably 200 stories on this issue over the last decade or so, so this will just be --

THE COURT: Why don’t you tell, you know, tell everybody a little bit about yourself, because other people introduced themselves. Tell us why you went into the media. Tell us why you care about it. Tell us what's important.

MR. STANTON: Why I went into journalism?

THE COURT: Yeah.

MR. STANTON: Initially in high school I wanted to be a sports reporter, and then I started writing for my college paper and decided there were much more important issues to write about, so I got hooked on those and have been a government reporter for, you know, what, I think 17 years now. So yeah, I've been writing for the Ann Arbor News for well over a decade now and covering this issue at that time, and a lot more since 2016 when things started heating up and following it ever since and reporting at every twist and turn, and this is, like I said, probably just one of many more to come, and I'll probably be writing about this issue for as long as I'm with the Ann Arbor News, and whoever takes over my job after me will pick it up.

THE COURT: Well, come back any time. You're always welcome.

MR. STANTON: Thanks.

THE COURT: All right.

MS. ELIAS: Your Honor, can I add one thing?

THE COURT: Yes. Yes, Ms. Elias, it's great to see you again.

MS. ELIAS: Well, I retired two years ago, so who knows, but the, all of the pleadings that were filed on Friday, all the briefs, exhibits, and technical reports, have now been posted on the City's website under the Gelman Litigation Information page. So if the members of the public or anyone else who hasn’t read them all, it's like 1,500 pages, it's at a2gov.org, and you can just search for Gelman. Look for the litigation page and scroll down to briefings, and you can read them individually at your pleasure, et cetera, et cetera.

THE COURT: Good. That's helpful. Thank you, Ms. Elias. And it's good to see you again. I know you retired, but I'm really glad you're still back on the case.

All right, anything else? We're good?

(No verbal response).

THE COURT: Good luck. Stay safe.

MR. STAPLETON: Thank you, Your Honor.

MR. DUPES: Thank you, Your Honor.

MULTIPLE PARTICIPANTS: Thank you.

(At 1:00 p.m., proceedings concluded; off the record.)

STATE OF MICHIGAN
COUNTY OF WASHTENAW)ss.

I certify that this transcript is a complete, true, and correct transcript to the best of my ability of the Zoom videoconference hearing in the matter of ATTORNEY GENERAL FOR THE STATE OF MICHIGAN v. GELMAN SCIENCES, case number 88-34734-CE, held May 3, 2021.

Digital proceedings were recorded and provided to this transcriptionist by the court and this certified reporter accepts no responsibility for any events that occurred during the above proceedings, for any unintelligible, inaudible, and/or indiscernible response by any person or party involved in the proceeding or for the content of the digital media provided.

I also certify that I am not a relative or employee of the parties involved and have no financial interest in this case.

DATED: May 6, 2021

s/Kristen Shankleton

____

Transcription provided by:
Kristen Shankleton (CER6785)
Modern Court Reporting & Video, L.L.C.

From: William Stapleton <wstapleton@hooperhathaway.com>
Sent: Wednesday, May 5, 2021 8:56 AM
To: Michael Caldwell; Negele, Brian (AG)
Cc: Dindoffer, Frederick; Dupes, Nathan; Elias, Abigail; Postema, Stephen; Robert Davis; Erin Mette
Subject: Order
Attachments: ORDER TO IMPLEMENT PROPOSED FOURTH AMENDED AND RESTATED CONSENT JUDGMENT - Clean.DOCX

[EXTERNAL EMAIL]
Mike and Brian,

Proposed order attached for your review.

William J. Stapleton
Hooper Hathaway, P.C.
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Ann Arbor, MI 48104
(734) 662-4426
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HOOPER HATHAWAY
ATTORNEYS AT LAW

HOOPER HATHAWAY PRICE BEUCHE & WALLACE 2018
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STATE OF MICHIGAN

IN THE CIRCUIT COURT FOR THE COUNTY OF WASHTENAW

ATTORNEY GENERAL FOR THE STATE
OF MICHIGAN *ex rel.* MICHIGAN
DEPARTMENT OF NATURAL RESOURCES
AND ENVIRONMENT,

Plaintiffs,
-and-

Case No. 88-34734-CE
Hon. Timothy P. Connors

CITY OF ANN ARBOR; WASHTENAW COUNTY;
WASHTENAW COUNTY HEALTH DEPARTMENT;
WASHTENAW COUNTY HEALTH OFFICER
JIMENA LOVELUCK, in her official capacity;
HURON RIVER WATERSHED COUNCIL; and
SCIO TOWNSHIP,

Intervening Plaintiffs,

vs.

GELMAN SCIENCES, INC., a Michigan corporation,

Defendant.

_______________________________________/

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Robert Charles Davis (P41055) Attorney for Intervening Washtenaw County Entities DAVIS, BURKET, SAVAGE, LISTMAN, TAYLOR 10 S. Main Street, Suite 401 Mt Clemens, MI 48043 (586) 469-4300	Erin E. Mette (P83199) Attorneys for Intervenor Huron River Watershed Council GREAT LAKES ENVIRONMENTAL LAW CENTER 4444 2nd Avenue Detroit, MI 48201 (313) 782-3372

/

ORDER TO CONDUCT RESPONSE ACTIVITIES TO IMPLEMENT AND COMPLY WITH REVISED CLEANUP CRITERIA

This matter having come before the court for hearing on Response Activities necessary to implement and comply with revised cleanup criteria, all parties having filed briefs and technical reports, the court having heard argument of counsel and being otherwise fully advised in the premises;

IT IS HEREBY ORERED:

1. Gelman Sciences shall immediately implement and conduct all requirements and activities stated in the Proposed "Fourth Amended and Restated Consent Judgment" which is attached to this Order and incorporated by reference.

2. The court retains continuing jurisdiction and will hold further hearings on a quarterly basis to review the progress of Response Activities and other actions required by this order related to releases of 1,4-dioxane at and emanating from the Gelman site and consider the implementation of additional or modified Response Activities and other actions.

3. The first quarterly hearing is scheduled for September 1, 2021 at 9 a.m.

4. Intervening Plaintiffs shall retain their status as Intervenors in this action.

5. This is not a final order and does not close the case.

SO ORDERED.

Dated: ___________________ ______________________________________

Timothy P. Connors
Circuit Court Judge

From: Michael Caldwell <MCaldwell@zausmer.com>
Sent: Sunday, May 9, 2021 10:29 AM
To: Robert Davis; William Stapleton; 'Dindoffer, Frederick'; Dupes, Nathan; Erin Mette; Negele, Brian (AG); Elias, Abigail; Postema, Stephen
Subject: Revised Proposed Order
Attachments: Response Activity Order 5-7-21.docx; Response Activity Order 5-7-21 Redline.DOCX

Counsel, attached is Gelman's proposed order regarding the evidentiary hearing. The redline version shows the changes to Intervenors' version. Let me know what you think. Thanks.

Michael L. Caldwell
Shareholder
Zausmer
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32255 Northwestern Highway, Suite 225
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STATE OF MICHIGAN

IN THE CIRCUIT COURT FOR THE COUNTY OF WASHTENAW

ATTORNEY GENERAL FOR THE STATE
OF MICHIGAN *ex rel.* MICHIGAN
DEPARTMENT OF NATURAL RESOURCES
AND ENVIRONMENT,

Plaintiffs,
-and-

Case No. 88-34734-CE
Hon. Timothy P. Connors

CITY OF ANN ARBOR; WASHTENAW COUNTY;
WASHTENAW COUNTY HEALTH DEPARTMENT;
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Intervening Plaintiffs,

vs.

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Defendant.
_______________________________________/

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ORDER TO IMPLEMENT REVISED CLEANUP CRITERIA AND TO MODIFY RESPONSE ACTIVITY ORDERS AND JUDGMENTS

This matter having come before the court for an evidentiary hearing on the implementation of revised cleanup criteria and modification of response activity Orders and Judgments, all parties having filed briefs and expert reports, the court having heard statements of counsel and being otherwise advised in the premises;

IT IS HEREBY ORDERED:

1. Effective immediately, the Third Amended Consent Judgment is hereby modified and replaced by entry of this Order to Conduct Response Activities to Implement Revised Cleanup Criteria and to Modify Response Activity Orders and Judgments ("Response Activity Order").

2. Gelman Sciences shall conduct all requirements and activities stated in the attached "Fourth Amended and Restated Consent Judgment," which is fully incorporated by and made part of this Response Activity Order, subject to and consistent with the terms of that document.

Commented [CRL1]: @Intervenors – This title is consistent with the April 6 order setting the hearing:

"A hearing on implementation of revised cleanup criteria and modification of response activity
Orders and Judgments set for May 3, 4 and 5, 2021 at 9:00 AM."

Commented [CRL2]: @Intervenors – This is consistent with how the filings were described in the April 6 order setting the hearing.

3. The court will hold further hearings on a quarterly basis to review the progress of Response Activities and other actions required by this Response Activity Order and consider the implementation of additional or modified Response Activities and other actions.

4. The first quarterly hearing is scheduled for September 1, 2021 at 9 a.m.

5. Intervenors shall retain their status as Intervenors in this action.

6. This is a final, appealable order, but does not close the case and the court retains continuing jurisdiction over implementation of the Response Activity Order.

Commented [CRL3]: @Intervenors – The Court was explicit that this is a final, appealable order. *See* Hr'g Tr. 132:5–13:

THE COURT: And so, so I think he's right. He needs a final order so that if he has to go up to the Court of Appeals and say, "This is," whatever, he's got that ability.
MR. CALDWELL: Thank you.
THE COURT: Are you with me, Bill?
MR. STAPLETON: Yes, I -- absolutely, Your Honor. We will include that language.
THE COURT: Okay.

SO ORDERED.

Dated: ____________________

Timothy P. Connors
Circuit Court Judge

STATE OF MICHIGAN

IN THE CIRCUIT COURT FOR THE COUNTY OF WASHTENAW

ATTORNEY GENERAL FOR THE STATE
OF MICHIGAN *ex rel.* MICHIGAN
DEPARTMENT OF NATURAL RESOURCES
AND ENVIRONMENT,

Plaintiffs,

-and-

Case No. 88-34734-CE
Hon. Timothy P. Connors

CITY OF ANN ARBOR; WASHTENAW COUNTY;
WASHTENAW COUNTY HEALTH DEPARTMENT;
WASHTENAW COUNTY HEALTH OFFICER
JIMENA LOVELUCK, in her official capacity;
HURON RIVER WATERSHED COUNCIL; and
SCIO TOWNSHIP,

Intervening Plaintiffs,

vs.

GELMAN SCIENCES, INC., a Michigan corporation,

Defendant.

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ORDER TO ~~CONDUCT RESPONSE ACTIVITIES TO~~ IMPLEMENT ~~AND COMPLY WITH~~ REVISED CLEANUP CRITERIA AND TO MODIFY RESPONSE ACTIVITY ORDERS AND JUDGMENTS

Commented [CRL1]: @Intervenors – This title is consistent with the April 6 order setting the hearing:

"A hearing on implementation of revised cleanup criteria and modification of response activity
Orders and Judgments set for May 3, 4 and 5, 2021 at 9:00 AM."

This matter having come before the court for an evidentiary hearing on the implementation of revised cleanup criteria and modification of response activity Orders and Judgments~~Response Activities necessary to implement and comply with revised cleanup criteria~~, all parties having filed briefs and ~~technical~~ expert reports, the court having heard ~~argument~~ statements of counsel and being otherwise ~~fully~~ advised in the premises;

Commented [CRL2]: @Intervenors – This is consistent with how the filings were described in the April 6 order setting the hearing.

IT IS HEREBY ORDERED:

1. Effective immediately, the Third Amended Consent Judgment is hereby modified and replaced by entry of this Order to Conduct Response Activities to Implement Revised Cleanup Criteria and to Modify Response Activity Orders and Judgments ("Response Activity Order").

2. Gelman Sciences shall ~~immediately implement and~~ conduct all requirements and activities stated in the attached ~~Proposed~~ "Fourth Amended and Restated Consent Judgment,"

which is fully incorporated by and made part of this Response Activity Order, ~~which is attached to this Order and incorporated by reference~~subject to and consistent with the terms of that document.

3~~2~~. The court ~~retains continuing jurisdiction and~~ will hold further hearings on a quarterly basis to review the progress of Response Activities and other actions required by this ~~order~~ Response Activity Order ~~related to releases of 1,4 dioxane at and emanating from the Gelman site~~ and consider the implementation of additional or modified Response Activities and other actions.

4~~3~~. The first quarterly hearing is scheduled for September 1, 2021 at 9 a.m.

5~~4~~. Intervenors ~~ing Plaintiffs~~ shall retain their status as Intervenors in this action.

6~~5~~. This is ~~not~~ a final, appealable order ~~and~~ but does not close the case and the court retains continuing jurisdiction over implementation of the Response Activity Order.

Commented [CRL3]: @Intervenors – The Court was explicit that this is a final, appealable order. *See* Hr'g Tr. 132:5–13:

5 THE COURT: And so, so I think he's right. He
6 needs a final order so that if he has to go up to the
7 Court of Appeals and say, "This is," whatever, he's got
8 that ability.
9 MR. CALDWELL: Thank you.
10 THE COURT: Are you with me, Bill?
11 MR. STAPLETON: Yes, I -- absolutely, Your
12 Honor. We will include that language.
13 THE COURT: Okay.

SO ORDERED.

Dated: ____________________

____
Timothy P. Connors
Circuit Court Judge

STATE OF MICHIGAN

IN THE CIRCUIT COURT FOR THE COUNTY OF WASHTENAW

ATTORNEY GENERAL FOR THE STATE OF MICHIGAN *ex rel.* MICHIGAN DEPARTMENT OF NATURAL RESOURCES AND ENVIRONMENT,

Plaintiff,

and

THE CITY OF ANN ARBOR, WASHTENAW COUNTY, THE WASHTENAW COUNTY HEALTH DEPARTMENT, WASHTENAW COUNTY HEALTH OFFICER JIMENA LOVELUCK, THE HURON RIVER WATERSHED COUNCIL, AND SCIO TOWNSHIP,

Intervenors,

v

GELMAN SCIENCES, INC., a Michigan Corporation,

Defendant.

Washtenaw Circuit
Court N. 88-34734-CE

Hon. Timothy P. Connors

DEFENDANT GELMAN SCIENCES, INC.'S OBJECTIONS TO PROPOSED 7-DAY ORDER

/

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______________________________________/

DEFENDANT'S OBJECTIONS TO PROPOSED 7-DAY ORDER TO CONDUCT RESPONSE ACTIVITIES TO IMPLEMENT AND COMPLY WITH REVISED CLEANUP CRITERIA

Defendant Gelman Sciences, Inc. ("Gelman"), by and through its attorneys, ZAUSMER, P.C., hereby objects to the proposed Order to Conduct Response Activities to Implement and Comply with Revised Cleanup Criteria as submitted on May 10, 2021 ("Proposed Order") under MCR 2.602(B) for the following reasons:

1. The Proposed Order appears to only attach the Proposed "Fourth Amended and Restated Consent Judgment" ("4th Amended CJ") for purposes of identifying the "requirements and activities" Gelman is to implement and fails to unambiguously make the 4th Amended CJ itself an order/judgment of this Court. The Proposed Order therefore fails, among other things, to:

a. Adopt the more restrictive cleanup criteria set forth in the 4th Amended CJ;

b. Expand the Prohibition Zone (thus, among other things, rendering the detailed trigger/contingency planning process set forth in Sections V.A.4 and 5 meaningless) or adopt either the "clear and convincing" standard of proof applicable to requests for further expansion of the Prohibition Zone set forth in Section V.A.2.f or the Prohibition Zone Review process by which the Prohibition Zone may be contracted set forth in Section V.A.6; and

c. Adopt the coordinated and integrated terms and conditions set forth in the 4th Amended CJ with respect to each of the specific response activities Gelman is to implement and incorrectly suggests that Gelman can "immediately implement and conduct" the identified response activities without first, for example, obtaining access rights, submitting work plans to EGLE for approval, or obtaining a NPDES permit from the State for operation of the Parklake Well, etc.

2. The Proposed Order fails to address the status of the existing Third Amended Consent Judgment or describe the effect of the Proposed Order on the Third Amended Consent Judgment—i.e., whether the Proposed Order replaces and/or modifies the Third Amended Consent Judgment—so that it is clear which document controls the cleanup, which cleanup criteria apply, etc.

3. The Proposed Order improperly presupposes the outcome of the quarterly review hearings by suggesting that such hearings will involve "the implementation of additional or modified Response Activities and other actions."

4. The Proposed Order incorrectly states that "Intervening Plaintiffs" will maintain their status as Intervenors, suggesting that Intervenors have obtained the status of Intervening Plaintiffs. Having not filed their complaints, Intervenors are not Intervening Plaintiffs nor full parties to this enforcement action, but rather are simply Intervenors.

5. The Proposed Order inaccurately states that the Order is "not a final order" when in fact the Court made clear that the order to be issued was intended to be a "final, appealable" order subject to appellate review:

> The Court: And so, so I think he's right. He needs a final order so that if he has to go up to the Court of Appeals and say, "This is," whatever, he's got that ability.
>
> Mr. Caldwell: Thank you.
>
> The Court: Are you with me, Bill
>
> Mr. Stapleton: Yes, I – absolutely, Your Honor. We will include that language.
>
> The Court: Okay.

Evidentiary Hr'g Tr. 132:5-13. (**Exhibit A**). The Order should therefore state that it is "a final, appealable order, but does not close the case and the Court retains continuing jurisdiction over enforcement and implementation of" the Order.

6. The title of the Proposed Order is not consistent with the Court's April 6, 2021 Order Denying Motion for Reconsideration and Scheduling Hearing Dates, which the described the evidentiary hearing as a "hearing on implementation of revised cleanup criteria and modification of response activity Orders and Judgments." To be consistent, the order's title should therefore read: "Order to Implement Revised Cleanup Criteria and to Modify Response Activity Orders and Judgments".

7. Pursuant to MCR 2.602B(3)(c), Gelman submits the attached Order (**Exhibit B**), which addresses these deficiencies.

Dated: May 14, 2021

Respectfully submitted,

ZAUSMER, P.C.

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PROOF OF SERVICE

The undersigned certifies that a copy of the foregoing instrument was served upon all parties to the above cause to each of the attorneys of record herein at their respective addresses as directed on the pleadings on May 14, 2021 by:

☒ E-FILE ☐ US MAIL ☐ HAND DELIVERY ☐ UPS
☐ FEDERAL EXPRESS ☐ OTHER

EXHIBIT A

STATE OF MICHIGAN

IN THE 22nd CIRCUIT COURT (WASHTENAW COUNTY)

ATTORNEY GENERAL FOR THE STATE
OF MICHIGAN ex. rel. MICHIGAN
DEPARTMENT OF NATURAL RESOURCES
AND ENVIRONMENT,
Plaintiff,
And

Case No. 88-34734-CE

THE CITY OF ANN ARBOR,
Intervenor,
And

WASHTENAW COUNTY,
Intervenor,

And

WASHTENAW COUNTY HEALTH
DEPARTMENT,
Intervenor,

And

WASHTENAW COUNTY HEALTH OFFICER
JIMENA LOVELUCK,
Intervenor,
And

THE HURON RIVER WATERSHED COUNCIL,
Intervenor,
And

SCIO TOWNSHIP,
Intervenor,

V.

GELMAN SCIENCES, INC., a Michigan
Corporation,
Defendant.
____./

EVIDENTIARY HEARING HELD VIA ZOOM VIDEOCONFERENCE

BEFORE THE HONORABLE TIMOTHY P. CONNORS

Ann Arbor, Michigan - Monday, May 3, 2021

APPEARANCES:

FOR THE PLAINTIFF:

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(517) 373-7540

INTERVENING PLAINTIFFS:

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NATHAN D. DUPES (P75454)
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AND:

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ABIGAIL ELIAS (P34941)
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For Washtenaw County, Washtenaw County Health Department, and Washtenaw Health Officer:

ROBERT CHARLES DAVIS (P40155)
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(Appearances continued)

THE COURT: Right, and part, Mr. Caldwell, I mean part of it, all attorneys can sign off on this, but I'm ordering that you start this right now.

MR. CALDWELL: I understand.

THE COURT: And so, so I think he's right. He needs a final order so that if he has to go up to the Court of Appeals and say, "This is," whatever, he's got that ability.

MR. CALDWELL: Thank you.

THE COURT: Are you with me, Bill?

MR. STAPLETON: Yes, I -- absolutely, Your Honor. We will include that language.

THE COURT: Okay.

Mr. Negele, are you okay? Not with the substance, just --

MR. NEGELE: Yes, I -- I am.

THE COURT: Okay. All right. My Board of Commissioners, what do you think?

MS. SHINK: Thank you, Your Honor, for caring what we think, and for working hard to try to find a resolution. I hope that this results in cleanup happening, because really at the end of the day, that's what we need, so I'm hopeful.

THE COURT: Thank you.

Kevin, are you okay? I gotta see you in the

EXHIBIT B

STATE OF MICHIGAN

IN THE CIRCUIT COURT FOR THE COUNTY OF WASHTENAW

ATTORNEY GENERAL FOR THE STATE
OF MICHIGAN *ex rel.* MICHIGAN
DEPARTMENT OF NATURAL RESOURCES
AND ENVIRONMENT,

Plaintiffs,
-and-

Case No. 88-34734-CE
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vs.

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/

ORDER TO IMPLEMENT REVISED CLEANUP CRITERIA AND TO MODIFY RESPONSE ACTIVITY ORDERS AND JUDGMENTS

This matter having come before the Court for an evidentiary hearing on the implementation of revised cleanup criteria and modification of response activity Orders and Judgments, all parties having filed briefs and expert reports, the Court having heard statements of counsel and being otherwise advised in the premises;

IT IS HEREBY ORDERED:

1. Effective immediately, the Third Amended Consent Judgment is hereby modified and replaced by entry of this Order to Conduct Response Activities to Implement Revised Cleanup Criteria and to Modify Response Activity Orders and Judgments ("Response Activity Order").

2. The attached "Fourth Amended and Restated Consent Judgment" is fully incorporated into and made part of this Response Activity Order.

3. Gelman Sciences shall conduct all requirements and activities set forth in the attached "Fourth Amended and Restated Consent Judgment", subject to and consistent with the terms thereof.

4. The Court will hold further hearings on a quarterly basis to review the progress of Response Activities and other actions required by this Response Activity Order.

5. The first quarterly hearing is scheduled for September 1, 2021 at 9 a.m.

6. Intervenors shall retain their status as Intervenors in this action.

7. This is a final, appealable order, but does not close the case and the Court retains continuing jurisdiction over enforcement and implementation of this Response Activity Order.

SO ORDERED.

Dated: ____________________

Timothy P. Connors
Circuit Court Judge

STATE OF MICHIGAN
IN THE CIRCUIT COURT FOR THE COUNTY OF WASHTENAW

ATTORNEY GENERAL FOR THE
STATE OF MICHIGAN *ex rel.*
MICHIGAN DEPARTMENT OF
NATURAL RESOURCESAND
ENVIRONMENT,

Plaintiffs,
-and-

Case No. 88-34734-CE
Hon. Timothy P. Connors

CITY OF ANN ARBOR; WASHTENAW COUNTY;
WASHTENAW COUNTY HEALTH DEPARTMENT;
WASHTENAW COUNTY HEALTH OFFICER
JIMENA LOVELUCK, in her official
capacity; HURON RIVER WATERSHED
COUNCIL; andSCIO TOWNSHIP,

Intervening Plaintiffs,
vs.

GELMAN SCIENCES, INC., a Michigan

corporation,Defendant.
______________________________________/

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____/

INTERVENORS' RESPONSE TO DEFENDANT'S OBJECTIONS TO PROPOSED 7-DAY ORDER TO CONDUCT RESPONSE ACTIVITIES AND COMPLY WITH REVISED CLEANUP CRITERIA

The Intervenors respond to each of the numbered paragraphs in Gelman's Objections as follows:

1. The intent of Intervenors' proposed order was to make all of the provisions of the Proposed "Fourth Amended and Restated Consent Judgment" effective. By attaching that document to the proposed order and expressly incorporating it by reference, Intervenors' proposed order accomplishes that objective. Gelman's concern is misplaced.

2. Intervenors agree with Gelman that, for clarity, the Court's order should state that it replaces the Third Amended Consent Judgment. However, the Court's order does not "modify" the Third Amended Consent Judgment. It is an independent order entered after the Court's consideration of the legal briefs, technical reports, and presentations made at the evidentiary hearing. It represents the Court's finding of what is necessary and appropriate to implement the Court's existing orders and judgments and meet the objectives for the Gelman site in light of the change in cleanup criteria. The Court agreed at the hearing that it "was simply ordering all of the actions set forth in the proposed Consent, Fourth Amended Consent [Judgment], not adopting a new consent [judgment]." *See* Ex. A, Transcript, 124:14-24.

3. Gelman mischaracterizes and overstates how the quarterly process is described in the Intervenors' proposed order. The proposed order states that the Court will "consider the implementation of additional or modified Response Activities and other actions" (emphasis added). The Intervenors' proposed order does not presume that the Court will in fact order additional site activities at any quarterly hearing. Gelman apparently recognized this simple fact when it circulated a proposed order to Intervenors on May 9 containing the very language to which Gelman now objects. *See* Ex. B, May 9 Proposed Order.

4. The Intervenors do not object to replacing the term "Intervening Plaintiffs" with "Intervenors," although they do not believe that this is a material difference as Gelman suggests.

5. Gelman's position on the finality of the order is contrary to Michigan law. MCR 2.604(A) provides:

> an order or other form of decision adjudicating fewer than all the claims, or the rights and liabilities of fewer than all the parties, does not terminate the action as to any of the claims or parties, and the order is subject to revision before entry of final judgment adjudicating all the claims and the rights and liabilities of all the parties. Such an order or other form of decision is not appealable as of right before entry of final judgment. A party may file an application for leave to appeal from such an order.

The only exception is in "receivership and similar actions, the court may direct that an order entered before adjudication of all of the claims and rights and liabilities of all the parties constitutes a final order on an express determination that there is no just reason for delay." MCR 2.604(B). Similarly, MCL 7.202(6) of the appellate rules provides in relevant part that a "final order" is "the first judgment or order that disposes of all the claims and adjudicates the rights and liabilities of all the parties, including such an order entered after reversal of an earlier final judgment or order." Gelman concedes at paragraph 7 of its proposed order that the Court's order will not close the case because it will not adjudicate all of the rights and liabilities of all the parties to the case. It is therefore erroneous to call the order a "final, appealable order," as Gelman proposes. If Gelman wants to appeal the order, it must follow MCR 7.205 and apply for leave, as required for any interlocutory order.

The statements that the Court made at the hearing which Gelman cites do not change (nor could they change) this conclusion. When the Court was referring to a "final" order, it presumably was making clear that its decision was final on the particular issues before the Court such that Gelman could apply for leave to appeal from that decision. Intervenors do not believe that the Court meant that it was purporting to certify its decision for appeal. Unlike federal district courts, Michigan trial courts do not have the power to certify orders for appeal. *Cf.* 28 U.S.C. 1292(b); MCR 7.203(B). Gelman's suggestion to the contrary is erroneous.

6. The title of Intervenors' proposed order accurately describes what the order does—it requires Gelman to conduct response activities to implement and comply with revised cleanup criteria. Consistency with the order scheduling the hearing is irrelevant. It is unclear why Gelman is taking issue with something as non-controversial as the title of the proposed order, other than as a ploy to try to improve Gelman's chances on appeal.

7. Gelman's proposed order does not reflect the Court's ruling. The Intervenors' proposed order should be entered, with the minor modifications described above.

ANN ARBOR CITY ATTORNEY'S OFFICE

Dated: May 24, 2021

BY: /s/Stephen K. Postema
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Attorneys for Intervenor City of Ann Arbor

BODMAN PLC

Dated: May 24, 2021 BY: /s/Nathan D. Dupes
Nathan D. Dupes (P75454)
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DAVIS, BURKET, SAVAGE, LISTMAN

Dated: May 24, 2021 BY: /s/RobertCharles Davis
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Dated: May 24, 2021 BY: /s/Erin E. Mette
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HOOPER HATHAWAY, P.C.

Dated: May 24, 2021 BY: /s/William J. Stapleton
William J. Stapleton (P38339)
Attorneys for Intervenor Scio Township

EXHIBIT A

STATE OF MICHIGAN

IN THE 22nd CIRCUIT COURT (WASHTENAW COUNTY)

ATTORNEY GENERAL FOR THE STATE
OF MICHIGAN ex. rel. MICHIGAN
DEPARTMENT OF NATURAL RESOURCES
AND ENVIRONMENT,
Plaintiff, Case No. 88-34734-CE
And

THE CITY OF ANN ARBOR,
Intervenor,
And

WASHTENAW COUNTY,
Intervenor,

And

WASHTENAW COUNTY HEALTH
DEPARTMENT,
Intervenor,

And

WASHTENAW COUNTY HEALTH OFFICER
JIMENA LOVELUCK,
Intervenor,
And

THE HURON RIVER WATERSHED COUNCIL,
Intervenor,
And

SCIO TOWNSHIP,
Intervenor,

V.

GELMAN SCIENCES, INC., a Michigan

Corporation,

Defendant.

____./

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EVIDENTIARY HEARING HELD VIA ZOOM VIDEOCONFERENCE

BEFORE THE HONORABLE TIMOTHY P. CONNORS

Ann Arbor, Michigan - Monday, May 3, 2021

APPEARANCES:
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Health Department, and Washtenaw Health Officer:
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(Appearances continued)

For Huron River Watershed Council:

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For Scio Township:

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issues come up, we could do it that way.

Mr. McKee, what do you think?

MR. MCKEE: I think --

THE COURT: Is that good?

MR. MCKEE: -- a quarterly review would be fine if you, if we don't get stuck with the provisions in the order that are poison pills.

THE COURT: Well, we're going to start with that, and then we're going to do a quarterly review. I'll listen. But I just want to make sure, it's not like a motion or a, you know, a formal thing; that I'm on it, okay?

And the Court of Appeals is going to do whatever they want to do. Counsel, are you okay with that?

MR. DAVIS: I'm good, Judge. Quarterly sounds reasonable to me. It gives time for things to evolve.

MR. CALDWELL: Your Honor, we'll review whatever the order provides with our client.

THE COURT: Okay. I understand.

MR. POSTEMA: Judge, you're talking about -- excuse me, Judge. You're talking about the proposed interim order that would get started that you would like to take, us to take to our clients, and we've said that we would all take to them, and adding this additional thing

about quarterly reviews to actually deal with issues come up. Is that correct?

THE COURT: That's correct. Instead of -- so first of all, I'm going to order Mr. Stapleton, I'm going to order that we put into effect right now the proposed Consent Judgment. I would still like to have quarterly review of where things stand, because I know things go up to the Court of Appeals and then I finally -- I mean, I heard on Thursday or Friday. So I'd like to just be able to let people weigh in, where it stands, so it's more than an annual review. I'm just proposing quarterly review.

MR. STAPLETON: And Your Honor, just for clarification, if the Fourth CJ were to be entered now, would the Intervenors retain Intervenor status so we could --

THE COURT: Absolutely.

MR. POSTEMA: Yes.

THE COURT: Absolutely.

MR. STAPLETON: Thank you.

THE COURT: And so the idea, I'm going to have quarterly review just to see where things are standing, instead of annual review. I want to know what's going on. And then the Court of Appeals, you know, all parties are free to appeal me, and the Court of Appeals is free to tell me what I'm doing is wrong.

MR. POSTEMA: And Judge, did you, you talked about doing an interim order on your own, not a consent order --

THE COURT: No, it's my order. That's right, it's my order.

MR. POSTEMA: And the Fourth CJ, the additional documents that they had talked about, the settlement and the other orders, those are not part of it because --

THE COURT: No.

MR. POSTEMA: -- the EPA is gone --

THE COURT: No.

MR. POSTEMA: -- it doesn't make any sense. Right.

THE COURT: No. I am absolutely just saying I'm ordering the proposed Consent Judgment, and then I want to say on it every quarter, and the Court of Appeals, you know, can decide whether that's appropriate or not. And then I'd like the Court of Appeals to weigh in frankly before I take any additional steps. Are you with me?

MR. STAPLETON: So Your Honor, and just so I'm clear --

THE COURT: Sure.

MR. STAPLETON: -- because it sounds like you would like me to draft something and send it out to

counsel.

THE COURT: Yes, I do.

MR. STAPLETON: So three components: entry of the proposed Fourth Amended CJ now.

THE COURT: Yes.

MR. STAPLETON: Quarterly review where the parties and the Intervenors review the progress at the site.

THE COURT: Yes.

MR. STAPLETON: And Intervenors retain their status as Intervenors.

THE COURT: Yes.

MR. STAPLETON: Okay.

MR. DAVIS: Bill Stapleton and Judge, Bob Davis here. Bill, on the wording, wouldn't it be more appropriate if the Judge was simply ordering all of the actions set forth in the proposed Consent, Fourth Amended Consent, not adopting a new consent?

THE COURT: Yeah, that might be -- that might be smart.

MR. STAPLETON: Correct.

MR. DAVIS: All right.

MR. STAPLETON: Correct.

THE COURT: For appellate review, I agree.

MR. DAVIS: I learned -- I learned --

EXHIBIT B

From:	Michael Caldwell <MCaldwell@zausmer.com>
Sent:	Sunday, May 9, 2021 10:29 AM
To:	Robert Davis; William Stapleton; Dindoffer, Fredrick; Dupes, Nathan; Erin Mette; Negele, Brian (AG); Elias, Abigail; Postema, Stephen
Subject:	Revised Proposed Order
Attachments:	Response Activity Order 5-7-21.docx; Response Activity Order 5-7-21 Redline.DOCX

Counsel, attached is Gelman's proposed order regarding the evidentiary hearing. The redline version shows the changes to Intervenors' version. Let me know what you think. Thanks.

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STATE OF MICHIGAN

IN THE CIRCUIT COURT FOR THE COUNTY OF WASHTENAW

ATTORNEY GENERAL FOR THE STATE
OF MICHIGAN *ex rel.* MICHIGAN
DEPARTMENT OF NATURAL RESOURCES
AND ENVIRONMENT,

Plaintiffs,
-and-

Case No. 88-34734-CE
Hon. Timothy P. Connors

CITY OF ANN ARBOR; WASHTENAW COUNTY;
WASHTENAW COUNTY HEALTH DEPARTMENT;
WASHTENAW COUNTY HEALTH OFFICER
JIMENA LOVELUCK, in her official capacity;
HURON RIVER WATERSHED COUNCIL; and
SCIO TOWNSHIP,

Intervening Plaintiffs,

vs.

GELMAN SCIENCES, INC., a Michigan corporation,

Defendant.

/

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ORDER TO IMPLEMENT REVISED CLEANUP CRITERIA AND TO MODIFY RESPONSE ACTIVITY ORDERS AND JUDGMENTS

This matter having come before the court for an evidentiary hearing on the implementation of revised cleanup criteria and modification of response activity Orders and Judgments, all parties having filed briefs and expert reports, the court having heard statements of counsel and being otherwise advised in the premises;

IT IS HEREBY ORDERED:

1. Effective immediately, the Third Amended Consent Judgment is hereby modified and replaced by entry of this Order to Conduct Response Activities to Implement Revised Cleanup Criteria and to Modify Response Activity Orders and Judgments ("Response Activity Order").

2. Gelman Sciences shall conduct all requirements and activities stated in the attached "Fourth Amended and Restated Consent Judgment," which is fully incorporated by and made part of this Response Activity Order, subject to and consistent with the terms of that document.

3. The court will hold further hearings on a quarterly basis to review the progress of Response Activities and other actions required by this Response Activity Order and consider the implementation of additional or modified Response Activities and other actions.

4. The first quarterly hearing is scheduled for September 1, 2021 at 9 a.m.

5. Intervenors shall retain their status as Intervenors in this action.

6. This is a final, appealable order, but does not close the case and the court retains continuing jurisdiction over implementation of the Response Activity Order.

SO ORDERED.

Dated: ____________________

____
Timothy P. Connors
Circuit Court Judge

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STATE OF MICHIGAN

IN THE CIRCUIT COURT FOR THE COUNTY OF WASHTENAW

ATTORNEY GENERAL FOR THE STATE
OF MICHIGAN *ex rel.* MICHIGAN
DEPARTMENT OF NATURAL RESOURCES
AND ENVIRONMENT,

Plaintiffs,
-and-

Case No. 88-34734-CE
Hon. Timothy P. Connors

CITY OF ANN ARBOR; WASHTENAW COUNTY;
WASHTENAW COUNTY HEALTH DEPARTMENT;
WASHTENAW COUNTY HEALTH OFFICER
JIMENA LOVELUCK, in her official capacity;
HURON RIVER WATERSHED COUNCIL; and
SCIO TOWNSHIP,

Intervening Plaintiffs,

vs.

GELMAN SCIENCES, INC., a Michigan corporation,

Defendant.

____/

Brian J. Negele (P41846) Attorney for Plaintiff MICHIGAN DEPARTMENT OF ATTORNEY GENERAL 525 W. Ottawa Street P.O. Box 30212 Lansing, MI 48909-7712 (517) 373-7540	Michael L. Caldwell (P40554) Attorney for Defendant ZAUSMER, P.C. 31700 Middlebelt Road, Suite 150 Farmington Hills, MI 48334 (248) 851-4111
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ORDER TO ~~CONDUCT RESPONSE ACTIVITIES TO~~ IMPLEMENT ~~AND COMPLY WITH~~ REVISED CLEANUP CRITERIA AND TO MODIFY RESPONSE ACTIVITY ORDERS AND JUDGMENTS

This matter having come before the court for an evidentiary hearing on the implementation of revised cleanup criteria and modification of response activity Orders and Judgments~~Response Activities necessary to implement and comply with revised cleanup criteria~~, all parties having filed briefs and ~~technical~~ expert reports, the court having heard ~~argument~~ statements of counsel and being otherwise ~~fully~~ advised in the premises;

IT IS HEREBY ORDERED:

1. Effective immediately, the Third Amended Consent Judgment is hereby modified and replaced by entry of this Order to Conduct Response Activities to Implement Revised Cleanup Criteria and to Modify Response Activity Orders and Judgments ("Response Activity Order").

2. Gelman Sciences shall ~~immediately implement and~~ conduct all requirements and activities stated in the attached ~~Proposed~~ "Fourth Amended and Restated Consent Judgment,"

which is fully incorporated by and made part of this Response Activity Order, ~~which is attached to this Order and incorporated by reference~~subject to and consistent with the terms of that document.

3~~2~~. The court ~~retains continuing jurisdiction and~~ will hold further hearings on a quarterly basis to review the progress of Response Activities and other actions required by this ~~order~~ Response Activity Order ~~related to releases of 1,4-dioxane at and emanating from the Gelman site~~ and consider the implementation of additional or modified Response Activities and other actions.

4~~3~~. The first quarterly hearing is scheduled for September 1, 2021 at 9 a.m.

5~~4~~. Intervenors ~~ing Plaintiffs~~ shall retain their status as Intervenors in this action.

6~~5~~. This is ~~not~~ a final, appealable order ~~and~~ but does not close the case and the court retains continuing jurisdiction over implementation of the Response Activity Order.

SO ORDERED.

Dated: ____________________

____
Timothy P. Connors
Circuit Court Judge

STATE OF MICHIGAN

IN THE CIRCUIT COURT FOR THE COUNTY OF WASHTENAW

ATTORNEY GENERAL FOR THE STATE OF MICHIGAN *ex rel.* MICHIGAN DEPARTMENT OF NATURAL RESOURCES AND ENVIRONMENT,

Plaintiff,

and

THE CITY OF ANN ARBOR, WASHTENAW COUNTY, THE WASHTENAW COUNTY HEALTH DEPARTMENT, WASHTENAW COUNTY HEALTH OFFICER JIMENA LOVELUCK, THE HURON RIVER WATERSHED COUNCIL, AND SCIO TOWNSHIP,

Intervenors,

v

GELMAN SCIENCES, INC., a Michigan Corporation,

Defendant.

Washtenaw Circuit
Court N. 88-34734-CE

Hon. Timothy P. Connors

DEFENDANT GELMAN SCIENCES, INC.'S SUPPLEMENTAL BRIEF IN SUPPORT OF ITS OBJECTIONS TO PROPOSED 7-DAY ORDER

/

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/

DEFENDANT GELMAN SCIENCES, INC.'S SUPPLEMENTAL BRIEF IN SUPPORT OF ITS OBJECTIONS TO PROPOSED 7-DAY ORDER

Defendant Gelman Sciences, Inc. ("Gelman"), having reviewed Intervenors' Response to Gelman's Objections to Intervenors' Proposed 7-Day Order, intends to refer to the two additional documents described below during tomorrow's hearing and, because the hearing will be held via Zoom, felt it would be helpful if these documents were circulated prior to the hearing:

1. The initial draft order Gelman provided to Intervenors' counsel in an attempt to reach a compromise on the order language in the hopes of avoiding burdening this Court further. **Exhibit A.** The compromise order which Gelman circulated to counsel included explanatory comments and references to the evidentiary hearing transcript justifying Gelman's proposed changes to the order that Intervenors chose to delete from the version they attached to their 5/24/21 Response as Exhibit B.

2. Additional pages of the evidentiary hearing transcript to give context to the Court's ruling that the order to be issued was intended to be a "final, appealable" order subject to appellate review:

> The Court: And so, so I think he's right. He needs a final order so that if he has to go up to the Court of Appeals and say, "This is," whatever, he's got that ability.
>
> Mr. Caldwell: Thank you.
>
> The Court: Are you with me, Bill
>
> Mr. Stapleton: Yes, I – absolutely, Your Honor. We will include that language.
>
> The Court: Okay.

Evidentiary Hr'g Tr. 132:5-13. (**Exhibit B**, Evidentiary Hr'g Tr. 130-132).

Respectfully submitted,

ZAUSMER, P.C.

/s/Michael L. Caldwell

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Dated: May 26, 2021

PROOF OF SERVICE

The undersigned certifies that a copy of the foregoing instrument was served upon all parties to the above cause to each of the attorneys of record herein at their respective addresses as directed on the pleadings on May 26, 2021 by:

☒ E-FILE ☐ US MAIL ☐ HAND DELIVERY ☐ UPS
☐ FEDERAL EXPRESS ☐ OTHER

/s/Brenda Ann Smith

EXHIBIT A

STATE OF MICHIGAN

IN THE CIRCUIT COURT FOR THE COUNTY OF WASHTENAW

ATTORNEY GENERAL FOR THE STATE
OF MICHIGAN *ex rel.* MICHIGAN
DEPARTMENT OF NATURAL RESOURCES
AND ENVIRONMENT,

Plaintiffs,

-and-

Case No. 88-34734-CE
Hon. Timothy P. Connors

CITY OF ANN ARBOR; WASHTENAW COUNTY;
WASHTENAW COUNTY HEALTH DEPARTMENT;
WASHTENAW COUNTY HEALTH OFFICER
JIMENA LOVELUCK, in her official capacity;
HURON RIVER WATERSHED COUNCIL; and
SCIO TOWNSHIP,

Intervening Plaintiffs,

vs.

GELMAN SCIENCES, INC., a Michigan corporation,

Defendant.

/

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ORDER TO ~~CONDUCT RESPONSE ACTIVITIES TO~~ IMPLEMENT ~~AND COMPLY WITH~~ REVISED CLEANUP CRITERIA AND TO MODIFY RESPONSE ACTIVITY ORDERS AND JUDGMENTS

Commented [CRL1]: @Intervenors – This title is consistent with the April 6 order setting the hearing:

"A hearing on implementation of revised cleanup criteria and modification of response activity
Orders and Judgments set for May 3, 4 and 5, 2021 at 9:00 AM."

This matter having come before the court for an evidentiary hearing on the implementation of revised cleanup criteria and modification of response activity Orders and Judgments~~Response Activities necessary to implement and comply with revised cleanup criteria~~, all parties having filed briefs and ~~technical~~ expert reports, the court having heard ~~argument~~ statements of counsel and being otherwise ~~fully~~ advised in the premises;

Commented [CRL2]: @Intervenors – This is consistent with how the filings were described in the April 6 order setting the hearing.

IT IS HEREBY ORDERED:

1. Effective immediately, the Third Amended Consent Judgment is hereby modified and replaced by entry of this Order to Conduct Response Activities to Implement Revised Cleanup Criteria and to Modify Response Activity Orders and Judgments ("Response Activity Order").

2. Gelman Sciences shall ~~immediately implement and~~ conduct all requirements and activities stated in the attached ~~Proposed~~ "Fourth Amended and Restated Consent Judgment,"

which is fully incorporated by and made part of this Response Activity Order, ~~which is attached to this Order and incorporated by reference~~subject to and consistent with the terms of that document.

3~~2~~. The court ~~retains continuing jurisdiction and~~ will hold further hearings on a quarterly basis to review the progress of Response Activities and other actions required by this ~~order~~ Response Activity Order ~~related to releases of 1,4-dioxane at and emanating from the Gelman site~~ and consider the implementation of additional or modified Response Activities and other actions.

4~~3~~. The first quarterly hearing is scheduled for September 1, 2021 at 9 a.m.

5~~4~~. Intervenors ~~ing Plaintiffs~~ shall retain their status as Intervenors in this action.

6~~5~~. This is ~~not~~ a final, appealable order ~~and~~ but does not close the case and the court retains continuing jurisdiction over implementation of the Response Activity Order.

Commented [CRL3]: @Intervenors – The Court was explicit that this is a final, appealable order. *See* Hr'g Tr. 132:5–13:

5 THE COURT: And so, so I think he's right. He
6 needs a final order so that if he has to go up to the
7 Court of Appeals and say, "This is," whatever, he's got
8 that ability.
9 MR. CALDWELL: Thank you.
10 THE COURT: Are you with me, Bill?
11 MR. STAPLETON: Yes, I -- absolutely, Your
12 Honor. We will include that language.
13 THE COURT: Okay.

SO ORDERED.

Dated: ____________________

Timothy P. Connors
Circuit Court Judge

EXHIBIT B

STATE OF MICHIGAN

IN THE 22nd CIRCUIT COURT (WASHTENAW COUNTY)

ATTORNEY GENERAL FOR THE STATE OF MICHIGAN ex. rel. MICHIGAN DEPARTMENT OF NATURAL RESOURCES AND ENVIRONMENT,
Plaintiff,

Case No. 88-34734-CE

And

THE CITY OF ANN ARBOR,
Intervenor,

And

WASHTENAW COUNTY,
Intervenor,

And

WASHTENAW COUNTY HEALTH DEPARTMENT,
Intervenor,

And

WASHTENAW COUNTY HEALTH OFFICER JIMENA LOVELUCK,
Intervenor,

And

THE HURON RIVER WATERSHED COUNCIL,
Intervenor,

And

SCIO TOWNSHIP,
Intervenor,

V.

GELMAN SCIENCES, INC., a Michigan Corporation,
Defendant.

____./

EVIDENTIARY HEARING HELD VIA ZOOM VIDEOCONFERENCE

BEFORE THE HONORABLE TIMOTHY P. CONNORS

Ann Arbor, Michigan - Monday, May 3, 2021

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MR. DAVIS: So can the order be --

THE COURT: I hope you all keep it up.

MR. DAVIS: Can we title the order "Interim"?

THE COURT: Huh?

MR. DAVIS: Will the title of the order be "Interim order"?

MR. POSTEMA: Yes.

THE COURT: Probably a good idea.

MR. CALDWELL: Your Honor, we lose some -- I can't see who --

THE COURT: I --

MR. CALDWELL: Your Honor, from our position --

THE COURT: Okay, I can see him now.

MR. CALDWELL: -- I don't know what the interim nature of this order is.

THE COURT: No, that's --

MR. CALDWELL: It's an order --

THE COURT: No, Mr. Caldwell --

MR. CALDWELL: -- that is undertaken --

THE COURT: Yeah, Mr. Caldwell is -- I think we have to have it in the language, it's order to implement the proposed judgment with all the things we talked about, Mr. Stapleton --

MR. STAPLETON: Yes.

THE COURT: -- annual review, but I think it could be, maybe we should add some language saying it's an order with review waiting for -- we're wordsmithing here. But something about sensitive to appellate review.

MR. DAVIS: Well, it's not a final order, right, Judge?

MR. POSTEMA: Right.

MR. DAVIS: It’s not a final --

THE COURT: No, not -- what Mr. Caldwell is saying, we need to have a final order.

MR. CALDWELL: Yes, Your Honor.

THE COURT: Language in the final order to say this is, you know, this is the steps we're going to go forward, ongoing jurisdiction, on -- you know, still open to let's see how this works, and still keeping open the idea of arguments, you know, with, as we go forward. Because I understand what Mr. Caldwell is saying; he's got to have a final order --

MR. CALDWELL: Yes, Your Honor.

THE COURT: -- to deal with.

MR. CALDWELL: Yes. And we don’t want to -- as we sample them, if we have to implement activities, I mean, you know, there's nothing interim about that, but, yes, we do need --

THE COURT: Right.

MR. CALDWELL: -- for appellate --

THE COURT: Right, and part, Mr. Caldwell, I mean part of it, all attorneys can sign off on this, but I'm ordering that you start this right now.

MR. CALDWELL: I understand.

THE COURT: And so, so I think he's right. He needs a final order so that if he has to go up to the Court of Appeals and say, "This is," whatever, he's got that ability.

MR. CALDWELL: Thank you.

THE COURT: Are you with me, Bill?

MR. STAPLETON: Yes, I -- absolutely, Your Honor. We will include that language.

THE COURT: Okay.

Mr. Negele, are you okay? Not with the substance, just --

MR. NEGELE: Yes, I -- I am.

THE COURT: Okay. All right. My Board of Commissioners, what do you think?

MS. SHINK: Thank you, Your Honor, for caring what we think, and for working hard to try to find a resolution. I hope that this results in cleanup happening, because really at the end of the day, that's what we need, so I'm hopeful.

THE COURT: Thank you.

Kevin, are you okay? I gotta see you in the

STATE OF MICHIGAN

IN THE 22nd CIRCUIT COURT (WASHTENAW COUNTY)

ATTORNEY GENERAL FOR THE STATE OF MICHIGAN ex. rel. MICHIGAN DEPARTMENT OF NATURAL RESOURCES AND ENVIRONMENT,
Plaintiff,
And

Case No. 88-34734-CE

THE CITY OF ANN ARBOR,
Intervenor,
And

WASHTENAW COUNTY,
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And

WASHTENAW COUNTY HEALTH DEPARTMENT,
Intervenor,

And

WASHTENAW COUNTY HEALTH OFFICER JIMENA LOVELUCK,
Intervenor,
And

THE HURON RIVER WATERSHED COUNCIL,
Intervenor,
And

SCIO TOWNSHIP,
Intervenor,

V.

GELMAN SCIENCES, INC., a Michigan Corporation,
Defendant.
____./

MOTION HEARING ON DEFENDANT'S OBJECTION TO SEVEN DAY ORDER

HELD VIA ZOOM VIDEOCONFERENCE

BEFORE THE HONORABLE TIMOTHY P. CONNORS

Ann Arbor, Michigan - Thursday, May 27, 2021

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TABLE OF CONTENTS

WITNESSES:	PAGE:
None.	

EXHIBITS:	PAGE:
None.	

Ann Arbor, Michigan

Thursday, May 27, 2021 - 9:02 a.m.

REFEREE SULLIVAN: Now on record, Frank J. Kelley versus Gelman Sciences, case number 88-34734-CE. This is Defendant's objections to the proposed seven day order.

THE COURT: Good morning. This is Judge Connors. Can we have appearances on the record, please?

MR. CALDWELL: Hello, Your Honor. Mike Caldwell on behalf of Gelman Sciences. With me I have Ray Ludwiszewski and Bruce Courtade.

MR. DAVIS: Your Honor, Robert Davis for the County Defendants.

MR. POSTEMA: Your Honor, Stephen Postema on behalf of the City of Ann Arbor. With me today are outside counsel Fred Dindoffer and Nathan Dupes.

MR. STAPLETON: Your Honor, William Stapleton for Scio Township.

MS. METTE: Good morning, Your Honor. Erin Mette on behalf of Huron River Watershed Council.

MR. NEGELE: Good morning, Your Honor. Assistant Attorney General Brian Negele representing the Michigan Department of Environment, Great Lakes, and Energy.

MR. CALDWELL: I believe that's everybody.

Your Honor, if I may proceed, I believe these are our objections to the Intervenors' proposed order regarding the evidentiary hearing; a few discrete issues for the Court's consideration.

I want to be clear at the outset that we tried to work out the language of this order with the Intervenors and repeatedly followed up with the Intervenor counsels' designated contact, but they simply didn't respond. I think it's clear that the intent is, of the Intervenors, is to take another bite at the apple following the hearing that was governed by a process that they wanted regardless of what the Court ordered on May 3rd.

We sent an annotated response to the Intervenors' proposed order which includes specific references to the hearing transcript that supported our proposed order. Intervenors chose to attach that offer of compromise to their response, which is bad enough, but they also deleted the comments, the explanatory comments and the references in the record that supported our proposals. And after receiving that initial response that I interpreted as positive that indicated we might be able to reach a settlement on this, we got barely the Intervenors, the larger group of Intervenors caucused and they decided to not respond to any of our suggested revisions, so we're unfortunately forced to bring this matter to the Court's attention.

The Intervenors', in my mind, clear intent in seeking another bite at the apple is, nowhere is that more clear than with respect to the issue of the finality of the order. The Court could not have been more clear that this order was to be a final order, not an interim order or an interlocutory order, but a final order specifically because that type of order was necessary for appellate purposes. The Intervenors' counsel promised that that language would be included in the order. That exchange is, was included in our annotated order that we sent the Intervenors, and it was also included in objections we provided to the Court. And yesterday, just so there was no question about the context of that discussion and the Court's unequivocal ruling, I provided the entire conversation that led up to that ruling.

Nevertheless, the Intervenors' order provides that the order is not final. That's simply not what the Court ordered. Again, we refer the Court to the transcripts we provided that note that the Intervenors haven't provided -- excuse me -- any transcript citation in support of their position. I would again believe that the Intervenors are simply playing games, trying to put as many hurdles in front in the way of our appellate rights as they can. And the thing is, Your Honor, I don't -- I think the Court was clear during the hearing that it was not interested in those types of games. I would agree with the transcript, and I believe the Court referred to the likely appellate review of its decision 17 times. At one point you said you were ordering the proposed Consent Judgment and with the quarterly reviews, and then you said, and I quote:

> "And then I'd like the Court of Appeals to weigh in frankly before I take any additional steps. Are you with me?"

To Intervenors' counsel.

As the Court recognized, it would be completely inappropriate for the Court to order Gelman to implement the Fourth Amended CJ and not make that order final for appellate purposes. Implementing the Fourth Amended CJ is a massive undertaking, and it impacts not just Gelman, but the community as a whole.

So I would ask the Court to stick to its guns. The Court ordered what it did. I'm sure you did that because you felt that that was the best thing for the community. So, you know, I would urge the Court to stand by its order and let the appellate chips fall where they may and not to engage in the kind of gamesmanship that the Intervenors are suggesting.

In terms of, I don't think there's any question that the order itself is final. It disposes of all claims and adjudicates the rights and liabilities of all the parties. And first of all, the only parties to this action are Gelman and the State, but certainly, this order certainly resolves our pending claims and rights. But even with respect to the Intervenors, they're not full parties because they haven't filed their Complaints. There are no pending claims. They have not been adjudicated. The Court ordered this evidentiary hearing process in lieu of filing their Complaints, and the Intervenors did not object, but rather enthusiastically supported that process. And the Court ordered the relief it did, and there are no other claims pending. So this order is in fact a final order.

Now, the, clearly the Court has inherent authority to enforce its orders, and we have quarterly reviews and status conferences that the Court has scheduled, first one for September 1st, and in our proposed order, although an inherent authority of the Court, our proposed order explicitly reserves that right of enforcement and continuing jurisdiction for the Court. But that doesn't mean that the order's not final and resolves all pending claims. It is, and it is final.

Now, Intervenors appear to argue that the fact that we put in our last sentence, as we're required to do by MCR 2.602, we stated that this order does not close the case, and they argue that that is somehow inconsistent with the order being final. In fact, as the Court is probably more aware than the rest of us, that provision is a docket management issue or provision that a group of, a Judge's Association suggested be inserted. Ironically I believe that originated from a Wayne County local court rule that I believe my former partner, Rick Kaufman, was the Chief Judge at the time, put in place. And it doesn't have, that docket management provision doesn't have anything to do with whether the order is final for appeal purposes, and the Intervenors conflating those two issues is simply misplaced. So I think that's it for the finality issue.

There are a few other issues that I think to some degree we agree on that we can quickly march through if I could. I think Intervenors' response indicates that they're in agreement with us that the order should reflect the, what the status of the Third Amended Consent Judgment is, and that their order was deficient because it didn't include that provision. Our paragraph number 1 clearly resolves this by stating that the Court's order would modify and replace the Third Amended CJ as the Fourth Amended CJ would have done if it were entered by the, by consent.

Now, I don't understand, the only objection they have it seems in this regard is that they don’t want to include the word "modify," but clearly entry of this order is going to modify and replace the previous Consent Judgment, so I frankly don't understand how that is not proper to conclude that.

And I would also point out they refer to other, you know, remedial orders. The stipulation that was entered in March 2011 with the, that led to the entry of the Third Amended Consent Judgment made it clear that the Third Amended Consent Judgment is the operable document; that the objectives of the previous remediation orders were either incorporated into the Third Amended Consent Judgment or eliminated to the extent that they were inconsistent. So the Third Amended Consent Judgment is the only operable document at this point.

The next issue relates to how clearly, frankly how clearly we do what the, I think everybody agrees, was the Judge's, the Court's intent. And it's -- they do, the Intervenors concede that they do intend for the Fourth Amended Consent Judgment to be an order of the Court and be incorporated into the Court's order. I would suggest that their, you know, incorporation by reference is not clear enough in that regard; that it should be using the language of our proposed order where we say that the proposed Fourth Amended CJ is fully incorporated and made part of this order. I think that language is necessary to make that issue clear.

And the other issue related to this paragraph is the Intervenors' order says we should implement the requirements of the Fourth Amended, set forth in the Fourth Amended CJ, but it fails to say that that work, that implementation is to be conducted subject to and consistent with the terms of the Fourth Amended CJ, which is necessary for a couple reasons. There are, the Fourth Amended CJ is an integrated document, and you can't just say implement, install a bunch of monitoring wells without the related, the terms and conditions set forth in the Fourth Amended CJ. For instance, we have to get access, we have to submit a work plan to -- work plans to EGLE. And without the provision that the work to be ordered by the Court is subject to and consistent with those terms that are in the Fourth Amended CJ, you really kind of usurp EGLE's role as the regulator of this work.

You know, I think, at least I hope I have some credibility in this regard because unlike the other counsel involved in this matter, I have very extensive experience with this site in putting the pedal to metal, if you will. When we were ordered to attempt to restore, you know, clean up the site within five years, we had to really move, and we, you know, quadrupled our purge rate over the objection of some of the Intervenors. We installed, you know, multiple wells, but we just didn’t run off like, you know, my farm girl mother used to say, like a chicken with its head cut off. We had a plan. We had a plan, a five-year plan that was approved by the EGLE and approved by the Court. And that's what the Consent Judgment is. That's what the Fourth Amended CJ would be.

And so the work that we're doing has to be subject to and consistent with those terms for it to make any sense, and in order to, for EGLE to retain its role as the regulator in this matter. And I think that's important, and may be a subtle issue that the Intervenors didn’t -- weren't aware of.

The next objection I think Intervenors are on board with regarding their status as Intervenors rather than Intervening Plaintiffs, and so I think we can recommend that the Court use our paragraph 6 rather than their paragraph 4.

And then the only remaining issue relates to whether the order, the purpose of the quarterly meetings and the description of those quarterly meetings and whether the order should presuppose that the purpose of the quarterly meetings as the Intervenors suggest is to consider implementation of additional or modified response activities. Obviously the Court, and as recognized by our order, the Court has inherent authority to enforce its orders and Gelman's proposed order reflects that, and it's certainly possible that after being advised of the progress and any changes of circumstances that have occurred between each quarterly meeting, that there may need to -- that there may need to be further actions taken by the Court to enforce its response activity order. I don't think it's likely, but we may not be proceeding as fast as the Court would like. We may, you know, as provided in the Fourth Amended CJ, if data, you know, we get from the response activities we're required to do comes back and it suggests that there are additional response activities required, we'll have to take those.

But I don't think we should, and I mean, my goodness, as we know, there could be a new cleanup criteria imposed at some point in the future, and we'll all have to react to those change in circumstances. But I don't think the order should presuppose or assumes facts not in evidence and state that the purpose of the quarterly reviews is to consider additional response activities, and we certainly don’t think that it was the Court's intent to turn these quarterly meetings into quarterly evidentiary hearings like the one we just had.

I mean, that would be more expensive than simply litigating the Intervenors' claims. And perhaps more importantly and more to the point, that type of process would, again, usurp EGLE's role as the regulating agency, which I don't think this, was this Court's intent, or even the Intervenors' intent to do that.

But in a nutshell, Your Honor, those are our objections to the Intervenors' proposed order. I think our order reflects both the Court's rulings. It's clearly announced during the hearing, and the proper process to follow going forward. Thank you.

THE COURT: Thank you, Mr. Caldwell.

Mr. Negele, did you have a position on this?

MR. NEGELE: Yeah. I only have some, you know, very brief comments. You know, we're looking for, looking for certainty in the order, and appreciate the changes that the parties have made, and those, you know, they confirm EGLE's understanding of what you ordered during the May 3rd hearing, and most importantly two of those are the, you know, the order makes all provisions of proposed Fourth CJ effective, and also clarifying the status of the Third Amended CJ so we don't have, you know, two conflicting orders basically there.

Regarding the quarterly hearings though, however, we do have some issues, and we interpreted Your

Honor's discussion of them to be more along the lines of a traditional status conference, not as an avenue to open up the order and obtain additional or modified response activities. The order itself, the Fourth CJ, provides, you know, for, you know, that as a possibility, you know, under certain circumstances. And, you know, among other things with that we're concerned that, you know, this could bog down the actual on the ground work through attempts to basically relitigate the issues from the May 3rd hearing.

And I also want to point out, too, is that you should understand that implement, and I think Mr. Caldwell was kind of, address this, it's like implementation of these remedies doesn’t move very quickly necessarily. There are many time consuming steps that are, you know, involved, including, you know, getting access to rights of way for properties to install the extraction and monitoring wells, and also to, you know, install a pipeline to connect to the pipeline that brings contaminated groundwater back to the treatment system.

So it might be that for the first quarterly report there might not be much to report, you know, so at least, you know, that, you know, we should understand that. You know, these things move kind of slowly. There's a lot involved.

And that's really all I have. Thanks.

THE COURT: Thank you.

Mr. Stapleton, I think I had asked you to prepare the proposed order. Would you be the one responding?

MR. STAPLETON: Yes, Your Honor, you did, however Mr. Dupes is going to be responding on behalf of Intervenors.

THE COURT: Thank you.

Mr. Dupes.

MR. DUPES: Thank you, Your Honor.

First of all, I'm a little beside myself that we're, the Intervenors are being accused of gamesmanship by a party that fought us tooth and nail on a scheduling order. Something I've never experienced. I'm sure some of my senior colleagues on the phone have never experienced. So it's a little ridiculous to be now being accused of gamesmanship or a second bite at the apple. We've done everything we can in our power to try and work things out with Gelman over the past several years and, you know, they're a scorched earth, you know, litigation type of party, and I think we've all seen that. So we certainly object to that characterization.

And it's also pretty rich to hear that after Gelman files yet another unsanctioned brief last night, you know, before this hearing, as the Court knows, a party doesn't just get to willy-nilly file briefs without leave of the Court, and they don't bother to seek Your Honor's permission to file supplemental reply briefs; they just do it, okay? And so anyway, I just think, I hope Your Honor doesn't give that point that Mr. Caldwell made several times even the time of day, but I feel like I needed to address it.

The first --

THE COURT: Let me just say to both of you, I could care less about arguments like that. I'm just waiting for you to finish so we can get to the substance.

MR. DUPES: Okay.

THE COURT: You can call each other names --

MR. DUPES: Thank you, Your Honor.

THE COURT: You two can call each other names on the phone, on a Zoom, in front of me, I don't pay any attention to it. I just wait to get, wait for the two of you to get it out of your system and let's get to the substance of it, all right?

MR. DUPES: Yeah, I appreciate that, Your Honor.

THE COURT: So what about the final, what about this issue of the final order? Because it -- I do know that we need to have something that the Court of Appeals would accept, and I do want the Court of Appeals to weigh in. The last thing I want to do is go down a path only to have it reversed by the Court of Appeals and we're starting all over somewhere.

MR. DUPES: Your Honor, that is --

THE COURT: That -- it makes sense.

MR. DUPES: Yeah, so that is, as you point out, that's the main issue. And, you know, and I apologize for starting with the other issue, but we're on the record and I felt like I needed to respond, okay?

But on the finality issue, as we all know, that's a term of art, right? So this came out of the hearing because we had proposed whether the title of the order should be called "interim," and Mr. Caldwell responded, "Well, Judge, there's nothing interim about what we're being asked to do," and you agreed saying, "Yes," you know, "I'm asking you to immediately implement all these activities," right? So it wasn't a, you know, "Maybe I'll think about it decision." It was, "No, you are to immediately start implementing these activities." So that was our understanding of what Your Honor meant by final.

But in terms of what goes in the order, a final order is a term of art, and in the court rules the only time we use a final order is, again, in that, in the court rule that we cited, which is, which Gelman doesn't even respond to. It's 2.604, and it says a final order is only one that resolves all pending claims, and all the rights and liabilities of the parties. Okay. This order doesn't do that, okay? And Mr. Caldwell has gone to pains in other hearings to say that it's the rights and liabilities of the Intervenors haven't been determined. The Intervenors' claims and potential claims, Gelman's potential defenses of those claims haven't been determined; none of that was addressed at this hearing, right?

So the hearing was final I guess in the sense of it requires immediate action by Gelman and it resolved what the Court thought was appropriate to do to reflect the change in cleanup criteria, but for purposes of the court rules it’s not a final order. And the reason -- so the question is, well, does that mean that the Court of Appeals can't look at it? Well, of course not. All it means is that Gelman has to apply for leave just like they would for any other interlocutory order, right? And if the Court of Appeals thinks, "Yeah, we need to take a look at this," then they'll take the appeal.

So it's a difference between a direct appeal, so if we had gone through a full trial, everybody's rights and liabilities were determined, there's a final judgment and order, case closed, Gelman takes a direct appeal, versus they just have to file the interlocutory route, just like they did, by the way, years ago when Your Honor let us into the case as Intervenors, right? There were no claims filed by the Intervenors at that time, there were no pending claims by the State of Michigan, and then Gelman applied for leave to the Michigan Court of Appeals and then applied for leave to the Supreme Court.

So this order being an interlocutory order, which is exactly what it is, does not prevent Gelman from applying for leave to the Michigan Court of Appeals, which is presumably what they're planning on doing. All this is, all that Mr. Caldwell wants to do is to try and avoid that process and somehow convince the Court of Appeals that this is a direct appeal after all the rights and liabilities have been determined, which of course this order did not do.

So this, it's kind of, Mr. Caldwell is misusing this term finality to try and create a new basis for a direct appeal which aren't, which simply isn't in the Michigan Court Rules. So the federal court process, which is maybe what he's trying to import in this case, will occasion -- will allow a district court to certify an interlocutory order for appeal. Okay, there is no such process in the Michigan Court Rules. In the Michigan Court Rules you either have a directly appealable final order that resolves everything in the case, in which case you just file a notice of appeal. Or, all other orders that aren't such an order are interlocutory and you apply for leave, and then it's up to the Court of Appeals to decide whether it wants the case. So that's what we're talking about. We're not talking about barring the doors to the Court of Appeals to Gelman. We're just saying you need to follow the proper process, just like you did appealing the order letting us into the case as Intervenors. So that's that --

THE COURT: I --

MR. DUPES: -- Your Honor. It's not --

THE COURT: Right. I understand that. What about the status, their proposed paragraph 6 rather than your proposed paragraph 4 as the status of the Intervenors?

MR. DUPES: So, Your Honor, again I hope you have -- you appreciate, or Mr. Caldwell appreciates that we tried in our response to meet Gelman halfway to, again, avoid this back and forth. If we're called Intervenors versus Intervening Plaintiffs, I don't think we really care because in our mind there isn't really a material difference. Have we filed our Complaints? No, but we don’t -- all we need to do -- I went back and read one of the orders Your Honor entered last night that let in the

Intervenors to the case, all we need to do to file our Complaints is to provide notice to Gelman and then we can file our Complaints. So there is no, you know, we don’t need to attain further leave to file claims, you know, so I -- if we don’t, if Gelman doesn't want us to be called Intervening Plaintiffs, and we use the term Intervenors for ourselves frequently, then so be it, you know. So we don’t object to calling ourselves Intervenors.

And then, Your Honor, moving on, which I think was the other point of contention is, and Mr. Negele also addressed this, was the words "Consider the implementation of additional or modified response activities and other actions," right? That's the characterizing the nature of the quarterly reviews. So again, when we first sent a order to Gelman and they responded, they kept in this language. So I'm not sure if they, they all of a sudden had a change of heart, they wanted one more thing to fight about, but they, Gelman had this very sentence, this very phrase, "Consider the implementation of additional response activities" in their own order. So I'm not exactly sure what the dispute is now.

But they're also just misreading that provision. Okay, it says, "Consider the implementation." That doesn't presuppose anything. Your Honor doesn't -- Your Honor isn't saying in this language that you're going to order anything at those further quarterly reviews. But, we think it's fair to say that this is a possible topic for those reviews, and that was something that was discussed at the hearing. Your Honor made clear that you wanted Gelman to immediately implement the activities in the Fourth, proposed Fourth CJ because you didn't want further delay, but you also recognized I think that from the Intervenors' perspective we were looking for more to be done. We have some particular concerns about what was in the proposed Fourth CJ, and we thought that one of the purposes of having those quarterly reviews was not only to just check-in status as Mr. Negele suggested, but if things aren't working, we should be able to discuss those with the Court and the Court's perfectly capable of managing that process.

So I'll just give you one example. One of the issues is the Park Lake well, right? Under the proposed Fourth CJ, Gelman is to apply for a permit from the State of Michigan to be able to, you know, put in an extraction well and discharge water to First Sister Lake, okay? The way the proposed Fourth CJ is worded is if Gelman is not granted that permit, it doesn't have to install the extraction well, okay, and our position as Intervenors is, well that's not really -- we all recognize that well is an important well, and so if it turns out down the road, and maybe it's not in the first quarterly review, maybe it's in the second or third, depending on EGLE's process, that there's issues with that permit, then I think we would come back to Your Honor and say, "Okay, Your Honor, that permit maybe isn't going to work. Let's talk about an alternative discharge solution for that well so that the meaningful contribution to, or mediation of the plume that that well would cause is still being implemented."

So it's that type of thing. It's not -- it's not re-opening the evidentiary hearing. It's the parties coming to you and saying, where are we, you know, how are things moving along, and I think it's also just to keep a check on the parties to make sure they're implementing your clear order at the hearing, which is this needs to happen right away, right? We don’t want to wait, you know, several months before Gelman applies for a permit for something, or if it's taking EGLE a certain period of time to review something, you know, then let's all talk about what the bottlenecks are and see if we can loosen those.

So, but in any event, getting back to our language, all it says is we're going to consider whether there be an implementation of an additional modified response activities. It does not presuppose any result. So that's the point.

And then as for -- sorry, I'm just -- bear with me, Your Honor. I'm pulling up Gelman's proposed order again making sure I'm addressing all these.

As for the status of the Third Amendment Consent Judgment, Your Honor, part of what's a little confusing about this is previously, there was the 1992 Consent Judgment, and then there were three amendments, okay? Every time it was amended there was no restatement of the prior provisions. So basically you had to look back at the First Consent Judgment and then look at what the first amendment did, and then what the second amendment did, and the third amended. So when the parties were putting together proposed Fourth, I think the idea was, okay, that's confusing; let's get rid of that. It's a proposed Fourth Amended and Restated, right? So basically all you need to do is look at the terms in the proposed Fourth. It's an all-inclusive document.

So in our mind, you know, we're fine with language, and maybe, you know, maybe the language is as simple as, you know, the provisions in the proposed Fourth Amended are, you know, adopted by this order, and we think the language above incorporated by reference is sufficiently clear. If the Court thinks that that's, there needs to be a little more clarity, we're fine with making that clear, but it's not a modification, okay, and

I think Your Honor was clear about that at the hearing. Your Honor is not modifying the Consent Judgment. You made clear at the hearing that this order you were entering was an independent order in reaction to what the parties had filed, reaction to the change in cleanup criteria, okay, and something that you felt was needed to be done right away to address these things, okay?

The reason we're referring to the proposed Fourth Amended CJ is really for convenience. It's like, instead of incorporating the pages and pages and pages of that document into this order, it's being attached and incorporated by reference. So I don’t, I think the issue of it being not clear is pretty overstated. I mean, it's going to be attached to the order, and the provisions are going to be effective. So, again, we're not exactly sure what Gelman's objection is there because we think it's clear.

And then, sorry, let me just look at my notes, Your Honor.

We talked about the Park Lake well.

Oh, there was comments made about usurping EGLE's role. I really don’t know where that's coming from. I mean, the intent is, the proposed Amended Fourth Consent Judgment by its terms, which, again, in our proposal would be incorporated by reference and attached to the order, reserves the regulatory oversight and authority of EGLE. And remember, EGLE at the hearing was advocating for adoption of that document, okay, so EGLE still retains its role to review work plans that Gelman submits, and to, you know, give the first, you know, give the review and order certain activities or order approvals of certain work plans, so all that's incorporated in the Fourth Consent Judgment. That's not going anywhere. So again I'm not sure what the concern is.

So again, Your Honor, I think, I believe that resolves the remaining issues that were, you know, between the parties, unless Your Honor has other questions for us, or obviously if any of the other Intervenor attorneys want to chime in, but I think that addresses the remaining disputes.

MR. CALDWELL: Your Honor --

THE COURT: I think that -- go ahead.

MR. CALDWELL: I'm happy to, if the Court has questions. I do have a few brief comments in response, but I can wait.

THE COURT: Well, I think -- I think that it is, while I would like to also urge the Court of Appeals to weigh in on this, it is not a final order. It is an interim order. And Mr. Dupes is right, I mean, I think that unfortunately for you, you have to ask for leave to appeal on it.

And frankly, I think Mr. Stapleton the order that you proposed captures the spirit and my intent. These are quarterly reviews. I don't know where they're, you know, where things are going to be. Certainly I'll consider what developments might have occurred. Certainly I'll consider what the Court of Appeals may have done. I have three-and-a-half years left on the bench, so a different Judge is going to be taking this over. So part of the reason I have the quarterly reviews is I want to make sure it's being addressed and looked at and that, you know, there's been some concern in the community that the Court hasn't been involved with it or there hasn't, you know, there hasn't been full transparency, and I want to make sure that isn't the way it is viewed from here.

So I'm comfortable, Mr. Stapleton, with the order that you proposed.

MR. STAPLETON: Thank you, Your Honor.

MR. POSTEMA: Thank you, Your Honor.

MR. CALDWELL: Your Honor, if I may briefly make a couple points for the record?

THE COURT: Sure.

MR. CALDWELL: Your Honor, with regard to the finality issue, this order does resolve all pending claims. I mean, the evidentiary hearing we had was in lieu of them, the Intervenors filing their Complaints. They have not been filed. And I don't know how, what circumstances -- and frankly perhaps for the benefit of the appellate review, I mean what, I'm going to ask the Court, what claims are out there that have not been resolved that are actually pending?

THE COURT: I always thought the Judge is the one that asked the lawyers --

MR. CALDWELL: And I don't mean to cross-examine the Court, but I --

THE COURT: I always thought that the lawyer had to defend their position, not the Judge.

I'll sign the interim order, Mr. Stapleton. I'll sign the order that you proposed, and I'll see you in September and get --

MR. STAPLETON: Thank you, Your Honor.

MR. POSTEMA: Thank you, Your Honor.

THE COURT: All right.

MR. DUPES: Thank you, Your Honor.

THE COURT: So Ms. Fire or Ms. Rolowski?

THE CLERK: I'm here.

THE COURT: Yeah. Make sure that that, the order that I'm talking, the one that's proposed, we get the signature on it, okay?

THE CLERK: Can you -- can they re-efile it?

Sorry.

THE COURT: Mr. Stapleton, could you re-efile it?

MR. STAPLETON: Yes.

MR. POSTEMA: Yes.

MR. STAPLETON: Yes, we will do that, Your Honor. Thank you.

THE COURT: All right.

MR. DAVIS: Your Honor, I think I actually filed that. Bill, I'll work with you and we'll get it re-filed today.

MR. STAPLETON: Okay, great.

THE COURT: Thank you.

MR. POSTEMA: Thank you, Judge.

THE COURT: Okay.

MR. NEGELE: Thank you, Your Honor.

(At 9:40 a.m., proceedings concluded; off the record.)

STATE OF MICHIGAN
COUNTY OF WASHTENAW)ss.

I certify that this transcript is a complete, true, and correct transcript to the best of my ability of the Zoom videoconference hearing in the matter of ATTORNEY GENERAL FOR THE STATE OF MICHIGAN v. GELMAN SCIENCES, case number 88-34734-CE, held May 27, 2021.

Digital proceedings were recorded and provided to this transcriptionist by the court and this certified reporter accepts no responsibility for any events that occurred during the above proceedings, for any unintelligible, inaudible, and/or indiscernible response by any person or party involved in the proceeding or for the content of the digital media provided.

I also certify that I am not a relative or employee of the parties involved and have no financial interest in this case.

DATED: June 1, 2021

s/Kristen Shankleton

____

Transcription provided by:
Kristen Shankleton (CER6785)
Modern Court Reporting & Video, L.L.C.

STATE OF MICHIGAN

IN THE CIRCUIT COURT FOR THE COUNTY OF WASHTENAW

ATTORNEY GENERAL FOR THE STATE
OF MICHIGAN *ex rel.* MICHIGAN
DEPARTMENT OF NATURAL RESOURCES
AND ENVIRONMENT,

Plaintiffs,
-and-

Case No. 88-34734-CE
Hon. Timothy P. Connors

CITY OF ANN ARBOR; WASHTENAW COUNTY;
WASHTENAW COUNTY HEALTH DEPARTMENT;
WASHTENAW COUNTY HEALTH OFFICER
JIMENA LOVELUCK, in her official capacity;
HURON RIVER WATERSHED COUNCIL; and
SCIO TOWNSHIP,

Intervening Plaintiffs,

vs.

GELMAN SCIENCES, INC., a Michigan corporation,

Defendant.
______________________________________/

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____/

ORDER TO CONDUCT RESPONSE ACTIVITIES TO IMPLEMENT AND COMPLY WITH REVISED CLEANUP CRITERIA

This matter having come before the court for hearing on Response Activities necessary to implement and comply with revised cleanup criteria, all parties having filed briefs and technical reports, the court having heard argument of counsel and being otherwise fully advised in the premises;

IT IS HEREBY ORDERED:

1. Gelman Sciences shall immediately implement and conduct all requirements and activities stated in the Proposed "Fourth Amended and Restated Consent Judgment" which is attached to this Order and incorporated by reference.

2. The court retains continuing jurisdiction and will hold further hearings on a quarterly basis to review the progress of Response Activities and other actions required by this order related to releases of 1,4 dioxane at and emanating from the Gelman site and consider the implementation of additional or modified Response Activities and other actions.

3. The first quarterly hearing is scheduled for September 1, 2021 at 9 a.m.

4. Intervening Plaintiffs shall retain their status as Intervenors in this action.

5. This is not a final order and does not close the case.

SO ORDERED.

Dated: 6/1/2021

/s/ Timothy Connors 6/1/2021

Timothy P. Connors
Circuit Court Judge

Drafted/Presented By:

By: /s/Robert Charles Davis
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Dated: May 27, 2021

STATE OF MICHIGAN

IN THE CIRCUIT COURT FOR THE COUNTY OF WASHTENAW

ATTORNEY GENERAL FOR THE STATE OF
MICHIGAN *ex rel.* MICHIGAN DEPARTMENT
OF ENVIRONMENT, GREAT LAKES, AND
ENERGY,

Plaintiffs,

-v-

GELMAN SCIENCES INC.,
a Michigan Corporation,

Defendant.

File No. 88-34734-CE
Honorable Timothy P. Connors

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FOURTH AMENDED AND RESTATED CONSENT JUDGMENT

The Parties enter this Fourth Amended and Restated Consent Judgment ("Consent Judgment" or "Fourth Amended Consent Judgment") in recognition of, and with the intention of, furtherance of the public interest by (1) addressing environmental concerns raised in Plaintiffs' Complaint; (2) expediting Remedial Action at the Site; and (3) avoiding further litigation concerning matters covered by this Consent Judgment. Among other things, the Parties enter this Consent Judgment to reflect EGLE's revision of the generic state-wide residential and non-residential generic drinking water cleanup criteria for 1,4-dioxane in groundwater to 7.2 micrograms per liter ("ug/L") and 350 ug/L, respectively, and of the generic groundwater-surface water interface cleanup criterion for 1,4-dioxane in groundwater to 280 ug/L. The Parties agree to be bound by the terms of this Consent Judgment and stipulate to its entry by the Court.

The Parties recognize that this Consent Judgment is a compromise of disputed claims. By entering into this Consent Judgment, Defendant does not admit any of the allegations of the Complaint, does not admit any fault or liability under any statutory or common law, and does not waive any rights, claims, or defenses with respect to any person, including the State of Michigan, its agencies, and employees, except as otherwise provided herein. By entering into this Consent Judgment, Plaintiffs do not admit the validity or factual basis of any of the defenses asserted by Defendant, do not admit the validity of any factual or legal determinations previously made by the Court in this matter, and do not waive any rights with respect to any person, including Defendant, except as otherwise provided herein. The Parties agree, and the Court by entering this Consent Judgment finds, that the terms and conditions of the Consent Judgment are reasonable, adequately resolve the environmental issues covered by the Consent Judgment, and properly protect the public interest.

NOW, THEREFORE, upon the consent of the Parties, by their attorneys, it is hereby ORDERED and ADJUDGED:

I. JURISDICTION

A. This Court has jurisdiction over the subject matter of this action. This Court also has personal jurisdiction over the Defendant.

B. This Court shall retain jurisdiction over the Parties and the subject matter of this action to enforce this Consent Judgment and to resolve disputes arising under the Consent Judgment.

II. PARTIES BOUND

This Consent Judgment applies to, is binding upon, and inures to the benefit of Plaintiffs, Defendant, and their successors and assigns.

III. DEFINITIONS

Whenever the terms listed below are used in this Consent Judgment or the Attachments that are appended hereto, the following definitions shall apply:

A. "Consent Judgment" or ""Fourth Amended Consent Judgment" shall mean this Fourth Amended and Restated Consent Judgment and all Attachments appended hereto. All Attachments to this Consent Judgment are incorporated herein and made enforceable parts of this Consent Judgment.

B. "Day" shall mean a calendar day unless expressly stated to be a working day. "Working Day" shall mean a day other than a Saturday, Sunday, or a State legal holiday. In computing any period of time under this Consent Judgment, where the last day would fall on a Saturday, Sunday, or State legal holiday, the period shall run until the end of the next working day.

C. "Defendant" shall mean Gelman Sciences Inc.

D. "1,4-dioxane" shall mean 1,4-dioxane released to or migrating from the Gelman Property. This term as it is used in this Consent Judgment shall not include any 1,4-dioxane that Defendant establishes by a preponderance of the evidence to have originated from a release for which Defendant is not legally responsible, except to the extent that such 1,4-dioxane is commingled with 1,4-dioxane released to or migrating from the Gelman Property. Nothing in this Consent Judgment shall preclude Defendant's right to seek contribution or cost recovery from other parties responsible for such commingled 1,4-dioxane.

E. “Eastern Area” shall mean the part of the Site that is located east of Wagner Road, including the areas encompassed by the Prohibition Zone.

F. “EGLE” shall mean the Michigan Department of Environment, Great Lakes, and Energy, the successor to the Michigan Department of Environmental Quality, the Michigan Department of Natural Resources and Environment, the Michigan Department of Natural Resources, and the Water Resources Commission. Pursuant to Executive Order 2019-06, effective April 22, 2019, the Michigan Department of Environmental Quality was renamed the Michigan Department of Environment, Great Lakes, and Energy.

G. “Evergreen Subdivision Area” shall mean the residential subdivision generally located north of I-94 and between Wagner and Maple Roads, bounded on the west by Rose Street, on the north by Dexter Road, and on the south and east by Valley Drive.

H. “Gelman” shall mean Gelman Sciences Inc.

I. “Gelman Property” shall mean the real property described in Attachment A, where Defendant formerly operated a manufacturing facility in Scio Township, Michigan. The Defendant sold portions of the property and retains one parcel only for purposes of operating a water treatment system (the “Wagner Road Treatment Facility”).

J. “Generic GSI Criterion” shall mean the generic groundwater-surface water interface (“GSI”) cleanup criterion for 1,4-dioxane of 280 ug/L established pursuant to MCL 324.20120e(1)(a).

K. “Groundwater Contamination” shall mean the 1,4-dioxane in the groundwater at a concentration in excess of 7.2 ug/L, as determined by the analytical method(s) described in Attachment B to this Consent Judgment, subject to review and approval by EGLE.

L. “Municipal Water Connection Contingency Plan” or “MWCCP” shall mean a contingency plan developed to identify the steps necessary to connect properties that rely on a private drinking water well to municipal water in the event those wells are threatened by 1,4-dioxane concentrations in excess of the applicable drinking water cleanup criterion and the estimated time necessary to implement each step of the water connection process.

M. "Part 201" shall mean Part 201 of the Natural Resources and Environmental Protection Act, MCL 324.20101, *et seq*.

N. "Parties" shall mean Plaintiffs and Defendant.

O. "Plaintiffs" shall mean the Attorney General of the State of Michigan *ex rel.* EGLE.

P. "Prohibition Zone" or "PZ" shall mean the area that is subject to the institutional control established by the Prohibition Zone Order and this Consent Judgment. A map depicting the Prohibition Zone established by this Fourth Amended Consent Judgment is attached as Attachment C.

Q. "Prohibition Zone Order" shall collectively mean the Court's Order Prohibiting Groundwater Use, dated May 17, 2005, which established a judicial institutional control, and the March 8, 2011 Stipulated Order Amending Previous Remediation Orders, which incorporated the Prohibition Zone Order into this Consent Judgment and applied the institutional control to the Expanded Prohibition Zone, as defined in the Third Amendment to Consent Judgment.

R. "PZ Boundary Wells" shall mean those wells on or near the boundary of the Prohibition Zone and designated in Section V.A.3.b herein, whose purpose is to detect movement of 1,4-dioxane near the Prohibition Zone boundary.

S. "Remedial Action" or "Remediation" shall mean removal, treatment, and proper disposal of Groundwater and Soil Contamination, land use or resource restrictions, and institutional controls, pursuant to the terms and conditions of this Consent Judgment and work plans approved by EGLE under this Consent Judgment.

T. “Response Activity” or “Response Activities” shall have the same meaning as that term is defined in Part 201, MCL 324.20101(vv).

U. “Sentinel Wells” shall mean those wells designated in Section V.A.3.a herein, whose purpose is to detect movement of 1,4-dioxane toward the Prohibition Zone boundary.

V. “Site” shall mean the Gelman Property and other areas affected by the migration of 1,4-dioxane emanating from the Gelman Property.

W. “Soil Contamination” or “Soil Contaminant” shall mean 1,4-dioxane in soil at a concentration in excess of 500 micrograms per kilogram (“ug/kg”), as determined by the analytical method(s) described in Attachment D or another higher concentration limit derived by means consistent with Mich Admin Code R 299.18 or MCL 324.20120a.

X. “Verification Process” shall mean the process through which Defendant shall test for and verify concentrations of 1,4-dioxane in excess of the applicable threshold at the relevant monitoring and drinking water wells, using the sampling and analytical method(s) described in Attachment B to this Consent Judgment. Specifically, Defendant shall sample the wells on a quarterly basis unless an alternative schedule is agreed upon with EGLE. Groundwater samples will be analyzed for 1,4-dioxane, either by Defendant’s laboratory or a third-party laboratory retained by Defendant. In the event that 1,4-dioxane concentrations in groundwater sampled from any well exceed the applicable threshold, Defendant shall notify EGLE by phone or electronic mail within 48 hours of completion of the data verification and validation specified in the Quality Assurance Project Plan (“QAPP”) described in Section V.E. Defendant will resample the same well within five days after the data verification and validation of the original result or at a time agreed upon with EGLE, if EGLE opts to take split samples. If a second sample analyzed by Defendant's laboratory or a third-party laboratory retained by Defendant has contaminant concentrations exceeding the applicable threshold, the exceedance will be considered verified and Defendant shall undertake the required Response Activities.

In the event that EGLE opts to take split samples, Defendant shall also collect an additional split sample for potential analysis within the applicable holding time by a mutually agreed-upon third-party laboratory at Defendant's expense. If the results from one sample, but not both, confirm a verified exceedance, the third sample analyzed by the mutually agreed-upon third-party laboratory, using the sampling and analytical method(s) described in Attachment B to this Consent Judgment, shall serve as the relevant result for verification purposes.

Y. "Western Area" shall mean that part of the Site located west of Wagner Road.

IV. IMPLEMENTATION OF REMEDIAL ACTION BY DEFENDANT

Defendant shall implement the Remedial Action to address Groundwater and Soil Contamination at, and emanating from, the Gelman Property in accordance with (1) the terms and conditions of this Consent Judgment; and (2) work plans approved by EGLE pursuant to this Consent Judgment. Notwithstanding any requirements set forth in this Consent Judgment obligating Defendant to operate remedial systems on a continuous basis, at a minimum rate, or until certain circumstances occur, Defendant may temporarily reduce or shut-down such remedial systems for reasonably necessary maintenance according to EGLE-approved operation and maintenance plans.

V. GROUNDWATER REMEDIATION

Defendant shall design, install, operate, and maintain the systems described below to satisfy the objectives described below. Defendant also shall implement a monitoring program to verify the effectiveness of these systems.

A. Eastern Area

1. Objectives. The remedial objectives of the Eastern Area ("Eastern Area Objectives") shall be the following:

a. Prohibition Zone Containment Objective. Defendant shall prevent Groundwater Contamination, regardless of the aquifer designation or the depth of the groundwater or Groundwater Contamination, from migrating beyond the boundaries of the Prohibition Zone as may be amended pursuant to Section V.A.2.f. Compliance with the Prohibition Zone Containment Objective shall be determined as provided in Section V.A.4.b, below.

b. Groundwater-Surface Water Interface Objective. Defendant shall prevent 1,4-dioxane from venting into surface waters in the Eastern Area at concentrations above the Generic GSI Cleanup Criterion, except in compliance with Part 201, including MCL 324.20120e ("Groundwater-Surface Water Interface Objective" for the Eastern Area).

2. Prohibition Zone Institutional Control. Pursuant to MCL 324.20121(8) and the Prohibition Zone Order, the following land and resource use restrictions shall apply to the Prohibition Zone depicted on the map attached hereto as Attachment C:

a. The installation by any person of a new water supply well in the Prohibition Zone for drinking, irrigation, commercial, or industrial use is prohibited.

b. The Washtenaw County Health Officer or any other entity authorized to issue well construction permits shall not issue a well construction permit for any well in the Prohibition Zone.

c. The consumption or use by any person of groundwater from the

Prohibition Zone is prohibited.

d. The prohibitions listed in Subsections V.A.2.a–c do not apply to the installation and use of:

i. Groundwater extraction and monitoring wells as part of Response Activities approved by EGLE or otherwise authorized under Parts 201 or 213 of the Natural Resources and Environmental Protection Act ("NREPA"), or other legal authority;

ii. Dewatering wells for lawful construction or maintenance activities, provided that appropriate measures are taken to prevent unacceptable human or environmental exposures to hazardous substances and comply with MCL 324.20107a;

iii. Wells supplying heat pump systems that either operate in a closed loop system or if not, are demonstrated to operate in a manner sufficient to prevent unacceptable human or environmental exposures to hazardous substances and comply with MCL 324.20107a;

iv. Emergency measures necessary to protect public health, safety, welfare or the environment;

v. Any existing water supply well that has been demonstrated, on a case-by-case basis and with the written approval of EGLE, to draw water from a formation that is not likely to become contaminated with 1,4-dioxane emanating from the Gelman Property. Such wells shall be monitored for 1,4-dioxane by Defendant at a frequency determined by EGLE; and

vi. The City of Ann Arbor's Northwest Supply Well, provided that the City of Ann Arbor operates the Northwest Supply Well in a manner that does not prevent its municipal water supply system from complying with all applicable state and federal laws and regulations.

e. Attachment E (consisting of the map depicting the Prohibition Zone and the above list of prohibitions/exceptions) shall be published and maintained in the same manner as a zoning ordinance at Defendant's sole expense, which may be accomplished by the City of Ann Arbor maintaining a hyperlink on its public webpage that includes the City of Ann Arbor zoning maps, or another appropriate webpage, that directs the visitor to the portion of EGLE's Gelman Sciences website that identifies the extent of the Prohibition Zone and the Summary of Restrictions. EGLE-approved legal notice of the Prohibition Zone expansion reflected in Attachment F shall be provided at Defendant's sole expense.

f. The Prohibition Zone Institutional Control shall remain in effect in this form until such time as it is modified through amendment of this Consent Judgment, with a minimum of 30 days' prior notice to all Parties. The Defendant or EGLE may move to amend this Consent Judgment to modify the boundaries of the Prohibition Zone to reflect material changes in the boundaries or fate and transport of the Groundwater Contamination as determined by future hydrogeological investigations or EGLE-approved monitoring of the fate and transport of the Groundwater Contamination. The dispute resolution procedures of Section XVI shall not apply to such motion. Rather, the Prohibition Zone boundary may not be expanded unless the moving Party demonstrates by clear and convincing evidence that there are compelling reasons that the proposed expansion is needed to prevent an unacceptable risk to human health. The above-described showing shall not apply to a motion if the Prohibition Zone expansion being sought arises from or is related to: (1) inclusion of the Triangle Property under the following subsection; (2) the incorporation of a more restrictive definition of Groundwater Contamination (i.e., a criterion less than 7.2 ug/L) into this Consent Judgment; or (3) expansion under V.A.6.c up to and including back to the boundary established by this Fourth Amended Consent Judgment.

g. Future Inclusion of Triangle Property in the Prohibition Zone. The triangular piece of property located along Dexter Road/M-14 ("Triangle Property"), depicted in Attachment C, will be included in the Prohibition Zone if the data obtained from monitoring wells MW-121s and MW-121d and other nearby wells, including any water supply well installed on the property, as validated by the Verification Process, indicate that the Groundwater Contamination has migrated to the Triangle Property.

h. Well Identification. To identify any wells newly included in the Prohibition Zone as a result of this modification or any future modification to the Prohibition Zone, pursuant to an EGLE-approved schedule, Defendant shall implement a well identification plan for the affected area that is consistent with the Expanded Prohibition Zone Well Identification Work Plan approved by EGLE on February 4, 2011.

i. Plugging of Private Water Wells. Defendant shall plug and replace any private drinking water wells identified in any areas newly included in the Prohibition Zone by connecting those properties to the municipal water supply. Unless otherwise approved by EGLE, Defendant shall also properly plug non-drinking water wells in any areas newly included in the Prohibition Zone.

j. Municipal Water Connection Contingency Plan ("MWCCP"). Defendant shall develop a MWCCP addressing the potential provision of municipal water to properties using private drinking water wells in the Calvin Street, Wagner Road, and Lakeview Avenue areas. The MWCCP will be developed according to a schedule to be approved by

EGLE.

3. Monitoring and Extraction Well Installation and Operation. Defendant shall install the following additional wells in the Eastern Area according to a schedule approved by EGLE and subject to access and receipt of any required approvals pursuant to Section VII.D:

a. Sentinel Well Installation. Defendant shall install the following three monitoring well clusters to monitor movement of 1,4-dioxane south of the northern Prohibition Zone boundary, in addition to MW-120, MW-123, and MW-129 that are already in place (collectively referred to herein as "Sentinel Wells"):

i. Residential area in the general vicinity of Ravenwood and Barber Avenues (Location "A" on map attached as Attachment G);
ii. Residential area in the general vicinity of Sequoia Parkway and Archwood Avenues between Delwood and Center (Location "B" on map attached as Attachment G); and
iii. Residential area in the general vicinity of Maple Road and North Circle Drive (Location "C" on the map attached as Attachment G).

b. PZ Boundary Well Installation. Defendant shall install the following two monitoring well clusters to monitor the movement of 1,4-dioxane near the PZ Boundary (collectively referred to herein as "PZ Boundary Wells"):

i. Residential, commercial, and vacant area east of South Wagner Road, north of West Liberty Road, west of Lakeview Avenue, and south of Second Sister Lake (Location "D" on map attached as Attachment G); and
ii. Residential area south/southeast of the MW-112 cluster (Location "E" on map attached as Attachment G).

c. Sentinel and PZ Boundary Well Installation and Sampling. Defendant shall install the new well clusters according to a schedule to be approved by EGLE. Each new Sentinel or PZ Boundary Well cluster will include two to three monitoring wells, and the determination of the number of wells shall be based on EGLE's and the Defendant's evaluation of the geologic conditions present at each location, consistent with past practice. The frequency of sampling these monitoring wells and the analytical methodology for sample analysis will be included in the Eastern Area System Monitoring Plan, as amended.

d. Drilling Techniques. Borings for new wells installed pursuant to Section V.A.3 shall be drilled to bedrock unless a different depth is approved by EGLE or if conditions make such installation impracticable. EGLE reserves the right to require alternate drilling techniques to reach bedrock if standard methods are not able to do so. If the Defendant believes that drilling one or more of these wells to bedrock is not practical due to the geologic conditions encountered and/or that such conditions do not warrant the alternative drilling technique required by EGLE, Defendant may initiate dispute resolution under Section XVI of this Consent Judgment. The wells shall be installed using Defendant's current vertical profiling techniques, which are designed to minimize the amount of water introduced during drilling, unless EGLE agrees to alternate techniques. Any material excavated as the result of well installation shall be properly characterized and disposed of or transferred to an appropriate facility for preservation and future scientific investigation, at Defendant's discretion.

e. Installation of Additional Groundwater Extraction Wells.

i. Defendant shall install an additional groundwater extraction well (the "Rose Well") and associated infrastructure in the general area bounded by Rose Street and Pinewood Street as designated on Attachment G or convert former injection well IW-2 to a groundwater extraction well, or both. The decision to install the Rose Well or to convert IW-2 to an extraction well (or to do both) and exact location of the Rose Well if installed will be based on an evaluation of relevant geologic conditions, water quality, and other relevant factors, including access.

ii. Subject to V.A.3.g., below, Defendant shall install an additional groundwater extraction well (the "Parklake Well") and associated infrastructure in the parcel owned by the City of Ann Arbor bounded by Parklake Avenue and Jackson Road as designated on Attachment G (the "City of Ann Arbor-owned parcel"). The exact location of the Parklake Well within the City of Ann Arbor-owned parcel will be based on an evaluation of relevant geologic conditions, water quality, and other relevant factors, including access. Terms of access to the City of Ann Arbor-owned parcel shall be governed by an access or license agreement between Defendant and the City of Ann Arbor and Defendant's obligation to install and operate the Parklake Well shall be conditioned on negotiation of a mutually acceptable agreement with the City of Ann Arbor.

f. Eastern Area Groundwater Extraction.

i. The Defendant shall operate the Evergreen Subdivision Area extraction wells, LB-4 and either the Rose Well or IW-2, or both (including EGLE-approved replacement well(s)) (collectively, the "Evergreen Wells"), and TW-19 and TW-23 (or EGLE-approved replacement well(s)) (the "Maple Road Wells"), at a combined minimum purge rate of approximately 200 gallons per minute ("gpm") or the maximum capacity of the existing deep transmission pipeline, whichever is less provided Defendant properly maintains the pipeline, in order to reduce the mass of 1,4-dioxane migrating through the Evergreen Subdivision Area and the mass of 1,4-dioxane migrating east of Maple Road, until such time as the Eastern Area Objectives will be met at a reduced extraction rate or without the need to operate these extraction wells. In the event the maximum capacity of the existing deep transmission pipeline is ever reduced to below 180 gpm, Defendant shall repair and/or reconfigure the pipeline and related infrastructure, or take other action, including potentially replacing the pipeline or treating and disposing of some portion of the extracted groundwater at a different location, as needed to once again achieve a capacity of 190 – 200 gpm. Defendant shall have the discretion to adjust the individual well purge rates in order to optimize mass removal and compliance with the Eastern Area Objectives, provided that it shall operate the Evergreen Wells at a combined minimum purge rate of approximately 100 gpm, until such time as the Eastern Area Objectives will be met at a reduced extraction rate without the need to operate these wells. Before significantly reducing extraction below the minimum purge rates described above or permanently terminating extraction from either the Evergreen Wells or the Maple Road Wells, Defendant shall consult with EGLE and provide a written analysis, together with the data that supports its conclusion that the Eastern Area Objectives can be met at a reduced extraction rate or without the need to operate these extraction wells. EGLE will review the analysis and data and provide a written response to Defendant within 56 days after receiving Defendant's written analysis and data. If Defendant disagrees with the EGLE's conclusion, Defendant may initiate dispute resolution under Section XVI of this Consent Judgment. The Defendant shall not significantly reduce or terminate extraction from the Evergreen Wells or the Maple Road Wells during the 56-day review period or while Defendant is disputing EGLE's conclusion.

ii. Defendant shall operate the Parklake Well, at a purge rate of approximately 200 gpm, subject to the yield of the aquifer in that area and discharge volume restrictions imposed in connection with the method of water disposal including discharge restrictions during wet weather events, in order to reduce the mass of 1,4-dioxane migrating from that area. Purged groundwater from the Parklake Well shall be treated with ozone/hydrogen peroxide or ultraviolet light and oxidizing agents at the City of Ann Arbor-owned parcel.

Defendant shall operate this extraction and treatment system until the 1,4-dioxane concentration in the groundwater extracted from the Parklake Well has been reduced below 500 ug/L. Once concentrations have been reduced below 500 ug/L, Defendant shall cycle the Parklake Well off and on for several periods of time approved by EGLE to demonstrate that significant concentration rebound is not occurring. Defendant shall not permanently terminate extraction and treatment of water from the Parklake Well before the second anniversary of the date extraction was commenced. Before significantly reducing or terminating extraction from the Parklake Well (beyond the discharge volume restrictions/variations arising from the approved discharge option/above-described cycling), Defendant shall consult with EGLE and provide a written analysis, together with the data that supports its conclusion that the foregoing conditions have been satisfied. EGLE will review the analysis and data and provide a written response to Defendant within 56 days after receiving Defendant's written analysis and data. If Defendant disagrees with EGLE's conclusion, Defendant may initiate dispute resolution under Section XVI of this Consent Judgment. The Defendant shall not significantly reduce or terminate extraction from the Parklake Well during the 56-day review period or while Defendant is disputing EGLE's conclusion.

g. Prerequisites for Parklake Well. Notwithstanding anything else in this Consent Judgment, Defendant shall not be obligated to install and operate the Parklake Well unless and until EGLE issues Defendant an NPDES permit with effluent limitations, discharge limits (other than volume) and other conditions no more restrictive than those included in Defendant's NPDES Permit No. MI-0048453 dated October 1, 2014 ("2014 NPDES Permit") that authorizes discharge of groundwater extracted by the Parklake Well to First Sister Lake following treatment with ozone/hydrogen peroxide technology .

4. Verification Monitoring. Defendant shall amend its Eastern Area System Monitoring Plan dated December 22, 2011 to include the monitoring wells installed under Section V.A.3 within 60 days of their installation. The Eastern Area System Monitoring Plan, as amended (hereinafter the "Verification Plan"), shall be sufficient to meet the objectives of this Section.

a. Objectives of Verification Plan. The Verification Plan shall include the collection of data sufficient to measure the effectiveness of the Remediation and to: (i) ensure that any potential migration of Groundwater Contamination outside of the Prohibition Zone is detected before such migration occurs and with sufficient time to allow Defendant to maintain compliance with the Prohibition Zone Containment Objective; (ii) verify that the Groundwater-Surface Water Interface Objective is satisfied; (iii) track the migration of the Groundwater Contamination to determine the need for additional investigation and monitoring points to meet the objectives in Section V.A.1, including the determination of the fate and transport of Groundwater Contamination when and if it reaches the Allen Creek Drain (including its branches) and the portion of the Huron River that is the easternmost extent of the Prohibition Zone; and (iv) evaluate potential changes in groundwater flow resulting from adjustments in extraction rates at different extraction well locations. The Verification Plan shall be continued until terminated pursuant to Section V.D.

b. Compliance Determination. The Verification Plan shall include the following steps for verifying sampling results and confirming compliance or noncompliance with the Eastern Area Objectives.

i. Verification Process for Sentinel Wells. Defendant shall conduct the Verification Process as defined in Section III.X for each Sentinel Well to verify any exceedance of 7.2 ug/L. A verified detection above 7.2 ug/L will be considered a "Verified Sentinel Well Exceedance" and Defendant shall take the Response Activities set forth in Section V.A.5.a.

ii. Verification Process for PZ Boundary Wells. Defendant shall conduct the Verification Process as defined in Section III.X for each PZ Boundary Well to verify any exceedance of 4.6 ug/L and/or 7.2 ug/L. A verified detection above 4.6 ug/L will be considered a "Verified PZ Boundary Well Exceedance" and Defendant shall take the Response Activities set forth in Section V.5.b. A verified detection above 7.2 ug/L will be considered a "Confirmed PZ Boundary Well Noncompliance" and Defendant shall take the Response Activities set forth in Section V.5.c.

5. <u>Eastern Area Response Activities</u>. Defendant shall take the following Response Activities:

a. Verified Sentinel Well Exceedance. In the event of a Verified Sentinel Well Exceedance, Defendant shall sample that Sentinel Well monthly. If the concentrations of 1,4-dioxane are less than 7.2 ug/L in samples from any two successive monthly sampling events, Defendant shall return to sampling that Sentinel Well quarterly. If, however, the concentrations of 1,4-dioxane exceed 7.2 ug/L in samples collected from the same Sentinel Well in any three successive monthly sampling events, Defendant shall take the following actions:

i. If involving a Sentinel Well in the north, installation of up to two additional well clusters near the Prohibition Zone boundary (the location of which shall be determined based on the location of the initial exceedance). If more than one Sentinel Well in the north exceeds the trigger level, Defendant and EGLE will mutually agree on the number of

PZ Boundary Wells to be installed. Defendant shall sample the new PZ Boundary Wells monthly until Defendant completes the hydrogeological assessment described in Section V.A.5.a.ii below.

ii. Completion of a focused hydrogeological assessment of the applicable area that analyzes the likelihood that 1,4-dioxane at levels above 7.2 ug/L will migrate outside the Prohibition Zone. The assessment shall also opine on the mechanism causing the exceedances and the potential risk of impact to private drinking water wells. Defendant shall provide this assessment to EGLE within 60 days after installation of the new PZ Boundary Well(s). If the focused hydrogeological assessment determines that there is a low potential for the Groundwater Contamination to migrate beyond the Prohibition Zone boundary, normal quarterly monitoring of the Sentinel Well and applicable PZ Boundary Wells will resume. If the focused hydrogeological assessment determines that there is a reasonable likelihood for 1,4-dioxane greater than 7.2 ug/L to migrate beyond the Prohibition Zone boundary, the Defendant shall initiate the following Response Activities:

(A) Defendant shall continue to monitor the affected Sentinel Well(s) and the Prohibition Zone Boundary Wells on a monthly basis.

(B) If the Verified Sentinel Well Exceedance occurs in a Sentinel Well to be installed near the northern boundary of the Prohibition Zone, Defendant shall develop a "Remedial Contingency Plan" that identifies the Response Activities that could be implemented to prevent Groundwater Contamination from migrating beyond the Prohibition Zone Boundary. The Remedial Contingency Plan may identify expansion of the Prohibition Zone as an option, subject to Section V.A.2.f. Defendant shall submit the Remedial Contingency Plan to EGLE within 45 days after the focused hydrogeological assessment is completed.

(C) Defendant will review the Municipal Water Connection Contingency Plan, if applicable, and initiate preliminary activities related to provision of municipal water to potentially impacted private drinking water wells. The amount of work to be completed will be based on the anticipated time frame for water extension and the projected time of migration to potential receptors.

b. Verified PZ Boundary Well Exceedance. In the event of a Verified PZ Boundary Well Exceedance, Defendant shall sample that PZ Boundary Well monthly. If the concentrations of 1,4-dioxane are less than 4.6 ug/L in samples from any two successive monthly sampling events, Defendant shall return to sampling that PZ Boundary Well quarterly. If, however, the concentrations of 1,4-dioxane exceed 4.6 ug/L in samples collected from the same PZ Boundary Well in any three successive monthly sampling events, Defendant shall take the following actions:

i. Defendant, in consultation with EGLE, shall sample select private drinking water wells in the immediate vicinity of the impacted PZ Boundary Well.

ii. Defendant will review the Municipal Water Connection Contingency Plan, and initiate further activities related to potential provision of municipal water to potentially impacted private drinking water wells as appropriate. The amount of work to be completed will be based on the anticipated time frames for water extension and the projected time of migration to potential receptors.

iii. Subject to Section V.A.2.f, Defendant shall implement the Remedial Contingency Plan as necessary to prevent contaminant levels above 7.2 ug/L from migrating beyond the Prohibition Zone Boundary.

c. Confirmed PZ Boundary Well Noncompliance. In the event of a

Confirmed PZ Boundary Well Noncompliance, Defendant shall sample that PZ Boundary Well monthly. If the concentrations of 1,4-dioxane are less than 7.2 ug/L in samples from any two successive monthly sampling events, Defendant shall return to sampling that PZ Boundary Well quarterly. If, however, the concentrations of 1,4-dioxane exceed 7.2 ug/L in samples collected from the same PZ Boundary Well in any four successive monthly sampling events, Defendant shall take the following actions:

i. Defendant shall sample any active drinking water wells in the immediate vicinity of the impacted PZ Boundary Well on a monthly basis.

ii. Defendant will review the Municipal Water Connection Contingency Plan and implement the remaining activities necessary to provide municipal water to properties serviced by private drinking water wells potentially impacted by 1,4-dioxane concentrations above the applicable drinking water cleanup criterion.

iii. Defendant shall connect any such properties to municipal water on a case-by-case basis as determined by EGLE or if requested by the property owner.

iv. Subject to Section V.A.2.f, Defendant shall undertake Response Actions as necessary to reduce concentrations in the affected PZ Boundary Well(s) to less than 7.2 ug/L.

d. Bottled Water. At any time, Defendant shall supply the occupants of any property with a threatened drinking water well with bottled water if, prior to connection to municipal water, 1,4-dioxane concentrations in the drinking water well servicing the property exceed 3.0 ug/L. This obligation shall terminate if either (i) the 1,4-dioxane concentration in the well drops below 3.0 ug/L during two consecutive sampling events or (ii) the property is connected to an alternative water supply.

e. Triangle Property. If a drinking water well is installed on the Triangle Property in the future, Defendant shall take the necessary steps to obtain permission to sample the well on a schedule approved by EGLE. Defendant shall monitor such well(s) on EGLE-approved schedule unless or until that property is included in the Prohibition Zone, at which time, any water well(s) shall be addressed as part of the well identification process described in Section V.A.2.h.

f. Downgradient Investigation. The Defendant shall continue to implement its Downgradient Investigation Work Plan as approved by EGLE on February 4, 2005, as may be amended, to track the Groundwater Contamination as it migrates to ensure any potential migration of Groundwater Contamination outside of the Prohibition Zone is detected before such migration occurs with sufficient time to allow Defendant to maintain compliance with the Prohibition Zone Containment Objective and to ensure compliance with the Groundwater-Surface Water Interface Objective. Defendant shall, as the next phase of this iterative investigation process investigate the area depicted on the map attached as Attachment G, including the installation of monitoring wells at the following locations subject to access and receipt of any required approvals pursuant to Section VII.D:

i. A monitoring well nest in the residential area in the general vicinity of intersection of Washington and 7th Streets (Location "F" on Attachment G);
iii. A shallow well in the residential area in the general vicinity of current monitoring well nest MW-98 (Location "G" on Attachment G); and
iv. A monitoring well nest in the residential area in the general vicinity of Brierwood and Linwood Streets (Location "H" on Attachment G).

The data from these wells will be used to guide additional downgradient investigations as necessary to ensure compliance with the Eastern Area Objectives.

6. Prohibition Zone Boundary Review.

a. Five years after entry of this Fourth Amended Consent Judgment and then every five years thereafter, Defendant and EGLE shall confer and determine whether the boundary of the Prohibition Zone can be contracted without either: (i) posing a current or future risk to the public health and welfare, including maintaining an adequate distance between the Groundwater Contamination and the Prohibition Zone boundary; or (ii) requiring Defendant to undertake additional Response Activities to contain the Groundwater Contamination within the contracted Prohibition Zone boundary beyond those Response Activities otherwise required immediately before the proposed contraction. This determination will be based on consideration of the totality of all data from existing Eastern Area monitoring wells.

b. If EGLE and Defendant jointly agree that the Prohibition Zone boundary may be contracted under these conditions, the Parties shall move to amend Attachments C and E of this Consent Judgment for the sole purpose of establishing a revised boundary for the Prohibition Zone. If only one Party concludes that the Prohibition Zone boundary may be contracted under these conditions, that Party may move to amend Attachments C and E of this Consent Judgment for the sole purpose of establishing a revised boundary for the Prohibition Zone, but must demonstrate by clear and convincing evidence that the above conditions are satisfied. The non-moving Party may oppose or otherwise respond to such motion and the showing required under Section XVI shall not apply to the Court's resolution of the motion.

c. If the Prohibition Zone boundary is contracted under Section V.A.6 and the Parties, either jointly or independently, subsequently determine that based on the totality of the data, the Prohibition Zone boundary should be expanded up to and including back to the boundary established by this Fourth Amended Consent Judgment in order to protect the public health and welfare, the Party(ies) may move to amend Attachments C and E of this Consent Judgment for the sole purpose of establishing a revised boundary for the Prohibition Zone. Neither Section XVI nor the showing required under SectionV.A.2.f shall apply to the Court's resolution of the motion, provided that the expansion sought does not extend beyond the boundary established by this Fourth Amended Consent Judgment.

d. To the extent the Prohibition Zone boundary is contracted under Section V.A.6.a, Defendant shall not be required to undertake Response Activities to contain the Groundwater Contamination within the contracted boundary beyond those Response Activities required immediately before the Prohibition Zone was contracted.

7. Operation and Maintenance. Subject to Sections V.A.3.f, V.A.9, and reasonably necessary maintenance according to EGLE-approved operation and maintenance plans, Defendant shall operate and maintain the Eastern Area System as necessary to meet the Prohibition Zone Containment Objective until Defendant is authorized to terminate extraction well operations pursuant to Section V.C.1.

8. Treatment and Disposal. Groundwater extracted by the extraction well(s) in the Eastern Area System shall be treated (as necessary depending on the disposal method(s) utilized) with ozone/hydrogen peroxide or ultraviolet light and oxidizing agent(s), or such other method approved by EGLE to reduce 1,4-dioxane concentrations to the required level and disposed of using methods approved by EGLE, including, but not limited to, the following options:

a. Groundwater Discharge. The purged groundwater shall be treated to reduce 1,4-dioxane concentrations to the level required by EGLE, and discharged to groundwater at locations approved by EGLE in compliance with a permit or exemption authorizing such discharge.

b. Sanitary Sewer Discharge. Use of the sanitary sewer leading to the Ann Arbor Wastewater Treatment Plant is conditioned upon approval of the City of Ann Arbor. If discharge is made to the sanitary sewer, the Evergreen and Maple Road Wells shall be operated and monitored in compliance with the terms and conditions of an Industrial User's Permit from the City of Ann Arbor, and any subsequent written amendment of that permit made by the City of Ann Arbor. The terms and conditions of any such permit and any subsequent amendment shall be directly enforceable by EGLE against Defendant as requirements of this Consent Judgment.

c. Storm Sewer Discharge. Use of the storm drain or sewer is conditioned upon issuance of an NPDES permit and approval of the appropriate regulatory authority(ies). Discharge to the Huron River via a storm water system shall be in accordance with the relevant NPDES permit and conditions required by the relevant regulatory authority(ies). If a storm drain or sewer is to be used for disposal of purged groundwater, Defendant shall submit to EGLE and the appropriate local regulatory authority(ies) for their review and approval, a protocol under which the purge system shall be temporarily shut down: (i) for maintenance of the storm drain or sewer and (ii) during storm events to assure that the storm water system retains adequate capacity to handle run-off created during such events. Defendant shall not be permitted or be under any obligation under this subsection to discharge purged groundwater to the storm drain or sewer unless the protocol for temporary shutdown is approved by all necessary authorities. Following approval of the protocol, the purge system shall be operated in accordance with the approved protocol.

d. Existing or Additional/Replacement Pipeline to Wagner Road Treatment Facility.

i. The existing deep transmission pipeline, an additional pipeline, or a pipeline replacing the existing deep transmission pipeline may be used to convey purged groundwater from the existing Evergreen Area infrastructure to the Wagner Road Treatment Facility where the purged groundwater shall be treated to reduce 1,4-dioxane concentrations to the level required by NPDES Permit No. MI-0048453, as amended or reissued.

ii. Installation of an additional pipeline or a replacement pipeline from the existing Evergreen Area to the Wagner Road Treatment Facility is conditioned upon approval of such installation by EGLE. If the pipeline is proposed to be installed on public property, the pipeline installation is conditioned upon approval of such installation by the appropriate local authority(ies), if required by statute or ordinance, or by Order of the Court pursuant to the authority under MCL 324.20135a. Defendant shall design and install the pipeline in compliance with all state requirements and install the pipeline with monitoring devices to detect any leaks. If leaks are detected, the system will automatically shut down and notify an operator of the condition. In the event that any leakage is detected, Defendant shall take any measures necessary to repair any leaks and perform any remediation that may be necessary. To reduce the possibility of accidental damage to the pipeline during any future construction, Defendant shall participate in the notification system provided by MISS DIG Systems, Inc., or its successor ("MISS DIG"), and shall comply with the provisions of MCL 460.721, *et seq.,* as may be amended and with the regulations promulgated thereunder. Defendant shall properly mark its facilities upon notice from MISS DIG.

e. Existing, Replacement, or Additional Pipeline from Maple Road

Extraction Well(s). Defendant may operate the existing pipeline or install and operate a replacement pipeline or an additional pipeline from the Maple Road Extraction Well(s) to the existing Evergreen area infrastructure to convey groundwater extracted from the Maple Road Extraction Wells to the Wagner Road Treatment Facility, where the purged groundwater shall be treated to reduce 1,4-dioxane concentrations to the level required by NPDES Permit No. MI-0048453, as amended or reissued. Installation and operation of an additional or replacement pipeline from the Maple Road area to Evergreen area is conditioned upon approval of such installation and operation by EGLE. If the pipeline is proposed to be installed on public property, the pipeline installation is conditioned upon approval of such installation by the appropriate local authorities, if required by statute or ordinance, or Order of the Court pursuant to the authority under MCL 324.20135a. Defendant shall design any such pipeline in compliance with all state requirements and install it with monitoring devices to detect any leaks. In the event any leakage is detected, Defendant shall take any measures necessary to repair any leaks and perform any remediation that may be necessary. To reduce the possibility of accidental damage to the pipeline, Defendant shall participate in the notification system provided by MISS DIG and shall comply with the provisions of MCL 460.721, *et seq*., as may be amended, and with the regulations promulgated thereunder. Defendant shall properly mark its facilities upon notice from MISS DIG.

f. Pipeline from Rose Well. Installation and operation of a proposed pipeline from the Rose Well to the existing Evergreen area infrastructure is conditioned upon approval of such installation and operation by EGLE. If the pipeline is proposed to be installed on public property, the pipeline installation is conditioned upon approval of such installation by the appropriate local authorities, if required by statute or ordinance, or Order of the Court pursuant to the authority under MCL 324.20135a. Defendant shall design and install any such pipeline in compliance with all state requirements and install it with monitoring devices to detect any leaks. In the event any leakage is detected, Defendant shall take any measures necessary to repair any leaks and perform any remediation that may be necessary. To reduce the possibility of accidental damage to the pipeline, Defendant shall participate in the notification system provided by MISS DIG and shall comply with the provisions of MCL 460.721, *et seq*., as may be amended, and with the regulations promulgated thereunder. Defendant shall properly mark its facilities upon notice from MISS DIG. Defendant may operate such pipeline to, among other things, convey groundwater extracted from the Rose Well to the existing Evergreen Area infrastructure and then to the Wagner Road Treatment Facility, where the purged groundwater shall be treated to reduce 1,4-dioxane concentrations to the level required by NPDES Permit No. MI-0048453, as amended or reissued.

g. Surface Water Discharge to First Sister Lake. Groundwater extracted from the Parklake Well may be discharged to First Sister Lake, conditioned on EGLE's issuance of an NPDES permit with effluent limitations, discharge limits (other than volume), and other conditions no more restrictive than those included in Defendant's 2014 NPDES Permit that authorizes discharge of groundwater to First Sister Lake following treatment with ozone/hydrogen peroxide technology. Defendant shall submit a protocol to EGLE and the appropriate local authority(ies) for their review and approval, a protocol under which the Parklake Well shall be temporarily shut down during storm events or high water levels in First Sister Lake as necessary to avoid flooding. Defendant shall not be under any obligation to operate the Parklake Well unless the protocol for temporary shutdown is approved by all necessary authorities. Following approval of the protocol, Defendant shall operate the Parklake

Well in accordance with the approved protocol.

9. Wagner Road Extraction. The extraction wells currently or in the future located just west of Wagner Road (the "Wagner Road Wells") shall be considered part of the Eastern Area System even though they are located west of Wagner Road. The Defendant shall initially operate the Wagner Road Wells at a combined 200 gpm extraction rate. The Defendant shall continue to operate the Wagner Road Wells in order to reduce the migration of 1,4-dioxane east of Wagner Road at this rate until such time as the Eastern Area Objectives will be met with a lower combined extraction rate or without the need to operate these wells or that reduction of the Wagner Road extraction rate would enhance 1,4-dioxane mass removal from the Parklake Well and/or the Rose Well/IW-2 and Defendant's efforts to reduce the mass of 1,4-dioxane migrating east of Maple Road and/or through the Evergreen Subdivision Area. Before significantly reducing or terminating extraction from the Wagner Road Wells, Defendant shall consult with EGLE and provide a written analysis, together with the data that supports its conclusion that the above-objectives can be met at a reduced extraction rate or without the need to operate these extraction wells. EGLE will review the analysis and data and provide a written response to Defendants within 56 days after receiving Defendant's written analysis and data. If Defendant disagrees with EGLE's conclusion, Defendant may initiate dispute resolution under Section XVI of this Consent Judgment. The Defendant shall not significantly reduce or terminate the Wagner Road extraction during the 56-day review period or while Defendant is disputing EGLE's conclusion.

10. Options Array for Transmission Line Failure/Inadequate Capacity. The Defendant has provided EGLE with documentation regarding the life expectancy of the deep transmission line and an Options Array (attached as Attachment H). The Options Array

describes the various options that may be available if the deep transmission line fails or the 200 gpm capacity of the existing deep transmission line that transports groundwater from the Eastern Area System to the treatment system located on the Gelman Property proves to be insufficient to meet the Prohibition Zone Containment Objective.

B. Western Area

1. Western Area Non-Expansion Cleanup Objective. The Defendant shall prevent the horizontal extent of the Groundwater Contamination in the Western Area, regardless of the depth (as established under Section V.B.3.b and c), from expanding. Compliance with this objective shall be determined as set forth in Section V.B.4, below. Continued migration of Groundwater Contamination into the Prohibition Zone, as may be modified, shall not be considered expansion and is allowed. A change in the horizontal extent of Groundwater Contamination resulting solely from the Court's application of a new cleanup criterion shall not constitute expansion. Nothing in this Section prohibits EGLE from seeking additional response activities pursuant to Section XVIII.E of this Consent Judgment. Compliance with the Non-Expansion Cleanup Objective shall be established and verified by the network of monitoring wells in the Western Area to be selected and/or installed by the Defendant as provided in Sections V.B.3.b and c, below ("Western Area Compliance Well Network") and the Compliance Process set forth in Section V.B.4 ("Western Area Compliance Process"). Except as provided in Section VI.C.1, there is no independent mass removal requirement or a requirement that Defendant operate any particular Western Area extraction well(s) at any particular rate beyond what is necessary to prevent the prohibited expansion, provided that Defendant's ability to terminate all groundwater extraction in the Western Area is subject to Section V.C.1.c and the establishment of property use restrictions as required by Section V.B.3.a. If prohibited

expansion occurs, as determined by the Western Area Compliance Well Network and the Western Area Compliance Process, Defendant shall undertake additional response activities to return the Groundwater Contamination to the boundary established by the Western Area Compliance Well Network (such response activities may include groundwater extraction at particular locations).

As part of the Third Amendment to Consent Judgment, EGLE agreed to modify the remedial objective for the Western Area as provided herein to a no expansion performance objective in reliance on Defendant's agreement to comply with a no expansion performance objective for the Western Area. To ensure compliance with this objective, Defendant acknowledges that in addition to taking further response action to return the horizontal extent of Groundwater Contamination to the boundary established by the Compliance Well Network, Defendant shall be subject to stipulated penalties for violation of the objective as provided in Section XVII. Nothing in this Section shall limit Defendant's ability to contest the assessment of such stipulated penalties as provided in this Consent Judgment.

2. Western Area Groundwater-Surface Water Interface Objective.

a. Defendant shall prevent 1,4-dioxane from venting into surface waters in the Western Area at concentrations above the Generic GSI Cleanup Criterion, except in compliance with Part 201, including MCL 324.20120e ("Groundwater-Surface Water Interface Objective" for the Western Area).

b. GSI Investigation Work Plan. Within 90 days of entry of this Consent Judgment, Defendant shall submit to EGLE for its review and approval a work plan for investigation of the groundwater-surface water interface in the Western Area and a schedule for implementing the work plan. Defendant's work plan shall include:

i. An evaluation of the Western Area and identification of any areas where the GSI pathway is relevant, i.e., any areas where 1,4-dioxane in groundwater is reasonably expected to vent to surface water in concentrations that exceed the Generic GSI Criterion based on evaluation of the factors listed in MCL 324.20120e(3); and

ii. A description of the Response Activities Defendant will take to determine whether 1,4-dioxane in groundwater is venting to surface water in any such areas in concentrations that exceed the Generic GSI Criterion.

c. GSI Response Activity Work Plan. With respect to any areas where the above-described GSI investigation demonstrates that 1,4-dioxane in groundwater is venting to surface water in any such areas in concentrations that exceed the Generic GSI Criterion, Defendant shall submit for EGLE review and approval a work plan and a schedule for implementing the work plan that describes the Response Activities, including any evaluations under MCL 324.20120e, Defendant will undertake to ensure compliance with Groundwater-Surface Water Interface Objective within a reasonable timeframe.

d. Compliance with Groundwater-Surface Water Interface Objective. Defendant shall undertake such Response Activities and/or evaluations as necessary to achieve compliance with the Groundwater-Surface Water Interface Objective. It shall not be a violation of this Consent Judgment nor shall Defendant be subject to stipulated penalties unless and until Defendant fails to achieve compliance with the Groundwater-Surface Water Interface Objective within a reasonable timeframe established by EGLE and then only from that point forward. EGLE's determination of a reasonable timeframe for compliance with the Groundwater-Surface Water Interface Objective is subject to dispute resolution under Section XVI.

3. <u>Western Area Response Activities</u>. Defendant shall implement the

following response activities:

a. Groundwater Extraction. The Western Area Response Activities shall include the operation of groundwater extraction wells as necessary to meet the objectives described in Section V.B.1 and 2, including operation of the Marshy Area groundwater extraction system described in Defendant's May 5, 2000 Final Design and Effectiveness Monitoring Plan, as subsequently modified and approved by EGLE. Defendant shall also install and operate additional groundwater extraction wells at the Gelman Property as described in Section VI, below, in order to reduce the mass of 1,4-dioxane in the groundwater. Purged groundwater from the Western Area shall be treated with ozone/hydrogen peroxide or ultraviolet light and oxidizing agent(s), or such other method approved by EGLE to reduce 1,4-dioxane concentrations to the level required by NPDES Permit No. MI-0048453, as amended or reissued. Discharge to the Honey Creek tributary shall be in accordance with NPDES Permit No. MI-0048453, as amended or reissued. The Defendant shall have property use restrictions that are sufficient to prevent unacceptable exposures in place for any properties affected by Soil Contamination or Groundwater Contamination before completely terminating extraction in the Western Area.

b. Western Area Delineation Investigation. Defendant shall install the following additional groundwater monitoring wells pursuant to a schedule approved by EGLE and subject to the accessibility of the locations and obtaining access and any required approvals under Section VII.D at the approximate locations described below and on the map attached as Attachment G to address gaps in the current definition of the Groundwater Contamination and to further define the horizontal extent of Groundwater Contamination in the Western Area:

i. Commercial area north of Jackson Road (across from April Drive) and south of US-Highway I-94, near MW-40s&d. (Deep well only) (Location "I" on Attachment G);

ii. Commercial area north of Jackson Road (across from Nancy Drive) and south of US-Highway I-94, east of MW-40s&d and west of the MW-133 cluster (Location "J" on Attachment G);

iii. Residential area west of West Delhi, north of Jackson Road and south of US-Highway I-94 (Location "K" on Attachment G);

iv. Residential area southwest of the MW-141 cluster in the vicinity of Kilkenny and Birkdale (Location "L" on Attachment G);

v. Residential area along Myrtle between Jackson Road and Park Road (Shallow Well only) (Location "M" on Attachment G); and

vi. Residential and vacant area within approximately 250 feet of Honey Creek southwest of Dexter Road (Location "N" on Attachment G).

This investigation may be amended by agreement of EGLE and the Defendant to reflect data obtained during the investigation. Defendant shall promptly provide the data/results from the investigation to EGLE so that EGLE receives them prior to Defendant's submission of the Compliance Monitoring Plan described in Subsection V.B.3.c, below. Based on the data obtained from the wells described above, Defendant may propose to install additional monitoring wells to potentially serve as Compliance Wells rather than one or more of the wells identified above. EGLE reserves the right to request the installation of additional borings/monitoring wells, if the totality of the data indicate that the horizontal extent of Groundwater Contamination has not been completely defined.

c. Compliance Well Network and Compliance Monitoring Plan.

Within 30 days of completing the investigation described in Subsection V.B.3.b, above, Defendant shall amend its Western Area Monitoring Plan dated April 18, 2011, including Defendant's analysis of the data obtained during the investigation for review and approval by

EGLE, to identify the network of compliance wells that will be used to confirm compliance with the Western Area Non-Expansion Cleanup Objective (hereinafter referred to as the "Compliance Monitoring Plan"). The Compliance Monitoring Plan shall include the collection of data from a compliance well network sufficient to verify the effectiveness of the Western Area System in meeting the Western Area Non-Expansion Cleanup Objective. The locations and/or number of the Compliance Wells for the Compliance Monitoring Plan will be determined based on the data obtained from the investigation Defendant shall conduct pursuant to Section V.B.3.b, and shall be made up of existing monitoring wells. EGLE shall approve the Compliance Monitoring Plan, submit to Defendant changes in the Compliance Monitoring Plan that would result in approval, or deny the Compliance Monitoring Plan within 35 days of receiving the Compliance Monitoring Plan. Defendant shall either implement the EGLE-approved Compliance Monitoring Plan, including any changes required by EGLE, or initiate dispute resolution pursuant to Section XVI of this Consent Judgment. Defendant shall implement the EGLE- (or Court)-approved Compliance Monitoring Plan to verify the effectiveness of the Western Area System in meeting the Western Area Non-Expansion Cleanup Objective. Defendant shall continue to implement the current EGLE-approved monitoring plan(s) until EGLE approves the Compliance Monitoring Plan required by this Section. The monitoring program shall be continued until terminated pursuant to Section V.D.

d. Municipal Water Connection Contingency Plan ("MWCCP"). Defendant shall develop a MWCCP addressing the potential provision of township water to properties using private drinking water wells on Elizabeth Road. The MWCCP will be developed according to a schedule to be approved by EGLE.

4. Compliance Determination for Non-Expansion Objective. The Compliance Monitoring Plan shall include the following steps for verifying sampling results and confirming compliance or noncompliance with the Western Area Non-Expansion Cleanup Objective.

a. Monitoring Frequency/Analytical Method. Defendant will sample groundwater from the Compliance Wells on a quarterly basis unless an alternative schedule is agreed upon on with EGLE. Groundwater samples will be submitted to a laboratory owned, operated or contracted by Defendant for 1,4-dioxane analysis.

b. Verification Process. Defendant shall conduct the Verification Process as defined in Section III.X for each Compliance Well to verify any exceedance of 7.2 ug/L. A verified detection above 7.2 ug/L will be considered a "Verified Compliance Well Exceedance." If a second sample does not exceed 7.2 ug/L, monitoring of the well will increase to monthly until the pattern of exceedances is broken by two successive sampling events below 7.2 ug/L. At that point, a quarterly monitoring frequency will resume.

c. Response Activities. In the event of a Verified Compliance Well Exceedance, Defendant shall take the following Response Activities:

i. Sample selected nearby private drinking water wells. Defendant shall sample select private drinking water wells unless otherwise the Parties otherwise agree. Prior to sampling the selected wells, Defendant shall submit a list of the wells to be sampled and other sampling details to EGLE for approval. In selecting wells to be sampled, Defendant shall consider data collected from monitoring and private drinking water wells within 1,000 feet of the Compliance Well(s) that exceeded 7.2 ug/L, groundwater flow, hydrogeology and well depth. EGLE shall respond within seven days after receipt of Defendant's list of select private drinking water wells and shall either approve the list or propose alternate or additional wells to be sampled.

ii. If a Verified Compliance Well Exceedance occurs in the same Compliance Well in any two successive monthly sampling events, Defendant shall take the following Response Activities:

(A) Continue to sample the previously selected private drinking water well(s) on a monthly basis unless otherwise agreed upon with EGLE.

(B) Conduct focused hydrogeological investigation to determine whether the Verified Compliance Well Exceedance is a temporary fluctuation or evidence of plume expansion. The investigation shall include the measurement of groundwater levels in relevant monitoring wells in the vicinity of the Compliance Well with the Verified Compliance Well Exceedance. Defendant shall report its findings to EGLE within 30 days of completing the hydrogeological investigation.

(C) Conduct Statistical Analysis. During the eight month period after the second consecutive Verified Compliance Well Exceedance, Defendant shall complete a statistical analysis of the data using a Mann-Kendall Trend Test or other statistical technique approved by EGLE.

(D) Interim Measures Feasibility Study. During the eight month period after the second consecutive Verified Compliance Well Exceedance, Defendant shall evaluate affirmative measures to control expansion of the Groundwater Contamination as necessary to reduce the concentration of 1,4-dioxane in the relevant Compliance Well to below 7.2 ug/L, including adjustments in groundwater extraction rates, the installation of additional groundwater extraction wells or other remedial technologies.

Defendant shall submit to EGLE a feasibility study within 240 days of the Verified Compliance Well Exceedance. The feasibility study shall include an evaluation of the feasibility and effectiveness of all applicable measures to control expansion of the Groundwater Contamination as necessary to reduce the concentration of 1,4-dioxane in the relevant Compliance Well to below 7.2 ug/L in light of the geology and current understanding of the fate and transport of the Groundwater Contamination.

iii. If, after conducting the focused hydrogeological investigation and statistical analysis, the totality of the data evidences a reasonable likelihood that the Western Area Non-Expansion Cleanup Objective is not being met, Defendant shall evaluate and, subject to EGLE approval, implement one or more of the potential response activities identified in the feasibility study, or other response activities, as necessary to achieve compliance with the Western Area Non-Expansion Cleanup Objective. Nothing in this Section shall prevent Defendant from implementing response activities as necessary to achieve the Western Area Non-Expansion Cleanup Objective at an earlier time.

d. Stipulated Penalties/Exacerbation. Defendant shall not be subject to stipulated penalties until concentrations in at least four consecutive monthly samples from a given Compliance Well exceed 7.2 ug/L, at which point Defendant shall be subject to stipulated penalties for violation of the Western Area Non-Expansion Cleanup Objective as provided in Section XVII, provided, however, that Defendant shall not be subject to stipulated penalties with respect to prohibited expansion of the horizontal extent of the Groundwater Contamination if Defendant can demonstrate by a preponderance of the evidence that the migration of the Groundwater Contamination is caused in whole or in part by the actions of an unrelated third party that have contributed to or exacerbated the Groundwater Contamination. In such event, although Defendant is not subject to stipulated penalties, Defendant shall remain responsible for mitigating the migration of the Groundwater Contamination. Nothing in this Consent Judgment shall preclude Defendant from seeking contribution or cost recovery from other parties responsible for or contributing to exacerbation of the Groundwater Contamination.

e. Private Drinking Water Well Response Activities. If, after conducting the focused hydrogeological investigation and statistical analysis, the totality of the data evidences a reasonable likelihood that 1,4-dioxane will be present at concentrations above 7.2 ug/L in a residential drinking water well and/or at concentrations above 350 ug/L in an active non-residential drinking water well, Defendant shall evaluate and, if appropriate, implement response activities, including, without limitation, the following:

i. Sampling of at risk drinking water well(s) on a monthly basis;

ii. Implementation of affirmative interim measures to mitigate the expansion of 1,4-dioxane at concentrations above the applicable drinking water standard toward the drinking water well(s) as determined in the feasibility study described in Section V.B.4.c.ii.(D);

iii. Evaluation of land use restrictions and/or institutional controls to eliminate drinking water exposures to 1,4-dioxane in the groundwater at concentrations above the applicable drinking water standard; and

iv. Evaluation of water supply alternatives including, but not limited to, providing bottled water, a township water connection, installation of a new drinking water well completed in an uncontaminated portion of the subsurface, and point-of-use treatment systems.

v. If at any time 1,4-dioxane is detected in an active private drinking water well above 3.0 ug/L, Defendant shall promptly at its expense, offer the occupants of the property the option of receiving bottled water and shall sample the well monthly. These obligations shall terminate if either (i) the 1,4-dioxane concentration in the well drops below 3.0 ug/L during two consecutive sampling events or (ii) the property is connected to a permanent alternative water supply. Furthermore, Defendant shall work with EGLE and municipal authorities to evaluate long-term and economically reasonable water supply options.

vi. If 1,4-dioxane is detected at concentrations above 7.2 ug/L in an active residential drinking water well and/or at concentrations above 350 ug/L in an active non-residential drinking water well, Defendant shall conduct the Verification Process as defined in Section III.X for each such private drinking water well. If the detection above 7.2 ug/L is verified, Defendant shall monitor each such private drinking water well on a monthly basis if not already doing so and shall continue monthly monitoring until the well is no longer considered at risk under Section V.B.4.e.i. If 1,4-dioxane is detected at concentrations above 7.2 ug/L in four consecutive monthly samples or any seven monthly samples in any 12 month period, Defendant shall provide at its expense a long-term alternative water supply to the property serviced by the affected well. Such long-term alternative water supply may be in the form of a township water connection, installation of a new drinking water well completed in an uncontaminated portion of the subsurface, or a point-of-use treatment system, or other long-term drinking water supply option approved by EGLE. Defendant shall also provide at its expense bottled water to the property owner until the property is serviced by a long-term alternative water supply.

5. Groundwater Contamination Delineation. Additional delineation of the extent of Groundwater Contamination, including within the plume boundary, and/or

characterization of source areas shall not be required except as provided in Section V.B.3.c. EGLE reserves the right to petition the Court to require additional work if there are findings that EGLE determines warrant additional Groundwater Contamination delineation.

C. Termination of Groundwater Extraction Systems

1. Defendant may only terminate the Groundwater Extraction Systems listed below as provided below:

a. Termination Criteria for Evergreen Wells/Maple Road Wells/Wagner Road Wells. Except as otherwise provided pursuant to Section V.C.2, Defendant may only reduce (below the stated minimum purge rates) or terminate operation of the Evergreen Wells/Maple Road Wells as provided in Section V.A.3.f.i. and of the Wagner Road Wells as provided in Section V.A.9.

b. Termination Criteria for Parklake Well. Except as otherwise provided pursuant to Section V.C.2, Defendant may reduce or terminate operation of the Parklake Well as provided in Section V.A.3.f.ii.

c. Termination Criteria for Western Area. Defendant may terminate the groundwater extraction described in Section VI.C.1 as provided in that Section. Except as otherwise provided pursuant to Section V.C.2, and subject to Section V.B.1., Defendant shall not terminate all groundwater extraction in the Western Area until all of the following are established:

i. Defendant can establish to EGLE's satisfaction that groundwater extraction is no longer necessary to prevent the expansion of Groundwater Contamination prohibited under Section V.B.1;

ii. Defendant's demonstration shall also establish that groundwater extraction is no longer necessary to satisfy the Groundwater-Surface Water Interface Objective under Section V.B.2; and

iii. Defendant has the land use or resource use restrictions described in Section V.B.3.a in place.

Defendant's request to terminate extraction in the Western Area must be made in writing for review and approval pursuant to Section X of this Consent Judgment. The request must include all supporting documentation demonstrating compliance with the termination criteria. Defendant may initiate dispute resolution pursuant to Section XVI of this Consent Judgment if EGLE does not approve the Defendant's request/demonstration. Defendant may terminate Western Area groundwater extraction upon: (i) receipt of notice of approval from EGLE; or (ii) receipt of notice of a final decision approving termination pursuant to dispute resolution procedures of Section XVI of this Consent Judgment.

2. Modification of Termination Criteria/Cleanup Criteria. The termination criteria provided in Section V.C.1. and/or the definition of "Groundwater Contamination" or "Soil Contamination" may be modified as follows:

a. After entry of this Fourth Amended Consent Judgment, Defendant may propose to EGLE that the termination criteria be modified based upon either or both of the following:

i. a change in legally applicable or relevant and appropriate regulatory criteria since the entry of this Fourth Amended Consent Judgment; for purposes for this Subsection, "regulatory criteria" shall mean any promulgated standard criterion or limitation under federal or state environmental law specifically applicable to 1,4-dioxane; or

ii. scientific evidence newly released since the date of the

United States Environmental Protection Agency's IRIS risk assessment for 1,4-dioxane (August 11, 2010), which, in combination with the existing scientific evidence, establishes that different termination criteria/definitions for 1,4-dioxane are appropriate and will assure protection of public health, safety, welfare, the environment, and natural resources.

b. Defendant shall submit any such proposal in writing, together with supporting documentation, to EGLE for review.

c. If the Defendant and EGLE agree to a proposed modification, the agreement shall be made by written Stipulation filed with the Court pursuant to Section XXIV of this Consent Judgment.

d. If EGLE disapproves the proposed modification, Defendant may invoke the dispute resolution procedures contained in Section XVI of this Consent Judgment. Alternatively, if EGLE disapproves a proposed modification, Defendant may seek to have the dispute resolved pursuant to Subsection V.C.3.

3. If the Defendant invokes the procedures of this Subsection, Defendant and EGLE shall prepare a list of the items of difference to be submitted to a scientific advisory panel for review and recommendations. The scientific advisory panel shall be comprised of three persons with scientific expertise in the discipline(s) relevant to the items of difference. No member of the panel may be a person who has been employed or retained by either Party, except persons compensated solely for providing peer review of the Hartung Report, in connection with the subject of this litigation.

a. If this procedure is invoked, each Party shall, within 14 days, select one member of the panel. Those two members of the panel shall select the third member. Defendant shall, within 28 days after this procedure is invoked, establish a fund of at least

$10,000.00, from which each member of the panel shall be paid reasonable compensation for their services, including actual and necessary expenses. If EGLE and Defendant do not agree concerning the qualifications, eligibility, or compensation of panel members, they may invoke the dispute resolution procedures contained in Section XVI of this Consent Judgment.

b. Within a reasonable period of time after selection of all panel members, the panel shall confer and establish a schedule for acceptance of submissions from EGLE and the Defendant completing review and making recommendations on the items of difference.

c. The scientific advisory panel shall make its recommendations concerning resolution of the items of difference to EGLE and the Defendant. If both EGLE and Defendant accept those recommendations, the termination criteria shall be modified in accordance with such recommendations. If EGLE and the Defendant disagree with the recommendations, EGLE's proposed resolution of the dispute shall be final unless Defendant invokes the procedures for judicial dispute resolution as provided in Section XVI of this Consent Judgment. The recommendation of the scientific advisory panel and any related documents shall be submitted to the Court as part of the record to be considered by the Court in resolving the dispute.

D. Post-Termination Monitoring

1. Eastern Area

a. Prohibition Zone Containment Objective. Except as otherwise provided pursuant to Section V.C.2, Defendant shall continue to monitor the Groundwater Contamination as it migrates within the Prohibition Zone until all approved monitoring wells are below 7.2 ug/L or such other applicable criterion for 1,4-dioxane for six consecutive months, or

Defendant can establish to EGLE's satisfaction that continued monitoring is not necessary to satisfy the Prohibition Zone Containment Objective. Defendant's request to terminate monitoring must be made in writing for review and approval pursuant to Section X of this Consent Judgment. Defendant may initiate dispute resolution pursuant to Section XVI of this Consent Judgment if EGLE does not approve its termination request.

b. Groundwater-Surface Water Interface Objective. Except as provided in Section V.D.1.a, for Prohibition Zone monitoring wells, post-termination monitoring is required for Eastern Area wells for a minimum of ten years after purging is terminated under Section V.C.1.a with cessation subject to EGLE approval. Defendant's request to terminate monitoring must be made in writing for review and approval pursuant to Section X of this Consent Judgment. Defendant may initiate dispute resolution pursuant to Section XVI of this Consent Judgment if EGLE does not approve its termination request.

2. Western Area. Post-termination monitoring will be required for a minimum of ten years after termination of extraction with cessation subject to EGLE approval. Except as otherwise provided pursuant to Section V.C.2, Defendant shall continue to monitor the groundwater in accordance with approved monitoring plan(s), to verify that it remains in compliance with the Non-Expansion Cleanup Objective set forth in Section V.B.1 and the Groundwater-Surface Water Interface Objective set forth in Section V.B.2. If any exceedance is detected, Defendant shall immediately notify EGLE and take whatever steps are necessary to comply with the requirements of Section V.B.1, or V.B.2, as applicable.

E. Quality Assurance Project Plan (QAPP). Defendant previously voluntarily submitted to EGLE for review and approval a QAPP, which is intended to describe the quality control, quality assurance, sampling protocol, and chain of custody procedures that will be used in carrying out the tasks required by this Consent Judgment. EGLE shall review, and Defendant shall revise accordingly, the QAPP to ensure that it is in general accordance with the United States Environmental Protection Agency's ("U.S. EPA" or "EPA") "Guidance for Quality Assurance Project Plans," EPA QA/G-5, December 2002; and American National Standard ANSI/ASQC E4-2004, "Quality Systems For Environmental Data And Technology Programs – Requirements With Guidance For Use."

VI. GELMAN PROPERTY RESPONSE ACTIVITIES

A. Gelman Property Objectives. The objectives for the Gelman Property shall be to prevent the migration of 1,4-dioxane from contaminated soils on the Gelman Property into any aquifer at concentrations or locations that cause non-compliance with the Western Area objectives set forth in Sections V.B.1 and V.B.2.

B. Response Activities.

1. Remedial Systems. Defendant shall design and implement remedial systems at the Gelman Property as necessary to achieve the Gelman Property Objectives.

2. Monitoring. Defendant shall implement an EGLE-approved Compliance Monitoring Plan to verify that the Gelman Property Soil Contamination does not cause or contribute to non-compliance with the Western Area objectives set forth in Sections V.B.1 and V.B.2, and to verify the effectiveness of any implemented remedial system.

C. Additional Source Control. Defendant shall implement the following Response Activities to reduce the mass of and/or exposure to 1,4-dioxane present in the soils and/or shallow groundwater on the Gelman Property subject to receipt of any required approvals pursuant to Section VII.D:

1. Additional Groundwater Extraction. Defendant shall install and operate

three "Phase I" extraction wells (one of which was previously installed) at the general locations depicted in the attached Attachment I to enhance control and mass removal of 1,4-dioxane from this area of shallow groundwater contamination. Defendant shall operate these extraction wells at a combined purge rate of approximately 75 gpm, subject to aquifer yield. Defendant shall have the discretion to adjust the individual well purge rates in order to optimize mass removal. Subject to Defendant's ability to adjust individual well purge rates, Defendant shall continue to extract a combined purge rate of approximately 75 gpm, subject to aquifer yield, from this system until the 1,4-dioxane concentration in the groundwater extracted from each of these extraction wells has been reduced below 500 ug/L and, once the concentrations in all three of the wells have been reduced below 500 ug/L, Defendant shall cycle those wells off and on for several periods of time approved by EGLE to demonstrate that significant concentration rebound is not occurring. Before otherwise significantly reducing or terminating extraction from this system, Defendant shall consult with EGLE and provide a written analysis, together with the data that supports its conclusion that the concentration of 1,4-dioxane in the groundwater extracted from each of these wells has been reduced below 500 ug/L, as stated above. EGLE will review the analysis and data and provide a written response to Defendants within 56 days after receiving Defendant's written analysis and data. If Defendant disagrees with EGLE's conclusion, Defendant may initiate dispute resolution under Section XVI of this Consent Judgment. The Defendant shall not significantly reduce or terminate the extraction from this system during the 56-day review period or while Defendant is disputing EGLE's conclusion.

Based on the performance achieved from these extraction wells, the Parties shall evaluate whether installation of up to three additional extraction wells at the general locations indicated on Attachment I would accelerate mass removal to a degree that meaningfully benefits the

Remediation. If EGLE determines that additional mass removal from these locations would be beneficial, Defendant shall, subject to its right to invoke Dispute Resolution under Section XVI, install and operate these additional wells pursuant to a work plan approved by EGLE.

Groundwater extracted from the extraction wells described in this subparagraph will be conveyed to the Wagner Road Treatment Facility for treatment and disposal pursuant to Defendant's NPDES Permit No. MI-0048453, as amended or re-issued.

2. Phytoremediation—Former Pond 1 and 2 Area. Defendant shall apply phytoremediation techniques in the treatment area depicted on Attachment I to reduce the potential mass flux of 1,4-dioxane from vadose zone soils in this area to the groundwater aquifers. Defendant shall plant and maintain trees in the treatment area in order to: (i) remove 1,4-dioxane mass by via biodegradation and transpiration; and (ii) extract and reduce the volume of shallow perched groundwater in this area. Defendant shall install and maintain the trees in a healthy state and replace trees as necessary to assure continued success of the phytoremediation system. Defendant shall continue to operate the phytoremediation system as set forth above until it determines that the further reduction of the mass flux of 1,4-dioxane from the vadose zone soils to the groundwater aquifers is not necessary to achieve compliance with the Gelman Property Objectives. Before significantly reducing or terminating phytoremediation in the Former Pond 1 and 2 area, Defendant shall consult with EGLE and provide a written analysis, together with the data that supports its conclusions. EGLE will review the analysis and data and provide a written response to Defendants within 56 days after receiving Defendant's written analysis and data. If Defendant disagrees with EGLE's conclusion, Defendant may initiate dispute resolution under Section XVI of this Consent Judgment. The Defendant shall not significantly reduce or terminate the phytoremediation during the 56-day review period or while

Defendant is disputing EGLE’s conclusion.

3. Phytoremediation—Marshy Area. Defendant will undertake actions to reduce the percolation/infiltration of 1,4-dioxane from Marshy Area to the underlying groundwater through the application of phytoremediation techniques in the area depicted in Attachment I. The initial phase of these Response Activities may include further investigation of the Marshy Area as needed to complete the phytoremediation design regarding methods of enabling roots from trees grown in the Marshy Area to extend into deeper soils containing elevated concentrations of 1,4-dioxane. Defendant shall install and maintain the trees in a healthy state as necessary to assure continued success of the phytoremediation system. Defendant shall continue to operate the phytoremediation system as set forth above until it determines that the further reduction of the percolation/infiltration of 1,4-dioxane from the Marshy Area to the underlying groundwater is not necessary to achieve compliance with the Gelman Property Objectives. Before significantly reducing or terminating phytoremediation in the Marshy Area, Defendant shall consult with EGLE and provide a written analysis, together with the data that supports its conclusions. EGLE will review the analysis and data and provide a written response to Defendants within 56 days after receiving Defendant’s written analysis and data. If Defendant disagrees with EGLE’s decision to reduce or terminate the phytoremediation in the Marshy Area, Defendant may initiate dispute resolution under Section XVI of this Consent Judgment. The Defendant shall not significantly reduce or terminate the phytoremediation in the Marshy Area during the 56-day review period or while Defendant is disputing EGLE’s conclusion.

4. Former Burn Pit Area. Defendant shall undertake the following Response Activities with respect to the former Burn Pit area depicted on Attachments I and J:

a. Install, operate, and maintain a Heated Soil Vapor Extraction System ("HSVE System"). The HSVE System shall be designed to reduce the mass of 1,4-dioxane present in the soils in the portion of the former Burn Pit area identified as "Heated Soil Vapor Extraction" on Attachment J. Defendant shall operate the HSVE system until 1,4-dioxane concentrations in the HSVE System's effluent/exhaust has been reduced to levels that indicate that continued operation of the HVSE system will no longer contribute to meaningful reduction of 1,4-dioxane mass in the Former Burn Pit Area Soils or the Soil Contamination in the treatment area is eliminated, whichever occurs first. Before significantly reducing or terminating operation of the HSVE system, Defendant shall consult with EGLE and provide a written analysis, together with the data that supports its conclusion, that one or both of the above conditions has been satisfied. EGLE will review the analysis and data and provide a written response to Defendant within 56 days after receiving Defendant's written analysis and data. If Defendant disagrees with EGLE's conclusion, Defendant may initiate dispute resolution under Section XVI of this Consent Judgment. The Defendant shall not significantly reduce or terminate operation of the HSVE system during the 56-day review period or while Defendant is disputing EGLE's conclusion.

Following completion of the HSVE treatment, Defendant shall install an impervious barrier over the HSVE Treatment Area to inhibit water from percolating through the soils in the former Burn Pit Area, except with regard to any areas where Defendant can demonstrate to EGLE's satisfaction that Soil Contamination does not exist. Defendant shall maintain the impervious barrier in place until Soil Contamination is no longer present in the underlying soils.

b. Cap the portion of the former Burn Pit area identified as "Capped Area" on Attachment J with an impervious barrier to inhibit water from percolating through the soils in the former Burn Pit area. Defendant shall maintain the impervious barrier in place until Soil Contamination is no longer present in the underlying soils.

5. After completing installation of the Response Activity systems listed in Sections VI.C.2, VI.C.3 and VI.C.4, the Defendant shall submit a separate installation report (i.e., as-built report) for each of the systems. The reports shall describe the systems as installed including, but not limited to, components of a system, location of components within the specific areas, depths of components of a system, and operational specifications of components of a system.

6. Required Approvals. Notwithstanding the above, Defendant's obligation to implement any of the additional source control Response Activities described in Section VI.C is conditioned upon receipt of any required approvals pursuant to Section VII.D.

VII. COMPLIANCE WITH OTHER LAWS AND PERMITS

A. Defendant shall undertake all activities pursuant to this Consent Judgment in accordance with the requirements of all applicable laws, regulations, and permits.

B. Defendant shall apply for all permits necessary for implementation of this Consent Judgment including, without limitation, surface water discharge permit(s) and air discharge permit(s).

C. Defendant shall include in all contracts entered into by the Defendant for Remedial Action required under this Consent Judgment (and shall require that any contractor include in all subcontracts), a provision stating that such contractors and subcontractors, including their agents and employees, shall perform all activities required by such contracts or subcontracts in compliance with and all applicable laws, regulations, and permits. Defendant shall provide a copy of relevant approved work plans to any such contractor or subcontractor.

D. The Plaintiffs agree to provide reasonable cooperation and assistance to the Defendant in obtaining necessary approvals and permits for Remedial Action. Plaintiffs shall not unreasonably withhold or delay any required approvals or permits for Defendant's performance of Remedial Action. Plaintiffs expressly acknowledge that one or more of the following permits and approvals may be a necessary prerequisite for one or more of the Response Activities set forth in this Consent Judgment:

1. Renewal of NPDES Permit No. MI-0048453 with respect to the discharge of treated groundwater to the unnamed tributary of Honey Creek.

2. An NPDES Permit that authorizes the discharge of groundwater to First Sister Lake in connection with operation of the Parklake Well following treatment with ozone/hydrogen peroxide technology that has effluent limitations, discharge limits (other than volume), and other conditions no more restrictive than those included in Defendant's 2014 NPDES Permit.

3. Negotiation and execution of an access agreement between Defendant and the City of Ann Arbor providing reasonable and necessary access to the City-owned parcel at Parklake Avenue and Jackson Road with respect to installation and operation of an extraction well, operation and maintenance of a groundwater treatment unit, and disposal of treated groundwater.

4. An Air Permit for discharges of contaminants to the atmosphere for vapor extraction systems, including the HSVE system described in Subsection VI.C.4, under terms reasonably acceptable to Defendant and as necessary if such systems are part of the remedial design.

5. Wetlands Permit(s) from EGLE and/or Scio Township if necessary for the

response activities described in Section VI.C.3 with terms reasonably acceptable to Defendant.

6. An Industrial User's Permit to be issued by the City of Ann Arbor for use of the sewer to dispose of treated or untreated purged groundwater from the Evergreen and/or Maple Road Wells. Plaintiffs have no objection to receipt by the Ann Arbor Wastewater Treatment Plant of the purged groundwater extracted pursuant to the terms and conditions of this Consent Judgment, and acknowledge that receipt of the purged groundwater would not necessitate any change in current and proposed residual management programs of the Ann Arbor Wastewater Treatment Plant.

7. Permit(s) or permit exemptions to be issued by EGLE to authorize the reinjection of purged and treated groundwater in the Eastern Area and Western Area.

8. Surface water discharge permit(s) for discharge into surface waters in the area of Little Lake, if necessary.

9. Approval of the City of Ann Arbor and the Washtenaw County Drain Commissioner to use storm drains or sewers for the remedial programs.

10. Washtenaw County permits as necessary for the installation of extraction wells, monitoring wells, and borings.

VIII. SAMPLING AND ANALYSIS

Defendant shall make available to EGLE the results of all sampling, tests, and/or other data generated in the performance or monitoring of any requirement under this Consent Judgment. Sampling data generated consistent with this Consent Judgment shall be admissible in evidence in any proceeding related to enforcement of this Consent Judgment without waiver by any Party of any objection as to weight or relevance. EGLE and/or their authorized representatives, at their discretion, may take split or duplicate samples and observe the sampling event. EGLE shall make available to Defendant the results of all sampling, tests, and/or other data generated in the performance or monitoring of any requirement under this Consent Judgment. Defendant will provide EGLE with reasonable notice of changes in the schedule of data collection activities included in the progress reports submitted pursuant to Section XII.

IX. ACCESS

A. From the effective date of this Consent Judgment, EGLE, its authorized employees, agents, representatives, contractors, and consultants, upon presentation of proper identification, shall have the right at all reasonable times to enter the Site and any property to which access is required for the implementation of this Consent Judgment, to the extent access to the property is owned, controlled by, or available to the Defendant, for the purpose of conducting any activity authorized by this Consent Judgment, including, but not limited to:

1. Monitoring of the Remedial Action or any other activities taking place pursuant to this Consent Judgment on the property;
2. Verification of any data or information submitted to EGLE;
3. Conduct of investigations related to 1,4-dioxane concentrations at the Site;
4. Collection of samples;
5. Assessment of the need for, or planning and implementing of, Response Activities at the Site; and
6. Inspection and copying of non-privileged documents including records, operating logs, contracts, or other documents required to assess Defendant's compliance with this Consent Judgment.

All Parties with access to the Site or other property pursuant to this Section shall comply with all applicable health and safety laws and regulations.

B. To the extent that the Site or any other area where Remedial Action is to be performed by the Defendant under this Consent Judgment is owned or controlled by persons other than the Defendant, Defendant shall use its best efforts to secure from such persons access for Defendant, EGLE, and their authorized employees, agents, representatives, contractors, and consultants. Defendant shall provide EGLE with a copy of each access agreement secured pursuant to this Section. For purposes of this Section, "best efforts" includes, but is not limited to, seeking judicial assistance to secure such access pursuant to MCL 324.20135a.

X. APPROVALS OF SUBMISSIONS

Upon receipt of any plan, report, or other item that is required to be submitted for approval pursuant to this Consent Judgment, as soon as practicable, but in no event later than 56 days after receipt of such submission, EGLE will: (1) approve the submission or (2) submit to Defendant changes in the submission that would result in approval of the submission. EGLE will (1) approve a feasibility study or plan that proposes a risk based cleanup or a remedy that requires public comment, or (2) submit to Defendant changes in such submittal that would result in approval in the time provided under Part 201. If EGLE does not respond within 56 days, Defendant may submit the matter to dispute resolution pursuant to Section XVI. Upon receipt of a notice of approval or changes from EGLE, Defendant shall proceed to take any action required by the plan, report, or other item, as approved or as may be modified to address the deficiencies identified by EGLE. If Defendant does not accept the changes proposed by EGLE, Defendant may submit the matter to dispute resolution pursuant to Section XVI.

XI. PROJECT COORDINATORS

A. Plaintiffs designate Daniel Hamel as EGLE's Project Coordinator. Defendant designates Lawrence Gelb as Defendant's Project Coordinator. Defendant's Project Coordinator shall have primary responsibility for implementation of the Remedial Action at the Site. EGLE's Project Coordinator will be the primary designated representative for Plaintiffs with respect to implementation of the Remedial Action at the Site. All communication between Defendant and EGLE, including all documents, reports, approvals, other submissions, and correspondence concerning the activities performed pursuant to the terms and conditions of this Consent Judgment, shall be directed through the Project Coordinators. If any Party changes its designated Project Coordinator, that Party shall provide the name, address, email address and telephone number of the successor in writing to the other Party seven days prior to the date on which the change is to be effective. This Section does not relieve Defendant from other reporting obligations under the law.

B. EGLE may designate other authorized representatives, employees, contractors, and consultants to observe and monitor the progress of any activity undertaken pursuant to this Consent Judgment. EGLE's Project Coordinator shall provide Defendant's Project Coordinator with the names, addresses, telephone numbers, positions, and responsibilities of any person designated pursuant to this Section.

XII. PROGRESS REPORTS

Defendant shall provide to EGLE written quarterly progress reports that shall: (1) describe the actions which have been taken toward achieving compliance with this Consent Judgment during the previous three months; (2) describe data collection and activities scheduled for the next three months; and (3) include all results of sampling and tests and other data received by Defendant, its consultants, engineers, or agents during the previous three months relating to Remedial Action performed pursuant to this Consent Judgment. Defendant shall submit the first quarterly report to EGLE within 120 days after entry of this Consent Judgment, and by the 30th day of the month following each quarterly period thereafter, as feasible, until termination of this Consent Judgment as provided in Section XXV.

XIII. RESTRICTIONS ON ALIENATION

A. Defendant shall not sell, lease, or alienate the Gelman Property until: (1) it places an EGLE-approved land use or resource use restrictions on the affected portion(s) of the Gelman Property; and (2) any purchaser, lessee, or grantee provides to EGLE its written agreement providing that the purchaser, lessee, or grantee will not interfere with any term or condition of this Consent Judgment. Notwithstanding any purchase, lease, or grant, Defendant shall remain obligated to comply with all terms and conditions of this Consent Judgment.

B. Any deed, title, or other instrument of conveyance regarding the Gelman Property shall contain a notice that Defendant's Property is the subject of this Consent Judgment, setting forth the caption of the case, the case number, and the court having jurisdiction herein.

XIV. FORCE MAJEURE

Any delay attributable to a Force Majeure shall not be deemed a violation of Defendant's obligations under this Consent Judgment.

A. "Force Majeure" is defined as an occurrence or nonoccurrence arising from causes beyond the control of Defendant or of any entity controlled by the Defendant performing Remedial Action, such as Defendant's employees, contractors, and subcontractors. Such occurrence or nonoccurrence includes, but is not limited to: (1) an Act of God; (2) untimely review of permit applications or submissions; (3) acts or omissions of third parties for which Defendant is not responsible; (4) insolvency of any vendor, contractor, or subcontractor retained by Defendant as part of implementation of this Consent Judgment; and (5) delay in obtaining necessary access agreements under Section IX that could not have been avoided or overcome by due diligence. "Force Majeure" does not include unanticipated or increased costs, changed financial circumstances, or nonattainment of the treatment and termination standards set forth in Sections V and VI.

B. When circumstances occur that Defendant believes constitute Force Majeure, Defendant shall notify EGLE by telephone of the circumstances within 48 hours after Defendant first believes those circumstances to apply. Within 14 working days after Defendant first believes those circumstances to apply, Defendant shall supply to EGLE, in writing, an explanation of the cause(s) of any actual or expected delay, the anticipated duration of the delay, the measures taken and the measures to be taken by Defendant to avoid, minimize, or overcome the delay, and the timetable for implementation of such measures. Failure of Defendant to comply with the written notice provisions of this Section shall constitute a waiver of Defendant's right to assert a claim of Force Majeure with respect to the circumstances in question.

C. A determination by EGLE that an event does not constitute Force Majeure, that a delay was not caused by Force Majeure, or that the period of delay was not necessary to compensate for Force Majeure may be subject to dispute resolution under Section XVI of this Consent Judgment.

D. EGLE shall respond, in writing, to any request by Defendant for a Force Majeure extension within 30 days of receipt of the Defendant's request. If EGLE does not respond within that time period, Defendant's request shall be deemed granted. If EGLE agrees that a delay is or was caused by Force Majeure, Defendant's delays shall be excused, stipulated penalties shall not accrue, and EGLE shall provide Defendant such additional time as may be necessary to compensate for the Force Majeure event.

E. Delay in achievement of any obligation established by this Consent Judgment shall not automatically justify or excuse delay in achievement of any subsequent obligation unless the subsequent obligation automatically follows from the delayed obligation.

XV. REVOCATION OR MODIFICATION OF LICENSES OR PERMITS

Any delay attributable to the revocation or modification of licenses or permits obtained by Defendant to implement remediation actions as set forth in this Consent Judgment shall not be deemed a violation of Defendant's obligations under this Consent Judgment, provided that such revocation or modification arises from causes beyond the control of Defendant or of any entity controlled by the Defendant performing Remedial Action, such as Defendant's employees, contractors, and subcontractors.

A. Licenses or permits that may need to be obtained or modified by Defendant to implement the Remedial Actions are those specified in Section VII.D. and licenses, easements, and other agreements for access to property or rights of way on property necessary for the installation of remedial systems required by this Consent Judgment.

B. A revocation or modification of a license or permit within the meaning of this Section means withdrawal of permission, denial of permission, a limitation or a change in license or permit conditions that delays the implementation of all or part of a remedial system. Revocation or modification due to Defendant's violation of a license or permit (or any conditions of a license or permit) shall not constitute a revocation or modification covered by this Section.

C. When circumstances occur that Defendant believes constitute revocation or modification of a license or permit, Defendant shall notify EGLE by telephone of the circumstances within 48 hours after Defendant first believes those circumstances to apply.

Within 14 working days after Defendant first believes those circumstances to apply, Defendant shall supply to EGLE, in writing, an explanation of the cause(s) of any actual or expected delay, the anticipated duration of the delay, the measures taken and the measures to be taken by Defendant to avoid, minimize, or overcome the delay, and the timetable for implementation of such measures. Failure of Defendant to comply with the written notice provisions of this Section shall constitute a waiver of Defendant's right to assert a claim of revocation or modification of a license or permit with respect to the circumstances in question.

D. A determination by EGLE that an event does not constitute revocation or modification of a license or permit, that a delay was not caused by revocation or modification of a license or permit, or that the period of delay was not necessary to compensate for revocation or modification of a license or permit may be subject to dispute resolution under Section XVI of this Consent Judgment.

E. EGLE shall respond, in writing, to any request by Defendant for a revocation or modification of a license or permit extension within 30 days of receipt of the Defendant's request. If EGLE does not respond within that time period, Defendant's request shall be deemed granted. If EGLE agrees that a delay is or was caused by revocation or modification of a license or permit, Defendant's delays shall be excused, stipulated penalties shall not accrue, and EGLE shall provide Defendant such additional time as may be necessary to compensate for the revocation or modification of a license or permit.

F. Delay in achievement of any obligation established by this Consent Judgment shall not automatically justify or excuse delay in achievement of any subsequent obligation unless the subsequent obligation automatically follows from the delayed obligation.

XVI. DISPUTE RESOLUTION

A. The dispute resolution procedures of this Section shall be the exclusive mechanism to resolve disputes arising under this Consent Judgment and shall apply to all provisions of this Consent Judgment except for disputes related to Prohibition Zone boundary modification under Sections V.A.2.f and V.A.6, whether or not particular provisions of this Consent Judgment in question make reference to the dispute resolution provisions of this Section. Any dispute that arises under this Consent Judgment initially shall be the subject of informal negotiations between the Parties. The period of negotiations shall not exceed ten working days from the date of written notice by EGLE or the Defendant that a dispute has arisen. This period may be extended or shortened by agreement of EGLE or the Defendant.

B. Immediately upon expiration of the informal negotiation period (or sooner if upon agreement of the parties), EGLE shall provide to Defendant a written statement setting forth EGLE’s proposed resolution of the dispute. Such resolution shall be final unless, within 15 days after receipt of EGLE’s proposed resolution (clearly identified as such under this Section), Defendant files a petition for resolution with the Washtenaw County Circuit Court setting forth the matter in dispute, the efforts made by the Parties to resolve it, the relief requested, and the schedule, if any, within which the dispute must be resolved to ensure orderly implementation of this Consent Judgment.

C. Within ten days of the filing of the petition, EGLE may file a response to the petition, and unless a dispute arises from the alleged failure of EGLE to timely make a decision, EGLE will submit to the Court all documents containing information related to the matters in dispute, including documents provided to EGLE by Defendant. In the event of a dispute arising from the alleged failure of EGLE to timely make a decision, within ten days of filing of the petition, each party shall submit to the Court correspondence, reports, affidavits, maps, diagrams, and other documents setting forth facts pertaining to the matters in dispute. Those documents and this Consent Judgment shall comprise the record upon which the Court shall resolve the dispute. Additional evidence may be taken by the Court on its own motion or at the request of either party if the Court finds that the record is incomplete or inadequate. Review of the petition shall be conducted by the Court and shall be confined to the record. The review shall be independent of any factual or legal conclusions made by the Court prior to the date of entry of this Consent Judgment.

D. The Court shall uphold the decision of EGLE on the issue in dispute unless the Court determines that the decision is any of the following:

1. Inconsistent with this Consent Judgment;
2. Not supported by competent, material, and substantial evidence on the whole record;
3. Arbitrary, capricious, or clearly an abuse or unwarranted exercise of discretion; or
4. Affected by other substantial and material error of law.

E. The filing of a petition for resolution of a dispute shall not by itself extend or postpone any obligation of Defendant under this Consent Judgment, provided, however, that payment of stipulated penalties with respect to the disputed matter shall be stayed pending resolution of the dispute. Notwithstanding the stay of payment, stipulated penalties shall accrue as provided in Section XVII. Stipulated penalties that have accrued with respect to the matter in dispute shall not be assessed by the Court and shall be dissolved if Defendant prevails on the matter. The Court may also direct that stipulated penalties shall not be assessed and paid as provided in Section XVII upon a determination that there was a substantial basis for Defendant's position on the disputed matter.

XVII. STIPULATED PENALTIES

A. Except as otherwise provided, if Defendant fails or refuses to comply with any term or condition in Sections IV, V, VI, VII, or VIII, or with any plan, requirement, or schedule established pursuant to those Sections, then Defendant shall pay stipulated penalties in the following amounts for each working day for every failure or refusal to comply or conform:

Period of Delay	Penalty Per Violation Per Day
1st through 15th Day	$ 1,000
15th through 30th Day	$ 1,500
Beyond 30 Days	$ 2,000

B. Except as otherwise provided if Defendant fails or refuses to comply with any other term or condition of this Consent Judgment, Defendant shall pay to EGLE stipulated penalties of $500.00 per working day for each and every failure to comply.

C. If Defendant is in violation of this Consent Judgment, Defendant shall notify EGLE of any violation no later than five working days after first becoming aware of such violation, and shall describe the violation.

D. Stipulated penalties shall begin to accrue upon the next day after performance was due or other failure or refusal to comply occurred. Penalties shall continue to accrue until the final day of correction of the noncompliance. Separate penalties shall accrue for each separate failure or refusal to comply with the terms and conditions of this Consent Judgment. Penalties may be waived in whole or in part by EGLE or may be dissolved by the Court pursuant to Section XVII.

E. Stipulated penalties shall be paid no later than 14 working days after receipt by

Defendant of a written demand from EGLE. Defendant shall make payment by transmitting a check in the amount due, payable to the "State of Michigan," addressed to the Revenue Control Unit; Finance Section, Administration Division; Michigan Department of Environment, Great Lakes, and Energy; P.O. Box 30657; Lansing, MI 48909-8157. The check shall be transmitted via Courier to the Revenue Control Unit; Finance Section, Administration Division; Michigan Department of Environment, Great Lakes, and Energy; Constitution Hall, 5th Floor South Tower; 525 West Allegan Street; Lansing, MI 48933-2125. To ensure proper credit, Defendant shall include the settlement ID - ERD1902 on the payment.

F. Plaintiffs agree that, in the event that an act or omission of Defendant constitutes a violation of this Consent Judgment subject to stipulated penalties and a violation of other applicable law, Plaintiffs will not impose upon Defendant for that violation both the stipulated penalties provided under this Consent Judgment and the civil penalties permitted under other applicable laws. EGLE reserves the right to pursue any other remedy or remedies to which they may be entitled under this Consent Judgment or any applicable law for any failure or refusal of the Defendant to comply with the requirements of this Consent Judgment.

XVIII. PLAINTIFFS' COVENANT NOT TO SUE AND RESERVATION OF RIGHTS

A. Except as otherwise provided in this Consent Judgment, Plaintiffs covenant not to sue or take administrative action for Covered Matters against Defendant, its officers, employees, agents, directors, and any persons acting on its behalf or under its control.

B. "Covered Matters" shall mean any and all claims available to Plaintiffs under federal and state law arising out of the subject matter of the Plaintiffs' Complaint with respect to the following:

1. Claims for injunctive relief to address soil, groundwater, and surface water

contamination at or emanating from the Gelman Property;

2. Claims for civil penalties and costs;

3. Claims for natural resource damages;

4. Claims for reimbursement of response costs incurred prior to entry of this Consent Judgment or incurred by Plaintiffs for provision of alternative water supplies in the Evergreen Subdivision; and

5. Claims for reimbursement of costs incurred by Plaintiffs for overseeing the implementation of this Consent Judgment.

C. "Covered Matters" does not include:

1. Claims based upon a failure by Defendant to comply with the requirements of this Consent Judgment;

2. Liability for violations of federal or state law which occur during implementation of the Remedial Action; and

3. Liability arising from the disposal, treatment, or handling of any hazardous substance removed from the Site.

D. With respect to liability for alleged past violations of law, this covenant not to sue shall take effect on the effective date of this Consent Judgment. With respect to future liability for performance of response activities required to be performed under this Consent Judgment, the covenant not to sue shall take effect upon issuance by EGLE of the Certificate of Completion in accordance with Section XXV.

E. Notwithstanding any other provision in this Consent Judgment: (1) EGLE reserves the right to institute proceedings in this action or in a new action seeking to require Defendant to perform any additional response activity at the Site; and (2) EGLE reserves the right to institute proceedings in this action or in a new action seeking to reimburse EGLE for response costs incurred by the State of Michigan relating to the Site. EGLE's rights in Sections XVIII.E.1 and E.2 apply if the following conditions are met:

1. For proceedings prior to EGLE's certification of completion of the Remedial Action concerning the Site,

a. (i) conditions at the Site, previously unknown to EGLE, are discovered after entry of this Consent Judgment, (ii) new information previously unknown to EGLE is received after entry of this Consent Judgment, or (iii) EGLE adopts one or more new, more restrictive cleanup criteria for 1,4-dioxane pursuant to Part 201 after entry of this Consent Judgment; and

b. these previously unknown conditions, new information, and/or change in criteria indicate that the Remedial Action is not protective of the public health, safety, welfare, and the environment; and

2. For proceedings subsequent to EGLE's certification of completion of the Remedial Action concerning the Site,

a. (i) conditions at the Site, previously unknown to EGLE, are discovered after certification of completion by EGLE, (ii) new information previously unknown to EGLE is received after certification of completion by EGLE, or (iii) EGLE adopts one or more new, more restrictive cleanup criteria for 1,4-dioxane pursuant to Part 201, after certification of completion by EGLE; and

b. these previously unknown conditions, new information, and/or change in criteria indicate that the Remedial Action is not protective of the public health, safety, welfare, and the environment.

If EGLE adopts one or more new, more restrictive, cleanup criteria, EGLE's rights in Sections XVIII.E.1 and E.2 shall also be subject to Defendant's right to seek another site-specific criterion(ia) that is protective of public health, safety, welfare, and the environment and/or to argue that EGLE has not made the demonstration(s) required under this Section.

F. Nothing in this Consent Judgment shall in any manner restrict or limit the nature or scope of Response Activities that may be taken by EGLE in fulfilling its responsibilities under federal and state law, and this Consent Judgment does not release, waive, limit, or impair in any manner the claims, rights, remedies, or defenses of EGLE against a person or entity not a party to this Consent Judgment.

G. Except as expressly provided in this Consent Judgment, EGLE reserves all other rights and defenses that they may have, and this Consent Judgment is without prejudice, and shall not be construed to waive, estop, or otherwise diminish EGLE's right to seek other relief with respect to all matters other than Covered Matters.

XIX. DEFENDANT'S COVENANT NOT TO SUE AND RESERVATION OF RIGHTS

A. Defendant hereby covenants not to sue and agrees not to assert any claim or cause of action against EGLE or any other agency of the State of Michigan with respect to environmental contamination at the Site or response activities relating to the Site arising from this Consent Judgment.

B. Notwithstanding any other provision in this Consent Judgment, for matters that are not Covered Matters as defined in Section XVIII.B, or in the event that Plaintiffs institute proceedings as allowed under Section XVIII.E., Defendant reserves all other rights, defenses, or counterclaims that it may have with respect to such matters and this Consent Judgment is without prejudice, and shall not be construed to waive, estop, or otherwise diminish Defendant's right to seek other relief and to assert any other rights and defenses with respect to such other matters.

C. Nothing in this Consent Judgment shall in any way impair Defendant's rights, claims, or defenses with respect to any person not a party to this Consent Judgment.

XX. INDEMNIFICATION, INSURANCE, AND FINANCIAL ASSURANCE

A. Defendant shall indemnify and save and hold harmless the State of Michigan and its departments, agencies, officials, agents, employees, contractors, and representatives from any and all claims or causes of action arising from, or on account of, acts or omissions of Defendant, its officers, employees, agents, and any persons acting on its behalf or under its control in carrying out Remedial Action pursuant to this Consent Judgment. EGLE shall not be held out as a party to any contract entered into by or on behalf of Defendant in carrying out activities pursuant to this Consent Judgment. Neither the Defendant nor any contractor shall be considered an agent of EGLE. Defendant shall not indemnify or save and hold harmless Plaintiffs from their own negligence pursuant to this Section.

B. Prior to commencing any Remedial Action on the Gelman Property, Defendant shall secure, and shall maintain for the duration of the Remedial Action, comprehensive general liability insurance with limits of $1,000,000.00, combined single limit, naming as an additional insured the State of Michigan. If Defendant demonstrates by evidence satisfactory to EGLE that any contractor or subcontractor maintains insurance equivalent to that described above, or insurance covering the same risks but in a lesser amount, then with respect to that contractor or subcontractor, Defendant need provide only that portion, if any, of the insurance described above that is not maintained by the contractor or subcontractor.

C. Financial Assurance

1. Defendant shall be responsible for providing and maintaining financial assurance in a mechanism approved by EGLE in an amount sufficient to cover the estimated cost to assure performance of the response activities required to meet the remedial objectives of this Consent Judgment including, but not limited to, investigation, monitoring, operation and maintenance, and other costs (collectively referred to as “Long-Term Remedial Action Costs”). Defendant shall continuously maintain a financial assurance mechanism (“FAM”) until EGLE’s Remediation and Redevelopment Division (“RRD”) Chief or his or her authorized representative notifies it in writing that it is no longer required to maintain a FAM.

2. The Letter of Credit provided in Attachment K is the initial FAM approved by EGLE. Defendant shall be responsible for providing and maintaining financial assurance in a mechanism acceptable to EGLE to assure the performance of the Long Term Remedial Action Costs required by Defendant’s selected remedial action.

3. The FAM shall remain in an amount sufficient to cover Long Term Remedial Action Costs for a 30-year period. Unless Defendant opts to use and satisfies the Financial Test or Financial Test/Corporate Guarantee as provided in Section XX.C.8, the FAM shall remain in a form that allows EGLE to immediately contract for the response activities for which financial assurance is required in the event Defendant fails to implement the required tasks, subject to Defendant’s rights under Sections XIV and XVI.

4. Within 120 days of the Effective Date of this Fourth Amended Consent Judgment, Defendant shall provide EGLE with an estimate of the amount of funds necessary to assure Long Term Remedial Action Costs for the following 30-year period based upon an annual estimate of costs for the response activities required by this Fourth Amended Consent Judgment

as if they were to be conducted by a person under contract to EGLE (the "Updated Long Term Remedial Action Cost Estimate"). The Updated Long Term Remedial Action Cost Estimate shall include all assumptions and calculations used in preparing the cost estimate and shall be signed by an authorized representative of Defendant who shall confirm the validity of the data. Defendant may only use a present worth analysis if an interest accruing FAM is selected. Within 60 days after Defendant's submittal of the Updated Long Term Remedial Action Cost Estimate, Defendant shall capitalize or revise the FAM in a manner acceptable to EGLE to address Long Term Remedial Action Costs unless otherwise notified by EGLE. If EGLE disagrees with the conclusions of the Updated Long Term Remedial Action Cost Estimate, Defendant shall capitalize the FAM to a level acceptable to EGLE within 30 days of EGLE notification, subject to Dispute Resolution under Section XVI.

5. Sixty days prior to the 5-year anniversary of the Effective Date of this Fourth Amended Consent Judgment and each subsequent 5-year anniversary, Defendant shall provide to EGLE a report containing the actual Long Term Remedial Action Costs for the previous 5-year period and an estimate of the amount of funds necessary to assure Long Term Remedial Action Costs for the following 30-year period given the financial trends in existence at the time of preparation of the report ("Long Term Remedial Action Cost Report"). The cost estimate shall be based upon an annual estimate of maximum costs for the response activities required by this Fourth Amended Consent Judgment as if they were to be conducted by a person under contract to EGLE, provided that, if Defendant is using the Financial Test or Corporate Guarantee/Financial Test under Section XX.C.8, below, Defendant may use an estimate on its internal costs to satisfy the Financial Test. The Long Term Remedial Action Cost Report shall also include all assumptions and calculations used in preparing the necessary cost estimate and

shall be signed by an authorized representative of Defendant who shall confirm the validity of the data. Defendant may only use a present worth analysis if an interest accruing FAM is selected.

6. Within 60 days after Defendant's submittal of the Long Term Remedial Action Cost Report to EGLE, Defendant shall capitalize or revise the FAM in a manner acceptable to EGLE to address Long Term Remedial Action Costs consistent with the conclusions of the Long Term Remedial Action Cost Report unless otherwise notified by EGLE. If EGLE disagrees with the conclusions of the Long Term Remedial Action Cost Report, Defendant shall capitalize the FAM to a level acceptable to EGLE within 30 days of EGLE notification, subject to dispute resolution under Section XVI. If, at any time, EGLE determines that the FAM does not secure sufficient funds to address Long Term Remedial Action Costs, Defendant shall capitalize the FAM or provide an alternate FAM to secure any additional costs within 30 days of request by EGLE, subject to dispute resolution under Section XVI.

7. If, pursuant to the Long Term Remedial Action Cost Report, Defendant can demonstrate that the FAM provides funds in excess of those needed for Long Term Remedial Action Costs, Defendant may request a modification in the amount. Any requested FAM modifications must be accompanied by a demonstration that the proposed FAM provides adequate funds to address future Long Term Remedial Action Costs. Upon EGLE approval of the request, Defendant may modify the FAM as approved by EGLE. Modifications to the FAM pursuant to this Section shall be approved by EGLE RRD Chief or his or her authorized representative, subject to dispute resolution under Section XVI.

8. If Defendant chooses to use the Financial Test or Corporate Guarantee/Financial Test attached as Attachment L (hereinafter, the term "Financial Test" refers

to both an independent financial test or a financial test utilized in conjunction with a corporate guarantee), Defendant shall, within 90 days after the end of Defendant's next fiscal year and the end of each succeeding fiscal year, submit to EGLE the necessary forms and supporting documents to demonstrate to the satisfaction of EGLE that Defendant can continue to meet the Financial Test requirements. If Defendant can no longer meet the financial test requirements, Defendant shall submit a proposal for an alternate FAM to satisfy its financial obligations with respect to this Consent Judgment.

9. If the Financial Test is being used as the FAM, EGLE, based on a reasonable belief that Defendant may no longer meet the requirements for the Financial Test, may require reports of financial condition at any time from Defendant, and/or require Defendant to submit updated Financial Test information to determine whether it meets the Financial Test criteria. Defendant shall provide, with reasonable promptness to EGLE, any other data and information that may reasonably be expected to materially adversely affect Defendant's ability to meet the Financial Test requirements. If EGLE finds that Defendant no longer meets the Financial Test requirements, Defendant shall, within 30 days after notification from EGLE, submit a proposal for an alternate FAM to satisfy its financial obligations with respect to this Consent Judgment, subject to dispute resolution under Section XVI.

10. If the Financial Test/Corporate Guarantee is used as the FAM, Defendant shall comply with the terms of the Corporate Guarantee. The Corporate Guarantee shall remain in place until Long-Term Remedial Action Costs are no longer required or Defendant establishes an alternate FAM acceptable to EGLE.

11. If Defendant wishes to change the type of FAM or establish a new FAM, Defendant shall submit a request to EGLE for approval. Upon EGLE approval of the request,

Defendant may change the type of FAM or establish the new FAM as approved by EGLE. Modifications to the FAM pursuant to this Section shall be approved by EGLE RRD Chief or his or her authorized representative, subject to dispute resolution under Section XVI.

12. If Defendant dissolves or otherwise ceases to conduct business and fails to make arrangements acceptable to EGLE for the continued implementation of all activities required by this Consent Judgment, all rights under this Consent Judgment regarding the FAM shall immediately and automatically vest in EGLE in accordance with the FAM.

XXI. RECORD RETENTION

Defendant, Plaintiffs, and their representatives, consultants, and contractors shall preserve and retain, during the pendency of this Consent Judgment and for a period of ten years after its termination, all records, sampling or test results, charts, and other documents that are maintained or generated pursuant to any requirement of this Consent Judgment, including, but not limited to, documents reflecting the results of any sampling or tests or other data or information generated or acquired by Plaintiffs or Defendant, or on their behalf, with respect to the implementation of this Consent Judgment. After the ten-year period of document retention, the Defendant and its successors shall notify EGLE, in writing, at least 90 days prior to the destruction of such documents or records, and upon request, the Defendant and/or its successor shall relinquish custody of all records and documents to EGLE.

XXII. ACCESS TO INFORMATION

Upon request, EGLE and Defendant shall provide to each other copies of or access to all non-privileged documents and information within their possession and/or control or that of their employees, contractors, agents, or representatives, relating to activities at the Site or to the implementation of this Consent Judgment, including, but not limited to, sampling, analysis, chain of custody records, manifests, trucking logs, receipts, reports, sample traffic routing, correspondence, or other documents or information related to the Remedial Action. Upon request, Defendant shall also make available to EGLE, their employees, contractors, agents, or representatives with knowledge or relevant facts concerning the performance of the Remedial Action. The Plaintiffs shall treat as confidential all documents provided to Plaintiffs by the Defendant marked "confidential" or "proprietary."

XXIII. NOTICES

Whenever under the terms of this Consent Judgment notice is required to be given or a report, sampling data, analysis, or other document is required to be forwarded by one Party to the other, such notice or document shall be directed to the following individuals at the specified addresses or at such other address as may subsequently be designated in writing:

For Plaintiffs:

Daniel Hamel
Project Coordinator
Michigan Department
of Environment, Great
Lakes, and Energy,
Remediation and Redevelopment
Division
301 East Louis Glick Highway
Jackson, MI 49201

For Defendants:

Lawrence Gelb
Gelman Sciences Inc.
642 South Wagner Road
Ann Arbor, MI 48106

and

Michael L. Caldwell
Zausmer, P.C.
32255 Northwestern Hwy., Ste. 225
Farmington Hills, MI 48334

Any party may substitute for those designated to receive such notices by providing prior written notice to the other parties.

XXIV. MODIFICATION

This Consent Judgment may not be modified unless such modification is in writing, signed by the Plaintiffs and the Defendant, and approved and entered by the Court. Remedial Plans, work plans, or other submissions made pursuant to this Consent Judgment may be modified by mutual agreement of the Defendant and EGLE.

XXV. CERTIFICATION AND TERMINATION

A. When Defendant determines that it has completed all Remedial Action required by this Consent Judgment, Defendant shall submit to EGLE a Notification of Completion and a draft final report. The draft final report must summarize all Remedial Action performed under this Consent Judgment and the performance levels achieved. The draft final report shall include or refer to any supporting documentation.

B. Upon receipt of the Notification of Completion, EGLE will review the Notification of Completion and the accompanying draft final report, any supporting documentation, and the actual Remedial Action performed pursuant to this Consent Judgment. After conducting this review, and not later than three months after receipt of the Notification of Completion, EGLE shall issue a Certificate of Completion upon a determination by EGLE that Defendant has completed satisfactorily all requirements of this Consent Decree, including, but not limited to, completion of all Remedial Action, achievement of all termination and treatment standards required by this Consent Judgment, compliance with all terms and conditions of this Consent Judgment, and payment of any and all stipulated penalties owed to EGLE. If EGLE does not respond to the Notification of Completion within three months after receipt of the Notification of Completion, Defendant may submit the matter to dispute resolution pursuant to Section XVI. This Consent Judgment shall terminate upon motion and order of this Court after issuance of the Certificate of Completion. Upon issuance, the Certificate of Completion may be recorded.

XXVI. EFFECTIVE DATE

The effective date of this Consent Judgment shall be the date upon which this Consent Judgment is entered by the Court.

XXVII. SEVERABILITY

The provisions of this Consent Judgment shall be severable. Should any provision be declared by a court of competent jurisdiction to be inconsistent with federal or state law, and therefore unenforceable, the remaining provisions of this Consent Judgment shall remain in full force and effect.

XXVIII. SIGNATORIES

Each undersigned representatives of a Party to this Consent Judgment certifies that he or she is fully authorized by the Party to enter into this Consent Judgment and to legally bind such Party to the respective terms and conditions of this Consent Judgment.

ATTORNEY GENERAL, et al v GELMAN SCIENCES, INC.

(Washtenaw County Circuit Court No. 88-34734-CE)

ATTACHMENT A

GELMAN PROPERTY

Legal Information for H -08-26-100-001 (234 Nancy Dr.)

COM AT N 1/4 POST OF SEC, TH E 1446.11 FT IN N LINE OF SEC, TH S 1199 FT FOR PL OF BEG, TH S 1479.11 FT, TH W 113.7 FT IN E & W 1/4 LINE. TH N 1478.76 FT. TH E 113.7 FT TO PL OF BEG, BEING PART OF NE 1/4 SEC. 26 T2S R5E 3.86 AC.

Legal Information for H -08-26-100-002 (Jackson Plaza – vacant)

COM AT N 1/4 POST OF SEC, TH E 886.06 FT IN N LINE OF SEC, TH DEFL 91 DEG RIGHT 1199 FT FOR PL OF BEG, TH DEFL 91 DEG LEFT 446.36 FT, TH DEFL 91 DEG RIGHT 1478.76 FT, TH W 446.36 FT IN E & W 1/4 LINE, TH N 1477.34 FT TO PL OF BEG, BEING PART OF NE 1/4 SEC 26 T2S-R5E 15.14 AC.

Legal Information for H -08-26-100-020 (April Drive – vacant)

COM AT N 1/4 COR OF SEC 26, TH S 2-6-15 W 1102.76 FT TO POB TH N 69-0 E 71.74 FT, TH S 80-46 E 141.53 FT, TH S 60-22 E 215.47 FT, TH S 83-27 E 366.02 FT, TH S 58-36 E 141.63 FT, TH S 2-6-15 W 1371.36 FT, TH N 88-42-15 W 886.06 FT, TH N 2-6-15 E 1570.79 FT TO POB, PART NE 1/4 SEC 26 T2S R5E 30.43 AC

Legal Information for H -08-26-110-013 (Jackson Plaza – vacant)

BEG AT SE COR OF LOT 22, TH N 88-42-15 W 344.35 FT TH N 2-06-15 E 348.30 FT, TH N 87-24-40 E 463.07 FT TH 69.56 FT IN ARC OF CURVE LEFT, RADIUS 376.77 FT, CHORD S 55-32-20 E 69.47 FT, TH S 29-10-30 W 386.49 FT TO POB, BEING PART OF LOT 22 JACKSON PLAZA BUSINESS PARK

Legal Information for H -08-26-400-007 (S. Wagner – vacant)

COM AT E 1/4 COR OF SEC 26, TH S 2-8-15 W 976.97 FT TO POB, TH S 2-8-15 W 326.10 FT, TH N 88-42-15 W 1337.18 FT, TH N 2-8-30 E 326.10 FT, TH S 88-42-15 E 1337.16 FT TO POB, PART E 1/4 SEC 26 T2S R5E 10.01 AC

Legal Information for H -08-26-400-011 (602 S. Wagner)

COM AT E 1/4 COR SEC 26, T2S-R5E; TH N 88-14-19 W 571.00 FT TO POB; TH S 01-18-41 W 490.00 FT; TH N 88-41-19 W 773.65 FT; TH N 02-07-21 E 490.05 FT; TH S 88-41-19 E 766.72 FT TO POB. 8.664 AC. SPLIT ON 08/16/2007 FROM H -08-26-400-008 INTO H-08-26-400-011 & -012

Legal Information for H -08-26-400-012 (600 S. Wagner)

BEG AT E 1/4 COR SEC 26, T2S-R5E; TH S 02-08-15 W 976.65 FT; TH N 88-41-19 W 1337.46 FT; TH N 02-07-21 E 486.60 FT; TH S 88-41-19 E 773.65 FT; TH N 01-18-42 E 490.00 FT; TH S 88-41-19 E 571.00 FT TO POB. 21.43 AC. SPLIT ON 08/16/2007 FROM H -08-26-400-008, INTO H-08-26-400-011 & -012

Legal Information for H -08-26-400-013 (S. Wagner – vacant)

COM AT SE COR SEC 26, T2S, R5E; TH N 02-09-20 E 1144.49 FT TO POB; TH N 88-42-15 W 1219.79 FT; TH N 30-40-35 W 106.07 FT; TH N 88-42-15 W 60 FT; TH N 02-08-30 E 146.10 FT; TH S 88-42-15 E 1337.34 FT; TH S 02-09-20 W 236.09 FT TO POB. 7.19 AC. SPLIT ON 08/20/2007 FROM H -08-26-400-005, H -08-26-400-006; INTO CHILDREN H-08-26-400-013 & -014

ATTORNEY GENERAL, et al v GELMAN SCIENCES, INC.

(Washtenaw County Circuit Court No. 88-34734-CE)

ATTACHMENT B

EPA United States
Environmental Protection
Agency

Office of Water

www.epa.gov [illegible]

Method 1624, Revision C: Volatile Organic Compounds by Isotope Dilution GCMS

Method 1624

Revision C

Volatile Organic Compounds by Isotope Dilution GCMS

Method 1624

Volatile Organic Compounds by Isotope Dilution GCMS

1. SCOPE AND APPLICATION

1.1 This method is designed to meet the survey requirements of the USEPA ITD. The method is used to determine the volatile toxic organic pollutants associated with the Clean Water Act (as amended 1987); the Resource Conservation and Recovery Act (as amended in 1986); the Comprehensive Environmental Response, Compensation, and Liability Act (as amended in 1986); and other compounds amenable to purge and trap gas chromatography/mass spectrometry (GCMS).

1.2 The chemical compounds listed in Tables 1 and 2 may be determined in waters, soils, and municipal sludges by the method.

1.3 The detection limits of the method are usually dependent on the level of interferences rather than instrumental limitations. The levels in Table 3 typify the minimum quantities that can be detected with no interferences present.

1.4 The GCMS portions of the method are for use only by analysts experienced with GCMS or under the close supervision of such qualified persons. Laboratories unfamiliar with analysis of environmental samples by GCMS should run the performance tests in Reference 1 before beginning.

2. SUMMARY OF METHOD

2.1 The percent solids content of the sample is determined. If the solids content is known or determined to be less than 1%, stable isotopically labeled analogs of the compounds of interest are added to a 5-mL sample and the sample is purged with an inert gas at 20 to 25°C in a chamber designed for soil or water samples. If the solids content is greater than one, mL of reagent water and the labeled compounds are added to a 5-aliquot of sample and the mixture is purged at 40°C. Compounds that will not purge at 20 to 25°C or at 40°C are purged at 75 to 85°C (see Table 2). In the purging process, the volatile compounds are transferred from the aqueous phase into the gaseous phase where they are passed into a sorbent column and trapped. After purging is completed, the trap is backflushed and heated rapidly to desorb the compounds into a gas chromatograph (GC). The compounds are separated by the GC and detected by a mass spectrometer (MS) (References 2 and 3). The labeled compounds serve to correct the variability of the analytical technique.

2.2 Identification of a pollutant (qualitative analysis) is performed in one of three ways: (1) For compounds listed in Table 1 and other compounds for which authentic standards are available, the GCMS system is calibrated and the mass spectrum and retention time for each standard are stored in a user created library. A compound is identified when its retention time and mass spectrum agree with the library retention time and spectrum. (2) For compounds listed in Table 2 and other compounds for which standards are not available, a compound is identified when the retention time and mass spectrum agree with those specified in this method. (3) For chromatographic peaks which are not identified by (1) and (2) above, the background corrected spectrum at the peak maximum is compared with spectra in the EPA/NIH mass spectral file (Reference 4). Tentative identification is established when the spectrum agrees (see Section 12).

2.3 Quantitative analysis is performed in one of four ways by GCMS using extracted ion current profile (EICP) areas: (1) For compounds listed in Table 1 and other compounds for which standards and labeled analogs are available, the GCMS system iscalibrated and the compound concentration is determined using an isotope dilution technique. (2) For compounds listed in Table 1 and for other compounds for which authentic standards but no labeled compounds are available, the GCMS system is calibrated and the compound concentration is determined using an internal standard technique. (3) For compounds listed in Table 2 and other compounds for which standards are not available, compound concentrations are determined using known response factors. (4) For compounds for which neither standards nor known response factors are available, compound concentration is determined using the sum of the EICP areas relative to the sum of the EICP areas of the nearest eluted internal standard.

2.4 The quality of the analysis is assured through reproducible calibration and testing of the purge and trap and GCMS systems.

Table 1. Volatile Organic Compounds Determined by GCMS Using Isotope Dilution and Internal Standard Techniques

	Pollutant				Labeled Compound		
Compound	STORET	CAS Registry	EPA EGD	NPDES	Analog	CAS Registry	EPA EGD
Acetone	81552	67-64-1	516 V		d_6	666-52-4	616 V
Acrolein	34210	107-02-8	002 V	001 V	d_4	33984-05-3	202 V
Acrylonitrile	34215	107-13-1	003 V	002 V	d_3	53807-26-4	203 V
Benzene	34030	71-43-2	004 V	003 V	d_6	1076-43-3	204 V
Bromodichloromethane	32101	75-27-4	048 V	012 V	^{13}C	93952-10-4	248 V
Bromoform	32104	75-25-2	047 V	005 V	^{13}C	72802-81-4	247 V
Bromomethane	34413	74-83-9	046 V	020 V	d_3	1111-88-2	246 V
Carbon tetrachloride	32102	56-23-5	006 V	006 V	^{13}C	32488-50-9	206 V
Chlorobenzene	34301	108-90-7	007 V	007 V	d_5	3114-55-4	207 V
Chloroethane	34311	75-00-3	016 V	009 V	d_5	19199-91-8	216 V
2-Chloroethylvinyl ether	34576	110-75-8	019 V	010 V			
Chloroform	32106	67-66-3	023 V	011 V	^{13}C	31717-44-9	223 V
Chloromethane	34418	74-87-3	045 V	021 V	d_3	1111-89-3	245 V
Dibromochloromethane	32105	124-48-1	051 V	008 V	^{13}C	93951-99-6	251 V
1,1-Dichloroethane	34496	75-34-3	013 V	014 V	d_3	56912-77-7	213 V
1,2-Dichloroethane	32103	107-06-2	010 V	015 V	d_4	17070-07-0	210 V
1,1-Dichloroethene	34501	75-35-4	029 V	016 V	d_2	22280-73-5	229 V
trans-1,2-Dichlorethene	34546	156-60-5	030 V	026 V	d_3	42366-47-2	230 V
1,2-Dichloropropane	34541	78-87-5	032 V	017 V	d_6	93952-08-0	232 V
trans-1,3-Dichloropropene	34699	10061-02-6	033 V		d_4	93951-86-1	233 V
Diethyl ether	81576	60-29-7	515 V		d_{10}	2679-89-2	615 V
p-Dioxane	81582	123-91-1	527 V		d_8	17647-74-4	627 V
Ethylbenzene	34371	100-41-4	038 V	019 V	d_{10}	25837-05-2	238 V
Methylene chloride	34423	75-09-2	044 V	022 V	d_2	1665-00-5	244 V
Methyl ethyl ketone	81595	78-93-3	514 V		d_3	53389-26-7	614 V
1,1,2,2-Tetrachloroethane	34516	79-34-5	015 V	023 V	d_2	33685-54-0	215 V
Tetrachloroethene	34475	127-18-4	085 V	024 V	$^{13}C_2$	32488-49-6	285 V
Toluene	34010	108-88-3	086 V	025 V	d_8	2037-26-5	286 V
1,1,1-Trichloroethane	34506	71-55-6	011 V	027 V	d_3	2747-58-2	211 V
1,1,2-Trichloroethane	34511	79-00-5	014 V	028 V	$^{13}C_2$	93952-09-1	214 V
Trichloroethene	39180	79-01-6	087 V	029 V	$^{13}C_2$	93952-00-2	287 V
Vinyl chloride	39175	75-01-4	088 V	031 V	d_3	6745-35-3	288 V

Table 2. Volatile Organic Compounds to be Determined by Reverse Search and Quantitation Using Known Retention Times, Response Factors, Reference Compounds, and Mass Spectra

EGD No.	**Compound**	**CAS Registry**
532	Allyl alcohol [1]	107-18-6
533	Carbon disulfide	75-15-0
534	2-Chloro-1,3-butadiene (Chloroprene)	126-99-8
535	Chloroacetonitrile[1]	107-14-2
536	3-Chloropropene	107-05-1
537	Crotonaldehyde[1]	123-73-9
538	1,2-Dibromoethane (EDB)	106-93-3
539	Dibromomethane	74-95-3
540	trans-1,4-Dichloro-2-butene	110-57-6
541	1,3-Dichloropropane	142-28-9
542	cis-1,3-Dichloropropene	10061-01-5
543	Ethyl cyanide [1]	107-12-0
544	Ethyl methacrylate	97-63-2
545	2-Hexanone	591-78-6
546	Iodomethane	74-88-4
547	Isobutyl alcohol[1]	78-83-1
548	Methacrylonitrile	126-98-7
549	Methyl methacrylate	78-83-1
550	4-Methyl-2-pentanone	108-10-1
551	1,1,1,2-Tetrachloroethane	630-20-6
552	Trichlorofluoromethane	75-69-4
553	1,2,3-Trichloropropane	96-18-4
554	Vinyl acetate	108-05-4
951	*m*-Xylene	108-38-3
952	*o*- and *p*-Xylene	

[1] Determined at a purge temperature of 75–85°C.

3. CONTAMINATION AND INTERFERENCES

3.1 Impurities in the purge gas, organic compounds out-gassing from the plumbing upstream of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system is demonstrated to be free from interferences under conditions of the analysis by analyzing reagent water blanks initially and with each sample batch (samples analyzed on the same 8-hour shift), as described in Section 8.5.

3.2 Samples can be contaminated by diffusion of volatile organic compounds (particularly methylene chloride) through the bottle seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol may serve as a check on such contamination.

3.3 Contamination by carry-over can occur when high level and low level samples are analyzed sequentially. To reduce carry-over, the purging device (Figure 1 for samples containing less than one percent solids; Figure 2 for samples containing one percent solids or greater) is cleaned or replaced with a clean purging device after each sample is analyzed. When an unusually concentrated sample is encountered, it is followed by analysis of a reagent water blank to check for carry-over. Purging devices are cleaned by washing with soap solution, rinsing with tap and distilled water, and drying in an oven at 100 to 125°C. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

3.4 Interferences resulting from samples will vary considerably from source to source, depending on the diversity of the site being sampled.

Table 3. Gas Chromatography of Purgeable Organic Compounds

		Retention Time				Method Detection Limit [4]	
EGD No.[1]	Compound	Mean (sec)	EGD Ref	Relative [2]	Minimum Level [3] (µg/L)	Low Solids (µg/kg)	High Solids (µg/kg)
245	Chloromethane-d_3	147	181	0.141–0.270	50		
345	Chloromethane	148	245	0.922–1.210	50	207 [7]	13
246	Bromomenthane-d_3	243	181	0.233–0.423	50		
346	Bromomethane	246	246	0.898–1.195	50	148 [7]	11
288	Vinyl chloride-d_3	301	181	0.286–0.501	50		
388	Vinyl chloride	304	288	0.946–1.023	10	190 [7]	11
216	Chloroethane-d_5	378	181	0.373–0.620	50		
316	Chloroethane	386	216	0.999–1.060	50	789 [7]	24
244	Methylene chloride-d_2	512	181	0.582–0.813	10		
344	Methylene chloride	517	244	0.999–1.017	10	566 [7]	280 [7]
546	Iodomethane	498	181	0.68			
616	Acetone-d_6	554	181	0.628–0.889	50		
716	Acetone	565	616	0.984–1.019 [7]	50	3561	322 [7]
202	Acrolein-d_4	564	181	0.641–0.903	-- [5]	50	
302	Acrolein	566	202	0.984–1.018[5]	50	377 [7]	18
203	Acrylonitrile-d_3	606	181	0.735–0.926	50		
303	Acrylonitrile	612	203	0.985–1.030	50	360 [7]	9
533	Carbon disulfide	631	181	0.86			
552	Trichlorofluoromethane	663	181	0.91			
543	Ethyl cyanide	672	181	0.92			
229	1,1-Dichloroethene-d_2	696	181	0.903–0.976	10		
329	1,1-Dichloroethene	696	229	0.999–1.011	10	31	5
536	3-Chloropropene	696	181	0.95			
532	Allyl alcohol	703	181	0.96			
181	Bromochloromethane (I.S.)	730	181	1.000–1.000	10		
213	1,1-Dichloroethane-d_3	778	181	1.031–1.119	10		
313	1,1-Dichloroethane	786	213	0.999–1.014	10	16	1
615	Diethyl ether-d_{10}	804	181	1.067–1.254	50		
715	Diethyl ether	820	615	1.010–1.048	50	63	12
230	trans-1,2-Dichloroethene-d_2	821	181	1.056–1.228	10		
330	trans-1,2-Dichloroethene	821	230	0.996–1.011	10	41	3
614	Methyl ethyl ketone-d_3	840	181	0.646–1.202	50		
714	Methyl ethyl ketone	848	614	0.992–1.055	50	241 [7]	80 [7]
223	Chloroform-$^{13}C_1$	861	181	1.092–1.322	10		
323	Chloroform	861	223	0.961–1.009	10	21	2

EGD No.[1]	Compound	Retention Time			Minimum Level [3] (µg/L)	Method Detection Limit [4]	
		Mean (sec)	EGD Ref	Relative [2]		Low Solids (µg/kg)	High Solids (µg/kg)
535	Chloroacetonitrile	884	181	1.21			
210	1,2-Dichloroethane-d_4	901	181	1.187–1.416	10		
310	1,2-Dichloroethane	910	210	0.973–1.032	10	23	3
539	Dibromomethane	910	181	1.25			
548	Methacrylonitrile	921	181	1.26			
547	Isobutyl alcohol	962	181	1.32			
211	1,1,1-Trichloroethane-$^{13}C_2$	989	181	1.293–1.598	10		
311	1,1,1-Trichloroethane	999	211	0.989–1.044	10	16	4
627	*p*-Dioxane-d_8	982	181	1.262–1.448[5]	50		
727	*p*-Dioxane	1001	627	1.008–1.040[5]	50	--	140 [7]
206	Carbon tetrachloride-$^{13}C_2$	1018	182	0.754–0.805	10		
306	Carbon tetrachloride	1018	206	0.938–1.005	10	87	9
554	Vinyl acetate	1031	182	0.79			
248	Bromodichloromethane-$^{13}C_1$	1045	182	0.766–0.825	10		
348	Bromodichloromethane	1045	248	0.978–1.013	10	28	3
534	2-Chloro-1,3-butadiene	1084	182	0.83			
537	Crotonaldehyde	1098	182	0.84			
232	1,2-Dichloropropane-d_6	1123	182	0.830–0.880	10		
332	1,2-Dichloropropane	1134	232	0.984–1.018	10	29	5
542	cis-1,3-Dichloropropene	1138	182	0.87			
287	Trichloroethene-$^{13}C_2$	1172	182	0.897–0.917	10		
387	Trichloroethene	1187	287	0.991–1.037	10	41	2
541	1,3-Dichloropropane	1196	182	0.92			
204	Benezene-d_6	1200	182	0.888–0.952	10		
304	Benezene	1212	204	1.002–1.026	10	23	8
251	Chlorodibromomethane-$^{13}C_1$	1222	182	0.915–0.949	10		
351	Chlorodibromomethane	1222	231	0.989–1.030	10	15	2
214	1,1,2-Trichloroethane-$^{13}C_2$	1224	182	0.922–0.953	10		
314	1,1,2-Trichloroethane	1224	214	0.975–1.027	10	26	1
233	trans-1,3-Dichloropropene-d_4	1226	182	0.922–0.959	10		
333	trans-1,3-Dichloropropene	1226	233	0.993–1.016	10	--[6,7]	--[6,7]
019	2-Chloroethyvinyl ether	1278	182	0.983–1.026	10	122	21
538	1,2-Dibromoethane	1279	182	0.98			
182	2-bromo-1-chloropropane (I.S.)	1306	182	1.000–1.000	10		

		Retention Time				Method Detection Limit [4]	
EGD No.[1]	Compound	Mean (sec)	EGD Ref	Relative [2]	Minimum Level [3] (µg/L)	Low Solids (µg/kg)	High Solids (µg/kg)
549	Methyl methacrylate	1379	182	1.06			
247	Bromoform-$^{13}C_1$	1386	182	1.048–1.087	10		
347	Bromoform	1386	247	0.992–1.003	10	91	7
551	1,1,1,2-Tetrachloroethane	1408	182	1.08			
550	4-Methyl-2-pentanone	1435	183	0.92			
553	1,2,3-Trichloropropane	1520	183	0.98			
215	1,1,2,2-Tetrachloroethane-d_2	1525	183	0.969–0.996	10		
315	1,1,2,2-Tetrachloroethane	1525	215	0.890–1.016	10	20	6
545	2-Hexanone	1525	183	0.98			
285	Tetrachloroethene-$^{13}C_2$	1528	183	0.966–0.996	10		
385	Tetrachloroethene	1528	285	0.997–1.003	10	106	10
540	trans-1,4-Dichloro-2-butene	1551	183	1.00			
183	1,4-Dichlorobutane (int std)	1555	183	1.000–1.000	10		
544	Ethyl methacrylate	1594	183	1.03			
286	Toluene-d_8	1603	183	1.016–1.054	10		
386	Toluene	1619	286	1.001–1.019	10	27	4
207	Chlorobenzene-d_5	1679	183	1.066–1.135	10		
307	Chlorobenzene	1679	207	0.914–1.019	10	21	58 [7]
238	Ethylbenzene-d_{10}	1802	183	1.144–1.293	10		
338	Ethylbenzene	1820	238	0.981–1.018	10	28	4
185	Bromofluorobenzene	1985	183	1.255–1.290	10		
951	*m*-Xylene	2348	183	1.51	10		
952	*o*- and *p*-Xylene	2446	183	1.57	10		

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[1] Reference numbers beginning with 0, 1, 5, or 9 indicate a pollutant quantified by the internal standard method; reference numbers beginning with 2 or 6 indicate a labeled compound quantified by the internal standard method; reference numbers beginning with 3 or 7 indicate a pollutant quantified by isotope dilution.

[2] The retention time limits in this column are based on data from four wastewater laboratories. The single values for retention times in this column are based on data from one wastewater laboratory.

[3] This is a minimum level at which the analytical system shall give recognizable mass spectra (background corrected) and acceptable calibration points when calibrated using reagent water. The concentration in the aqueous or solid phase is determined using the equations in Section 13.

[4] Method detection limits determined in digested sludge (low solids) and in filter cake or compost (high solids).

[5] Specification derived from related compound.

[6] An unknown interference in the particular sludge studied precluded measurement of the method detection limit (MDL) for this compound.

[7] Background levels of these compounds were present in the sludge resulting in higher than expected MDLs. The MDL for these compounds is expected to be approximately 20 µg/kg (100 to 200 µg/kg for the gases and water soluble compounds) for the low solids method and 5 to 10 µg/kg (25 to 50 µg/kg for the gases and water soluble compounds) for the high solids methods, with no interferences present.

Column: 2.4 m (8 ft) × 2 mm I.D. glass, packed with 1% SP-1000 coated on 60/80 Carbopak B.
Carrier gas: Helium at 40 mL/min.
Temperature program: 3 min at 45°C, 8°C/min to 240°C, hold at 240°C for 15 minutes.

4. SAFETY

4.1 The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined; however, each chemical compound should be treated as a potential health hazard.

Exposure to these compounds should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets should also be made available to all personnel involved in these analyses. Additional information on laboratory safety can be found in References 5 through 7.

4.2 The following compounds covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, chloroform, and vinyl chloride. Primary standards of these toxic compounds should be prepared in a hood, and a NIOSH/MESA approved toxic gas respirator should be worn when high concentrations are handled.

5. APPARATUS AND MATERIALS

5.1 Sample bottles for discrete sampling.

5.1.1 Bottle: 25– to 40–mL with screw—cap (Pierce 13075, or equivalent). Detergent —wash, rinse with tap and distilled water, and dry at >105°C for a minimum of 1 hour before use.

5.1.2 Septum: Teflon-faced silicone (Pierce 12722, or equivalent), cleaned as above and baked at 100 to 200°C for 1 hour minimum.

5.2 Purge and trap device: Consists of purging device, trap, and desorber.

5.2.1 Purging devices for water and soil samples.

5.2.1.1 Purging device for water samples Designed to accept 5-mL samples with water column at least 3 cm deep. The volume of the gaseous head space between the water and trap shall be less than 15 mL. The purge gas shall be introduced less than 5 mm from the base of the water column and shall pass through the water as bubbles with a diameter less than 3 mm. The purging device shown in Figure 1 meets these criteria.

5.2.1.2 Purging device for solid samples: Designed to accept 5 g of solids plus 5 mL of water. The volume of the gaseous head space between the water and trap shall be less than 25 mL. The purge gas shall be introduced less than 5 mm from the base of the sample and shall pass through the water as bubbles with a diameter less than 3 mm. The purging device shall be capable of operating at ambient temperature (20 to 25°C) and of being controlled at temperatures of 40°C (±2°C) and 80°C (±5°C) while the sample is being purged. The purging device shown in Figure 2 meets these criteria.

5.2.2 Trap: 25 to 30 cm long × 2.5 mm I.D. minimum, containing the following:

5.2.2.1 Methyl silicone packing: 1cm (±0.2cm), 3% OV-1 on 60/80 mesh Chromosorb W, or equivalent.

5.2.2.2 Porous polymer: 15cm (±1.0 cm), Tenax GC (2,6-diphenylene oxide polymer), 60/80 mesh, chromatographic grade, or equivalent.

5.2.2.3 Silica gel: 8cm (±1.0 cm), Davison Chemical, 35/60 mesh, grade 15, or equivalent. The trap shown in Figure 3 meets these specifications.

5.2.3 Desorber: Shall heat the trap to 175°C (±5°C) in 45 seconds or less. The polymer section of the trap shall not exceed a temperature of 180°C and the remaining sections shall not exceed 220°C during desorb, and no portion of the trap shall exceed 225°C during bakeout. The desorber shown in Figure 3 meets these specifications.

5.2.4 The purge and trap device may be a separate unit, or coupled to a GC as shown in Figures 4 and 5.

5.3 Gas chromatograph: Shall be linearly temperature programmable with initial and final holds, shall contain a glass jet separator as the MS interface, and shall produce results which meet the calibration (Section 7), quality assurance (Section 8), and performance tests (Section 11) of this method.

5.3.1 Column: 2.8 · 0.4 m x 2 · 0.5 mm I.D. glass, packed with 1% SP-1000 on Carbopak B, 60/80 mesh, or equivalent.

5.4 Mass spectrometer: 70 eV electron impact ionization; shall repetitively scan from 20 to 250 amu every 2 to 3 seconds, and produce a unit resolution (valleys between m/z 174 to 176 less than 10% of the height of the m/z 175 peak), background corrected mass spectrum from 50 ng 4-bromofluorobenzene (BFB) injected into the GC. The BFB spectrum shall meet the mass-intensity criteria in Table 4. All portions of the GC column, transfer lines, and separator which connect the GC column to the ion source shall remain at or above the column temperature during analysis to preclude condensation of less volatile compounds.

5.5 Data system: Shall collect and record MS data, store mass-intensity data in spectral libraries, process GCMS data and generate reports, and shall calculate and record response factors.

Table 4
BFB Mass-Intensity Specifications

m/z	Intensity Required
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	base peak, 100%
96	5 to 9% of m/z 95
173	less than 2% of m/z 174
174	greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	95 to 101% of m/z 174
177	5 to 9% of m/z 176

5.5.1 Data acquisition: Mass spectra shall be collected continuously throughout the analysis and stored on a mass storage device.

5.5.2 Mass spectral libraries: User-created libraries containing mass spectra obtained from analysis of authentic standards shall be employed to reverse search GCMS runs for the compounds of interest (Section 7.2).

5.5.3 Data processing: The data system shall be used to search, locate, identify, and quantify the compounds of interest in each GCMS analysis. Software routines shall be employed to compute retention times and EICP areas. Displays of spectra, mass chromatograms, and library comparisons are required to verify results.

5.5.4 Response factors and multipoint calibrations: The data system shall be used to record and maintain lists of response factors (response ratios for isotope dilution) and generate multi-point calibration curves (Section 7). Computations of relative standard deviation (coefficient of variation) are useful for testing calibration linearity. Statistics on initial and ongoing performance shall be maintained (Sections 8 and 11).

5.6 Syringes: 5-mL glass hypodermic, with Luer-lok tips.

5.7 Micro syringes: 10-, 25-, and 100 µL.

5.8 Syringe valves: 2-way, with Luer ends (Teflon or Kel-F).

5.9 Syringe: 5-mL, gas-tight, with shut-off valve.

5.10 Bottles: 15-mL, screw-cap with Teflon liner.

5.11 Balances.

5.11.1 Analytical, capable of weighing 0.1 mg.

5.11.2 Top-loading, capable of weighing 10 mg.

5.12 Equipment for determining percent moisture.

5.12.1 Oven,capable of being temperature-controlled at 110°C (±5°C).

5.12.2 Dessicator.

5.12.3 Beakers: 50 to 100-mL.

6. REAGENTS AND STANDARDS

6.1 Reagent water: Water in which the compounds of interest and interfering compounds are not detected by this method (Section 11.7). It may be generated by any of the following methods:

6.1.1 Activated carbon: pass tap water through a carbon bed (Calgon Filtrasorb-300, or equivalent).

6.1.2 Water purifier: Pass tap water through a purifier (Millipore Super Q, or equivalent).

6.1.3 Boil and purge: Heat tap water to between 90 and 100°C and bubble contaminant free inert gas through it for approximately 1 hour. While still hot, transfer the water to screw-cap bottles and seal with a Teflon-lined cap.

6.2 Sodium thiosulfate: ACS granular.

6.3 Methanol: Pesticide-quality or equivalent.

6.4 Standard solutions: Purchased as solutions or mixtures with certification to their purity, concentration, and authenticity, or prepared from materials of known purity and composition. If compound purity is 96% or greater, the weight may be used without correction to calculate the concentration of the standard.

6.5 Preparation of stock solutions: Prepare in methanol using liquid or gaseous standards per the steps below. Observe the safety precautions given in Section 4.

6.5.1 Place approximately 9.8 mL of methanol in a 10-mL ground-glass-stoppered volumetric flask. Allow the flask to stand unstoppered for approximately 10 minutes or until all methanol wetted surfaces have dried. In each case, weigh the flask, immediately add the compound, then immediately reweigh to prevent evaporation losses from affecting the measurement.

6.5.1.1 Liquids: Using a 100 µL syringe, permit 2 drops of liquid to fall into the methanol without contacting the neck of the flask. Alternatively, inject a known volume of the compound into the methanol in the flask using a micro-syringe.

6.5.1.2 Gases (chloromethane, bromomethane, chloroethane, vinyl chloride): Fill a valved 5-mL gas-tight syringe with the compound. Lower the needle to approximately 5 mm above the methanol meniscus. Slowly introduce the compound above the surface of the meniscus. The gas will dissolve rapidly in the methanol.

6.5.2 Fill the flask to volume, stopper, then mix by inverting several times. Calculate the concentration in mg/mL (µg/µL) from the weight gain (or density if a known volume was injected).

6.5.3 Transfer the stock solution to a Teflon–sealed screw-cap bottle. Store, with minimal headspace, in the dark at –10 to –20°C.

6.5.4 Prepare fresh standards weekly for the gases and 2-chloroethylvinyl ether. All other standards are replaced after one month, or sooner if comparison with check standards indicate a change in concentration. Quality control check standards that can be used to determine the accuracy of calibration standards are available from the US Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

6.6 Labeled compound spiking solution: From stock standard solutions prepared as above, or from mixtures, prepare the spiking solution to contain a concentration such that a 5- to 10- µL spike into each 5-mL sample, blank, or aqueous standard analyzed will result in a concentration of 20 ug/L of each labeled compound. For the gases and for the water soluble compounds (acrolein, acrylonitrile, acetone, diethyl ether, p-dioxane, and MEK), a concentration of 100 ug/L may be used. Include the internal standards (Section 7.5) in this solution so that a concentration of 20 ug/L in each sample, blank, or aqueous standard will be produced.

6.7 Secondary standards: Using stock solutions, prepare a secondary standard in methanol to contain each pollutant at a concentration of 500 µg/mL. For the gases and water soluble compounds (Section 6.6), a concentration of 2.5 mg/mL may be used.

6.7.1 Aqueous calibration standards: Using a 25-µL syringe, add 20 µL of the secondary standard (Section 6.7) to 50, 100, 200, 500, and 1000 mL of reagent water to produce concentrations of 200, 100, 50, 20, and 10 µg/L, respectively. If the higher concentration standard for the gases and water soluble compounds was chosen (Section 6.6), these compounds will be at concentrations of 1000, 500, 250, 100, and 50 µg/L in the aqueous calibration standards.

6.7.2 Aqueous performance standard: An aqueous standard containing all pollutants, internal standards, labeled compounds, and BFB is prepared daily, and analyzed each shift to demonstrate performance (Section 11). This standard shall contain either 20 or 100 µg/L of the labeled and pollutant gases and water soluble compounds, 10 µg/L BFB, and 20 µg/L of all other pollutants, labeled compounds, and internal standards. It may be the nominal 20 µg/L aqueous calibration standard (Section 6.7.1).

6.7.3 A methanolic standard containing all pollutants and internal standards is prepared to demonstrate recovery of these compounds when syringe injection and purge-and-trap analyses are compared. This standard shall contain either 100 µg/mL or 500 µg/mL of the gases and water soluble compounds, and 100 µg/mL of the remaining pollutants and internal standards (consistent with the amounts in the aqueous performance standard in 6.7.2).

6.7.4 Other standards which may be needed are those for test of BFB performance (Section 7.1) and for collection of mass spectra for storage in spectral libraries (Section 7.2).

7. CALIBRATION

Calibration of the GCMS system is performed by purging the compounds of interest and their labeled analogs from reagent water at the temperature to be used for analysis of samples.

7.1 Assemble the gas chromatographic apparatus and establish operating conditions given in Table 3. By injecting standards into the GC, demonstrate that the analytical system meets the minimum levels in Table 3 for the compounds for which calibration is to be performed, and the mass-intensity criteria in Table 4 for 50 ng BFB.

7.2 Mass spectral libraries: Detection and identification of the compounds of interest are dependent upon the spectra stored in user created libraries.

7.2.1 For the compounds in Table 1 and other compounds for which the GCMS is to be calibrated, obtain a mass spectrum of each pollutant and labeled compound and each internal standard by analyzing an authentic standard either singly or as part of a mixture in which there is no interference between closely eluted components. Examine the spectrum to determine that only a single compound is present. Fragments not attributable to the compound under study indicate the presence of an interfering compound. Adjust the analytical conditions and scan rate (for this test only) to produce an undistorted spectrum at the GC peak maximum. An undistorted spectrum will usually be obtained if five complete spectra are collected across the upper half of the GC peak. Software algorithms designed to "enhance" the spectrum may eliminate distortion, but may also eliminate authentic m/z's or introduce other distortion.

7.2.2 The authentic reference spectrum is obtained under BFB tuning conditions (Section 7.1 and Table 4) to normalize it to spectra from other instruments.

7.2.3 The spectrum is edited by saving the five most intense mass spectral peaks and all other mass spectral peaks greater than 10% of the base peak. The spectrum may be further edited to remove common interfering masses. If five mass spectral peaks cannot be obtained under the scan conditions given in Section 5.4, the mass spectrometer may be scanned to an m/z lower than 20 to gain additional spectral information. The spectrum obtained is stored for reverse search and for compound confirmation.

7.2.4 For the compounds in Table 2 and other compounds for which the mass spectra, quantitation m/z's, and retention times are known but the instrument is not to be calibrated, add the retention time and reference compound (Table 3); the response factor and the quantitation m/z (Table 5); and spectrum (Appendix A) to the reverse search library. Edit the spectrum per Section 7.2.3, if necessary.

7.3 Assemble the purge-and-trap device. Pack the trap as shown in Figure 3 and condition overnight at 170 to 180°C by backflushing with an inert gas at a flow rate of 20 to 30 mL/min. Condition traps daily for a minimum of 10 minutes prior to use.

7.3.1 Analyze the aqueous performance standard (Section 6.7.2) according to the purge–and–trap procedure in Section 10. Compute the area at the primary m/z (Table 5) for each compound. Compare these areas to those obtained by injecting 1 µL of the methanolic standard (Section 6.7.3) to determine compound recovery. The recovery shall be greater than 20% for the water soluble compounds (Section 6.6), and 60 to 110% for all other compounds. This recovery is demonstrated initially for each purge-and-trap GCMS system. The test is repeated only if the purge-and-trap or GCMS systems are modified in any way that might result in a change in recovery.

7.3.2 Demonstrate that 100 ng toluene (or toluene-d_8) produces an area at m/z 91 (or 99) approximately one-tenth that required to exceed the linear range of the system. The exact value must be determined by experience for each instrument. It is used to match the calibration range of the instrument to the analytical range and detection limits required.

Table 5. Volatile Organic Compound Characteristic M/Z'S

Compound	labeled Analog	Primary m/z[1]	Reference Compound[2]	Response purge temp. Of: 20 °C	80 °C
Acetone	d_6	58/64			
Acrolein	d_4	56/60			
Acrylonitrile	d_3	53/56			
Allyl alcohol		57	181	--[3]	0.20
Benzene	d_6	78/84			
2-Bromo-1-chloropropane[4]		77			
Bromochloromethane[4]		128			
Bromodichloromethane	13$_c$	83/86			
Bromoform	13$_c$	173/176			
Bromomethane	d_3	96/99			
Carbon disulfide		76	181	1.93	2.02
Carbon tetrachloride	13$_c$	47/48			
2-Chloro-1,3-butadiene		53	182	0.29	0.50
Chloroacetonitrile		75	181	--[3]	1.12
Chlorobenzene	d_5	112/117			
Chloroethane	d_5	64/71			
2-Chloroethylvinyl ether	d_7	106/113			
Chloroform	13$_C$	85/86			
Chloromethane	d_3	50/53			
3-Chloropropene		76	181	0.43	0.63
Crotonaldehyde		70	182	--[3]	0.090
Dibromochloromethane	13$_c$	129/130			
1,2-Dibromoethane		107	182	0.86	0.68
Dibromomethane		93	181	1.35	1.91
1,4-Dichlorobutane		55			
trans-1,4-Dichloro-2-butene		75	183	0.093	0.014
1,1-Dichloroethane	d_3	63/66			

Compound	labeled Analog	Primary m/z[1]	Reference Compound[2]	Response purge temp. Of:	
				20 °C	80 °C
1,2-Dichloroethane	d_4	62/67			
1,1-Dichloroethene	d_2	61/65			
trans-1,2-Dichlorethene	d_2	61/65			
1,2-Dichloropropane	d_6	63/67			
1,3-Dichloropropane		76	182	0.89	0.88
cis-1,3-Dichloropropene		75	182	0.29	0.41
trans-1,3-Dichloropropene	d_4	75/79			
Diethyl ether	d_{10}	74/84			
p-Dioxane	d_8	88/96			
Ethyl cyanide		54	181	(3)	1.26
Ethyl methacrylate		69	183	0.69	0.52
Ethylbenzene	d_{10}	106/116			
2-Hexanone		58	183	0.076	0.33
Iodomethane		142	181	4.55	2.55
Isobutyl alcohol		74	181	(3)	0.22
Methylene chloride	d_2	84/88			
Methyl ethyl ketone	d_8	72/80			
Methyl methacrylate		69	182	0.23	0.79
4-Methyl-2-pentanone		58	183	0.15	0.29
Methacrylonitrile		67	181	0.25	0.79
1,1,1,2-Tetrachloroethane		131	182	0.20	0.25
1,1,2,2-Tetrachloroethane	d_2	83/84			
Tetrachloroethene	13_{C}^{2}	164/172			
Toluene	d_8	92/100			
1,1,1-Trichloroethane	d_3	97/102			
1,1,2-Trichloroethane	13_{C}^{2}	83/84			
Trichloroethene	13_{C}^{2}	95/136			
Trichlorofluoromethane		101	181	2.31	2.19
1,2,3-Trichloropropane		75	183	0.89	0.72
Vinyl acetate		86	182	0.054	0.19
Vinyl chloride	d_3	62/65			
m-Xylene		106	183	1.69	-
0- and *p*-Xylene		106	183	3.33	-

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[1] Native/labeled

[2] 181 = bromochloromethane
182 = 2-bromo-1-chloropropane
183 = 1,4-dichlorobutane

[3] Not detected at a purge temperature of 20°C

[4] Internal standard

Note: Because the composition and purity of commercially-supplied isotopically labeled standard's may vary, the primary m/z of the labeled analogs given in this table should be used as guidance. The appropriate m/z of the labeled analogs should be determined prior to use for sample analysis. Deviations from the m/z's listed here must be documented by the laboratory and submitted with the data.

7.4 Calibration by isotope dilution: The isotope dilution approach is used for the purgeable organic compounds when appropriate labeled compounds are available and when interferences do not preclude the analysis. If labeled compounds are not available, or interferences are present, the internal standard method (Section 7.5) is used. A calibration curve encompassing the concentration range of interest is prepared for each compound determined. The relative response (RR) vs. concentration (µg/L) is plotted or computed using a linear regression. An example of a calibration curve for toluene using toluene-d_8 is given in Figure 6. Also shown are the ±10% error limits (dotted lines). Relative response is determined according to the procedures described below. A minimum of five data points are required for calibration (Section 7.4.4).

7.4.1 The relative response (RR) of pollutant to labeled compound is determined from isotope ratio values calculated from acquired data. Three isotope ratios are used in this process:

R_x = *the isotope ratio measured in the pure pollutant (Figure 7A).*
R_y = *the isotope ratio of pure labeled compound (Figure 7B).*
R_m = *the isotope ratio measured in the analytical mixture of the pollutant and labeled compounds (Figure 7C.)*

The correct way to calculate RR is:

$$RR = \frac{(R_y - R_m)\ (R_x + 1)}{(R_m - R_x)\ (R_y + 1)}$$

If R_m is not between $2R_y$ and $0.5R_x$, the method does not apply and the sample is analyzed by the internal standard method (Section 7.5).

7.4.2 In most cases, the retention times of the pollutant and labeled compound are the same, and isotope ratios (R's) can be calculated from the EICP areas, where:

$$R = \frac{(area\ at\ m_1/z)}{(area\ at\ m_2/z)}$$

If either of the areas is zero, it is assigned a value of one in the calculations; that is, if: area of m_1/z = 50721,
area of mz/Z = 0,
then R = 50721/1 = 50720

The data from these analyses are reported to three significant figures (see Section 13.6). In order to prevent rounding errors from affecting the values to be reported, all calculations performed prior to the final determination of concentrations should be carried out using at least four significant figures. Therefore, the calculation of R above is rounded to four significant figures.

The m/z's are always selected such that $R_x > R_y$. When there is a difference in retention times (RT) between the pollutant and labeled compounds, special precautions are required to determine the isotope ratios.

R_x, R_y, and R_m are defined as follows:

$$R_x = \frac{[area\ m_1/z\ (at\ RT_1)]}{1}$$

$$R_y = \frac{1}{[area\ m_2/z\ (at\ RT_2)]}$$

$$R_m = \frac{[area\ m_1/z\ (at\ RT_1)]}{[area\ m_2/z\ (at\ RT_2)]}$$

7.4.3 An example of the above calculations can be taken from the data plotted in Figure 7 for toluene and toluene-d_8. For these data:

$$R_x = \frac{168920}{1} = 168900$$

$$R_y = \frac{1}{60960} = 0.00001640$$

$$R_m = \frac{96868}{82508} = 1.174$$

The RR for the above data is then calculated using the equation given in Section 7.4.1. For the example, rounded to four significant figures, RR = 1.174. Not all labeled compounds elute before their pollutant analogs.

7.4.4 To calibrate the analytical system by isotope dilution, analyze a 5-mL aliquot of each of the aqueous calibration standards (Section 6.7.1) spiked with an appropriate constant amount of the labeled compound spiking solution (Section 6.6), using the purge-and-trap procedure in Section 10. Compute the RR at each concentration.

7.4.5 Linearity: If the ratio of relative response to concentration for any compound is constant (less than 20% coefficient of variation) over the five point calibration range, an averaged relative response/concentration ratio may be used for that compound; otherwise, the complete calibration curve for that compound shall be used over the five point calibration range.

7.5 Calibration by internal standard: Used when criteria for isotope dilution (Section 7.4) cannot be met. The method is applied to pollutants having no labeled analog and to the labeled compounds. The internal standards used for volatiles analyses are bromochloromethane, 2-bromo-1-chloropropane, and 1,4-dichlorobutane. Concentrations of the labeled compounds and pollutants without labeled analogs are computed relative to the nearest eluting internal standard, as shown in Tables 3 and 5.

7.5.1 Response factors: Calibration requires the determination of response factors (RF) which are defined by the following equation:

$$R = \frac{(A_s \times C_{is}),}{(a_{is} \times C_s)}$$

Where:

A = *is the EICP area at the characteristic m/z for the compound in the daily standard.*
A_{is} = *is the EICP area at the characteristic m/z for the internal standard.*
C_{is} = *is the concentration* (µg/L) *of the internal standard.*
C_s = *is the concentration of the pollutant in the daily standard.*

7.5.2 The response factor is determined at 10, 20, 50, 100, and 200 µg/L for the pollutants (optionally at five times these concentrations for gases and water soluble pollutants; see Section 6.7), in a way analogous to that for calibration by isotope dilution (Section 7.4.4). The RF is plotted against concentration for each compound in the standard (C_s) to produce a calibration curve.

7.5.3 Linearity: If the response factor (RF) for any compound is constant (less than 35% coefficient of variation) over the five-point calibration range, an averaged response factor may be used for that compound; otherwise, the complete calibration curve for that compound shall be used over the five-point range.

7.6 Combined calibration: By adding the isotopically labeled compounds and internal standards (Section 6.6) to the aqueous calibration standards (Section 6.7.1), a single set of analyses can be used to produce calibration curves for the isotope dilution and internal standard methods. These curves are verified each shift (Section 11.5) by purging the aqueous performance standard (Section 6.7.2). Recalibration is required only if calibration and ongoing performance (Section 11.5) criteria cannot be met.

7.7 Elevated purge temperature calibration: Samples containing greater than 1% solids are analyzed at a temperature of 40°C (±2°C) (Section 10). For these samples, the analytical system may be calibrated using a purge temperature of 40°C(±2°C) in order to more closely approximate the behavior of the compounds of interest in high solids samples.

8. QUALITY ASSURANCE/QUALITY CONTROL

8.1 Each laboratory that uses this method is required to operate a formal quality assurance program (Reference 8). The minimum requirements of this program consist of an initial demonstration of laboratory capability, analysis of samples spiked with labeled compounds to evaluate and document data quality, and analysis of standards and blanks as tests of continued performance. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

8.1.1 The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 The analyst is permitted to modify this method to improve separations or lower the costs of measurements, provided all performance specifications are met. Each time a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2 to demonstrate method performance.

8.1.3 Analyses of blanks are required to demonstrate freedom from contamination and that the compounds of interest and interfering compounds have not been carried over from a previous analysis (Section 3). The procedures and criteria for analysis of a blank are described in Section 8.5.

8.1.4 The laboratory shall spike all samples with labeled compounds to monitor method performance. This test is described in Section 8.3. When results of these spikes indicate atypical method performance for samples, the samples are diluted to bring method performance within acceptable limits (Section 14.2).

8.1.5 The laboratory shall, on an ongoing basis, demonstrate through the analysis of the aqueous performance standard (Section 6.7.2) that the analysis system is in control. This procedure is described in Sections 11.1 and 11.5.

8.1.6 The laboratory shall maintain records to define the quality of data that is generated. Development of accuracy statements is described in Sections 8.4 and 11.5.2.

Table 6. Acceptance Criteria for Performance Tests

EGD No.[1]	Compound	Acceptance criteria at 20 µg/L or as noted			
		Labeled and native compound initial precision and accuracy (Sect. 8.2.3)		Labeled compound recovery (Sect. 8.3 and 14.2)	Labeled and native compound ongoing accuracy (Sect. 11.5)
		s (µg/L)	X (µg/L)	P (%)	R (µg/L)
516	acetone*	51.0	77 - 153	35 - 165	55 - 145
002	acrolein*	72.0	32 - 168	37 - 163	7 - 190
003	acrylonitrile*	16.0	70 - 132	ns - 204	58 - 144
004	benzene	9.0	13 - 28	ns - 196	4 - 33
048	bromodichloromethane	8.2	7 - 32	ns - 199	4 - 34
047	bromoform	7.0	7 - 35	ns - 214	6 - 36
046	bromomethane	25.0	d - 54	ns - 414	d - 61
006	carbon tetrachloride	6.9	16 - 25	42 - 165	12 - 30
007	chlorobenzene	8.2	14 - 30	ns - 205	4 - 35
016	chloroethane	15.0	d - 47	ns - 308	d - 51
019	2-chloroethylvinyl ether	36.0	d - 70	ns - 554	d - 79
023	chloroform	7.9	12 - 26	18 - 172	8 - 30
045	chloromethane	26.0	d - 56	ns - 410	d - 64
051	dibromochloromethane	7.9	11 - 29	16 - 185	8 - 32
013	1,1-dichloroethane	6.7	11 - 31	23 - 191	9 - 33
010	1,2-dichloroethane	7.7	12 - 30	12 - 192	8 - 33
029	1,1-dichloroethene	12.0	d - 50	ns - 315	d - 52
030	trans-1,2-dichloroethene	7.4	11 - 32	15 - 195	8 - 34
032	1,2-dichloropropane	19.0	d - 47	ns - 343	d - 51
033	trans-1,3-dichloropropene	15.0	d - 40	ns - 284	d - 44
515	diethyl ether*	44.0	75 - 146	44 - 156	55 - 145

		Acceptance criteria at 20 µg/L or as noted			
EGD		Labeled and native compound initial precision and accuracy (Sect. 8.2.3)		Labeled compound recovery (Sect. 8.3 and 14.2)	Labeled and native compound ongoing accuracy (Sect. 11.5)
No.[1]	Compound	s (µg/L)	X (µg/L)	P (%)	R (µg/L)
527	p-dioxane*	7.2	13 - 27	ns - 239	11 - 29
038	ethylbenzene	9.6	16 - 29	ns - 203	5 - 35
044	methylene chloride	9.7	d - 50	ns - 316	d - 50
514	methyl ethyl ketone*	57.0	66 - 159	36 - 164	42 - 158
015	1,1,2,2-tetrachloroethane	9.6	11 - 30	5 - 199	7 - 34
085	tetrachloroethane	6.6	15 - 29	31 - 181	11 - 32
086	toluene	6.3	15 - 29	4 - 193	6 - 33
011	1,1,1-trichloroethane	5.9	11 - 33	12 - 200	8 - 35
014	1,1,2-trichloroethane	7.1	12 - 30	21 - 184	9 - 32
087	trichloroethene	8.9	17 - 30	35 - 196	12 - 34
088	vinyl chloride	28.0	d - 59	ns - 452	d - 65

* acceptance criteria at 100 µg/L
d = detected; result must be greater than zero.
ns = no specification; limit would be below detection limit.

[1] Reference numbers beginning with 0, 1, or 5 indicate a pollutant quantified by the internal standard method; reference numbers beginning with 2 or 6 indicate a labeled compound quantified by the internal Standard method; reference numbers beginning with 3 or 7 indicate a pollutant quantified by isotope dilution.

8.2 Initial precision and accuracy: To establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations for compounds to be calibrated:

8.2.1 Analyze two sets of four 5-mL aliquots (8 aliquots total) of the aqueous performance standard (Section 6.7.2) according to the method beginning in Section 10.

8.2.2 Using results of the first set of four analyses in Section 8.2.1, compute the average recovery (X) in µg/L and the standard deviation of the recovery (s) in µg/L for each compound, by isotope dilution for pollutants with a labeled analog, and by internal standard for labeled compounds and pollutants with no labeled analog.

8.2.3 For each compound, compare s and X with the corresponding limits for initial precision and accuracy found in Table 6. If s and X for all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may begin. If, however, any individual s exceeds the precision limit or any individual X falls outside the range for accuracy, system performance is unacceptable for that compound.

NOTE: *The large number of compounds in Table 6 present a substantial probability that one or more will fail one of the acceptance criteria when all compounds are analyzed. To determine if the analytical system is out of control, or if the failure can be attributed to probability, proceed as follows:*

8.2.4 Using the results of the second set of four analyses, compute s and X for only those compounds which failed the test of the first set of four analyses (Section 8.2.3). If these compounds now pass, system performance is acceptable for all compounds and analysis of blanks and samples may begin. If, however, any of the same compounds fail again, the analysis system is not performing properly for the compound (s) in question. In this event, correct the problem and repeat the entire test (Section 8.2.1).

8.3 The laboratory shall spike all samples with labeled compounds to assess method performance on the sample matrix.

8.3.1 Spike and analyze each sample according to the method beginning in Section 10.

8.3.2 Compute the percent recovery (P) of the labeled compounds using the internal standard method (Section 7.5).

8.3.3 Compare the percent recovery for each compound with the corresponding labeled compound recovery limit in Table 6. If the recovery of any compound falls outside its warning limit, method performance is unacceptable for that compound in that sample. Therefore, the sample matrix is complex and the sample is to be diluted and reanalyzed, per Section 14.2.

8.4 As part of the QA program for the laboratory, method accuracy for wastewater samples shall be assessed and records shall be maintained. After the analysis of five wastewater samples for which the labeled compounds pass the tests in Section 8.3.3, compute the average percent recovery (P) and the standard deviation of the percent recovery (sp) for the labeled compounds only. Express the accuracy assessment as a percent recovery interval from P - 2sp to P + 2sp. For example, if P = 90% and sp = 10%, the accuracy interval is expressed as 70 to 110%. Update the accuracy assessment for each compound on a regular basis (e.g., after each 5 to 10 new accuracy measurements).

8.5 Blanks: Reagent water blanks are analyzed to demonstrate freedom from carry-over (Section 3) and contamination.

8.5.1 The level at which the purge and trap system will carry greater than 5 µg/L of a pollutant of interest (Tables 1 and 2) into a succeeding blank shall be determined by analyzing successively larger concentrations of these compounds. When a sample contains this concentration or more, a blank shall be analyzed immediately following this sample to demonstrate no carry-over at the 5 µg/L level.

8.5.2 With each sample lot (samples analyzed on the same 8-hour shift), a blank shall be analyzed immediately after analysis of the aqueous performance standard (Section 11.1) to demonstrate freedom from contamination. If any of the compounds of interest (Tables 1 and 2) or any potentially interfering compound is found in a blank at greater than 10 µg/L (assuming a response factor of 1 relative to the nearest eluted internal standard for compounds not listed in Tables 1 and 2), analysis of samples is halted until the source of contamination is eliminated and a blank shows no evidence of contamination at this level.

8.6 The specifications contained in this method can be met if the apparatus used is calibrated properly, then maintained in a calibrated state. The standards used for calibration (Section 7), calibration verification (Section 11.5) and for initial (Section 8.2) and ongoing (Section 11.5) precision and accuracy should be identical, so that the most precise results will be obtained. The GCMS instrument in particular will provide the most reproducible results if dedicated to the settings and conditions required for the analyses of volatiles by this method.

8.7 Depending on specific program requirements, field replicates may be collected to determine the precision of the sampling technique, and spiked samples may be required to determine the accuracy of the analysis when the internal method is used.

9. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

9.1 Grab samples are collected in glass containers having a total volume greater than 20 mL. For aqueous samples which pour freely, fill sample bottles so that no air bubbles pass through the sample as the bottle is filled and seal each bottle so that no air bubbles are entrapped. Maintain the hermetic seal on the sample bottle until time of analysis.

9.2 Samples are maintained at 0 to 4°C from the time of collection until analysis. If an aqueous sample contains residual chlorine, add sodium thiosulfate preservative (10 mg/40 mL) to the empty sample bottles just prior to shipment to the sample site. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine (Reference 9). If preservative has been added, shake the bottle vigorously for one minute immediately after filling.

9.3 For aqueous samples, experimental evidence indicates that some aromatic compounds, notably benzene, toluene, and ethyl benzene are susceptible to rapid biological degradation under certain environmental conditions. Refrigeration alone may not be adequate to preserve these compounds in wastewaters for more than seven days. For this reason, a separate sample should be collected, acidified, and analyzed when these aromatics are to be determined. Collect about 500 mL of sample in a clean container. Adjust the pH of the sample to about 2 by adding HCl (1+1) while stirring. Check pH with narrow range (1.4 to 2.8) pH paper. Fill a sample container as described in Section 9.1. If residual chlorine is present, add sodium thiosulfate to a separate sample container and fill as in Section 9.1.

9.4 All samples shall be analyzed within 14 days of collection.

10. PURGE, TRAP, AND GCMS ANALYSIS

Samples containing less than one percent solids are analyzed directly as aqueous samples (Section 10.4). Samples containing one percent solids or greater are analyzed as solid samples utilizing one of two methods, depending on the levels of pollutants in the sample. Samples containing one percent solids or greater and low to moderate levels of pollutants are analyzed by purging a known weight of sample added to 5 mL of reagent water (Section 10.5). Samples containing 1% solids or greater and high levels of pollutants are extracted with methanol, and an aliquot of the methanol extract is added to reagent water and purged (Section 10.6).

10.1 Determination of percent solids.

10.1.1 Weigh 5 to 10 g of sample into a tared beaker.

10.1.2 Dry overnight (12 hours minimum) at 110°C (±5°C), and cool in a dessicator.

10.1.3 Determine percent solids as follows:

$$\%\ solids = \frac{weight\ of\ sample\ dry}{weight\ of\ sample\ wet} \times 100$$

10.2 Remove standards and samples from cold storage and bring to 20 to 25°C.

10.3 Adjust the purge gas flow rate to 40 (±4mL/min).

10.4 Samples containing less than 1% solids.

10.4.1 Mix the sample by shaking vigorously. Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample bottle and carefully pour the sample into the syringe barrel until it overflows. Replace the plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5 mL (±0.1 mL). Because this process of taking an aliquot destroys the validity of the sample for future analysis, fill a second syringe at this time to protect against possible loss of data.

10.4.2 Add an appropriate amount of the labeled compound spiking solution (Section 6.6) through the valve bore, then close the valve.

10.4.3 Attach the syringe valve assembly to the syringe valve on the purging device. Open both syringe valves and inject the sample into the purging chamber. Purge the sample per Section 10.7.

10.5 Samples containing 1% solids or greater and low to moderate levels of pollutants.

10.5.1 Mix the sample thoroughly using a clean spatula.

10.5.2 Weigh 5 g (±1 g) of sample into a purging vessel (Figure 2). Record the weight to three significant figures.

10.5.3 Add 5 mL (±0.1 mL) of reagent water to the vessel.

10.5.4 Using a metal spatula, break up any lumps of sample to disperse the sample in the water.

10.5.5 Add an appropriate amount of the labeled compound spiking solution (Section 6.6) to the sample in the purge vessel. Place a cap on the purging vessel and and shake vigorously to further disperse the sample. Attach the purge vessel to the purging device, and purge the sample per Section 10.7.

10.6 Samples containing 1% solids or greater and high levels of pollutants, or samples requiring dilution by a factor of more than 100 (see Section 13.4).

10.6.1 Mix the sample thoroughly using a clean spatula.

10.6.2 Weigh 5g (±1 g) of sample into a calibrated 15- to 25-mL centrifuge tube. Record the weight of the sample to three significant figures.

10.6.3 Add 10 mL of methanol to the centrifuge tube. Cap the tube and shake it vigorously for 15 to 20 seconds to disperse the sample in the methanol. Allow the sample to settle in the tube. If necessary, centrifuge the sample to settle suspended particles.

10.6.4 Remove approximately 0.1% of the volume of the supernatant methanol using a 15- to 25- µL syringe. This volume will be in the range of 10 to 15 µL.

10.6.5 Add this volume of the methanol extract to 5 mL reagent water in a 5 mL syringe, and analyze per Section 10.4.1.

10.6.6 For further dilutions, dilute 1 mL of the supernatant methanol (Section 10.6.4) to 10 mL, 100 mL, 1000 mL, etc., in reagent water. Remove a volume of this methanol extract/reagent water mixture equivalent to the volume in Section 10.6.4, add it to 5 mL reagent water in a 5 mL syringe, and analyze per Section 10.4.1.

10.7 Purge the sample for 11 minutes (±0.1 minute) at 20 to 25°C for samples containing less than 1% solids. Purge samples containing one percent solids or greater at 40°(±2°). If the compounds in Table 2 that do not purge at 20 to 40°C are to be determined, a purge temperature of 80°C (±5°C) is used.

10.8 After the 11 minute purge time, attach the trap to the chromatograph and set the purge-and- trap apparatus to the desorb mode (Figure 5). Desorb the trapped compounds into the GC column by heating the trap to between 170 and 180°C while backflushing with carrier gas at 20 to 60 mL/min for 4 minutes. Start MS data acquisition upon start of the desorb cycle, and start the GC column temperature program 3 minutes later. Table 3 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times and minimum levels that can be achieved under these conditions. An example of the separations achieved by the column listed is shown in Figure 9. Other columns may be used provided the requirements in Section 8 are met.

If the priority pollutant gases produce GC peaks so broad that the precision and recovery specifications (Section 8.2) cannot be met, the column may be cooled to ambient or subambient temperatures to sharpen these peaks.

10.9 After desorbing the sample for four minutes, recondition the trap by purging with purge gas while maintaining the trap temperature at between 170 and 180°C. After approximately 7 minutes, turn off the trap heater to stop the gas flow through the trap. When cool, the trap is ready for the next sample.

10.10 While analysis of the desorbed compounds proceeds, remove and clean the purge device. Rinse with tap water, clean with detergent and water, rinse with tap and distilled water, and dry for aminimum of 1 hour in an oven at a temperature greater than 150°C.

11. SYSTEM PERFORMANCE

11.1 At the beginning of each 8 hour shift during which analyses are performed, system calibration and performance shall be verified for the pollutants and labeled compounds (Table 1). For these tests, analysis of the aqueous performance standard (Section 6.7.2) shall be used to verify all performance criteria. Adjustment and/or recalibration (per Section 7) shall be performed until all performance criteria are met. Only after all performance criteria are met may blanks and samples be analyzed.

11.2 BFB spectrum validity: The criteria in Table 4 shall be met.

11.3 Retention times: The absolute retention times of the internal standards shall be as follows: bromochloromethane: 653 to 782 seconds; 2-bromo-1-chloropropane: 1270 to 1369 seconds; 1,4-dichlorobutane: 1510 to 1605 seconds. The relative retention times of all pollutants and labeled compounds shall fall within the limits given in Table 3.

11.4 GC resolution: The valley height between toluene and toluene-d_8 (at m/z 91 and 99 plotted on the same graph) shall be less than 10% of the taller of the two peaks.

11.5 Calibration verification and ongoing precision and accuracy: Compute the concentration of each pollutant (Table 1) by isotope dilution (Section 7.4) for those compounds which have labeled analogs. Compute the concentration of each pollutant which has no labeled analog by the internal standard method (Section 7.5). Compute the concentrations of the labeled compounds themselves by the internal standard method. These concentrations are computed based on the calibration data determined in Section 7.

11.5.1 For each pollutant and labeled compound, compare the concentration with the corresponding limit for ongoing accuracy in Table 6. If all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may continue. If any individual value falls outside the range given, system performance is unacceptable for that compound.

NOTE: *The large number of compounds in Table 6 present a substantial probability that one or more will fail the acceptance criteria when all compounds are analyzed. To determine if the analytical system is out of control, or if the failure may be attributed to probability, proceed as follows:*

11.5.1.1 Analyze a second aliquot of the aqueous performance standard (Section 6.7.2).

11.5.1.2 Compute the concentration for only those compounds which failed the first test (Section 11.5.1). If these compounds now pass, system performance is acceptable for all compounds, and analyses of blanks and samples may proceed. If, however, any of the compounds fail again, the measurement system is not performing properly for these compounds. In this event, locate and correct the problem or recalibrate the system (Section 7), and repeat the entire test (Section 11.1) for all compounds.

11.5.2 Add results which pass the specification in Section 11.5.1.2 to initial (Section 8.2) and previous on-going data. Update QC charts to form a graphic representation of laboratory performance (Figure 8). Develop a statement of accuracy for each pollutant and labeled compound by calculating the average percent recovery (R) and the standard deviation of percent recovery (sr). Express the accuracy as a recovery interval from R – 2sr to R + 2sr. For example, if R = 95% and sr = 5%, the accuracy is 85 to 105%.

12. QUALITATIVE DETERMINATION

Identification is accomplished by comparison of data from analysis of a sample or blank with data stored in the mass-spectral libraries. For compounds for which the relative retention times and mass spectra are known, identification is confirmed per Sections 12.1 and 12.2. For unidentified GC peaks, the spectrum is compared to spectra in the EPA/NIH mass spectral file per Section 12.3.

12.1 Labeled compounds and pollutants having no labeled analog (Tables 1 and 2):

12.1.1 The signals for all characteristic m/z's stored in the spectral library (Section 7.2.3) shall be present and shall maximize within the same two consecutive scans.

12.1.2 Either (1) the background corrected EICP areas or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of 2 (0.5 to 2 times) for all masses stored in the library.

12.1.3 In order for the compounds for which the system has been calibrated (Table 1) to be identified, their relative retention times shall be within the retention-time windows specified in Table 3.

12.1.4 The system has not been calibrated for the compounds listed in Table 2; however, the relative retention times and mass spectra of these compounds are known. Therefore, for a compound in Table 2 to be identified, its relative retention time must fall within a retention-time window of ±60 seconds or ±20 scans (whichever is greater) of the nominal retention time of the compound specified in Table 3.

12.2 Pollutants having a labeled analog (Table 1):

12.2.1 The signals for all characteristic m/z's stored in the spectral library (Section 7.2.3) shall be present and shall maximize within the same two consecutive scans.

12.2.2 Either (1) the background corrected EICP areas or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two for all masses stored in the spectral library.

12.2.3 The relative retention time between the pollutant and its labeled analog shall be within the windows specified in Table 3.

12.3 Unidentified GC peaks.

12.3.1 The signals for m/z's specific to a GC peak shall all maximize within the same two consecutive scans.

12.3.2 Either (1) the background corrected EICP areas or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of 2 with the masses stored in the EPA/NIH mass-spectral file.

12.4 The m/z's present in the sample mass spectrum that are not present in the reference mass spectrum shall be accounted for by contaminant or background ions. If the sample mass spectrum is contaminated, or if identification is ambiguous, an experienced spectrometrist (Section 1.4) is to determine the presence or absence of the compound.

13. QUANTITATIVE DETERMINATION

13.1 Isotope dilution: Because the pollutant and its labeled analog exhibit the same effects upon purging, desorption, and gas chromatography, correction for recovery of the pollutant can be made by adding a known amount of a labeled compound to every sample prior to purging. Relative response (RR) values for sample mixtures are used in conjunction with the calibration curves described in Section 7.4 to determine concentrations directly, so long as labeled compound spiking levels are constant. For the toluene example given in Figure 7 (Section 7.4.3), RR would be equal to 1.174. For this RR value, the toluene calibration curve given in Figure 6 indicates a concentration of 31.8 µg/L.

13.2 Internal standard: For the compounds for which the system was calibrated (Table 1) according to Section 7.5, use the response factor determined during the calibration to calculate the concentration from the following equation.

$$Concentration = \frac{(A_s \times C_{is})}{(A_{is} \times RF)}$$

where the terms are as defined in Section 7.5.1. For the compounds for which the system was not calibrated (Table 2), use the response factors in Table 5 to calculate the concentration.

13.3 The concentration of the pollutant in the solid phase of the sample is computed using the concentration of the pollutant detected in the aqueous solution, as follows:

$$Concentration\ in\ solid\ (\mu g/kg) = \frac{0.005\ L \times aqueous\ conc\ (\mu g/L)}{0.01 \times percent\ solids(g)}$$

where

"percent solids" is from Section 10.1.3

13.4 Dilution of samples: If the EICP area at the quantitation m/z exceeds the calibration range of the system, samples are diluted by successive factors of 10 until the area is within the calibration range.

13.4.1 For aqueous samples, bring 0.50 mL, 0.050 mL, 0.0050 mL, etc., to 5-mL volume with reagent water and analyze per Section 10.4.

13.4.2 For samples containing high solids, substitute 0.50 or 0.050 g in Section 10.5.2 to achieve a factor of 10 or 100 dilution, respectively.

13.4.3 If dilution of high solids samples by greater than a factor of 100 is required, then extract the sample with methanol, as described in Section 10.6.

13.5 Dilution of samples containing high concentrations of compounds not in Table 1: When the EICP area of the quantitation m/z of a compound to be identified per Section 12.3 exceeds the linear range of the GCMS system, or when any peak in the mass spectrum is saturated, dilute the sample per Sections 13.4.1 through 13.4.3.

13.6 Report results for all pollutants, labeled compounds, and tentatively identified compounds found in all standards, blanks, and samples to three significant figures. For samples containing less than 1% solids, the units are µg/L; and for undiluted samples containing 1% solids or greater, units are µg/kg.

13.6.1 Results for samples which have been diluted are reported at the least dilute level at which the area at the quantitation m/z is within the calibration range (Section 13.4), or at which no m/z in the spectrum is saturated (Section 13.5). For compounds having a labeled analog, results are reported at the least dilute level at which the area at the quantitation m/z is within the calibration range (Section 13.4) and the labeled compound recovery is within the normal range for the method (Section 14.2).

14. ANALYSIS OF COMPLEX SAMPLES

14.1 Some samples may contain high levels (>1000 µg/kg) of the compounds of interest and of interfering compounds. Some samples will foam excessively when purged. Others will overload the trap or the GC column.

14.2 When the recovery of any labeled compound is outside the range given in Table 6, dilute 0.5 mL of samples containing less than 1% solids, or 0.5 g of samples containing 1% solids or greater, with 4.5 mL of reagent water and analyze this diluted sample. If the recovery remains outside of the range for this diluted sample, the aqueous performance standard shall be analyzed (Section 11) and calibration verified (Section 11.5). If the recovery for the labeled compound in the aqueous performance standard is outside the range given in Table 6, the analytical system is out of control. In this case, the instrument shall be repaired, the performance specifications in Section 11 shall be met, and the analysis of the undiluted sample shall be repeated. If the recovery for the aqueous performance standard is within the range given in Table 6, then the method does not apply to the sample being analyzed, and the result may not be reported for regulatory compliance purposes.

14.3 When a high level of the pollutant is present, reverse search computer programs may misinterpret the spectrum of chromatographically unresolved pollutant and labeled compound pairs with overlapping spectra. Examine each chromatogram for peaks greater than the height of the internal standard peaks. These peaks can obscure the compounds of interest.

15. METHOD PERFORMANCE

15.1 The specifications for this method were taken from the interlaboratory validation of EPA Method 624 (Reference 10). Method 1624 has been shown to yield slightly better performance on treated effluents than method 624. Results of initial tests of this method at a purge temperature of 80°C can be found in Reference 11 and results of initial tests of this method on municipal sludge can be found in Reference 12.

15.2 A chromatogram of the 20 µg/L aqueous performance standards (Sections 6.7.2 and 11.1) is shown in Figure 9.

Reference

1. "Performance Tests for the Evaluation of Computerized Gas Chromatography/Mass Spectrometry Equipment and Laboratories," USEPA, EMSL Cincinnati, OH 45268, EPA-600/4-80-025 (April 1980).
2. Bellar, T. A. and Lichtenberg, J. J., "Journal American Water Works Association," 66, 739 (1974).
3. Bellar, T. A. and Lichtenberg, J. J., "Semi-Automated Headspace Analysis of Drinking Waters and Industrial Waters for Purgeable Volatile Organic Compounds," in *Measurement of Organic Pollutants in Water and Wastewater*, C. E. VanHall, ed., American Society for Testing Materials, Philadelphia, PA, Special Technical Publication 686, (1978).
4. National Standard Reference Data System, "Mass Spectral Tape Format," U.S. National Bureau of Standards (1979 and later attachments).
5. "Working with Carcinogens," DHEW, PHS, NIOSH, Publication 77-206 (1977).
6. "OSHA Safety and Health Standards, General Industry," 29 *CFR* 1910, OSHA 2206, (1976).
7. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety (1979).
8. "Handbook of Analytical Quality Control in Water and Wastewater Laboratories," USEPA, EMSL Cincinnati, OH 45268, EPA-4-79-019 (March 1979).
9. "Methods 330.4 and 330.5 for Total Residual Chlorine," USEPA, EMSL Cincinnati, OH 45268, EPA-4-79-020 (March 1979).
10. "Method 624--Purgeables", 40 *CFR* Part 136 (49 *FR* 43234), 26 October 1984.
11. "Narrative for SAS 106: Development of an Isotope Dilution GC/MS Method for Hot Purge and-Trap Volatiles Analysis," S-CUBED Division of Maxwell Laboratories, Inc., Prepared for W. A. Telliard, Industrial Technology Division (WH-552), USEPA, 401 M St. SW, Washington DC 20460 (July 1986).
12. Colby, Bruce N. and Ryan, Philip W., "Initial Evaluation of Methods 1634 and 1635 for the Analysis of Municipal Wastewater Treatment Sludges by Isotope Dilution GCMS," Pacific Analytical Inc., Prepared for W. A. Telliard, Industrial Technology Division (WH-552), USEPA, 401 M St. SW, Washington DC 20460 (July 1986).

532 allyl alcohol

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
42	30	43	39	44	232	45	12	53	13	55	59
56	58	57	1000	58	300	61	15				

533 carbon disulfide

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
44	282	46	10	64	14	76	1000	77	27	78	82

534 2-chloro-1,3-butadiene (chloroprene)

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
48	21	49	91	50	223	51	246	52	241	53	1000
54	41	61	30	62	54	63	11	64	16	73	21
87	12	88	452	89	22	90	137				

535 chloroacetonitrile

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
47	135	48	1000	49	88	50	294	51	12	73	22
74	43	75	884	76	39	77	278				

536 3-chloropropene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
35	39	36	40	40	44	42	206	47	40	58	35
49	176	51	64	52	31	61	29	73	22	75	138
76	1000	77	74	78	324						

537 crotonaldehyde

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
35	26	40	28	42	339	43	48	44	335	49	27
50	40	51	20	52	21	53	31	55	55	68	24
69	511	70	1000	71	43						

538 1,2-dibromoethane (EDB)

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
79	50	80	13	31	51	82	15	93	54	95	42
105	32	106	29	107	1000	108	38	109	922	110	19
186	13	188	27	190	13						

539 dibromomethane

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
43	99	44	101	45	30	79	184	80	35	81	175
91	142	92	61	93	1000	94	64	95	875	160	18
172	375	173	14	174	719	175	12	176	342		

540 trans-1,4-dichloro-2-butene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
49	166	50	171	51	289	52	85	53	878	54	273
62	286	64	91	75	1000	77	323	88	246	89	415
90	93	91	129	124	138	126	86	128	12		

541 1,3-dichloropropane

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
40	15	42	44	47	19	48	20	49	193	51	55
61	18	62	22	63	131	65	38	75	47	76	1000
77	46	78	310	79	12						

542 cis-1,3-dichloropropene

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
37	262	38	269	39	998	49	596	51	189	75	1000
77	328	110	254	112	161						

543 ethyl cyanide

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
44	115	50	34	51	166	52	190	53	127	54	1000
55	193										

544 ethyl methacrylate

m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
42	127	43	48	45	155	55	32	58	39	68	60
69	1000	70	83	71	25	85	14	86	169	87	21
96	17	99	93	113	11	114	119				

545 2-hexanone (methyl butyl ketone)											
m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
42	61	43	1000	44	24	55	12	57	130	58	382
59	21	71	36	85	37	100	56				
546 iodomethane											
m/z	int.	m/z	int.	m/z	int.	m/z.	int.	m/z	int.	m/z	int.
44	57	127	328	128	17	139	39	140	34	141	120
142	1000	143	12								
547 isobutyl alcohol											
m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
34	21	35	13	36	13	37	11	39	10	42	575
43	1000	44	42	45	21	55	40	56	37	57	21
59	25	73	12	74	63						
548 methacrylonitrile											
m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
38	24	39	21	41	26	42	100	49	19	50	60
51	214	52	446	53	19	62	24	63	59	64	136
65	55	66	400	67	1000	68	51				
549 methyl methacrylate											
m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
42	127	43	52	45	48	53	30	55	100	56	49
59	124	68	28	69	1000	70	51	82	26	85	45
98	20	99	89	100	442	101	22				
550 4-methyl-2-pentanone (methyl isoboutyl ketone; MIBK)											
m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
42	69	43	1000	44	54	53	11	55	15	56	13
57	205	58	346	59	20	67	12	69	10	85	96
100	94										
551 1,1,1,2-tetrachloroethane											
m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
47	144	49	163	60	303	61	330	62	98	82	45
84	31	95	416	96	152	97	270	98	84	117	804
121	236	131	1000	133	955	135	301				

552 trichlorofluoromethane											
m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
44	95	47	153	49	43	51	21	52	14	66	162
68	53	82	40	84	28	101	1000	102	10	103	671
105	102	117	16	119	14						
553 1,2,3-trichloropropane											
m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
49	285	51	87	61	300	62	107	63	98	75	1000
76	38	77	302	83	23	96	29	97	166	98	20
99	103	110	265	111	28	112	164	114	25		
554 vinyl acetate											
m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
36	5	42	103	43	1000	44	70	45	8	86	57
951 m-xylene											
m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
65	62	77	124	91	1000	105	245	106	580		
951 0- + p-xylene											
m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.	m/z	int.
51	88	77	131	91	1000	105	229	106	515		

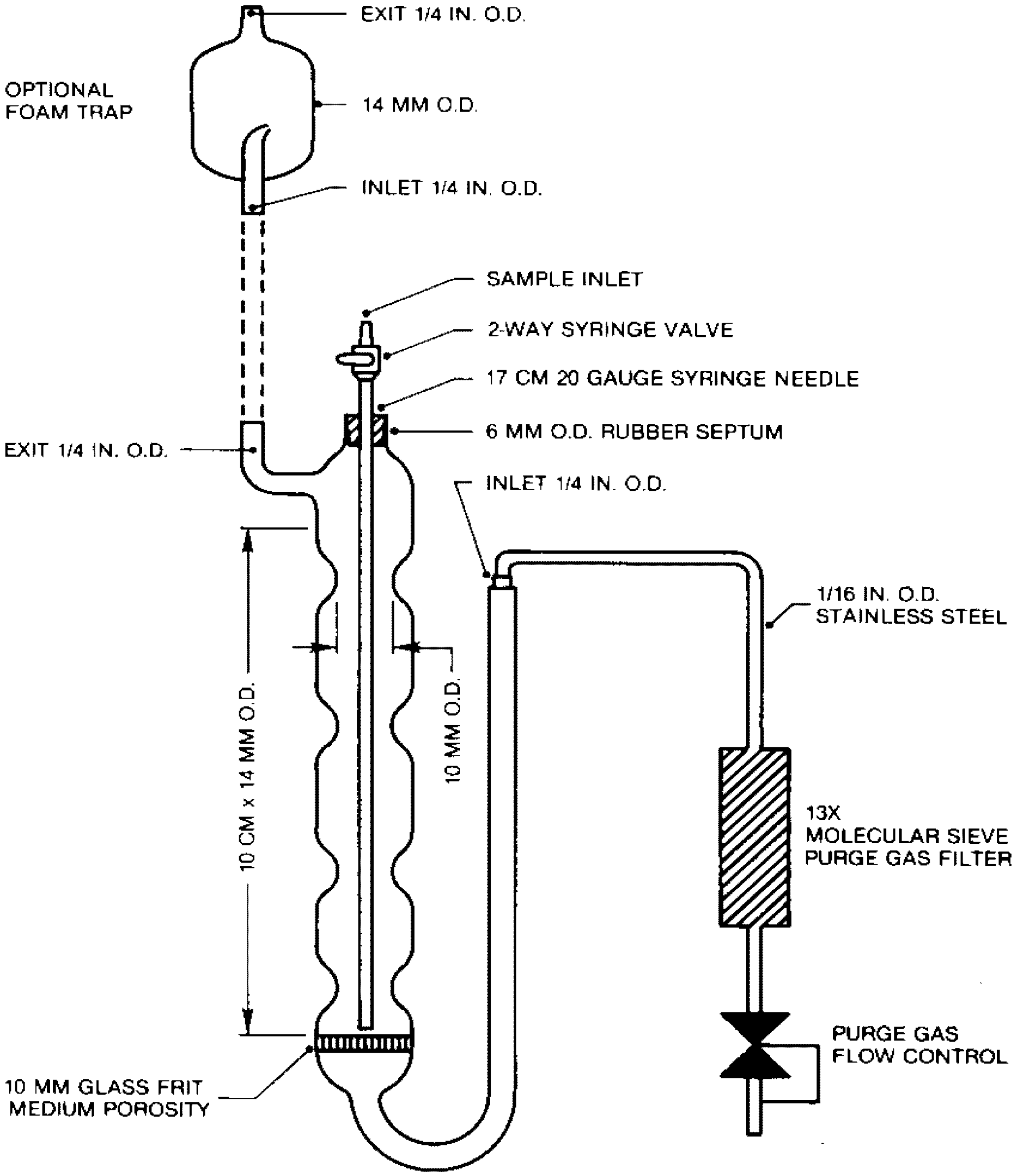


FIGURE 1 Purging Device for Waters

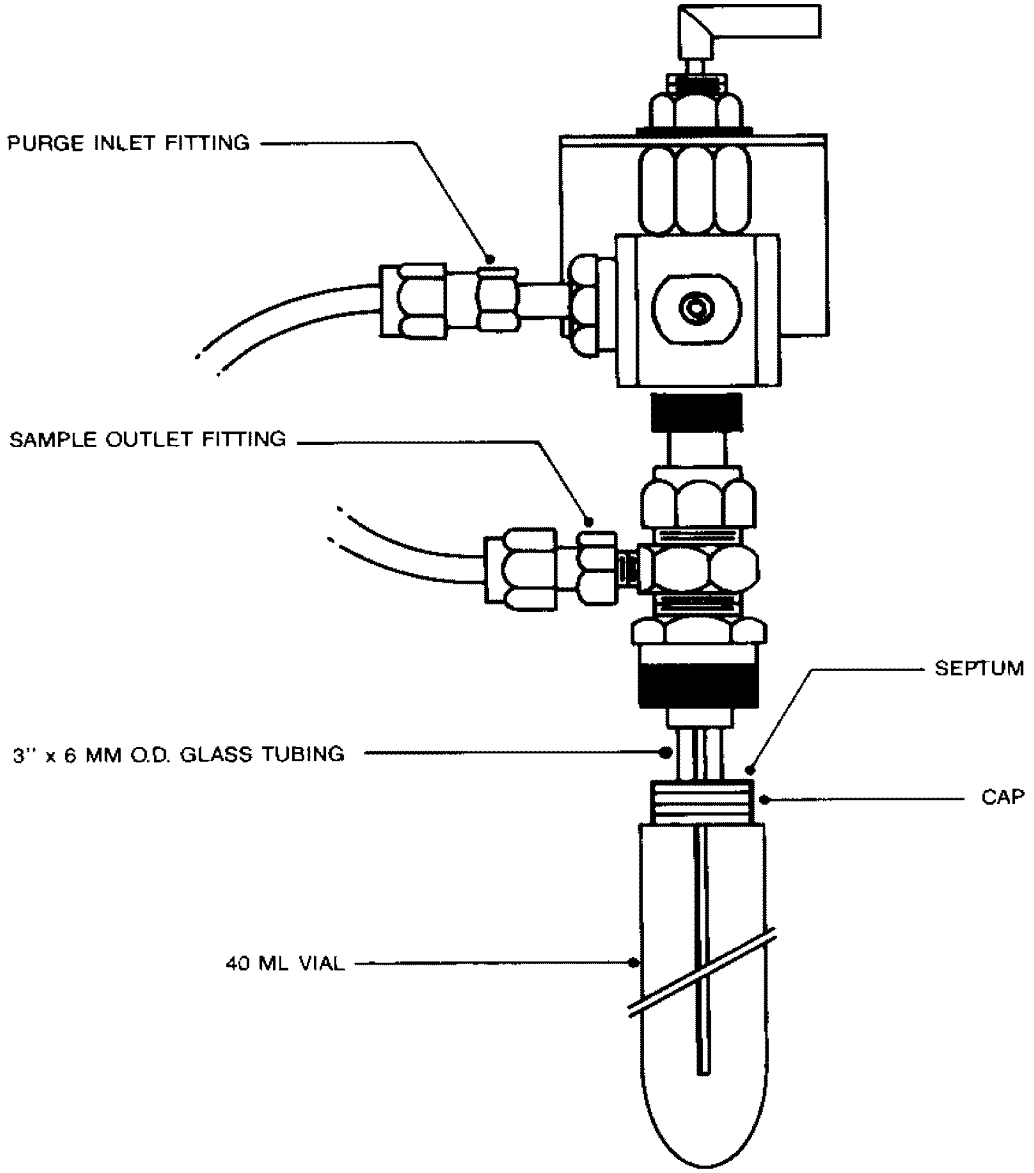


FIGURE 2 Purging Device for Soils or Waters

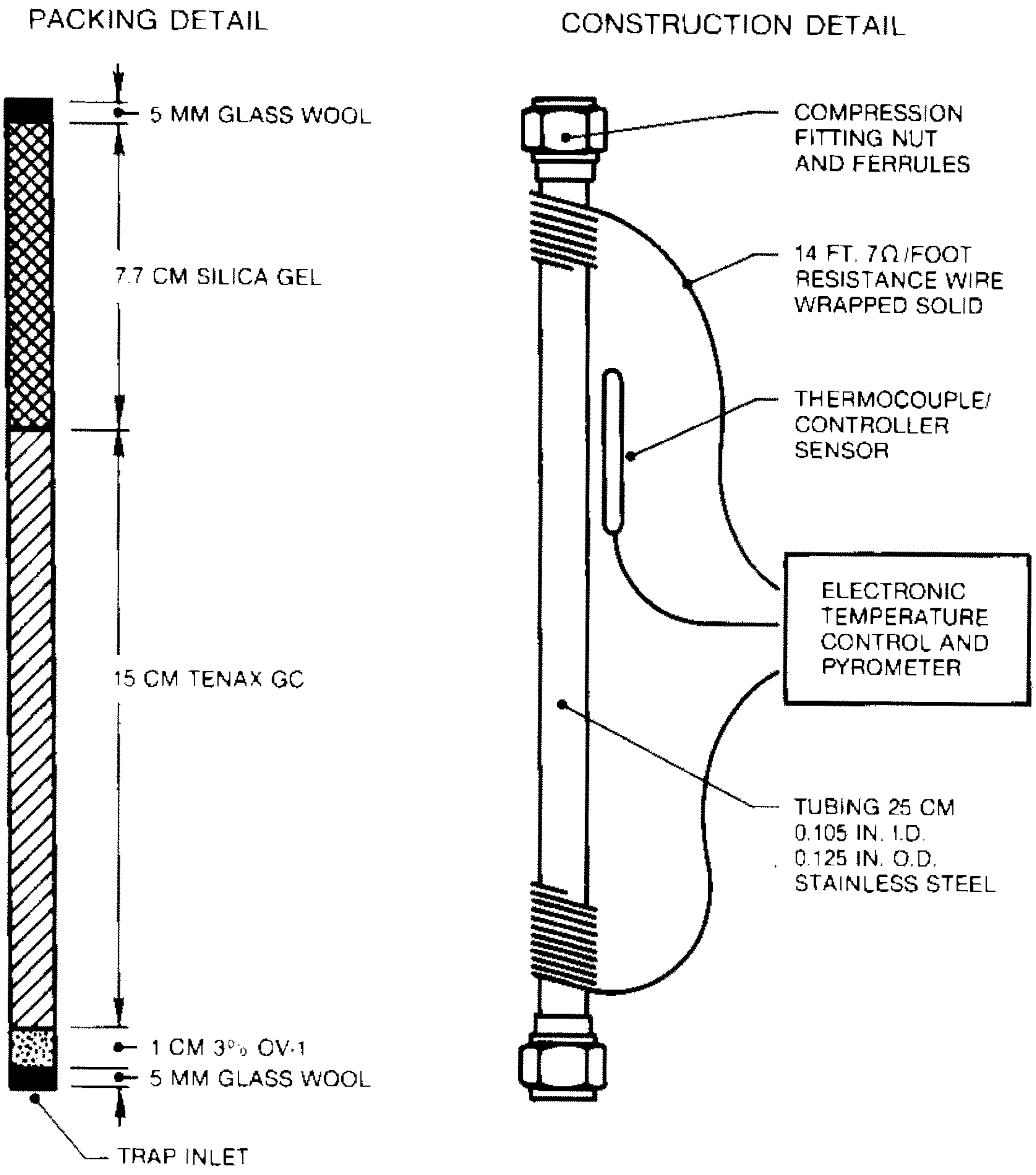


FIGURE 3 Trap Construction and Packings

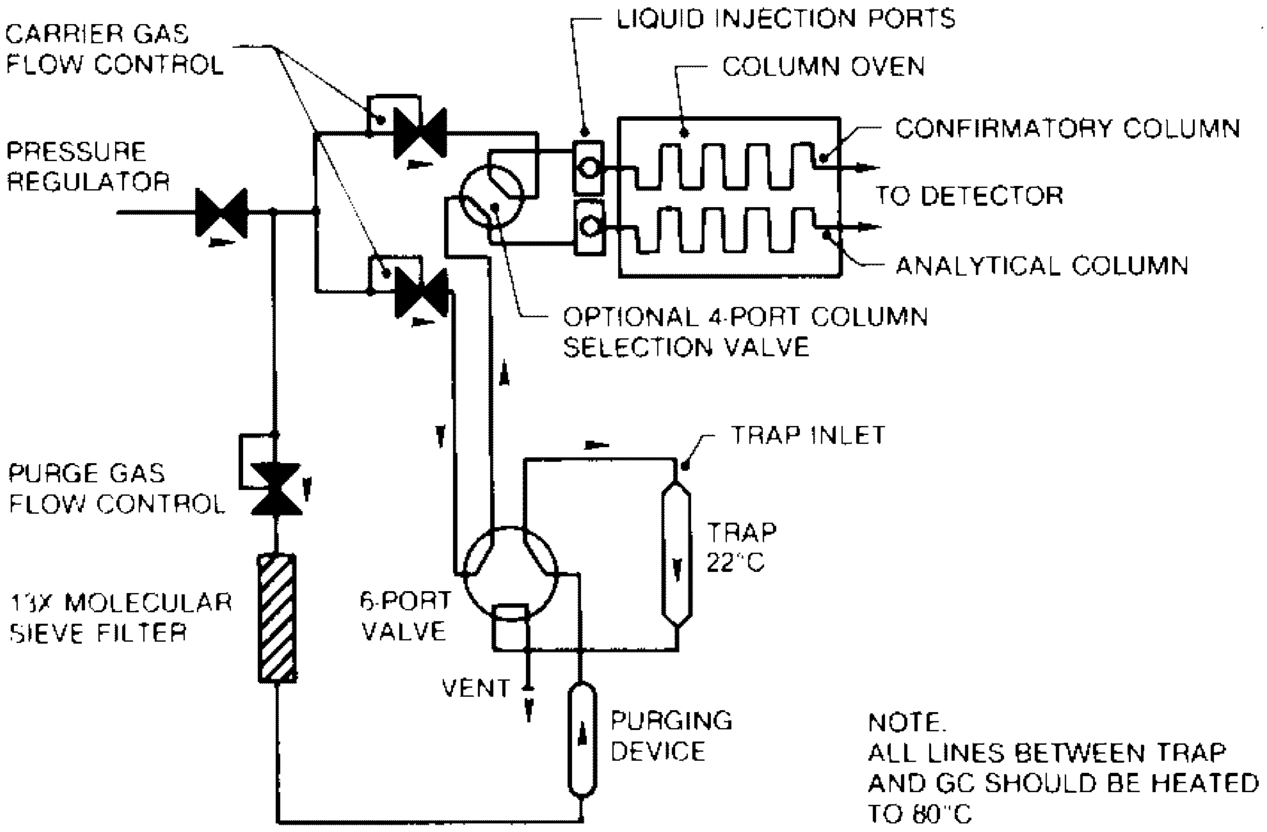


FIGURE 4 Schematic of Purge and Trap Device--Purge Mode

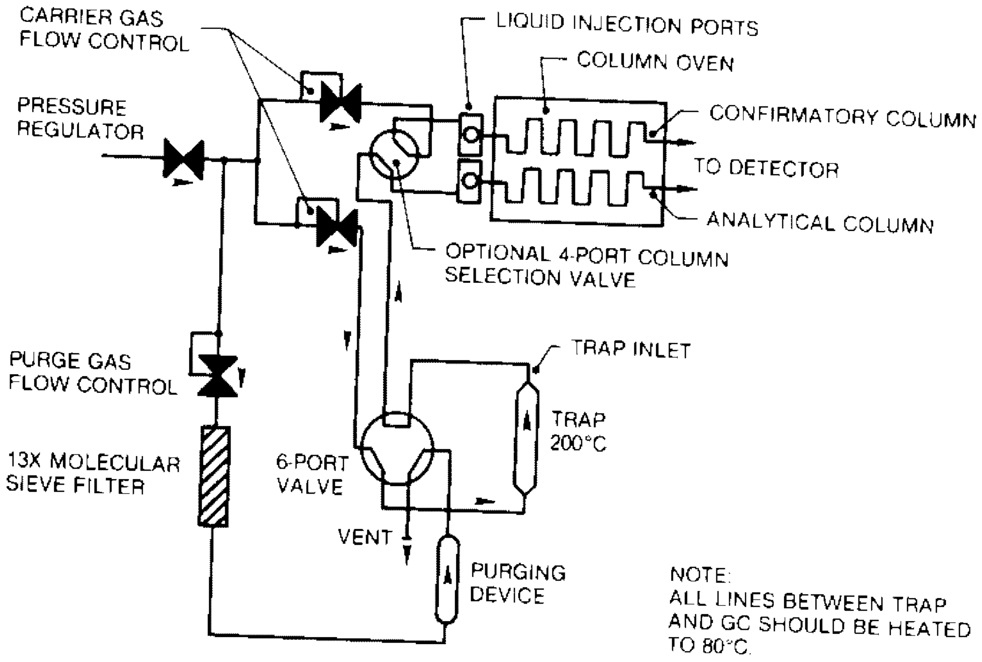


FIGURE 5 Schematic of Purge and Trap Device--Desorb Mode

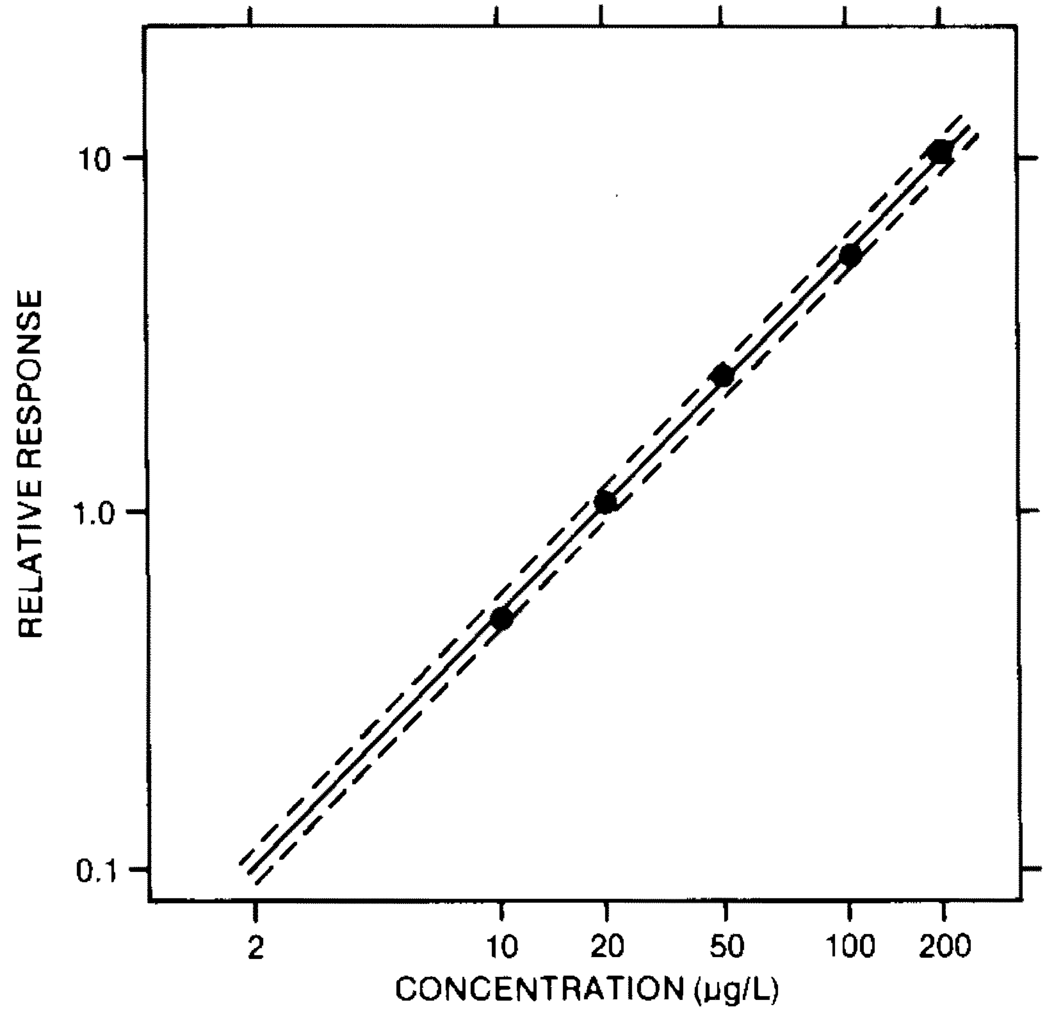


FIGURE 6 Relative Response Calibration Curve for Toluene. The Dotted Lines Enclose a +/- 10 Percent Error Window

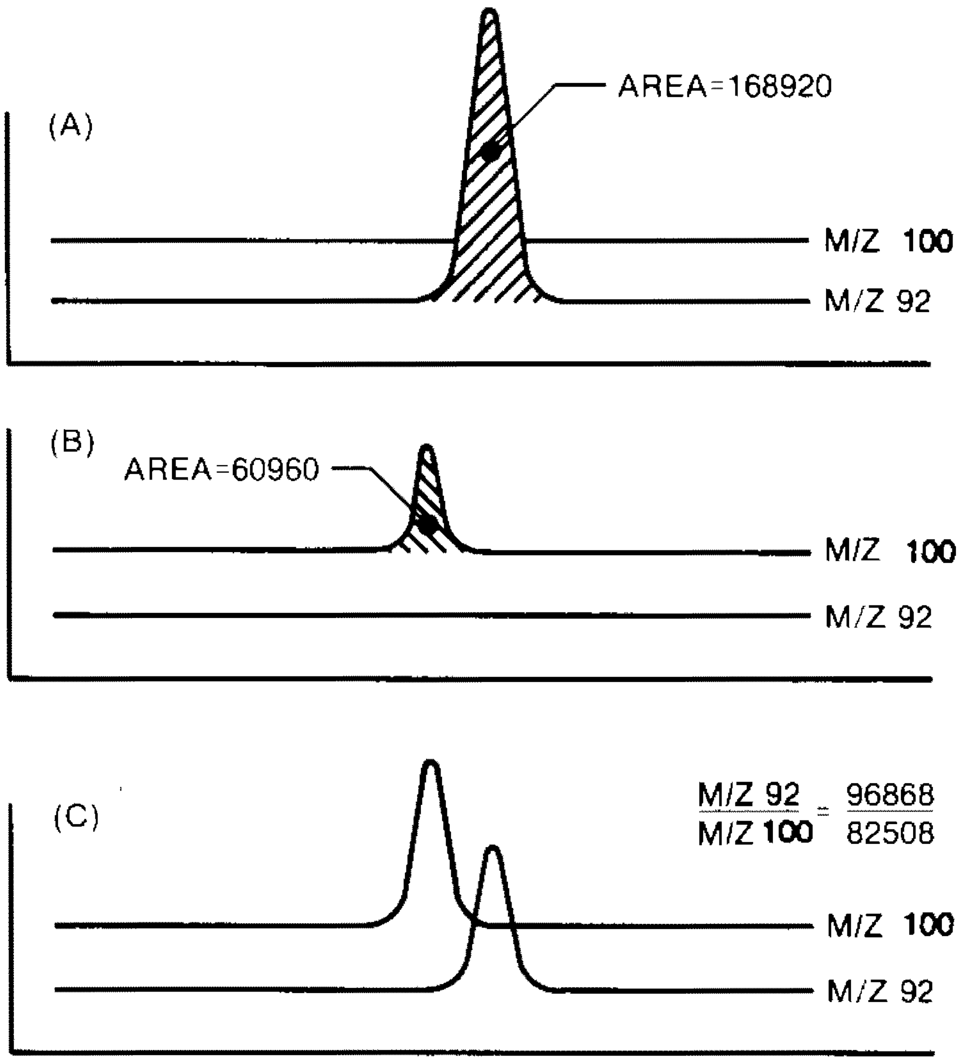


FIGURE 7 Extracted Ion Current Profiles for (A) Toluene, (B) Toluene-d_8, and (C) a Mixture of Toluene and Toluene-d_8

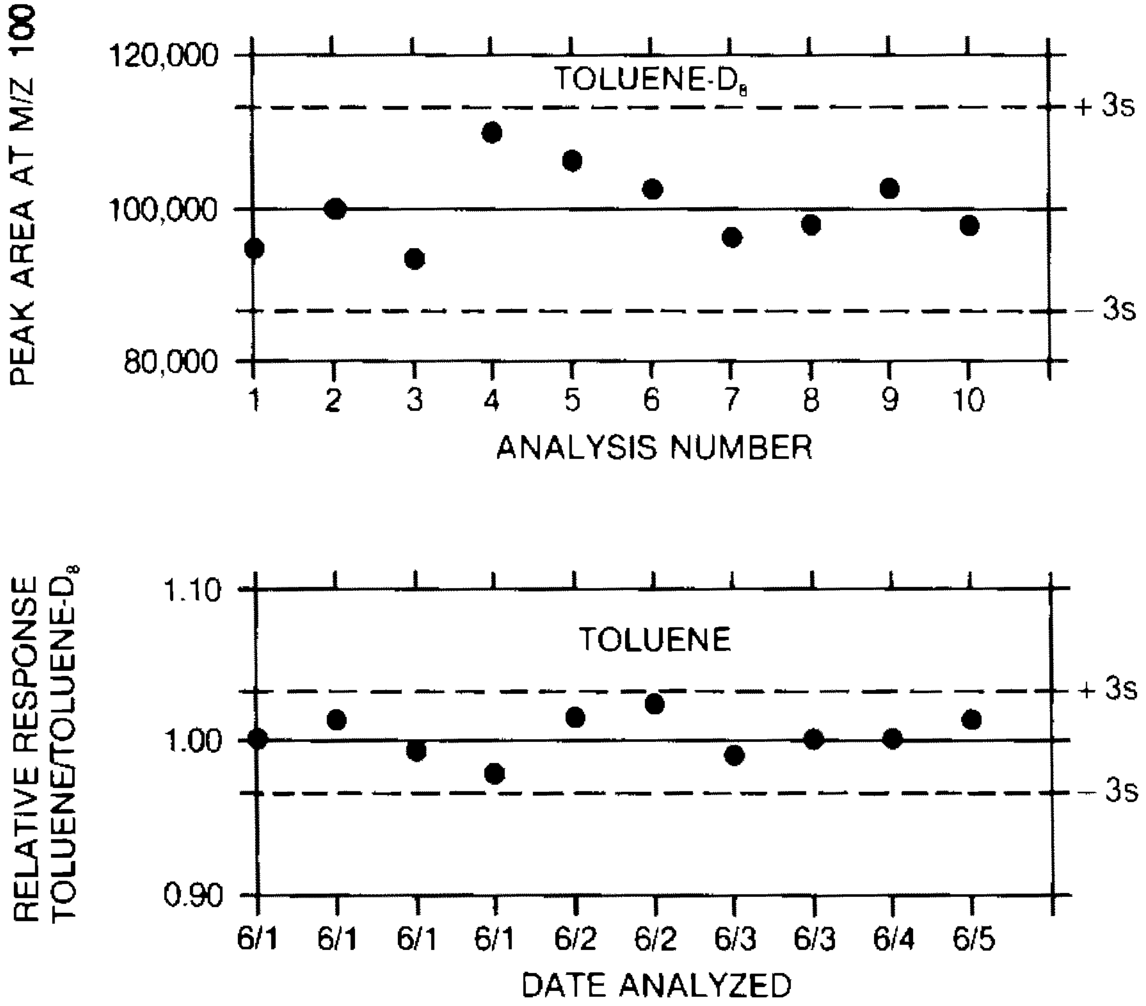
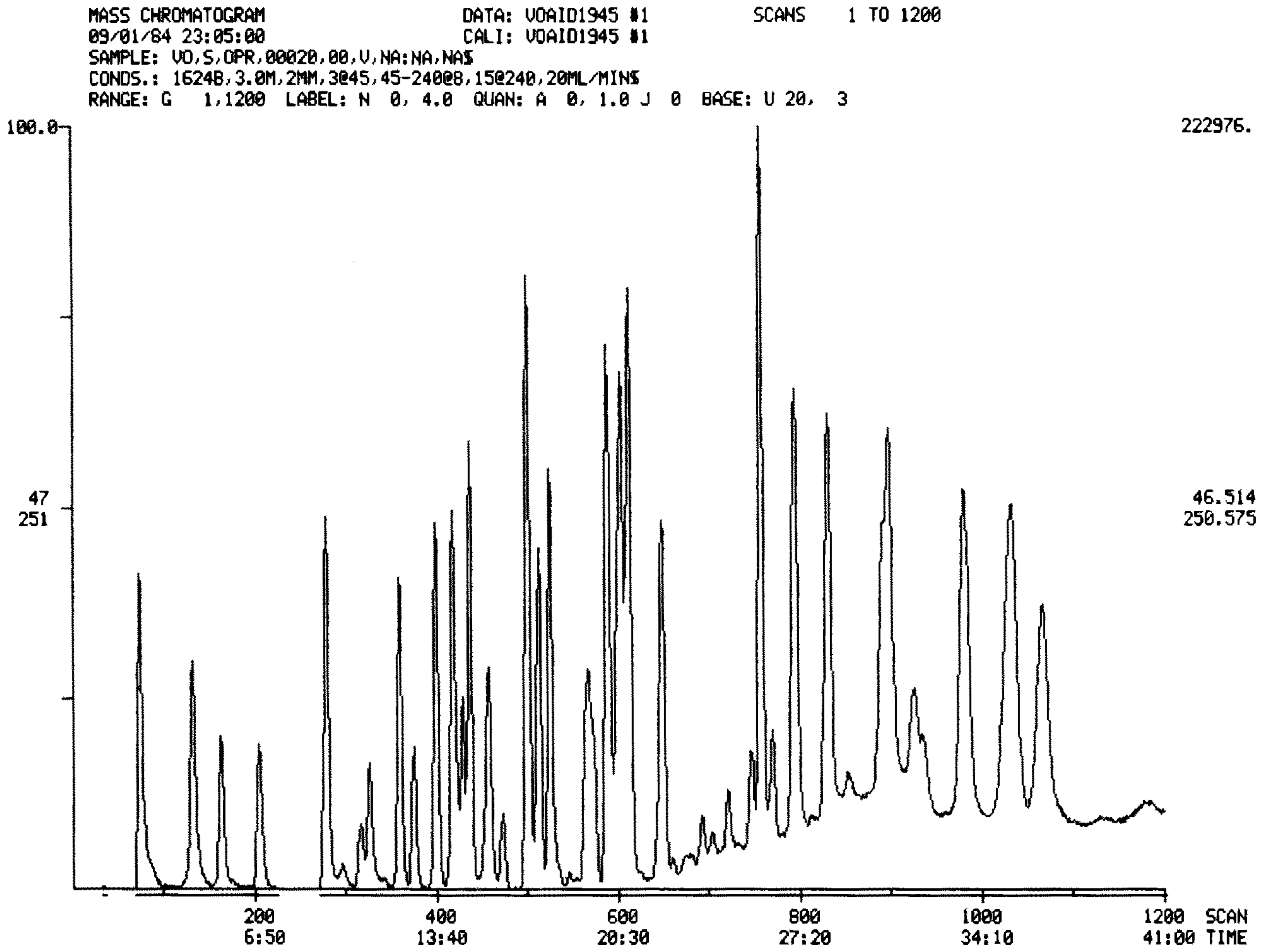


FIGURE 8 Quality Control Charts Showing Area (top graph) and Relative Response of Toluene to Toluene-d$_8$ (lower graph) Plotted as Function of Time or Analysis Number



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MASS CHROMATOGRAM                    DATA: VOAID1945 #1          SCANS    1 TO 1200
09/01/84 23:05:00                    CALI: VOAID1945 #1
SAMPLE: VO,S,OPR,00020,00,V,NA:NA,NA$
CONDS.: 1624B,3.0M,2MM,3@45,45-240@8,15@240,20ML/MIN$
RANGE: G   1,1200  LABEL: N  0, 4.0  QUAN: A  0, 1.0 J  0  BASE: U 20,  3
```

FIGURE 9 Chromatogram of Aqueous Performance Standard

METHOD 8260B
VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/ MASS SPECTROMETRY (GC/MS)

1.0 SCOPE AND APPLICATION

1.1 Method 8260 is used to determine volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including various air sampling trapping media, ground and surface water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments. The following compounds can be determined by this method:

		Appropriate Preparation Technique[a]					
Compound	CAS No.[b]	5030/ 5035	5031	5032	5021	5041	Direct Inject.
Acetone	67-64-1	pp	c	c	nd	c	c
Acetonitrile	75-05-8	pp	c	nd	nd	nd	c
Acrolein (Propenal)	107-02-8	pp	c	c	nd	nd	c
Acrylonitrile	107-13-1	pp	c	c	nd	c	c
Allyl alcohol	107-18-6	ht	c	nd	nd	nd	c
Allyl chloride	107-05-1	c	nd	nd	nd	nd	c
Benzene	71-43-2	c	nd	c	c	c	c
Benzyl chloride	100-44-7	c	nd	nd	nd	nd	c
Bis(2-chloroethyl)sulfide	505-60-2	pp	nd	nd	nd	nd	c
Bromoacetone	598-31-2	pp	nd	nd	nd	nd	c
Bromochloromethane	74-97-5	c	nd	c	c	c	c
Bromodichloromethane	75-27-4	c	nd	c	c	c	c
4-Bromofluorobenzene (surr)	460-00-4	c	nd	c	c	c	c
Bromoform	75-25-2	c	nd	c	c	c	c
Bromomethane	74-83-9	c	nd	c	c	c	c
n-Butanol	71-36-3	ht	c	nd	nd	nd	c
2-Butanone (MEK)	78-93-3	pp	c	c	nd	nd	c
t-Butyl alcohol	75-65-0	pp	c	nd	nd	nd	c
Carbon disulfide	75-15-0	pp	nd	c	nd	c	c
Carbon tetrachloride	56-23-5	c	nd	c	c	c	c
Chloral hydrate	302-17-0	pp	nd	nd	nd	nd	c
Chlorobenzene	108-90-7	c	nd	c	c	c	c
Chlorobenzene-d_5 (IS)		c	nd	c	c	c	c
Chlorodibromomethane	124-48-1	c	nd	c	nd	c	c
Chloroethane	75-00-3	c	nd	c	c	c	c
2-Chloroethanol	107-07-3	pp	nd	nd	nd	nd	c
2-Chloroethyl vinyl ether	110-75-8	c	nd	c	nd	nd	c
Chloroform	67-66-3	c	nd	c	c	c	c
Chloromethane	74-87-3	c	nd	c	c	c	c
Chloroprene	126-99-8	c	nd	nd	nd	nd	c
3-Chloropropionitrile	542-76-7	I	nd	nd	nd	nd	pc

(continued)

		Appropriate Preparation Technique[a]					
Compound	CAS No.[b]	5030/ 5035	5031	5032	5021	5041	Direct Inject.
Crotonaldehyde	4170-30-3	pp	c	nd	nd	nd	c
1,2-Dibromo-3-chloropropane	96-12-8	pp	nd	nd	c	nd	c
1,2-Dibromoethane	106-93-4	c	nd	nd	c	nd	c
Dibromomethane	74-95-3	c	nd	c	c	c	c
1,2-Dichlorobenzene	95-50-1	c	nd	nd	c	nd	c
1,3-Dichlorobenzene	541-73-1	c	nd	nd	c	nd	c
1,4-Dichlorobenzene	106-46-7	c	nd	nd	c	nd	c
1,4-Dichlorobenzene-d_4 (IS)		c	nd	nd	c	nd	c
cis-1,4-Dichloro-2-butene	1476-11-5	c	nd	c	nd	nd	c
trans-1,4-Dichloro-2-butene	110-57-6	pp	nd	c	nd	nd	c
Dichlorodifluoromethane	75-71-8	c	nd	c	c	nd	c
1,1-Dichloroethane	75-34-3	c	nd	c	c	c	c
1,2-Dichloroethane	107-06-2	c	nd	c	c	c	c
1,2-Dichloroethane-d_4 (surr)		c	nd	c	c	c	c
1,1-Dichloroethene	75-35-4	c	nd	c	c	c	c
trans-1,2-Dichloroethene	156-60-5	c	nd	c	c	c	c
1,2-Dichloropropane	78-87-5	c	nd	c	c	c	c
1,3-Dichloro-2-propanol	96-23-1	pp	nd	nd	nd	nd	c
cis-1,3-Dichloropropene	10061-01-5	c	nd	c	nd	c	c
trans-1,3-Dichloropropene	10061-02-6	c	nd	c	nd	c	c
1,2,3,4-Diepoxybutane	1464-53-5	c	nd	nd	nd	nd	c
Diethyl ether	60-29-7	c	nd	nd	nd	nd	c
1,4-Difluorobenzene (IS)	540-36-3	nd	nd	nd	nd	c	nd
1,4-Dioxane	123-91-1	pp	c	c	nd	nd	c
Epichlorohydrin	106-89-8	I	nd	nd	nd	nd	c
Ethanol	64-17-5	I	c	c	nd	nd	c
Ethyl acetate	141-78-6	I	c	nd	nd	nd	c
Ethylbenzene	100-41-4	c	nd	c	c	c	c
Ethylene oxide	75-21-8	pp	c	nd	nd	nd	c
Ethyl methacrylate	97-63-2	c	nd	c	nd	nd	c
Fluorobenzene (IS)	462-06-6	c	nd	nd	nd	nd	nd
Hexachlorobutadiene	87-68-3	c	nd	nd	c	nd	c
Hexachloroethane	67-72-1	I	nd	nd	nd	nd	c
2-Hexanone	591-78-6	pp	nd	c	nd	nd	c
2-Hydroxypropionitrile	78-97-7	I	nd	nd	nd	nd	pc
Iodomethane	74-88-4	c	nd	c	nd	c	c
Isobutyl alcohol	78-83-1	pp	c	nd	nd	nd	c
Isopropylbenzene	98-82-8	c	nd	nd	c	nd	c
Malononitrile	109-77-3	pp	nd	nd	nd	nd	c
Methacrylonitrile	126-98-7	pp	I	nd	nd	nd	c
Methanol	67-56-1	I	c	nd	nd	nd	c
Methylene chloride	75-09-2	c	nd	c	c	c	c
Methyl methacrylate	80-62-6	c	nd	nd	nd	nd	c
4-Methyl-2-pentanone (MIBK)	108-10-1	pp	c	c	nd	nd	c
Naphthalene	91-20-3	c	nd	nd	c	nd	c

(continued)

		Appropriate Preparation Technique[a]					
Compound	CAS No.[b]	5030/ 5035	5031	5032	5021	5041	Direct Inject.
Nitrobenzene	98-95-3	c	nd	nd	nd	nd	c
2-Nitropropane	79-46-9	c	nd	nd	nd	nd	c
N-Nitroso-di-n-butylamine	924-16-3	pp	c	nd	nd	nd	c
Paraldehyde	123-63-7	pp	c	nd	nd	nd	c
Pentachloroethane	76-01-7	I	nd	nd	nd	nd	c
2-Pentanone	107-87-9	pp	c	nd	nd	nd	c
2-Picoline	109-06-8	pp	c	nd	nd	nd	c
1-Propanol	71-23-8	pp	c	nd	nd	nd	c
2-Propanol	67-63-0	pp	c	nd	nd	nd	c
Propargyl alcohol	107-19-7	pp	I	nd	nd	nd	c
β-Propiolactone	57-57-8	pp	nd	nd	nd	nd	c
Propionitrile (ethyl cyanide)	107-12-0	ht	c	nd	nd	nd	pc
n-Propylamine	107-10-8	c	nd	nd	nd	nd	c
Pyridine	110-86-1	I	c	nd	nd	nd	c
Styrene	100-42-5	c	nd	c	c	c	c
1,1,1,2-Tetrachloroethane	630-20-6	c	nd	nd	c	c	c
1,1,2,2-Tetrachloroethane	79-34-5	c	nd	c	c	c	c
Tetrachloroethene	127-18-4	c	nd	c	c	c	c
Toluene	108-88-3	c	nd	c	c	c	c
Toluene-d_8 (surr)	2037-26-5	c	nd	c	c	c	c
o-Toluidine	95-53-4	pp	c	nd	nd	nd	c
1,2,4-Trichlorobenzene	120-82-1	c	nd	nd	c	nd	c
1,1,1-Trichloroethane	71-55-6	c	nd	c	c	c	c
1,1,2-Trichloroethane	79-00-5	c	nd	c	c	c	c
Trichloroethene	79-01-6	c	nd	c	c	c	c
Trichlorofluoromethane	75-69-4	c	nd	c	c	c	c
1,2,3-Trichloropropane	96-18-4	c	nd	c	c	c	c
Vinyl acetate	108-05-4	c	nd	c	nd	nd	c
Vinyl chloride	75-01-4	c	nd	c	c	c	c
o-Xylene	95-47-6	c	nd	c	c	c	c
m-Xylene	108-38-3	c	nd	c	c	c	c
p-Xylene	106-42-3	c	nd	c	c	c	c

[a] See Sec. 1.2 for other appropriate sample preparation techniques
[b] Chemical Abstract Service Registry Number

c = Adequate response by this technique
ht = Method analyte only when purged at 80°C
nd = Not determined
I = Inappropriate technique for this analyte
pc = Poor chromatographic behavior
pp = Poor purging efficiency resulting in high Estimated Quantitation Limits
surr = Surrogate
IS = Internal Standard

1.2 There are various techniques by which these compounds may be introduced into the GC/MS system. The more common techniques are listed in the table above. Purge-and-trap, by Methods 5030 (aqueous samples) and 5035 (solid and waste oil samples), is the most commonly used technique for volatile organic analytes. However, other techniques are also appropriate and necessary for some analytes. These include direct injection following dilution with hexadecane (Method 3585) for waste oil samples; automated static headspace by Method 5021 for solid samples; direct injection of an aqueous sample (concentration permitting) or injection of a sample concentrated by azeotropic distillation (Method 5031); and closed system vacuum distillation (Method 5032) for aqueous, solid, oil and tissue samples. For air samples, Method 5041 provides methodology for desorbing volatile organics from trapping media (Methods 0010, 0030, and 0031). In addition, direct analysis utilizing a sample loop is used for sub-sampling from Tedlar® bags (Method 0040). Method 5000 provides more general information on the selection of the appropriate introduction method.

1.3 Method 8260 can be used to quantitate most volatile organic compounds that have boiling points below 200°C. Volatile, water soluble compounds can be included in this analytical technique by the use of azeotropic distillation or closed-system vacuum distillation. Such compounds include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides. See Tables 1 and 2 for analytes and retention times that have been evaluated on a purge-and-trap GC/MS system. Also, the method detection limits for 25-mL sample volumes are presented. The following compounds are also amenable to analysis by Method 8260:

Bromobenzene
n-Butylbenzene
sec-Butylbenzene
tert-Butylbenzene
Chloroacetonitrile
1-Chlorobutane
1-Chlorohexane
2-Chlorotoluene
4-Chlorotoluene
Dibromofluoromethane
cis-1,2-Dichloroethene
1,3-Dichloropropane
2,2-Dichloropropane
1,1-Dichloropropene
p-Isopropyltoluene
Methyl acrylate
Methyl-t-butyl ether
Pentafluorobenzene
n-Propylbenzene
1,2,3-Trichlorobenzene
1,2,4-Trimethylbenzene
1,3,5-Trimethylbenzene

1.4 The estimated quantitation limit (EQL) of Method 8260 for an individual compound is somewhat instrument dependent and also dependent on the choice of sample preparation/introduction method. Using standard quadrapole instrumentation and the purge-and-trap technique, limits should be approximately 5 µg/kg (wet weight) for soil/sediment samples, 0.5 mg/kg (wet weight) for wastes, and 5 µg/L for ground water (see Table 3). Somewhat lower limits may be achieved using an ion trap mass spectrometer or other instrumentation of improved design. No matter which instrument is used, EQLs will be proportionately higher for sample extracts and samples that require dilution or when a reduced sample size is used to avoid saturation of the detector.

1.5 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatograph/mass spectrometers, and skilled in the interpretation of mass spectra and their use as a quantitative tool.

2.0 SUMMARY OF METHOD

2.1 The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method or by other methods (see Sec. 1.2). The analytes are introduced directly to a wide-bore capillary column or cryofocussed on a capillary pre-column before being flash evaporated to a narrow-bore capillary for analysis. The column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) interfaced to the gas chromatograph (GC).

2.2 Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or a direct connection. (Wide-bore capillary columns normally require a jet separator, whereas narrow-bore capillary columns may be directly interfaced to the ion source). Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve.

2.3 The method includes specific calibration and quality control steps that supersede the general requirements provided in Method 8000.

3.0 INTERFERENCES

3.1 Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components should be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of calibration and reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter. Subtracting blank values from sample results is not permitted. If reporting values without correcting for the blank results in what the laboratory feels is a false positive result for a sample, the laboratory should fully explained this in text accompanying the uncorrected data.

3.2 Contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. A technique to prevent this problem is to rinse the purging apparatus and sample syringes with two portions of organic-free reagent water between samples. After the analysis of a sample containing high concentrations of volatile organic compounds, one or more blanks should be analyzed to check for cross-contamination. Alternatively, if the sample immediately following the high concentration sample does not contain the volatile organic compounds present in the high level sample, freedom from contamination has been established.

3.3 For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds, or high concentrations of compounds being determined, it may be necessary to wash the purging device with a soap solution, rinse it with organic-free reagent water, and then dry the purging device in an oven at 105°C. In extreme situations, the entire purge-and-trap device may require dismantling and cleaning. Screening of the samples prior to purge-and-trap GC/MS analysis is highly recommended to prevent contamination of the system. This is especially true for soil and waste samples. Screening may be accomplished with an automated headspace technique (Method 5021) or by Method 3820 (Hexadecane Extraction and Screening of Purgeable Organics).

3.4 Many analytes exhibit low purging efficiencies from a 25-mL sample. This often results in significant amounts of these analytes remaining in the sample purge vessel after analysis. After removal of the sample aliquot that was purged, and rinsing the purge vessel three times with organic-free water, the empty vessel should be subjected to a heated purge cycle prior to the analysis of another sample in the same purge vessel. This will reduce sample-to-sample carryover.

3.5 Special precautions must be taken to analyze for methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride. Otherwise, random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing should be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean, since clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.

3.6 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample container into the sample during shipment and storage. A trip blank prepared from organic-free reagent water and carried through the sampling, handling, and storage protocols can serve as a check on such contamination.

3.7 Use of sensitive mass spectrometers to achieve lower detection level will increase the potential to detect laboratory contaminants as interferences.

3.8 Direct injection - Some contamination may be eliminated by baking out the column between analyses. Changing the injector liner will reduce the potential for cross-contamination. A portion of the analytical column may need to be removed in the case of extreme contamination. The use of direct injection will result in the need for more frequent instrument maintenance.

3.9 If hexadecane is added to waste samples or petroleum samples that are analyzed, some chromatographic peaks will elute after the target analytes. The oven temperature program must include a post-analysis bake out period to ensure that semivolatile hydrocarbons are volatilized.

4.0 APPARATUS AND MATERIALS

4.1 Purge-and-trap device for aqueous samples - Described in Method 5030.

4.2 Purge-and-trap device for solid samples - Described in Method 5035.

4.3 Automated static headspace device for solid samples - Described in Method 5021.

4.4 Azeotropic distillation apparatus for aqueous and solid samples - Described in Method 5031.

4.5 Vacuum distillation apparatus for aqueous, solid and tissue samples - Described in Method 5032.

4.6 Desorption device for air trapping media for air samples - Described in Method 5041.

4.7 Air sampling loop for sampling from Tedlar® bags for air samples - Described in Method 0040.

4.8 Injection port liners (HP Catalog #18740-80200, or equivalent) - modified for direct injection analysis by placing a 1-cm plug of glass wool approximately 50-60 mm down the length of the injection port towards the oven (see illustration below). A 0.53-mm ID column is mounted 1 cm into the liner from the oven side of the injection port, according to manufacturer's specifications.

4.9 Gas chromatography/mass spectrometer/data system

4.9.1 Gas chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection with appropriate interface for sample introduction device. The system includes all required accessories, including syringes, analytical columns, and gases.

4.9.1.1 The GC should be equipped with variable constant differential flow controllers so that the column flow rate will remain constant throughout desorption and temperature program operation.

4.9.1.2 For some column configurations, the column oven must be cooled to less than 30°C, therefore, a subambient oven controller may be necessary.

4.9.1.3 The capillary column is either directly coupled to the source or interfaced through a jet separator, depending on the size of the capillary and the requirements of the GC/MS system.

4.9.1.4 Capillary pre-column interface - This device is the interface between the sample introduction device and the capillary gas chromatograph, and is necessary when using cryogenic cooling. The interface condenses the desorbed sample components and focuses them into a narrow band on an uncoated fused-silica capillary pre-column. When the interface is flash heated, the sample is transferred to the analytical capillary column.

4.9.1.5 During the cryofocussing step, the temperature of the fused-silica in the interface is maintained at -150°C under a stream of liquid nitrogen. After the desorption period, the interface must be capable of rapid heating to 250°C in 15 seconds or less to complete the transfer of analytes.

4.9.2 Gas chromatographic columns

4.9.2.1 Column 1 - 60 m x 0.75 mm ID capillary column coated with VOCOL (Supelco), 1.5-µm film thickness, or equivalent.

4.9.2.2 Column 2 - 30 - 75 m x 0.53 mm ID capillary column coated with DB-624 (J&W Scientific), Rt$_x$-502.2 (RESTEK), or VOCOL (Supelco), 3-µm film thickness, or equivalent.

4.9.2.3 Column 3 - 30 m x 0.25 - 0.32 mm ID capillary column coated with 95% dimethyl - 5% diphenyl polysiloxane (DB-5, Rt$_x$-5, SPB-5, or equivalent), 1-µm film thickness.

4.9.2.4 Column 4 - 60 m x 0.32 mm ID capillary column coated with DB-624 (J&W Scientific), 1.8-µm film thickness, or equivalent.

4.9.3 Mass spectrometer - Capable of scanning from 35 to 300 amu every 2 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for 4-Bromofluorobenzene (BFB) which meets all of the criteria in Table 4 when 5-50 ng of the GC/MS tuning standard (BFB) are injected through the GC. To ensure sufficient precision of mass spectral data, the desirable MS scan rate allows acquisition of at least five spectra while a sample component elutes from the GC.

An ion trap mass spectrometer may be used if it is capable of axial modulation to reduce ion-molecule reactions and can produce electron impact-like spectra that match those in the EPA/NIST Library. Because ion-molecule reactions with water and methanol in an ion trap mass spectrometer may produce interferences that coelute with chloromethane and chloroethane, the base peak for both of these analytes will be at m/z 49. This ion should be used as the quantitation ion in this case. The mass spectrometer must be capable of producing a mass spectrum for BFB which meets all of the criteria in Table 3 when 5 or 50 ng are introduced.

4.9.4 GC/MS interface - Two alternatives may be used to interface the GC to the mass spectrometer.

4.9.4.1 Direct coupling, by inserting the column into the mass spectrometer, is generally used for 0.25 - 0.32 mm ID columns.

4.9.4.2 A jet separator, including an all-glass transfer line and glass enrichment device or split interface, is used with a 0.53 mm column.

4.9.4.3 Any enrichment device or transfer line may be used, if all of the performance specifications described in Sec. 8.0 (including acceptable calibration at 50 ng or less) can be achieved. GC/MS interfaces constructed entirely of glass or of glass-lined materials are recommended. Glass may be deactivated by silanizing with dichlorodimethylsilane.

4.9.5 Data system - A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program must be interfaced to the mass spectrometer. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

4.10 Microsyringes - 10-, 25-, 100-, 250-, 500-, and 1,000-µL.

4.11 Syringe valve - Two-way, with Luer ends (three each), if applicable to the purging device.

4.12 Syringes - 5-, 10-, or 25-mL, gas-tight with shutoff valve.

4.13 Balance - Analytical, capable of weighing 0.0001 g, and top-loading, capable of weighing 0.1 g.

4.14 Glass scintillation vials - 20-mL, with PTFE-lined screw-caps or glass culture tubes with PTFE-lined screw-caps.

4.15 Vials - 2-mL, for GC autosampler.

4.16 Disposable pipets - Pasteur.

4.17 Volumetric flasks, Class A - 10-mL and 100-mL, with ground-glass stoppers.

4.18 Spatula - Stainless steel.

5.0 REAGENTS

5.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all inorganic reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

5.3 Methanol, CH_3OH - Pesticide quality or equivalent, demonstrated to be free of analytes. Store apart from other solvents.

5.4 Reagent Hexadecane - Reagent hexadecane is defined as hexadecane in which interference is not observed at the method detection limit of compounds of interest. Hexadecane quality is demonstrated through the analysis of a solvent blank injected directly into the GC/MS. The results of such a blank analysis must demonstrate that all interfering volatiles have been removed from the hexadecane.

5.5 Polyethylene glycol, $H(OCH_2CH_2)_nOH$ - Free of interferences at the detection limit of the target analytes.

5.6 Hydrochloric acid (1:1 v/v), HCl - Carefully add a measured volume of concentrated HCl to an equal volume of organic-free reagent water.

5.7 Stock solutions - Stock solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol, using assayed liquids or gases, as appropriate.

5.7.1 Place about 9.8 mL of methanol in a 10-mL tared ground-glass-stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 minutes or until all alcohol-wetted surfaces have dried. Weigh the flask to the nearest 0.0001 g.

5.7.2 Add the assayed reference material, as described below.

5.7.2.1 Liquids - Using a 100-µL syringe, immediately add two or more drops of assayed reference material to the flask; then reweigh. The liquid must fall directly into the alcohol without contacting the neck of the flask.

5.7.2.2 Gases - To prepare standards for any compounds that boil below 30°C (e.g., bromomethane, chloroethane, chloromethane, or vinyl chloride), fill a 5-mL valved gas-tight syringe with the reference standard to the 5.0 mL mark. Lower the needle to

5 mm above the methanol meniscus. Slowly introduce the reference standard above the surface of the liquid. The heavy gas will rapidly dissolve in the methanol. Standards may also be prepared by using a lecture bottle equipped with a septum. Attach PTFE tubing to the side arm relief valve and direct a gentle stream of gas into the methanol meniscus.

5.7.3 Reweigh, dilute to volume, stopper, and then mix by inverting the flask several times. Calculate the concentration in milligrams per liter (mg/L) from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially-prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

5.7.4 Transfer the stock standard solution into a bottle with a PTFE-lined screw-cap. Store, with minimal headspace and protected from light, at -10°C or less or as recommended by the standard manufacturer. Standards should be returned to the freezer as soon as the analyst has completed mixing or diluting the standards to prevent the evaporation of volatile target compounds.

5.7.5 Frequency of Standard Preparation

5.7.5.1 Standards for the permanent gases should be monitored frequently by comparison to the initial calibration curve. Fresh standards should be prepared if this check exceeds a 20% drift. Standards for gases usually need to be replaced after one week or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. Dichlorodifluoromethane and dichloromethane will usually be the first compounds to evaporate from the standard and should, therefore, be monitored very closely when standards are held beyond one week.

5.7.5.2 Standards for the non-gases should be monitored frequently by comparison to the initial calibration. Fresh standards should be prepared if this check exceeds a 20% drift. Standards for non-gases usually need to be replaced after six months or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. Standards of reactive compounds such as 2-chloroethyl vinyl ether and styrene may need to be prepared more frequently.

5.7.6 Preparation of Calibration Standards From a Gas Mixture

An optional calibration procedure involves using a certified gaseous mixture daily, utilizing a commercially-available gaseous analyte mixture of bromomethane, chloromethane, chloroethane, vinyl chloride, dichloro-difluoromethane and trichlorofluoromethane in nitrogen. Mixtures of documented quality are stable for as long as six months without refrigeration. (VOA-CYL III, RESTEK Corporation, Cat. #20194 or equivalent).

5.7.6.1 Before removing the cylinder shipping cap, be sure the valve is completely closed (turn clockwise). The contents are under pressure and should be used in a well-ventilated area.

5.7.6.2 Wrap the pipe thread end of the Luer fitting with PTFE tape. Remove the shipping cap from the cylinder and replace it with the Luer fitting.

5.7.6.3 Transfer half the working standard containing other analytes, internal standards, and surrogates to the purge apparatus.

5.7.6.4 Purge the Luer fitting and stem on the gas cylinder prior to sample removal using the following sequence:

a) Connect either the 100-µL or 500-µL Luer syringe to the inlet fitting of the cylinder.

b) Make sure the on/off valve on the syringe is in the open position.

c) Slowly open the valve on the cylinder and withdraw a full syringe volume.

d) Be sure to close the valve on the cylinder before you withdraw the syringe from the Luer fitting.

e) Expel the gas from the syringe into a well-ventilated area.

f) Repeat steps a through e one more time to fully purge the fitting.

5.7.6.5 Once the fitting and stem have been purged, quickly withdraw the volume of gas you require using steps 5.6.6.1.4(a) through (d). Be sure to close the valve on the cylinder and syringe before you withdraw the syringe from the Luer fitting.

5.7.6.6 Open the syringe on/off valve for 5 seconds to reduce the syringe pressure to atmospheric pressure. The pressure in the cylinder is ~30 psi.

5.7.6.7 The gas mixture should be quickly transferred into the reagent water through the female Luer fitting located above the purging vessel.

NOTE: Make sure the arrow on the 4-way valve is pointing toward the female Luer fitting when transferring the sample from the syringe. Be sure to switch the 4-way valve back to the closed position before removing the syringe from the Luer fitting.

5.7.6.8 Transfer the remaining half of the working standard into the purging vessel. This procedure insures that the total volume of gas mix is flushed into the purging vessel, with none remaining in the valve or lines.

5.7.6.9 The concentration of each compound in the cylinder is typically 0.0025 µg/µL.

5.7.6.10 The following are the recommended gas volumes spiked into 5 mL of water to produce a typical 5-point calibration:

Gas Volume	Calibration Concentration
40 µL	20 µg/L
100 µL	50 µg/L
200 µL	100 µg/L
300 µL	150 µg/L
400 µL	200 µg/L

5.7.6.11 The following are the recommended gas volumes spiked into 25 mL of water to produce a typical 5-point calibration:

Gas Volume	Calibration Concentration
10 µL	1 µg/L
20 µL	2 µg/L
50 µL	5 µg/L
100 µL	10 µg/L
250 µL	25 µg/L

5.8 Secondary dilution standards - Using stock standard solutions, prepare secondary dilution standards in methanol containing the compounds of interest, either singly or mixed together. Secondary dilution standards must be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Store in a vial with no headspace. Replace after one week. Secondary standards for gases should be replaced after one week unless the acceptability of the standard can be documented. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations. The analyst should also handle and store standards as stated in Sec. 5.7.4 and return them to the freezer as soon as standard mixing or diluting is completed to prevent the evaporation of volatile target compounds.

5.9 Surrogate standards - The recommended surrogates are toluene-d_8, 4-bromofluorobenzene, 1,2-dichloroethane-d_4, and dibromofluoromethane. Other compounds may be used as surrogates, depending upon the analysis requirements. A stock surrogate solution in methanol should be prepared as described above, and a surrogate standard spiking solution should be prepared from the stock at a concentration of 50-250 µg/10 mL, in methanol. Each sample undergoing GC/MS analysis must be spiked with 10 µL of the surrogate spiking solution prior to analysis. If a more sensitive mass spectrometer is employed to achieve lower detection levels, then more dilute surrogate solutions may be required.

5.10 Internal standards - The recommended internal standards are fluorobenzene, chlorobenzene-d_5, and 1,4-dichlorobenzene-d_4. Other compounds may be used as internal standards as long as they have retention times similar to the compounds being detected by GC/MS. Prepare internal standard stock and secondary dilution standards in methanol using the procedures described in Secs. 5.7 and 5.8. It is recommended that the secondary dilution standard be prepared at a concentration of 25 mg/L of each internal standard compound. Addition of 10 µL of this standard to 5.0 mL of sample or calibration standard would be the equivalent of 50 µg/L. If a more sensitive mass spectrometer is employed to achieve lower detection levels, then more dilute internal standard solutions may be required. Area counts of the internal standard peaks should be between 50-200% of the areas of the target analytes in the mid-point calibration analysis.

5.11 4-Bromofluorobenzene (BFB) standard - A standard solution containing 25 ng/µL of BFB in methanol should be prepared. If a more sensitive mass spectrometer is employed to achieve lower detection levels, then a more dilute BFB standard solution may be required.

5.12 Calibration standards -There are two types of calibration standards used for this method: initial calibration standards and calibration verification standards. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

5.12.1 Initial calibration standards should be prepared at a minimum of five different concentrations from the secondary dilution of stock standards (see Secs. 5.7 and 5.8) or from a premixed certified solution. Prepare these solutions in organic-free reagent water. At least one of the calibration standards should correspond to a sample concentration at or below that necessary to meet the data quality objectives of the project. The remaining standards should correspond to the range of concentrations found in typical samples but should not exceed the working range of the GC/MS system. Initial calibration standards should be mixed from fresh stock standards and dilution standards when generating an initial calibration curve.

5.12.2 Calibration verification standards should be prepared at a concentration near the mid-point of the initial calibration range from the secondary dilution of stock standards (see Secs. 5.7 and 5.8) or from a premixed certified solution. Prepare these solutions in organic-free reagent water. See Sec. 7.4 for guidance on calibration verification.

5.12.3 It is the intent of EPA that all target analytes for a particular analysis be included in the initial calibration and calibration verification standard(s). These target analytes may not include the entire list of analytes (Sec. 1.1) for which the method has been demonstrated. However, the laboratory shall not report a quantitative result for a target analyte that was not included in the calibration standard(s).

5.12.4 The calibration standards must also contain the internal standards chosen for the analysis.

5.13 Matrix spiking and laboratory control sample (LCS) standards - Matrix spiking standards should be prepared from volatile organic compounds which are representative of the compounds being investigated. At a minimum, the matrix spike should include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. The matrix spiking solution should contain compounds that are expected to be found in the types of samples to be analyzed.

5.13.1 Some permits may require the spiking of specific compounds of interest, especially if polar compounds are a concern, since the spiking compounds listed above would not be representative of such compounds. The standard should be prepared in methanol, with each compound present at a concentration of 250 µg/10.0 mL.

5.13.2 The spiking solutions should not be prepared from the same standards as the calibration standards. However, the same spiking standard prepared for the matrix spike may be used for the LCS.

5.13.3 If a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute matrix spiking solutions may be required.

5.14 Great care must be taken to maintain the integrity of all standard solutions. It is recommended all standards in methanol be stored at -10°C or less, in amber bottles with PTFE-lined screw-caps.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

See the introductory material to this chapter, Organic Analytes, Sec. 4.1.

7.0 PROCEDURE

7.1 Various alternative methods are provided for sample introduction. All internal standards, surrogates, and matrix spiking compounds (when applicable) must be added to the samples before introduction into the GC/MS system. Consult the sample introduction method for the procedures by which to add such standards.

7.1.1 Direct injection - This includes: injection of an aqueous sample containing a very high concentration of analytes; injection of aqueous concentrates from Method 5031 (azeotropic distillation); and injection of a waste oil diluted 1:1 with hexadecane (Method 3585). Direct injection of aqueous samples (non-concentrated) has very limited applications. It is only used for the determination of volatiles at the toxicity characteristic (TC) regulatory limits or at concentrations in excess of 10,000 µg/L. It may also be used in conjunction with the test for ignitability in aqueous samples (along with Methods 1010 and 1020), to determine if alcohol is present at greater than 24%.

7.1.2 Purge-and-trap - This includes purge-and-trap for aqueous samples (Method 5030) and purge-and-trap for solid samples (Method 5035). Method 5035 also provides techniques for extraction of high concentration solid and oily waste samples by methanol (and other water-miscible solvents) with subsequent purge-and-trap from an aqueous matrix using Method 5030.

7.1.2.1 Traditionally, the purge-and-trap of aqueous samples is performed at ambient temperature, while purging of soil/solid samples is performed at 40°C, to improve purging efficiency.

7.1.2.2 Aqueous and soil/solid samples may also be purged at temperatures above those being recommended as long as all calibration standards, samples, and QC samples are purged at the same temperature, appropriate trapping material is used to handle the excess water, and the laboratory demonstrates acceptable method performance for the project. Purging of aqueous samples at elevated temperatures (e.g., 40°C) may improve the purging performance of many of the water soluble compounds which have poor purging efficiencies at ambient temperatures.

7.1.3 Vacuum distillation - this technique may be used for the introduction of volatile organics from aqueous, solid, or tissue samples (Method 5032) into the GC/MS system.

7.1.4 Automated static headspace - this technique may be used for the introduction of volatile organics from solid samples (Method 5021) into the GC/MS system.

7.1.5 Cartridge desorption - this technique may be for the introduction of volatile organics from sorbent cartridges (Method 5041) used in the sampling of air. The sorbent cartridges are from the volatile organics sampling train (VOST) or SMVOC (Method 0031).

7.2 Recommended chromatographic conditions

7.2.1 General conditions

Injector temperature:	200 - 225°C
Transfer line temperature:	250 - 300°C

7.2.2 Column 1 and Column 2 with cryogenic cooling (example chromatograms are presented in Figures 1 and 2)

Carrier gas (He) flow rate:	15 mL/min
Initial temperature:	10°C, hold for 5 minutes
Temperature program:	6°C/min to 70°C, then 15°C/min to 145°C
Final temperature:	145°C, hold until all expected compounds have eluted.

7.2.5 Direct injection - Column 2

Carrier gas (He) flow rate:	4 mL/min
Column:	J&W DB-624, 70m x 0.53 mm
Initial temperature:	40°C, hold for 3 minutes
Temperature program:	8°C/min
Final temperature:	260°C, hold until all expected compounds have eluted.
Column Bake out:	75 minutes
Injector temperature:	200-225°C
Transfer line temperature:	250-300°C

7.2.6 Direct split interface - Column 4

Carrier gas (He) flow rate:	1.5 mL/min
Initial temperature:	35°C, hold for 2 minutes
Temperature program:	4°C/min to 50°C 10°C/min to 220°C
Final temperature:	220°C, hold until all expected compounds have eluted
Split ratio:	100:1
Injector temperature:	125°C

7.3 Initial calibration

Establish the GC/MS operating conditions, using the following as guidance:

Mass range:	35 - 260 amu
Scan time:	0.6 - 2 sec/scan
Source temperature:	According to manufacturer's specifications
Ion trap only:	Set axial modulation, manifold temperature, and emission current to manufacturer's recommendations

7.3.1 Each GC/MS system must be hardware-tuned to meet the criteria in Table 4 for a 5-50 ng injection or purging of 4-bromofluorobenzene (2-µL injection of the BFB standard). Analyses must not begin until these criteria are met.

7.3.1.1 In the absence of specific recommendations on how to acquire the mass spectrum of BFB from the instrument manufacturer, the following approach has been shown to be useful: The mass spectrum of BFB may be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of

BFB. Do not background subtract part of the BFB peak. Alternatively, the analyst may use other documented approaches suggested by the instrument manufacturer.

7.3.1.2 Use the BFB mass intensity criteria in Table 4 as tuning acceptance criteria. Alternatively, other documented tuning criteria may be used (e.g., CLP, Method 524.2, or manufacturer's instructions), provided that method performance is not adversely affected.

NOTE: All subsequent standards, samples, MS/MSDs, LCSs, and blanks associated with a BFB analysis must use identical mass spectrometer instrument conditions.

7.3.2 Set up the sample introduction system as outlined in the method of choice (see Sec. 7.1). A different calibration curve is necessary for each method because of the differences in conditions and equipment. A set of at least five different calibration standards is necessary (see Sec. 5.12 and Method 8000). Calibration must be performed using the sample introduction technique that will be used for samples. For Method 5030, the purging efficiency for 5 mL of water is greater than for 25 mL. Therefore, develop the standard curve with whichever volume of sample that will be analyzed.

7.3.2.1 To prepare a calibration standard, add an appropriate volume of a secondary dilution standard solution to an aliquot of organic-free reagent water in a volumetric flask. Use a microsyringe and rapidly inject the alcoholic standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection. Mix by inverting the flask three times only. Discard the contents contained in the neck of the flask. Aqueous standards are not stable and should be prepared daily. Transfer 5.0 mL (or 25 mL if lower detection limits are required) of each standard to a gas tight syringe along with 10 µL of internal standard. Then transfer the contents to the appropriate device or syringe. Some of the introduction methods may have specific guidance on the volume of calibration standard and the way the standards are transferred to the device.

7.3.2.2 The internal standards selected in Sec. 5.10 should permit most of the components of interest in a chromatogram to have retention times of 0.80 - 1.20, relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation (see Table 1). If interferences are noted, use the next most intense ion as the quantitation ion.

7.3.2.3 To prepare a calibration standard for direct injection analysis of waste oil, dilute standards in hexadecane.

7.3.3 Proceed with the analysis of the calibration standards following the procedure in the introduction method of choice. For direct injection, inject 1 - 2 µL into the GC/MS system. The injection volume will depend upon the chromatographic column chosen and the tolerance of the specific GC/MS system to water.

7.3.4 Tabulate the area response of the characteristic ions (see Table 5) against the concentration for each target analyte and each internal standard. Calculate response factors (RF) for each target analyte relative to one of the internal standards. The internal standard selected for the calculation of the RF for a target analyte should be the internal standard that has a retention time closest to the analyte being measured (Sec. 7.6.2).

The RF is calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

A_s = Peak area (or height) of the analyte or surrogate.
A_{is} = Peak area (or height) of the internal standard.
C_s = Concentration of the analyte or surrogate.
C_{is} = Concentration of the internal standard.

7.3.5 System performance check compounds (SPCCs) - Calculate the mean RF for each target analyte using the five RF values calculated from the initial (5-point) calibration curve. A system performance check should be made before this calibration curve is used. Five compounds (the System Performance Check Compounds, or SPCCs) are checked for a minimum average response factor. These compounds are chloromethane; 1,1-dichloroethane; bromoform; chlorobenzene; and 1,1,2,2-tetrachloroethane. These compounds are used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system. Example problems include:

7.3.5.1 Chloromethane is the most likely compound to be lost if the purge flow is too fast.

7.3.5.2 Bromoform is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve bromoform response.

7.3.5.3 Tetrachloroethane and 1,1-dichloroethane are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

7.3.5.4 The minimum mean response factors for the volatile SPCCs are as follows:

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

7.3.6 Calibration check compounds (CCCs)

7.3.6.1 The purpose of the CCCs are to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites on the column. Meeting the CCC criteria is not a substitute for successful calibration of the target analytes using one of the approaches described in Sec. 7.0 of Method 8000.

7.3.6.2 Calculate the standard deviation (SD) and relative standard deviation (RSD) of the response factors for all target analytes from the initial calibration, as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^{n}(RF_i - \overline{RF})^2}{n-1}} \qquad RSD = \frac{SD}{\overline{RF}} \times 100$$

where:

RF_i = RF for each of the calibration standards
$\overline{RF}$ = mean RF for each compound from the initial calibration
n = Number of calibration standards, e.g., 5

7.3.6.3 The RSD should be less than or equal to 15% for each target analyte. However, the RSD for each individual Calibration Check Compound (CCC) must be equal or less than 30%. If the CCCs are not included in the list of analytes for a project, and therefore not included in the calibration standards, refer to Sec. 7.0 of Method 8000. The CCCs are:

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl chloride

7.3.6.4 If an RSD of greater than 30% is measured for any CCC, then corrective action to eliminate a system leak and/or column reactive sites is necessary before reattempting calibration.

7.3.7 Evaluation of retention times - The relative retention times of each target analyte in each calibration standard should agree within 0.06 relative retention time units. Late-eluting compounds usually have much better agreement.

7.3.8 Linearity of target analytes

7.3.8.1 If the RSD of any target analyte is 15% or less, then the response factor is assumed to be constant over the calibration range, and the average response factor may be used for quantitation (Sec. 7.7.2).

7.3.8.2 If the RSD of any target analyte is greater than 15%, refer to Sec. 7.0 of Method 8000 for additional calibration options. One of the options must be applied to GC/MS calibration in this situation, or a new initial calibration must be performed.

NOTE: Method 8000 specifies a linearity criterion of 20% RSD. That criterion pertains to GC and HPLC methods other than GC/MS. Method 8260 requires 15% RSD as evidence of sufficient linearity to employ an average response factor.

7.3.8.3 When the RSD exceeds 15%, the plotting and visual inspection of a calibration curve can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

NOTE: The 20% RSD criteria in Method 8000 pertains to GC and HPLC methods other than GC/MS. Method 8260 requires 15% RSD.

7.4 GC/MS calibration verification - Calibration verification consists of three steps that are performed at the beginning of each 12-hour analytical shift.

7.4.1 Prior to the analysis of samples or calibration standards, inject or introduce 5-50 ng of the 4-bromofluorobenzene standard into the GC/MS system. The resultant mass spectra for the BFB must meet the criteria given in Table 4 before sample analysis begins. These criteria must be demonstrated each 12-hour shift during which samples are analyzed.

7.4.2 The initial calibration curve (Sec. 7.3) for each compound of interest should be verified once every 12 hours prior to sample analysis, using the introduction technique used for samples. This is accomplished by analyzing a calibration standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS. The results from the calibration standard analysis should meet the verification acceptance criteria provided in Secs. 7.4.4 through 7.4.7.

NOTE: The BFB and calibration verification standard may be combined into a single standard as long as both tuning and calibration verification acceptance criteria for the project can be met without interferences.

7.4.3 A method blank should be analyzed after the calibration standard, or at any other time during the analytical shift, to ensure that the total system (introduction device, transfer lines and GC/MS system) is free of contaminants. If the method blank indicates contamination, then it may be appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples. See Sec. 8.0 of Method 8000 for method blank performance criteria.

7.4.4 System Performance Check Compounds (SPCCs)

7.4.4.1 A system performance check must be made during every 12-hour analytical shift. Each SPCC compound in the calibration verification standard must meet its minimum response factor (see Sec. 7.3.5.4). This is the same check that is applied during the initial calibration.

7.4.4.2 If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before sample analysis begins.

7.4.5 Calibration Check Compounds (CCCs)

7.4.5.1 After the system performance check is met, the CCCs listed in Sec. 7.3.6 are used to check the validity of the initial calibration. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model. Refer to Sec. 7.0 of Method 8000 for guidance on calculating percent difference and drift.

7.4.5.2 If the percent difference or drift for each CCC is less than or equal to 20%, the initial calibration is assumed to be valid. If the criterion is not met (i.e., greater than 20% difference or drift), for any one CCC, then corrective action must be taken prior to the analysis of samples. If the CCC's are not included in the list of analytes for a project, and therefore not included in the calibration standards, then all analytes must meet the 20% difference or drift criterion.

7.4.5.3 Problems similar to those listed under SPCCs could affect the CCCs. If the problem cannot be corrected by other measures, a new five-point initial calibration must be generated. The CCC criteria must be met before sample analysis begins.

7.4.6 Internal standard retention time - The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

7.4.7 Internal standard response - If the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to + 100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

7.5 GC/MS analysis of samples

7.5.1 It is highly recommended that the sample be screened to minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds. Some of the screening options available utilizing SW-846 methods are automated headspace-GC/FID (Methods 5021/8015), automated headspace-GC/PID/ELCD (Methods 5021/8021), or waste dilution-GC/PID/ELCD (Methods 3585/8021) using the same type of capillary column. When used only for screening purposes, the quality control requirements in the methods above may be reduced as appropriate. Sample screening is particularly important when Method 8260 is used to achieve low detection levels.

7.5.2 BFB tuning criteria and GC/MS calibration verification criteria must be met before analyzing samples.

7.5.3 All samples and standard solutions must be allowed to warm to ambient temperature before analysis. Set up the introduction device as outlined in the method of choice.

7.5.4 The process of taking an aliquot destroys the validity of remaining volume of an aqueous sample for future analysis. Therefore, if only one VOA vial is provided to the laboratory, the analyst should prepare two aliquots for analysis at this time, to protect against possible loss of sample integrity. This second sample is maintained only until such time when the analyst has determined that the first sample has been analyzed properly. For aqueous samples, one 20-mL syringe could be used to hold two 5-mL aliquots. If the second aliquot is to be taken from the syringe, it must be analyzed within 24 hours. Care must be taken to prevent air from leaking into the syringe.

7.5.5 Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample or standard bottle, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. If lower detection limits are required, use a 25-mL syringe, and adjust the final volume to 25.0 mL.

7.5.6 The following procedure may be used to dilute aqueous samples for analysis of volatiles. All steps must be performed without delays, until the diluted sample is in a gas-tight syringe.

7.5.6.1 Dilutions may be made in volumetric flasks (10- to 100-mL). Select the volumetric flask that will allow for the necessary dilution. Intermediate dilution steps may be necessary for extremely large dilutions.

7.5.6.2 Calculate the approximate volume of organic-free reagent water to be added to the volumetric flask, and add slightly less than this quantity of organic-free reagent water to the flask.

7.5.6.3 Inject the appropriate volume of the original sample from the syringe into the flask. Aliquots of less than 1 mL are not recommended. Dilute the sample to the mark with organic-free reagent water. Cap the flask, invert, and shake three times. Repeat above procedure for additional dilutions.

7.5.6.4 Fill a 5-mL syringe with the diluted sample, as described in Sec. 7.5.5.

7.5.7 Compositing aqueous samples prior to GC/MS analysis

7.5.7.1 Add 5 mL of each sample (up to 5 samples are allowed) to a 25-mL glass syringe. Special precautions must be made to maintain zero headspace in the syringe. Larger volumes of a smaller number of samples may be used, provided that equal volumes of each sample are composited.

7.5.7.2 The samples must be cooled to 4°C or less during this step to minimize volatilization losses. Sample vials may be placed in a tray of ice during the processing.

7.5.7.3 Mix each vial well and draw out a 5-mL aliquot with the 25-mL syringe.

7.5.7.4 Once all the aliquots have been combined on the syringe, invert the syringe several times to mix the aliquots. Introduce the composited sample into the instrument, using the method of choice (see Sec. 7.1).

7.5.7.5 If less than five samples are used for compositing, a proportionately smaller syringe may be used, unless a 25-mL sample is to be purged.

7.5.8 Add 10 µL of the surrogate spiking solution and 10 µL of the internal standard spiking solution to each sample either manually or by autosampler. The surrogate and internal standards may be mixed and added as a single spiking solution. The addition of 10 µL of the surrogate spiking solution to 5 mL of aqueous sample will yield a concentration of 50 µg/L of each surrogate standard. The addition of 10 µL of the surrogate spiking solution to 5 g of a non-aqueous sample will yield a concentration of 50 µg/kg of each standard.

If a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute surrogate and internal standard solutions may be required.

7.5.9 Add 10 µL of the matrix spike solution (Sec. 5.13) to a 5-mL aliquot of the sample chosen for spiking. Disregarding any dilutions, this is equivalent to a concentration of 50 µg/L of each matrix spike standard.

7.5.9.1 Follow the same procedure in preparing the laboratory control sample (LCS), except the spike is added to a clean matrix. See Sec. 8.4 and Method 5000 for more guidance on the selection and preparation of the matrix spike and the LCS.

7.5.9.2 If a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute matrix spiking and LCS solutions may be required.

7.5.10 Analyze the sample following the procedure in the introduction method of choice.

7.5.10.1 For direct injection, inject 1 to 2 µL into the GC/MS system. The volume limitation will depend upon the chromatographic column chosen and the tolerance of the specific GC/MS system to water (if an aqueous sample is being analyzed).

7.5.10.2 The concentration of the internal standards, surrogates, and matrix spiking standards (if any) added to the injection aliquot must be adjusted to provide the same concentration in the 1-2 µL injection as would be introduced into the GC/MS by purging a 5-mL aliquot.

NOTE: It may be a useful diagnostic tool to monitor internal standard retention times and responses (area counts) in all samples, spikes, blanks, and standards to effectively check drifting method performance, poor injection execution, and anticipate the need for system inspection and/or maintenance.

7.5.11 If the initial analysis of the sample or a dilution of the sample has a concentration of any analyte that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. Secondary ion quantitation is allowed only when there are sample interferences with the primary ion.

7.5.11.1 When ions from a compound in the sample saturate the detector, this analysis must be followed by the analysis of an organic-free reagent water blank. If the blank analysis is not free of interferences, then the system must be decontaminated. Sample analysis may not resume until the blank analysis is demonstrated to be free of interferences.

7.5.11.2 All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

7.5.12 The use of selected ion monitoring (SIM) is acceptable in situations requiring detection limits below the normal range of full EI spectra. However, SIM may provide a lesser degree of confidence in the compound identification unless multiple ions are monitored for each compound.

7.6 Qualitative analysis

7.6.1 The qualitative identification of each compound determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met.

7.6.1.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

7.6.1.2 The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

7.6.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

7.6.1.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

7.6.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

7.6.1.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

7.6.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Use the following guidelines for making tentative identifications:

(1) Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

(2) The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

(3) Molecular ions present in the reference spectrum should be present in the sample spectrum.

(4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

(5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

7.7 Quantitative analysis

7.7.1 Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. The internal standard used shall be the one nearest the retention time of that of a given analyte.

7.7.2 If the RSD of a compound's response factors is 15% or less, then the concentration in the extract may be determined using the average response factor ($\overline{RF}$) from initial calibration data (7.3.6). See Method 8000, Sec. 7.0, for the equations describing internal standard calibration and either linear or non-linear calibrations.

7.7.3 Where applicable, the concentration of any non-target analytes identified in the sample (Sec. 7.6.2) should be estimated. The same formulae should be used with the following modifications: The areas A_x and A_{is} should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1.

7.7.4 The resulting concentration should be reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One and Method 8000 for specific quality control (QC) procedures. Quality control procedures to ensure the proper operation of the various sample preparation and/or sample introduction techniques can be found in Methods 3500 and 5000. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated.

8.2 Quality control procedures necessary to evaluate the GC system operation are found in Method 8000, Sec. 7.0 and include evaluation of retention time windows, calibration verification and chromatographic analysis of samples. In addition, instrument QC requirements may be found in the following sections of Method 8260:

8.2.1 The GC/MS system must be tuned to meet the BFB specifications in Secs. 7.3.1 and 7.4.1.

8.2.2 There must be an initial calibration of the GC/MS system as described in Sec. 7.3.

8.2.3 The GC/MS system must meet the SPCC criteria described in Sec. 7.4.4 and the CCC criteria in Sec. 7.4.5, each 12 hours.

8.3 Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made. See Method 8000, Sec. 8.0 for information on how to accomplish this demonstration.

8.4 Sample Quality Control for Preparation and Analysis - The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank, matrix spike, a duplicate, and a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample.

8.4.1 Before processing any samples, the analyst should demonstrate, through the analysis of a method blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is analyzed or there is a change in reagents, a method blank should be analyzed as a safeguard against chronic laboratory contamination. The blanks should be carried through all stages of sample preparation and measurement.

8.4.2 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair.

8.4.3 A Laboratory Control Sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

8.4.4 See Method 8000, Sec. 8.0 for the details on carrying out sample quality control procedures for preparation and analysis.

8.5 Surrogate recoveries - The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 8000, Sec. 8.0 for information on evaluating surrogate data and developing and updating surrogate limits.

8.6 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the calibration verification standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g., the column changed), recalibration of the system must take place.

8.7 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9.0 METHOD PERFORMANCE

9.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

9.2 This method has been tested using purge-and-trap (Method 5030) in a single laboratory using spiked water. Using a wide-bore capillary column, water was spiked at concentrations between 0.5 and 10 µg/L. Single laboratory accuracy and precision data are presented for the method analytes in Table 6. Calculated MDLs are presented in Table 1.

9.3 The method was tested using purge-and-trap (Method 5030) with water spiked at 0.1 to 0.5 µg/L and analyzed on a cryofocussed narrow-bore column. The accuracy and precision data for these compounds are presented in Table 7. MDL values were also calculated from these data and are presented in Table 2.

9.4 Direct injection (Method 3585) has been used for the analysis of waste motor oil samples using a wide-bore column. Single laboratory precision and accuracy data are presented in Tables 10 and 11 for TCLP volatiles in oil. The performance data were developed by spiking and analyzing seven replicates each of new and used oil. The oils were spiked at the TCLP regulatory concentrations for most analytes, except for the alcohols, ketones, ethyl acetate and chlorobenzene which are spiked at 5 ppm, well below the regulatory concentrations. Prior to spiking, the new oil (an SAE 30-weight motor oil) was heated at 80°C overnight to remove volatiles. The used oil (a mixture of used oil drained from passenger automobiles) was not heated and was contaminated with 20 - 300 ppm of BTEX compounds and isobutanol. These contaminants contributed to the extremely high recoveries of the BTEX compounds in the used oil. Therefore, the data from the deuterated analogs of these analytes represent more typical recovery values.

9.5 Single laboratory accuracy and precision data were obtained for the Method 5035 analytes in three soil matrices: sand; a soil collected 10 feet below the surface of a hazardous landfill, called C-Horizon; and a surface garden soil. Sample preparation was by Method 5035. Each sample was fortified with the analytes at a concentration of 4 µg/kg. These data are listed in Tables 17, 18, and 19. All data were calculated using fluorobenzene as the internal standard added to the soil sample prior to extraction. This causes some of the results to be greater than 100% recovery because the precision of results is sometimes as great as 28%.

9.5.1 In general, the recoveries of the analytes from the sand matrix are the highest, the C-Horizon soil results are somewhat less, and the surface garden soil recoveries are the lowest. This is due to the greater adsorptive capacity of the garden soil. This illustrates the necessity of analyzing matrix spike samples to assess the degree of matrix effects.

9.5.2 The recoveries of some of the gases, or very volatile compounds, such as vinyl chloride, trichlorofluoromethane, and 1,1-dichloroethene, are somewhat greater than 100%. This is due to the difficulty encountered in fortifying the soil with these compounds, allowing an equilibration period, then extracting them with a high degree of precision. Also, the garden soil results in Table 19 include some extraordinarily high recoveries for some aromatic compounds, such as toluene, xylenes, and trimethylbenzenes. This is due to contamination of the soil prior to sample collection, and to the fact that no background was subtracted.

9.6 Performance data for nonpurgeable volatiles using azeotropic distillation (Method 5031) are included in Tables 12 to 16.

9.7 Performance data for volatiles prepared using vacuum distillation (Method 5032) in soil, water, oil and fish tissue matrices are included in Tables 20 to 27.

9.8 Single laboratory accuracy and precision data were obtained for the Method 5021 analytes in two soil matrices: sand and a surface garden soil. Replicate samples were fortified with the analytes at concentrations of 10 µg/kg. These data are listed in Table 30. All data were calculated using the internal standards listed for each analyte in Table 28. The recommended internal standards were selected because they generated the best accuracy and precision data for the analyte in both types of soil.

9.8.1 If a detector other than an MS is used for analysis, consideration must be given to the choice of internal standards and surrogates. They must not coelute with any other analyte and must have similar properties to the analytes. The recoveries of the analytes are 50% or higher for each matrix studied. The recoveries of the gases or very volatile compounds are greater than 100% in some cases. Also, results include high recoveries of some aromatic compounds, such as toluene, xylenes, and trimethylbenzenes. This is due to contamination of the soil prior to sample collection.

9.8.2 The method detection limits using Method 5021 listed in Table 29 were calculated from results of seven replicate analyses of the sand matrix. Sand was chosen because it demonstrated the least degree of matrix effect of the soils studied. These MDLs were calculated utilizing the procedure described in Chapter One and are intended to be a general indication of the capabilities of the method.

9.9 The MDL concentrations listed in Table 31 were determined using Method 5041 in conjunction with Method 8260. They were obtained using cleaned blank VOST tubes and reagent water. Similar results have been achieved with field samples. The MDL actually achieved in a given analysis will vary depending upon instrument sensitivity and the effects of the matrix. Preliminary spiking studies indicate that under the test conditions, the MDLs for spiked compounds in extremely complex matrices may be larger by a factor of 500 - 1000.

9.10 The EQL of sample taken by Method 0040 and analyzed by Method 8260 is estimated to be in the range of 0.03 to 0.9 ppm (See Table 33). Matrix effects may cause the individual compound detection limits to be higher.

10.0 REFERENCES

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TABLE 1

CHROMATOGRAPHIC RETENTION TIMES AND METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC COMPOUNDS ON WIDE-BORE CAPILLARY COLUMNS

Compound	Retention Time (minutes)			MDL[d]
	Column 1[a]	Column 2[b]	Column 2'[c]	(µg/L)
Dichlorodifluoromethane	1.35	0.70	3.13	0.10
Chloromethane	1.49	0.73	3.40	0.13
Vinyl Chloride	1.56	0.79	3.93	0.17
Bromomethane	2.19	0.96	4.80	0.11
Chloroethane	2.21	1.02	--	0.10
Trichlorofluoromethane	2.42	1.19	6.20	0.08
Acrolein	3.19			
Iodomethane	3.56			
Acetonitrile	4.11			
Carbon disulfide	4.11			
Allyl chloride	4.11			
Methylene chloride	4.40	2.06	9.27	0.03
1,1-Dichloroethene	4.57	1.57	7.83	0.12
Acetone	4.57			
trans-1,2-Dichloroethene	4.57	2.36	9.90	0.06
Acrylonitrile	5.00			
1,1-Dichloroethane	6.14	2.93	10.80	0.04
Vinyl acetate	6.43			
2,2-Dichloropropane	8.10	3.80	11.87	0.35
2-Butanone	--			
cis-1,2-Dichloroethene	8.25	3.90	11.93	0.12
Propionitrile	8.51			
Chloroform	9.01	4.80	12.60	0.03
Bromochloromethane	--	4.38	12.37	0.04
Methacrylonitrile	9.19			
1,1,1-Trichloroethane	10.18	4.84	12.83	0.08
Carbon tetrachloride	11.02	5.26	13.17	0.21
1,1-Dichloropropene	--	5.29	13.10	0.10
Benzene	11.50	5.67	13.50	0.04
1,2-Dichloroethane	12.09	5.83	13.63	0.06
Trichloroethene	14.03	7.27	14.80	0.19
1,2-Dichloropropane	14.51	7.66	15.20	0.04
Bromodichloromethane	15.39	8.49	15.80	0.08
Dibromomethane	15.43	7.93	5.43	0.24
Methyl methacrylate	15.50			
1,4-Dioxane	16.17			
2-Chloroethyl vinyl ether	--			
4-Methyl-2-pentanone	17.32			
trans-1,3-Dichloropropene	17.47	--	16.70	--
Toluene	18.29	10.00	17.40	0.11
cis-1,3-Dichloropropene	19.38	--	17.90	--

TABLE 1 (cont.)

Compound	Retention Time (minutes)			MDL^{d}
	Column 1^{a}	Column 2^{b}	Column 2"^{c}	(µg/L)
1,1,2-Trichloroethane	19.59	11.05	18.30	0.10
Ethyl methacrylate	20.01			
2-Hexanone	20.30			
Tetrachloroethene	20.26	11.15	18.60	0.14
1,3-Dichloropropane	20.51	11.31	18.70	0.04
Dibromochloromethane	21.19	11.85	19.20	0.05
1,2-Dibromoethane	21.52	11.83	19.40	0.06
1-Chlorohexane	--	13.29	--	0.05
Chlorobenzene	23.17	13.01	20.67	0.04
1,1,1,2-Tetrachloroethane	23.36	13.33	20.87	0.05
Ethylbenzene	23.38	13.39	21.00	0.06
p-Xylene	23.54	13.69	21.30	0.13
m-Xylene	23.54	13.68	21.37	0.05
o-Xylene	25.16	14.52	22.27	0.11
Styrene	25.30	14.60	22.40	0.04
Bromoform	26.23	14.88	22.77	0.12
Isopropylbenzene (Cumene)	26.37	15.46	23.30	0.15
cis-1,4-Dichloro-2-butene	27.12			
1,1,2,2-Tetrachloroethane	27.29	16.35	24.07	0.04
Bromobenzene	27.46	15.86	24.00	0.03
1,2,3-Trichloropropane	27.55	16.23	24.13	0.32
n-Propylbenzene	27.58	16.41	24.33	0.04
2-Chlorotoluene	28.19	16.42	24.53	0.04
trans-1,4-Dichloro-2-butene	28.26			
1,3,5-Trimethylbenzene	28.31	16.90	24.83	0.05
4-Chlorotoluene	28.33	16.72	24.77	0.06
Pentachloroethane	29.41			
1,2,4-Trimethylbenzene	29.47	17.70	31.50	0.13
sec-Butylbenzene	30.25	18.09	26.13	0.13
tert-Butylbenzene	30.59	17.57	26.60	0.14
p-Isopropyltoluene	30.59	18.52	26.50	0.12
1,3-Dichlorobenzene	30.56	18.14	26.37	0.12
1,4-Dichlorobenzene	31.22	18.39	26.60	0.03
Benzyl chloride	32.00			
n-Butylbenzene	32.23	19.49	27.32	0.11
1,2-Dichlorobenzene	32.31	19.17	27.43	0.03
1,2-Dibromo-3-chloropropane	35.30	21.08	--	0.26
1,2,4-Trichlorobenzene	38.19	23.08	31.50	0.04
Hexachlorobutadiene	38.57	23.68	32.07	0.11
Naphthalene	39.05	23.52	32.20	0.04
1,2,3-Trichlorobenzene	40.01	24.18	32.97	0.03

Compound	Retention Time (minutes)			MDL[d]
	Column 1[a]	Column 2[b]	Column 2"[c]	(µg/L)
INTERNAL STANDARDS/SURROGATES				
1,4-Difluorobenzene	13.26			
Chlorobenzene-d_5	23.10			
1,4-Dichlorobenzene-d_4	31.16			
4-Bromofluorobenzene	27.83	15.71	23.63	
1,2-Dichlorobenzene-d_4	32.30	19.08	27.25	
Dichloroethane-d_4	12.08			
Dibromofluoromethane	--			
Toluene-d_8	18.27			
Pentafluorobenzene	--			
Fluorobenzene	13.00	6.27	14.06	

[a] Column 1 - 60 meter x 0.75 mm ID VOCOL capillary. Hold at 10°C for 8 minutes, then program to 180°C at 4°C/min.

[b] Column 2 - 30 meter x 0.53 mm ID DB-624 wide-bore capillary using cryogenic oven. Hold at 10°C for 5 minutes, then program to 160°C at 6°C/min.

[c] Column 2" - 30 meter x 0.53 mm ID DB-624 wide-bore capillary, cooling GC oven to ambient temperatures. Hold at 10°C for 6 minutes, program to 70°C at 10 °C/min, program to 120°C at 5°C/min, then program to 180°C at 8°C/min.

[d] MDL based on a 25-mL sample volume.

TABLE 2

CHROMATOGRAPHIC RETENTION TIMES AND METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC COMPOUNDS ON NARROW-BORE CAPILLARY COLUMNS

Compound	Retention Time (minutes) Column 3[a]	MDL[b] (µg/L)
Dichlorodifluoromethane	0.88	0.11
Chloromethane	0.97	0.05
Vinyl chloride	1.04	0.04
Bromomethane	1.29	0.03
1,1-Dichloroethane	4.03	0.03
cis-1,2-Dichloroethene	5.07	0.06
2,2-Dichloropropane	5.31	0.08
Chloroform	5.55	0.04
Bromochloromethane	5.63	0.09
1,1,1-Trichloroethane	6.76	0.04
1,2-Dichloroethane	7.00	0.02
1,1-Dichloropropene	7.16	0.12
Carbon tetrachloride	7.41	0.02
Benzene	7.41	0.03
1,2-Dichloropropane	8.94	0.02
Trichloroethene	9.02	0.02
Dibromomethane	9.09	0.01
Bromodichloromethane	9.34	0.03
Toluene	11.51	0.08
1,1,2-Trichloroethane	11.99	0.08
1,3-Dichloropropane	12.48	0.08
Dibromochloromethane	12.80	0.07
Tetrachloroethene	13.20	0.05
1,2-Dibromoethane	13.60	0.10
Chlorobenzene	14.33	0.03
1,1,1,2-Tetrachloroethane	14.73	0.07
Ethylbenzene	14.73	0.03
p-Xylene	15.30	0.06
m-Xylene	15.30	0.03
Bromoform	15.70	0.20
o-Xylene	15.78	0.06
Styrene	15.78	0.27
1,1,2,2-Tetrachloroethane	15.78	0.20
1,2,3-Trichloropropane	16.26	0.09
Isopropylbenzene	16.42	0.10
Bromobenzene	16.42	0.11
2-Chlorotoluene	16.74	0.08
n-Propylbenzene	16.82	0.10
4-Chlorotoluene	16.82	0.06

TABLE 2 (cont.)

Compound	Retention Time (minutes) Column 3[a]	MDL[b] (µg/L)
1,3,5-Trimethylbenzene	16.99	0.06
tert-Butylbenzene	17.31	0.33
1,2,4-Trimethylbenzene	17.31	0.09
sec-Butylbenzene	17.47	0.12
1,3-Dichlorobenzene	17.47	0.05
p-Isopropyltoluene	17.63	0.26
1,4-Dichlorobenzene	17.63	0.04
1,2-Dichlorobenzene	17.79	0.05
n-Butylbenzene	17.95	0.10
1,2-Dibromo-3-chloropropane	18.03	0.50
1,2,4-Trichlorobenzene	18.84	0.20
Naphthalene	19.07	0.10
Hexachlorobutadiene	19.24	0.10
1,2,3-Trichlorobenzene	19.24	0.14

[a] Column 3 - 30 meter x 0.32 mm ID DB-5 capillary with 1 µm film thickness.

[b] MDL based on a 25-mL sample volume.

TABLE 3

ESTIMATED QUANTITATION LIMITS FOR VOLATILE ANALYTES[a]

Estimated Quantitation Limits		
5-mL Ground Water Purge (µg/L)	25-mL Ground water Purge (µg/L)	Low Soil/Sediment[b] µg/kg
5	1	5

[a] Estimated Quantitation Limit (EQL) - The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL is generally 5 to 10 times the MDL. However, it may be nominally chosen within these guidelines to simplify data reporting. For many analytes the EQL analyte concentration is selected for the lowest non-zero standard in the calibration curve. Sample EQLs are highly matrix-dependent. The EQLs listed herein are provided for guidance and may not always be achievable. See the following footnote for further guidance on matrix-dependent EQLs.

[b] EQLs listed for soil/sediment are based on wet weight. Normally data are reported on a dry weight basis; therefore, EQLs will be higher, based on the percent dry weight in each sample.

Other Matrices	Factor[c]
Water miscible liquid waste	50
High concentration soil and sludge	125
Non-water miscible waste	500

[c] EQL = [EQL for low soil sediment (Table 3)] x [Factor].

For non-aqueous samples, the factor is on a wet-weight basis.

TABLE 4

BFB (4-BROMOFLUOROBENZENE) MASS INTENSITY CRITERIA[a]

m/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to 9% of m/z 176

[a] Alternate tuning criteria may be used, (e.g. CLP, Method 524.2, or manufacturers" instructions), provided that method performance is not adversely affected.

TABLE 5

CHARACTERISTIC MASSES (m/z) FOR PURGEABLE ORGANIC COMPOUNDS

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Acetone	58	43
Acetonitrile	41	40, 39
Acrolein	56	55, 58
Acrylonitrile	53	52, 51
Allyl alcohol	57	58, 39
Allyl chloride	76	41, 39, 78
Benzene	78	-
Benzyl chloride	91	126, 65, 128
Bromoacetone	136	43, 138, 93, 95
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
iso-Butanol	74	43
n-Butanol	56	41
2-Butanone	72	43
n-Butylbenzene	91	92, 134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91, 134
Carbon disulfide	76	78
Carbon tetrachloride	117	119
Chloral hydrate	82	44, 84, 86, 111
Chloroacetonitrile	48	75
Chlorobenzene	112	77, 114
1-Chlorobutane	56	49
Chlorodibromomethane	129	208, 206
Chloroethane	64 (49*)	66 (51*)
2-Chloroethanol	49	44, 43, 51, 80
Bis(2-chloroethyl) sulfide	109	111, 158, 160
2-Chloroethyl vinyl ether	63	65, 106
Chloroform	83	85
Chloromethane	50 (49*)	52 (51*)
Chloroprene	53	88, 90, 51
3-Chloropropionitrile	54	49, 89, 91
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
1,2-Dibromo-3-chloropropane	75	155, 157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174

TABLE 5 (cont.)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
1,2-Dichlorobenzene	146	111, 148
1,2-Dichlorobenzene-d_4	152	115, 150
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
cis-1,4-Dichloro-2-butene	75	53, 77, 124, 89
trans-1,4-Dichloro-2-butene	53	88, 75
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	96	61, 98
trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,3-Dichloro-2-propanol	79	43, 81, 49
1,1-Dichloropropene	75	110, 77
cis-1,3-Dichloropropene	75	77, 39
trans-1,3-Dichloropropene	75	77, 39
1,2,3,4-Diepoxybutane	55	57, 56
Diethyl ether	74	45, 59
1,4-Dioxane	88	58, 43, 57
Epichlorohydrin	57	49, 62, 51
Ethanol	31	45, 27, 46
Ethyl acetate	88	43, 45, 61
Ethylbenzene	91	106
Ethylene oxide	44	43, 42
Ethyl methacrylate	69	41, 99, 86, 114
Hexachlorobutadiene	225	223, 227
Hexachloroethane	201	166, 199, 203
2-Hexanone	43	58, 57, 100
2-Hydroxypropionitrile	44	43, 42, 53
Iodomethane	142	127, 141
Isobutyl alcohol	43	41, 42, 74
Isopropylbenzene	105	120
p-Isopropyltoluene	119	134, 91
Malononitrile	66	39, 65, 38
Methacrylonitrile	41	67, 39, 52, 66
Methyl acrylate	55	85
Methyl-t-butyl ether	73	57
Methylene chloride	84	86, 49
Methyl ethyl ketone	72	43
Methyl iodide	142	127, 141

TABLE 5 (cont.)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Methyl methacrylate	69	41, 100, 39
4-Methyl-2-pentanone	100	43, 58, 85
Naphthalene	128	-
Nitrobenzene	123	51, 77
2-Nitropropane	46	-
2-Picoline	93	66, 92, 78
Pentachloroethane	167	130, 132, 165, 169
Propargyl alcohol	55	39, 38, 53
β-Propiolactone	42	43, 44
Propionitrile (ethyl cyanide)	54	52, 55, 40
n-Propylamine	59	41, 39
n-Propylbenzene	91	120
Pyridine	79	52
Styrene	104	78
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	164	129, 131, 166
Toluene	92	91
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	97, 130, 132
Trichlorofluoromethane	151	101, 153
1,2,3-Trichloropropane	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl acetate	43	86
Vinyl chloride	62	64
o-Xylene	106	91
m-Xylene	106	91
p-Xylene	106	91
Internal Standards/Surrogates:		
Benzene-d_6	84	83
Bromobenzene-d_5	82	162
Bromochloromethane-d_2	51	131
1,4-Difluorobenzene	114	
Chlorobenzene-d_5	117	
1,4-Dichlorobenzene-d_4	152	115, 150
1,1,2-Trichloroethane-d_3	100	
4-Bromofluorobenzene	95	174, 176
Chloroform-d_1	84	
Dibromofluoromethane	113	

TABLE 5 (cont.)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Internal Standards/Surrogates		
Dichloroethane-d_4	102	
Toluene-d_8	98	
Pentafluorobenzene	168	
Fluorobenzene	96	77

* Characteristic ion for an ion trap mass spectrometer (to be used when ion-molecule reactions are observed).

TABLE 6

SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR PURGEABLE VOLATILE ORGANIC COMPOUNDS IN WATER DETERMINED WITH A WIDE-BORE CAPILLARY COLUMN (METHOD 5030)

Compound	Conc. Range (µg/L)	Number of Samples	% Recovery[a]	Standard Deviation of Recovery[b]	RSD
Benzene	0.1 - 10	31	97	6.5	5.7
Bromobenzene	0.1 - 10	30	100	5.5	5.5
Bromochloromethane	0.5 - 10	24	90	5.7	6.4
Bromodichloromethane	0.1 - 10	30	95	5.7	6.1
Bromoform	0.5 - 10	18	101	6.4	6.3
Bromomethane	0.5 - 10	18	95	7.8	8.2
n-Butylbenzene	0.5 - 10	18	100	7.6	7.6
sec-Butylbenzene	0.5 - 10	16	100	7.6	7.6
tert-Butylbenzene	0.5 - 10	18	102	7.4	7.3
Carbon tetrachloride	0.5 - 10	24	84	7.4	8.8
Chlorobenzene	0.1 - 10	31	98	5.8	5.9
Chloroethane	0.5 - 10	24	89	8.0	9.0
Chloroform	0.5 - 10	24	90	5.5	6.1
Chloromethane	0.5 - 10	23	93	8.3	8.9
2-Chlorotoluene	0.1 - 10	31	90	5.6	6.2
4-Chlorotoluene	0.1 - 10	31	99	8.2	8.3
1,2-Dibromo-3-Chloropropane	0.5 - 10	24	83	16.6	19.9
Dibromochloromethane	0.1 - 10	31	92	6.5	7.0
1,2-Dibromoethane	0.5 - 10	24	102	4.0	3.9
Dibromomethane	0.5 - 10	24	100	5.6	5.6
1,2-Dichlorobenzene	0.1 - 10	31	93	5.8	6.2
1,3-Dichlorobenzene	0.5 - 10	24	99	6.8	6.9
1,4-Dichlorobenzene	0.2 - 20	31	103	6.6	6.4
Dichlorodifluoromethane	0.5 - 10	18	90	6.9	7.7
1,1-Dichlorobenzene	0.5 - 10	24	96	5.1	5.3
1,2-Dichlorobenzene	0.1 - 10	31	95	5.1	5.4
1,1-Dichloroethene	0.1 - 10	34	94	6.3	6.7
cis-1,2-Dichloroethene	0.5 - 10	18	101	6.7	6.7
trans-1,2-Dichloroethene	0.1 - 10	30	93	5.2	5.6
1,2-Dichloropropane	0.1 - 10	30	97	5.9	6.1
1,3-Dichloropropane	0.1 - 10	31	96	5.7	6.0
2,2-Dichloropropane	0.5 - 10	12	86	14.6	16.9
1,1-Dichloropropene	0.5 - 10	18	98	8.7	8.9
Ethylbenzene	0.1 - 10	31	99	8.4	8.6
Hexachlorobutadiene	0.5 - 10	18	100	6.8	6.8
Isopropylbenzene	0.5 - 10	16	101	7.7	7.6
p-Isopropyltoluene	0.1 - 10	23	99	6.7	6.7
Methylene chloride	0.1 - 10	30	95	5.0	5.3

TABLE 6 (cont.)

Compound	Conc. Range (µg/L)	Number of Samples	% Recovery[a]	Standard Deviation of Recovery[b]	RSD
Naphthalene	0.1 -100	31	104	8.6	8.2
n-Propylbenzene	0.1 - 10	31	100	5.8	5.8
Styrene	0.1 -100	39	102	7.3	7.2
1,1,1,2-Tetrachloroethane	0.5 - 10	24	90	6.1	6.8
1,1,2,2-Tetrachloroethane	0.1 - 10	30	91	5.7	6.3
Tetrachloroethene	0.5 - 10	24	89	6.0	6.8
Toluene	0.5 - 10	18	102	8.1	8.0
1,2,3-Trichlorobenzene	0.5 - 10	18	109	9.4	8.6
1,2,4-Trichlorobenzene	0.5 - 10	18	108	9.0	8.3
1,1,1-Trichloroethane	0.5 - 10	18	98	7.9	8.1
1,1,2-Trichloroethane	0.5 - 10	18	104	7.6	7.3
Trichloroethene	0.5 - 10	24	90	6.5	7.3
Trichlorofluoromethane	0.5 - 10	24	89	7.2	8.1
1,2,3-Trichloropropane	0.5 - 10	16	108	15.6	14.4
1,2,4-Trimethylbenzene	0.5 - 10	18	99	8.0	8.1
1,3,5-Trimethylbenzene	0.5 - 10	23	92	6.8	7.4
Vinyl chloride	0.5 - 10	18	98	6.5	6.7
o-Xylene	0.1 - 31	18	103	7.4	7.2
m-Xylene	0.1 - 10	31	97	6.3	6.5
p-Xylene	0.5 - 10	18	104	8.0	7.7

[a] Recoveries were calculated using internal standard method. The internal standard was fluorobenzene.

[b] Standard deviation was calculated by pooling data from three concentrations.

TABLE 7

SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR PURGEABLE VOLATILE ORGANIC COMPOUNDS IN WATER DETERMINED WITH A NARROW-BORE CAPILLARY COLUMN (METHOD 5030)

Compound	Conc. (µg/L)	Number of Samples	% Recovery[a]	Standard Deviation of Recovery[b]	RSD
Benzene	0.1	7	99	6.2	6.3
Bromobenzene	0.5	7	97	7.4	7.6
Bromochloromethane	0.5	7	97	5.8	6.0
Bromodichloromethane	0.1	7	100	4.6	4.6
Bromoform	0.5	7	101	5.4	5.3
Bromomethane	0.5	7	99	7.1	7.2
n-Butylbenzene	0.5	7	94	6.0	6.4
sec-Butylbenzene	0.5	7	110	7.1	6.5
tert-Butylbenzene	0.5	7	110	2.5	2.3
Carbon tetrachloride	0.1	7	108	6.8	6.3
Chlorobenzene	0.1	7	91	5.8	6.4
Chloroethane	0.1	7	100	5.8	5.8
Chloroform	0.1	7	105	3.2	3.0
Chloromethane	0.5	7	101	4.7	4.7
2-Chlorotoluene	0.5	7	99	4.6	4.6
4-Chlorotoluene	0.5	7	96	7.0	7.3
1,2-Dibromo-3-chloropropane	0.5	7	92	10.0	10.9
Dibromochloromethane	0.1	7	99	5.6	5.7
1,2-Dibromoethane	0.5	7	97	5.6	5.8
Dibromomethane	0.5	7	93	5.6	6.0
1,2-Dichlorobenzene	0.1	7	97	3.5	3.6
1,3-Dichlorobenzene	0.1	7	101	6.0	5.9
1,4-Dichlorobenzene	0.1	7	106	6.5	6.1
Dichlorodifluoromethane	0.1	7	99	8.8	8.9
1,1-Dichloroethane	0.5	7	98	6.2	6.3
1,2-Dichloroethane	0.1	7	100	6.3	6.3
1,1-Dichloroethene	0.1	7	95	9.0	9.5
cis-1,2-Dichloroethene	0.1	7	100	3.5	3.7
trans-1,2-Dichloroethene	0.1	7	98	7.2	7.3
1,2-Dichloropropane	0.5	7	96	6.0	6.3
1,3-Dichloropropane	0.5	7	99	5.8	5.9
2,2-Dichloropropane	0.5	7	99	4.9	4.9
1,1-Dichloropropene	0.5	7	102	7.4	7.3
Ethylbenzene	0.5	7	99	5.2	5.3
Hexachlorobutadiene	0.5	7	100	6.7	6.7
Isopropylbenzene	0.5	7	102	6.4	6.3
p-Isopropyltoluene	0.5	7	113	13.0	11.5
Methylene chloride	0.5	7	97	13.0	13.4
Naphthalene	0.5	7	98	7.2	7.3

TABLE 7 (cont.)

Compound	Conc. (µg/L)	Number of Samples	% Recovery[a]	Standard Deviation of Recovery[b]	RSD
n-Propylbenzene	0.5	7	99	6.6	6.7
Styrene	0.5	7	96	19.0	19.8
1,1,1,2-Tetrachloroethane	0.5	7	100	4.7	4.7
1,1,2,2-Tetrachloroethane	0.5	7	100	12.0	12.0
Tetrachloroethene	0.1	7	96	5.0	5.2
Toluene	0.5	7	100	5.9	5.9
1,2,3-Trichlorobenzene	0.5	7	102	8.9	8.7
1,2,4-Trichlorobenzene	0.5	7	91	16.0	17.6
1,1,1-Trichloroethane	0.5	7	100	4.0	4.0
1,1,2-Trichloroethane	0.5	7	102	4.9	4.8
Trichloroethene	0.1	7	104	2.0	1.9
Trichlorofluoromethane	0.1	7	97	4.6	4.7
1,2,3-Trichloropropane	0.5	7	96	6.5	6.8
1,2,4-Trimethylbenzene	0.5	7	96	6.5	6.8
1,3,5-Trimethylbenzene	0.5	7	101	4.2	4.2
Vinyl chloride	0.1	7	104	0.2	0.2
o-Xylene	0.5	7	106	7.5	7.1
m-Xylene	0.5	7	106	4.6	4.3
p-Xylene	0.5	7	97	6.1	6.3

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[a] Recoveries were calculated using internal standard method. Internal standard was fluorobenzene.

TABLE 8

SURROGATE SPIKE RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES

Surrogate Compound	Water	Soil/Sediment
4-Bromofluorobenzene[a]	86-115	74-121
Dibromofluoromethane[a]	86-118	80-120
Toluene-d_8[a]	88-110	81-117
Dichloroethane-d_4[a]	80-120	80-120

[a] Single laboratory data, for guidance only.

TABLE 9

QUANTITY OF EXTRACT REQUIRED FOR ANALYSIS OF HIGH CONCENTRATION SAMPLES

Approximate Concentration Range (µg/kg)	Volume of Extract[a]
500 - 10,000	100 µL
1,000 - 20,000	50 µL
5,000 - 100,000	10 µL
25,000 - 500,000	100 µL of 1/50 dilution[b]

Calculate appropriate dilution factor for concentrations exceeding this table.

[a] The volume of solvent added to 5 mL of water being purged should be kept constant. Therefore, add to the 5-mL syringe whatever volume of solvent is necessary to maintain a volume of 100 µL added to the syringe.

[b] Dilute an aliquot of the solvent extract and then take 100 µL for analysis.

TABLE 10

DIRECT INJECTION ANALYSIS OF NEW OIL AT 5 PPM (METHOD 3585)

Compound	Recovery (%)	%RSD	Blank (ppm)	Spike (ppm)
Acetone	91	14.8	1.9	5.0
Benzene	86	21.3	0.1	0.5
n-Butanol*,**	107	27.8	0.5	5.0
iso-Butanol*,**	95	19.5	0.9	5.0
Carbon tetrachloride	86	44.7	0.0	0.5
Carbon disulfide**	53	22.3	0.0	5.0
Chlorobenzene	81	29.3	0.0	5.0
Chloroform	84	29.3	0.0	6.0
1,4-Dichlorobenzene	98	24.9	0.0	7.5
1,2-Dichloroethane	101	23.1	0.0	0.5
1,1-Dichloroethene	97	45.3	0.0	0.7
Diethyl ether	76	24.3	0.0	5.0
Ethyl acetate	113	27.4	0.0	5.0
Ethylbenzene	83	30.1	0.2	5.0
Hexachloroethane	71	30.3	0.0	3.0
Methylene chloride	98	45.3	0.0	5.0
Methyl ethyl ketone	79	24.6	0.4	5.0
MIBK	93	31.4	0.0	5.0
Nitrobenzene	89	30.3	0.0	2.0
Pyridine	31	35.9	0.0	5.0
Tetrachloroethene	82	27.1	0.0	0.7
Trichlorofluoromethane	76	27.6	0.0	5.0
1,1,2-Trichlorotrifluoroethane	69	29.2	0.0	5.0
Toluene	73	21.9	0.6	5.0
Trichloroethene	66	28.0	0.0	0.5
Vinyl chloride	63	35.2	0.0	0.2
o-Xylene	83	29.5	0.4	5.0
m/p-Xylene	84	29.5	0.6	10.0

* Alternate mass employed
** IS quantitation

Data are taken from Reference 9.

TABLE 11

SINGLE LABORATORY PERFORMANCE
DATA FOR THE DIRECT INJECTION METHOD - USED OIL (METHOD 3585)

Compound	Recovery (%)	%RSD	Blank (ppm)	Spike (ppm)
Acetone**	105	54	2.0	5.0
Benzene	3135	44	14	0.5
Benzene-d_6	56	44	2.9	0.5
n-Butanol**	100	71	12	5.0
iso-Butanol*,**	132	27	0	5.0
Carbon tetrachloride	143	68	0	0.5
Carbon tetrachloride-^{13}C	99	44	5.1	0.5
Carbon disulfide**	95	63	0	5.0
Chlorobenzene	148	71	0	5.0
Chlorobenzene-d_5	60	44	3.6	5.0
Chloroform	149	74	0	6.0
Chloroform-d_1	51	44	2.6	6.0
1,4-Dichlorobenzene	142	72	0	7.5
1,4-Dichlorobenzene-d_4	53	44	3.4	7.5
1,2-Dichloroethane**	191	54	0	0.5
1,1-Dichloroethene*	155	51	0	0.7
1,1-Dichloroethene-d_2	68	44	3.4	0.7
Diethyl ether**	95	66	0	5.0
Ethyl acetate*,**	126	39	0	5.0
Ethylbenzene	1298	44	54	5.0
Ethylbenzene-d_{10}	63	44	3.6	5.0
Hexachloroethane	132	72	0	3.0
Hexachloroethane-^{13}C	54	45	3.5	3.0
Methylene chloride**	86	65	0.3	5.0
Methyl ethyl ketone**	107	64	0	5.0
4-Methyl-2-pentanone (MIBK)**	100	74	0.1	5.0
Nitrobenzene	111	80	0	2.0
Nitrobenzene-d_5	65	53	4.0	2.0
Pyridine**	68	85	0	5.0
Pyridine-d_5	ND	--	0	5.0
Tetrachloroethene**	101	73	0	0.7
Trichlorofluoromethane**	91	70	0	5.0
1,1,2-Cl_3F_3ethane**	81	70	0	5.0
Toluene	2881	44	128	5.0
Toluene-d_8	63	44	3.6	5.0
Trichloroethene	152	57	0	0.5
Trichloroethene-d_1	55	44	2.8	0.5

TABLE 11 (cont.)

Compound	Recovery (%)	%RSD	Blank (ppm)	Spike (ppm)
Vinyl chloride**	100	69	0	0.2
o-Xylene	2292	44	105	5.0
o-Xylene-d_{10}	76	44	4.2	5.0
m-/p-Xylene	2583	44	253	10.0
p-Xylene-d_{10}	67	44	3.7	10.0

* Alternate mass employed
** IS quantitation
ND = Not Detected

Data are based on seven measurements and are taken from Reference 9.

TABLE 12

METHOD DETECTION LIMITS (METHOD 5031)

Compound	MDL (µg/L) Macro[a]	Concentration Factor Macro	Concentration Factor Micro
Acetone	31	25-500	-
Acetonitrile	57	25-500	200
Acrolein	-	-	100
Acrylonitrile	16	25-500	100
Allyl Alcohol	7	25-500	-
1-Butanol	-	-	250
Crotonaldehyde	12	25-500	-
1,4-Dioxane	12	25-500	150
Ethyl Acetate	-	-	100
Isobutyl alcohol	7	25-500	-
Methanol	38	25-500	140
Methyl Ethyl Ketone	16	25-500	-
2-Methyl-1-propanol	-	-	250
n-Nitroso-di-n-butylamine	14	25-500	-
Paraldehyde	10	25-500	-
2-Picoline	7	25-500	-
1-Propanol	-	-	240
Propionitrile	11	25-500	200
Pyridine	4	25-500	-
o-Toluidine	13	25-500	-

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[a] Produced by analysis of seven aliquots of reagent water spiked at 25 ppb at the listed compounds; calculations based on internal standard technique and use of the following equation:

MDL = 3.134 x Std. Dev. of low concentration spike (ppb).

[b] When a 40-mL sample is used, and the first 100 µL of distillate are collected.

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TABLE 13

TARGET COMPOUNDS, SURROGATES, AND INTERNAL STANDARDS (METHOD 5031)

Target Compound	Surrogate	Internal Standard
Acetone	d_6-Acetone	d_8-Isopropyl alcohol
Acetonitrile	d_3-Acetonitrile	d_8-Isopropyl alcohol
Acrylonitrile	d_8-Isopropyl alcohol	
Allyl alcohol	d_7-Dimethyl formamide	
Crotonaldehyde	d_8-Isopropyl alcohol	
1,4-Dioxane	d_8-1,4-Dioxane	d_7-Dimethyl formamide
Isobutyl alcohol	d_7-Dimethyl formamide	
Methanol	d_3-Methanol	d_8-Isopropyl alcohol
Methyl ethyl ketone	d_8-Isopropyl alcohol	
N-Nitroso-di-n-butylamine	d_7-Dimethyl formamide	
Paraldehyde	d_7-Dimethyl formamide	
2-Picoline	d_7-Dimethyl formamide	
Propionitrile	d_8-Isopropyl alcohol	
Pyridine	d_5-Pyridine	d_7-Dimethyl formamide
o-Toluidine	d_7-Dimethyl formamide	

TABLE 14

RECOMMENDED CONCENTRATIONS FOR CALIBRATION SOLUTIONS (METHOD 5031)

Compound	Concentration(s) (ng/μL)
Internal Standards	
d_5-benzyl alcohol	10.0
d_{14}-Diglyme	10.0
d_7-Dimethyl formamide	10.0
d_8-Isopropyl alcohol	10.0
Surrogates	
d_6-Acetone	10.0
d_3-Acetonitrile	10.0
d_8-1,4-Dioxane	10.0
d_3-Methanol	10.0
d_5-Pyridine	10.0
Target Compounds	
Acetone	1.0, 5.0, 10.0, 25.0, 100.0
Acetonitrile	1.0, 5.0, 10.0, 25.0, 100.0
Acrylonitrile	1.0, 5.0, 10.0, 25.0, 100.0
Allyl alcohol	1.0, 5.0, 10.0, 25.0, 100.0
Crotonaldehyde	1.0, 5.0, 10.0, 25.0, 100.0
1,4-Dioxane	1.0, 5.0, 10.0, 25.0, 100.0
Isobutyl alcohol	1.0, 5.0, 10.0, 25.0, 100.0
Methanol	1.0, 5.0, 10.0, 25.0, 100.0
Methyl ethyl ketone	1.0, 5.0, 10.0, 25.0, 100.0
N-Nitroso-di-n-butylamine	1.0, 5.0, 10.0, 25.0, 100.0
Paraldehyde	1.0, 5.0, 10.0, 25.0, 100.0
2-Picoline	1.0, 5.0, 10.0, 25.0, 100.0
Propionitrile	1.0, 5.0, 10.0, 25.0, 100.0
Pyridine	1.0, 5.0, 10.0, 25.0, 100.0
o-Toluidine	1.0, 5.0, 10.0, 25.0, 100.0

TABLE 15

CHARACTERISTIC IONS AND RETENTION TIMES FOR VOCs (METHOD 5031)

Compound	Quantitation Ion[a]	Secondary Ions	Retention Time (min)[b]
Internal Standards			
d_8-Isopropyl alcohol	49		1.75
d_{14}-Diglyme	66	98,64	9.07
d_7-Dimethyl formamide	50	80	9.20
Surrogates			
d_6-Acetone	46	64,42	1.03
d_3-Methanol	33	35,30	1.75
d_3-Acetonitrile	44	42	2.63
d_8-1,4-Dioxane	96	64,34	3.97
d_5-Pyridine	84	56,79	6.73
d_5-Phenol[c]	99	71	15.43
Target Compounds			
Acetone	43	58	1.05
Methanol	31	29	1.52
Methyl ethyl ketone	43	72,57	1.53
Methacrylonitrile[c]	67	41	2.38
Acrylonitrile	53	52,51	2.53
Acetonitrile	41	40,39	2.73
Methyl isobutyl ketone[c]	85	100,58	2.78
Propionitrile	54	52,55	3.13
Crotonaldehyde	41	70	3.43
1,4-Dioxane	58	88,57	4.00
Paraldehyde	45	89	4.75
Isobutyl alcohol	43	33,42	5.05
Allyl alcohol	57	39	5.63
Pyridine	79	50,52	6.70
2-Picoline	93	66	7.27
N-Nitroso-di-n-butylamine	84	116	12.82
Aniline[c]	93	66,92	13.23
o-Toluidine	106	107	13.68
Phenol[c]	94	66,65	15.43

[a] These ions were used for quantitation in selected ion monitoring.

[b] GC column: DB-Wax, 30 meter x 0.53 mm, 1 µm film thickness.
Oven program: 45°C for 4 min, increased to 220°C at 12°C/min.

[c] Compound removed from target analyte list due to poor accuracy and precision.

TABLE 16

METHOD ACCURACY AND PRECISION BY MEAN PERCENT RECOVERY AND PERCENT RELATIVE STANDARD DEVIATION[a] (METHOD 5031 - MACRODISTILLATION TECHNIQUE)

(Single Laboratory and Single Operator)

	25 ppb Spike		100 ppb Spike		500 ppb Spike	
Compound	Mean %R	%RSD	Mean %R	%RSD	Mean %R	%RSD
d_6-Acetone	66	24	69	14	65	16
d_3-Acetonitrile	89	18	80	18	70	10
d_8-1,4-Dioxane	56	34	58	11	61	18
d_3-Methanol	43	29	48	19	56	14
d_5-Pyridine	83	6.3	84	7.8	85	9.0
Acetone	67	45	63	14	60	14
Acetonitrile	44	35	52	15	56	15
Acrylonitrile	49	42	47	27	45	27
Allyl alcohol	69	13	70	9.7	73	10
Crotonaldehyde	68	22	68	13	69	13
1,4-Dioxane	63	25	55	16	54	13
Isobutyl alcohol	66	14	66	5.7	65	7.9
Methanol	50	36	46	22	49	18
Methyl ethyl ketone	55	37	56	20	52	19
N-Nitroso-di-n-butylamine	57	21	61	15	72	18
Paraldehyde	65	20	66	11	60	8.9
Picoline	81	12	81	6.8	84	8.0
Propionitrile	67	22	69	13	68	13
Pyridine	74	7.4	72	6.7	74	7.3
o-Toluidine	52	31	54	15	58	12

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[a] Data from analysis of seven aliquots of reagent water spiked at each concentration, using a quadrapole mass spectrometer in the selected ion monitoring mode.

TABLE 17

RECOVERIES IN SAND SAMPLES FORTIFIED AT 4 µg/kg (ANALYSIS BY METHOD 5035)

		Recovery per Replicate (ng)						Mean
Compound	1	2	3	4	5	Mean	RSD	Rec
Vinyl chloride	8.0	7.5	6.7	5.4	6.6	6.8	13.0	34.2
Trichlorofluoromethane	13.3	16.5	14.9	13.0	10.3	13.6	15.2	68.0
1,1-Dichloroethene	17.1	16.7	15.1	14.8	15.6	15.9	5.7	79.2
Methylene chloride	24.5	22.7	19.7	19.4	20.6	21.4	9.1	107
trans-1,2-Dichloroethene	22.7	23.6	19.4	18.3	20.1	20.8	0.7	104
1,2-Dichloroethane	18.3	18.0	16.7	15.6	15.9	16.9	6.4	84.4
cis-1,2-Dichloroethene	26.1	23.1	22.6	20.3	20.8	22.6	9.0	113
Bromochloromethane	24.5	25.4	20.9	20.1	20.1	22.2	10.2	111
Chloroform	26.5	26.0	22.1	18.9	22.1	23.1	12.2	116
1,1,1-Trichloroethane	21.5	23.0	23.9	16.7	31.2	23.4	21.2	117
Carbon tetrachloride	23.6	24.2	22.6	18.3	23.3	22.4	9.4	112
Benzene	22.4	23.9	20.4	17.4	19.2	20.7	11.2	103
Trichloroethene	21.5	20.5	19.2	14.4	19.1	18.9	12.7	94.6
1,2-Dichloropropane	24.9	26.3	23.1	19.0	23.3	23.3	10.5	117
Dibromomethane	25.4	26.4	21.6	20.4	23.6	23.5	9.6	117
Bromodichloromethane	25.7	26.7	24.1	17.9	23.0	23.5	13.1	117
Toluene	28.3	25.0	24.8	16.3	23.6	23.6	16.9	118
1,1,2-Trichloroethane	25.4	24.5	21.6	17.7	22.1	22.2	12.1	111
1,3-Dichloropropane	25.4	24.2	22.7	17.0	22.2	22.3	12.8	112
Dibromochloromethane	26.3	26.2	23.7	18.2	23.2	23.5	12.5	118
Chlorobenzene	22.9	22.5	19.8	14.6	19.4	19.9	15.0	99.3
1,1,1,2-Tetrachloroethane	22.4	27.7	25.1	19.4	22.6	23.4	12.0	117
Ethylbenzene	25.6	25.0	22.1	14.9	24.0	22.3	17.5	112
p-Xylene	22.5	22.0	19.8	13.9	20.3	19.7	15.7	98.5
o-Xylene	24.2	23.1	21.6	14.0	20.4	20.7	17.3	103
Styrene	23.9	21.5	20.9	14.3	20.5	20.2	15.7	101
Bromoform	26.8	25.6	26.0	20.1	23.5	24.4	9.9	122
iso-Propylbenzene	25.3	25.1	24.2	15.4	24.6	22.9	16.6	114
Bromobenzene	19.9	21.8	20.0	15.5	19.1	19.3	10.7	96.3
1,2,3-Trichloropropane	25.9	23.0	25.6	15.9	21.4	22.2	15.8	111
n-Propylbenzene	26.0	23.8	22.6	13.9	21.9	21.6	19.0	106
2-Chlorotoluene	23.6	23.8	21.3	13.0	21.5	20.6	19.2	103
4-Chlorotoluene	21.0	19.7	18.4	12.1	18.3	17.9	17.1	89.5
1,3,5-Trimethylbenzene	24.0	22.1	22.5	13.8	22.9	21.1	17.6	105
sec-Butylbenzene	25.9	25.3	27.8	16.1	28.6	24.7	18.1	124
1,2,4-Trimethylbenzene	30.6	39.2	22.4	18.0	22.7	26.6	28.2	133
1,3-Dichlorobenzene	20.3	20.6	18.2	13.0	17.6	17.9	15.2	89.7
p-iso-Propyltoluene	21.6	22.1	21.6	16.0	22.8	20.8	11.8	104
1,4-Dichlorobenzene	18.1	21.2	20.0	13.2	17.4	18.0	15.3	90.0
1,2-Dichlorobenzene	18.4	22.5	22.5	15.2	19.9	19.7	13.9	96.6
n-Butylbenzene	13.1	20.3	19.5	10.8	18.7	16.5	23.1	82.4
1,2,4-Trichlorobenzene	14.5	14.9	15.7	8.8	12.3	13.3	18.8	66.2
Hexachlorobutadiene	17.6	22.5	21.6	13.2	21.6	19.3	18.2	96.3
1,2,3-Trichlorobenzene	14.9	15.9	16.5	11.9	13.9	14.6	11.3	73.1

Data in Tables 17, 18, and 19 are from Reference 15.

TABLE 18
RECOVERIES IN C-HORIZON SOILS FORTIFIED AT 4 µg/kg (ANALYSIS BY METHOD 5035)

	Recovery per Replicate (ng)							Mean
Compound	1	2	3	4	5	Mean	RSD	Rec
Vinyl chloride	33.4	31.0	30.9	29.7	28.6	30.8	5.2	154
Trichlorofluoromethane	37.7	20.8	20.0	21.8	20.5	24.1	28.2	121
1,1-Dichloroethene	21.7	33.5	39.8	30.2	32.5	31.6	18.5	158
Methylene chloride	20.9	19.4	18.7	18.3	18.4	19.1	5.1	95.7
trans-1,2-Dichloroethene	21.8	18.9	20.4	17.9	17.8	19.4	7.9	96.8
1,1-Dichloroethane	23.8	21.9	21.3	21.3	20.5	21.8	5.2	109
cis-1,2-Dichloroethene	21.6	18.8	18.5	18.2	18.2	19.0	6.7	95.2
Bromochloromethane	22.3	19.5	19.3	19.0	19.2	20.0	6.0	100
Chloroform	20.5	17.1	17.3	16.5	15.9	17.5	9.2	87.3
1,1,1-Trichloroethane	16.4	11.9	10.7	9.5	9.4	11.6	22.4	57.8
Carbon tetrachloride	13.1	11.3	13.0	11.8	11.2	12.1	6.7	60.5
Benzene	21.1	19.3	18.7	18.2	16.9	18.8	7.4	94.1
Trichloroethene	19.6	16.4	16.5	16.5	15.5	16.9	8.3	84.5
1,2-Dichloropropane	21.8	19.0	18.3	18.8	16.5	18.9	9.0	94.4
Dibromomethane	20.9	17.9	17.9	17.2	18.3	18.4	6.9	92.1
Bromodichloromethane	20.9	18.0	18.9	18.2	17.3	18.6	6.6	93.2
Toluene	22.2	17.3	18.8	17.0	15.9	18.2	12.0	91.2
1,1,2-Trichloroethane	21.0	16.5	17.2	17.2	16.5	17.7	9.6	88.4
1,3-Dichloropropane	21.4	17.3	18.7	18.6	16.7	18.5	8.8	92.6
Dibromochloromethane	20.9	18.1	19.0	18.8	16.6	18.7	7.5	93.3
Chlorobenzene	20.8	18.4	17.6	16.8	14.8	17.7	11.2	88.4
1,1,1,2-Tetrachloroethane	19.5	19.0	17.8	17.2	16.5	18.0	6.2	90.0
Ethylbenzene	21.1	18.3	18.5	16.9	15.3	18.0	10.6	90.0
p-Xylene	20.0	17.4	18.2	16.3	14.4	17.3	10.9	86.3
o-Xylene	20.7	17.2	16.8	16.2	14.8	17.1	11.4	85.7
Styrene	18.3	15.9	16.2	15.3	13.7	15.9	9.3	79.3
Bromoform	20.1	15.9	17.1	17.5	16.1	17.3	8.6	86.7
iso-Propylbenzene	21.0	18.1	19.2	18.4	15.6	18.4	9.6	92.2
Bromobenzene	20.4	16.2	17.2	16.7	15.4	17.2	10.1	85.9
1,1,2,2-Tetrachloroethane	23.3	17.9	21.2	18.8	16.8	19.6	12.1	96.0
1,2,3-Trichloropropane	18.4	14.6	15.6	16.1	15.6	16.1	8.0	80.3
n-Propylbenzene	20.4	18.9	17.9	17.0	14.3	17.7	11.6	88.4
2-Chlorotoluene	19.1	17.3	16.1	16.0	14.4	16.7	9.2	83.6
4-Chlorotoluene	19.0	15.5	16.8	15.9	13.6	16.4	10.6	81.8
1,3,5-Trimethylbenzene	20.8	18.0	17.4	16.1	14.7	17.4	11.7	86.9
sec-Butylbenzene	21.4	18.3	18.9	17.0	14.9	18.1	11.8	90.5
1,2,4-Trimethylbenzene	20.5	18.6	16.8	15.3	13.7	17.0	14.1	85.0
1,3-Dichlorobenzene	17.6	15.9	15.6	14.2	14.4	15.6	7.9	77.8
p-iso-Propyltoluene	20.5	17.0	17.1	15.6	13.4	16.7	13.9	83.6
1,4-Dichlorobenzene	18.5	13.8	14.8	16.7	14.9	15.7	10.5	78.7
1,2-Dichlorobenzene	18.4	15.0	15.4	15.3	13.5	15.5	10.5	77.6
n-Butylbenzene	19.6	15.9	15.9	14.4	18.9	16.9	11.7	84.6
1,2,4-Trichlorobenzene	15.2	17.2	17.4	13.6	12.1	15.1	13.5	75.4
Hexachlorobutadiene	18.7	16.2	15.5	13.8	16.6	16.1	10.0	80.7
Naphthalene	13.9	11.1	10.2	10.8	11.4	11.5	11.0	57.4
1,2,3-Trichlorobenzene	14.9	15.2	16.8	13.7	12.7	14.7	9.5	73.2

TABLE 19
RECOVERIES IN GARDEN SOIL FORTIFIED AT 4 µg/kg (ANALYSIS BY METHOD 5035)

	Recovery per Replicate (ng)							Mean
Compound	1	2	3	4	5	Mean	RSD	Rec
Vinyl chloride	12.7	10.9	9.8	8.1	7.2	9.7	20.2	48.7
Trichlorofluoromethane	33.7	6.4	30.3	27.8	22.9	24.2	39.6	121
1,1-Dichloroethene	27.7	20.5	24.1	15.1	13.2	20.1	26.9	101
Methylene chloride	25.4	23.9	24.7	22.2	24.2	24.1	4.4	120
trans-1,2-Dichloroethene	2.8	3.0	3.3	2.2	2.4	2.7	15.0	13.6
1,1-Dichloroethane	24.1	26.3	27.0	20.5	21.2	23.8	11.0	119
cis-1,2-Dichloroethene	8.3	10.2	8.7	5.8	6.4	7.9	20.1	39.4
Bromochloromethane	11.1	11.8	10.2	8.8	9.0	10.2	11.2	50.9
Chloroform	16.7	16.9	17.0	13.8	15.0	15.9	7.9	79.3
1,1,1-Trichloroethane	24.6	22.8	22.1	16.2	20.9	21.3	13.4	107
Carbon tetrachloride	19.4	20.3	22.2	20.0	20.2	20.4	4.6	102
Benzene	21.4	22.0	22.4	19.6	20.4	21.2	4.9	106
Trichloroethene	12.4	16.5	14.9	9.0	9.9	12.5	22.9	62.7
1,2-Dichloropropane	19.0	18.8	19.7	16.0	17.6	18.2	7.1	91.0
Dibromomethane	7.3	8.0	6.9	5.6	6.8	6.9	11.3	34.6
Bromodichloromethane	14.9	15.9	15.9	12.8	13.9	14.7	8.3	73.3
Toluene	42.6	39.3	45.1	39.9	45.3	42.4	5.9	212
1,1,2-Trichloroethane	13.9	15.2	1.4	21.3	14.9	15.9	17.0	79.6
1,3-Dichloropropane	13.3	16.7	11.3	10.9	9.5	12.3	20.3	61.7
Dibromochloromethane	14.5	13.1	14.5	11.9	14.4	13.7	7.6	68.3
Chlorobenzene	8.4	10.0	8.3	6.9	7.8	8.3	12.1	41.3
1,1,1,2-Tetrachloroethane	16.7	16.7	15.6	15.8	15.7	16.1	3.2	80.4
Ethylbenzene	22.1	21.4	23.1	20.1	22.6	21.9	4.8	109
p-Xylene	41.4	38.4	43.8	38.3	44.0	41.2	6.1	206
o-Xylene	31.7	30.8	34.3	30.4	33.2	32.1	4.6	160
Styrene	0	0	0	0	0	0	0	0
Bromoform	8.6	8.9	9.1	7.0	7.7	8.3	9.4	41.4
iso-Propylbenzene	18.1	18.8	9.7	18.3	19.6	18.9	3.5	94.4
Bromobenzene	5.1	5.4	5.3	4.4	4.0	4.8	11.6	24.1
1,1,2,2-Tetrachloroethane	14.0	13.5	14.7	15.3	17.1	14.9	8.5	74.5
1,2,3-Trichloropropane	11.0	12.7	11.7	11.7	11.9	11.8	4.5	59.0
n-Propylbenzene	13.4	13.3	14.7	12.8	13.9	13.6	4.7	68.1
2-Chlorotoluene	8.3	9.0	11.7	8.7	7.9	9.1	14.8	45.6
4-Chlorotoluene	5.1	5.4	5.5	4.8	4.5	5.0	7.9	25.2
1,3,5-Trimethylbenzene	31.3	27.5	33.0	31.1	33.6	31.3	6.8	157
sec-Butylbenzene	13.5	13.4	16.4	13.8	15.4	14.5	8.3	72.5
1,2,4-Trimethylbenzene	38.7	32.4	40.8	34.1	40.3	37.3	9.1	186
1,3-Dichlorobenzene	3.6	3.6	3.7	3.0	3.2	3.4	8.0	17.2
p-iso-Propyltoluene	14.7	14.1	16.1	13.9	15.1	14.8	5.2	73.8
1,4-Dichlorobenzene	3.0	3.5	3.3	2.6	2.8	3.0	10.2	15.0
1,2-Dichlorobenzene	3.6	4.3	4.0	3.5	3.6	3.8	8.3	19.0
n-Butylbenzene	17.4	13.8	14.0	18.9	24.0	17.6	21.2	88.0
1,2,4-Trichlorobenzene	2.8	2.9	3.3	2.6	3.2	3.0	8.5	15.0
Hexachlorobutadiene	4.8	4.0	6.1	5.6	6.0	5.3	15.1	26.4
Naphthalene	5.5	5.1	5.5	4.7	5.6	5.3	6.2	26.5
1,2,3-Trichlorobenzene	2.2	2.3	2.4	2.2	2.3	2.3	3.5	11.4

Data in Table 19 are from Reference 15.

TABLE 20

VOLATILE ORGANIC ANALYTE RECOVERY FROM SOIL USING VACUUM DISTILLATION (METHOD 5032)[a]

	Soil/H_2O[b] Recovery		Soil/Oil[c] Recovery		Soil/Oil/H_2O Recovery	
Compound	Mean	RSD	Mean	RSD	Mean	RSD
Chloromethane	61	20	40	18	108	68
Bromomethane	58	20	47	13	74	13
Vinyl chloride	54	12	46	11	72	20
Chloroethane	46	10	41	8	52	14
Methylene chloride	60	2	65	8	76	11
Acetone	INT[e]	INT	44	8		
Carbon disulfide	47	13	53	10	47	4
1,1-Dichloroethene	48	9	47	5	58	3
1,1-Dichloroethane	61	6	58	9	61	6
trans-1,2-Trichloroethane	54	7	60	7	56	5
cis-1,2-Dichloroethene	60	4	72	6	63	8
Chloroform	104	11	93	6	114	15
1,2-Dichloroethane	177	50	117	8	151	22
2-Butanone	INT	36	38	INT		
1,1,1-Trichloroethane	124	13	72	16	134	26
Carbon tetrachloride	172	122	INT	INT		
Vinyl acetate	88	11	INT			
Bromodichloromethane	93	4	91	23	104	23
1,1,2,2-Tetrachloroethane	96	13	50	12	104	7
1,2-Dichloropropane	105	8	102	6	111	6
trans-1,3-Dichloropropene	134	10	84	16	107	8
Trichloroethene	98	9	99	10	100	5
Dibromochloromethane	119	8	125	31	142	16
1,1,2-Trichloroethane	126	10	72	16	97	4
Benzene	99	7	CONT[f]	CONT		
cis-1,3-Dichloropropene	123	12	94	13	112	9
Bromoform	131	13	58	18	102	9
2-Hexanone	155	18	164	19	173	29
4-Methyl-2-pentanone	152	20	185	20	169	18
Tetrachloroethene	90	9	123	14	128	7
Toluene	94	3	CONT	CONT		
Chlorobenzene	98	7	93	18	112	5
Ethylbenzene	114	13	CONT	CONT		
Styrene	106	8	93	18	112	5
p-Xylene	97	9	CONT	CONT		
o-Xylene	105	8	112	12	144	13

TABLE 20 (cont.)

Compound	Soil/H_2O[b] Recovery Mean	Soil/H_2O[b] Recovery RSD	Soil/Oil[c] Recovery Mean	Soil/Oil[c] Recovery RSD	Soil/Oil/H_2O Recovery Mean	Soil/Oil/H_2O Recovery RSD
Surrogates						
1,2-Dichloroethane	177	50	117	8	151	22
Toluene-d_8	96	6	79	12	82	6
Bromofluorobenzene	139	13	37	13	62	5

[a] Results are for 10 min. distillations times, and condenser temperature held at -10°C. A 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness was used for chromatography. Standards and samples were replicated and precision value reflects the propagated errors. Each analyte was spiked at 50 ppb. Vacuum distillation efficiencies (Method 5032) are modified by internal standard corrections. Method 8260 internal standards may introduce bias for some analytes. See Method 5032 to identify alternate internal standards with similar efficiencies to minimize bias.

[b] Soil samples spiked with 0.2 mL water containing analytes and then 5 mL water added to make slurry.

[c] Soil sample + 1 g cod liver oil, spiked with 0.2 mL water containing analytes.

[d] Soil samples + 1 g cod liver oil, spiked as above with 5 mL of water added to make slurry.

[e] Interference by co-eluting compounds prevented accurate measurement of analyte.

[f] Contamination of sample matrix by analyte prevented assessment of efficiency.

TABLE 21

VACUUM DISTILLATION EFFICIENCIES FOR VOLATILE ORGANIC ANALYTES IN FISH TISSUE (METHOD 5032)[a]

Compound	Efficiency Mean (%)	RSD (%)
Chloromethane	N/A[b]	
Bromomethane	N/A[b]	
Vinyl chloride	N/A[b]	
Chloroethane	N/A[b]	
Methylene chloride	CONT[c]	
Acetone	CONT[c]	
Carbon disulfide	79	36
1,1-Dichloroethene	122	39
1,1-Dichloroethane	126	35
trans-1,2-Trichloroethene	109	46
cis-1,2-Dichloroethene	106	22
Chloroform	111	32
1,2-Dichloroethane	117	27
2-Butanone	INT[d]	
1,1,1-Trichloroethane	106	30
Carbon tetrachloride	83	34
Vinyl acetate	INT[d]	
Bromodichloromethane	97	22
1,1,2,2-Tetrachloroethane	67	20
1,2-Dichloropropane	117	23
trans-1,3-Dichloropropene	92	22
Trichloroethene	98	31
Dibromochloromethane	71	19
1,1,2-Trichloroethane	92	20
Benzene	129	35
cis-1,3-Dichloropropene	102	24
Bromoform	58	19
2-Hexanone	INT[d]	
4-Methyl-2-pentanone	113	37
Tetrachloroethene	66	20
Toluene	CONT[c]	
Chlorobenzene	65	19
Ethylbenzene	74	19
Styrene	57	14
p-Xylene	46	13
o-Xylene	83	20

TABLE 21 (cont.)

Compound	Efficiency Mean (%)	RSD (%)
Surrogates		
1,2-Dichloroethane	115	27
Toluene-d_8	88	24
Bromofluorobenzene	52	15

[a] Results are for 10 min. distillation times and condenser temperature held at -10°C. Five replicate 10-g aliquots of fish spiked at 25 ppb were analyzed using GC/MS external standard quantitation. A 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness was used for chromatography. Standards were replicated and results reflect 1 sigma propagated standard deviation.

[b] No analyses.

[c] Contamination of sample matrix by analyte prevented accurate assessment of analyte efficiency.

[d] Interfering by co-eluting compounds prevented accurate measurement of analyte.

TABLE 22

METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC ANALYTES IN FISH TISSUE (METHOD 5032)[a]

	Method Detection Limit (ppb)	
Compound	External Standard Method	Internal Standard Method
Chloromethane	7.8	7.3
Bromomethane	9.7	9.8
Vinyl chloride	9.5	9.4
Chloroethane	9.2	10.0
Methylene chloride	CONT[b]	CONT[b]
Acetone	CONT[b]	CONT[b]
Carbon disulfide	5.4	4.9
1,1-Dichloroethene	4.0	5.7
1,1-Dichloroethane	4.0	3.5
trans-1,2-Dichloroethene	4.4	4.0
cis-1,2-Dichloroethene	4.7	4.1
Chloroform	5.6	5.0
1,2-Dichloroethane	3.3	3.2
2-Butanone	INT[c]	INT[c]
1,1,1-Trichloroethane	1.1	4.2
Carbon tetrachloride	3.2	3.5
Vinyl acetate	INT[c]	INT[c]
Bromodichloromethane	3.2	2.8
1,1,2,2-Tetrachloroethane	4.4	3.8
1,2-Dichloropropane	3.8	3.7
trans-1,3-Dichloropropene	3.4	3.0
Trichloroethene	3.1	4.0
Dibromochloromethane	3.5	3.2
1,1,2-Trichloroethane	4.4	3.3
Benzene	3.6	3.2
cis-1,3-Dichloropropene	3.5	3.0
Bromoform	4.9	4.0
2-Hexanone	7.7	8.0
4-Methyl-2-pentanone	7.5	8.0
Tetrachloroethene	4.3	4.0
Toluene	3.0	2.5
Chlorobenzene	3.3	2.8
Ethylbenzene	3.6	3.5
Styrene	3.5	3.3
p-Xylene	3.7	3.5
o-Xylene	3.3	4.7

Footnotes are on the following page.

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[a] Values shown are the average MDLs for studies on three non-consecutive days, involving seven replicate analyses of 10 g of fish tissue spiked a 5 ppb. Daily MDLs were calculated as three times the standard deviation. Quantitation was performed by GC/MS Method 8260 and separation with a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness.

[b] Contamination of sample by analyte prevented determination.

[c] Interference by co-eluting compounds prevented accurate quantitation.

TABLE 23

VOLATILE ORGANIC ANALYTES RECOVERY FOR WATER USING VACUUM DISTILLATION (METHOD 5032)[a]

Compound	5 mL H_2O^b Recovery Mean	RSD	20 mL H_2O^c Recovery Mean	RSD	20 mL H_2O/Oil Recovery Mean	RSD
Chloromethane	114	27	116	29	176	67
Bromomethane	131	14	121	14	113	21
Vinyl chloride	131	13	120	16	116	23
Chloroethane	110	15	99	8	96	16
Methylene chloride	87	16	105	15	77	6
Acetone	83	22	65	34	119	68
Carbon disulfide	138	17	133	23	99	47
1,1-Dichloroethene	105	11	89	4	96	18
1,1-Dichloroethane	118	10	119	11	103	25
trans-1,2-Dichloroethene	105	11	107	14	96	18
cis-1,2-Dichloroethene	106	7	99	5	104	23
Chloroform	114	6	104	8	107	21
1,2-Dichloroethane	104	6	109	8	144	19
2-Butanone	83	50	106	31	INT[c]	
1,1,1-Trichloroethane	118	9	109	9	113	23
Carbon tetrachloride	102	6	108	12	109	27
Vinyl acetate	90	16	99	7	72	36
Bromodichloromethane	104	3	110	5	99	5
1,1,2,2-Tetrachloroethane	85	17	81	7	111	43
1,2-Dichloropropane	100	6	103	2	104	7
trans-1,3-Dichloropropene	105	8	105	4	92	4
Trichloroethene	98	4	99	2	95	5
Dibromochloroethane	99	8	99	6	90	25
1,1,2-Trichloroethane	98	7	100	4	76	12
Benzene	97	4	100	5	112	10
cis-1,3-Dichloropropene	106	5	105	4	98	3
Bromoform	93	16	94	8	57	21
2-Hexanone	60	17	63	16	78	23
4-Methyl-2-pentanone	79	24	63	14	68	15
Tetrachloroethene	101	3	97	7	77	14
Toluene	100	6	97	8	85	5
Chlorobenzene	98	6	98	4	88	16
Ethylbenzene	100	3	92	8	73	13
Styrene	98	4	97	9	88	16
p-Xylene	96	4	94	8	60	12
o-Xylene	96	7	95	6	72	14

TABLE 23 (cont.)

Compound	5 mL H_2O^b Recovery Mean	RSD	20 mL H_2O^c Recovery Mean	RSD	20 mL H_2O/Oil Recovery Mean	RSD
Surrogates						
1,2-Dichloroethane	104	6	109	6	144	19
Toluene-d_8	104	5	102	2	76	7
Bromofluorobenzene	106	6	106	9	40	8

[a] Results are for 10 min. distillation times, and condenser temperature held at -10°C. A 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness was used for chromatography. Standards and samples were replicated and precision values reflect the propagated errors. Concentrations of analytes were 50 ppb for 5-mL samples and 25 ppb for 20-mL samples. Recovery data generated with comparison to analyses of standards without the water matrix.

[b] Sample contained 1 gram cod liver oil and 20 mL water. An emulsion was created by adding 0.2 mL of water saturated with lecithin.

[c] Interference by co-eluting compounds prevented accurate assessment of recovery.

TABLE 24

METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC ANALYTES USING VACUUM DISTILLATION (METHOD 5032) (INTERNAL STANDARD METHOD)[a]

Compound	Water[b] (µg/L)	Soil[c] (µg/kg)	Tissue[d] (µg/kg)	Oil[e] (mg/kg)
Chloromethane	3.2	8.0	7.3	N/A[f]
Bromomethane	2.8	4.9	9.8	N/A[f]
Vinyl chloride	3.5	6.0	9.4	N/A[f]
Chloroethane	5.9	6.0	10.0	N/A[f]
Methylene chloride	3.1	4.0	CONT[g]	0.05
Acetone	5.6	CONT[g]	CONT[g]	0.06
Carbon disulfide	2.5	2.0	4.9	0.18
1,1-Dichloroethene	2.9	3.2	5.7	0.18
1,1-Dichloroethane	2.2	2.0	3.5	0.14
trans-1,2-Dichloroethene	2.2	1.4	4.0	0.10
cis-1,2-Dichloroethene	2.0	2.3	4.1	0.07
Chloroform	2.4	1.8	5.0	0.07
1,2-Dichloroethane	1.7	1.5	3.2	0.06
2-Butanone	7.4	INT[h]	INT[h]	INT[h]
1,1,1-Trichloroethane	1.8	1.7	4.2	0.10
Carbon tetrachloride	1.4	1.5	3.5	0.13
Vinyl acetate	11.8	INT[h]	INT[h]	INT[h]
Bromodichloromethane	1.6	1.4	2.8	0.06
1,1,2,2-Tetrachloroethane	2.5	2.1	3.8	0.02
1,2-Dichloropropane	2.2	2.1	3.7	0.15
trans-1,3-Dichloropropene	1.5	1.7	3.0	0.05
Trichloroethene	1.6	1.7	4.0	0.04
Dibromochloromethane	1.7	1.5	3.2	0.07
1,1,2-Trichloroethane	2.1	1.7	3.3	0.05
Benzene	0.5	1.5	3.2	0.05
cis-1,3-Dichloropropene	1.4	1.7	3.0	0.04
Bromoform	1.8	1.5	4.0	0.05
2-Hexanone	4.6	3.6	8.0	INT[h]
4-Methyl-2-pentanone	3.5	4.6	8.0	INT[h]
Tetrachloroethene	1.4	1.6	4.0	0.10
Toluene	1.0	3.3	2.5	0.05
Chlorobenzene	1.4	1.4	2.8	0.06
Ethylbenzene	1.5	2.8	3.5	0.04
Styrene	1.4	1.4	3.3	0.18
p-Xylene	1.5	2.9	3.5	0.20
o-Xylene	1.7	3.4	4.7	0.07

Footnotes are found on the following page.

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[a] Quantitation was performed using GC/MS Method 8260 and chromatographic separation with a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness. Method detection limits are the average MDLs for studies on three non-consecutive days.

[b] Method detection limits are the average MDLs for studies of three non-consecutive days. Daily studies were seven replicated analyses of 5 mL aliquots of 4 ppb soil. Daily MDLs were three times the standard deviation.

[c] Daily studies were seven replicated analyses of 10 g fish tissue spiked at 5 ppb. Daily MDLs were three times the standard deviation. Quantitation was performed using GC/MS Method 8260 and chromatographic separation with a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness.

[d] Method detection limits are estimated analyzing 1 g of cod liver oil samples spiked at 250 ppm. Five replicates were analyzed using Method 8260.

[e] No analyses.

[f] Contamination of sample by analyte prevented determination.

[g] Interference by co-eluting compounds prevented accurate quantitation.

TABLE 25

METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC ANALYTES (METHOD 5032) (EXTERNAL STANDARD METHOD)[a]

Compound	Water[b] (µg/L)	Soil[c] (µg/kg)	Tissue[d] (µg/kg)	Oil[e] (mg/kg)
Chloromethane	3.1	8.6[f]	7.8	N/A[g]
Bromomethane	2.5	4.9[f]	9.7	N/A[g]
Vinyl chloride	4.0	7.1[f]	9.5	N/A[g]
Chloroethane	6.1	7.5[f]	9.2	N/A[g]
Methylene chloride	3.1	3.3	CONT[h]	0.08
Acetone	33.0[f]	CONT[h]	CONT[h]	0.12
Carbon disulfide	2.5	3.2	5.4	0.19
1,1-Dichloroethene	3.4	3.8	4.0	0.19
1,1-Dichloroethane	2.3	1.7	4.0	0.13
trans-1,2-Dichloroethene	3.0	3.2	4.4	0.09
cis-1,2-Dichloroethene	2.4	2.7	4.7	0.08
Chloroform	2.7	2.6	5.6	0.06
1,2-Dichloroethane	1.6	1.7	3.3	0.06
2-Butanone	57.0[f]	INT[i]	INT[i]	INT[i]
1,1,1-Trichloroethane	1.6	2.4	1.1	0.08
Carbon tetrachloride	1.5	1.7	3.2	0.15
Vinyl acetate	23.0[f]	INT[i]	INT[i]	INT[i]
Bromodichloromethane	2.0	2.3	3.2	0.05
1,1,2,2-Tetrachloroethane	3.6	3.2	4.4	0.09
1,2-Dichloropropane	2.9	3.7	3.8	0.12
trans-1,3-Dichloropropene	2.3	2.4	3.8	0.08
Trichloroethene	2.5	3.0	3.1	0.06
Dibromochloromethane	2.1	2.9	3.5	0.04
1,1,2-Trichloroethane	2.7	2.8	4.4	0.07
Benzene	1.7	2.9	3.6	0.03
cis-1,3-Dichloropropene	2.1	2.5	3.5	0.06
Bromoform	2.3	2.5	4.9	0.10
2-Hexanone	4.6	4.6	7.7	INT[i]
4-Methyl-2-pentanone	3.8	3.9	7.5	INT[i]
Tetrachloroethene	1.8	2.6	4.3	0.12
Toluene	1.8	4.4	3.0	0.09
Chlorobenzene	2.4	2.6	3.3	0.07
Ethylbenzene	2.4	4.1	3.6	0.09
Styrene	2.0	2.5	3.5	0.16
p-Xylene	2.3	3.9	3.7	0.18
o-Xylene	2.4	4.1	3.3	0.08

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[a] Method detection limits are the average MDLs for studies on three non-consecutive days. Daily studies were seven replicate analyses of 5-mL aliquots of water spiked at 4 ppb. Daily MDLs were three times the standard deviation.

[b] Daily studies were seven replicate analyses of 5-mL aliquots of water spiked at 4 ppb.

[c] These studies were seven replicate analyses of 5-g aliquots of soil spiked at 4 ppb.

[d] These studies were seven replicate analyses of 10-g aliquots of fish tissue spiked at 5 ppb.

[e] Method detection limits were estimated by analyzing cod liver oil samples spiked at 250 ppb. Five replicates were analyzed using Method 8260.

[f] Method detection limits were estimated by analyzing replicate 50 ppb standards five times over a single day.

[g] No analyses.

[h] Contamination of sample by analyte prevented determination.

[I] Interference by co-eluting compound prevented accurate quantitation.

TABLE 26

VOLATILE ORGANIC ANALYTE RECOVERY FROM OIL USING VACUUM DISTILLATION (METHOD 5032)[a]

Compound	Recovery Mean (%)	RSD (%)
Chloromethane	N/A[b]	
Bromomethane	N/A[b]	
Vinyl chloride	N/A[b]	
Chloroethane	N/A[b]	
Methylene chloride	62	32
Acetone	108	55
Carbon disulfide	98	46
1,1-Dichloroethene	97	24
1,1-Dichloroethane	96	22
trans-1,2-Trichloroethene	86	23
cis-1,2-Dichloroethene	99	11
Chloroform	93	14
1,2-Dichloroethane	138	31
2-Butanone	INT[c]	
1,1,1-Trichloroethane	89	14
Carbon tetrachloride	129	23
Vinyl acetate	INT[c]	
Bromodichloromethane	106	14
1,1,2,2-Tetrachloroethane	205	46
1,2-Dichloropropane	107	24
trans-1,3-Dichloropropene	98	13
Trichloroethene	102	8
Dibromochloromethane	168	21
1,1,2-Trichloroethane	95	7
Benzene	146	10
cis-1,3-Dichloropropene	98	11
Bromoform	94	18
2-Hexanone	INT[c]	
4-Methyl-2-pentanone	INT[c]	
Tetrachloroethene	117	22
Toluene	108	8
Chlorobenzene	101	12
Ethylbenzene	96	10
Styrene	120	46
p-Xylene	87	23
o-Xylene	90	10

TABLE 26 (cont.)

Compound	Recovery Mean (%)	RSD (%)
Surrogates		
1,2-Dichloroethane	137	30
Toluene-d_8	84	6
Bromofluorobenzene	48	2

[a] Results are for 10 min. distillation times and condenser temperature held at -10°C. Five replicates of 10-g fish aliquots spiked at 25 ppb were analyzed. Quantitation was performed with a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness. Standards and samples were replicated and precision value reflects the propagated errors. Vacuum distillation efficiencies (Method 5032) are modified by internal standard corrections. Method 8260 internal standards may bias for some analytes. See Method 5032 to identify alternate internal standards with similar efficiencies to minimize bias.

[b] Not analyzed.

[c] Interference by co-evaluating compounds prevented accurate measurement of analyte.

TABLE 27

METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC ANALYTES IN OIL (METHOD 5032)[a]

	Method Detection Limit (ppb)	
Compound	External Standard Method	Internal Standard Method
Chloromethane	N/A[b]	N/A[b]
Bromomethane	N/A[b]	N/A[b]
Vinyl chloride	N/A[b]	N/A[b]
Chloroethane	N/A[b]	N/A[b]
Methylene chloride	80	50
Acetone	120	60
Carbon disulfide	190	180
1,1-Dichloroethene	190	180
1,1-Dichloroethane	130	140
trans-1,2-Dichloroethene	90	100
cis-1,2-Dichloroethene	80	70
Chloroform	60	70
1,2-Dichloroethane	60	60
2-Butanone	INT[c]	INT[c]
1,1,1-Trichloroethane	80	100
Carbon tetrachloride	150	130
Vinyl acetate	INT[c]	INT[c]
Bromodichloromethane	50	60
1,1,2,2-Tetrachloroethane	90	20
1,2-Dichloropropane	120	150
trans-1,3-Dichloropropene	80	50
Trichloroethene	60	40
Dibromochloromethane	40	70
1,1,2-Trichloroethane	70	50
Benzene	30	50
cis-1,3-Dichloropropene	60	40
Bromoform	100	50
2-Hexanone	INT[c]	INT[c]
4-Methyl-2-pentanone	INT[c]	INT[c]
Tetrachloroethene	120	100
Toluene	90	50
Chlorobenzene	70	60
Ethylbenzene	90	40
Styrene	160	180
p-Xylene	180	200
o-Xylene	80	70

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[a] Method detection limits are estimated as the result of five replicated analyses of 1 g cod liver oil spiked at 25 ppb. MDLs were calculated as three times the standard deviation. Quantitation was performed using a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness.

[b] No analyses.

[c] Interference by co-eluting compounds prevented accurate quantitation.

TABLE 28

INTERNAL STANDARDS FOR ANALYTES AND SURROGATES PREPARED USING EQUILIBRIUM HEADSPACE ANALYSIS (METHOD 5021)

Chloroform-d_1	1,1,2-TCA-d_3	Bromobenzene-d_5
Dichlorodifluoromethane	1,1,1-Trichloroethane	Chlorobenzene
Chloromethane	1,1-Dichloropropene	Bromoform
Vinyl chloride	Carbon tetrachloride	Styrene
Bromomethane	Benzene	iso-Propylbenzene
Chloroethane	Dibromomethane	Bromobenzene
Trichlorofluoromethane	1,2-Dichloropropane	n-Propylbenzene
1,1-Dichloroethene	Trichloroethene	2-Chlorotoluene
Methylene chloride	Bromodichloromethane	4-Chlorotoluene
trans-1,2-Dichloroethene	cis-1,3-Dichloropropene	1,3,5-Trimethylbenzene
1,1-Dichloroethane	trans-1,3-Dichloropropene	tert-Butylbenzene
cis-1,2-Dichloroethene	1,1,2-Trichloroethane	1,2,4-Trimethylbenzene
Bromochloromethane	Toluene	sec-Butylbenzene
Chloroform	1,3-Dichloropropane	1,3-Dichlorobenzene
2,2-Dichloropropane	Dibromochloromethane	1,4-Dichlorobenzene
1,2-Dichloroethane	1,2-Dibromoethane	p-iso-Propyltoluene
	Tetrachloroethene	1,2-Dichlorobenzene
	1,1,2-Trichloroethane	n-Butylbenzene
	Ethylbenzene	1,2-Dibromo-3-chloropropane
	m-Xylene	1,2,4-Trichlorobenzene
	p-Xylene	Naphthalene
	o-Xylene	Hexachlorobutadiene
	1,1,2,2-Tetrachloroethane	1,2,3-Trichlorobenzene
	1,2,3-Trichloropropane	

TABLE 29

PRECISION AND MDL DETERMINED FOR ANALYSIS OF FORTIFIED SAND[a] (METHOD 5021)

Compound	% RSD	MDL (µg/kg)
Benzene	3.0	0.34
Bromochloromethane	3.4	0.27
Bromodichloromethane	2.4	0.21
Bromoform	3.9	0.30
Bromomethane	11.6	1.3
Carbon tetrachloride	3.6	0.32
Chlorobenzene	3.2	0.24
Chloroethane	5.6	0.51
Chloroform	3.1	0.30
Chloromethane	4.1	3.5[b]
1,2-Dibromo-3-chloropropane	5.7	0.40
1,2-Dibromoethane	3.2	0.29
Dibromomethane	2.8	0.20
1,2-Dichlorobenzene	3.3	0.27
1,3-Dichlorobenzene	3.4	0.24
1,4-Dichlorobenzene	3.7	0.30
Dichlorodifluoromethane	3.0	0.28
1,1-Dichloroethane	4.5	0.41
1,2-Dichloroethane	3.0	0.24
1,1-Dichloroethene	3.3	0.28
cis-1,2-Dichloroethene	3.2	0.27
trans-1,2-Dichloroethene	2.6	0.22
1,2-Dichloropropane	2.6	0.21
1,1-Dichloropropene	3.2	0.30
cis-1,3-Dichloropropene	3.4	0.27
Ethylbenzene	4.8	0.47
Hexachlorobutadiene	4.1	0.38
Methylene chloride	8.2	0.62[c]
Naphthalene	16.8	3.4[c]
Styrene	7.9	0.62
1,1,1,2-Tetrachloroethane	3.6	0.27
1,1,2,2-Tetrachloroethane	2.6	0.20
Tetrachloroethene	9.8	1.2[c]
Toluene	3.5	0.38
1,2,4-Trichlorobenzene	4.2	0.44
1,1,1-Trichloroethane	2.7	0.27
1,1,2-Trichloroethane	2.6	0.20
Trichloroethene	2.3	0.19

TABLE 29 (cont.)

Compound	% RSD	MDL (µg/kg)
Trichlorofluoromethane	2.7	0.31
1,2,3-Trichloropropane	1.5	0.11
Vinyl chloride	4.8	0.45
m-Xylene/p-Xylene	3.6	0.37
o-Xylene	3.6	0.33

[a] Most compounds spiked at 2 ng/g (2 µg/kg)
[b] Incorrect ionization due to methanol
[c] Compound detected in unfortified sand at >1 ng

TABLE 30

RECOVERIES IN GARDEN SOIL FORTIFIED AT 20 µg/kg (ANALYSIS BY METHOD 5021)

Compound	Recovery per Replicate (ng) Sample 1	 Sample 2	 Sample 3	Mean (ng)	RSD	Recovery (%)
Benzene	37.6	35.2	38.4	37.1	3.7	185[a]
Bromochloromethane	20.5	19.4	20.0	20.0	2.3	100
Bromodichloromethane	21.1	20.3	22.8	21.4	4.9	107
Bromoform	23.8	23.9	25.1	24.3	2.4	121
Bromomethane	21.4	19.5	19.7	20.2	4.2	101
Carbon tetrachloride	27.5	26.6	28.6	27.6	3.0	138
Chlorobenzene	25.6	25.4	26.4	25.8	1.7	129
Chloroethane	25.0	24.4	25.3	24.9	1.5	125
Chloroform	21.9	20.9	21.7	21.5	2.0	108
Chloromethane	21.0	19.9	21.3	20.7	2.9	104[a]
1,2-Dibromo-3-chloro-propane	20.8	20.8	21.0	20.9	0.5	104
1,2-Dibromoethane	20.1	19.5	20.6	20.1	2.2	100
Dibromomethane	22.2	21.0	22.8	22.0	3.4	110
1,2-Dichlorobenzene	18.0	17.7	17.1	17.6	2.1	88.0
1,3-Dichlorobenzene	21.2	21.0	20.1	20.8	2.3	104
1,4-Dichlorobenzene	20.1	20.9	19.9	20.3	2.1	102
Dichlorodifluoromethane	25.3	24.1	25.4	24.9	2.4	125
1,1-Dichloroethane	23.0	22.0	22.7	22.6	1.9	113
1,2-Dichloroethane	20.6	19.5	19.8	20.0	2.3	100
1,1-Dichloroethene	24.8	23.8	24.4	24.3	1.7	122
cis-1,2-Dichloroethene	21.6	20.0	21.6	21.1	3.6	105
trans-1,2-Dichloroethene	22.4	21.4	22.2	22.0	2.0	110
1,2-Dichloropropane	22.8	22.2	23.4	22.8	2.1	114
1,1-Dichloropropene	26.3	25.7	28.0	26.7	3.7	133
cis-1,3-Dichloropropene	20.3	19.5	21.1	20.3	3.2	102
Ethylbenzene	24.7	24.5	25.5	24.9	1.7	125
Hexachlorobutadiene	23.0	25.3	25.2	24.5	4.3	123
Methylene chloride	26.0	25.7	26.1	25.9	0.7	130[a]
Naphthalene	13.8	12.7	11.8	12.8	6.4	63.8[a]
Styrene	24.2	23.3	23.3	23.6	1.8	118
1,1,1,2-Tetrachloroethane	21.4	20.2	21.3	21.0	2.6	105
1,1,2,2-Tetrachloroethane	18.6	17.8	19.0	18.5	2.7	92.3
Tetrachloroethene	25.2	24.8	26.4	25.5	2.7	127
Toluene	28.6	27.9	30.9	29.1	4.4	146[a]
1,2,4-Trichlorobenzene	15.0	14.4	12.9	14.1	6.3	70.5
1,1,1-Trichloroethane	28.1	27.2	29.9	28.4	4.0	142
1,1,2-Trichloroethane	20.8	19.6	21.7	20.7	4.2	104

TABLE 30 (cont.)

Compound	Recovery per Replicate (ng) Sample 1	 Sample 2	 Sample 3	Mean (ng)	RSD	Recovery (%)
Trichloroethene	26.3	24.9	26.8	26.0	3.1	130
Trichlorofluoromethane	25.9	24.8	26.5	25.7	2.7	129
1,2,3-Trichloropropane	18.8	18.3	19.3	18.8	2.2	94.0
Vinyl chloride	24.8	23.2	23.9	24.0	2.7	120
m-Xylene/p-Xylene	24.3	23.9	25.3	24.5	2.4	123
o-Xylene	23.1	22.3	23.4	22.9	2.0	115

[a] Compound found in unfortified garden soil matrix at >5 ng.

TABLE 31

METHOD DETECTION LIMITS AND BOILING POINTS FOR VOLATILE ORGANICS (ANALYSIS BY METHOD 5041)[a]

Compound	Detection Limit (ng)	Boiling Point (°C)
Chloromethane	58	-24
Bromomethane	26	4
Vinyl chloride	14	-13
Chloroethane	21	13
Methylene chloride	9	40
Acetone	35	56
Carbon disulfide	11	46
1,1-Dichloroethene	14	32
1,1-Dichloroethane	12	57
trans-1,2-Dichloroethene	11	48
Chloroform	11	62
1,2-Dichloroethane	13	83
1,1,1-Trichloroethane	8	74
Carbon tetrachloride	8	77
Bromodichloromethane	11	88
1,1,2,2-Tetrachloroethane**	23	146
1,2-Dichloropropane	12	95
trans-1,3-Dichloropropene	17	112
Trichloroethene	11	87
Dibromochloromethane	21	122
1,1,2-Trichloroethane	26	114
Benzene	26	80
cis-1,3-Dichloropropene	27	112
Bromoform**	26	150
Tetrachloroethene	11	121
Toluene	15	111
Chlorobenzene	15	132
Ethylbenzene**	21	136
Styrene**	46	145
Trichlorofluoromethane	17	24
Iodomethane	9	43
Acrylonitrile	13	78
Dibromomethane	14	97
1,2,3-Trichloropropane**	37	157
total Xylenes**	22	138-144

Footnotes are found on the following page.

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* The method detection limit (MDL) is defined in Chapter One. The detection limits cited above were determined according to 40 CFR, Part 136, Appendix B, using standards spiked onto clean VOST tubes. Since clean VOST tubes were used, the values cited above represent the best that the methodology can achieve. The presence of an emissions matrix will affect the ability of the methodology to perform at its optimum level.

** Boiling Point greater than 130°C. Not appropriate for quantitative sampling by Method 0030.

TABLE 32

VOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION (METHOD 5041)

Bromochloromethane

Acetone
Acrylonitrile
Bromomethane
Carbon disulfide
Chloroethane
Chloroform
Chloromethane
1,1-Dichloroethane
1,2-Dichloroethane
1,2-Dichloroethane-d_4 (surrogate)
1,1-Dichloroethene
Trichloroethene
trans-1,2-Dichloroethene
Iodomethane
Methylene chloride
Trichlorofluoromethane
Vinyl chloride

Chlorobenzene-d_5

4-Bromofluorobenzene (surrogate)
Chlorobenzene
Ethylbenzene
Styrene
1,1,2,2-Tetrachloroethane
Tetrachloroethene
Toluene
Toluene-d_8 (surrogate)
1,2,3-Trichloropropane
Xylenes

1,4-Difluorobenzene

Benzene
Bromodichloromethane
Bromoform
Carbon tetrachloride
Chlorodibromomethane
Dibromomethane
1,2-Dichloropropane
cis-1,3-Dichloropropene
trans-1,3-Dichloropropene
1,1,1-Trichloroethane
1,1,2-Trichloroethane

TABLE 33

METHOD 0040 - COMPOUNDS DEMONSTRATED TO BE APPLICABLE TO THE METHOD

Compound	Boiling Point (°C)	Condensation Point at 20°C (%)	Estimated Detection Limit[a] (ppm)
Dichlorodifluoromethane	-30	Gas	0.20
Vinyl chloride	-19	Gas	0.11
1,3-Butadiene	-4	Gas	0.90
1,2-Dichloro-1,1,2,2-tetrafluoroethane	4	Gas	0.14
Methyl bromide	4	Gas	0.14
Trichlorofluoromethane	24	88	0.18
1,1-Dichloroethene	31	22	0.07
Methylene chloride	40	44	0.05
1,1,2-Trichloro-trifluoroethane	48	37	0.13
Chloroform	61	21	0.04
1,1,1-Trichloroethane	75	13	0.03
Carbon tetrachloride	77	11	0.03
Benzene	80	10	0.16
Trichloroethene	87	8	0.04
1,2-Dichloropropane	96	5	0.05
Toluene	111	3	0.08
Tetrachloroethene	121	2	0.03

[a] Since this value represents a direct injection (no concentration) from the Tedlar® bag, these values are directly applicable as stack detection limits.

FIGURE 1
GAS CHROMATOGRAM OF VOLATILE ORGANICS

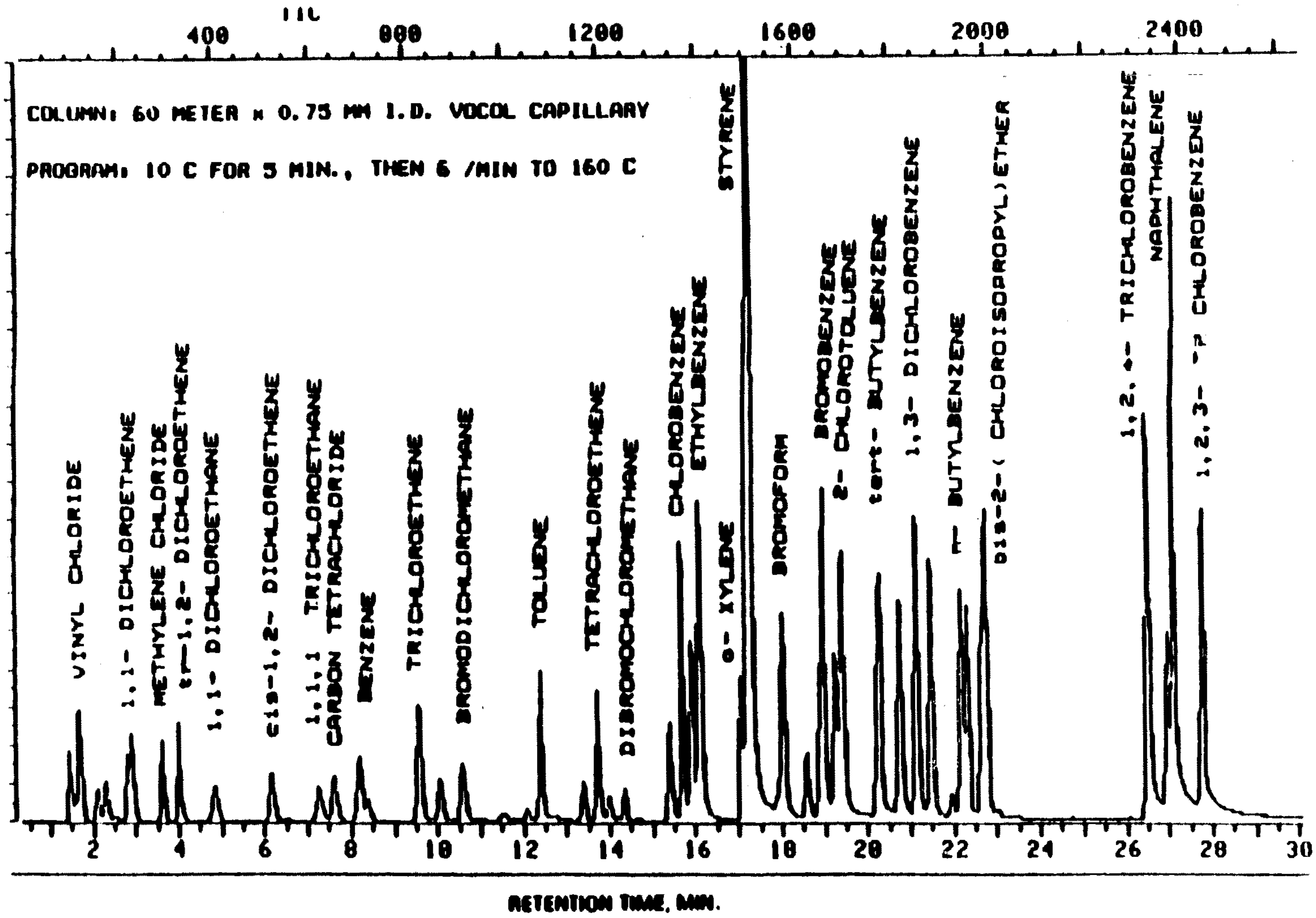


FIGURE 2
GAS CHROMATOGRAM OF VOLATILE ORGANICS

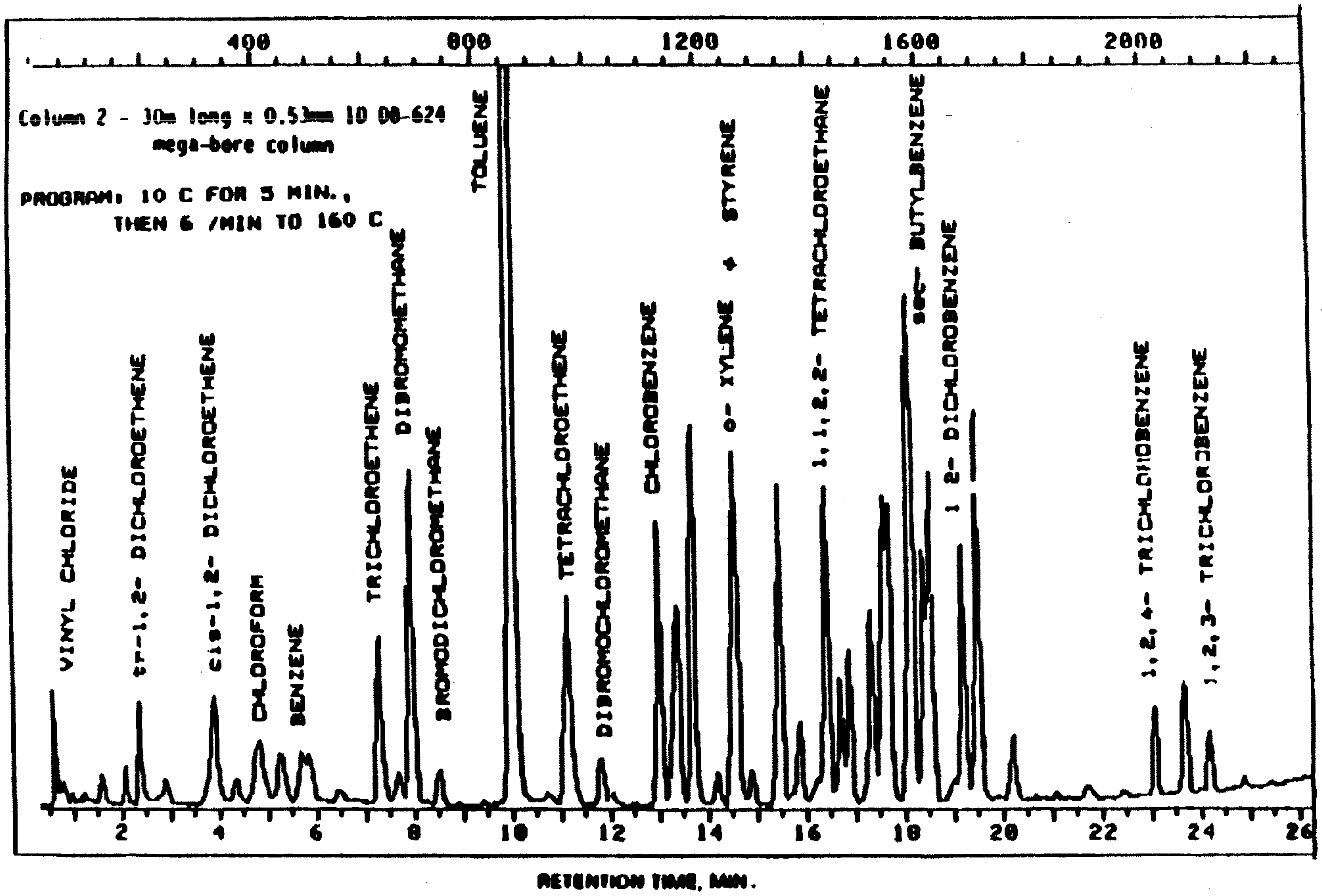


FIGURE 3
GAS CHROMATOGRAM OF VOLATILE ORGANICS

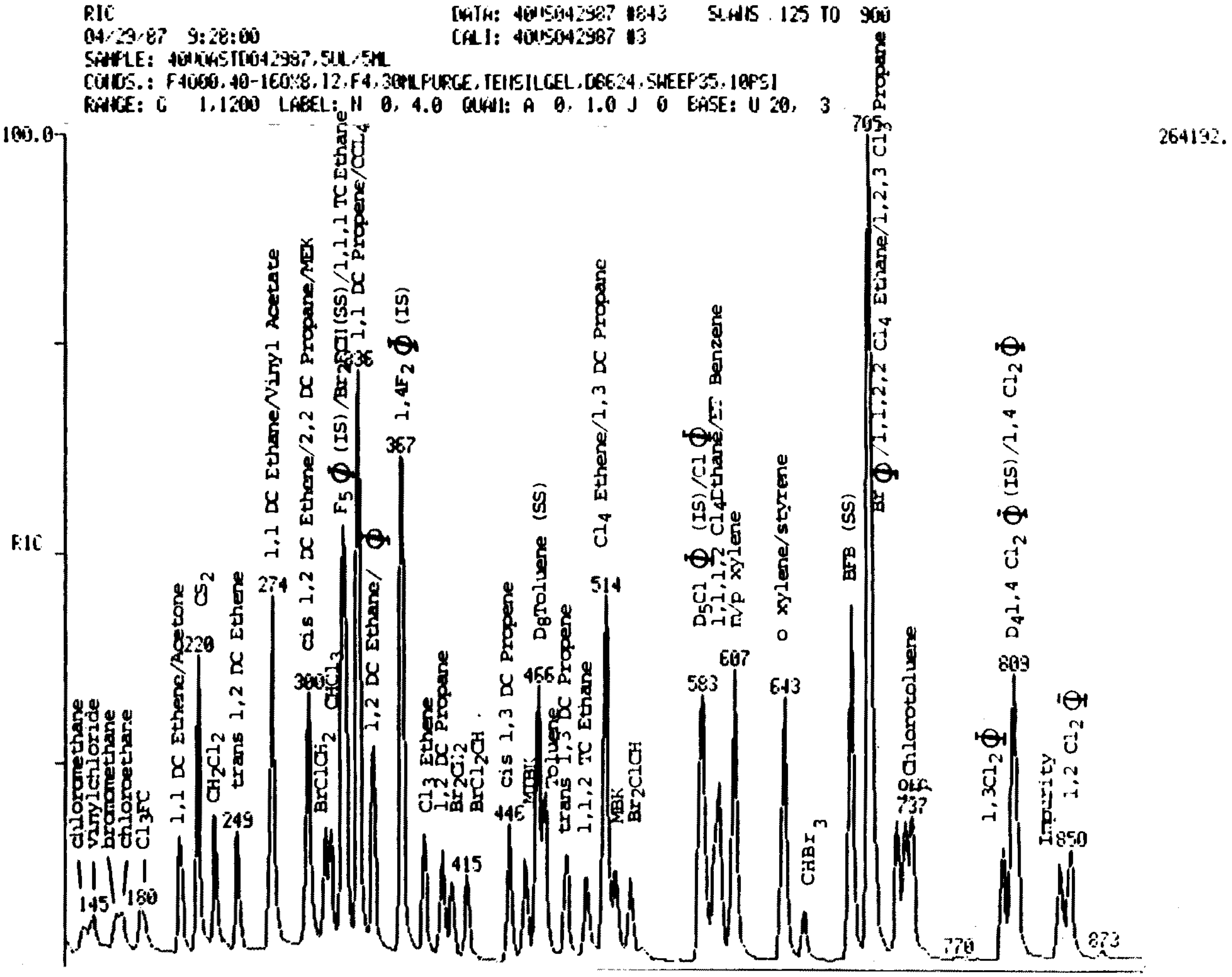
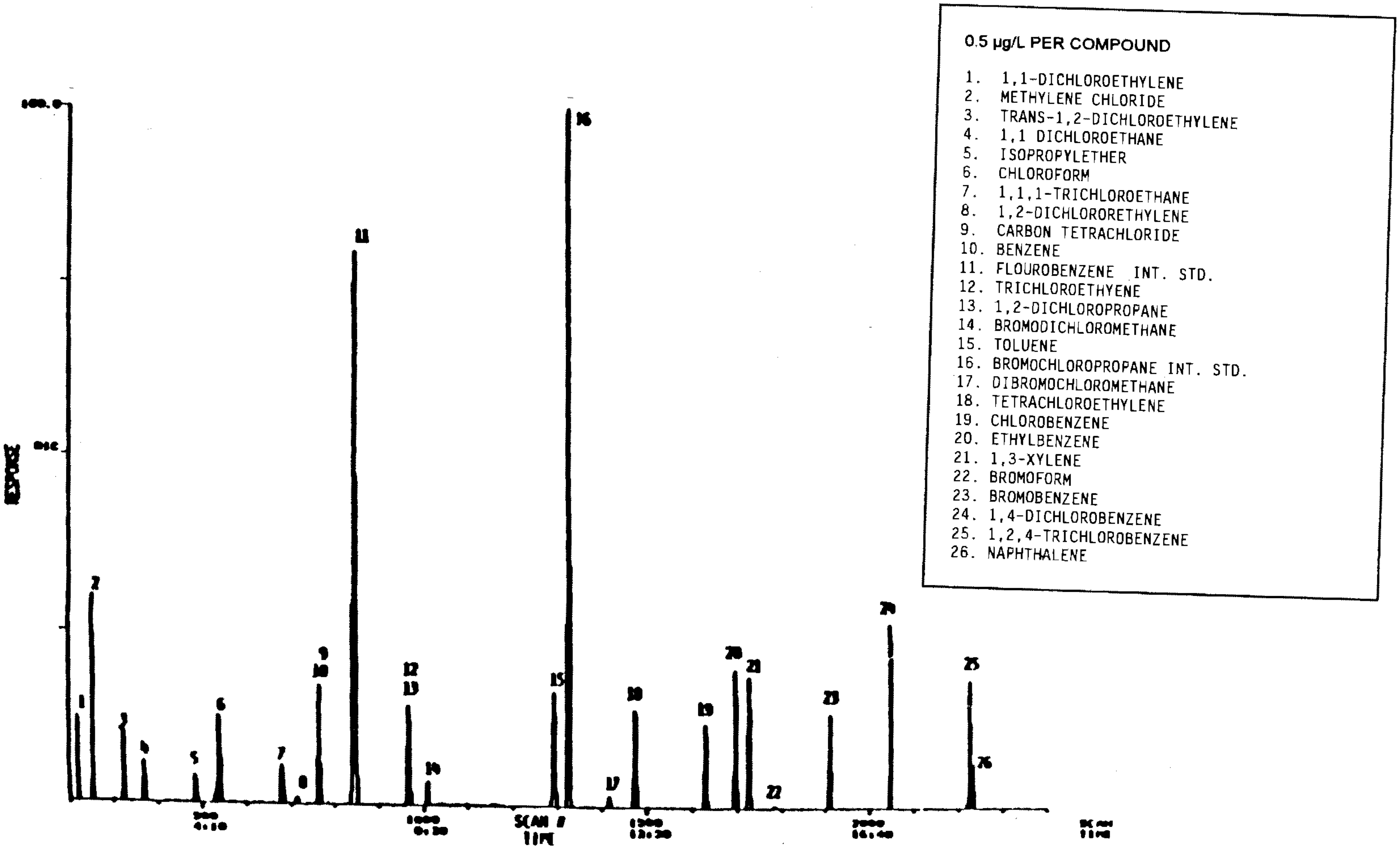
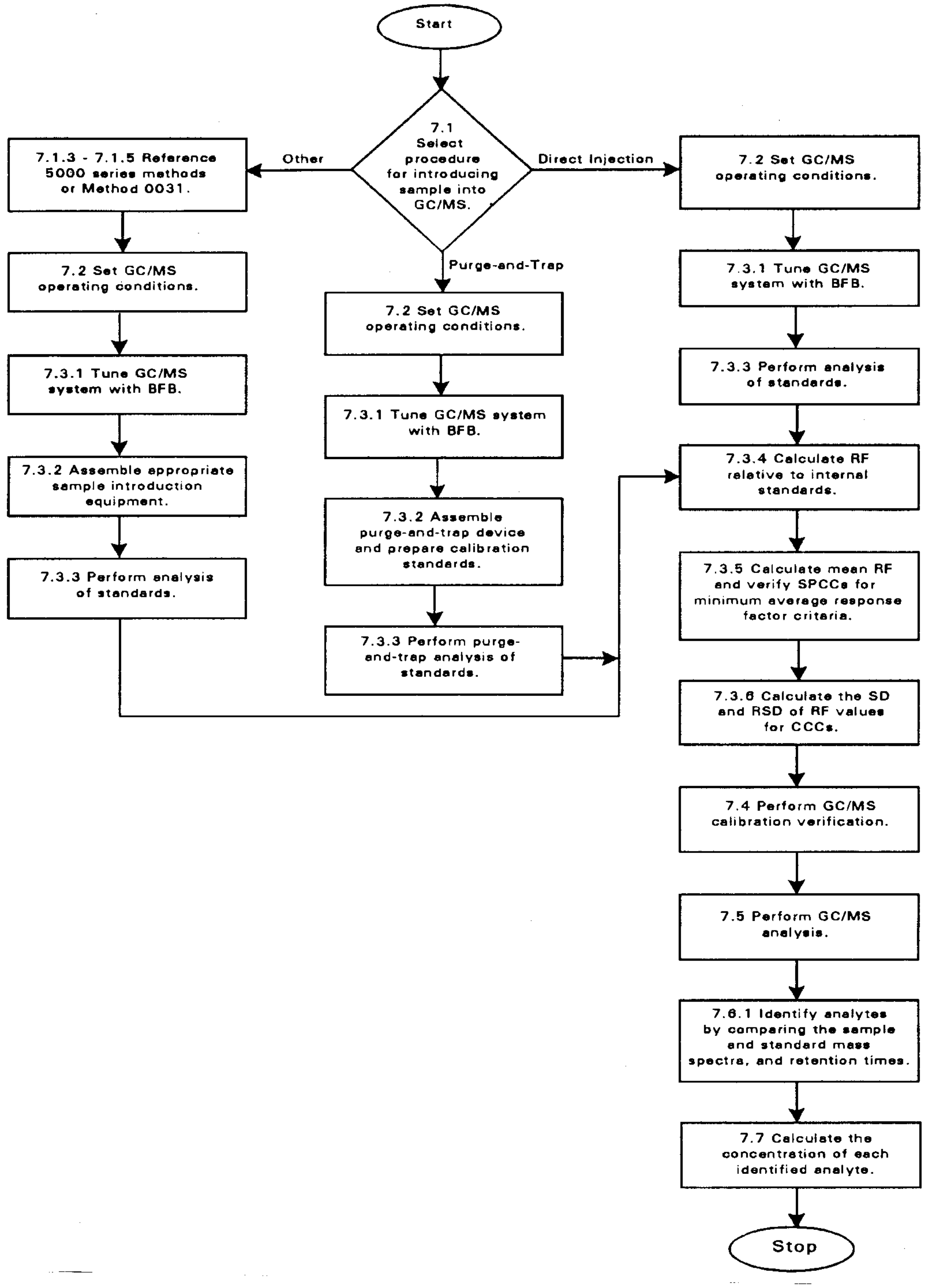


FIGURE 4
GAS CHROMATOGRAM OF TEST MIXTURE



METHOD 8260B
VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)



METHOD 5035

CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES

1.0 SCOPE AND APPLICATION

1.1 This method describes a closed-system purge-and-trap process for the analysis of volatile organic compounds (VOCs) in solid materials (e.g., soils, sediments, and solid waste). While the method is designed for use on samples containing low levels of VOCs, procedures are also provided for collecting and preparing solid samples containing high concentrations of VOCs and for oily wastes. For these high concentration and oily materials, sample collection and preparation are performed using the procedures described here, and sample introduction is performed using the aqueous purge-and-trap procedure in Method 5030. These procedures may be used in conjunction with any appropriate determinative gas chromatographic procedure, including, but not limited to, Methods 8015, 8021, and 8260.

1.2 The low soil method utilizes a hermetically-sealed sample vial, the seal of which is never broken from the time of sampling to the time of analysis. Since the sample is never exposed to the atmosphere after sampling, the losses of VOCs during sample transport, handling, and analysis are negligible. The applicable concentration range of the low soil method is dependent on the determinative method, matrix, and compound. However, it will generally fall in the 0.5 to 200 µg/kg range.

1.3 Procedures are included for preparing high concentration samples for purging by Method 5030. High concentration samples are those containing VOC levels of >200 µg/kg.

1.4 Procedures are also included for addressing oily wastes that are soluble in a water-miscible solvent. These samples are also purged using Method 5030..

1.5 Method 5035 can be used for most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile, water-soluble compounds can be included in this analytical technique. However, quantitation limits (by GC or GC/MS) are approximately ten times higher because of poor purging efficiency.

1.6 Method 5035, in conjunction with Method 8015 (GC/FID), may be used for the analysis of the aliphatic hydrocarbon fraction in the light ends of total petroleum hydrocarbons, e.g., gasoline. For the aromatic fraction (BTEX), use Method 5035 and Method 8021 (GC/PID). A total determinative analysis of gasoline fractions may be obtained using Method 8021 in series with Method 8015.

1.7 As with any preparative method for volatiles, samples should be screened to avoid contamination of the purge-and-trap system by samples that contain very high concentrations of purgeable material above the calibration range of the low concentration method. In addition, because the sealed sample container cannot be opened to remove a sample aliquot without compromising the integrity of the sample, multiple sample aliquots should be collected to allow for screening and reanalysis.

1.8 The closed-system purge-and-trap equipment employed for low concentration samples is not appropriate for soil samples preserved in the field with methanol. Such samples should be analyzed using Method 5030 (see the note in Sec. 6.2.2).

1.9 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 Low concentration soil method - generally applicable to and soils and other solid samples with VOC concentrations in the range of 0.5 to 200 µg/kg.

Volatile organic compounds (VOCs) are determined by collecting an approximately 5-g sample, weighed in the field at the time of collection, and placing it in a pre-weighed vial with a septum-sealed screw-cap (see Sec. 4) that already contains a stirring bar and a sodium bisulfate preservative solution. The vial is sealed and shipped to a laboratory or appropriate analysis site. The entire vial is then placed, unopened, into the instrument carousel. Immediately before analysis, organic-free reagent water, surrogates, and internal standards (if applicable) are automatically added without opening the sample vial. The vial containing the sample is heated to 40°C and the volatiles purged into an appropriate trap using an inert gas combined with agitation of the sample. Purged components travel via a transfer line to a trap. When purging is complete, the trap is heated and backflushed with helium to desorb the trapped sample components into a gas chromatograph for analysis by an appropriate determinative method.

2.2 High concentration soil method - generally applicable to soils and other solid samples with VOC concentrations greater than 200 µg/kg.

The sample introduction technique in Sec. 2.1 is not applicable to all samples, particularly those containing high concentrations (generally greater than 200 µg/kg) of VOCs which may overload either the volatile trapping material or exceed the working range of the determinative instrument system (e.g., GC/MS, GC/FID, GC/EC, etc.). In such instances, this method describes two sample collection options and the corresponding sample purging procedures.

2.2.1 The first option is to collect a bulk sample in a vial or other suitable container without the use of the preservative solution described in Sec. 2.1. A portion of that sample is removed from the container in the laboratory and is dispersed in a water-miscible solvent to dissolve the volatile organic constituents. An aliquot of the solution is added to 5 mL of reagent water in a purge tube. Surrogates and internal standards (if applicable) are added to the solution, then purged using Method 5030, and analyzed by an appropriate determinative method. Because the procedure involves opening the vial and removing a portion of the soil, some volatile constituents may be lost during handling.

2.2.2 The second option is to collect an approximately 5-g sample in a pre-weighed vial with a septum-sealed screw-cap (see Sec 4) that contains 5 mL of a water-miscible organic solvent (e.g., methanol). At the time of analysis, surrogates are added to the vial, then an aliquot of the solvent is removed from the vial, purged using Method 5030 and analyzed by an appropriate determinative method.

2.3 High concentration oily waste method - generally applicable to oily samples with VOC concentrations greater than 200 µg/kg that can be diluted in a water-miscible solvent.

Samples that are comprised of oils or samples that contain significant amounts of oil present additional analytical challenges. This procedure is generally appropriate for such samples when they are soluble in a water-miscible solvent.

2.3.1 After demonstrating that a test aliquot of the sample is soluble in methanol or polyethylene glycol (PEG), a separate aliquot of the sample is spiked with surrogates and diluted in the appropriate solvent. An aliquot of the solution is added to 5 mL of reagent water in a purge tube, taking care to ensure that a floating layer of oil is not present in the purge tube. Internal standards (if applicable) are added to the solution which is then purged using Method 5030 and analyzed by an appropriate determinative method.

2.3.2 Samples that contain oily materials that are not soluble in water-miscible solvents must be prepared according to Method 3585.

3.0 INTERFERENCES

3.1 Impurities in the purge gas and from organic compounds out-gassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running method blanks. The use of non-polytetrafluoroethylene (non-PTFE) plastic coating, non-PTFE thread sealants, or flow controllers with rubber components in the purging device must be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. These compounds will result in interferences or false positives in the determinative step.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank prepared from organic-free reagent water and carried through sampling and handling protocols serves as a check on such contamination.

3.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. Where practical, samples with unusually high concentrations of analytes should be followed by an analysis of organic-free reagent water to check for cross-contamination. If the target compounds present in an unusually concentrated sample are also found to be present in the subsequent samples, the analyst must demonstrate that the compounds are not due to carryover. Conversely, if those target compounds are not present in the subsequent sample, then the analysis of organic-free reagent water is not necessary.

3.4 The laboratory where volatile analysis is performed should be completely free of solvents. Special precautions must be taken to determine methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride, otherwise random background levels will result. Since methylene chloride will permeate through PTFE tubing, all GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory workers' clothing previously exposed to methylene chloride fumes during common liquid/liquid extraction procedures can contribute to sample contamination. The presence of other organic solvents in the laboratory where volatile organics are analyzed will also lead to random background levels and the same precautions must be taken.

4.0 APPARATUS AND MATERIALS

4.1 Sample Containers

The specific sample containers required will depend on the purge-and-trap system to be employed (see Sec. 4.2). Several systems are commercially available. Some systems employ 40-mL clear vials with a special frit and equipped with two PTFE-faced silicone septa. Other systems permit the use of any good quality glass vial that is large enough to contain at least 5 g of soil or solid material and at least 10 mL of water and that can be sealed with a screw-cap containing a PTFE-faced silicone septum. Consult the purge-and-trap system manufacturer's instructions regarding the suitable specific vials, septa, caps, and mechanical agitation devices.

4.2 Purge-and-Trap System

The purge-and-trap system consists of a unit that automatically adds water, surrogates, and internal standards (if applicable) to a vial containing the sample, purges the VOCs using an inert gas stream while agitating the contents of the vial, and also traps the released VOCs for subsequent desorption into the gas chromatograph. Such systems are commercially available from several sources and shall meet the following specifications.

4.2.1 The purging device should be capable of accepting a vial sufficiently large to contain a 5-g soil sample plus a magnetic stirring bar and 10 mL of water. The device must be capable of heating a soil vial to 40°C and holding it at that temperature while the inert purge gas is allowed to pass through the sample. The device should also be capable of introducing at least 5 mL of organic-free reagent water into the sample vial while trapping the displaced headspace vapors. It must also be capable of agitating the sealed sample during purging, (e.g., using a magnetic stirring bar added to the vial prior to sample collection, sonication, or other means). The analytes being purged must be quantitatively transferred to an absorber trap. The trap must be capable of transferring the absorbed VOCs to the gas chromatograph (see 4.2.2).

NOTE: The equipment used to develop this method was a Dynatech PTA-30 W/S Autosampler. This device was subsequently sold to Varian, and is now available as the Archon Purge and Trap Autosampler. See the Disclaimer at the front of this manual for guidance on the use of alternative equipment.

4.2.2 A variety of traps and trapping materials may be employed with this method. The choice of trapping material may depend on the analytes of interest. Whichever trap is employed, it must demonstrate sufficient adsorption and desorption characteristics to meet the quantitation limits of all the target analytes for a given project and the QC requirements in Method 8000 and the determinative method. The most difficult analytes are generally the gases, especially dichlorodifluoromethane. The trap must be capable of desorbing the late eluting target analytes.

NOTE: Check the responses of the brominated compounds when using alternative charcoal traps (especially Vocarb 4000), as some degradation has been noted when higher desorption temperatures (especially above 240 - 250°C) are employed. 2-Chloroethyl vinyl ether is degraded on Vocarb 4000 but performs adequately when Vocarb 3000 is used. The primary criterion, as stated above, is that all target analytes meet the sensitivity requirements for a given project.

4.2.2.1 The trap used to develop this method was 25 cm long, with an inside diameter of 0.105 inches, and was packed with Carbopack/Carbosieve (Supelco, Inc.).

4.2.2.2 The standard trap used in other EPA purge-and-trap methods is also acceptable. That trap is 25 cm long and has an inside diameter of at least 0.105 in. Starting from the inlet, the trap contains the equal amounts of the adsorbents listed below. It is recommended that 1.0 cm of methyl silicone-coated packing (35/60 mesh, Davison, grade 15 or equivalent) be inserted at the inlet to extend the life of the trap. If the analysis of dichlorodifluoromethane or other fluorocarbons of similar volatility is not required, then the charcoal can be eliminated and the polymer increased to fill 2/3 of the trap. If only compounds boiling above 35°C are to be analyzed, both the silica gel and charcoal can be eliminated and the polymer increased to fill the entire trap.

4.2.2.2.1 2,6-Diphenylene oxide polymer - 60/80 mesh, chromatographic grade (Tenax GC or equivalent).

4.2.2.2.2 Methyl silicone packing - OV-1 (3%) on Chromosorb-W, 60/80 mesh or equivalent.

4.2.2.2.3 Coconut charcoal - Prepare from Barnebey Cheney, CA-580-26, or equivalent, by crushing through 26 mesh screen.

4.2.2.3 Trapping materials other than those listed above also may be employed, provided that they meet the specifications in Sec. 4.2.3, below.

4.2.3 The desorber for the trap must be capable of rapidly heating the trap to the temperature recommended by the trap material manufacturer, prior to the beginning of the flow of desorption gas. Several commercial desorbers (purge-and-trap units) are available.

4.3 Syringe and Syringe Valves

4.3.1 25-mL glass hypodermic syringes with Luer-Lok (or equivalent) tip (other sizes are acceptable depending on sample volume used).

4.3.2 2-way syringe valves with Luer ends.

4.3.3 25-µL micro syringe with a 2 inch x 0.006 inch ID, 22° bevel needle (Hamilton #702N or equivalent).

4.3.4 Micro syringes - 10-, 100-µL.

4.3.5 Syringes - 0.5-, 1.0-, and 5-mL, gas-tight with shut-off valve.

4.4 Miscellaneous

4.4.1 Glass vials

4.4.1.1 60-mL, septum-sealed, to collect samples for screening, dry weight determination.

4.4.1.2 40-mL, screw-cap, PTFE lined, septum-sealed. Examine each vial prior to use to ensure that the vial has a flat, uniform sealing surface.

4.4.2 Top-loading balance - Capable of accurately weighing to 0.01 g.

4.4.3 Glass scintillation vials - 20-mL, with screw-caps and PTFE liners, or glass culture tubes with screw-caps and PTFE liners, for dilution of oily waste samples.

4.4.4 Volumetric flasks - Class A, 10-mL and 100-mL, with ground-glass stoppers.

4.4.5 2-mL glass vials, for GC autosampler - Used for oily waste samples extracted with methanol or PEG.

4.4.6 Spatula, stainless steel - narrow enough to fit into a sample vial.

4.4.7 Disposable Pasteur pipettes.

4.4.8 Magnetic stirring bars - PTFE- or glass-coated, of the appropriate size to fit the sample vials. Consult manufacturer's recommendation for specific stirring bars. Stirring bars may be reused, provided that they are thoroughly cleaned between uses. Consult the manufacturers of the purging device and the stirring bars for suggested cleaning procedures.

4.5 Field Sampling Equipment

4.5.1 Purge-and-Trap Soil Sampler - Model 3780PT (Associated Design and Manufacturing Company, 814 North Henry Street, Alexandria, VA 22314), or equivalent.

4.5.2 EnCore^{TM} sampler - (En Chem, Inc., 1795 Industrial Drive, Green Bay, WI 54302), or equivalent.

4.5.3 Alternatively, disposable plastic syringes with a barrel smaller than the neck of the soil vial may be used to collect the sample. The syringe end of the barrel is cut off prior to sampling. One syringe is needed for each sample aliquot to be collected.

4.5.4 Portable balance - For field use, capable of weighing to 0.01 g.

4.5.5 Balance weights - Balances employed in the field should be checked against an appropriate reference weight at least once daily, prior to weighing any samples, or as described in the sampling plan. The specific weights used will depend on the total weight of the sample container, sample, stirring bar, reagent water added, cap, and septum.

5.0 REAGENTS

5.1 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

5.2 Methanol, CH_3OH - purge-and-trap quality or equivalent. Store away from other solvents.

5.3 Polyethylene glycol (PEG), $H(OCH_2CH_2)_nOH$ - free of interferences at the detection limit of the target analytes.

5.4 Low concentration sample preservative

5.4.1 Sodium bisulfate, $NaHSO_4$ - ACS reagent grade or equivalent.

5.4.2 The preservative should be added to the vial prior to shipment to the field, and must be present in the vial prior to adding the sample.

5.5 See the determinative method and Method 5000 for guidance on internal standards and surrogates to be employed in this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Refer to the introductory material in this chapter, Organic Analytes, Sec. 4.1, for general sample collection information. The low concentration portion of this method employs sample vials that are filled and weighed in the field and never opened during the analytical process. As a result, sampling personnel should be equipped with a portable balance capable of weighing to 0.01 g.

6.1 Preparation of sample vials

The specific preparation procedures for sample vials depend on the expected concentration range of the sample, with separate preparation procedures for low concentration soil samples and high concentration soil and solid waste samples. Sample vials should be prepared in a fixed laboratory or other controlled environment, sealed, and shipped to the field location. Gloves should be worn during the preparation steps.

6.1.1 Low concentration soil samples

The following steps apply to the preparation of vials used in the collection of low concentration soil samples to be analyzed by the closed-system purge-and-trap equipment described in Method 5035.

6.1.1.1 Add a clean magnetic stirring bar to each clean vial. If the purge-and-trap device (Sec. 4.2) employs a means of stirring the sample other than a magnetic stirrer (e.g., sonication or other mechanical means), then the stir bar is omitted.

6.1.1.2 Add preservative to each vial. The preservative is added to each vial prior to shipping the vial to the field. Add approximately 1 g of sodium bisulfate to each vial. If samples markedly smaller or larger than 5 g are to be collected, adjust the amount of preservative added to correspond to approximately 0.2 g of preservative for each 1 g of sample. Enough sodium bisulfate should be present to ensure a sample pH of ≤ 2.

6.1.1.3 Add 5 mL of organic-free reagent water to each vial. The water and the preservative will form an acid solution that will reduce or eliminate the majority of the biological activity in the sample, thereby preventing biodegradation of the volatile target analytes.

6.1.1.4 Seal the vial with the screw-cap and septum seal. If the double-ended, fritted, vials are used, seal both ends as recommended by the manufacturer.

6.1.1.5 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is negligible).

6.1.1.6 Weigh the prepared vial to the nearest 0.01 g, record the tare weight, and write it on the label.

6.1.1.7 Because volatile organics will partition into the headspace of the vial from the aqueous solution and will be lost when the vial is opened, surrogates, matrix spikes, and internal standards (if applicable) should only be added to the vials after the sample has been added to the vial. These standards should be introduced back in the laboratory, either manually by puncturing the septum with a small-gauge needle or automatically by the sample introduction system, just prior to analysis.

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6.1.2 High concentration soil samples collected without a preservative

When high concentration samples are collected without a preservative, a variety of sample containers may be employed, including 60-mL glass vials with septum seals (see Sec. 4.4).

6.1.3 High concentration soil samples collected and preserved in the field

The following steps apply to the preparation of vials used in the collection of high concentration soil samples to be preserved in the field with methanol and analyzed by the aqueous purge-and-trap equipment described in Method 5030.

6.1.3.1 Add 10 mL of methanol to each vial.

6.1.3.2 Seal the vial with the screw-cap and septum seal.

6.1.3.3 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is negligible).

6.1.3.4 Weigh the prepared vial to the nearest 0.01 g, record the tare weight, and write it on the label.

NOTE: Vials containing methanol should be weighed a second time on the day that they are to be used. Vials found to have lost methanol (reduction in weight of >0.01 g) should not be used for sample collection.

6.1.3.5 Surrogates, internal standards and matrix spikes (if applicable) should be added to the sample after it is returned to the laboratory and prior to analysis.

6.1.4 Oily waste samples

When oily waste samples are known to be soluble in methanol or PEG, sample vials may be prepared as described in Sec. 6.1.3, using the appropriate solvent. However, when the solubility of the waste is unknown, the sample should be collected without the use of a preservative, in a vial such as that described in Sec. 6.1.2.

6.2 Sample collection

Collect the sample according to the procedures outlined in the sampling plan. As with any sampling procedure for volatiles, care must be taken to minimize the disturbance of the sample in order to minimize the loss of the volatile components. Several techniques may be used to transfer a sample to the relatively narrow opening of the low concentration soil vial. These include devices such as the EnCore^{TM} sampler, the Purge-and-Trap Soil Sampler ^{TM}, and a cut plastic syringe. Always wear gloves whenever handling the tared sample vials.

6.2.1 Low concentration soil samples

6.2.1.1 Using an appropriate sample collection device, collect approximately 5 g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere: generally within a few minutes at most. Carefully wipe the exterior of the sample collection device with a clean cloth or towel.

6.2.1.2 Using the sample collection device, add about 5 g (2 - 3 cm) of soil to the sample vial containing the preservative solution. Quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw-cap. Store samples on ice at 4°C.

<u>NOTE</u>: Soil samples that contain carbonate minerals (either from natural sources or applied as an amendment) may effervesce upon contact with the acidic preservative solution in the low concentration sample vial. If the amount of gas generated is very small (i.e., several mL), any loss of volatiles as a result of such effervescence may be minimal if the vial is sealed quickly. However, if larger amounts of gas are generated, not only may the sample lose a significant amount of analyte, but the gas pressure may shatter the vial if the sample vial is sealed. Therefore, when samples are known or suspected to contain high levels of carbonates, a test sample should be collected, added to a vial, and checked for effervescence. If a rapid or vigorous reaction occurs, discard the sample and collect low concentration samples in vials that do not contain the preservative solution.

6.2.1.3 When practical, use a portable balance to weigh the sealed vial containing the sample to ensure that 5.0 ± 0.5 g of sample were added. The balance should be calibrated in the field using an appropriate weight for the sample containers employed (Sec. 4.5.5). Record the weight of the sealed vial containing the sample to the nearest 0.01 g.

6.2.1.4 Alternatively, collect several trial samples with plastic syringes. Weigh each trial sample and note the length of the soil column in the syringe. Use these data to determine the length of soil in the syringe that corresponds to 5.0 ± 0.5 g. Discard each trial sample.

6.2.1.5 As with the collection of aqueous samples for volatiles, collect <u>at least</u> two replicate samples. This will allow the laboratory an additional sample for reanalysis. The second sample should be taken from the same soil stratum or the same section of the solid waste being sampled, and within close proximity to the location from which the original sample was collected.

6.2.1.6 In addition, since the soil vial cannot be opened without compromising the integrity of the sample, at least one additional aliquot of sample must be collected for screening, dry weight determination, and high concentration analysis (if necessary). This third aliquot may be collected in a 60-mL glass vial or a third 40-mL soil sample vial. However, this third vial must *not* contain the sample preservative solution, as an aliquot will be used to determine dry weight. If high concentration samples are collected in vials containing methanol, then two additional aliquots should be collected, one for high concentration analysis collected in a vial containing methanol, and another for the dry weight determination in a vial without either methanol or the low concentration aqueous preservative solution.

6.2.1.7 If samples are known or expected to contain target analytes over a wide range of concentrations, thereby requiring the analyses of multiple sample aliquots, it may be advisable and practical to take an additional sample aliquot in a low concentration soil vial containing the preservative, but collecting only 1-2 g instead of the 5 g collected in Sec. 6.2.1.1. This aliquot may be used for those analytes that exceed the instrument calibration range in the 5-g analysis.

6.2.1.8 The EnCore™ sampler has not been thoroughly evaluated by EPA as a sample storage device. While preliminary results indicate that storage in the EnCore™ device may be appropriate for up to 48 hours, samples collected in this device should be transferred to the soil sample vials as soon as possible, or analyzed within 48 hours.

6.2.1.9 The collection of low concentration soil samples in vials that contain methanol is <u>not</u> appropriate for samples analyzed with the closed-system purge-and-trap equipment described in this method (see Sec. 6.2.2).

6.2.2 High concentration soil samples preserved in the field

The collection of soil samples in vials that contain methanol has been suggested by some as a combined preservation and extraction procedure. However, this procedure is <u>not</u> appropriate for use with the low concentration soil procedure described in this method.

<u>NOTE</u>: The use of methanol preservation has not been formally evaluated by EPA and analysts must be aware of two potential problems. First, the use of methanol as a preservative and extraction solvent introduces a significant dilution factor that will raise the method quantitation limit beyond the operating range of the low concentration direct purge-and-trap procedure (0.5-200 µg/kg). The exact dilution factor will depend on the masses of solvent and sample, but generally exceeds 1000, and may make it difficult to demonstrate compliance with regulatory limits or action levels for some analytes. Because the analytes of interest are volatile, the methanol extract cannot be concentrated to overcome the dilution problem. Thus, for samples of unknown composition, it may still be necessary to collect an aliquot for analysis by this closed-system procedure and another aliquot preserved in methanol and analyzed by other procedures. The second problem is that the addition of methanol to the sample is likely to cause the sample to fail the ignitability characteristic, thereby making the unused sample volume a hazardous waste.

6.2.2.1 When samples are <u>known</u> to contain volatiles at concentrations high enough that the dilution factor will not preclude obtaining results within the calibration range of the appropriate determinative method, a sample may be collected and immediately placed in a sample vial containing purge-and-trap grade methanol.

6.2.2.2 Using an appropriate sample collection device, collect approximately 5 g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere: generally within a few minutes at most. Carefully wipe the exterior of the sample collection device with a clean cloth or towel.

6.2.2.3 Using the sample collection device, add about 5 g (2 - 3 cm) of soil to the vial containing 10 mL of methanol. Quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw-cap. Store samples on ice at 4°C.

6.2.2.4 When practical, use a portable balance to weigh the sealed vial containing the sample to ensure that 5.0 ± 0.5 g of sample were added. The balance should be calibrated in the field using an appropriate weight for the sample containers employed (Sec. 4.5.5). Record the weight of the sealed vial containing the sample to the nearest 0.01 g.

6.2.2.5 Alternatively, collect several trial samples with plastic syringes. Weigh each trial sample and note the length of the soil column in the syringe. Use these data to determine the length of soil in the syringe that corresponds to 5.0 ± 0.5 g. Discard each trial sample.

6.2.2.6 Other sample weights and volumes of methanol may be employed, provided that the analyst can demonstrate that the sensitivity of the overall analytical procedure is appropriate for the intended application.

6.2.2.7 The collection of at least one additional sample aliquot is required for the determination of the dry weight, as described in Sec. 6.2.1.6. Samples collected in methanol should be shipped as described in Sec. 6.3, and must be clearly labeled as containing methanol, so that the samples are not analyzed using the closed-system purge-and-trap equipment described in this procedure.

6.2.3 High concentration soil sample <u>not</u> preserved in the field

The collection of high concentration soil samples that are not preserved in the field generally follows similar procedures as for the other types of samples described in Secs. 6.2.1 and 6.2.2, with the obvious exception that the sample vials contain neither the aqueous preservative solution nor methanol. However, when field preservation is not employed, it is better to collect a larger volume sample, filling the sample container as full as practical in order to minimize the headspace. Such collection procedures generally do not require the collection of a separate aliquot for dry weight determination, but it may be advisable to collect a second sample aliquot for screening purposes, in order to minimize the loss of volatiles in either aliquot.

6.2.4 Oily waste samples

The collection procedures for oily samples depend on knowledge of the waste and its solubility in methanol or other solvents.

6.2.4.1 When an oily waste is <u>known</u> to be soluble in methanol or PEG, the sample may be collected in a vial containing such a solvent (see Sec. 6.1.4), using procedures similar to those described in Sec. 6.2.2.

6.2.4.2 When the solubility of the oily waste is <u>not</u> known, the sample should either be collected in a vial without a preservative, as described in Sec. 6.2.3, or the solubility of a trial sample should be tested in the field, using a vial containing solvent. If the trial sample is soluble in the solvent, then collect the oily waste sample as described in Sec. 6.2.2. Otherwise, collect an unpreserved sample as described in Sec. 6.2.3.

6.3 Sample handling and shipment

All samples for volatiles analysis should be cooled to approximately 4°C, packed in appropriate containers, and shipped to the laboratory on ice, as described in the sampling plan.

6.4 Sample storage

6.4.1 Once in the laboratory, store samples at 4°C until analysis. The sample storage area should be free of organic solvent vapors.

6.4.2 All samples should be analyzed as soon as practical, and within the designated holding time from collection. Samples not analyzed within the designated holding time must be noted and the data are considered minimum values.

6.4.3 When the low concentration samples are strongly alkaline or highly calcareous in nature, the sodium bisulfate preservative solution may not be strong enough to reduce the pH of the soil/water solution to below 2. Therefore, when low concentration soils to be sampled are known or suspected to be strongly alkaline or highly calcareous, additional steps may be required to preserve the samples. Such steps include: addition of larger amounts of the sodium bisulfate preservative to non-calcareous samples, storage of low concentration samples at -10°C (taking care not to fill the vials so full that the expansion of the water in the vial breaks the vial), or significantly reducing the maximum holding time for low concentration soil samples. Whichever steps are employed, they should be clearly described in the sampling and QA project plans and distributed to both the field and laboratory personnel. See Sec. 6.2.1.2 for additional information.

7.0 PROCEDURE

This section describes procedures for sample screening, the low concentration soil method, the high concentration soil method, and the procedure for oily waste samples. High concentration samples are to be introduced into the GC system using Method 5030. Oily waste samples are to be introduced into the GC system using Method 5030 if they are soluble in a water-miscible solvent, or using Method 3585 if they are not.

7.1 Sample screening

7.1.1 It is highly recommended that all samples be screened prior to the purge-and-trap GC or GC/MS analysis. Samples may contain higher than expected quantities of purgeable organics that will contaminate the purge-and-trap system, thereby requiring extensive cleanup and instrument maintenance. The screening data are used to determine which is the appropriate sample preparation procedure for the particular sample, the low concentration closed-system direct purge-and-trap method (Sec. 7.2), the high concentration (methanol extraction) method (Sec. 7.3), or the nonaqueous liquid (oily waste) methanol or PEG dilution procedure (Sec. 7.4).

7.1.2 The analyst may employ any appropriate screening technique. Two suggested screening techniques employing SW-846 methods are:

7.1.2.1 Automated headspace (Method 5021) using a gas chromatograph (GC) equipped with a photoionization detector (PID) and an electrolytic conductivity detector (HECD) in series, or, 7.1.2.2 Extraction of the sample with hexadecane (Method 3820) and analysis of the extract on a GC equipped with a FID and/or an ECD.

7.1.3 The analyst may inject a calibration standard containing the analytes of interest at a concentration equivalent to the upper limit of the calibration range of the low concentration soil method. The results from this standard may be used to determine when the screening results approach the upper limit of the low concentration soil method. There are no linearity or other performance criteria associated with the injection of such a standard, and other approaches may be employed to estimate sample concentrations.

7.1.4 Use the low concentration closed-system purge-and-trap method (Sec. 7.2) if the estimated concentration from the screening procedure falls within the calibration range of the selected determinative method. If the concentration exceeds the calibration range of the low concentration soil method, then use either the high concentration soil method (Sec. 7.3), or the oily waste method (Sec. 7.4).

7.2 Low concentration soil method (Approximate concentration range of 0.5 to 200 µg/kg - the concentration range is dependent upon the determinative method and the sensitivity of each analyte.)

7.2.1 Initial calibration

Prior to using this introduction technique for any GC or GC/MS method, the system must be calibrated. General calibration procedures are discussed in Method 8000, while the determinative methods and Method 5000 provide specific information on calibration and preparation of standards. Normally, external standard calibration is preferred for the GC methods (non-MS detection) because of possible interference problems with internal standards. If interferences are not a problem, or when a GC/MS method is used, internal standard calibration may be employed.

7.2.1.1 Assemble a purge-and-trap device that meets the specification in Sec. 4.2 and that is connected to a gas chromatograph or a gas chromatograph/mass spectrometer system.

7.2.1.2 Before initial use, a Carbopack/Carbosieve trap should be conditioned overnight at 245°C by backflushing with an inert gas flow of at least 20 mL/minute. If other trapping materials are substituted for the Carbopack/Carbosieve, follow the manufacturers recommendations for conditioning. Vent the trap effluent to the hood, not to the analytical column. Prior to daily use, the trap should be conditioned for 10 minutes at 245°C with backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.

7.2.1.3 If the standard trap in Sec. 4.2.2.2 is employed, prior to initial use, the trap should be conditioned overnight at 180°C by backflushing with an inert gas flow of at least 20 mL/min, or according to the manufacturer's recommendations. Vent the trap effluent to the hood, not to the analytical column. Prior to daily use, the trap should be conditioned for 10 min at 180°C with backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.

7.2.1.4 Establish the purge-and-trap instrument operating conditions. Adjust the instrument to inject 5 mL of water, to heat the sample to 40°C, and to hold the sample at 40°C for 1.5 minutes before commencing the purge process, or as recommended by the instrument manufacturer.

7.2.1.5 Prepare a minimum of five initial calibration standards containing all the analytes of interest and surrogates, as described in Method 8000, and following the instrument manufacturer's instructions. The calibration standards are prepared in organic-free reagent water. The volume of organic-free reagent water used for calibration must be the same volume used for sample analysis (normally 5 mL added to the vial before shipping it to the field <u>plus</u> the organic-free reagent water added by the instrument). The calibration standards should also contain approximately the same amount of the sodium bisulfate preservative as the sample (e.g., ~1 g), as the presence of the preservative will affect the purging efficiencies of the analytes. The internal standard solution must be added automatically, by the instrument, in the same fashion as used for the samples. Place the soil vial containing the solution in the instrument carousel. In order to calibrate the surrogates using standards at five concentrations, it may be necessary to disable the automatic addition of surrogates to each vial containing a calibration standard (consult the manufacturer's instructions). Prior to purging, heat the sample vial to 40°C for 1.5 minutes, or as recommended by the manufacturer.

7.2.1.6 Carry out the purge-and-trap procedure as outlined in Secs. 7.2.3. to 7.2.5.

7.2.1.7 Calculate calibration factors (CF) or response factors (RF) for each analyte of interest using the procedures described in Method 8000. Calculate the average CF (external standards) or RF (internal standards) for each compound, as described in Method 8000. Evaluate the linearity of the calibration data, or choose another calibration model, as described in Method 8000 and the specific determinative method.

7.2.1.8 For GC/MS analysis, a system performance check must be made before this calibration curve is used (see Method 8260). If the purge-and-trap procedure is used with Method 8021, evaluate the response for the following four compounds: chloromethane; 1,1-dichloroethane; bromoform; and 1,1,2,2-tetrachloroethane. They are used to check for proper purge flow and to check for degradation caused by contaminated lines or active sites in the system.

7.2.1.8.1 Chloromethane is the most likely compound to be lost if the purge flow is too fast.

7.2.1.8.2 Bromoform is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response.

7.2.1.8.3 Tetrachloroethane and 1,1-dichloroethane are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

7.2.1.9 When analyzing for very late eluting compounds with Method 8021 (i.e., hexachlorobutadiene, 1,2,3-trichlorobenzene, etc.), cross-contamination and memory effects from a high concentration sample or even the standard are a common problem.

Extra rinsing of the purge chamber after analysis normally corrects this. The newer purge-and-trap systems often overcome this problem with better bakeout of the system following the purge-and-trap process. Also, the charcoal traps retain less moisture and decrease the problem.

7.2.2 Calibration verification

Refer to Method 8000 for details on calibration verification. A single standard near the mid-point of calibration range is used for verification. This standard should also contain approximately 1 g of sodium bisulfate.

7.2.3 Sample purge-and-trap

This method is designed for a 5-g sample size, but smaller sample sizes may be used. Consult the instrument manufacturer's instructions regarding larger sample sizes, in order to avoid clogging of the purging apparatus. The soil vial is hermetically sealed at the sampling site, and MUST remain so in order to guarantee the integrity of the sample. Gloves must be worn when handling the sample vial since the vial has been tared. If any soil is noted on the exterior of the vial or cap, it must be carefully removed prior to weighing. Weigh the vial and contents to the nearest 0.01 g, even if the sample weight was determined in the field, and record this weight. This second weighing provides a check on the field sampling procedures and provides additional assurance that the reported sample weight is accurate. Data users should be advised on significant discrepancies between the field and laboratory weights.

7.2.3.1 Remove the sample vial from storage and allow it to warm to room temperature. Shake the vial gently, to ensure that the contents move freely and that stirring will be effective. Place the sample vial in the instrument carousel according to the manufacturer's instructions.

7.2.3.2 Without disturbing the hermetic seal on the sample vial, add 5 mL of organic-free reagent water, the internal standards, and the surrogate compounds. This is carried out using the automated sampler. Other volumes of organic-free reagent water may be used, however, it is imperative that all samples, blanks, and calibration standards have exactly the same final volume of organic-free reagent water. Prior to purging, heat the sample vial to 40°C for 1.5 minutes, or as described by the manufacturer.

7.2.3.3 For the sample selected for matrix spiking, add the matrix spiking solution described in Sec. 5.0 of Method 5000, either manually, or automatically, following the manufacturer's instructions. The concentration of the spiking solution and the amount added should be established as described in Sec. 8.0 of Method 8000.

7.2.3.4 Purge the sample with helium or another inert gas at a flow rate of up to 40 mL/minute (the flow rate may vary from 20 to 40 mL/min, depending on the target analyte group) for 11 minutes while the sample is being agitated with the magnetic stirring bar or other mechanical means. The purged analytes are allowed to flow out of the vial through a glass-lined transfer line to a trap packed with suitable sorbent materials.

7.2.4 Sample Desorption

7.2.4.1 Non-cryogenic interface - After the 11 minute purge, place the purge-and-trap system in the desorb mode and preheat the trap to 245°C without a flow of desorption gas. Start the flow of desorption gas at 10 mL/minute for about four minutes (1.5 min is normally adequate for analytes in Method 8015). Begin the temperature program of the gas chromatograph and start data acquisition.

7.2.4.2 Cryogenic interface - After the 11 minute purge, place the purge-and-trap system in the desorb mode, make sure that the cryogenic interface is at -150°C or lower, and rapidly heat the trap to 245°C while backflushing with an inert gas at 4 mL/minute for about 5 minutes (1.5 min is normally adequate for analytes in Methods 8015). At the end of the 5-minute desorption cycle, rapidly heat the cryogenic trap to 250°C. Begin the temperature program of the gas chromatograph and start the data acquisition.

7.2.5 Trap Reconditioning

After desorbing the sample for 4 minutes, recondition the trap by returning the purge-and-trap system to the purge mode. Maintain the trap temperature at 245°C (or other temperature recommended by the manufacturer of the trap packing materials). After approximately 10 minutes, turn off the trap heater and halt the purge flow through the trap. When the trap is cool, the next sample can be analyzed.

7.2.6 Data Interpretation

Perform qualitative and quantitative analysis following the guidance given in the determinative method and Method 8000. If the concentration of any target analyte exceeds the calibration range of the instrument, it will be necessary to reanalyze the sample by the high concentration method. Such reanalyses need only address those analytes for which the concentration exceeded the calibration range of the low concentration method. Alternatively, if a sample aliquot of 1-2 g was also collected (see Sec. 6.2.1.7), it may be practical to analyze that aliquot for the analytes that exceeded the instrument calibration range in the 5-g analysis. If results are to be reported on a dry weight basis, proceed to Sec. 7.5

7.3 High concentration method for soil samples with concentrations generally greater than 200 µg/kg.

The high concentration method for soil is based on a solvent extraction. A solid sample is either extracted or diluted, depending on sample solubility in a water-miscible solvent. An aliquot of the extract is added to organic-free reagent water containing surrogates and, if applicable, internal and matrix spiking standards, purged according to Method 5030, and analyzed by an appropriate determinative method. Wastes that are insoluble in methanol (i.e., petroleum and coke wastes) are diluted with hexadecane (see Sec. 7.3.8).

The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were <u>not</u> preserved in the field are prepared using the steps below, beginning at Sec. 7.3.1. If solvent preservation was employed in the field, then the preparation begins with Sec. 7.3.4.

7.3.1 When the high concentration sample is <u>not</u> preserved in the field, the sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Whenever practical, mix the contents of the sample container by shaking or other mechanical means without opening the vial. When shaking is not practical, quickly mix the contents of the vial with a narrow metal spatula and immediately reseal the vial.

7.3.2 If the sample is from an unknown source, perform a solubility test before proceeding. Remove several grams of material from the sample container. Quickly reseal the container to minimize the loss of volatiles. Weigh 1-g aliquots of the sample into several test tubes or other suitable containers. Add 10 mL of methanol to the first tube, 10 mL of PEG to the second, and 10 mL of hexadecane to the third. Swirl the sample and determine if it is soluble in the solvent. Once the solubility has been evaluated, discard these test solutions. If the sample is soluble in either methanol or PEG, proceed with Sec. 7.3.3. If the sample is only soluble in hexadecane, proceed with Sec. 7.3.8.

7.3.3 For soil and solid waste samples that are soluble in methanol, add 9.0 mL of methanol and 1.0 mL of the surrogate spiking solution to a tared 20-mL vial. Using a top-loading balance, weigh 5 g (wet weight) of sample into the vial. Quickly cap the vial and reweigh the vial. Record the weight to 0.1 g. Shake the vial for 2 min. If the sample was not soluble in methanol, but was soluble in PEG, employ the same procedure described above, but use 9.0 mL of PEG in place of the methanol. Proceed with Sec. 7.3.5.

NOTE: The steps in Secs. 7.3.1, 7.3.2, and 7.3.3 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.

7.3.4 For soil and solid waste samples that were collected in methanol or PEG (see Sec. 6.2.2), weigh the vial to 0.1 g as a check on the weight recorded in the field, add the surrogate spiking solution to the vial by injecting it through the septum, shake for 2 min, as described above, and proceed with Sec. 7.3.5.

7.3.5 Pipet approximately 1 mL of the extract from either Sec. 7.3.3 or 7.3.4 into a GC vial for storage, using a disposable pipet, and seal the vial. The remainder of the extract may be discarded. Add approximately 1 mL of methanol or PEG to a separate GC vial for use as the method blank for each set of samples extracted with the same solvent.

7.3.6 The extracts must be stored at 4°C in the dark, prior to analysis. Add an appropriate aliquot of the extract (see Table 2) to 5.0 mL of organic-free reagent water and analyze by Method 5030 in conjunction with the appropriate determinative method. Proceed to Sec. 7.0 in Method 5030 and follow the procedure for purging high concentration samples.

7.3.7 If results are to be reported on a dry weight basis, determine the dry weight of a separate aliquot of the sample, using the procedure in Sec. 7.5, after the sample extract has been transferred to a GC vial and the vial sealed.

7.3.8 For solids that are not soluble in methanol or PEG (including those samples consisting primarily of petroleum or coking waste) dilute or extract the sample with hexadecane using the procedures in Sec. 7.0 of Method 3585.

7.4 High concentration method for oily waste samples

This procedure for the analysis of oily waste samples involves the dilution of the sample in methanol or PEG. However, care must be taken to avoid introducing any of the floating oil layer into the instrument. A portion of the diluted sample is then added to 5.0 mL of organic-free reagent water, purged according to Method 5030, and analyzed using an appropriate determinative method.

For oily samples that are not soluble in methanol or PEG (including those samples consisting primarily of petroleum or coking waste), dilute or extract with hexadecane using the procedures in Sec. 7.0 of Method 3585.

The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were not preserved in the field are prepared using the steps below, beginning at Sec. 7.4.1. If methanol preservation was employed in the field, then the preparation begins with Sec. 7.4.3.

7.4.1 If the waste was not preserved in the field and it is soluble in methanol or PEG, weigh 1 g (wet weight) of the sample into a tared 10-mL volumetric flask, a tared scintillation vial, or a tared culture tube. If a vial or tube is used instead of a volumetric flask, it must be calibrated prior to use. This operation must be performed prior to opening the sample vial and weighing out the aliquot for analysis.

7.4.1.1 To calibrate the vessel, pipet 10.0 mL of methanol or PEG into the vial or tube and mark the bottom of the meniscus.

7.4.1.2 Discard this solvent, and proceed with weighing out the 1-g sample aliquot.

7.4.2 Quickly add 1.0 mL of surrogate spiking solution to the flask, vial, or tube, and dilute to 10.0 mL with the appropriate solvent (methanol or PEG). Swirl the vial to mix the contents and then shake vigorously for 2 minutes.

7.4.3 If the sample was collected in the field in a vial containing methanol or PEG, weigh the vial to 0.1 g as a check on the weight recorded in the field, add the surrogate spiking solution to the vial by injecting it through the septum. Swirl the vial to mix the contents and then shake vigorously for 2 minutes and proceed with Sec. 7.4.4.

7.4.4 Regardless of how the sample was collected, the target analytes are extracted into the solvent along with the majority of the oily waste (i.e., some of the oil may still be floating on the surface). If oil is floating on the surface, transfer 1 to 2 mL of the extract to a clean GC vial using a Pasteur pipet. Ensure that no oil is transferred to the vial.

7.4.5 Add 10 - 50 µL of the methanol extract to 5 mL of organic-free reagent water for purge-and-trap analysis, using Method 5030.

7.4.6 Prepare a matrix spike sample by adding 10 - 50 µL of the matrix spike standard dissolved in methanol to a 1-g aliquot of the oily waste. Shake the vial to disperse the matrix spike solution throughout the oil. Then add 10 mL of extraction solvent and proceed with the extraction and analysis, as described in Secs. 7.4.2 - 7.4.5. Calculate the recovery of the spiked analytes as described in Method 8000. If the recovery is not within the acceptance limits for the application, use the hexadecane dilution technique in Sec. 7.0 of Method 3585.

7.5 Determination of % Dry Weight

If results are to be reported on a dry weight basis, it is necessary to determine the dry weight of the sample.

NOTE: It is highly recommended that the dry weight determination only be made after the analyst has determined that no sample aliquots will be taken from the 60-mL vial for high concentration analysis. This is to minimize loss of volatiles and to avoid sample contamination from the laboratory atmosphere. There is no holding time associated with the dry weight determination. Thus, this determination can be made any time prior to reporting the sample results, as long as the vial containing the additional sample has remained sealed and properly stored.

7.5.1 Weigh 5-10 g of the sample from the 60-mL VOA vial into a tared crucible.

7.5.2 Dry this aliquot overnight at 105°C. Allow to cool in a desiccator before weighing. Calculate the % dry weight as follows:

$$\%\ \text{dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

WARNING: The drying oven should be contained in a hood or vented. Significant laboratory contamination may result from a heavily contaminated hazardous waste sample.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control procedures and Method 5000 for sample preparation QC procedures.

8.2 Before processing any samples, the analyst should demonstrate through the analysis of an organic-free reagent water method blank that all glassware and reagents are interference free. Each time a set of samples is extracted, or there is a change in reagents, a method blank should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement.

8.3 Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat this demonstration whenever new staff are trained or significant changes in instrumentation are made. See Sec. 8.0 of Methods 5000 and 8000 for information on how to accomplish this demonstration.

8.4 Sample Quality Control for Preparation and Analysis - See Sec. 8.0 in Method 5000 and Method 8000 for procedures to follow to demonstrate acceptable continuing performance on each set of samples to be analyzed. These include the method blank, either a matrix spike/matrix spike duplicate or a matrix spike and duplicate sample analysis, a laboratory control sample (LCS), and the addition of surrogates to each sample and QC sample.

8.5 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9.0 METHOD PERFORMANCE

9.1 Single laboratory accuracy and precision data were obtained for the method analytes in three soil matrices, sand, a soil collected 10 feet below the surface of a hazardous landfill, called the

C-Horizon, and a surface garden soil. Each sample was fortified with the analytes at a concentration of 20 ng/5 g, which is equivalent to 4 μg/kg. These data are listed in tables found in Method 8260.

9.2 Single laboratory accuracy and precision data were obtained for certain method analytes when extracting oily liquid using methanol as the extraction solvent. The data are presented in a table in Method 8260. The compounds were spiked into three portions of an oily liquid (taken from a waste site) following the procedure for matrix spiking described in Sec. 7.4. This represents a worst case set of data based on recovery data from many sources of oily liquid.

10.0 REFERENCES

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TABLE 1

QUANTITY OF METHANOL EXTRACT REQUIRED FOR ANALYSIS OF HIGH CONCENTRATION SOILS/SEDIMENTS

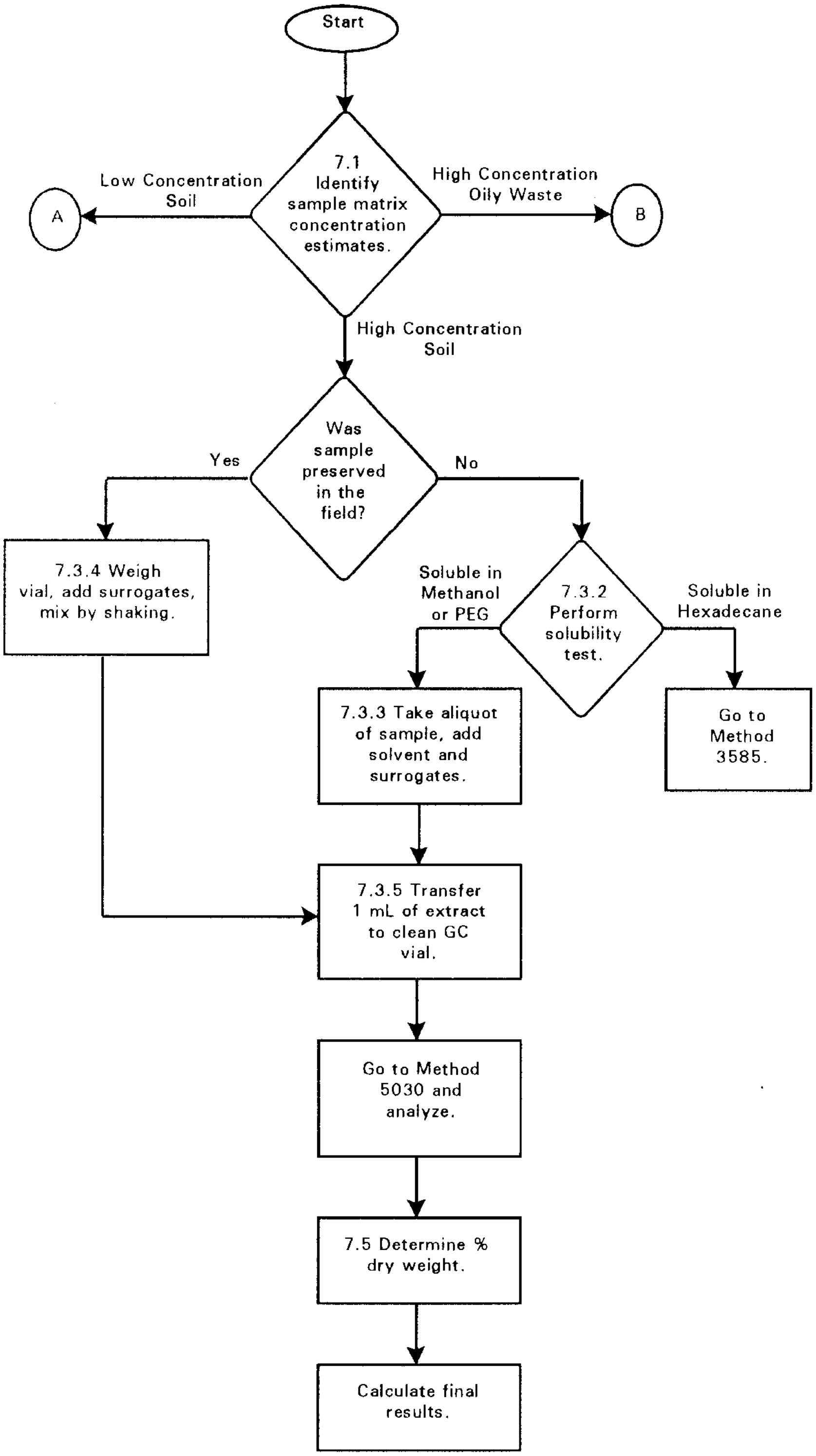
Approximate Concentration Range	Volume of Methanol Extract[a]
500 - 10,000 µg/kg	100 µL
1,000 - 20,000 µg/kg	50 µL
5,000 - 100,000 µg/kg	10 µL
25,000 - 500,000 µg/kg	100 µL of 1/50 dilution[b]

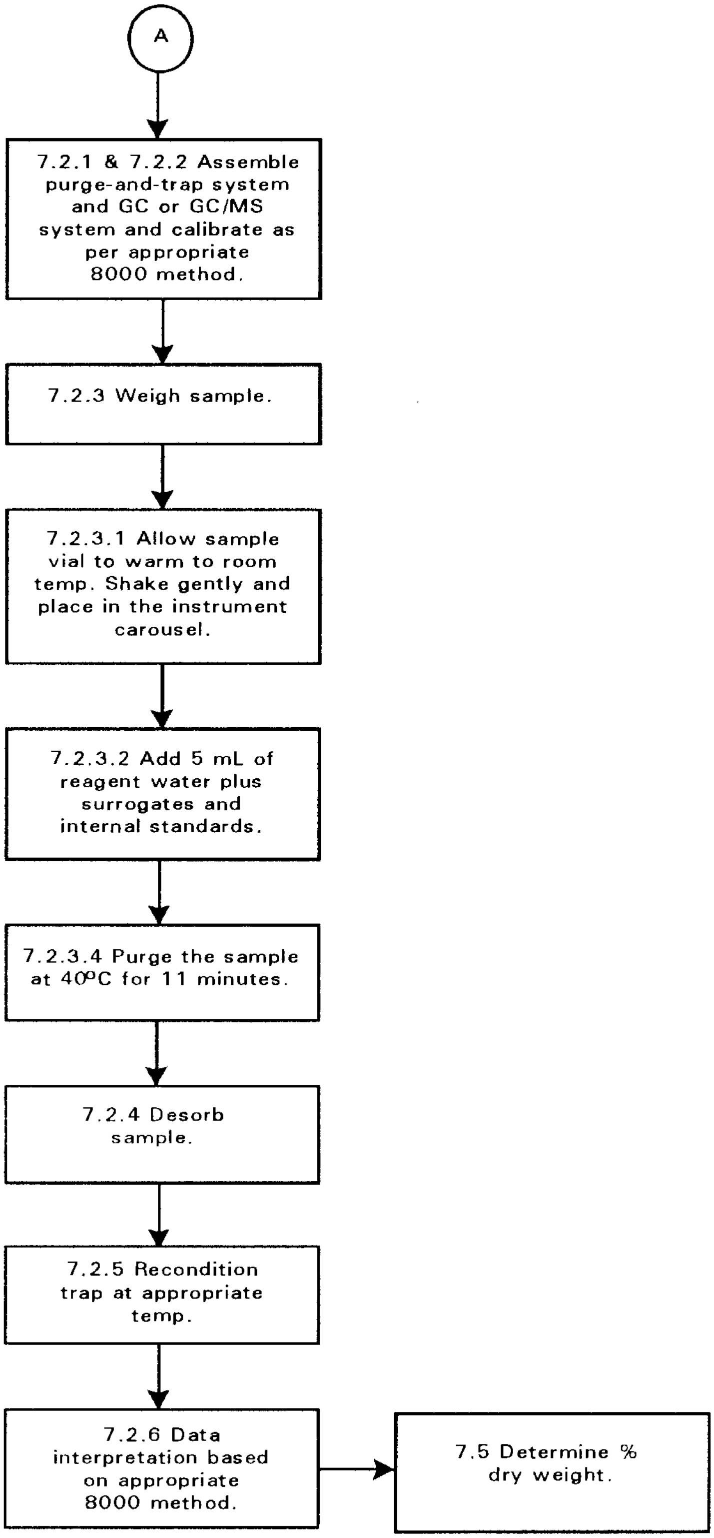
Calculate appropriate dilution factor for concentrations exceeding those in this table.

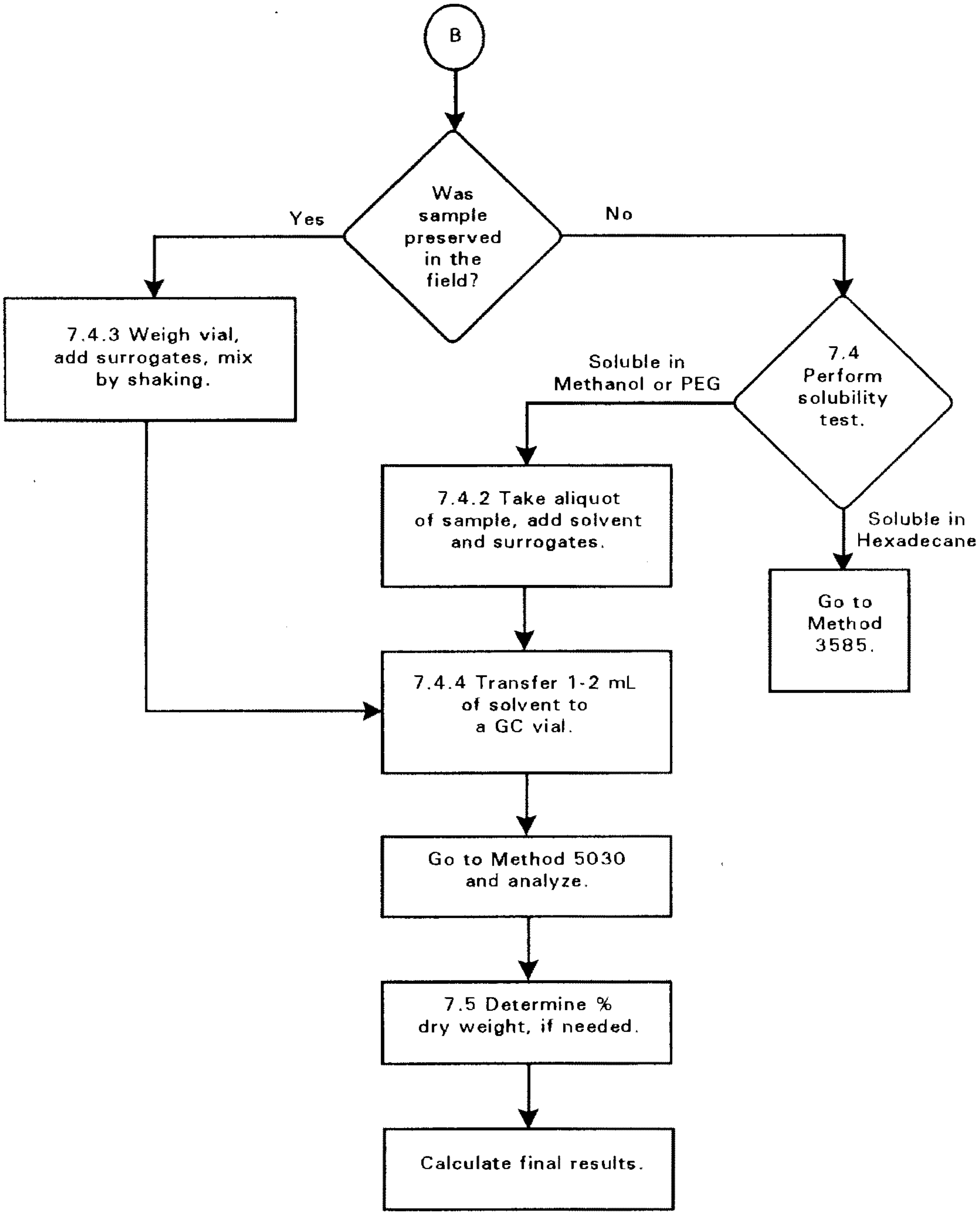
[a] The volume of methanol added to 5 mL of water being purged should be kept constant. Therefore, add to the 5-mL syringe whatever volume of methanol is necessary to maintain a total volume of 100 µL of methanol.

[b] Dilute an aliquot of the methanol extract and then take 100 µL for analysis.

METHOD 5035
CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES




A
7.2.1 & 7.2.2 Assemble purge-and-trap system and GC or GC/MS system and calibrate as per appropriate 8000 method.
7.2.3 Weigh sample.
7.2.3.1 Allow sample vial to warm to room temp. Shake gently and place in the instrument carousel.
7.2.3.2 Add 5 mL of reagent water plus surrogates and internal standards.
7.2.3.4 Purge the sample at 40°C for 11 minutes.
7.2.4 Desorb sample.
7.2.5 Recondition trap at appropriate temp.
7.2.6 Data interpretation based on appropriate 8000 method.
7.5 Determine % dry weight.


B
Was sample preserved in the field?
Yes
No
7.4.3 Weigh vial, add surrogates, mix by shaking.
7.4 Perform solubility test.
Soluble in Methanol or PEG
Soluble in Hexadecane
7.4.2 Take aliquot of sample, add solvent and surrogates.
Go to Method 3585.
7.4.4 Transfer 1-2 mL of solvent to a GC vial.
Go to Method 5030 and analyze.
7.5 Determine % dry weight, if needed.
Calculate final results.

ATTORNEY GENERAL, et al v GELMAN SCIENCES, INC.

(Washtenaw County Circuit Court No. 88-34734-CE)

ATTACHMENT C

LEGEND

- MONITOR WELL
- EXTRACTION WELL
- ARTESIAN WELL
- FORMER RESIDENTIAL WELL ROUTINELY MONITORED
- INJECTION WELL
- PROHIBITION ZONE BOUNDARY

NORTH

0 800 1600
SCALE IN FEET

Hard copy is intended to be 24"x36" when plotted.

FLEIS&VANDENBRINK
DESIGN. BUILD. OPERATE.

GELMAN SCIENCES
Scio Twp., Washtenaw County, Michigan

FOURTH AMENDED CONSENT JUDGEMENT - ATTACHMENT C

PROJECT MGR.	DATE 05/01/20
DRAWN BY	DATE 05/01/20
GEOLOGIST	DATE 05/01/20
CAD FILE	
EDIT	
SCALE	
	DRAWING
PROJECT	806500
SHEET NO.	

<u>ATTORNEY GENERAL, et al</u> v <u>GELMAN SCIENCES, INC.</u>

(Washtenaw County Circuit Court No. 88-34734-CE)

ATTACHMENT D

METHOD 8260B
VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/ MASS SPECTROMETRY (GC/MS)

1.0 SCOPE AND APPLICATION

1.1 Method 8260 is used to determine volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including various air sampling trapping media, ground and surface water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments. The following compounds can be determined by this method:

		Appropriate Preparation Technique[a]					
Compound	CAS No.[b]	5030/ 5035	5031	5032	5021	5041	Direct Inject.
Acetone	67-64-1	pp	c	c	nd	c	c
Acetonitrile	75-05-8	pp	c	nd	nd	nd	c
Acrolein (Propenal)	107-02-8	pp	c	c	nd	nd	c
Acrylonitrile	107-13-1	pp	c	c	nd	c	c
Allyl alcohol	107-18-6	ht	c	nd	nd	nd	c
Allyl chloride	107-05-1	c	nd	nd	nd	nd	c
Benzene	71-43-2	c	nd	c	c	c	c
Benzyl chloride	100-44-7	c	nd	nd	nd	nd	c
Bis(2-chloroethyl)sulfide	505-60-2	pp	nd	nd	nd	nd	c
Bromoacetone	598-31-2	pp	nd	nd	nd	nd	c
Bromochloromethane	74-97-5	c	nd	c	c	c	c
Bromodichloromethane	75-27-4	c	nd	c	c	c	c
4-Bromofluorobenzene (surr)	460-00-4	c	nd	c	c	c	c
Bromoform	75-25-2	c	nd	c	c	c	c
Bromomethane	74-83-9	c	nd	c	c	c	c
n-Butanol	71-36-3	ht	c	nd	nd	nd	c
2-Butanone (MEK)	78-93-3	pp	c	c	nd	nd	c
t-Butyl alcohol	75-65-0	pp	c	nd	nd	nd	c
Carbon disulfide	75-15-0	pp	nd	c	nd	c	c
Carbon tetrachloride	56-23-5	c	nd	c	c	c	c
Chloral hydrate	302-17-0	pp	nd	nd	nd	nd	c
Chlorobenzene	108-90-7	c	nd	c	c	c	c
Chlorobenzene-d_5 (IS)		c	nd	c	c	c	c
Chlorodibromomethane	124-48-1	c	nd	c	nd	c	c
Chloroethane	75-00-3	c	nd	c	c	c	c
2-Chloroethanol	107-07-3	pp	nd	nd	nd	nd	c
2-Chloroethyl vinyl ether	110-75-8	c	nd	c	nd	nd	c
Chloroform	67-66-3	c	nd	c	c	c	c
Chloromethane	74-87-3	c	nd	c	c	c	c
Chloroprene	126-99-8	c	nd	nd	nd	nd	c
3-Chloropropionitrile	542-76-7	I	nd	nd	nd	nd	pc

(continued)

		Appropriate Preparation Technique[a]					
Compound	CAS No.[b]	5030/ 5035	5031	5032	5021	5041	Direct Inject.
Crotonaldehyde	4170-30-3	pp	c	nd	nd	nd	c
1,2-Dibromo-3-chloropropane	96-12-8	pp	nd	nd	c	nd	c
1,2-Dibromoethane	106-93-4	c	nd	nd	c	nd	c
Dibromomethane	74-95-3	c	nd	c	c	c	c
1,2-Dichlorobenzene	95-50-1	c	nd	nd	c	nd	c
1,3-Dichlorobenzene	541-73-1	c	nd	nd	c	nd	c
1,4-Dichlorobenzene	106-46-7	c	nd	nd	c	nd	c
1,4-Dichlorobenzene-d_4 (IS)		c	nd	nd	c	nd	c
cis-1,4-Dichloro-2-butene	1476-11-5	c	nd	c	nd	nd	c
trans-1,4-Dichloro-2-butene	110-57-6	pp	nd	c	nd	nd	c
Dichlorodifluoromethane	75-71-8	c	nd	c	c	nd	c
1,1-Dichloroethane	75-34-3	c	nd	c	c	c	c
1,2-Dichloroethane	107-06-2	c	nd	c	c	c	c
1,2-Dichloroethane-d_4 (surr)		c	nd	c	c	c	c
1,1-Dichloroethene	75-35-4	c	nd	c	c	c	c
trans-1,2-Dichloroethene	156-60-5	c	nd	c	c	c	c
1,2-Dichloropropane	78-87-5	c	nd	c	c	c	c
1,3-Dichloro-2-propanol	96-23-1	pp	nd	nd	nd	nd	c
cis-1,3-Dichloropropene	10061-01-5	c	nd	c	nd	c	c
trans-1,3-Dichloropropene	10061-02-6	c	nd	c	nd	c	c
1,2,3,4-Diepoxybutane	1464-53-5	c	nd	nd	nd	nd	c
Diethyl ether	60-29-7	c	nd	nd	nd	nd	c
1,4-Difluorobenzene (IS)	540-36-3	nd	nd	nd	nd	c	nd
1,4-Dioxane	123-91-1	pp	c	c	nd	nd	c
Epichlorohydrin	106-89-8	l	nd	nd	nd	nd	c
Ethanol	64-17-5	l	c	c	nd	nd	c
Ethyl acetate	141-78-6	l	c	nd	nd	nd	c
Ethylbenzene	100-41-4	c	nd	c	c	c	c
Ethylene oxide	75-21-8	pp	c	nd	nd	nd	c
Ethyl methacrylate	97-63-2	c	nd	c	nd	nd	c
Fluorobenzene (IS)	462-06-6	c	nd	nd	nd	nd	nd
Hexachlorobutadiene	87-68-3	c	nd	nd	c	nd	c
Hexachloroethane	67-72-1	l	nd	nd	nd	nd	c
2-Hexanone	591-78-6	pp	nd	c	nd	nd	c
2-Hydroxypropionitrile	78-97-7	l	nd	nd	nd	nd	pc
Iodomethane	74-88-4	c	nd	c	nd	c	c
Isobutyl alcohol	78-83-1	pp	c	nd	nd	nd	c
Isopropylbenzene	98-82-8	c	nd	nd	c	nd	c
Malononitrile	109-77-3	pp	nd	nd	nd	nd	c
Methacrylonitrile	126-98-7	pp	l	nd	nd	nd	c
Methanol	67-56-1	l	c	nd	nd	nd	c
Methylene chloride	75-09-2	c	nd	c	c	c	c
Methyl methacrylate	80-62-6	c	nd	nd	nd	nd	c
4-Methyl-2-pentanone (MIBK)	108-10-1	pp	c	c	nd	nd	c
Naphthalene	91-20-3	c	nd	nd	c	nd	c

(continued)

		Appropriate Preparation Technique[a]					
Compound	CAS No.[b]	5030/ 5035	5031	5032	5021	5041	Direct Inject.
Nitrobenzene	98-95-3	c	nd	nd	nd	nd	c
2-Nitropropane	79-46-9	c	nd	nd	nd	nd	c
N-Nitroso-di-n-butylamine	924-16-3	pp	c	nd	nd	nd	c
Paraldehyde	123-63-7	pp	c	nd	nd	nd	c
Pentachloroethane	76-01-7	I	nd	nd	nd	nd	c
2-Pentanone	107-87-9	pp	c	nd	nd	nd	c
2-Picoline	109-06-8	pp	c	nd	nd	nd	c
1-Propanol	71-23-8	pp	c	nd	nd	nd	c
2-Propanol	67-63-0	pp	c	nd	nd	nd	c
Propargyl alcohol	107-19-7	pp	I	nd	nd	nd	c
β-Propiolactone	57-57-8	pp	nd	nd	nd	nd	c
Propionitrile (ethyl cyanide)	107-12-0	ht	c	nd	nd	nd	pc
n-Propylamine	107-10-8	c	nd	nd	nd	nd	c
Pyridine	110-86-1	I	c	nd	nd	nd	c
Styrene	100-42-5	c	nd	c	c	c	c
1,1,1,2-Tetrachloroethane	630-20-6	c	nd	nd	c	c	c
1,1,2,2-Tetrachloroethane	79-34-5	c	nd	c	c	c	c
Tetrachloroethene	127-18-4	c	nd	c	c	c	c
Toluene	108-88-3	c	nd	c	c	c	c
Toluene-d_8 (surr)	2037-26-5	c	nd	c	c	c	c
o-Toluidine	95-53-4	pp	c	nd	nd	nd	c
1,2,4-Trichlorobenzene	120-82-1	c	nd	nd	c	nd	c
1,1,1-Trichloroethane	71-55-6	c	nd	c	c	c	c
1,1,2-Trichloroethane	79-00-5	c	nd	c	c	c	c
Trichloroethene	79-01-6	c	nd	c	c	c	c
Trichlorofluoromethane	75-69-4	c	nd	c	c	c	c
1,2,3-Trichloropropane	96-18-4	c	nd	c	c	c	c
Vinyl acetate	108-05-4	c	nd	c	nd	nd	c
Vinyl chloride	75-01-4	c	nd	c	c	c	c
o-Xylene	95-47-6	c	nd	c	c	c	c
m-Xylene	108-38-3	c	nd	c	c	c	c
p-Xylene	106-42-3	c	nd	c	c	c	c

[a] See Sec. 1.2 for other appropriate sample preparation techniques
[b] Chemical Abstract Service Registry Number

c = Adequate response by this technique
ht = Method analyte only when purged at 80°C
nd = Not determined
I = Inappropriate technique for this analyte
pc = Poor chromatographic behavior
pp = Poor purging efficiency resulting in high Estimated Quantitation Limits
surr = Surrogate
IS = Internal Standard

1.2 There are various techniques by which these compounds may be introduced into the GC/MS system. The more common techniques are listed in the table above. Purge-and-trap, by Methods 5030 (aqueous samples) and 5035 (solid and waste oil samples), is the most commonly used technique for volatile organic analytes. However, other techniques are also appropriate and necessary for some analytes. These include direct injection following dilution with hexadecane (Method 3585) for waste oil samples; automated static headspace by Method 5021 for solid samples; direct injection of an aqueous sample (concentration permitting) or injection of a sample concentrated by azeotropic distillation (Method 5031); and closed system vacuum distillation (Method 5032) for aqueous, solid, oil and tissue samples. For air samples, Method 5041 provides methodology for desorbing volatile organics from trapping media (Methods 0010, 0030, and 0031). In addition, direct analysis utilizing a sample loop is used for sub-sampling from Tedlar® bags (Method 0040). Method 5000 provides more general information on the selection of the appropriate introduction method.

1.3 Method 8260 can be used to quantitate most volatile organic compounds that have boiling points below 200°C. Volatile, water soluble compounds can be included in this analytical technique by the use of azeotropic distillation or closed-system vacuum distillation. Such compounds include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides. See Tables 1 and 2 for analytes and retention times that have been evaluated on a purge-and-trap GC/MS system. Also, the method detection limits for 25-mL sample volumes are presented. The following compounds are also amenable to analysis by Method 8260:

Bromobenzene
n-Butylbenzene
sec-Butylbenzene
tert-Butylbenzene
Chloroacetonitrile
1-Chlorobutane
1-Chlorohexane
2-Chlorotoluene
4-Chlorotoluene
Dibromofluoromethane
cis-1,2-Dichloroethene
1,3-Dichloropropane
2,2-Dichloropropane
1,1-Dichloropropene
p-Isopropyltoluene
Methyl acrylate
Methyl-t-butyl ether
Pentafluorobenzene
n-Propylbenzene
1,2,3-Trichlorobenzene
1,2,4-Trimethylbenzene
1,3,5-Trimethylbenzene

1.4 The estimated quantitation limit (EQL) of Method 8260 for an individual compound is somewhat instrument dependent and also dependent on the choice of sample preparation/introduction method. Using standard quadrapole instrumentation and the purge-and-trap technique, limits should be approximately 5 µg/kg (wet weight) for soil/sediment samples, 0.5 mg/kg (wet weight) for wastes, and 5 µg/L for ground water (see Table 3). Somewhat lower limits may be achieved using an ion trap mass spectrometer or other instrumentation of improved design. No matter which instrument is used, EQLs will be proportionately higher for sample extracts and samples that require dilution or when a reduced sample size is used to avoid saturation of the detector.

1.5 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatograph/mass spectrometers, and skilled in the interpretation of mass spectra and their use as a quantitative tool.

2.0 SUMMARY OF METHOD

2.1 The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method or by other methods (see Sec. 1.2). The analytes are introduced directly to a wide-bore capillary column or cryofocussed on a capillary pre-column before being flash evaporated to a narrow-bore capillary for analysis. The column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) interfaced to the gas chromatograph (GC).

2.2 Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or a direct connection. (Wide-bore capillary columns normally require a jet separator, whereas narrow-bore capillary columns may be directly interfaced to the ion source). Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve.

2.3 The method includes specific calibration and quality control steps that supersede the general requirements provided in Method 8000.

3.0 INTERFERENCES

3.1 Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components should be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of calibration and reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter. Subtracting blank values from sample results is not permitted. If reporting values without correcting for the blank results in what the laboratory feels is a false positive result for a sample, the laboratory should fully explained this in text accompanying the uncorrected data.

3.2 Contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. A technique to prevent this problem is to rinse the purging apparatus and sample syringes with two portions of organic-free reagent water between samples. After the analysis of a sample containing high concentrations of volatile organic compounds, one or more blanks should be analyzed to check for cross-contamination. Alternatively, if the sample immediately following the high concentration sample does not contain the volatile organic compounds present in the high level sample, freedom from contamination has been established.

3.3 For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds, or high concentrations of compounds being determined, it may be necessary to wash the purging device with a soap solution, rinse it with organic-free reagent water, and then dry the purging device in an oven at 105°C. In extreme situations, the entire purge-and-trap device may require dismantling and cleaning. Screening of the samples prior to purge-and-trap GC/MS analysis is highly recommended to prevent contamination of the system. This is especially true for soil and waste samples. Screening may be accomplished with an automated headspace technique (Method 5021) or by Method 3820 (Hexadecane Extraction and Screening of Purgeable Organics).

3.4 Many analytes exhibit low purging efficiencies from a 25-mL sample. This often results in significant amounts of these analytes remaining in the sample purge vessel after analysis. After removal of the sample aliquot that was purged, and rinsing the purge vessel three times with organic-free water, the empty vessel should be subjected to a heated purge cycle prior to the analysis of another sample in the same purge vessel. This will reduce sample-to-sample carryover.

3.5 Special precautions must be taken to analyze for methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride. Otherwise, random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing should be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean, since clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.

3.6 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample container into the sample during shipment and storage. A trip blank prepared from organic-free reagent water and carried through the sampling, handling, and storage protocols can serve as a check on such contamination.

3.7 Use of sensitive mass spectrometers to achieve lower detection level will increase the potential to detect laboratory contaminants as interferences.

3.8 Direct injection - Some contamination may be eliminated by baking out the column between analyses. Changing the injector liner will reduce the potential for cross-contamination. A portion of the analytical column may need to be removed in the case of extreme contamination. The use of direct injection will result in the need for more frequent instrument maintenance.

3.9 If hexadecane is added to waste samples or petroleum samples that are analyzed, some chromatographic peaks will elute after the target analytes. The oven temperature program must include a post-analysis bake out period to ensure that semivolatile hydrocarbons are volatilized.

4.0 APPARATUS AND MATERIALS

4.1 Purge-and-trap device for aqueous samples - Described in Method 5030.

4.2 Purge-and-trap device for solid samples - Described in Method 5035.

4.3 Automated static headspace device for solid samples - Described in Method 5021.

4.4 Azeotropic distillation apparatus for aqueous and solid samples - Described in Method 5031.

4.5 Vacuum distillation apparatus for aqueous, solid and tissue samples - Described in Method 5032.

4.6 Desorption device for air trapping media for air samples - Described in Method 5041.

4.7 Air sampling loop for sampling from Tedlar® bags for air samples - Described in Method 0040.

4.8 Injection port liners (HP Catalog #18740-80200, or equivalent) - modified for direct injection analysis by placing a 1-cm plug of glass wool approximately 50-60 mm down the length of the injection port towards the oven (see illustration below). A 0.53-mm ID column is mounted 1 cm into the liner from the oven side of the injection port, according to manufacturer's specifications.

4.9 Gas chromatography/mass spectrometer/data system

4.9.1 Gas chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection with appropriate interface for sample introduction device. The system includes all required accessories, including syringes, analytical columns, and gases.

4.9.1.1 The GC should be equipped with variable constant differential flow controllers so that the column flow rate will remain constant throughout desorption and temperature program operation.

4.9.1.2 For some column configurations, the column oven must be cooled to less than 30°C, therefore, a subambient oven controller may be necessary.

4.9.1.3 The capillary column is either directly coupled to the source or interfaced through a jet separator, depending on the size of the capillary and the requirements of the GC/MS system.

4.9.1.4 Capillary pre-column interface - This device is the interface between the sample introduction device and the capillary gas chromatograph, and is necessary when using cryogenic cooling. The interface condenses the desorbed sample components and focuses them into a narrow band on an uncoated fused-silica capillary pre-column. When the interface is flash heated, the sample is transferred to the analytical capillary column.

4.9.1.5 During the cryofocussing step, the temperature of the fused-silica in the interface is maintained at -150°C under a stream of liquid nitrogen. After the desorption period, the interface must be capable of rapid heating to 250°C in 15 seconds or less to complete the transfer of analytes.

4.9.2 Gas chromatographic columns

4.9.2.1 Column 1 - 60 m x 0.75 mm ID capillary column coated with VOCOL (Supelco), 1.5-µm film thickness, or equivalent.

4.9.2.2 Column 2 - 30 - 75 m x 0.53 mm ID capillary column coated with DB-624 (J&W Scientific), Rt_x-502.2 (RESTEK), or VOCOL (Supelco), 3-µm film thickness, or equivalent.

4.9.2.3 Column 3 - 30 m x 0.25 - 0.32 mm ID capillary column coated with 95% dimethyl - 5% diphenyl polysiloxane (DB-5, Rt_x-5, SPB-5, or equivalent), 1-µm film thickness.

4.9.2.4 Column 4 - 60 m x 0.32 mm ID capillary column coated with DB-624 (J&W Scientific), 1.8-µm film thickness, or equivalent.

4.9.3 Mass spectrometer - Capable of scanning from 35 to 300 amu every 2 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for 4-Bromofluorobenzene (BFB) which meets all of the criteria in Table 4 when 5-50 ng of the GC/MS tuning standard (BFB) are injected through the GC. To ensure sufficient precision of mass spectral data, the desirable MS scan rate allows acquisition of at least five spectra while a sample component elutes from the GC.

An ion trap mass spectrometer may be used if it is capable of axial modulation to reduce ion-molecule reactions and can produce electron impact-like spectra that match those in the EPA/NIST Library. Because ion-molecule reactions with water and methanol in an ion trap mass spectrometer may produce interferences that coelute with chloromethane and chloroethane, the base peak for both of these analytes will be at m/z 49. This ion should be used as the quantitation ion in this case. The mass spectrometer must be capable of producing a mass spectrum for BFB which meets all of the criteria in Table 3 when 5 or 50 ng are introduced.

4.9.4 GC/MS interface - Two alternatives may be used to interface the GC to the mass spectrometer.

4.9.4.1 Direct coupling, by inserting the column into the mass spectrometer, is generally used for 0.25 - 0.32 mm ID columns.

4.9.4.2 A jet separator, including an all-glass transfer line and glass enrichment device or split interface, is used with a 0.53 mm column.

4.9.4.3 Any enrichment device or transfer line may be used, if all of the performance specifications described in Sec. 8.0 (including acceptable calibration at 50 ng or less) can be achieved. GC/MS interfaces constructed entirely of glass or of glass-lined materials are recommended. Glass may be deactivated by silanizing with dichlorodimethylsilane.

4.9.5 Data system - A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program must be interfaced to the mass spectrometer. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

4.10 Microsyringes - 10-, 25-, 100-, 250-, 500-, and 1,000-µL.

4.11 Syringe valve - Two-way, with Luer ends (three each), if applicable to the purging device.

4.12 Syringes - 5-, 10-, or 25-mL, gas-tight with shutoff valve.

4.13 Balance - Analytical, capable of weighing 0.0001 g, and top-loading, capable of weighing 0.1 g.

4.14 Glass scintillation vials - 20-mL, with PTFE-lined screw-caps or glass culture tubes with PTFE-lined screw-caps.

4.15 Vials - 2-mL, for GC autosampler.

4.16 Disposable pipets - Pasteur.

4.17 Volumetric flasks, Class A - 10-mL and 100-mL, with ground-glass stoppers.

4.18 Spatula - Stainless steel.

5.0 REAGENTS

5.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all inorganic reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

5.3 Methanol, CH_3OH - Pesticide quality or equivalent, demonstrated to be free of analytes. Store apart from other solvents.

5.4 Reagent Hexadecane - Reagent hexadecane is defined as hexadecane in which interference is not observed at the method detection limit of compounds of interest. Hexadecane quality is demonstrated through the analysis of a solvent blank injected directly into the GC/MS. The results of such a blank analysis must demonstrate that all interfering volatiles have been removed from the hexadecane.

5.5 Polyethylene glycol, $H(OCH_2CH_2)_nOH$ - Free of interferences at the detection limit of the target analytes.

5.6 Hydrochloric acid (1:1 v/v), HCl - Carefully add a measured volume of concentrated HCl to an equal volume of organic-free reagent water.

5.7 Stock solutions - Stock solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol, using assayed liquids or gases, as appropriate.

5.7.1 Place about 9.8 mL of methanol in a 10-mL tared ground-glass-stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 minutes or until all alcohol-wetted surfaces have dried. Weigh the flask to the nearest 0.0001 g.

5.7.2 Add the assayed reference material, as described below.

5.7.2.1 Liquids - Using a 100-µL syringe, immediately add two or more drops of assayed reference material to the flask; then reweigh. The liquid must fall directly into the alcohol without contacting the neck of the flask.

5.7.2.2 Gases - To prepare standards for any compounds that boil below 30°C (e.g., bromomethane, chloroethane, chloromethane, or vinyl chloride), fill a 5-mL valved gas-tight syringe with the reference standard to the 5.0 mL mark. Lower the needle to

5 mm above the methanol meniscus. Slowly introduce the reference standard above the surface of the liquid. The heavy gas will rapidly dissolve in the methanol. Standards may also be prepared by using a lecture bottle equipped with a septum. Attach PTFE tubing to the side arm relief valve and direct a gentle stream of gas into the methanol meniscus.

5.7.3 Reweigh, dilute to volume, stopper, and then mix by inverting the flask several times. Calculate the concentration in milligrams per liter (mg/L) from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially-prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

5.7.4 Transfer the stock standard solution into a bottle with a PTFE-lined screw-cap. Store, with minimal headspace and protected from light, at -10°C or less or as recommended by the standard manufacturer. Standards should be returned to the freezer as soon as the analyst has completed mixing or diluting the standards to prevent the evaporation of volatile target compounds.

5.7.5 Frequency of Standard Preparation

5.7.5.1 Standards for the permanent gases should be monitored frequently by comparison to the initial calibration curve. Fresh standards should be prepared if this check exceeds a 20% drift. Standards for gases usually need to be replaced after one week or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. Dichlorodifluoromethane and dichloromethane will usually be the first compounds to evaporate from the standard and should, therefore, be monitored very closely when standards are held beyond one week.

5.7.5.2 Standards for the non-gases should be monitored frequently by comparison to the initial calibration. Fresh standards should be prepared if this check exceeds a 20% drift. Standards for non-gases usually need to be replaced after six months or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. Standards of reactive compounds such as 2-chloroethyl vinyl ether and styrene may need to be prepared more frequently.

5.7.6 Preparation of Calibration Standards From a Gas Mixture

An optional calibration procedure involves using a certified gaseous mixture daily, utilizing a commercially-available gaseous analyte mixture of bromomethane, chloromethane, chloroethane, vinyl chloride, dichloro-difluoromethane and trichlorofluoromethane in nitrogen. Mixtures of documented quality are stable for as long as six months without refrigeration. (VOA-CYL III, RESTEK Corporation, Cat. #20194 or equivalent).

5.7.6.1 Before removing the cylinder shipping cap, be sure the valve is completely closed (turn clockwise). The contents are under pressure and should be used in a well-ventilated area.

5.7.6.2 Wrap the pipe thread end of the Luer fitting with PTFE tape. Remove the shipping cap from the cylinder and replace it with the Luer fitting.

5.7.6.3 Transfer half the working standard containing other analytes, internal standards, and surrogates to the purge apparatus.

5.7.6.4 Purge the Luer fitting and stem on the gas cylinder prior to sample removal using the following sequence:

a) Connect either the 100-µL or 500-µL Luer syringe to the inlet fitting of the cylinder.

b) Make sure the on/off valve on the syringe is in the open position.

c) Slowly open the valve on the cylinder and withdraw a full syringe volume.

d) Be sure to close the valve on the cylinder before you withdraw the syringe from the Luer fitting.

e) Expel the gas from the syringe into a well-ventilated area.

f) Repeat steps a through e one more time to fully purge the fitting.

5.7.6.5 Once the fitting and stem have been purged, quickly withdraw the volume of gas you require using steps 5.6.6.1.4(a) through (d). Be sure to close the valve on the cylinder and syringe before you withdraw the syringe from the Luer fitting.

5.7.6.6 Open the syringe on/off valve for 5 seconds to reduce the syringe pressure to atmospheric pressure. The pressure in the cylinder is ~30 psi.

5.7.6.7 The gas mixture should be quickly transferred into the reagent water through the female Luer fitting located above the purging vessel.

NOTE: Make sure the arrow on the 4-way valve is pointing toward the female Luer fitting when transferring the sample from the syringe. Be sure to switch the 4-way valve back to the closed position before removing the syringe from the Luer fitting.

5.7.6.8 Transfer the remaining half of the working standard into the purging vessel. This procedure insures that the total volume of gas mix is flushed into the purging vessel, with none remaining in the valve or lines.

5.7.6.9 The concentration of each compound in the cylinder is typically 0.0025 µg/µL.

5.7.6.10 The following are the recommended gas volumes spiked into 5 mL of water to produce a typical 5-point calibration:

Gas Volume	Calibration Concentration
40 µL	20 µg/L
100 µL	50 µg/L
200 µL	100 µg/L
300 µL	150 µg/L
400 µL	200 µg/L

5.7.6.11 The following are the recommended gas volumes spiked into 25 mL of water to produce a typical 5-point calibration:

Gas Volume	Calibration Concentration
10 µL	1 µg/L
20 µL	2 µg/L
50 µL	5 µg/L
100 µL	10 µg/L
250 µL	25 µg/L

5.8 Secondary dilution standards - Using stock standard solutions, prepare secondary dilution standards in methanol containing the compounds of interest, either singly or mixed together. Secondary dilution standards must be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Store in a vial with no headspace. Replace after one week. Secondary standards for gases should be replaced after one week unless the acceptability of the standard can be documented. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations. The analyst should also handle and store standards as stated in Sec. 5.7.4 and return them to the freezer as soon as standard mixing or diluting is completed to prevent the evaporation of volatile target compounds.

5.9 Surrogate standards - The recommended surrogates are toluene-d_8, 4-bromofluorobenzene, 1,2-dichloroethane-d_4, and dibromofluoromethane. Other compounds may be used as surrogates, depending upon the analysis requirements. A stock surrogate solution in methanol should be prepared as described above, and a surrogate standard spiking solution should be prepared from the stock at a concentration of 50-250 µg/10 mL, in methanol. Each sample undergoing GC/MS analysis must be spiked with 10 µL of the surrogate spiking solution prior to analysis. If a more sensitive mass spectrometer is employed to achieve lower detection levels, then more dilute surrogate solutions may be required.

5.10 Internal standards - The recommended internal standards are fluorobenzene, chlorobenzene-d_5, and 1,4-dichlorobenzene-d_4. Other compounds may be used as internal standards as long as they have retention times similar to the compounds being detected by GC/MS. Prepare internal standard stock and secondary dilution standards in methanol using the procedures described in Secs. 5.7 and 5.8. It is recommended that the secondary dilution standard be prepared at a concentration of 25 mg/L of each internal standard compound. Addition of 10 µL of this standard to 5.0 mL of sample or calibration standard would be the equivalent of 50 µg/L. If a more sensitive mass spectrometer is employed to achieve lower detection levels, then more dilute internal standard solutions may be required. Area counts of the internal standard peaks should be between 50-200% of the areas of the target analytes in the mid-point calibration analysis.

5.11 4-Bromofluorobenzene (BFB) standard - A standard solution containing 25 ng/µL of BFB in methanol should be prepared. If a more sensitive mass spectrometer is employed to achieve lower detection levels, then a more dilute BFB standard solution may be required.

5.12 Calibration standards -There are two types of calibration standards used for this method: initial calibration standards and calibration verification standards. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

5.12.1 Initial calibration standards should be prepared at a minimum of five different concentrations from the secondary dilution of stock standards (see Secs. 5.7 and 5.8) or from a premixed certified solution. Prepare these solutions in organic-free reagent water. At least one of the calibration standards should correspond to a sample concentration at or below that necessary to meet the data quality objectives of the project. The remaining standards should correspond to the range of concentrations found in typical samples but should not exceed the working range of the GC/MS system. Initial calibration standards should be mixed from fresh stock standards and dilution standards when generating an initial calibration curve.

5.12.2 Calibration verification standards should be prepared at a concentration near the mid-point of the initial calibration range from the secondary dilution of stock standards (see Secs. 5.7 and 5.8) or from a premixed certified solution. Prepare these solutions in organic-free reagent water. See Sec. 7.4 for guidance on calibration verification.

5.12.3 It is the intent of EPA that all target analytes for a particular analysis be included in the initial calibration and calibration verification standard(s). These target analytes may not include the entire list of analytes (Sec. 1.1) for which the method has been demonstrated. However, the laboratory shall not report a quantitative result for a target analyte that was not included in the calibration standard(s).

5.12.4 The calibration standards must also contain the internal standards chosen for the analysis.

5.13 Matrix spiking and laboratory control sample (LCS) standards - Matrix spiking standards should be prepared from volatile organic compounds which are representative of the compounds being investigated. At a minimum, the matrix spike should include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. The matrix spiking solution should contain compounds that are expected to be found in the types of samples to be analyzed.

5.13.1 Some permits may require the spiking of specific compounds of interest, especially if polar compounds are a concern, since the spiking compounds listed above would not be representative of such compounds. The standard should be prepared in methanol, with each compound present at a concentration of 250 µg/10.0 mL.

5.13.2 The spiking solutions should not be prepared from the same standards as the calibration standards. However, the same spiking standard prepared for the matrix spike may be used for the LCS.

5.13.3 If a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute matrix spiking solutions may be required.

5.14 Great care must be taken to maintain the integrity of all standard solutions. It is recommended all standards in methanol be stored at -10°C or less, in amber bottles with PTFE-lined screw-caps.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

See the introductory material to this chapter, Organic Analytes, Sec. 4.1.

7.0 PROCEDURE

7.1 Various alternative methods are provided for sample introduction. All internal standards, surrogates, and matrix spiking compounds (when applicable) must be added to the samples before introduction into the GC/MS system. Consult the sample introduction method for the procedures by which to add such standards.

7.1.1 Direct injection - This includes: injection of an aqueous sample containing a very high concentration of analytes; injection of aqueous concentrates from Method 5031 (azeotropic distillation); and injection of a waste oil diluted 1:1 with hexadecane (Method 3585). Direct injection of aqueous samples (non-concentrated) has very limited applications. It is only used for the determination of volatiles at the toxicity characteristic (TC) regulatory limits or at concentrations in excess of 10,000 µg/L. It may also be used in conjunction with the test for ignitability in aqueous samples (along with Methods 1010 and 1020), to determine if alcohol is present at greater than 24%.

7.1.2 Purge-and-trap - This includes purge-and-trap for aqueous samples (Method 5030) and purge-and-trap for solid samples (Method 5035). Method 5035 also provides techniques for extraction of high concentration solid and oily waste samples by methanol (and other water-miscible solvents) with subsequent purge-and-trap from an aqueous matrix using Method 5030.

7.1.2.1 Traditionally, the purge-and-trap of aqueous samples is performed at ambient temperature, while purging of soil/solid samples is performed at 40°C, to improve purging efficiency.

7.1.2.2 Aqueous and soil/solid samples may also be purged at temperatures above those being recommended as long as all calibration standards, samples, and QC samples are purged at the same temperature, appropriate trapping material is used to handle the excess water, and the laboratory demonstrates acceptable method performance for the project. Purging of aqueous samples at elevated temperatures (e.g., 40°C) may improve the purging performance of many of the water soluble compounds which have poor purging efficiencies at ambient temperatures.

7.1.3 Vacuum distillation - this technique may be used for the introduction of volatile organics from aqueous, solid, or tissue samples (Method 5032) into the GC/MS system.

7.1.4 Automated static headspace - this technique may be used for the introduction of volatile organics from solid samples (Method 5021) into the GC/MS system.

7.1.5 Cartridge desorption - this technique may be for the introduction of volatile organics from sorbent cartridges (Method 5041) used in the sampling of air. The sorbent cartridges are from the volatile organics sampling train (VOST) or SMVOC (Method 0031).

7.2 Recommended chromatographic conditions

7.2.1 General conditions

Injector temperature:	200 - 225°C
Transfer line temperature:	250 - 300°C

7.2.2 Column 1 and Column 2 with cryogenic cooling (example chromatograms are presented in Figures 1 and 2)

Carrier gas (He) flow rate:	15 mL/min
Initial temperature:	10°C, hold for 5 minutes
Temperature program:	6°C/min to 70°C, then 15°C/min to 145°C
Final temperature:	145°C, hold until all expected compounds have eluted.

7.2.5 Direct injection - Column 2

Carrier gas (He) flow rate:	4 mL/min
Column:	J&W DB-624, 70m x 0.53 mm
Initial temperature:	40°C, hold for 3 minutes
Temperature program:	8°C/min
Final temperature:	260°C, hold until all expected compounds have eluted.
Column Bake out:	75 minutes
Injector temperature:	200-225°C
Transfer line temperature:	250-300°C

7.2.6 Direct split interface - Column 4

Carrier gas (He) flow rate:	1.5 mL/min
Initial temperature:	35°C, hold for 2 minutes
Temperature program:	4°C/min to 50°C 10°C/min to 220°C
Final temperature:	220°C, hold until all expected compounds have eluted
Split ratio:	100:1
Injector temperature:	125°C

7.3 Initial calibration

Establish the GC/MS operating conditions, using the following as guidance:

Mass range:	35 - 260 amu
Scan time:	0.6 - 2 sec/scan
Source temperature:	According to manufacturer's specifications
Ion trap only:	Set axial modulation, manifold temperature, and emission current to manufacturer's recommendations

7.3.1 Each GC/MS system must be hardware-tuned to meet the criteria in Table 4 for a 5-50 ng injection or purging of 4-bromofluorobenzene (2-µL injection of the BFB standard). Analyses must not begin until these criteria are met.

7.3.1.1 In the absence of specific recommendations on how to acquire the mass spectrum of BFB from the instrument manufacturer, the following approach has been shown to be useful: The mass spectrum of BFB may be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of

BFB. Do not background subtract part of the BFB peak. Alternatively, the analyst may use other documented approaches suggested by the instrument manufacturer.

7.3.1.2 Use the BFB mass intensity criteria in Table 4 as tuning acceptance criteria. Alternatively, other documented tuning criteria may be used (e.g., CLP, Method 524.2, or manufacturer's instructions), provided that method performance is not adversely affected.

NOTE: All subsequent standards, samples, MS/MSDs, LCSs, and blanks associated with a BFB analysis must use identical mass spectrometer instrument conditions.

7.3.2 Set up the sample introduction system as outlined in the method of choice (see Sec. 7.1). A different calibration curve is necessary for each method because of the differences in conditions and equipment. A set of at least five different calibration standards is necessary (see Sec. 5.12 and Method 8000). Calibration must be performed using the sample introduction technique that will be used for samples. For Method 5030, the purging efficiency for 5 mL of water is greater than for 25 mL. Therefore, develop the standard curve with whichever volume of sample that will be analyzed.

7.3.2.1 To prepare a calibration standard, add an appropriate volume of a secondary dilution standard solution to an aliquot of organic-free reagent water in a volumetric flask. Use a microsyringe and rapidly inject the alcoholic standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection. Mix by inverting the flask three times only. Discard the contents contained in the neck of the flask. Aqueous standards are not stable and should be prepared daily. Transfer 5.0 mL (or 25 mL if lower detection limits are required) of each standard to a gas tight syringe along with 10 µL of internal standard. Then transfer the contents to the appropriate device or syringe. Some of the introduction methods may have specific guidance on the volume of calibration standard and the way the standards are transferred to the device.

7.3.2.2 The internal standards selected in Sec. 5.10 should permit most of the components of interest in a chromatogram to have retention times of 0.80 - 1.20, relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation (see Table 1). If interferences are noted, use the next most intense ion as the quantitation ion.

7.3.2.3 To prepare a calibration standard for direct injection analysis of waste oil, dilute standards in hexadecane.

7.3.3 Proceed with the analysis of the calibration standards following the procedure in the introduction method of choice. For direct injection, inject 1 - 2 µL into the GC/MS system. The injection volume will depend upon the chromatographic column chosen and the tolerance of the specific GC/MS system to water.

7.3.4 Tabulate the area response of the characteristic ions (see Table 5) against the concentration for each target analyte and each internal standard. Calculate response factors (RF) for each target analyte relative to one of the internal standards. The internal standard selected for the calculation of the RF for a target analyte should be the internal standard that has a retention time closest to the analyte being measured (Sec. 7.6.2).

The RF is calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

A_s = Peak area (or height) of the analyte or surrogate.
A_{is} = Peak area (or height) of the internal standard.
C_s = Concentration of the analyte or surrogate.
C_{is} = Concentration of the internal standard.

7.3.5 System performance check compounds (SPCCs) - Calculate the mean RF for each target analyte using the five RF values calculated from the initial (5-point) calibration curve. A system performance check should be made before this calibration curve is used. Five compounds (the System Performance Check Compounds, or SPCCs) are checked for a minimum average response factor. These compounds are chloromethane; 1,1-dichloroethane; bromoform; chlorobenzene; and 1,1,2,2-tetrachloroethane. These compounds are used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system. Example problems include:

7.3.5.1 Chloromethane is the most likely compound to be lost if the purge flow is too fast.

7.3.5.2 Bromoform is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve bromoform response.

7.3.5.3 Tetrachloroethane and 1,1-dichloroethane are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

7.3.5.4 The minimum mean response factors for the volatile SPCCs are as follows:

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

7.3.6 Calibration check compounds (CCCs)

7.3.6.1 The purpose of the CCCs are to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites on the column. Meeting the CCC criteria is not a substitute for successful calibration of the target analytes using one of the approaches described in Sec. 7.0 of Method 8000.

7.3.6.2 Calculate the standard deviation (SD) and relative standard deviation (RSD) of the response factors for all target analytes from the initial calibration, as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^{n}(RF_i - \overline{RF})^2}{n-1}} \qquad RSD = \frac{SD}{\overline{RF}} \times 100$$

where:

RF_i = RF for each of the calibration standards
$\overline{RF}$ = mean RF for each compound from the initial calibration
n = Number of calibration standards, e.g., 5

7.3.6.3 The RSD should be less than or equal to 15% for each target analyte. However, the RSD for each individual Calibration Check Compound (CCC) must be equal or less than 30%. If the CCCs are not included in the list of analytes for a project, and therefore not included in the calibration standards, refer to Sec. 7.0 of Method 8000. The CCCs are:

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl chloride

7.3.6.4 If an RSD of greater than 30% is measured for any CCC, then corrective action to eliminate a system leak and/or column reactive sites is necessary before reattempting calibration.

7.3.7 Evaluation of retention times - The relative retention times of each target analyte in each calibration standard should agree within 0.06 relative retention time units. Late-eluting compounds usually have much better agreement.

7.3.8 Linearity of target analytes

7.3.8.1 If the RSD of any target analyte is 15% or less, then the response factor is assumed to be constant over the calibration range, and the average response factor may be used for quantitation (Sec. 7.7.2).

7.3.8.2 If the RSD of any target analyte is greater than 15%, refer to Sec. 7.0 of Method 8000 for additional calibration options. One of the options must be applied to GC/MS calibration in this situation, or a new initial calibration must be performed.

NOTE: Method 8000 specifies a linearity criterion of 20% RSD. That criterion pertains to GC and HPLC methods other than GC/MS. Method 8260 requires 15% RSD as evidence of sufficient linearity to employ an average response factor.

7.3.8.3 When the RSD exceeds 15%, the plotting and visual inspection of a calibration curve can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

NOTE: The 20% RSD criteria in Method 8000 pertains to GC and HPLC methods other than GC/MS. Method 8260 requires 15% RSD.

7.4 GC/MS calibration verification - Calibration verification consists of three steps that are performed at the beginning of each 12-hour analytical shift.

7.4.1 Prior to the analysis of samples or calibration standards, inject or introduce 5-50 ng of the 4-bromofluorobenzene standard into the GC/MS system. The resultant mass spectra for the BFB must meet the criteria given in Table 4 before sample analysis begins. These criteria must be demonstrated each 12-hour shift during which samples are analyzed.

7.4.2 The initial calibration curve (Sec. 7.3) for each compound of interest should be verified once every 12 hours prior to sample analysis, using the introduction technique used for samples. This is accomplished by analyzing a calibration standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS. The results from the calibration standard analysis should meet the verification acceptance criteria provided in Secs. 7.4.4 through 7.4.7.

NOTE: The BFB and calibration verification standard may be combined into a single standard as long as both tuning and calibration verification acceptance criteria for the project can be met without interferences.

7.4.3 A method blank should be analyzed after the calibration standard, or at any other time during the analytical shift, to ensure that the total system (introduction device, transfer lines and GC/MS system) is free of contaminants. If the method blank indicates contamination, then it may be appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples. See Sec. 8.0 of Method 8000 for method blank performance criteria.

7.4.4 System Performance Check Compounds (SPCCs)

7.4.4.1 A system performance check must be made during every 12-hour analytical shift. Each SPCC compound in the calibration verification standard must meet its minimum response factor (see Sec. 7.3.5.4). This is the same check that is applied during the initial calibration.

7.4.4.2 If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before sample analysis begins.

7.4.5 Calibration Check Compounds (CCCs)

7.4.5.1 After the system performance check is met, the CCCs listed in Sec. 7.3.6 are used to check the validity of the initial calibration. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model. Refer to Sec. 7.0 of Method 8000 for guidance on calculating percent difference and drift.

7.4.5.2 If the percent difference or drift for each CCC is less than or equal to 20%, the initial calibration is assumed to be valid. If the criterion is not met (i.e., greater than 20% difference or drift), for any one CCC, then corrective action must be taken prior to the analysis of samples. If the CCC's are not included in the list of analytes for a project, and therefore not included in the calibration standards, then all analytes must meet the 20% difference or drift criterion.

7.4.5.3 Problems similar to those listed under SPCCs could affect the CCCs. If the problem cannot be corrected by other measures, a new five-point initial calibration must be generated. The CCC criteria must be met before sample analysis begins.

7.4.6 Internal standard retention time - The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

7.4.7 Internal standard response - If the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to + 100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

7.5 GC/MS analysis of samples

7.5.1 It is highly recommended that the sample be screened to minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds. Some of the screening options available utilizing SW-846 methods are automated headspace-GC/FID (Methods 5021/8015), automated headspace-GC/PID/ELCD (Methods 5021/8021), or waste dilution-GC/PID/ELCD (Methods 3585/8021) using the same type of capillary column. When used only for screening purposes, the quality control requirements in the methods above may be reduced as appropriate. Sample screening is particularly important when Method 8260 is used to achieve low detection levels.

7.5.2 BFB tuning criteria and GC/MS calibration verification criteria must be met before analyzing samples.

7.5.3 All samples and standard solutions must be allowed to warm to ambient temperature before analysis. Set up the introduction device as outlined in the method of choice.

7.5.4 The process of taking an aliquot destroys the validity of remaining volume of an aqueous sample for future analysis. Therefore, if only one VOA vial is provided to the laboratory, the analyst should prepare two aliquots for analysis at this time, to protect against possible loss of sample integrity. This second sample is maintained only until such time when the analyst has determined that the first sample has been analyzed properly. For aqueous samples, one 20-mL syringe could be used to hold two 5-mL aliquots. If the second aliquot is to be taken from the syringe, it must be analyzed within 24 hours. Care must be taken to prevent air from leaking into the syringe.

7.5.5 Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample or standard bottle, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. If lower detection limits are required, use a 25-mL syringe, and adjust the final volume to 25.0 mL.

7.5.6 The following procedure may be used to dilute aqueous samples for analysis of volatiles. All steps must be performed without delays, until the diluted sample is in a gas-tight syringe.

7.5.6.1 Dilutions may be made in volumetric flasks (10- to 100-mL). Select the volumetric flask that will allow for the necessary dilution. Intermediate dilution steps may be necessary for extremely large dilutions.

7.5.6.2 Calculate the approximate volume of organic-free reagent water to be added to the volumetric flask, and add slightly less than this quantity of organic-free reagent water to the flask.

7.5.6.3 Inject the appropriate volume of the original sample from the syringe into the flask. Aliquots of less than 1 mL are not recommended. Dilute the sample to the mark with organic-free reagent water. Cap the flask, invert, and shake three times. Repeat above procedure for additional dilutions.

7.5.6.4 Fill a 5-mL syringe with the diluted sample, as described in Sec. 7.5.5.

7.5.7 Compositing aqueous samples prior to GC/MS analysis

7.5.7.1 Add 5 mL of each sample (up to 5 samples are allowed) to a 25-mL glass syringe. Special precautions must be made to maintain zero headspace in the syringe. Larger volumes of a smaller number of samples may be used, provided that equal volumes of each sample are composited.

7.5.7.2 The samples must be cooled to 4°C or less during this step to minimize volatilization losses. Sample vials may be placed in a tray of ice during the processing.

7.5.7.3 Mix each vial well and draw out a 5-mL aliquot with the 25-mL syringe.

7.5.7.4 Once all the aliquots have been combined on the syringe, invert the syringe several times to mix the aliquots. Introduce the composited sample into the instrument, using the method of choice (see Sec. 7.1).

7.5.7.5 If less than five samples are used for compositing, a proportionately smaller syringe may be used, unless a 25-mL sample is to be purged.

7.5.8 Add 10 µL of the surrogate spiking solution and 10 µL of the internal standard spiking solution to each sample either manually or by autosampler. The surrogate and internal standards may be mixed and added as a single spiking solution. The addition of 10 µL of the surrogate spiking solution to 5 mL of aqueous sample will yield a concentration of 50 µg/L of each surrogate standard. The addition of 10 µL of the surrogate spiking solution to 5 g of a non-aqueous sample will yield a concentration of 50 µg/kg of each standard.

If a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute surrogate and internal standard solutions may be required.

7.5.9 Add 10 µL of the matrix spike solution (Sec. 5.13) to a 5-mL aliquot of the sample chosen for spiking. Disregarding any dilutions, this is equivalent to a concentration of 50 µg/L of each matrix spike standard.

7.5.9.1 Follow the same procedure in preparing the laboratory control sample (LCS), except the spike is added to a clean matrix. See Sec. 8.4 and Method 5000 for more guidance on the selection and preparation of the matrix spike and the LCS.

7.5.9.2 If a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute matrix spiking and LCS solutions may be required.

7.5.10 Analyze the sample following the procedure in the introduction method of choice.

7.5.10.1 For direct injection, inject 1 to 2 µL into the GC/MS system. The volume limitation will depend upon the chromatographic column chosen and the tolerance of the specific GC/MS system to water (if an aqueous sample is being analyzed).

7.5.10.2 The concentration of the internal standards, surrogates, and matrix spiking standards (if any) added to the injection aliquot must be adjusted to provide the same concentration in the 1-2 µL injection as would be introduced into the GC/MS by purging a 5-mL aliquot.

NOTE: It may be a useful diagnostic tool to monitor internal standard retention times and responses (area counts) in all samples, spikes, blanks, and standards to effectively check drifting method performance, poor injection execution, and anticipate the need for system inspection and/or maintenance.

7.5.11 If the initial analysis of the sample or a dilution of the sample has a concentration of any analyte that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. Secondary ion quantitation is allowed only when there are sample interferences with the primary ion.

7.5.11.1 When ions from a compound in the sample saturate the detector, this analysis must be followed by the analysis of an organic-free reagent water blank. If the blank analysis is not free of interferences, then the system must be decontaminated. Sample analysis may not resume until the blank analysis is demonstrated to be free of interferences.

7.5.11.2 All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

7.5.12 The use of selected ion monitoring (SIM) is acceptable in situations requiring detection limits below the normal range of full EI spectra. However, SIM may provide a lesser degree of confidence in the compound identification unless multiple ions are monitored for each compound.

7.6 Qualitative analysis

7.6.1 The qualitative identification of each compound determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met.

7.6.1.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

7.6.1.2 The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

7.6.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

7.6.1.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

7.6.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

7.6.1.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

7.6.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Use the following guidelines for making tentative identifications:

(1) Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

(2) The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

(3) Molecular ions present in the reference spectrum should be present in the sample spectrum.

(4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

(5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

7.7 Quantitative analysis

7.7.1 Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. The internal standard used shall be the one nearest the retention time of that of a given analyte.

7.7.2 If the RSD of a compound's response factors is 15% or less, then the concentration in the extract may be determined using the average response factor ($\overline{RF}$) from initial calibration data (7.3.6). See Method 8000, Sec. 7.0, for the equations describing internal standard calibration and either linear or non-linear calibrations.

7.7.3 Where applicable, the concentration of any non-target analytes identified in the sample (Sec. 7.6.2) should be estimated. The same formulae should be used with the following modifications: The areas A_x and A_{is} should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1.

7.7.4 The resulting concentration should be reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One and Method 8000 for specific quality control (QC) procedures. Quality control procedures to ensure the proper operation of the various sample preparation and/or sample introduction techniques can be found in Methods 3500 and 5000. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated.

8.2 Quality control procedures necessary to evaluate the GC system operation are found in Method 8000, Sec. 7.0 and include evaluation of retention time windows, calibration verification and chromatographic analysis of samples. In addition, instrument QC requirements may be found in the following sections of Method 8260:

8.2.1 The GC/MS system must be tuned to meet the BFB specifications in Secs. 7.3.1 and 7.4.1.

8.2.2 There must be an initial calibration of the GC/MS system as described in Sec. 7.3.

8.2.3 The GC/MS system must meet the SPCC criteria described in Sec. 7.4.4 and the CCC criteria in Sec. 7.4.5, each 12 hours.

8.3 Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made. See Method 8000, Sec. 8.0 for information on how to accomplish this demonstration.

8.4 Sample Quality Control for Preparation and Analysis - The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank, matrix spike, a duplicate, and a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample.

8.4.1 Before processing any samples, the analyst should demonstrate, through the analysis of a method blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is analyzed or there is a change in reagents, a method blank should be analyzed as a safeguard against chronic laboratory contamination. The blanks should be carried through all stages of sample preparation and measurement.

8.4.2 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair.

8.4.3 A Laboratory Control Sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

8.4.4 See Method 8000, Sec. 8.0 for the details on carrying out sample quality control procedures for preparation and analysis.

8.5 Surrogate recoveries - The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 8000, Sec. 8.0 for information on evaluating surrogate data and developing and updating surrogate limits.

8.6 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the calibration verification standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g., the column changed), recalibration of the system must take place.

8.7 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9.0 METHOD PERFORMANCE

9.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

9.2 This method has been tested using purge-and-trap (Method 5030) in a single laboratory using spiked water. Using a wide-bore capillary column, water was spiked at concentrations between 0.5 and 10 µg/L. Single laboratory accuracy and precision data are presented for the method analytes in Table 6. Calculated MDLs are presented in Table 1.

9.3 The method was tested using purge-and-trap (Method 5030) with water spiked at 0.1 to 0.5 µg/L and analyzed on a cryofocussed narrow-bore column. The accuracy and precision data for these compounds are presented in Table 7. MDL values were also calculated from these data and are presented in Table 2.

9.4 Direct injection (Method 3585) has been used for the analysis of waste motor oil samples using a wide-bore column. Single laboratory precision and accuracy data are presented in Tables 10 and 11 for TCLP volatiles in oil. The performance data were developed by spiking and analyzing seven replicates each of new and used oil. The oils were spiked at the TCLP regulatory concentrations for most analytes, except for the alcohols, ketones, ethyl acetate and chlorobenzene which are spiked at 5 ppm, well below the regulatory concentrations. Prior to spiking, the new oil (an SAE 30-weight motor oil) was heated at 80°C overnight to remove volatiles. The used oil (a mixture of used oil drained from passenger automobiles) was not heated and was contaminated with 20 - 300 ppm of BTEX compounds and isobutanol. These contaminants contributed to the extremely high recoveries of the BTEX compounds in the used oil. Therefore, the data from the deuterated analogs of these analytes represent more typical recovery values.

9.5 Single laboratory accuracy and precision data were obtained for the Method 5035 analytes in three soil matrices: sand; a soil collected 10 feet below the surface of a hazardous landfill, called C-Horizon; and a surface garden soil. Sample preparation was by Method 5035. Each sample was fortified with the analytes at a concentration of 4 µg/kg. These data are listed in Tables 17, 18, and 19. All data were calculated using fluorobenzene as the internal standard added to the soil sample prior to extraction. This causes some of the results to be greater than 100% recovery because the precision of results is sometimes as great as 28%.

9.5.1 In general, the recoveries of the analytes from the sand matrix are the highest, the C-Horizon soil results are somewhat less, and the surface garden soil recoveries are the lowest. This is due to the greater adsorptive capacity of the garden soil. This illustrates the necessity of analyzing matrix spike samples to assess the degree of matrix effects.

9.5.2 The recoveries of some of the gases, or very volatile compounds, such as vinyl chloride, trichlorofluoromethane, and 1,1-dichloroethene, are somewhat greater than 100%. This is due to the difficulty encountered in fortifying the soil with these compounds, allowing an equilibration period, then extracting them with a high degree of precision. Also, the garden soil results in Table 19 include some extraordinarily high recoveries for some aromatic compounds, such as toluene, xylenes, and trimethylbenzenes. This is due to contamination of the soil prior to sample collection, and to the fact that no background was subtracted.

9.6 Performance data for nonpurgeable volatiles using azeotropic distillation (Method 5031) are included in Tables 12 to 16.

9.7 Performance data for volatiles prepared using vacuum distillation (Method 5032) in soil, water, oil and fish tissue matrices are included in Tables 20 to 27.

9.8 Single laboratory accuracy and precision data were obtained for the Method 5021 analytes in two soil matrices: sand and a surface garden soil. Replicate samples were fortified with the analytes at concentrations of 10 µg/kg. These data are listed in Table 30. All data were calculated using the internal standards listed for each analyte in Table 28. The recommended internal standards were selected because they generated the best accuracy and precision data for the analyte in both types of soil.

9.8.1 If a detector other than an MS is used for analysis, consideration must be given to the choice of internal standards and surrogates. They must not coelute with any other analyte and must have similar properties to the analytes. The recoveries of the analytes are 50% or higher for each matrix studied. The recoveries of the gases or very volatile compounds are greater than 100% in some cases. Also, results include high recoveries of some aromatic compounds, such as toluene, xylenes, and trimethylbenzenes. This is due to contamination of the soil prior to sample collection.

9.8.2 The method detection limits using Method 5021 listed in Table 29 were calculated from results of seven replicate analyses of the sand matrix. Sand was chosen because it demonstrated the least degree of matrix effect of the soils studied. These MDLs were calculated utilizing the procedure described in Chapter One and are intended to be a general indication of the capabilities of the method.

9.9 The MDL concentrations listed in Table 31 were determined using Method 5041 in conjunction with Method 8260. They were obtained using cleaned blank VOST tubes and reagent water. Similar results have been achieved with field samples. The MDL actually achieved in a given analysis will vary depending upon instrument sensitivity and the effects of the matrix. Preliminary spiking studies indicate that under the test conditions, the MDLs for spiked compounds in extremely complex matrices may be larger by a factor of 500 - 1000.

9.10 The EQL of sample taken by Method 0040 and analyzed by Method 8260 is estimated to be in the range of 0.03 to 0.9 ppm (See Table 33). Matrix effects may cause the individual compound detection limits to be higher.

10.0 REFERENCES

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TABLE 1

CHROMATOGRAPHIC RETENTION TIMES AND METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC COMPOUNDS ON WIDE-BORE CAPILLARY COLUMNS

Compound	Retention Time (minutes)			MDL[d]
	Column 1[a]	Column 2[b]	Column 2'[c]	(µg/L)
Dichlorodifluoromethane	1.35	0.70	3.13	0.10
Chloromethane	1.49	0.73	3.40	0.13
Vinyl Chloride	1.56	0.79	3.93	0.17
Bromomethane	2.19	0.96	4.80	0.11
Chloroethane	2.21	1.02	--	0.10
Trichlorofluoromethane	2.42	1.19	6.20	0.08
Acrolein	3.19			
Iodomethane	3.56			
Acetonitrile	4.11			
Carbon disulfide	4.11			
Allyl chloride	4.11			
Methylene chloride	4.40	2.06	9.27	0.03
1,1-Dichloroethene	4.57	1.57	7.83	0.12
Acetone	4.57			
trans-1,2-Dichloroethene	4.57	2.36	9.90	0.06
Acrylonitrile	5.00			
1,1-Dichloroethane	6.14	2.93	10.80	0.04
Vinyl acetate	6.43			
2,2-Dichloropropane	8.10	3.80	11.87	0.35
2-Butanone	--			
cis-1,2-Dichloroethene	8.25	3.90	11.93	0.12
Propionitrile	8.51			
Chloroform	9.01	4.80	12.60	0.03
Bromochloromethane	--	4.38	12.37	0.04
Methacrylonitrile	9.19			
1,1,1-Trichloroethane	10.18	4.84	12.83	0.08
Carbon tetrachloride	11.02	5.26	13.17	0.21
1,1-Dichloropropene	--	5.29	13.10	0.10
Benzene	11.50	5.67	13.50	0.04
1,2-Dichloroethane	12.09	5.83	13.63	0.06
Trichloroethene	14.03	7.27	14.80	0.19
1,2-Dichloropropane	14.51	7.66	15.20	0.04
Bromodichloromethane	15.39	8.49	15.80	0.08
Dibromomethane	15.43	7.93	5.43	0.24
Methyl methacrylate	15.50			
1,4-Dioxane	16.17			
2-Chloroethyl vinyl ether	--			
4-Methyl-2-pentanone	17.32			
trans-1,3-Dichloropropene	17.47	--	16.70	--
Toluene	18.29	10.00	17.40	0.11
cis-1,3-Dichloropropene	19.38	--	17.90	--

TABLE 1 (cont.)

Compound	Retention Time (minutes)			MDL[d]
	Column 1[a]	Column 2[b]	Column 2"[c]	(µg/L)
1,1,2-Trichloroethane	19.59	11.05	18.30	0.10
Ethyl methacrylate	20.01			
2-Hexanone	20.30			
Tetrachloroethene	20.26	11.15	18.60	0.14
1,3-Dichloropropane	20.51	11.31	18.70	0.04
Dibromochloromethane	21.19	11.85	19.20	0.05
1,2-Dibromoethane	21.52	11.83	19.40	0.06
1-Chlorohexane	--	13.29	--	0.05
Chlorobenzene	23.17	13.01	20.67	0.04
1,1,1,2-Tetrachloroethane	23.36	13.33	20.87	0.05
Ethylbenzene	23.38	13.39	21.00	0.06
p-Xylene	23.54	13.69	21.30	0.13
m-Xylene	23.54	13.68	21.37	0.05
o-Xylene	25.16	14.52	22.27	0.11
Styrene	25.30	14.60	22.40	0.04
Bromoform	26.23	14.88	22.77	0.12
Isopropylbenzene (Cumene)	26.37	15.46	23.30	0.15
cis-1,4-Dichloro-2-butene	27.12			
1,1,2,2-Tetrachloroethane	27.29	16.35	24.07	0.04
Bromobenzene	27.46	15.86	24.00	0.03
1,2,3-Trichloropropane	27.55	16.23	24.13	0.32
n-Propylbenzene	27.58	16.41	24.33	0.04
2-Chlorotoluene	28.19	16.42	24.53	0.04
trans-1,4-Dichloro-2-butene	28.26			
1,3,5-Trimethylbenzene	28.31	16.90	24.83	0.05
4-Chlorotoluene	28.33	16.72	24.77	0.06
Pentachloroethane	29.41			
1,2,4-Trimethylbenzene	29.47	17.70	31.50	0.13
sec-Butylbenzene	30.25	18.09	26.13	0.13
tert-Butylbenzene	30.59	17.57	26.60	0.14
p-Isopropyltoluene	30.59	18.52	26.50	0.12
1,3-Dichlorobenzene	30.56	18.14	26.37	0.12
1,4-Dichlorobenzene	31.22	18.39	26.60	0.03
Benzyl chloride	32.00			
n-Butylbenzene	32.23	19.49	27.32	0.11
1,2-Dichlorobenzene	32.31	19.17	27.43	0.03
1,2-Dibromo-3-chloropropane	35.30	21.08	--	0.26
1,2,4-Trichlorobenzene	38.19	23.08	31.50	0.04
Hexachlorobutadiene	38.57	23.68	32.07	0.11
Naphthalene	39.05	23.52	32.20	0.04
1,2,3-Trichlorobenzene	40.01	24.18	32.97	0.03

TABLE 1 (cont.)

Compound	Retention Time (minutes) Column 1[a]	Column 2[b]	Column 2"[c]	MDL[d] (µg/L)
INTERNAL STANDARDS/SURROGATES				
1,4-Difluorobenzene	13.26			
Chlorobenzene-d_5	23.10			
1,4-Dichlorobenzene-d_4	31.16			
4-Bromofluorobenzene	27.83	15.71	23.63	
1,2-Dichlorobenzene-d_4	32.30	19.08	27.25	
Dichloroethane-d_4	12.08			
Dibromofluoromethane	--			
Toluene-d_8	18.27			
Pentafluorobenzene	--			
Fluorobenzene	13.00	6.27	14.06	

[a] Column 1 - 60 meter x 0.75 mm ID VOCOL capillary. Hold at 10°C for 8 minutes, then program to 180°C at 4°C/min.

[b] Column 2 - 30 meter x 0.53 mm ID DB-624 wide-bore capillary using cryogenic oven. Hold at 10°C for 5 minutes, then program to 160°C at 6°C/min.

[c] Column 2" - 30 meter x 0.53 mm ID DB-624 wide-bore capillary, cooling GC oven to ambient temperatures. Hold at 10°C for 6 minutes, program to 70°C at 10 °C/min, program to 120°C at 5°C/min, then program to 180°C at 8°C/min.

[d] MDL based on a 25-mL sample volume.

TABLE 2

CHROMATOGRAPHIC RETENTION TIMES AND METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC COMPOUNDS ON NARROW-BORE CAPILLARY COLUMNS

Compound	Retention Time (minutes) Column 3[a]	MDL[b] (µg/L)
Dichlorodifluoromethane	0.88	0.11
Chloromethane	0.97	0.05
Vinyl chloride	1.04	0.04
Bromomethane	1.29	0.03
1,1-Dichloroethane	4.03	0.03
cis-1,2-Dichloroethene	5.07	0.06
2,2-Dichloropropane	5.31	0.08
Chloroform	5.55	0.04
Bromochloromethane	5.63	0.09
1,1,1-Trichloroethane	6.76	0.04
1,2-Dichloroethane	7.00	0.02
1,1-Dichloropropene	7.16	0.12
Carbon tetrachloride	7.41	0.02
Benzene	7.41	0.03
1,2-Dichloropropane	8.94	0.02
Trichloroethene	9.02	0.02
Dibromomethane	9.09	0.01
Bromodichloromethane	9.34	0.03
Toluene	11.51	0.08
1,1,2-Trichloroethane	11.99	0.08
1,3-Dichloropropane	12.48	0.08
Dibromochloromethane	12.80	0.07
Tetrachloroethene	13.20	0.05
1,2-Dibromoethane	13.60	0.10
Chlorobenzene	14.33	0.03
1,1,1,2-Tetrachloroethane	14.73	0.07
Ethylbenzene	14.73	0.03
p-Xylene	15.30	0.06
m-Xylene	15.30	0.03
Bromoform	15.70	0.20
o-Xylene	15.78	0.06
Styrene	15.78	0.27
1,1,2,2-Tetrachloroethane	15.78	0.20
1,2,3-Trichloropropane	16.26	0.09
Isopropylbenzene	16.42	0.10
Bromobenzene	16.42	0.11
2-Chlorotoluene	16.74	0.08
n-Propylbenzene	16.82	0.10
4-Chlorotoluene	16.82	0.06

TABLE 2 (cont.)

Compound	Retention Time (minutes) Column 3[a]	MDL[b] (µg/L)
1,3,5-Trimethylbenzene	16.99	0.06
tert-Butylbenzene	17.31	0.33
1,2,4-Trimethylbenzene	17.31	0.09
sec-Butylbenzene	17.47	0.12
1,3-Dichlorobenzene	17.47	0.05
p-Isopropyltoluene	17.63	0.26
1,4-Dichlorobenzene	17.63	0.04
1,2-Dichlorobenzene	17.79	0.05
n-Butylbenzene	17.95	0.10
1,2-Dibromo-3-chloropropane	18.03	0.50
1,2,4-Trichlorobenzene	18.84	0.20
Naphthalene	19.07	0.10
Hexachlorobutadiene	19.24	0.10
1,2,3-Trichlorobenzene	19.24	0.14

[a] Column 3 - 30 meter x 0.32 mm ID DB-5 capillary with 1 µm film thickness.

[b] MDL based on a 25-mL sample volume.

TABLE 3

ESTIMATED QUANTITATION LIMITS FOR VOLATILE ANALYTES[a]

Estimated Quantitation Limits		
5-mL Ground Water Purge (µg/L)	25-mL Ground water Purge (µg/L)	Low Soil/Sediment[b] µg/kg
5	1	5

[a] Estimated Quantitation Limit (EQL) - The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL is generally 5 to 10 times the MDL. However, it may be nominally chosen within these guidelines to simplify data reporting. For many analytes the EQL analyte concentration is selected for the lowest non-zero standard in the calibration curve. Sample EQLs are highly matrix-dependent. The EQLs listed herein are provided for guidance and may not always be achievable. See the following footnote for further guidance on matrix-dependent EQLs.

[b] EQLs listed for soil/sediment are based on wet weight. Normally data are reported on a dry weight basis; therefore, EQLs will be higher, based on the percent dry weight in each sample.

Other Matrices	Factor[c]
Water miscible liquid waste	50
High concentration soil and sludge	125
Non-water miscible waste	500

[c] EQL = [EQL for low soil sediment (Table 3)] x [Factor].

For non-aqueous samples, the factor is on a wet-weight basis.

TABLE 4

BFB (4-BROMOFLUOROBENZENE) MASS INTENSITY CRITERIA[a]

m/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to 9% of m/z 176

[a] Alternate tuning criteria may be used, (e.g. CLP, Method 524.2, or manufacturers" instructions), provided that method performance is not adversely affected.

TABLE 5

CHARACTERISTIC MASSES (m/z) FOR PURGEABLE ORGANIC COMPOUNDS

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Acetone	58	43
Acetonitrile	41	40, 39
Acrolein	56	55, 58
Acrylonitrile	53	52, 51
Allyl alcohol	57	58, 39
Allyl chloride	76	41, 39, 78
Benzene	78	-
Benzyl chloride	91	126, 65, 128
Bromoacetone	136	43, 138, 93, 95
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
iso-Butanol	74	43
n-Butanol	56	41
2-Butanone	72	43
n-Butylbenzene	91	92, 134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91, 134
Carbon disulfide	76	78
Carbon tetrachloride	117	119
Chloral hydrate	82	44, 84, 86, 111
Chloroacetonitrile	48	75
Chlorobenzene	112	77, 114
1-Chlorobutane	56	49
Chlorodibromomethane	129	208, 206
Chloroethane	64 (49*)	66 (51*)
2-Chloroethanol	49	44, 43, 51, 80
Bis(2-chloroethyl) sulfide	109	111, 158, 160
2-Chloroethyl vinyl ether	63	65, 106
Chloroform	83	85
Chloromethane	50 (49*)	52 (51*)
Chloroprene	53	88, 90, 51
3-Chloropropionitrile	54	49, 89, 91
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
1,2-Dibromo-3-chloropropane	75	155, 157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
1,2-Dichlorobenzene	146	111, 148
1,2-Dichlorobenzene-d_4	152	115, 150
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
cis-1,4-Dichloro-2-butene	75	53, 77, 124, 89
trans-1,4-Dichloro-2-butene	53	88, 75
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	96	61, 98
trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,3-Dichloro-2-propanol	79	43, 81, 49
1,1-Dichloropropene	75	110, 77
cis-1,3-Dichloropropene	75	77, 39
trans-1,3-Dichloropropene	75	77, 39
1,2,3,4-Diepoxybutane	55	57, 56
Diethyl ether	74	45, 59
1,4-Dioxane	88	58, 43, 57
Epichlorohydrin	57	49, 62, 51
Ethanol	31	45, 27, 46
Ethyl acetate	88	43, 45, 61
Ethylbenzene	91	106
Ethylene oxide	44	43, 42
Ethyl methacrylate	69	41, 99, 86, 114
Hexachlorobutadiene	225	223, 227
Hexachloroethane	201	166, 199, 203
2-Hexanone	43	58, 57, 100
2-Hydroxypropionitrile	44	43, 42, 53
Iodomethane	142	127, 141
Isobutyl alcohol	43	41, 42, 74
Isopropylbenzene	105	120
p-Isopropyltoluene	119	134, 91
Malononitrile	66	39, 65, 38
Methacrylonitrile	41	67, 39, 52, 66
Methyl acrylate	55	85
Methyl-t-butyl ether	73	57
Methylene chloride	84	86, 49
Methyl ethyl ketone	72	43
Methyl iodide	142	127, 141

TABLE 5 (cont.)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Methyl methacrylate	69	41, 100, 39
4-Methyl-2-pentanone	100	43, 58, 85
Naphthalene	128	-
Nitrobenzene	123	51, 77
2-Nitropropane	46	-
2-Picoline	93	66, 92, 78
Pentachloroethane	167	130, 132, 165, 169
Propargyl alcohol	55	39, 38, 53
β-Propiolactone	42	43, 44
Propionitrile (ethyl cyanide)	54	52, 55, 40
n-Propylamine	59	41, 39
n-Propylbenzene	91	120
Pyridine	79	52
Styrene	104	78
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	164	129, 131, 166
Toluene	92	91
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	97, 130, 132
Trichlorofluoromethane	151	101, 153
1,2,3-Trichloropropane	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl acetate	43	86
Vinyl chloride	62	64
o-Xylene	106	91
m-Xylene	106	91
p-Xylene	106	91
Internal Standards/Surrogates:		
Benzene-d_6	84	83
Bromobenzene-d_5	82	162
Bromochloromethane-d_2	51	131
1,4-Difluorobenzene	114	
Chlorobenzene-d_5	117	
1,4-Dichlorobenzene-d_4	152	115, 150
1,1,2-Trichloroethane-d_3	100	
4-Bromofluorobenzene	95	174, 176
Chloroform-d_1	84	
Dibromofluoromethane	113	

TABLE 5 (cont.)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Internal Standards/Surrogates		
Dichloroethane-d_4	102	
Toluene-d_8	98	
Pentafluorobenzene	168	
Fluorobenzene	96	77

* Characteristic ion for an ion trap mass spectrometer (to be used when ion-molecule reactions are observed).

TABLE 6

SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR PURGEABLE VOLATILE ORGANIC COMPOUNDS IN WATER DETERMINED WITH A WIDE-BORE CAPILLARY COLUMN (METHOD 5030)

Compound	Conc. Range (µg/L)	Number of Samples	% Recovery[a]	Standard Deviation of Recovery[b]	RSD
Benzene	0.1 - 10	31	97	6.5	5.7
Bromobenzene	0.1 - 10	30	100	5.5	5.5
Bromochloromethane	0.5 - 10	24	90	5.7	6.4
Bromodichloromethane	0.1 - 10	30	95	5.7	6.1
Bromoform	0.5 - 10	18	101	6.4	6.3
Bromomethane	0.5 - 10	18	95	7.8	8.2
n-Butylbenzene	0.5 - 10	18	100	7.6	7.6
sec-Butylbenzene	0.5 - 10	16	100	7.6	7.6
tert-Butylbenzene	0.5 - 10	18	102	7.4	7.3
Carbon tetrachloride	0.5 - 10	24	84	7.4	8.8
Chlorobenzene	0.1 - 10	31	98	5.8	5.9
Chloroethane	0.5 - 10	24	89	8.0	9.0
Chloroform	0.5 - 10	24	90	5.5	6.1
Chloromethane	0.5 - 10	23	93	8.3	8.9
2-Chlorotoluene	0.1 - 10	31	90	5.6	6.2
4-Chlorotoluene	0.1 - 10	31	99	8.2	8.3
1,2-Dibromo-3-Chloropropane	0.5 - 10	24	83	16.6	19.9
Dibromochloromethane	0.1 - 10	31	92	6.5	7.0
1,2-Dibromoethane	0.5 - 10	24	102	4.0	3.9
Dibromomethane	0.5 - 10	24	100	5.6	5.6
1,2-Dichlorobenzene	0.1 - 10	31	93	5.8	6.2
1,3-Dichlorobenzene	0.5 - 10	24	99	6.8	6.9
1,4-Dichlorobenzene	0.2 - 20	31	103	6.6	6.4
Dichlorodifluoromethane	0.5 - 10	18	90	6.9	7.7
1,1-Dichlorobenzene	0.5 - 10	24	96	5.1	5.3
1,2-Dichlorobenzene	0.1 - 10	31	95	5.1	5.4
1,1-Dichloroethene	0.1 - 10	34	94	6.3	6.7
cis-1,2-Dichloroethene	0.5 - 10	18	101	6.7	6.7
trans-1,2-Dichloroethene	0.1 - 10	30	93	5.2	5.6
1,2-Dichloropropane	0.1 - 10	30	97	5.9	6.1
1,3-Dichloropropane	0.1 - 10	31	96	5.7	6.0
2,2-Dichloropropane	0.5 - 10	12	86	14.6	16.9
1,1-Dichloropropene	0.5 - 10	18	98	8.7	8.9
Ethylbenzene	0.1 - 10	31	99	8.4	8.6
Hexachlorobutadiene	0.5 - 10	18	100	6.8	6.8
Isopropylbenzene	0.5 - 10	16	101	7.7	7.6
p-Isopropyltoluene	0.1 - 10	23	99	6.7	6.7
Methylene chloride	0.1 - 10	30	95	5.0	5.3

TABLE 6 (cont.)

Compound	Conc. Range (µg/L)	Number of Samples	% Recovery[a]	Standard Deviation of Recovery[b]	RSD
Naphthalene	0.1 -100	31	104	8.6	8.2
n-Propylbenzene	0.1 - 10	31	100	5.8	5.8
Styrene	0.1 -100	39	102	7.3	7.2
1,1,1,2-Tetrachloroethane	0.5 - 10	24	90	6.1	6.8
1,1,2,2-Tetrachloroethane	0.1 - 10	30	91	5.7	6.3
Tetrachloroethene	0.5 - 10	24	89	6.0	6.8
Toluene	0.5 - 10	18	102	8.1	8.0
1,2,3-Trichlorobenzene	0.5 - 10	18	109	9.4	8.6
1,2,4-Trichlorobenzene	0.5 - 10	18	108	9.0	8.3
1,1,1-Trichloroethane	0.5 - 10	18	98	7.9	8.1
1,1,2-Trichloroethane	0.5 - 10	18	104	7.6	7.3
Trichloroethene	0.5 - 10	24	90	6.5	7.3
Trichlorofluoromethane	0.5 - 10	24	89	7.2	8.1
1,2,3-Trichloropropane	0.5 - 10	16	108	15.6	14.4
1,2,4-Trimethylbenzene	0.5 - 10	18	99	8.0	8.1
1,3,5-Trimethylbenzene	0.5 - 10	23	92	6.8	7.4
Vinyl chloride	0.5 - 10	18	98	6.5	6.7
o-Xylene	0.1 - 31	18	103	7.4	7.2
m-Xylene	0.1 - 10	31	97	6.3	6.5
p-Xylene	0.5 - 10	18	104	8.0	7.7

[a] Recoveries were calculated using internal standard method. The internal standard was fluorobenzene.

[b] Standard deviation was calculated by pooling data from three concentrations.

TABLE 7

SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR PURGEABLE VOLATILE ORGANIC COMPOUNDS IN WATER DETERMINED WITH A NARROW-BORE CAPILLARY COLUMN (METHOD 5030)

Compound	Conc. (µg/L)	Number of Samples	% Recovery[a]	Standard Deviation of Recovery[b]	RSD
Benzene	0.1	7	99	6.2	6.3
Bromobenzene	0.5	7	97	7.4	7.6
Bromochloromethane	0.5	7	97	5.8	6.0
Bromodichloromethane	0.1	7	100	4.6	4.6
Bromoform	0.5	7	101	5.4	5.3
Bromomethane	0.5	7	99	7.1	7.2
n-Butylbenzene	0.5	7	94	6.0	6.4
sec-Butylbenzene	0.5	7	110	7.1	6.5
tert-Butylbenzene	0.5	7	110	2.5	2.3
Carbon tetrachloride	0.1	7	108	6.8	6.3
Chlorobenzene	0.1	7	91	5.8	6.4
Chloroethane	0.1	7	100	5.8	5.8
Chloroform	0.1	7	105	3.2	3.0
Chloromethane	0.5	7	101	4.7	4.7
2-Chlorotoluene	0.5	7	99	4.6	4.6
4-Chlorotoluene	0.5	7	96	7.0	7.3
1,2-Dibromo-3-chloropropane	0.5	7	92	10.0	10.9
Dibromochloromethane	0.1	7	99	5.6	5.7
1,2-Dibromoethane	0.5	7	97	5.6	5.8
Dibromomethane	0.5	7	93	5.6	6.0
1,2-Dichlorobenzene	0.1	7	97	3.5	3.6
1,3-Dichlorobenzene	0.1	7	101	6.0	5.9
1,4-Dichlorobenzene	0.1	7	106	6.5	6.1
Dichlorodifluoromethane	0.1	7	99	8.8	8.9
1,1-Dichloroethane	0.5	7	98	6.2	6.3
1,2-Dichloroethane	0.1	7	100	6.3	6.3
1,1-Dichloroethene	0.1	7	95	9.0	9.5
cis-1,2-Dichloroethene	0.1	7	100	3.5	3.7
trans-1,2-Dichloroethene	0.1	7	98	7.2	7.3
1,2-Dichloropropane	0.5	7	96	6.0	6.3
1,3-Dichloropropane	0.5	7	99	5.8	5.9
2,2-Dichloropropane	0.5	7	99	4.9	4.9
1,1-Dichloropropene	0.5	7	102	7.4	7.3
Ethylbenzene	0.5	7	99	5.2	5.3
Hexachlorobutadiene	0.5	7	100	6.7	6.7
Isopropylbenzene	0.5	7	102	6.4	6.3
p-Isopropyltoluene	0.5	7	113	13.0	11.5
Methylene chloride	0.5	7	97	13.0	13.4
Naphthalene	0.5	7	98	7.2	7.3

TABLE 7 (cont.)

Compound	Conc. (µg/L)	Number of Samples	% Recovery[a]	Standard Deviation of Recovery[b]	RSD
n-Propylbenzene	0.5	7	99	6.6	6.7
Styrene	0.5	7	96	19.0	19.8
1,1,1,2-Tetrachloroethane	0.5	7	100	4.7	4.7
1,1,2,2-Tetrachloroethane	0.5	7	100	12.0	12.0
Tetrachloroethene	0.1	7	96	5.0	5.2
Toluene	0.5	7	100	5.9	5.9
1,2,3-Trichlorobenzene	0.5	7	102	8.9	8.7
1,2,4-Trichlorobenzene	0.5	7	91	16.0	17.6
1,1,1-Trichloroethane	0.5	7	100	4.0	4.0
1,1,2-Trichloroethane	0.5	7	102	4.9	4.8
Trichloroethene	0.1	7	104	2.0	1.9
Trichlorofluoromethane	0.1	7	97	4.6	4.7
1,2,3-Trichloropropane	0.5	7	96	6.5	6.8
1,2,4-Trimethylbenzene	0.5	7	96	6.5	6.8
1,3,5-Trimethylbenzene	0.5	7	101	4.2	4.2
Vinyl chloride	0.1	7	104	0.2	0.2
o-Xylene	0.5	7	106	7.5	7.1
m-Xylene	0.5	7	106	4.6	4.3
p-Xylene	0.5	7	97	6.1	6.3

[a] Recoveries were calculated using internal standard method. Internal standard was fluorobenzene.

TABLE 8

SURROGATE SPIKE RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES

Surrogate Compound	Water	Soil/Sediment
4-Bromofluorobenzene[a]	86-115	74-121
Dibromofluoromethane[a]	86-118	80-120
Toluene-d_8[a]	88-110	81-117
Dichloroethane-d_4[a]	80-120	80-120

[a] Single laboratory data, for guidance only.

TABLE 9

QUANTITY OF EXTRACT REQUIRED FOR ANALYSIS OF HIGH CONCENTRATION SAMPLES

Approximate Concentration Range (µg/kg)	Volume of Extract[a]
500 - 10,000	100 µL
1,000 - 20,000	50 µL
5,000 - 100,000	10 µL
25,000 - 500,000	100 µL of 1/50 dilution[b]

Calculate appropriate dilution factor for concentrations exceeding this table.

[a] The volume of solvent added to 5 mL of water being purged should be kept constant. Therefore, add to the 5-mL syringe whatever volume of solvent is necessary to maintain a volume of 100 µL added to the syringe.

[b] Dilute an aliquot of the solvent extract and then take 100 µL for analysis.

TABLE 10

DIRECT INJECTION ANALYSIS OF NEW OIL AT 5 PPM (METHOD 3585)

Compound	Recovery (%)	%RSD	Blank (ppm)	Spike (ppm)
Acetone	91	14.8	1.9	5.0
Benzene	86	21.3	0.1	0.5
n-Butanol*,**	107	27.8	0.5	5.0
iso-Butanol*,**	95	19.5	0.9	5.0
Carbon tetrachloride	86	44.7	0.0	0.5
Carbon disulfide**	53	22.3	0.0	5.0
Chlorobenzene	81	29.3	0.0	5.0
Chloroform	84	29.3	0.0	6.0
1,4-Dichlorobenzene	98	24.9	0.0	7.5
1,2-Dichloroethane	101	23.1	0.0	0.5
1,1-Dichloroethene	97	45.3	0.0	0.7
Diethyl ether	76	24.3	0.0	5.0
Ethyl acetate	113	27.4	0.0	5.0
Ethylbenzene	83	30.1	0.2	5.0
Hexachloroethane	71	30.3	0.0	3.0
Methylene chloride	98	45.3	0.0	5.0
Methyl ethyl ketone	79	24.6	0.4	5.0
MIBK	93	31.4	0.0	5.0
Nitrobenzene	89	30.3	0.0	2.0
Pyridine	31	35.9	0.0	5.0
Tetrachloroethene	82	27.1	0.0	0.7
Trichlorofluoromethane	76	27.6	0.0	5.0
1,1,2-Trichlorotrifluoroethane	69	29.2	0.0	5.0
Toluene	73	21.9	0.6	5.0
Trichloroethene	66	28.0	0.0	0.5
Vinyl chloride	63	35.2	0.0	0.2
o-Xylene	83	29.5	0.4	5.0
m/p-Xylene	84	29.5	0.6	10.0

* Alternate mass employed
** IS quantitation

Data are taken from Reference 9.

TABLE 11

SINGLE LABORATORY PERFORMANCE DATA FOR THE DIRECT INJECTION METHOD - USED OIL (METHOD 3585)

Compound	Recovery (%)	%RSD	Blank (ppm)	Spike (ppm)
Acetone**	105	54	2.0	5.0
Benzene	3135	44	14	0.5
Benzene-d_6	56	44	2.9	0.5
n-Butanol**	100	71	12	5.0
iso-Butanol*,**	132	27	0	5.0
Carbon tetrachloride	143	68	0	0.5
Carbon tetrachloride-^{13}C	99	44	5.1	0.5
Carbon disulfide**	95	63	0	5.0
Chlorobenzene	148	71	0	5.0
Chlorobenzene-d_5	60	44	3.6	5.0
Chloroform	149	74	0	6.0
Chloroform-d_1	51	44	2.6	6.0
1,4-Dichlorobenzene	142	72	0	7.5
1,4-Dichlorobenzene-d_4	53	44	3.4	7.5
1,2-Dichloroethane**	191	54	0	0.5
1,1-Dichloroethene*	155	51	0	0.7
1,1-Dichloroethene-d_2	68	44	3.4	0.7
Diethyl ether**	95	66	0	5.0
Ethyl acetate*,**	126	39	0	5.0
Ethylbenzene	1298	44	54	5.0
Ethylbenzene-d_{10}	63	44	3.6	5.0
Hexachloroethane	132	72	0	3.0
Hexachloroethane-^{13}C	54	45	3.5	3.0
Methylene chloride**	86	65	0.3	5.0
Methyl ethyl ketone**	107	64	0	5.0
4-Methyl-2-pentanone (MIBK)**	100	74	0.1	5.0
Nitrobenzene	111	80	0	2.0
Nitrobenzene-d_5	65	53	4.0	2.0
Pyridine**	68	85	0	5.0
Pyridine-d_5	ND	--	0	5.0
Tetrachloroethene**	101	73	0	0.7
Trichlorofluoromethane**	91	70	0	5.0
1,1,2-Cl_3F_3ethane**	81	70	0	5.0
Toluene	2881	44	128	5.0
Toluene-d_8	63	44	3.6	5.0
Trichloroethene	152	57	0	0.5
Trichloroethene-d_1	55	44	2.8	0.5

TABLE 11 (cont.)

Compound	Recovery (%)	%RSD	Blank (ppm)	Spike (ppm)
Vinyl chloride**	100	69	0	0.2
o-Xylene	2292	44	105	5.0
o-Xylene-d_{10}	76	44	4.2	5.0
m-/p-Xylene	2583	44	253	10.0
p-Xylene-d_{10}	67	44	3.7	10.0

* Alternate mass employed
** IS quantitation
ND = Not Detected

Data are based on seven measurements and are taken from Reference 9.

TABLE 12

METHOD DETECTION LIMITS (METHOD 5031)

	MDL (µg/L)	Concentration Factor	
Compound	Macro[a]	Macro	Micro
Acetone	31	25-500	-
Acetonitrile	57	25-500	200
Acrolein	-	-	100
Acrylonitrile	16	25-500	100
Allyl Alcohol	7	25-500	-
1-Butanol	-	-	250
Crotonaldehyde	12	25-500	-
1,4-Dioxane	12	25-500	150
Ethyl Acetate	-	-	100
Isobutyl alcohol	7	25-500	-
Methanol	38	25-500	140
Methyl Ethyl Ketone	16	25-500	-
2-Methyl-1-propanol	-	-	250
n-Nitroso-di-n-butylamine	14	25-500	-
Paraldehyde	10	25-500	-
2-Picoline	7	25-500	-
1-Propanol	-	-	240
Propionitrile	11	25-500	200
Pyridine	4	25-500	-
o-Toluidine	13	25-500	-

[a] Produced by analysis of seven aliquots of reagent water spiked at 25 ppb at the listed compounds; calculations based on internal standard technique and use of the following equation:

MDL = 3.134 x Std. Dev. of low concentration spike (ppb).

[b] When a 40-mL sample is used, and the first 100 µL of distillate are collected.

TABLE 13

TARGET COMPOUNDS, SURROGATES, AND INTERNAL STANDARDS (METHOD 5031)

Target Compound	Surrogate	Internal Standard
Acetone	d_6-Acetone	d_8-Isopropyl alcohol
Acetonitrile	d_3-Acetonitrile	d_8-Isopropyl alcohol
Acrylonitrile	d_8-Isopropyl alcohol	
Allyl alcohol	d_7-Dimethyl formamide	
Crotonaldehyde	d_8-Isopropyl alcohol	
1,4-Dioxane	d_8-1,4-Dioxane	d_7-Dimethyl formamide
Isobutyl alcohol	d_7-Dimethyl formamide	
Methanol	d_3-Methanol	d_8-Isopropyl alcohol
Methyl ethyl ketone	d_8-Isopropyl alcohol	
N-Nitroso-di-n-butylamine	d_7-Dimethyl formamide	
Paraldehyde	d_7-Dimethyl formamide	
2-Picoline	d_7-Dimethyl formamide	
Propionitrile	d_8-Isopropyl alcohol	
Pyridine	d_5-Pyridine	d_7-Dimethyl formamide
o-Toluidine	d_7-Dimethyl formamide	

TABLE 14

RECOMMENDED CONCENTRATIONS FOR CALIBRATION SOLUTIONS (METHOD 5031)

Compound	Concentration(s) (ng/µL)
Internal Standards	
d_5-benzyl alcohol	10.0
d_{14}-Diglyme	10.0
d_7-Dimethyl formamide	10.0
d_8-Isopropyl alcohol	10.0
Surrogates	
d_6-Acetone	10.0
d_3-Acetonitrile	10.0
d_8-1,4-Dioxane	10.0
d_3-Methanol	10.0
d_5-Pyridine	10.0
Target Compounds	
Acetone	1.0, 5.0, 10.0, 25.0, 100.0
Acetonitrile	1.0, 5.0, 10.0, 25.0, 100.0
Acrylonitrile	1.0, 5.0, 10.0, 25.0, 100.0
Allyl alcohol	1.0, 5.0, 10.0, 25.0, 100.0
Crotonaldehyde	1.0, 5.0, 10.0, 25.0, 100.0
1,4-Dioxane	1.0, 5.0, 10.0, 25.0, 100.0
Isobutyl alcohol	1.0, 5.0, 10.0, 25.0, 100.0
Methanol	1.0, 5.0, 10.0, 25.0, 100.0
Methyl ethyl ketone	1.0, 5.0, 10.0, 25.0, 100.0
N-Nitroso-di-n-butylamine	1.0, 5.0, 10.0, 25.0, 100.0
Paraldehyde	1.0, 5.0, 10.0, 25.0, 100.0
2-Picoline	1.0, 5.0, 10.0, 25.0, 100.0
Propionitrile	1.0, 5.0, 10.0, 25.0, 100.0
Pyridine	1.0, 5.0, 10.0, 25.0, 100.0
o-Toluidine	1.0, 5.0, 10.0, 25.0, 100.0

TABLE 15

CHARACTERISTIC IONS AND RETENTION TIMES FOR VOCs (METHOD 5031)

Compound	Quantitation Ion[a]	Secondary Ions	Retention Time (min)[b]
Internal Standards			
d_8-Isopropyl alcohol	49		1.75
d_{14}-Diglyme	66	98,64	9.07
d_7-Dimethyl formamide	50	80	9.20
Surrogates			
d_6-Acetone	46	64,42	1.03
d_3-Methanol	33	35,30	1.75
d_3-Acetonitrile	44	42	2.63
d_8-1,4-Dioxane	96	64,34	3.97
d_5-Pyridine	84	56,79	6.73
d_5-Phenol[c]	99	71	15.43
Target Compounds			
Acetone	43	58	1.05
Methanol	31	29	1.52
Methyl ethyl ketone	43	72,57	1.53
Methacrylonitrile[c]	67	41	2.38
Acrylonitrile	53	52,51	2.53
Acetonitrile	41	40,39	2.73
Methyl isobutyl ketone[c]	85	100,58	2.78
Propionitrile	54	52,55	3.13
Crotonaldehyde	41	70	3.43
1,4-Dioxane	58	88,57	4.00
Paraldehyde	45	89	4.75
Isobutyl alcohol	43	33,42	5.05
Allyl alcohol	57	39	5.63
Pyridine	79	50,52	6.70
2-Picoline	93	66	7.27
N-Nitroso-di-n-butylamine	84	116	12.82
Aniline[c]	93	66,92	13.23
o-Toluidine	106	107	13.68
Phenol[c]	94	66,65	15.43

[a] These ions were used for quantitation in selected ion monitoring.

[b] GC column: DB-Wax, 30 meter x 0.53 mm, 1 µm film thickness.
Oven program: 45°C for 4 min, increased to 220°C at 12°C/min.

[c] Compound removed from target analyte list due to poor accuracy and precision.

TABLE 16

METHOD ACCURACY AND PRECISION BY MEAN PERCENT RECOVERY AND PERCENT RELATIVE STANDARD DEVIATION[a] (METHOD 5031 - MACRODISTILLATION TECHNIQUE)

(Single Laboratory and Single Operator)

Compound	25 ppb Spike		100 ppb Spike		500 ppb Spike	
	Mean %R	%RSD	Mean %R	%RSD	Mean %R	%RSD
d_6-Acetone	66	24	69	14	65	16
d_3-Acetonitrile	89	18	80	18	70	10
d_8-1,4-Dioxane	56	34	58	11	61	18
d_3-Methanol	43	29	48	19	56	14
d_5-Pyridine	83	6.3	84	7.8	85	9.0
Acetone	67	45	63	14	60	14
Acetonitrile	44	35	52	15	56	15
Acrylonitrile	49	42	47	27	45	27
Allyl alcohol	69	13	70	9.7	73	10
Crotonaldehyde	68	22	68	13	69	13
1,4-Dioxane	63	25	55	16	54	13
Isobutyl alcohol	66	14	66	5.7	65	7.9
Methanol	50	36	46	22	49	18
Methyl ethyl ketone	55	37	56	20	52	19
N-Nitroso-di-n-butylamine	57	21	61	15	72	18
Paraldehyde	65	20	66	11	60	8.9
Picoline	81	12	81	6.8	84	8.0
Propionitrile	67	22	69	13	68	13
Pyridine	74	7.4	72	6.7	74	7.3
o-Toluidine	52	31	54	15	58	12

[a] Data from analysis of seven aliquots of reagent water spiked at each concentration, using a quadrapole mass spectrometer in the selected ion monitoring mode.

TABLE 17

RECOVERIES IN SAND SAMPLES FORTIFIED AT 4 µg/kg (ANALYSIS BY METHOD 5035)

	Recovery per Replicate (ng)							Mean
Compound	1	2	3	4	5	Mean	RSD	Rec
Vinyl chloride	8.0	7.5	6.7	5.4	6.6	6.8	13.0	34.2
Trichlorofluoromethane	13.3	16.5	14.9	13.0	10.3	13.6	15.2	68.0
1,1-Dichloroethene	17.1	16.7	15.1	14.8	15.6	15.9	5.7	79.2
Methylene chloride	24.5	22.7	19.7	19.4	20.6	21.4	9.1	107
trans-1,2-Dichloroethene	22.7	23.6	19.4	18.3	20.1	20.8	0.7	104
1,2-Dichloroethane	18.3	18.0	16.7	15.6	15.9	16.9	6.4	84.4
cis-1,2-Dichloroethene	26.1	23.1	22.6	20.3	20.8	22.6	9.0	113
Bromochloromethane	24.5	25.4	20.9	20.1	20.1	22.2	10.2	111
Chloroform	26.5	26.0	22.1	18.9	22.1	23.1	12.2	116
1,1,1-Trichloroethane	21.5	23.0	23.9	16.7	31.2	23.4	21.2	117
Carbon tetrachloride	23.6	24.2	22.6	18.3	23.3	22.4	9.4	112
Benzene	22.4	23.9	20.4	17.4	19.2	20.7	11.2	103
Trichloroethene	21.5	20.5	19.2	14.4	19.1	18.9	12.7	94.6
1,2-Dichloropropane	24.9	26.3	23.1	19.0	23.3	23.3	10.5	117
Dibromomethane	25.4	26.4	21.6	20.4	23.6	23.5	9.6	117
Bromodichloromethane	25.7	26.7	24.1	17.9	23.0	23.5	13.1	117
Toluene	28.3	25.0	24.8	16.3	23.6	23.6	16.9	118
1,1,2-Trichloroethane	25.4	24.5	21.6	17.7	22.1	22.2	12.1	111
1,3-Dichloropropane	25.4	24.2	22.7	17.0	22.2	22.3	12.8	112
Dibromochloromethane	26.3	26.2	23.7	18.2	23.2	23.5	12.5	118
Chlorobenzene	22.9	22.5	19.8	14.6	19.4	19.9	15.0	99.3
1,1,1,2-Tetrachloroethane	22.4	27.7	25.1	19.4	22.6	23.4	12.0	117
Ethylbenzene	25.6	25.0	22.1	14.9	24.0	22.3	17.5	112
p-Xylene	22.5	22.0	19.8	13.9	20.3	19.7	15.7	98.5
o-Xylene	24.2	23.1	21.6	14.0	20.4	20.7	17.3	103
Styrene	23.9	21.5	20.9	14.3	20.5	20.2	15.7	101
Bromoform	26.8	25.6	26.0	20.1	23.5	24.4	9.9	122
iso-Propylbenzene	25.3	25.1	24.2	15.4	24.6	22.9	16.6	114
Bromobenzene	19.9	21.8	20.0	15.5	19.1	19.3	10.7	96.3
1,2,3-Trichloropropane	25.9	23.0	25.6	15.9	21.4	22.2	15.8	111
n-Propylbenzene	26.0	23.8	22.6	13.9	21.9	21.6	19.0	106
2-Chlorotoluene	23.6	23.8	21.3	13.0	21.5	20.6	19.2	103
4-Chlorotoluene	21.0	19.7	18.4	12.1	18.3	17.9	17.1	89.5
1,3,5-Trimethylbenzene	24.0	22.1	22.5	13.8	22.9	21.1	17.6	105
sec-Butylbenzene	25.9	25.3	27.8	16.1	28.6	24.7	18.1	124
1,2,4-Trimethylbenzene	30.6	39.2	22.4	18.0	22.7	26.6	28.2	133
1,3-Dichlorobenzene	20.3	20.6	18.2	13.0	17.6	17.9	15.2	89.7
p-iso-Propyltoluene	21.6	22.1	21.6	16.0	22.8	20.8	11.8	104
1,4-Dichlorobenzene	18.1	21.2	20.0	13.2	17.4	18.0	15.3	90.0
1,2-Dichlorobenzene	18.4	22.5	22.5	15.2	19.9	19.7	13.9	96.6
n-Butylbenzene	13.1	20.3	19.5	10.8	18.7	16.5	23.1	82.4
1,2,4-Trichlorobenzene	14.5	14.9	15.7	8.8	12.3	13.3	18.8	66.2
Hexachlorobutadiene	17.6	22.5	21.6	13.2	21.6	19.3	18.2	96.3
1,2,3-Trichlorobenzene	14.9	15.9	16.5	11.9	13.9	14.6	11.3	73.1

Data in Tables 17, 18, and 19 are from Reference 15.

TABLE 18
RECOVERIES IN C-HORIZON SOILS FORTIFIED AT 4 µg/kg (ANALYSIS BY METHOD 5035)

	Recovery per Replicate (ng)							Mean
Compound	1	2	3	4	5	Mean	RSD	Rec
Vinyl chloride	33.4	31.0	30.9	29.7	28.6	30.8	5.2	154
Trichlorofluoromethane	37.7	20.8	20.0	21.8	20.5	24.1	28.2	121
1,1-Dichloroethene	21.7	33.5	39.8	30.2	32.5	31.6	18.5	158
Methylene chloride	20.9	19.4	18.7	18.3	18.4	19.1	5.1	95.7
trans-1,2-Dichloroethene	21.8	18.9	20.4	17.9	17.8	19.4	7.9	96.8
1,1-Dichloroethane	23.8	21.9	21.3	21.3	20.5	21.8	5.2	109
cis-1,2-Dichloroethene	21.6	18.8	18.5	18.2	18.2	19.0	6.7	95.2
Bromochloromethane	22.3	19.5	19.3	19.0	19.2	20.0	6.0	100
Chloroform	20.5	17.1	17.3	16.5	15.9	17.5	9.2	87.3
1,1,1-Trichloroethane	16.4	11.9	10.7	9.5	9.4	11.6	22.4	57.8
Carbon tetrachloride	13.1	11.3	13.0	11.8	11.2	12.1	6.7	60.5
Benzene	21.1	19.3	18.7	18.2	16.9	18.8	7.4	94.1
Trichloroethene	19.6	16.4	16.5	16.5	15.5	16.9	8.3	84.5
1,2-Dichloropropane	21.8	19.0	18.3	18.8	16.5	18.9	9.0	94.4
Dibromomethane	20.9	17.9	17.9	17.2	18.3	18.4	6.9	92.1
Bromodichloromethane	20.9	18.0	18.9	18.2	17.3	18.6	6.6	93.2
Toluene	22.2	17.3	18.8	17.0	15.9	18.2	12.0	91.2
1,1,2-Trichloroethane	21.0	16.5	17.2	17.2	16.5	17.7	9.6	88.4
1,3-Dichloropropane	21.4	17.3	18.7	18.6	16.7	18.5	8.8	92.6
Dibromochloromethane	20.9	18.1	19.0	18.8	16.6	18.7	7.5	93.3
Chlorobenzene	20.8	18.4	17.6	16.8	14.8	17.7	11.2	88.4
1,1,1,2-Tetrachloroethane	19.5	19.0	17.8	17.2	16.5	18.0	6.2	90.0
Ethylbenzene	21.1	18.3	18.5	16.9	15.3	18.0	10.6	90.0
p-Xylene	20.0	17.4	18.2	16.3	14.4	17.3	10.9	86.3
o-Xylene	20.7	17.2	16.8	16.2	14.8	17.1	11.4	85.7
Styrene	18.3	15.9	16.2	15.3	13.7	15.9	9.3	79.3
Bromoform	20.1	15.9	17.1	17.5	16.1	17.3	8.6	86.7
iso-Propylbenzene	21.0	18.1	19.2	18.4	15.6	18.4	9.6	92.2
Bromobenzene	20.4	16.2	17.2	16.7	15.4	17.2	10.1	85.9
1,1,2,2-Tetrachloroethane	23.3	17.9	21.2	18.8	16.8	19.6	12.1	96.0
1,2,3-Trichloropropane	18.4	14.6	15.6	16.1	15.6	16.1	8.0	80.3
n-Propylbenzene	20.4	18.9	17.9	17.0	14.3	17.7	11.6	88.4
2-Chlorotoluene	19.1	17.3	16.1	16.0	14.4	16.7	9.2	83.6
4-Chlorotoluene	19.0	15.5	16.8	15.9	13.6	16.4	10.6	81.8
1,3,5-Trimethylbenzene	20.8	18.0	17.4	16.1	14.7	17.4	11.7	86.9
sec-Butylbenzene	21.4	18.3	18.9	17.0	14.9	18.1	11.8	90.5
1,2,4-Trimethylbenzene	20.5	18.6	16.8	15.3	13.7	17.0	14.1	85.0
1,3-Dichlorobenzene	17.6	15.9	15.6	14.2	14.4	15.6	7.9	77.8
p-iso-Propyltoluene	20.5	17.0	17.1	15.6	13.4	16.7	13.9	83.6
1,4-Dichlorobenzene	18.5	13.8	14.8	16.7	14.9	15.7	10.5	78.7
1,2-Dichlorobenzene	18.4	15.0	15.4	15.3	13.5	15.5	10.5	77.6
n-Butylbenzene	19.6	15.9	15.9	14.4	18.9	16.9	11.7	84.6
1,2,4-Trichlorobenzene	15.2	17.2	17.4	13.6	12.1	15.1	13.5	75.4
Hexachlorobutadiene	18.7	16.2	15.5	13.8	16.6	16.1	10.0	80.7
Naphthalene	13.9	11.1	10.2	10.8	11.4	11.5	11.0	57.4
1,2,3-Trichlorobenzene	14.9	15.2	16.8	13.7	12.7	14.7	9.5	73.2

TABLE 19
RECOVERIES IN GARDEN SOIL FORTIFIED AT 4 μg/kg (ANALYSIS BY METHOD 5035)

	Recovery per Replicate (ng)							Mean
Compound	1	2	3	4	5	Mean	RSD	Rec
Vinyl chloride	12.7	10.9	9.8	8.1	7.2	9.7	20.2	48.7
Trichlorofluoromethane	33.7	6.4	30.3	27.8	22.9	24.2	39.6	121
1,1-Dichloroethene	27.7	20.5	24.1	15.1	13.2	20.1	26.9	101
Methylene chloride	25.4	23.9	24.7	22.2	24.2	24.1	4.4	120
trans-1,2-Dichloroethene	2.8	3.0	3.3	2.2	2.4	2.7	15.0	13.6
1,1-Dichloroethane	24.1	26.3	27.0	20.5	21.2	23.8	11.0	119
cis-1,2-Dichloroethene	8.3	10.2	8.7	5.8	6.4	7.9	20.1	39.4
Bromochloromethane	11.1	11.8	10.2	8.8	9.0	10.2	11.2	50.9
Chloroform	16.7	16.9	17.0	13.8	15.0	15.9	7.9	79.3
1,1,1-Trichloroethane	24.6	22.8	22.1	16.2	20.9	21.3	13.4	107
Carbon tetrachloride	19.4	20.3	22.2	20.0	20.2	20.4	4.6	102
Benzene	21.4	22.0	22.4	19.6	20.4	21.2	4.9	106
Trichloroethene	12.4	16.5	14.9	9.0	9.9	12.5	22.9	62.7
1,2-Dichloropropane	19.0	18.8	19.7	16.0	17.6	18.2	7.1	91.0
Dibromomethane	7.3	8.0	6.9	5.6	6.8	6.9	11.3	34.6
Bromodichloromethane	14.9	15.9	15.9	12.8	13.9	14.7	8.3	73.3
Toluene	42.6	39.3	45.1	39.9	45.3	42.4	5.9	212
1,1,2-Trichloroethane	13.9	15.2	1.4	21.3	14.9	15.9	17.0	79.6
1,3-Dichloropropane	13.3	16.7	11.3	10.9	9.5	12.3	20.3	61.7
Dibromochloromethane	14.5	13.1	14.5	11.9	14.4	13.7	7.6	68.3
Chlorobenzene	8.4	10.0	8.3	6.9	7.8	8.3	12.1	41.3
1,1,1,2-Tetrachloroethane	16.7	16.7	15.6	15.8	15.7	16.1	3.2	80.4
Ethylbenzene	22.1	21.4	23.1	20.1	22.6	21.9	4.8	109
p-Xylene	41.4	38.4	43.8	38.3	44.0	41.2	6.1	206
o-Xylene	31.7	30.8	34.3	30.4	33.2	32.1	4.6	160
Styrene	0	0	0	0	0	0	0	0
Bromoform	8.6	8.9	9.1	7.0	7.7	8.3	9.4	41.4
iso-Propylbenzene	18.1	18.8	9.7	18.3	19.6	18.9	3.5	94.4
Bromobenzene	5.1	5.4	5.3	4.4	4.0	4.8	11.6	24.1
1,1,2,2-Tetrachloroethane	14.0	13.5	14.7	15.3	17.1	14.9	8.5	74.5
1,2,3-Trichloropropane	11.0	12.7	11.7	11.7	11.9	11.8	4.5	59.0
n-Propylbenzene	13.4	13.3	14.7	12.8	13.9	13.6	4.7	68.1
2-Chlorotoluene	8.3	9.0	11.7	8.7	7.9	9.1	14.8	45.6
4-Chlorotoluene	5.1	5.4	5.5	4.8	4.5	5.0	7.9	25.2
1,3,5-Trimethylbenzene	31.3	27.5	33.0	31.1	33.6	31.3	6.8	157
sec-Butylbenzene	13.5	13.4	16.4	13.8	15.4	14.5	8.3	72.5
1,2,4-Trimethylbenzene	38.7	32.4	40.8	34.1	40.3	37.3	9.1	186
1,3-Dichlorobenzene	3.6	3.6	3.7	3.0	3.2	3.4	8.0	17.2
p-iso-Propyltoluene	14.7	14.1	16.1	13.9	15.1	14.8	5.2	73.8
1,4-Dichlorobenzene	3.0	3.5	3.3	2.6	2.8	3.0	10.2	15.0
1,2-Dichlorobenzene	3.6	4.3	4.0	3.5	3.6	3.8	8.3	19.0
n-Butylbenzene	17.4	13.8	14.0	18.9	24.0	17.6	21.2	88.0
1,2,4-Trichlorobenzene	2.8	2.9	3.3	2.6	3.2	3.0	8.5	15.0
Hexachlorobutadiene	4.8	4.0	6.1	5.6	6.0	5.3	15.1	26.4
Naphthalene	5.5	5.1	5.5	4.7	5.6	5.3	6.2	26.5
1,2,3-Trichlorobenzene	2.2	2.3	2.4	2.2	2.3	2.3	3.5	11.4

Data in Table 19 are from Reference 15.

TABLE 20

VOLATILE ORGANIC ANALYTE RECOVERY FROM SOIL USING VACUUM DISTILLATION (METHOD 5032)[a]

	Soil/H_2O[b] Recovery		Soil/Oil[c] Recovery		Soil/Oil/H_2O Recovery	
Compound	Mean	RSD	Mean	RSD	Mean	RSD
Chloromethane	61	20	40	18	108	68
Bromomethane	58	20	47	13	74	13
Vinyl chloride	54	12	46	11	72	20
Chloroethane	46	10	41	8	52	14
Methylene chloride	60	2	65	8	76	11
Acetone	INT[e]	INT	44	8		
Carbon disulfide	47	13	53	10	47	4
1,1-Dichloroethene	48	9	47	5	58	3
1,1-Dichloroethane	61	6	58	9	61	6
trans-1,2-Trichloroethane	54	7	60	7	56	5
cis-1,2-Dichloroethene	60	4	72	6	63	8
Chloroform	104	11	93	6	114	15
1,2-Dichloroethane	177	50	117	8	151	22
2-Butanone	INT	36	38	INT		
1,1,1-Trichloroethane	124	13	72	16	134	26
Carbon tetrachloride	172	122	INT	INT		
Vinyl acetate	88	11	INT			
Bromodichloromethane	93	4	91	23	104	23
1,1,2,2-Tetrachloroethane	96	13	50	12	104	7
1,2-Dichloropropane	105	8	102	6	111	6
trans-1,3-Dichloropropene	134	10	84	16	107	8
Trichloroethene	98	9	99	10	100	5
Dibromochloromethane	119	8	125	31	142	16
1,1,2-Trichloroethane	126	10	72	16	97	4
Benzene	99	7	CONT[f]	CONT		
cis-1,3-Dichloropropene	123	12	94	13	112	9
Bromoform	131	13	58	18	102	9
2-Hexanone	155	18	164	19	173	29
4-Methyl-2-pentanone	152	20	185	20	169	18
Tetrachloroethene	90	9	123	14	128	7
Toluene	94	3	CONT	CONT		
Chlorobenzene	98	7	93	18	112	5
Ethylbenzene	114	13	CONT	CONT		
Styrene	106	8	93	18	112	5
p-Xylene	97	9	CONT	CONT		
o-Xylene	105	8	112	12	144	13

TABLE 20 (cont.)

Compound	Soil/H_2O[b] Recovery Mean	Soil/H_2O[b] Recovery RSD	Soil/Oil[c] Recovery Mean	Soil/Oil[c] Recovery RSD	Soil/Oil/H_2O Recovery Mean	Soil/Oil/H_2O Recovery RSD
Surrogates						
1,2-Dichloroethane	177	50	117	8	151	22
Toluene-d_8	96	6	79	12	82	6
Bromofluorobenzene	139	13	37	13	62	5

[a] Results are for 10 min. distillations times, and condenser temperature held at -10°C. A 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness was used for chromatography. Standards and samples were replicated and precision value reflects the propagated errors. Each analyte was spiked at 50 ppb. Vacuum distillation efficiencies (Method 5032) are modified by internal standard corrections. Method 8260 internal standards may introduce bias for some analytes. See Method 5032 to identify alternate internal standards with similar efficiencies to minimize bias.

[b] Soil samples spiked with 0.2 mL water containing analytes and then 5 mL water added to make slurry.

[c] Soil sample + 1 g cod liver oil, spiked with 0.2 mL water containing analytes.

[d] Soil samples + 1 g cod liver oil, spiked as above with 5 mL of water added to make slurry.

[e] Interference by co-eluting compounds prevented accurate measurement of analyte.

[f] Contamination of sample matrix by analyte prevented assessment of efficiency.

TABLE 21

VACUUM DISTILLATION EFFICIENCIES FOR VOLATILE ORGANIC ANALYTES IN FISH TISSUE (METHOD 5032)[a]

Compound	Efficiency Mean (%)	RSD (%)
Chloromethane	N/A[b]	
Bromomethane	N/A[b]	
Vinyl chloride	N/A[b]	
Chloroethane	N/A[b]	
Methylene chloride	CONT[c]	
Acetone	CONT[c]	
Carbon disulfide	79	36
1,1-Dichloroethene	122	39
1,1-Dichloroethane	126	35
trans-1,2-Trichloroethene	109	46
cis-1,2-Dichloroethene	106	22
Chloroform	111	32
1,2-Dichloroethane	117	27
2-Butanone	INT[d]	
1,1,1-Trichloroethane	106	30
Carbon tetrachloride	83	34
Vinyl acetate	INT[d]	
Bromodichloromethane	97	22
1,1,2,2-Tetrachloroethane	67	20
1,2-Dichloropropane	117	23
trans-1,3-Dichloropropene	92	22
Trichloroethene	98	31
Dibromochloromethane	71	19
1,1,2-Trichloroethane	92	20
Benzene	129	35
cis-1,3-Dichloropropene	102	24
Bromoform	58	19
2-Hexanone	INT[d]	
4-Methyl-2-pentanone	113	37
Tetrachloroethene	66	20
Toluene	CONT[c]	
Chlorobenzene	65	19
Ethylbenzene	74	19
Styrene	57	14
p-Xylene	46	13
o-Xylene	83	20

TABLE 21 (cont.)

Compound	Efficiency Mean (%)	RSD (%)
Surrogates		
1,2-Dichloroethane	115	27
Toluene-d_8	88	24
Bromofluorobenzene	52	15

[a] Results are for 10 min. distillation times and condenser temperature held at -10°C. Five replicate 10-g aliquots of fish spiked at 25 ppb were analyzed using GC/MS external standard quantitation. A 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness was used for chromatography. Standards were replicated and results reflect 1 sigma propagated standard deviation.

[b] No analyses.

[c] Contamination of sample matrix by analyte prevented accurate assessment of analyte efficiency.

[d] Interfering by co-eluting compounds prevented accurate measurement of analyte.

TABLE 22

METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC ANALYTES IN FISH TISSUE (METHOD 5032)[a]

Compound	Method Detection Limit (ppb) External Standard Method	 Internal Standard Method
Chloromethane	7.8	7.3
Bromomethane	9.7	9.8
Vinyl chloride	9.5	9.4
Chloroethane	9.2	10.0
Methylene chloride	CONT[b]	CONT[b]
Acetone	CONT[b]	CONT[b]
Carbon disulfide	5.4	4.9
1,1-Dichloroethene	4.0	5.7
1,1-Dichloroethane	4.0	3.5
trans-1,2-Dichloroethene	4.4	4.0
cis-1,2-Dichloroethene	4.7	4.1
Chloroform	5.6	5.0
1,2-Dichloroethane	3.3	3.2
2-Butanone	INT[c]	INT[c]
1,1,1-Trichloroethane	1.1	4.2
Carbon tetrachloride	3.2	3.5
Vinyl acetate	INT[c]	INT[c]
Bromodichloromethane	3.2	2.8
1,1,2,2-Tetrachloroethane	4.4	3.8
1,2-Dichloropropane	3.8	3.7
trans-1,3-Dichloropropene	3.4	3.0
Trichloroethene	3.1	4.0
Dibromochloromethane	3.5	3.2
1,1,2-Trichloroethane	4.4	3.3
Benzene	3.6	3.2
cis-1,3-Dichloropropene	3.5	3.0
Bromoform	4.9	4.0
2-Hexanone	7.7	8.0
4-Methyl-2-pentanone	7.5	8.0
Tetrachloroethene	4.3	4.0
Toluene	3.0	2.5
Chlorobenzene	3.3	2.8
Ethylbenzene	3.6	3.5
Styrene	3.5	3.3
p-Xylene	3.7	3.5
o-Xylene	3.3	4.7

Footnotes are on the following page.

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[a] Values shown are the average MDLs for studies on three non-consecutive days, involving seven replicate analyses of 10 g of fish tissue spiked a 5 ppb. Daily MDLs were calculated as three times the standard deviation. Quantitation was performed by GC/MS Method 8260 and separation with a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness.

[b] Contamination of sample by analyte prevented determination.

[c] Interference by co-eluting compounds prevented accurate quantitation.

TABLE 23

VOLATILE ORGANIC ANALYTES RECOVERY FOR WATER USING VACUUM DISTILLATION (METHOD 5032)[a]

Compound	5 mL H_2O[b] Recovery		20 mL H_2O[c] Recovery		20 mL H_2O/Oil Recovery	
	Mean	RSD	Mean	RSD	Mean	RSD
Chloromethane	114	27	116	29	176	67
Bromomethane	131	14	121	14	113	21
Vinyl chloride	131	13	120	16	116	23
Chloroethane	110	15	99	8	96	16
Methylene chloride	87	16	105	15	77	6
Acetone	83	22	65	34	119	68
Carbon disulfide	138	17	133	23	99	47
1,1-Dichloroethene	105	11	89	4	96	18
1,1-Dichloroethane	118	10	119	11	103	25
trans-1,2-Dichloroethene	105	11	107	14	96	18
cis-1,2-Dichloroethene	106	7	99	5	104	23
Chloroform	114	6	104	8	107	21
1,2-Dichloroethane	104	6	109	8	144	19
2-Butanone	83	50	106	31	INT[c]	
1,1,1-Trichloroethane	118	9	109	9	113	23
Carbon tetrachloride	102	6	108	12	109	27
Vinyl acetate	90	16	99	7	72	36
Bromodichloromethane	104	3	110	5	99	5
1,1,2,2-Tetrachloroethane	85	17	81	7	111	43
1,2-Dichloropropane	100	6	103	2	104	7
trans-1,3-Dichloropropene	105	8	105	4	92	4
Trichloroethene	98	4	99	2	95	5
Dibromochloroethane	99	8	99	6	90	25
1,1,2-Trichloroethane	98	7	100	4	76	12
Benzene	97	4	100	5	112	10
cis-1,3-Dichloropropene	106	5	105	4	98	3
Bromoform	93	16	94	8	57	21
2-Hexanone	60	17	63	16	78	23
4-Methyl-2-pentanone	79	24	63	14	68	15
Tetrachloroethene	101	3	97	7	77	14
Toluene	100	6	97	8	85	5
Chlorobenzene	98	6	98	4	88	16
Ethylbenzene	100	3	92	8	73	13
Styrene	98	4	97	9	88	16
p-Xylene	96	4	94	8	60	12
o-Xylene	96	7	95	6	72	14

TABLE 23 (cont.)

	$5\ mL\ H_2O^b$ Recovery		$20\ mL\ H_2O^c$ Recovery		$20\ mL\ H_2O$/Oil Recovery	
Compound	Mean	RSD	Mean	RSD	Mean	RSD
Surrogates						
1,2-Dichloroethane	104	6	109	6	144	19
Toluene-d_8	104	5	102	2	76	7
Bromofluorobenzene	106	6	106	9	40	8

[a] Results are for 10 min. distillation times, and condenser temperature held at -10°C. A 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness was used for chromatography. Standards and samples were replicated and precision values reflect the propagated errors. Concentrations of analytes were 50 ppb for 5-mL samples and 25 ppb for 20-mL samples. Recovery data generated with comparison to analyses of standards without the water matrix.

[b] Sample contained 1 gram cod liver oil and 20 mL water. An emulsion was created by adding 0.2 mL of water saturated with lecithin.

[c] Interference by co-eluting compounds prevented accurate assessment of recovery.

TABLE 24

METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC ANALYTES USING VACUUM DISTILLATION (METHOD 5032) (INTERNAL STANDARD METHOD)[a]

Compound	Water[b] (µg/L)	Soil[c] (µg/kg)	Tissue[d] (µg/kg)	Oil[e] (mg/kg)
Chloromethane	3.2	8.0	7.3	N/A[f]
Bromomethane	2.8	4.9	9.8	N/A[f]
Vinyl chloride	3.5	6.0	9.4	N/A[f]
Chloroethane	5.9	6.0	10.0	N/A[f]
Methylene chloride	3.1	4.0	CONT[g]	0.05
Acetone	5.6	CONT[g]	CONT[g]	0.06
Carbon disulfide	2.5	2.0	4.9	0.18
1,1-Dichloroethene	2.9	3.2	5.7	0.18
1,1-Dichloroethane	2.2	2.0	3.5	0.14
trans-1,2-Dichloroethene	2.2	1.4	4.0	0.10
cis-1,2-Dichloroethene	2.0	2.3	4.1	0.07
Chloroform	2.4	1.8	5.0	0.07
1,2-Dichloroethane	1.7	1.5	3.2	0.06
2-Butanone	7.4	INT[h]	INT[h]	INT[h]
1,1,1-Trichloroethane	1.8	1.7	4.2	0.10
Carbon tetrachloride	1.4	1.5	3.5	0.13
Vinyl acetate	11.8	INT[h]	INT[h]	INT[h]
Bromodichloromethane	1.6	1.4	2.8	0.06
1,1,2,2-Tetrachloroethane	2.5	2.1	3.8	0.02
1,2-Dichloropropane	2.2	2.1	3.7	0.15
trans-1,3-Dichloropropene	1.5	1.7	3.0	0.05
Trichloroethene	1.6	1.7	4.0	0.04
Dibromochloromethane	1.7	1.5	3.2	0.07
1,1,2-Trichloroethane	2.1	1.7	3.3	0.05
Benzene	0.5	1.5	3.2	0.05
cis-1,3-Dichloropropene	1.4	1.7	3.0	0.04
Bromoform	1.8	1.5	4.0	0.05
2-Hexanone	4.6	3.6	8.0	INT[h]
4-Methyl-2-pentanone	3.5	4.6	8.0	INT[h]
Tetrachloroethene	1.4	1.6	4.0	0.10
Toluene	1.0	3.3	2.5	0.05
Chlorobenzene	1.4	1.4	2.8	0.06
Ethylbenzene	1.5	2.8	3.5	0.04
Styrene	1.4	1.4	3.3	0.18
p-Xylene	1.5	2.9	3.5	0.20
o-Xylene	1.7	3.4	4.7	0.07

Footnotes are found on the following page.

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[a] Quantitation was performed using GC/MS Method 8260 and chromatographic separation with a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness. Method detection limits are the average MDLs for studies on three non-consecutive days.

[b] Method detection limits are the average MDLs for studies of three non-consecutive days. Daily studies were seven replicated analyses of 5 mL aliquots of 4 ppb soil. Daily MDLs were three times the standard deviation.

[c] Daily studies were seven replicated analyses of 10 g fish tissue spiked at 5 ppb. Daily MDLs were three times the standard deviation. Quantitation was performed using GC/MS Method 8260 and chromatographic separation with a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness.

[d] Method detection limits are estimated analyzing 1 g of cod liver oil samples spiked at 250 ppm. Five replicates were analyzed using Method 8260.

[e] No analyses.

[f] Contamination of sample by analyte prevented determination.

[g] Interference by co-eluting compounds prevented accurate quantitation.

TABLE 25

METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC ANALYTES (METHOD 5032) (EXTERNAL STANDARD METHOD)[a]

Compound	Water[b] (µg/L)	Soil[c] (µg/kg)	Tissue[d] (µg/kg)	Oil[e] (mg/kg)
Chloromethane	3.1	8.6[f]	7.8	N/A[g]
Bromomethane	2.5	4.9[f]	9.7	N/A[g]
Vinyl chloride	4.0	7.1[f]	9.5	N/A[g]
Chloroethane	6.1	7.5[f]	9.2	N/A[g]
Methylene chloride	3.1	3.3	CONT[h]	0.08
Acetone	33.0[f]	CONT[h]	CONT[h]	0.12
Carbon disulfide	2.5	3.2	5.4	0.19
1,1-Dichloroethene	3.4	3.8	4.0	0.19
1,1-Dichloroethane	2.3	1.7	4.0	0.13
trans-1,2-Dichloroethene	3.0	3.2	4.4	0.09
cis-1,2-Dichloroethene	2.4	2.7	4.7	0.08
Chloroform	2.7	2.6	5.6	0.06
1,2-Dichloroethane	1.6	1.7	3.3	0.06
2-Butanone	57.0[f]	INT[i]	INT[i]	INT[i]
1,1,1-Trichloroethane	1.6	2.4	1.1	0.08
Carbon tetrachloride	1.5	1.7	3.2	0.15
Vinyl acetate	23.0[f]	INT[i]	INT[i]	INT[i]
Bromodichloromethane	2.0	2.3	3.2	0.05
1,1,2,2-Tetrachloroethane	3.6	3.2	4.4	0.09
1,2-Dichloropropane	2.9	3.7	3.8	0.12
trans-1,3-Dichloropropene	2.3	2.4	3.8	0.08
Trichloroethene	2.5	3.0	3.1	0.06
Dibromochloromethane	2.1	2.9	3.5	0.04
1,1,2-Trichloroethane	2.7	2.8	4.4	0.07
Benzene	1.7	2.9	3.6	0.03
cis-1,3-Dichloropropene	2.1	2.5	3.5	0.06
Bromoform	2.3	2.5	4.9	0.10
2-Hexanone	4.6	4.6	7.7	INT[i]
4-Methyl-2-pentanone	3.8	3.9	7.5	INT[i]
Tetrachloroethene	1.8	2.6	4.3	0.12
Toluene	1.8	4.4	3.0	0.09
Chlorobenzene	2.4	2.6	3.3	0.07
Ethylbenzene	2.4	4.1	3.6	0.09
Styrene	2.0	2.5	3.5	0.16
p-Xylene	2.3	3.9	3.7	0.18
o-Xylene	2.4	4.1	3.3	0.08

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[a] Method detection limits are the average MDLs for studies on three non-consecutive days. Daily studies were seven replicate analyses of 5-mL aliquots of water spiked at 4 ppb. Daily MDLs were three times the standard deviation.

[b] Daily studies were seven replicate analyses of 5-mL aliquots of water spiked at 4 ppb.

[c] These studies were seven replicate analyses of 5-g aliquots of soil spiked at 4 ppb.

[d] These studies were seven replicate analyses of 10-g aliquots of fish tissue spiked at 5 ppb.

[e] Method detection limits were estimated by analyzing cod liver oil samples spiked at 250 ppb. Five replicates were analyzed using Method 8260.

[f] Method detection limits were estimated by analyzing replicate 50 ppb standards five times over a single day.

[g] No analyses.

[h] Contamination of sample by analyte prevented determination.

[I] Interference by co-eluting compound prevented accurate quantitation.

TABLE 26

VOLATILE ORGANIC ANALYTE RECOVERY FROM OIL USING VACUUM DISTILLATION (METHOD 5032)[a]

Compound	Recovery Mean (%)	RSD (%)
Chloromethane	N/A[b]	
Bromomethane	N/A[b]	
Vinyl chloride	N/A[b]	
Chloroethane	N/A[b]	
Methylene chloride	62	32
Acetone	108	55
Carbon disulfide	98	46
1,1-Dichloroethene	97	24
1,1-Dichloroethane	96	22
trans-1,2-Trichloroethene	86	23
cis-1,2-Dichloroethene	99	11
Chloroform	93	14
1,2-Dichloroethane	138	31
2-Butanone	INT[c]	
1,1,1-Trichloroethane	89	14
Carbon tetrachloride	129	23
Vinyl acetate	INT[c]	
Bromodichloromethane	106	14
1,1,2,2-Tetrachloroethane	205	46
1,2-Dichloropropane	107	24
trans-1,3-Dichloropropene	98	13
Trichloroethene	102	8
Dibromochloromethane	168	21
1,1,2-Trichloroethane	95	7
Benzene	146	10
cis-1,3-Dichloropropene	98	11
Bromoform	94	18
2-Hexanone	INT[c]	
4-Methyl-2-pentanone	INT[c]	
Tetrachloroethene	117	22
Toluene	108	8
Chlorobenzene	101	12
Ethylbenzene	96	10
Styrene	120	46
p-Xylene	87	23
o-Xylene	90	10

TABLE 26 (cont.)

Compound	Recovery Mean (%)	RSD (%)
Surrogates		
1,2-Dichloroethane	137	30
Toluene-d_8	84	6
Bromofluorobenzene	48	2

[a] Results are for 10 min. distillation times and condenser temperature held at -10°C. Five replicates of 10-g fish aliquots spiked at 25 ppb were analyzed. Quantitation was performed with a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness. Standards and samples were replicated and precision value reflects the propagated errors. Vacuum distillation efficiencies (Method 5032) are modified by internal standard corrections. Method 8260 internal standards may bias for some analytes. See Method 5032 to identify alternate internal standards with similar efficiencies to minimize bias.

[b] Not analyzed.

[c] Interference by co-evaluating compounds prevented accurate measurement of analyte.

TABLE 27

METHOD DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC ANALYTES IN OIL (METHOD 5032)[a]

	Method Detection Limit (ppb)	
Compound	External Standard Method	Internal Standard Method
Chloromethane	N/A[b]	N/A[b]
Bromomethane	N/A[b]	N/A[b]
Vinyl chloride	N/A[b]	N/A[b]
Chloroethane	N/A[b]	N/A[b]
Methylene chloride	80	50
Acetone	120	60
Carbon disulfide	190	180
1,1-Dichloroethene	190	180
1,1-Dichloroethane	130	140
trans-1,2-Dichloroethene	90	100
cis-1,2-Dichloroethene	80	70
Chloroform	60	70
1,2-Dichloroethane	60	60
2-Butanone	INT[c]	INT[c]
1,1,1-Trichloroethane	80	100
Carbon tetrachloride	150	130
Vinyl acetate	INT[c]	INT[c]
Bromodichloromethane	50	60
1,1,2,2-Tetrachloroethane	90	20
1,2-Dichloropropane	120	150
trans-1,3-Dichloropropene	80	50
Trichloroethene	60	40
Dibromochloromethane	40	70
1,1,2-Trichloroethane	70	50
Benzene	30	50
cis-1,3-Dichloropropene	60	40
Bromoform	100	50
2-Hexanone	INT[c]	INT[c]
4-Methyl-2-pentanone	INT[c]	INT[c]
Tetrachloroethene	120	100
Toluene	90	50
Chlorobenzene	70	60
Ethylbenzene	90	40
Styrene	160	180
p-Xylene	180	200
o-Xylene	80	70

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[a] Method detection limits are estimated as the result of five replicated analyses of 1 g cod liver oil spiked at 25 ppb. MDLs were calculated as three times the standard deviation. Quantitation was performed using a 30 m x 0.53 mm ID stable wax column with a 1 µm film thickness.

[b] No analyses.

[c] Interference by co-eluting compounds prevented accurate quantitation.

TABLE 28

INTERNAL STANDARDS FOR ANALYTES AND SURROGATES PREPARED USING EQUILIBRIUM HEADSPACE ANALYSIS (METHOD 5021)

Chloroform-d_1	1,1,2-TCA-d_3	Bromobenzene-d_5
Dichlorodifluoromethane	1,1,1-Trichloroethane	Chlorobenzene
Chloromethane	1,1-Dichloropropene	Bromoform
Vinyl chloride	Carbon tetrachloride	Styrene
Bromomethane	Benzene	iso-Propylbenzene
Chloroethane	Dibromomethane	Bromobenzene
Trichlorofluoromethane	1,2-Dichloropropane	n-Propylbenzene
1,1-Dichloroethene	Trichloroethene	2-Chlorotoluene
Methylene chloride	Bromodichloromethane	4-Chlorotoluene
trans-1,2-Dichloroethene	cis-1,3-Dichloropropene	1,3,5-Trimethylbenzene
1,1-Dichloroethane	trans-1,3-Dichloropropene	tert-Butylbenzene
cis-1,2-Dichloroethene	1,1,2-Trichloroethane	1,2,4-Trimethylbenzene
Bromochloromethane	Toluene	sec-Butylbenzene
Chloroform	1,3-Dichloropropane	1,3-Dichlorobenzene
2,2-Dichloropropane	Dibromochloromethane	1,4-Dichlorobenzene
1,2-Dichloroethane	1,2-Dibromoethane	p-iso-Propyltoluene
	Tetrachloroethene	1,2-Dichlorobenzene
	1,1,2-Trichloroethane	n-Butylbenzene
	Ethylbenzene	1,2-Dibromo-3-chloropropane
	m-Xylene	1,2,4-Trichlorobenzene
	p-Xylene	Naphthalene
	o-Xylene	Hexachlorobutadiene
	1,1,2,2-Tetrachloroethane	1,2,3-Trichlorobenzene
	1,2,3-Trichloropropane	

TABLE 29

PRECISION AND MDL DETERMINED FOR ANALYSIS OF FORTIFIED SAND[a] (METHOD 5021)

Compound	% RSD	MDL (µg/kg)
Benzene	3.0	0.34
Bromochloromethane	3.4	0.27
Bromodichloromethane	2.4	0.21
Bromoform	3.9	0.30
Bromomethane	11.6	1.3
Carbon tetrachloride	3.6	0.32
Chlorobenzene	3.2	0.24
Chloroethane	5.6	0.51
Chloroform	3.1	0.30
Chloromethane	4.1	3.5[b]
1,2-Dibromo-3-chloropropane	5.7	0.40
1,2-Dibromoethane	3.2	0.29
Dibromomethane	2.8	0.20
1,2-Dichlorobenzene	3.3	0.27
1,3-Dichlorobenzene	3.4	0.24
1,4-Dichlorobenzene	3.7	0.30
Dichlorodifluoromethane	3.0	0.28
1,1-Dichloroethane	4.5	0.41
1,2-Dichloroethane	3.0	0.24
1,1-Dichloroethene	3.3	0.28
cis-1,2-Dichloroethene	3.2	0.27
trans-1,2-Dichloroethene	2.6	0.22
1,2-Dichloropropane	2.6	0.21
1,1-Dichloropropene	3.2	0.30
cis-1,3-Dichloropropene	3.4	0.27
Ethylbenzene	4.8	0.47
Hexachlorobutadiene	4.1	0.38
Methylene chloride	8.2	0.62[c]
Naphthalene	16.8	3.4[c]
Styrene	7.9	0.62
1,1,1,2-Tetrachloroethane	3.6	0.27
1,1,2,2-Tetrachloroethane	2.6	0.20
Tetrachloroethene	9.8	1.2[c]
Toluene	3.5	0.38
1,2,4-Trichlorobenzene	4.2	0.44
1,1,1-Trichloroethane	2.7	0.27
1,1,2-Trichloroethane	2.6	0.20
Trichloroethene	2.3	0.19

TABLE 29 (cont.)

Compound	% RSD	MDL (µg/kg)
Trichlorofluoromethane	2.7	0.31
1,2,3-Trichloropropane	1.5	0.11
Vinyl chloride	4.8	0.45
m-Xylene/p-Xylene	3.6	0.37
o-Xylene	3.6	0.33

[a] Most compounds spiked at 2 ng/g (2 µg/kg)
[b] Incorrect ionization due to methanol
[c] Compound detected in unfortified sand at >1 ng

TABLE 30

RECOVERIES IN GARDEN SOIL FORTIFIED AT 20 µg/kg (ANALYSIS BY METHOD 5021)

Compound	Recovery per Replicate (ng) Sample 1	Sample 2	Sample 3	Mean (ng)	RSD	Recovery (%)
Benzene	37.6	35.2	38.4	37.1	3.7	185[a]
Bromochloromethane	20.5	19.4	20.0	20.0	2.3	100
Bromodichloromethane	21.1	20.3	22.8	21.4	4.9	107
Bromoform	23.8	23.9	25.1	24.3	2.4	121
Bromomethane	21.4	19.5	19.7	20.2	4.2	101
Carbon tetrachloride	27.5	26.6	28.6	27.6	3.0	138
Chlorobenzene	25.6	25.4	26.4	25.8	1.7	129
Chloroethane	25.0	24.4	25.3	24.9	1.5	125
Chloroform	21.9	20.9	21.7	21.5	2.0	108
Chloromethane	21.0	19.9	21.3	20.7	2.9	104[a]
1,2-Dibromo-3-chloro-propane	20.8	20.8	21.0	20.9	0.5	104
1,2-Dibromoethane	20.1	19.5	20.6	20.1	2.2	100
Dibromomethane	22.2	21.0	22.8	22.0	3.4	110
1,2-Dichlorobenzene	18.0	17.7	17.1	17.6	2.1	88.0
1,3-Dichlorobenzene	21.2	21.0	20.1	20.8	2.3	104
1,4-Dichlorobenzene	20.1	20.9	19.9	20.3	2.1	102
Dichlorodifluoromethane	25.3	24.1	25.4	24.9	2.4	125
1,1-Dichloroethane	23.0	22.0	22.7	22.6	1.9	113
1,2-Dichloroethane	20.6	19.5	19.8	20.0	2.3	100
1,1-Dichloroethene	24.8	23.8	24.4	24.3	1.7	122
cis-1,2-Dichloroethene	21.6	20.0	21.6	21.1	3.6	105
trans-1,2-Dichloroethene	22.4	21.4	22.2	22.0	2.0	110
1,2-Dichloropropane	22.8	22.2	23.4	22.8	2.1	114
1,1-Dichloropropene	26.3	25.7	28.0	26.7	3.7	133
cis-1,3-Dichloropropene	20.3	19.5	21.1	20.3	3.2	102
Ethylbenzene	24.7	24.5	25.5	24.9	1.7	125
Hexachlorobutadiene	23.0	25.3	25.2	24.5	4.3	123
Methylene chloride	26.0	25.7	26.1	25.9	0.7	130[a]
Naphthalene	13.8	12.7	11.8	12.8	6.4	63.8[a]
Styrene	24.2	23.3	23.3	23.6	1.8	118
1,1,1,2-Tetrachloroethane	21.4	20.2	21.3	21.0	2.6	105
1,1,2,2-Tetrachloroethane	18.6	17.8	19.0	18.5	2.7	92.3
Tetrachloroethene	25.2	24.8	26.4	25.5	2.7	127
Toluene	28.6	27.9	30.9	29.1	4.4	146[a]
1,2,4-Trichlorobenzene	15.0	14.4	12.9	14.1	6.3	70.5
1,1,1-Trichloroethane	28.1	27.2	29.9	28.4	4.0	142
1,1,2-Trichloroethane	20.8	19.6	21.7	20.7	4.2	104

TABLE 30 (cont.)

Compound	Recovery per Replicate (ng) Sample 1	 Sample 2	 Sample 3	Mean (ng)	RSD	Recovery (%)
Trichloroethene	26.3	24.9	26.8	26.0	3.1	130
Trichlorofluoromethane	25.9	24.8	26.5	25.7	2.7	129
1,2,3-Trichloropropane	18.8	18.3	19.3	18.8	2.2	94.0
Vinyl chloride	24.8	23.2	23.9	24.0	2.7	120
m-Xylene/p-Xylene	24.3	23.9	25.3	24.5	2.4	123
o-Xylene	23.1	22.3	23.4	22.9	2.0	115

[a] Compound found in unfortified garden soil matrix at >5 ng.

TABLE 31

METHOD DETECTION LIMITS AND BOILING POINTS FOR VOLATILE ORGANICS (ANALYSIS BY METHOD 5041)[a]

Compound	Detection Limit (ng)	Boiling Point (°C)
Chloromethane	58	-24
Bromomethane	26	4
Vinyl chloride	14	-13
Chloroethane	21	13
Methylene chloride	9	40
Acetone	35	56
Carbon disulfide	11	46
1,1-Dichloroethene	14	32
1,1-Dichloroethane	12	57
trans-1,2-Dichloroethene	11	48
Chloroform	11	62
1,2-Dichloroethane	13	83
1,1,1-Trichloroethane	8	74
Carbon tetrachloride	8	77
Bromodichloromethane	11	88
1,1,2,2-Tetrachloroethane**	23	146
1,2-Dichloropropane	12	95
trans-1,3-Dichloropropene	17	112
Trichloroethene	11	87
Dibromochloromethane	21	122
1,1,2-Trichloroethane	26	114
Benzene	26	80
cis-1,3-Dichloropropene	27	112
Bromoform**	26	150
Tetrachloroethene	11	121
Toluene	15	111
Chlorobenzene	15	132
Ethylbenzene**	21	136
Styrene**	46	145
Trichlorofluoromethane	17	24
Iodomethane	9	43
Acrylonitrile	13	78
Dibromomethane	14	97
1,2,3-Trichloropropane**	37	157
total Xylenes**	22	138-144

Footnotes are found on the following page.

TABLE 31 (cont.)

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* The method detection limit (MDL) is defined in Chapter One. The detection limits cited above were determined according to 40 CFR, Part 136, Appendix B, using standards spiked onto clean VOST tubes. Since clean VOST tubes were used, the values cited above represent the best that the methodology can achieve. The presence of an emissions matrix will affect the ability of the methodology to perform at its optimum level.

** Boiling Point greater than 130°C. Not appropriate for quantitative sampling by Method 0030.

TABLE 32

VOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION (METHOD 5041)

Bromochloromethane

Acetone
Acrylonitrile
Bromomethane
Carbon disulfide
Chloroethane
Chloroform
Chloromethane
1,1-Dichloroethane
1,2-Dichloroethane
1,2-Dichloroethane-d_4 (surrogate)
1,1-Dichloroethene
Trichloroethene
trans-1,2-Dichloroethene
Iodomethane
Methylene chloride
Trichlorofluoromethane
Vinyl chloride

Chlorobenzene-d_5

4-Bromofluorobenzene (surrogate)
Chlorobenzene
Ethylbenzene
Styrene
1,1,2,2-Tetrachloroethane
Tetrachloroethene
Toluene
Toluene-d_8 (surrogate)
1,2,3-Trichloropropane
Xylenes

1,4-Difluorobenzene

Benzene
Bromodichloromethane
Bromoform
Carbon tetrachloride
Chlorodibromomethane
Dibromomethane
1,2-Dichloropropane
cis-1,3-Dichloropropene
trans-1,3-Dichloropropene
1,1,1-Trichloroethane
1,1,2-Trichloroethane

TABLE 33

METHOD 0040 - COMPOUNDS DEMONSTRATED TO BE APPLICABLE TO THE METHOD

Compound	Boiling Point (°C)	Condensation Point at 20°C (%)	Estimated Detection Limit[a] (ppm)
Dichlorodifluoromethane	-30	Gas	0.20
Vinyl chloride	-19	Gas	0.11
1,3-Butadiene	-4	Gas	0.90
1,2-Dichloro-1,1,2,2-tetrafluoroethane	4	Gas	0.14
Methyl bromide	4	Gas	0.14
Trichlorofluoromethane	24	88	0.18
1,1-Dichloroethene	31	22	0.07
Methylene chloride	40	44	0.05
1,1,2-Trichloro-trifluoroethane	48	37	0.13
Chloroform	61	21	0.04
1,1,1-Trichloroethane	75	13	0.03
Carbon tetrachloride	77	11	0.03
Benzene	80	10	0.16
Trichloroethene	87	8	0.04
1,2-Dichloropropane	96	5	0.05
Toluene	111	3	0.08
Tetrachloroethene	121	2	0.03

[a] Since this value represents a direct injection (no concentration) from the Tedlar® bag, these values are directly applicable as stack detection limits.

FIGURE 1
GAS CHROMATOGRAM OF VOLATILE ORGANICS

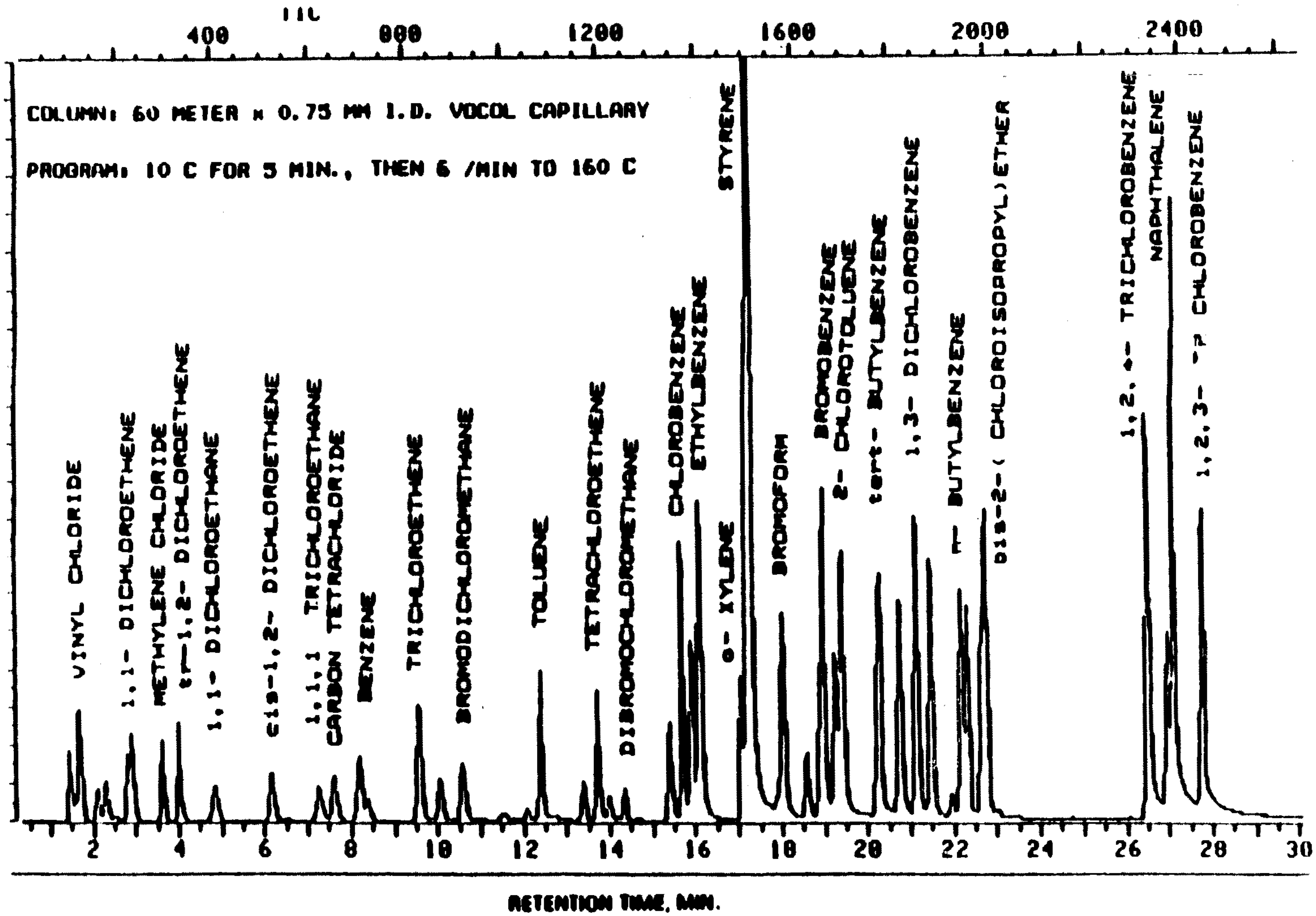


FIGURE 2
GAS CHROMATOGRAM OF VOLATILE ORGANICS

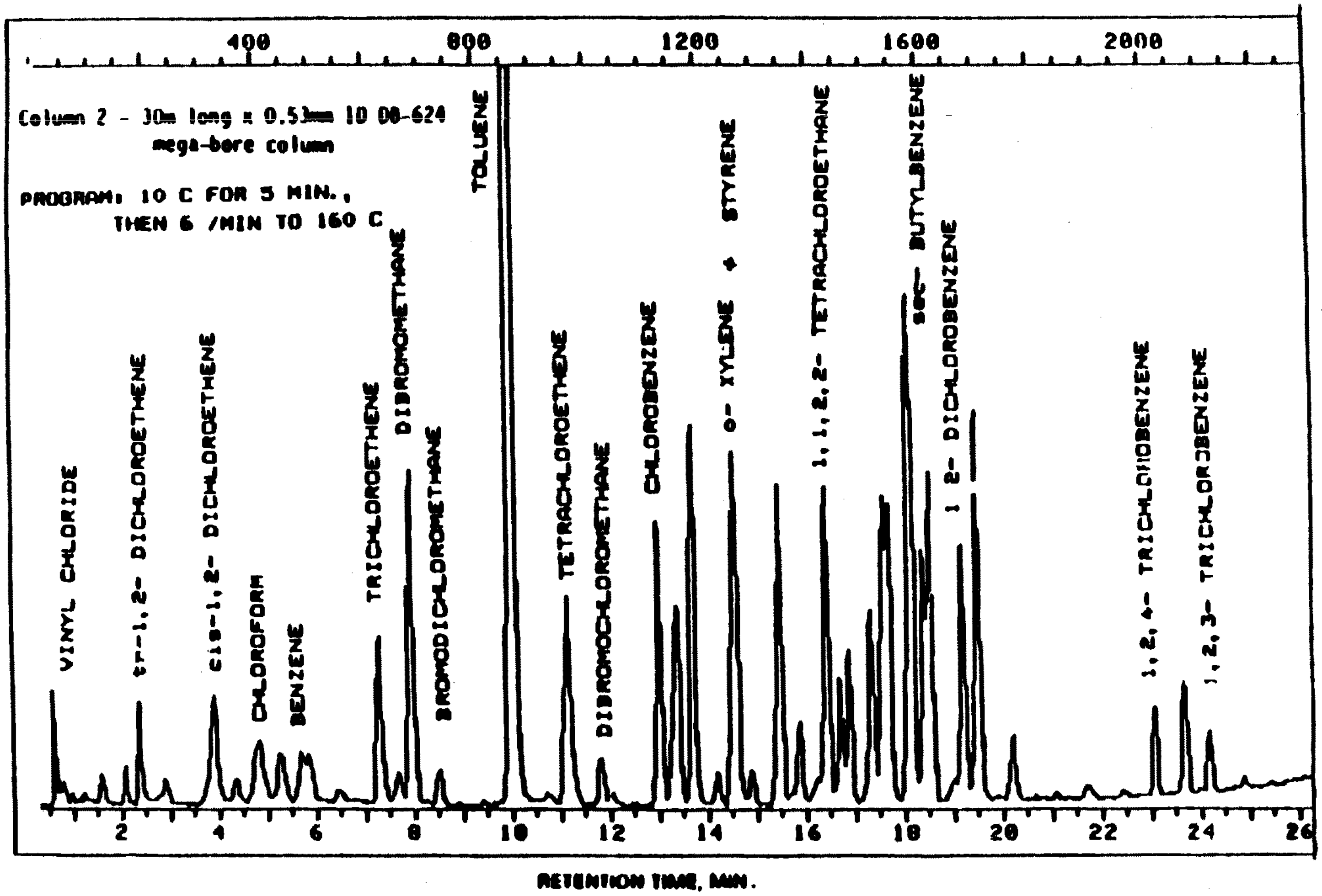


FIGURE 3

GAS CHROMATOGRAM OF VOLATILE ORGANICS

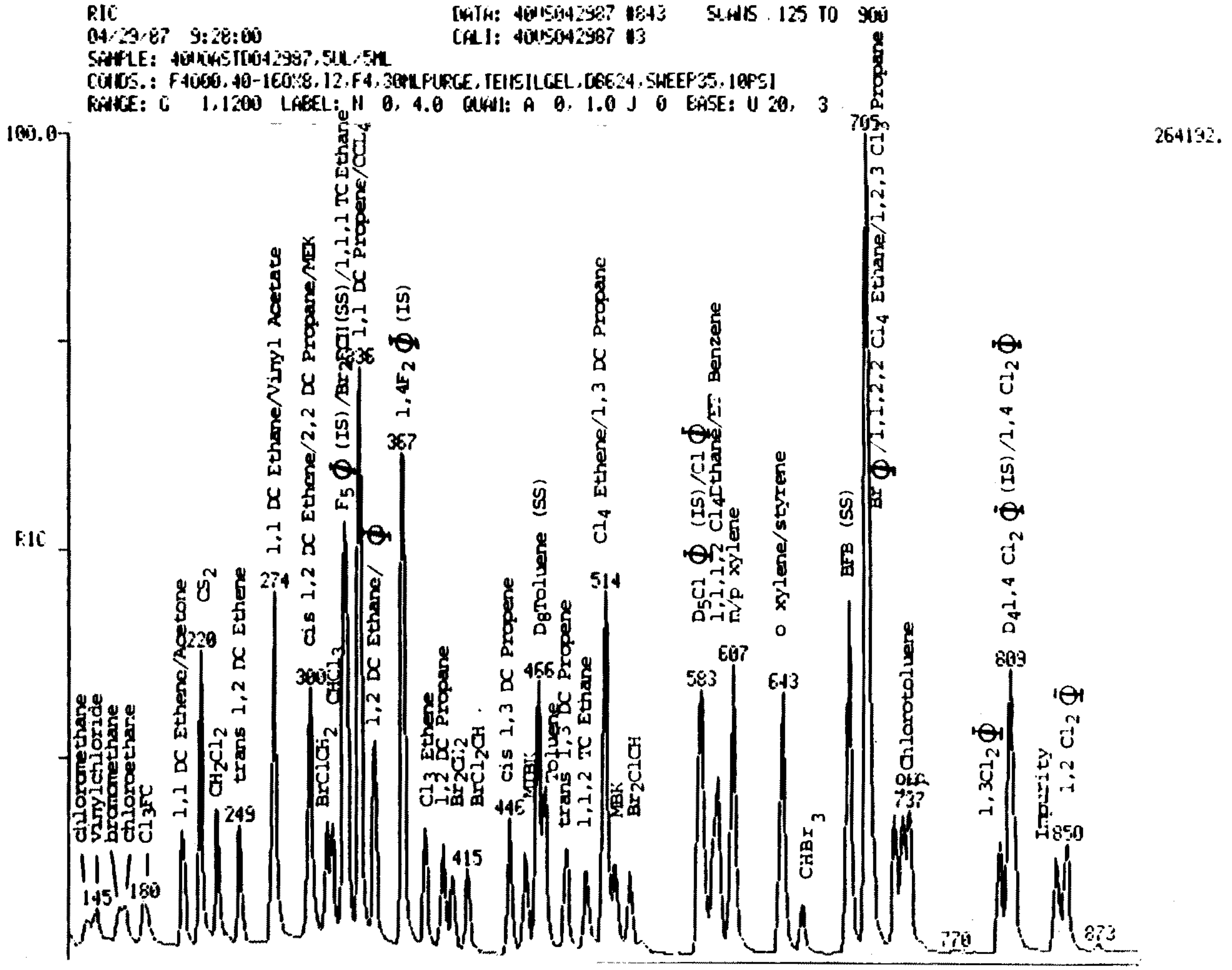
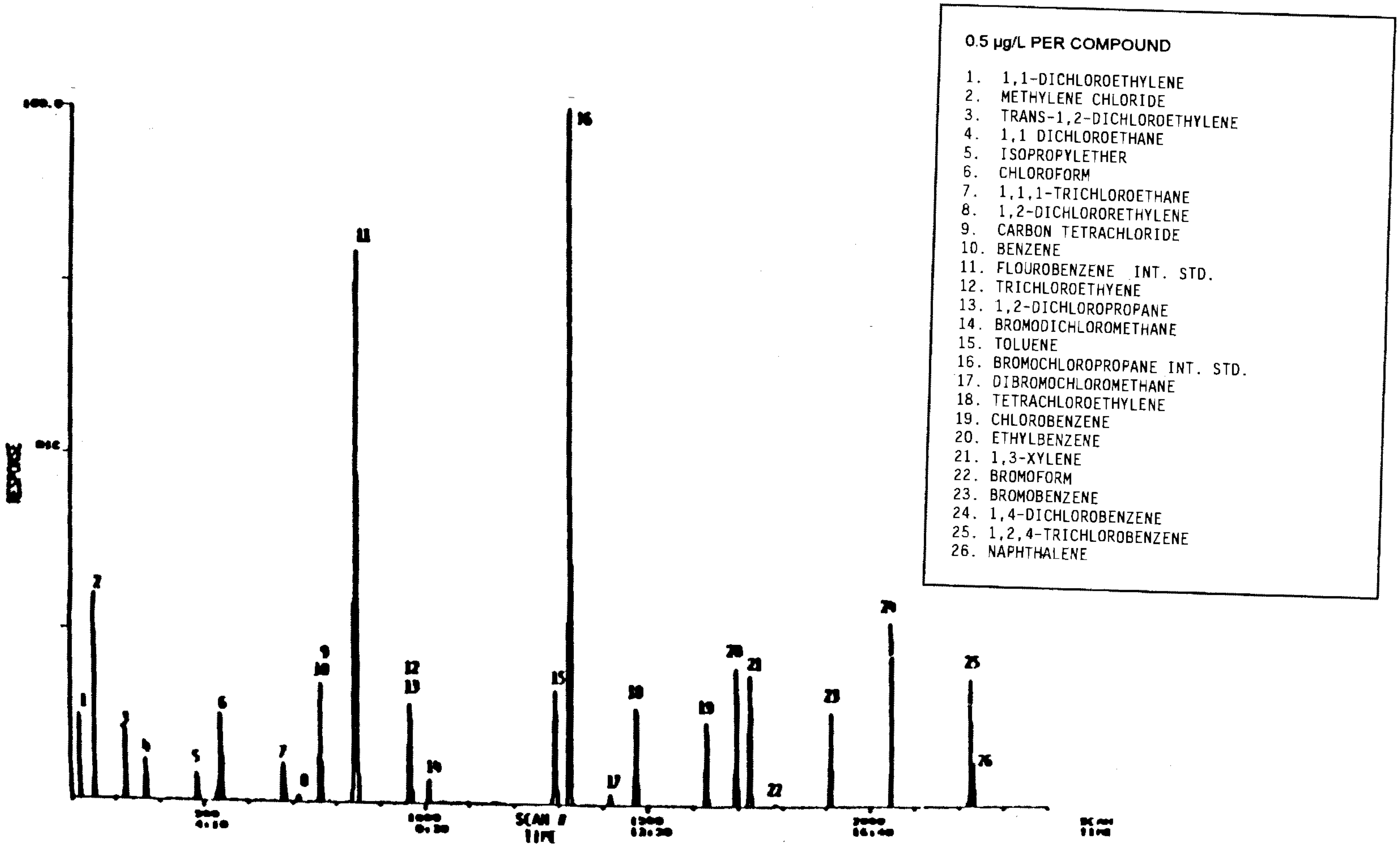
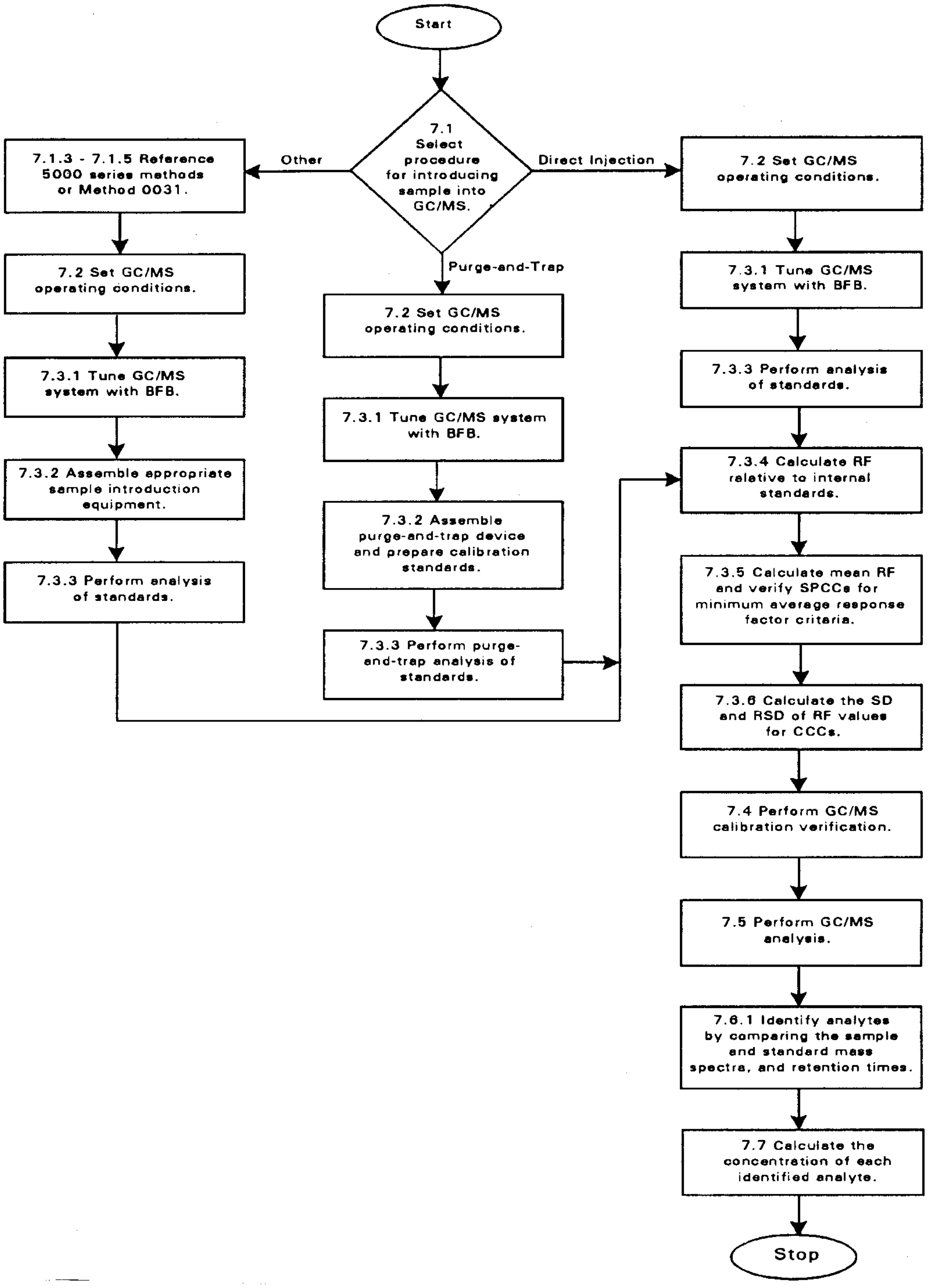


FIGURE 4
GAS CHROMATOGRAM OF TEST MIXTURE



0.5 µg/L PER COMPOUND

1. 1,1-DICHLOROETHYLENE
2. METHYLENE CHLORIDE
3. TRANS-1,2-DICHLOROETHYLENE
4. 1,1 DICHLOROETHANE
5. ISOPROPYLETHER
6. CHLOROFORM
7. 1,1,1-TRICHLOROETHANE
8. 1,2-DICHLORORETHYLENE
9. CARBON TETRACHLORIDE
10. BENZENE
11. FLOUROBENZENE INT. STD.
12. TRICHLOROETHYENE
13. 1,2-DICHLOROPROPANE
14. BROMODICHLOROMETHANE
15. TOLUENE
16. BROMOCHLOROPROPANE INT. STD.
17. DIBROMOCHLOROMETHANE
18. TETRACHLOROETHYLENE
19. CHLOROBENZENE
20. ETHYLBENZENE
21. 1,3-XYLENE
22. BROMOFORM
23. BROMOBENZENE
24. 1,4-DICHLOROBENZENE
25. 1,2,4-TRICHLOROBENZENE
26. NAPHTHALENE


Start
7.1 Select procedure for introducing sample into GC/MS.
Other
Direct Injection
Purge-and-Trap
7.1.3 - 7.1.5 Reference 5000 series methods or Method 0031.
7.2 Set GC/MS operating conditions.
7.3.1 Tune GC/MS system with BFB.
7.3.2 Assemble appropriate sample introduction equipment.
7.3.3 Perform analysis of standards.
7.2 Set GC/MS operating conditions.
7.3.1 Tune GC/MS system with BFB.
7.3.2 Assemble purge-and-trap device and prepare calibration standards.
7.3.3 Perform purge-and-trap analysis of standards.
7.2 Set GC/MS operating conditions.
7.3.1 Tune GC/MS system with BFB.
7.3.3 Perform analysis of standards.
7.3.4 Calculate RF relative to internal standards.
7.3.5 Calculate mean RF and verify SPCCs for minimum average response factor criteria.
7.3.6 Calculate the SD and RSD of RF values for CCCs.
7.4 Perform GC/MS calibration verification.
7.5 Perform GC/MS analysis.
7.6.1 Identify analytes by comparing the sample and standard mass spectra, and retention times.
7.7 Calculate the concentration of each identified analyte.
Stop

METHOD 5035

CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES

1.0 SCOPE AND APPLICATION

1.1 This method describes a closed-system purge-and-trap process for the analysis of volatile organic compounds (VOCs) in solid materials (e.g., soils, sediments, and solid waste). While the method is designed for use on samples containing low levels of VOCs, procedures are also provided for collecting and preparing solid samples containing high concentrations of VOCs and for oily wastes. For these high concentration and oily materials, sample collection and preparation are performed using the procedures described here, and sample introduction is performed using the aqueous purge-and-trap procedure in Method 5030. These procedures may be used in conjunction with any appropriate determinative gas chromatographic procedure, including, but not limited to, Methods 8015, 8021, and 8260.

1.2 The low soil method utilizes a hermetically-sealed sample vial, the seal of which is never broken from the time of sampling to the time of analysis. Since the sample is never exposed to the atmosphere after sampling, the losses of VOCs during sample transport, handling, and analysis are negligible. The applicable concentration range of the low soil method is dependent on the determinative method, matrix, and compound. However, it will generally fall in the 0.5 to 200 µg/kg range.

1.3 Procedures are included for preparing high concentration samples for purging by Method 5030. High concentration samples are those containing VOC levels of >200 µg/kg.

1.4 Procedures are also included for addressing oily wastes that are soluble in a water-miscible solvent. These samples are also purged using Method 5030..

1.5 Method 5035 can be used for most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile, water-soluble compounds can be included in this analytical technique. However, quantitation limits (by GC or GC/MS) are approximately ten times higher because of poor purging efficiency.

1.6 Method 5035, in conjunction with Method 8015 (GC/FID), may be used for the analysis of the aliphatic hydrocarbon fraction in the light ends of total petroleum hydrocarbons, e.g., gasoline. For the aromatic fraction (BTEX), use Method 5035 and Method 8021 (GC/PID). A total determinative analysis of gasoline fractions may be obtained using Method 8021 in series with Method 8015.

1.7 As with any preparative method for volatiles, samples should be screened to avoid contamination of the purge-and-trap system by samples that contain very high concentrations of purgeable material above the calibration range of the low concentration method. In addition, because the sealed sample container cannot be opened to remove a sample aliquot without compromising the integrity of the sample, multiple sample aliquots should be collected to allow for screening and reanalysis.

1.8 The closed-system purge-and-trap equipment employed for low concentration samples is not appropriate for soil samples preserved in the field with methanol. Such samples should be analyzed using Method 5030 (see the note in Sec. 6.2.2).

1.9 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 Low concentration soil method - generally applicable to and soils and other solid samples with VOC concentrations in the range of 0.5 to 200 µg/kg.

Volatile organic compounds (VOCs) are determined by collecting an approximately 5-g sample, weighed in the field at the time of collection, and placing it in a pre-weighed vial with a septum-sealed screw-cap (see Sec. 4) that already contains a stirring bar and a sodium bisulfate preservative solution. The vial is sealed and shipped to a laboratory or appropriate analysis site. The entire vial is then placed, unopened, into the instrument carousel. Immediately before analysis, organic-free reagent water, surrogates, and internal standards (if applicable) are automatically added without opening the sample vial. The vial containing the sample is heated to 40°C and the volatiles purged into an appropriate trap using an inert gas combined with agitation of the sample. Purged components travel via a transfer line to a trap. When purging is complete, the trap is heated and backflushed with helium to desorb the trapped sample components into a gas chromatograph for analysis by an appropriate determinative method.

2.2 High concentration soil method - generally applicable to soils and other solid samples with VOC concentrations greater than 200 µg/kg.

The sample introduction technique in Sec. 2.1 is not applicable to all samples, particularly those containing high concentrations (generally greater than 200 µg/kg) of VOCs which may overload either the volatile trapping material or exceed the working range of the determinative instrument system (e.g., GC/MS, GC/FID, GC/EC, etc.). In such instances, this method describes two sample collection options and the corresponding sample purging procedures.

2.2.1 The first option is to collect a bulk sample in a vial or other suitable container without the use of the preservative solution described in Sec. 2.1. A portion of that sample is removed from the container in the laboratory and is dispersed in a water-miscible solvent to dissolve the volatile organic constituents. An aliquot of the solution is added to 5 mL of reagent water in a purge tube. Surrogates and internal standards (if applicable) are added to the solution, then purged using Method 5030, and analyzed by an appropriate determinative method. Because the procedure involves opening the vial and removing a portion of the soil, some volatile constituents may be lost during handling.

2.2.2 The second option is to collect an approximately 5-g sample in a pre-weighed vial with a septum-sealed screw-cap (see Sec 4) that contains 5 mL of a water-miscible organic solvent (e.g., methanol). At the time of analysis, surrogates are added to the vial, then an aliquot of the solvent is removed from the vial, purged using Method 5030 and analyzed by an appropriate determinative method.

2.3 High concentration oily waste method - generally applicable to oily samples with VOC concentrations greater than 200 µg/kg that can be diluted in a water-miscible solvent.

Samples that are comprised of oils or samples that contain significant amounts of oil present additional analytical challenges. This procedure is generally appropriate for such samples when they are soluble in a water-miscible solvent.

2.3.1 After demonstrating that a test aliquot of the sample is soluble in methanol or polyethylene glycol (PEG), a separate aliquot of the sample is spiked with surrogates and diluted in the appropriate solvent. An aliquot of the solution is added to 5 mL of reagent water in a purge tube, taking care to ensure that a floating layer of oil is not present in the purge tube. Internal standards (if applicable) are added to the solution which is then purged using Method 5030 and analyzed by an appropriate determinative method.

2.3.2 Samples that contain oily materials that are not soluble in water-miscible solvents must be prepared according to Method 3585.

3.0 INTERFERENCES

3.1 Impurities in the purge gas and from organic compounds out-gassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running method blanks. The use of non-polytetrafluoroethylene (non-PTFE) plastic coating, non-PTFE thread sealants, or flow controllers with rubber components in the purging device must be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. These compounds will result in interferences or false positives in the determinative step.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank prepared from organic-free reagent water and carried through sampling and handling protocols serves as a check on such contamination.

3.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. Where practical, samples with unusually high concentrations of analytes should be followed by an analysis of organic-free reagent water to check for cross-contamination. If the target compounds present in an unusually concentrated sample are also found to be present in the subsequent samples, the analyst must demonstrate that the compounds are not due to carryover. Conversely, if those target compounds are not present in the subsequent sample, then the analysis of organic-free reagent water is not necessary.

3.4 The laboratory where volatile analysis is performed should be completely free of solvents. Special precautions must be taken to determine methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride, otherwise random background levels will result. Since methylene chloride will permeate through PTFE tubing, all GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory workers' clothing previously exposed to methylene chloride fumes during common liquid/liquid extraction procedures can contribute to sample contamination. The presence of other organic solvents in the laboratory where volatile organics are analyzed will also lead to random background levels and the same precautions must be taken.

4.0 APPARATUS AND MATERIALS

4.1 Sample Containers

The specific sample containers required will depend on the purge-and-trap system to be employed (see Sec. 4.2). Several systems are commercially available. Some systems employ 40-mL clear vials with a special frit and equipped with two PTFE-faced silicone septa. Other systems permit the use of any good quality glass vial that is large enough to contain at least 5 g of soil or solid material and at least 10 mL of water and that can be sealed with a screw-cap containing a PTFE-faced silicone septum. Consult the purge-and-trap system manufacturer's instructions regarding the suitable specific vials, septa, caps, and mechanical agitation devices.

4.2 Purge-and-Trap System

The purge-and-trap system consists of a unit that automatically adds water, surrogates, and internal standards (if applicable) to a vial containing the sample, purges the VOCs using an inert gas stream while agitating the contents of the vial, and also traps the released VOCs for subsequent desorption into the gas chromatograph. Such systems are commercially available from several sources and shall meet the following specifications.

4.2.1 The purging device should be capable of accepting a vial sufficiently large to contain a 5-g soil sample plus a magnetic stirring bar and 10 mL of water. The device must be capable of heating a soil vial to 40°C and holding it at that temperature while the inert purge gas is allowed to pass through the sample. The device should also be capable of introducing at least 5 mL of organic-free reagent water into the sample vial while trapping the displaced headspace vapors. It must also be capable of agitating the sealed sample during purging, (e.g., using a magnetic stirring bar added to the vial prior to sample collection, sonication, or other means). The analytes being purged must be quantitatively transferred to an absorber trap. The trap must be capable of transferring the absorbed VOCs to the gas chromatograph (see 4.2.2).

<u>NOTE</u>: The equipment used to develop this method was a Dynatech PTA-30 W/S Autosampler. This device was subsequently sold to Varian, and is now available as the Archon Purge and Trap Autosampler. See the Disclaimer at the front of this manual for guidance on the use of alternative equipment.

4.2.2 A variety of traps and trapping materials may be employed with this method. The choice of trapping material may depend on the analytes of interest. Whichever trap is employed, it must demonstrate sufficient adsorption and desorption characteristics to meet the quantitation limits of all the target analytes for a given project and the QC requirements in Method 8000 and the determinative method. The most difficult analytes are generally the gases, especially dichlorodifluoromethane. The trap must be capable of desorbing the late eluting target analytes.

<u>NOTE</u>: Check the responses of the brominated compounds when using alternative charcoal traps (especially Vocarb 4000), as some degradation has been noted when higher desorption temperatures (especially above 240 - 250°C) are employed. 2-Chloroethyl vinyl ether is degraded on Vocarb 4000 but performs adequately when Vocarb 3000 is used. The primary criterion, as stated above, is that all target analytes meet the sensitivity requirements for a given project.

4.2.2.1 The trap used to develop this method was 25 cm long, with an inside diameter of 0.105 inches, and was packed with Carbopack/Carbosieve (Supelco, Inc.).

4.2.2.2 The standard trap used in other EPA purge-and-trap methods is also acceptable. That trap is 25 cm long and has an inside diameter of at least 0.105 in. Starting from the inlet, the trap contains the equal amounts of the adsorbents listed below. It is recommended that 1.0 cm of methyl silicone-coated packing (35/60 mesh, Davison, grade 15 or equivalent) be inserted at the inlet to extend the life of the trap. If the analysis of dichlorodifluoromethane or other fluorocarbons of similar volatility is not required, then the charcoal can be eliminated and the polymer increased to fill 2/3 of the trap. If only compounds boiling above 35°C are to be analyzed, both the silica gel and charcoal can be eliminated and the polymer increased to fill the entire trap.

4.2.2.2.1 2,6-Diphenylene oxide polymer - 60/80 mesh, chromatographic grade (Tenax GC or equivalent).

4.2.2.2.2 Methyl silicone packing - OV-1 (3%) on Chromosorb-W, 60/80 mesh or equivalent.

4.2.2.2.3 Coconut charcoal - Prepare from Barnebey Cheney, CA-580-26, or equivalent, by crushing through 26 mesh screen.

4.2.2.3 Trapping materials other than those listed above also may be employed, provided that they meet the specifications in Sec. 4.2.3, below.

4.2.3 The desorber for the trap must be capable of rapidly heating the trap to the temperature recommended by the trap material manufacturer, prior to the beginning of the flow of desorption gas. Several commercial desorbers (purge-and-trap units) are available.

4.3 Syringe and Syringe Valves

4.3.1 25-mL glass hypodermic syringes with Luer-Lok (or equivalent) tip (other sizes are acceptable depending on sample volume used).

4.3.2 2-way syringe valves with Luer ends.

4.3.3 25-µL micro syringe with a 2 inch x 0.006 inch ID, 22° bevel needle (Hamilton #702N or equivalent).

4.3.4 Micro syringes - 10-, 100-µL.

4.3.5 Syringes - 0.5-, 1.0-, and 5-mL, gas-tight with shut-off valve.

4.4 Miscellaneous

4.4.1 Glass vials

4.4.1.1 60-mL, septum-sealed, to collect samples for screening, dry weight determination.

4.4.1.2 40-mL, screw-cap, PTFE lined, septum-sealed. Examine each vial prior to use to ensure that the vial has a flat, uniform sealing surface.

4.4.2 Top-loading balance - Capable of accurately weighing to 0.01 g.

4.4.3 Glass scintillation vials - 20-mL, with screw-caps and PTFE liners, or glass culture tubes with screw-caps and PTFE liners, for dilution of oily waste samples.

4.4.4 Volumetric flasks - Class A, 10-mL and 100-mL, with ground-glass stoppers.

4.4.5 2-mL glass vials, for GC autosampler - Used for oily waste samples extracted with methanol or PEG.

4.4.6 Spatula, stainless steel - narrow enough to fit into a sample vial.

4.4.7 Disposable Pasteur pipettes.

4.4.8 Magnetic stirring bars - PTFE- or glass-coated, of the appropriate size to fit the sample vials. Consult manufacturer's recommendation for specific stirring bars. Stirring bars may be reused, provided that they are thoroughly cleaned between uses. Consult the manufacturers of the purging device and the stirring bars for suggested cleaning procedures.

4.5 Field Sampling Equipment

4.5.1 Purge-and-Trap Soil Sampler - Model 3780PT (Associated Design and Manufacturing Company, 814 North Henry Street, Alexandria, VA 22314), or equivalent.

4.5.2 EnCore™ sampler - (En Chem, Inc., 1795 Industrial Drive, Green Bay, WI 54302), or equivalent.

4.5.3 Alternatively, disposable plastic syringes with a barrel smaller than the neck of the soil vial may be used to collect the sample. The syringe end of the barrel is cut off prior to sampling. One syringe is needed for each sample aliquot to be collected.

4.5.4 Portable balance - For field use, capable of weighing to 0.01 g.

4.5.5 Balance weights - Balances employed in the field should be checked against an appropriate reference weight at least once daily, prior to weighing any samples, or as described in the sampling plan. The specific weights used will depend on the total weight of the sample container, sample, stirring bar, reagent water added, cap, and septum.

5.0 REAGENTS

5.1 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

5.2 Methanol, CH_3OH - purge-and-trap quality or equivalent. Store away from other solvents.

5.3 Polyethylene glycol (PEG), $H(OCH_2CH_2)_nOH$ - free of interferences at the detection limit of the target analytes.

5.4 Low concentration sample preservative

5.4.1 Sodium bisulfate, $NaHSO_4$ - ACS reagent grade or equivalent.

5.4.2 The preservative should be added to the vial prior to shipment to the field, and must be present in the vial prior to adding the sample.

5.5 See the determinative method and Method 5000 for guidance on internal standards and surrogates to be employed in this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Refer to the introductory material in this chapter, Organic Analytes, Sec. 4.1, for general sample collection information. The low concentration portion of this method employs sample vials that are filled and weighed in the field and never opened during the analytical process. As a result, sampling personnel should be equipped with a portable balance capable of weighing to 0.01 g.

6.1 Preparation of sample vials

The specific preparation procedures for sample vials depend on the expected concentration range of the sample, with separate preparation procedures for low concentration soil samples and high concentration soil and solid waste samples. Sample vials should be prepared in a fixed laboratory or other controlled environment, sealed, and shipped to the field location. Gloves should be worn during the preparation steps.

6.1.1 Low concentration soil samples

The following steps apply to the preparation of vials used in the collection of low concentration soil samples to be analyzed by the closed-system purge-and-trap equipment described in Method 5035.

6.1.1.1 Add a clean magnetic stirring bar to each clean vial. If the purge-and-trap device (Sec. 4.2) employs a means of stirring the sample other than a magnetic stirrer (e.g., sonication or other mechanical means), then the stir bar is omitted.

6.1.1.2 Add preservative to each vial. The preservative is added to each vial prior to shipping the vial to the field. Add approximately 1 g of sodium bisulfate to each vial. If samples markedly smaller or larger than 5 g are to be collected, adjust the amount of preservative added to correspond to approximately 0.2 g of preservative for each 1 g of sample. Enough sodium bisulfate should be present to ensure a sample pH of ≤ 2.

6.1.1.3 Add 5 mL of organic-free reagent water to each vial. The water and the preservative will form an acid solution that will reduce or eliminate the majority of the biological activity in the sample, thereby preventing biodegradation of the volatile target analytes.

6.1.1.4 Seal the vial with the screw-cap and septum seal. If the double-ended, fritted, vials are used, seal both ends as recommended by the manufacturer.

6.1.1.5 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is negligible).

6.1.1.6 Weigh the prepared vial to the nearest 0.01 g, record the tare weight, and write it on the label.

6.1.1.7 Because volatile organics will partition into the headspace of the vial from the aqueous solution and will be lost when the vial is opened, surrogates, matrix spikes, and internal standards (if applicable) should only be added to the vials after the sample has been added to the vial. These standards should be introduced back in the laboratory, either manually by puncturing the septum with a small-gauge needle or automatically by the sample introduction system, just prior to analysis.

6.1.2 High concentration soil samples collected without a preservative

When high concentration samples are collected without a preservative, a variety of sample containers may be employed, including 60-mL glass vials with septum seals (see Sec. 4.4).

6.1.3 High concentration soil samples collected and preserved in the field

The following steps apply to the preparation of vials used in the collection of high concentration soil samples to be preserved in the field with methanol and analyzed by the aqueous purge-and-trap equipment described in Method 5030.

6.1.3.1 Add 10 mL of methanol to each vial.

6.1.3.2 Seal the vial with the screw-cap and septum seal.

6.1.3.3 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is negligible).

6.1.3.4 Weigh the prepared vial to the nearest 0.01 g, record the tare weight, and write it on the label.

NOTE: Vials containing methanol should be weighed a second time on the day that they are to be used. Vials found to have lost methanol (reduction in weight of >0.01 g) should not be used for sample collection.

6.1.3.5 Surrogates, internal standards and matrix spikes (if applicable) should be added to the sample after it is returned to the laboratory and prior to analysis.

6.1.4 Oily waste samples

When oily waste samples are known to be soluble in methanol or PEG, sample vials may be prepared as described in Sec. 6.1.3, using the appropriate solvent. However, when the solubility of the waste is unknown, the sample should be collected without the use of a preservative, in a vial such as that described in Sec. 6.1.2.

6.2 Sample collection

Collect the sample according to the procedures outlined in the sampling plan. As with any sampling procedure for volatiles, care must be taken to minimize the disturbance of the sample in order to minimize the loss of the volatile components. Several techniques may be used to transfer a sample to the relatively narrow opening of the low concentration soil vial. These include devices such as the EnCore^{TM} sampler, the Purge-and-Trap Soil Sampler $^{\mathbf{TM}}$, and a cut plastic syringe. Always wear gloves whenever handling the tared sample vials.

6.2.1 Low concentration soil samples

6.2.1.1 Using an appropriate sample collection device, collect approximately 5 g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere: generally within a few minutes at most. Carefully wipe the exterior of the sample collection device with a clean cloth or towel.

6.2.1.2 Using the sample collection device, add about 5 g (2 - 3 cm) of soil to the sample vial containing the preservative solution. Quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw-cap. Store samples on ice at 4°C.

<u>NOTE</u>: Soil samples that contain carbonate minerals (either from natural sources or applied as an amendment) may effervesce upon contact with the acidic preservative solution in the low concentration sample vial. If the amount of gas generated is very small (i.e., several mL), any loss of volatiles as a result of such effervescence may be minimal if the vial is sealed quickly. However, if larger amounts of gas are generated, not only may the sample lose a significant amount of analyte, but the gas pressure may shatter the vial if the sample vial is sealed. Therefore, when samples are known or suspected to contain high levels of carbonates, a test sample should be collected, added to a vial, and checked for effervescence. If a rapid or vigorous reaction occurs, discard the sample and collect low concentration samples in vials that do not contain the preservative solution.

6.2.1.3 When practical, use a portable balance to weigh the sealed vial containing the sample to ensure that 5.0 ± 0.5 g of sample were added. The balance should be calibrated in the field using an appropriate weight for the sample containers employed (Sec. 4.5.5). Record the weight of the sealed vial containing the sample to the nearest 0.01 g.

6.2.1.4 Alternatively, collect several trial samples with plastic syringes. Weigh each trial sample and note the length of the soil column in the syringe. Use these data to determine the length of soil in the syringe that corresponds to 5.0 ± 0.5 g. Discard each trial sample.

6.2.1.5 As with the collection of aqueous samples for volatiles, collect <u>at least</u> two replicate samples. This will allow the laboratory an additional sample for reanalysis. The second sample should be taken from the same soil stratum or the same section of the solid waste being sampled, and within close proximity to the location from which the original sample was collected.

6.2.1.6 In addition, since the soil vial cannot be opened without compromising the integrity of the sample, at least one additional aliquot of sample must be collected for screening, dry weight determination, and high concentration analysis (if necessary). This third aliquot may be collected in a 60-mL glass vial or a third 40-mL soil sample vial. However, this third vial must *not* contain the sample preservative solution, as an aliquot will be used to determine dry weight. If high concentration samples are collected in vials containing methanol, then two additional aliquots should be collected, one for high concentration analysis collected in a vial containing methanol, and another for the dry weight determination in a vial without either methanol or the low concentration aqueous preservative solution.

6.2.1.7 If samples are known or expected to contain target analytes over a wide range of concentrations, thereby requiring the analyses of multiple sample aliquots, it may be advisable and practical to take an additional sample aliquot in a low concentration soil vial containing the preservative, but collecting only 1-2 g instead of the 5 g collected in Sec. 6.2.1.1. This aliquot may be used for those analytes that exceed the instrument calibration range in the 5-g analysis.

6.2.1.8 The EnCore^{TM} sampler has not been thoroughly evaluated by EPA as a sample storage device. While preliminary results indicate that storage in the EnCore^{TM} device may be appropriate for up to 48 hours, samples collected in this device should be transferred to the soil sample vials as soon as possible, or analyzed within 48 hours.

6.2.1.9 The collection of low concentration soil samples in vials that contain methanol is <u>not</u> appropriate for samples analyzed with the closed-system purge-and-trap equipment described in this method (see Sec. 6.2.2).

6.2.2 High concentration soil samples preserved in the field

The collection of soil samples in vials that contain methanol has been suggested by some as a combined preservation and extraction procedure. However, this procedure is <u>not</u> appropriate for use with the low concentration soil procedure described in this method.

<u>NOTE</u>: The use of methanol preservation has not been formally evaluated by EPA and analysts must be aware of two potential problems. First, the use of methanol as a preservative and extraction solvent introduces a significant dilution factor that will raise the method quantitation limit beyond the operating range of the low concentration direct purge-and-trap procedure (0.5-200 µg/kg). The exact dilution factor will depend on the masses of solvent and sample, but generally exceeds 1000, and may make it difficult to demonstrate compliance with regulatory limits or action levels for some analytes. Because the analytes of interest are volatile, the methanol extract cannot be concentrated to overcome the dilution problem. Thus, for samples of unknown composition, it may still be necessary to collect an aliquot for analysis by this closed-system procedure and another aliquot preserved in methanol and analyzed by other procedures. The second problem is that the addition of methanol to the sample is likely to cause the sample to fail the ignitability characteristic, thereby making the unused sample volume a hazardous waste.

6.2.2.1 When samples are <u>known</u> to contain volatiles at concentrations high enough that the dilution factor will not preclude obtaining results within the calibration range of the appropriate determinative method, a sample may be collected and immediately placed in a sample vial containing purge-and-trap grade methanol.

6.2.2.2 Using an appropriate sample collection device, collect approximately 5 g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere: generally within a few minutes at most. Carefully wipe the exterior of the sample collection device with a clean cloth or towel.

6.2.2.3 Using the sample collection device, add about 5 g (2 - 3 cm) of soil to the vial containing 10 mL of methanol. Quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw-cap. Store samples on ice at 4°C.

6.2.2.4 When practical, use a portable balance to weigh the sealed vial containing the sample to ensure that 5.0 ± 0.5 g of sample were added. The balance should be calibrated in the field using an appropriate weight for the sample containers employed (Sec. 4.5.5). Record the weight of the sealed vial containing the sample to the nearest 0.01 g.

6.2.2.5 Alternatively, collect several trial samples with plastic syringes. Weigh each trial sample and note the length of the soil column in the syringe. Use these data to determine the length of soil in the syringe that corresponds to 5.0 ± 0.5 g. Discard each trial sample.

6.2.2.6 Other sample weights and volumes of methanol may be employed, provided that the analyst can demonstrate that the sensitivity of the overall analytical procedure is appropriate for the intended application.

6.2.2.7 The collection of at least one additional sample aliquot is required for the determination of the dry weight, as described in Sec. 6.2.1.6. Samples collected in methanol should be shipped as described in Sec. 6.3, and must be clearly labeled as containing methanol, so that the samples are not analyzed using the closed-system purge-and-trap equipment described in this procedure.

6.2.3 High concentration soil sample <u>not</u> preserved in the field

The collection of high concentration soil samples that are not preserved in the field generally follows similar procedures as for the other types of samples described in Secs. 6.2.1 and 6.2.2, with the obvious exception that the sample vials contain neither the aqueous preservative solution nor methanol. However, when field preservation is not employed, it is better to collect a larger volume sample, filling the sample container as full as practical in order to minimize the headspace. Such collection procedures generally do not require the collection of a separate aliquot for dry weight determination, but it may be advisable to collect a second sample aliquot for screening purposes, in order to minimize the loss of volatiles in either aliquot.

6.2.4 Oily waste samples

The collection procedures for oily samples depend on knowledge of the waste and its solubility in methanol or other solvents.

6.2.4.1 When an oily waste is <u>known</u> to be soluble in methanol or PEG, the sample may be collected in a vial containing such a solvent (see Sec. 6.1.4), using procedures similar to those described in Sec. 6.2.2.

6.2.4.2 When the solubility of the oily waste is <u>not</u> known, the sample should either be collected in a vial without a preservative, as described in Sec. 6.2.3, or the solubility of a trial sample should be tested in the field, using a vial containing solvent. If the trial sample is soluble in the solvent, then collect the oily waste sample as described in Sec. 6.2.2. Otherwise, collect an unpreserved sample as described in Sec. 6.2.3.

6.3 Sample handling and shipment

All samples for volatiles analysis should be cooled to approximately 4°C, packed in appropriate containers, and shipped to the laboratory on ice, as described in the sampling plan.

6.4 Sample storage

6.4.1 Once in the laboratory, store samples at 4°C until analysis. The sample storage area should be free of organic solvent vapors.

6.4.2 All samples should be analyzed as soon as practical, and within the designated holding time from collection. Samples not analyzed within the designated holding time must be noted and the data are considered minimum values.

6.4.3 When the low concentration samples are strongly alkaline or highly calcareous in nature, the sodium bisulfate preservative solution may not be strong enough to reduce the pH of the soil/water solution to below 2. Therefore, when low concentration soils to be sampled are known or suspected to be strongly alkaline or highly calcareous, additional steps may be required to preserve the samples. Such steps include: addition of larger amounts of the sodium bisulfate preservative to non-calcareous samples, storage of low concentration samples at -10°C (taking care not to fill the vials so full that the expansion of the water in the vial breaks the vial), or significantly reducing the maximum holding time for low concentration soil samples. Whichever steps are employed, they should be clearly described in the sampling and QA project plans and distributed to both the field and laboratory personnel. See Sec. 6.2.1.2 for additional information.

7.0 PROCEDURE

This section describes procedures for sample screening, the low concentration soil method, the high concentration soil method, and the procedure for oily waste samples. High concentration samples are to be introduced into the GC system using Method 5030. Oily waste samples are to be introduced into the GC system using Method 5030 if they are soluble in a water-miscible solvent, or using Method 3585 if they are not.

7.1 Sample screening

7.1.1 It is highly recommended that all samples be screened prior to the purge-and-trap GC or GC/MS analysis. Samples may contain higher than expected quantities of purgeable organics that will contaminate the purge-and-trap system, thereby requiring extensive cleanup and instrument maintenance. The screening data are used to determine which is the appropriate sample preparation procedure for the particular sample, the low concentration closed-system direct purge-and-trap method (Sec. 7.2), the high concentration (methanol extraction) method (Sec. 7.3), or the nonaqueous liquid (oily waste) methanol or PEG dilution procedure (Sec. 7.4).

7.1.2 The analyst may employ any appropriate screening technique. Two suggested screening techniques employing SW-846 methods are:

7.1.2.1 Automated headspace (Method 5021) using a gas chromatograph (GC) equipped with a photoionization detector (PID) and an electrolytic conductivity detector (HECD) in series, or, 7.1.2.2 Extraction of the sample with hexadecane (Method 3820) and analysis of the extract on a GC equipped with a FID and/or an ECD.

7.1.3 The analyst may inject a calibration standard containing the analytes of interest at a concentration equivalent to the upper limit of the calibration range of the low concentration soil method. The results from this standard may be used to determine when the screening results approach the upper limit of the low concentration soil method. There are no linearity or other performance criteria associated with the injection of such a standard, and other approaches may be employed to estimate sample concentrations.

7.1.4 Use the low concentration closed-system purge-and-trap method (Sec. 7.2) if the estimated concentration from the screening procedure falls within the calibration range of the selected determinative method. If the concentration exceeds the calibration range of the low concentration soil method, then use either the high concentration soil method (Sec. 7.3), or the oily waste method (Sec. 7.4).

7.2 Low concentration soil method (Approximate concentration range of 0.5 to 200 µg/kg - the concentration range is dependent upon the determinative method and the sensitivity of each analyte.)

7.2.1 Initial calibration

Prior to using this introduction technique for any GC or GC/MS method, the system must be calibrated. General calibration procedures are discussed in Method 8000, while the determinative methods and Method 5000 provide specific information on calibration and preparation of standards. Normally, external standard calibration is preferred for the GC methods (non-MS detection) because of possible interference problems with internal standards. If interferences are not a problem, or when a GC/MS method is used, internal standard calibration may be employed.

7.2.1.1 Assemble a purge-and-trap device that meets the specification in Sec. 4.2 and that is connected to a gas chromatograph or a gas chromatograph/mass spectrometer system.

7.2.1.2 Before initial use, a Carbopack/Carbosieve trap should be conditioned overnight at 245°C by backflushing with an inert gas flow of at least 20 mL/minute. If other trapping materials are substituted for the Carbopack/Carbosieve, follow the manufacturers recommendations for conditioning. Vent the trap effluent to the hood, not to the analytical column. Prior to daily use, the trap should be conditioned for 10 minutes at 245°C with backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.

7.2.1.3 If the standard trap in Sec. 4.2.2.2 is employed, prior to initial use, the trap should be conditioned overnight at 180°C by backflushing with an inert gas flow of at least 20 mL/min, or according to the manufacturer's recommendations. Vent the trap effluent to the hood, not to the analytical column. Prior to daily use, the trap should be conditioned for 10 min at 180°C with backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.

7.2.1.4 Establish the purge-and-trap instrument operating conditions. Adjust the instrument to inject 5 mL of water, to heat the sample to 40°C, and to hold the sample at 40°C for 1.5 minutes before commencing the purge process, or as recommended by the instrument manufacturer.

7.2.1.5 Prepare a minimum of five initial calibration standards containing all the analytes of interest and surrogates, as described in Method 8000, and following the instrument manufacturer's instructions. The calibration standards are prepared in organic-free reagent water. The volume of organic-free reagent water used for calibration must be the same volume used for sample analysis (normally 5 mL added to the vial before shipping it to the field <u>plus</u> the organic-free reagent water added by the instrument). The calibration standards should also contain approximately the same amount of the sodium bisulfate preservative as the sample (e.g., ~1 g), as the presence of the preservative will affect the purging efficiencies of the analytes. The internal standard solution must be added automatically, by the instrument, in the same fashion as used for the samples. Place the soil vial containing the solution in the instrument carousel. In order to calibrate the surrogates using standards at five concentrations, it may be necessary to disable the automatic addition of surrogates to each vial containing a calibration standard (consult the manufacturer’s instructions). Prior to purging, heat the sample vial to 40°C for 1.5 minutes, or as recommended by the manufacturer.

7.2.1.6 Carry out the purge-and-trap procedure as outlined in Secs. 7.2.3. to 7.2.5.

7.2.1.7 Calculate calibration factors (CF) or response factors (RF) for each analyte of interest using the procedures described in Method 8000. Calculate the average CF (external standards) or RF (internal standards) for each compound, as described in Method 8000. Evaluate the linearity of the calibration data, or choose another calibration model, as described in Method 8000 and the specific determinative method.

7.2.1.8 For GC/MS analysis, a system performance check must be made before this calibration curve is used (see Method 8260). If the purge-and-trap procedure is used with Method 8021, evaluate the response for the following four compounds: chloromethane; 1,1-dichloroethane; bromoform; and 1,1,2,2-tetrachloroethane. They are used to check for proper purge flow and to check for degradation caused by contaminated lines or active sites in the system.

7.2.1.8.1 Chloromethane is the most likely compound to be lost if the purge flow is too fast.

7.2.1.8.2 Bromoform is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response.

7.2.1.8.3 Tetrachloroethane and 1,1-dichloroethane are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

7.2.1.9 When analyzing for very late eluting compounds with Method 8021 (i.e., hexachlorobutadiene, 1,2,3-trichlorobenzene, etc.), cross-contamination and memory effects from a high concentration sample or even the standard are a common problem.

Extra rinsing of the purge chamber after analysis normally corrects this. The newer purge-and-trap systems often overcome this problem with better bakeout of the system following the purge-and-trap process. Also, the charcoal traps retain less moisture and decrease the problem.

7.2.2 Calibration verification

Refer to Method 8000 for details on calibration verification. A single standard near the mid-point of calibration range is used for verification. This standard should also contain approximately 1 g of sodium bisulfate.

7.2.3 Sample purge-and-trap

This method is designed for a 5-g sample size, but smaller sample sizes may be used. Consult the instrument manufacturer's instructions regarding larger sample sizes, in order to avoid clogging of the purging apparatus. The soil vial is hermetically sealed at the sampling site, and MUST remain so in order to guarantee the integrity of the sample. Gloves must be worn when handling the sample vial since the vial has been tared. If any soil is noted on the exterior of the vial or cap, it must be carefully removed prior to weighing. Weigh the vial and contents to the nearest 0.01 g, even if the sample weight was determined in the field, and record this weight. This second weighing provides a check on the field sampling procedures and provides additional assurance that the reported sample weight is accurate. Data users should be advised on significant discrepancies between the field and laboratory weights.

7.2.3.1 Remove the sample vial from storage and allow it to warm to room temperature. Shake the vial gently, to ensure that the contents move freely and that stirring will be effective. Place the sample vial in the instrument carousel according to the manufacturer's instructions.

7.2.3.2 Without disturbing the hermetic seal on the sample vial, add 5 mL of organic-free reagent water, the internal standards, and the surrogate compounds. This is carried out using the automated sampler. Other volumes of organic-free reagent water may be used, however, it is imperative that all samples, blanks, and calibration standards have exactly the same final volume of organic-free reagent water. Prior to purging, heat the sample vial to 40°C for 1.5 minutes, or as described by the manufacturer.

7.2.3.3 For the sample selected for matrix spiking, add the matrix spiking solution described in Sec. 5.0 of Method 5000, either manually, or automatically, following the manufacturer's instructions. The concentration of the spiking solution and the amount added should be established as described in Sec. 8.0 of Method 8000.

7.2.3.4 Purge the sample with helium or another inert gas at a flow rate of up to 40 mL/minute (the flow rate may vary from 20 to 40 mL/min, depending on the target analyte group) for 11 minutes while the sample is being agitated with the magnetic stirring bar or other mechanical means. The purged analytes are allowed to flow out of the vial through a glass-lined transfer line to a trap packed with suitable sorbent materials.

7.2.4 Sample Desorption

7.2.4.1 Non-cryogenic interface - After the 11 minute purge, place the purge-and-trap system in the desorb mode and preheat the trap to 245°C without a flow of desorption gas. Start the flow of desorption gas at 10 mL/minute for about four minutes (1.5 min is normally adequate for analytes in Method 8015). Begin the temperature program of the gas chromatograph and start data acquisition.

7.2.4.2 Cryogenic interface - After the 11 minute purge, place the purge-and-trap system in the desorb mode, make sure that the cryogenic interface is at -150°C or lower, and rapidly heat the trap to 245°C while backflushing with an inert gas at 4 mL/minute for about 5 minutes (1.5 min is normally adequate for analytes in Methods 8015). At the end of the 5-minute desorption cycle, rapidly heat the cryogenic trap to 250°C. Begin the temperature program of the gas chromatograph and start the data acquisition.

7.2.5 Trap Reconditioning

After desorbing the sample for 4 minutes, recondition the trap by returning the purge-and-trap system to the purge mode. Maintain the trap temperature at 245°C (or other temperature recommended by the manufacturer of the trap packing materials). After approximately 10 minutes, turn off the trap heater and halt the purge flow through the trap. When the trap is cool, the next sample can be analyzed.

7.2.6 Data Interpretation

Perform qualitative and quantitative analysis following the guidance given in the determinative method and Method 8000. If the concentration of any target analyte exceeds the calibration range of the instrument, it will be necessary to reanalyze the sample by the high concentration method. Such reanalyses need only address those analytes for which the concentration exceeded the calibration range of the low concentration method. Alternatively, if a sample aliquot of 1-2 g was also collected (see Sec. 6.2.1.7), it may be practical to analyze that aliquot for the analytes that exceeded the instrument calibration range in the 5-g analysis. If results are to be reported on a dry weight basis, proceed to Sec. 7.5

7.3 High concentration method for soil samples with concentrations generally greater than 200 µg/kg.

The high concentration method for soil is based on a solvent extraction. A solid sample is either extracted or diluted, depending on sample solubility in a water-miscible solvent. An aliquot of the extract is added to organic-free reagent water containing surrogates and, if applicable, internal and matrix spiking standards, purged according to Method 5030, and analyzed by an appropriate determinative method. Wastes that are insoluble in methanol (i.e., petroleum and coke wastes) are diluted with hexadecane (see Sec. 7.3.8).

The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were <u>not</u> preserved in the field are prepared using the steps below, beginning at Sec. 7.3.1. If solvent preservation was employed in the field, then the preparation begins with Sec. 7.3.4.

7.3.1 When the high concentration sample is <u>not</u> preserved in the field, the sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Whenever practical, mix the contents of the sample container by shaking or other mechanical means without opening the vial. When shaking is not practical, quickly mix the contents of the vial with a narrow metal spatula and immediately reseal the vial.

7.3.2 If the sample is from an unknown source, perform a solubility test before proceeding. Remove several grams of material from the sample container. Quickly reseal the container to minimize the loss of volatiles. Weigh 1-g aliquots of the sample into several test tubes or other suitable containers. Add 10 mL of methanol to the first tube, 10 mL of PEG to the second, and 10 mL of hexadecane to the third. Swirl the sample and determine if it is soluble in the solvent. Once the solubility has been evaluated, discard these test solutions. If the sample is soluble in either methanol or PEG, proceed with Sec. 7.3.3. If the sample is only soluble in hexadecane, proceed with Sec. 7.3.8.

7.3.3 For soil and solid waste samples that are soluble in methanol, add 9.0 mL of methanol and 1.0 mL of the surrogate spiking solution to a tared 20-mL vial. Using a top-loading balance, weigh 5 g (wet weight) of sample into the vial. Quickly cap the vial and reweigh the vial. Record the weight to 0.1 g. Shake the vial for 2 min. If the sample was not soluble in methanol, but was soluble in PEG, employ the same procedure described above, but use 9.0 mL of PEG in place of the methanol. Proceed with Sec. 7.3.5.

NOTE: The steps in Secs. 7.3.1, 7.3.2, and 7.3.3 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.

7.3.4 For soil and solid waste samples that were collected in methanol or PEG (see Sec. 6.2.2), weigh the vial to 0.1 g as a check on the weight recorded in the field, add the surrogate spiking solution to the vial by injecting it through the septum, shake for 2 min, as described above, and proceed with Sec. 7.3.5.

7.3.5 Pipet approximately 1 mL of the extract from either Sec. 7.3.3 or 7.3.4 into a GC vial for storage, using a disposable pipet, and seal the vial. The remainder of the extract may be discarded. Add approximately 1 mL of methanol or PEG to a separate GC vial for use as the method blank for each set of samples extracted with the same solvent.

7.3.6 The extracts must be stored at 4°C in the dark, prior to analysis. Add an appropriate aliquot of the extract (see Table 2) to 5.0 mL of organic-free reagent water and analyze by Method 5030 in conjunction with the appropriate determinative method. Proceed to Sec. 7.0 in Method 5030 and follow the procedure for purging high concentration samples.

7.3.7 If results are to be reported on a dry weight basis, determine the dry weight of a separate aliquot of the sample, using the procedure in Sec. 7.5, after the sample extract has been transferred to a GC vial and the vial sealed.

7.3.8 For solids that are not soluble in methanol or PEG (including those samples consisting primarily of petroleum or coking waste) dilute or extract the sample with hexadecane using the procedures in Sec. 7.0 of Method 3585.

7.4 High concentration method for oily waste samples

This procedure for the analysis of oily waste samples involves the dilution of the sample in methanol or PEG. However, care must be taken to avoid introducing any of the floating oil layer into the instrument. A portion of the diluted sample is then added to 5.0 mL of organic-free reagent water, purged according to Method 5030, and analyzed using an appropriate determinative method.

For oily samples that are not soluble in methanol or PEG (including those samples consisting primarily of petroleum or coking waste), dilute or extract with hexadecane using the procedures in Sec. 7.0 of Method 3585.

The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were not preserved in the field are prepared using the steps below, beginning at Sec. 7.4.1. If methanol preservation was employed in the field, then the preparation begins with Sec. 7.4.3.

7.4.1 If the waste was not preserved in the field and it is soluble in methanol or PEG, weigh 1 g (wet weight) of the sample into a tared 10-mL volumetric flask, a tared scintillation vial, or a tared culture tube. If a vial or tube is used instead of a volumetric flask, it must be calibrated prior to use. This operation must be performed prior to opening the sample vial and weighing out the aliquot for analysis.

7.4.1.1 To calibrate the vessel, pipet 10.0 mL of methanol or PEG into the vial or tube and mark the bottom of the meniscus.

7.4.1.2 Discard this solvent, and proceed with weighing out the 1-g sample aliquot.

7.4.2 Quickly add 1.0 mL of surrogate spiking solution to the flask, vial, or tube, and dilute to 10.0 mL with the appropriate solvent (methanol or PEG). Swirl the vial to mix the contents and then shake vigorously for 2 minutes.

7.4.3 If the sample was collected in the field in a vial containing methanol or PEG, weigh the vial to 0.1 g as a check on the weight recorded in the field, add the surrogate spiking solution to the vial by injecting it through the septum. Swirl the vial to mix the contents and then shake vigorously for 2 minutes and proceed with Sec. 7.4.4.

7.4.4 Regardless of how the sample was collected, the target analytes are extracted into the solvent along with the majority of the oily waste (i.e., some of the oil may still be floating on the surface). If oil is floating on the surface, transfer 1 to 2 mL of the extract to a clean GC vial using a Pasteur pipet. Ensure that no oil is transferred to the vial.

7.4.5 Add 10 - 50 µL of the methanol extract to 5 mL of organic-free reagent water for purge-and-trap analysis, using Method 5030.

7.4.6 Prepare a matrix spike sample by adding 10 - 50 µL of the matrix spike standard dissolved in methanol to a 1-g aliquot of the oily waste. Shake the vial to disperse the matrix spike solution throughout the oil. Then add 10 mL of extraction solvent and proceed with the extraction and analysis, as described in Secs. 7.4.2 - 7.4.5. Calculate the recovery of the spiked analytes as described in Method 8000. If the recovery is not within the acceptance limits for the application, use the hexadecane dilution technique in Sec. 7.0 of Method 3585.

7.5 Determination of % Dry Weight

If results are to be reported on a dry weight basis, it is necessary to determine the dry weight of the sample.

NOTE: It is highly recommended that the dry weight determination only be made after the analyst has determined that no sample aliquots will be taken from the 60-mL vial for high concentration analysis. This is to minimize loss of volatiles and to avoid sample contamination from the laboratory atmosphere. There is no holding time associated with the dry weight determination. Thus, this determination can be made any time prior to reporting the sample results, as long as the vial containing the additional sample has remained sealed and properly stored.

7.5.1 Weigh 5-10 g of the sample from the 60-mL VOA vial into a tared crucible.

7.5.2 Dry this aliquot overnight at 105°C. Allow to cool in a desiccator before weighing. Calculate the % dry weight as follows:

$$\%\ \text{dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

WARNING: The drying oven should be contained in a hood or vented. Significant laboratory contamination may result from a heavily contaminated hazardous waste sample.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control procedures and Method 5000 for sample preparation QC procedures.

8.2 Before processing any samples, the analyst should demonstrate through the analysis of an organic-free reagent water method blank that all glassware and reagents are interference free. Each time a set of samples is extracted, or there is a change in reagents, a method blank should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement.

8.3 Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat this demonstration whenever new staff are trained or significant changes in instrumentation are made. See Sec. 8.0 of Methods 5000 and 8000 for information on how to accomplish this demonstration.

8.4 Sample Quality Control for Preparation and Analysis - See Sec. 8.0 in Method 5000 and Method 8000 for procedures to follow to demonstrate acceptable continuing performance on each set of samples to be analyzed. These include the method blank, either a matrix spike/matrix spike duplicate or a matrix spike and duplicate sample analysis, a laboratory control sample (LCS), and the addition of surrogates to each sample and QC sample.

8.5 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9.0 METHOD PERFORMANCE

9.1 Single laboratory accuracy and precision data were obtained for the method analytes in three soil matrices, sand, a soil collected 10 feet below the surface of a hazardous landfill, called the

C-Horizon, and a surface garden soil. Each sample was fortified with the analytes at a concentration of 20 ng/5 g, which is equivalent to 4 µg/kg. These data are listed in tables found in Method 8260.

9.2 Single laboratory accuracy and precision data were obtained for certain method analytes when extracting oily liquid using methanol as the extraction solvent. The data are presented in a table in Method 8260. The compounds were spiked into three portions of an oily liquid (taken from a waste site) following the procedure for matrix spiking described in Sec. 7.4. This represents a worst case set of data based on recovery data from many sources of oily liquid.

10.0 REFERENCES

1. Bellar, T., "Measurement of Volatile Organic Compounds in Soils Using Modified Purge-and-Trap and Capillary Gas Chromatography/Mass Spectrometry" U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Cincinnati, OH, November 1991.

2. Siegrist, R. L., Jenssen, P. D., “Evaluation of Sampling Method Effects on Volatile Organic Compound Measurements in Contaminated Soils”, Envir Sci Technol, 1990; 24; 1387-92.

3. Hewitt, A. D., Jenkins, T. F., Grant, C. L., “Collection, Handling and Storage: Keys to Improved Data Quality for Volatile Organic Compounds in Soil”, Am Environ Lab, 1995; 7(1); 25-8.

4. Liikala, T. L., Olsen, K. B., Teel, S. S., Lanigan, D. C., “Volatile Organic Compounds: Comparison of Two Sample Collection and Preservation Methods”, Envir Sci Technol, 1996; 30; 3441-7.

5. Lewis, T. E., Crockett, A. B., Siegrist, R. L., Zarrabi, K., “Soil Sampling and Analysis for Volatile Organic Compounds”, Envir Monitoring & Assessment, 1994; 30; 213-46.

6. Hewitt, A. D., “Enhanced Preservation of Volatile Organic Compounds in Soil with Sodium Bisulfate”, SR95-26, U. S. Army Cold Regions Research and Engineering Laboratory, Hanover, NH.

7. Hewitt, A. D., Lukash, N. J. E., “Sampling for In-Vial Analysis of Volatile Organic Compounds in Soil”, Am Environ Lab, 1996; Aug; 15-9.

8. Hewitt, A. D., Miyares, P. H., Sletten, R. S., “Determination of Two Chlorinated Volatile Organic Compounds in Soil by Headspace Gas Chromatography and Purge-and-Trap Gas Chromatography/Mass Spectrometry”, Hydrocarbon Contaminated Soils, 1993, 3; 135-45, Chelsea, MI, Lewis Publishers.

9. Hewitt, A. D., “Methods of Preparing Soil Samples for Headspace Analysis of Volatile Organic Compounds: Emphasis on Salting Out”, 12th Annual Waste Testing and Quality Assurance Symposium, Washington, DC, 1996, 322-9.

10. Hewitt, A. D., Miyares, P. H., Leggett, D. C., Jenkins, T. F., “Comparison of Analytical Methods for Determination of Volatile Organic Compounds”, Envir Sci Tech, 1992; 26; 1932-8.

TABLE 1

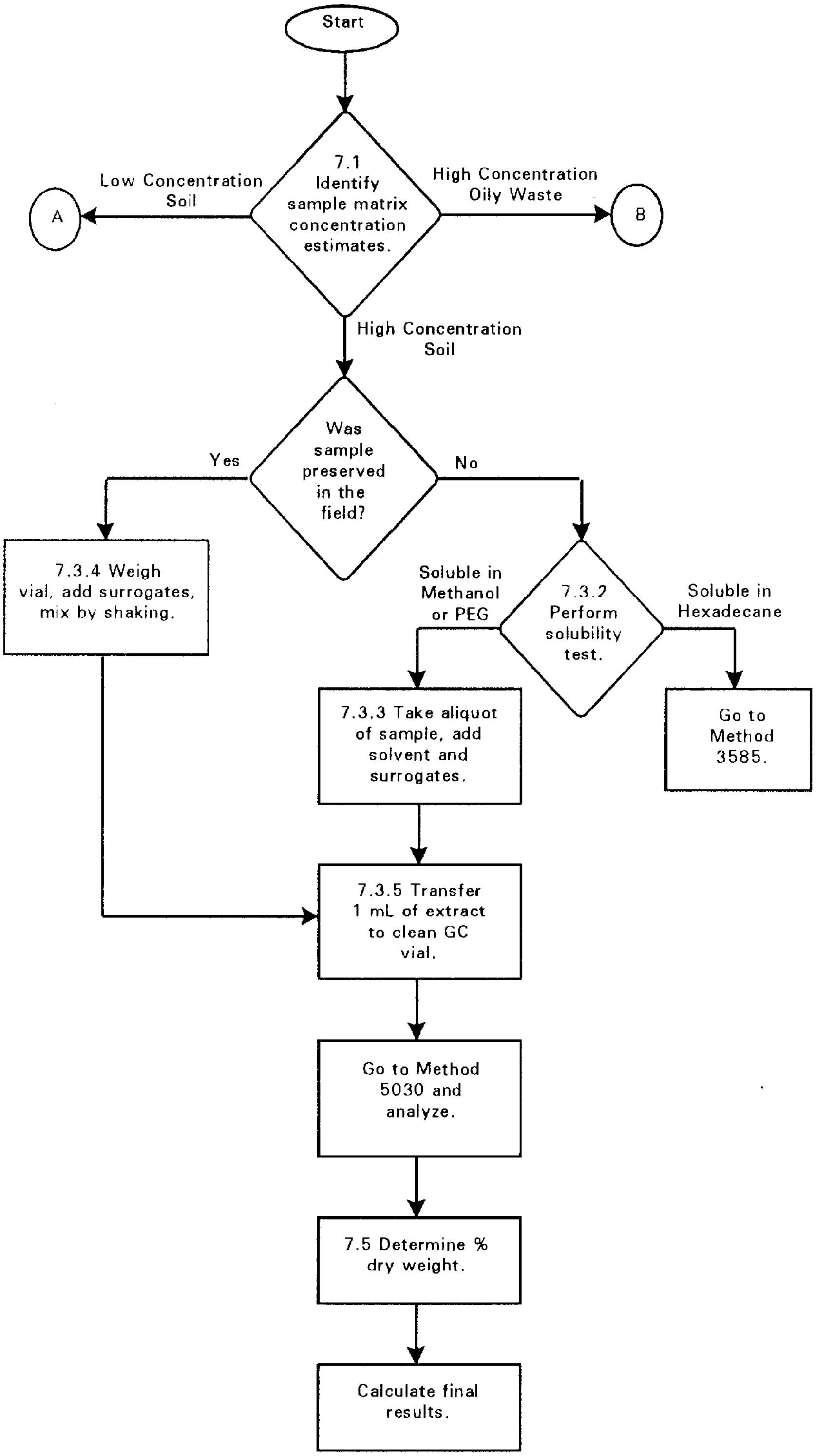
QUANTITY OF METHANOL EXTRACT REQUIRED FOR ANALYSIS OF HIGH CONCENTRATION SOILS/SEDIMENTS

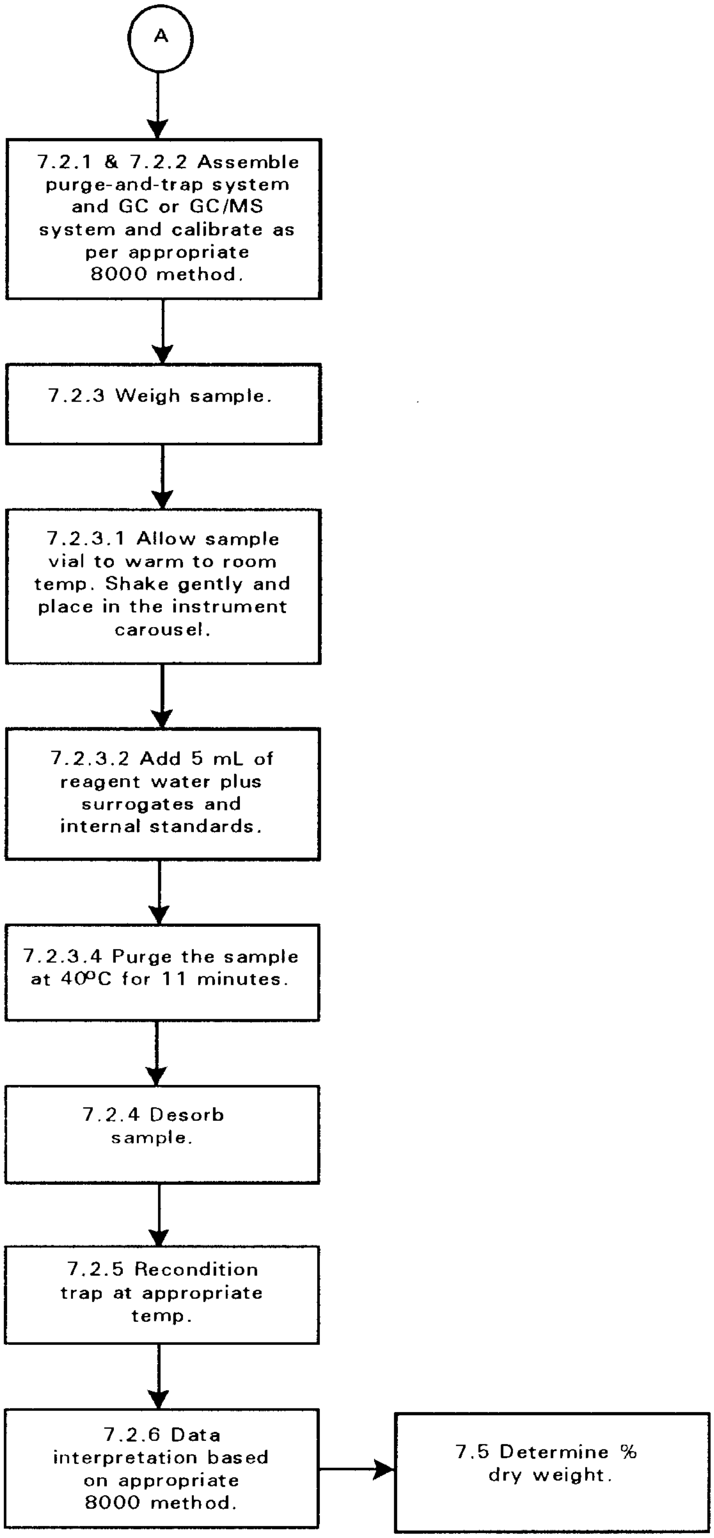
Approximate Concentration Range	Volume of Methanol Extract[a]
500 - 10,000 µg/kg	100 µL
1,000 - 20,000 µg/kg	50 µL
5,000 - 100,000 µg/kg	10 µL
25,000 - 500,000 µg/kg	100 µL of 1/50 dilution[b]

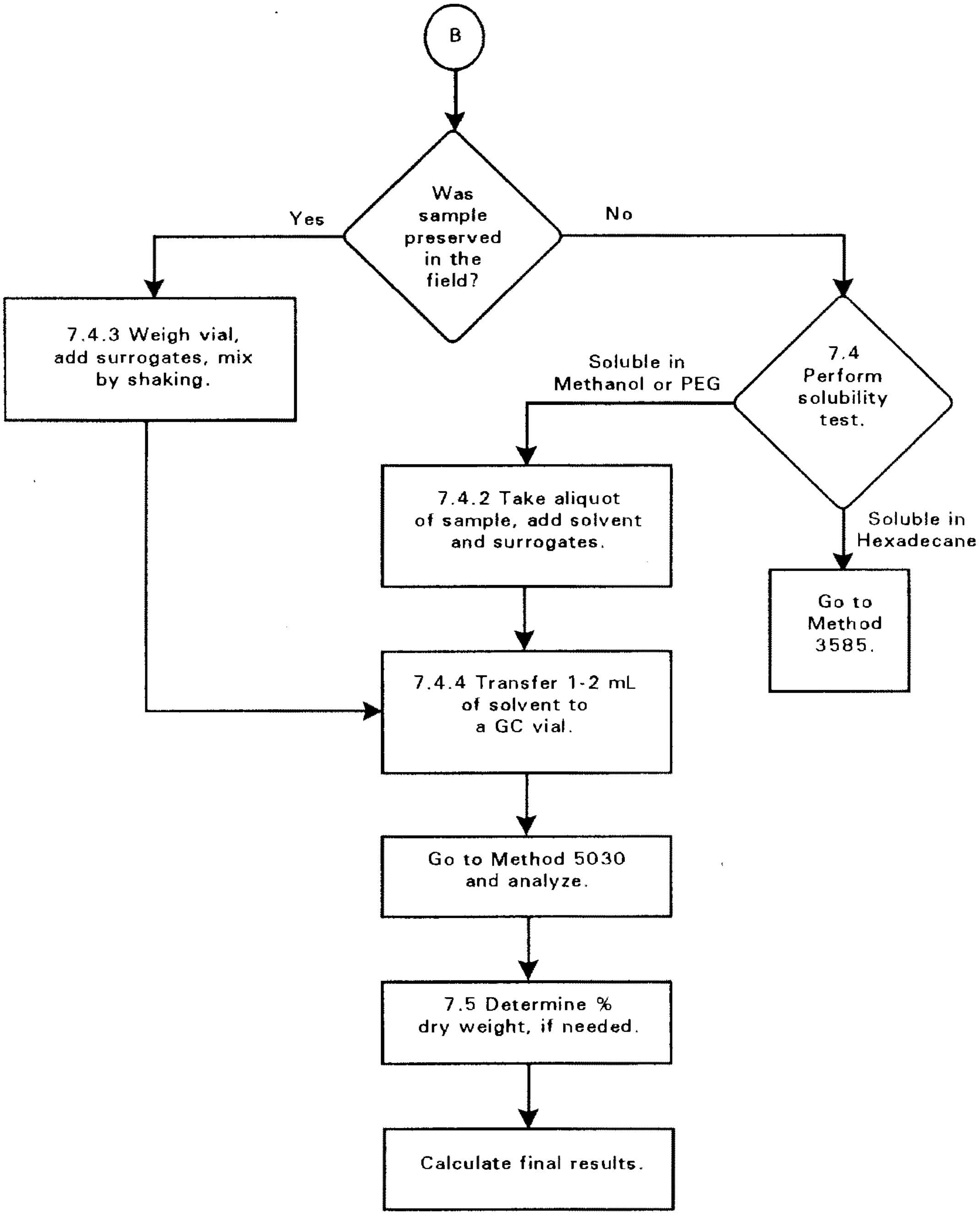
Calculate appropriate dilution factor for concentrations exceeding those in this table.

[a] The volume of methanol added to 5 mL of water being purged should be kept constant. Therefore, add to the 5-mL syringe whatever volume of methanol is necessary to maintain a total volume of 100 µL of methanol.

[b] Dilute an aliquot of the methanol extract and then take 100 µL for analysis.


Start
7.1 Identify sample matrix concentration estimates.
Low Concentration Soil
A
High Concentration Oily Waste
B
High Concentration Soil
Was sample preserved in the field?
Yes
No
7.3.4 Weigh vial, add surrogates, mix by shaking.
Soluble in Methanol or PEG
7.3.2 Perform solubility test.
Soluble in Hexadecane
7.3.3 Take aliquot of sample, add solvent and surrogates.
Go to Method 3585.
7.3.5 Transfer 1 mL of extract to clean GC vial.
Go to Method 5030 and analyze.
7.5 Determine % dry weight.
Calculate final results.


A
7.2.1 & 7.2.2 Assemble purge-and-trap system and GC or GC/MS system and calibrate as per appropriate 8000 method.
7.2.3 Weigh sample.
7.2.3.1 Allow sample vial to warm to room temp. Shake gently and place in the instrument carousel.
7.2.3.2 Add 5 mL of reagent water plus surrogates and internal standards.
7.2.3.4 Purge the sample at 40°C for 11 minutes.
7.2.4 Desorb sample.
7.2.5 Recondition trap at appropriate temp.
7.2.6 Data interpretation based on appropriate 8000 method.
7.5 Determine % dry weight.


B
Was sample preserved in the field?
Yes
No
7.4.3 Weigh vial, add surrogates, mix by shaking.
7.4 Perform solubility test.
Soluble in Methanol or PEG
Soluble in Hexadecane
7.4.2 Take aliquot of sample, add solvent and surrogates.
Go to Method 3585.
7.4.4 Transfer 1-2 mL of solvent to a GC vial.
Go to Method 5030 and analyze.
7.5 Determine % dry weight, if needed.
Calculate final results.

<u>ATTORNEY GENERAL, et al</u> v <u>GELMAN SCIENCES, INC.</u>

(Washtenaw County Circuit Court No. 88-34734-CE)

ATTACHMENT E

Prohibition Zone Institutional Control
Restrictions on Groundwater Use

Pursuant to MCL 324.20121(8) and the Fourth Amended and Restated Consent Judgment, entered in *Attorney General v Gelman Sciences, Inc.,* Washtenaw County Circuit Court Case No. 88-34734-CE, the following land and resource use restrictions shall apply to the "Prohibition Zone" depicted on the map attached hereto:

a. The installation by any person of a new water supply well in the Prohibition Zone for drinking, irrigation, commercial, or industrial use is prohibited.

b. The Washtenaw County Health Officer or any other entity authorized to issue well construction permits shall not issue a well construction permit for any well in the Prohibition Zone.

c. The consumption or use by any person of groundwater from the Prohibition Zone is prohibited.

d. The prohibitions listed in Subsections a–c, above, do not apply to the installation and use of:

i. Groundwater extraction and monitoring wells as part of Response Activities approved by EGLE or otherwise authorized under Parts 201 or 213 of the Natural Resources and Environmental Protection Act ("NREPA"), or other legal authority;

ii. Dewatering wells for lawful construction or maintenance activities, provided that appropriate measures are taken to prevent unacceptable human or environmental exposures to hazardous substances and comply with MCL 324.20107a;

iii. Wells supplying heat pump systems that either operate in a closed loop system or if not, are demonstrated to operate in a manner sufficient to prevent unacceptable human or environmental exposures to hazardous substances and comply with

MCL 324.20107a;

iv. Emergency measures necessary to protect public health, safety, welfare or the environment;

v. Any existing water supply well that has been demonstrated, on a case-by-case basis and with the written approval of EGLE, to draw water from a formation that is not likely to become contaminated with 1,4-dioxane emanating from the Gelman Property. Such wells shall be monitored for 1,4-dioxane by Defendant at a frequency determined by EGLE; and

vi. The City of Ann Arbor's Northwest Supply Well, provided that the City of Ann Arbor operates the Northwest Supply Well in a manner that does not prevent its municipal water supply system from complying with all applicable state and federal laws and regulations.

LEGEND

- MONITOR WELL
- EXTRACTION WELL
- ARTESIAN WELL
- FORMER RESIDENTIAL WELL ROUTINELY MONITORED
- INJECTION WELL
- PROHIBITION ZONE BOUNDARY

NORTH

0 800 1600
SCALE IN FEET

Hard copy is intended to be 24"x36" when plotted.

FLEIS&VANDENBRINK
DESIGN. BUILD. OPERATE.

GELMAN SCIENCES
Scio Twp., Washtenaw County, Michigan

FOURTH AMENDED CONSENT JUDGEMENT

PROJECT MGR.	DATE 05/01/20
DRAWN BY	DATE 05/01/20
GEOLOGIST	DATE 05/01/20
CAD FILE	
EDIT	
SCALE	
DRAWING	
PROJECT	806500
SHEET NO.	

ATTORNEY GENERAL, et al v GELMAN SCIENCES, INC.

(Washtenaw County Circuit Court No. 88-34734-CE)

ATTACHMENT F

On _____, 20__, the Washtenaw County Circuit Court (Court) entered the Fourth Amended and Restated Amendment to Consent Judgment (4th Amended CJ) in the matter of State of Michigan v Gelman Sciences Inc., case number 88-34734-CE (Hon. Timothy P. Connors). The 4th Amended CJ, among other things, provides for an expansion of the area covered by the "Prohibition Zone" previously established by the Third Amendment to Consent Judgment in connection with the groundwater cleanup project being undertaken by Gelman Sciences, Inc., ("Gelman"). The 4th Amended CJ, with limited exceptions, continues to prohibit the consumption or use of groundwater within the "Prohibition Zone" depicted on the map set forth below. The restrictions on groundwater use with in the Prohibition Zone and the map depicting the Prohibition Zone are also set forth at [LINK]. Gelman will provide, at its expense, connection to the City of Ann Arbor municipal water supply to replace any private drinking water wells within the newly established boundaries of the Prohibition Zone that must be abandoned. Such well abandonment and replacement will be performed in accordance with all applicable regulations and procedures at the expense of Gelman. Any private property owner within the Prohibition Zone that is aware of the existence of a water supply well on her or his property should contact Dan Hamel using the contact information listed below to arrange for well abandonment and if applicable, replacement, as provided in the 4th Amended CJ.

Dan Hamel
Project Coordinator
Michigan Department
 of Environment, Great
 Lakes, and Energy,
Remediation and Redevelopment
Division
301 East Louis Glick Highway
Jackson, MI 49201-1556
517-745-6595
HamelD@michigan.gov

You may contact Gelman at:

Lawrence Gelb
Project Coordinator
Gelman Sciences, Inc.
642 S. Wagner Road
Ann Arbor, MI 48106

Prohibition Zone Institutional Control
Restrictions on Groundwater Use

Pursuant to MCL 324.20121(8) and the Fourth Amended and Restated Consent Judgment, entered in *Attorney General v Gelman Sciences, Inc.,* Washtenaw County Circuit Court Case No. 88-34734-CE, the following land and resource use restrictions shall apply to the "Prohibition Zone" depicted on the map below:

a. The installation by any person of a new water supply well in the Prohibition Zone for drinking, irrigation, commercial, or industrial use is prohibited.

b. The Washtenaw County Health Officer or any other entity authorized to issue well construction permits shall not issue a well construction permit for any well in the Prohibition Zone.

c. The consumption or use by any person of groundwater from the Prohibition Zone is prohibited.

d. The prohibitions listed in Subsections a–c, above, do not apply to the installation and use of:

   i. Groundwater extraction and monitoring wells as part of Response Activities approved by EGLE or otherwise authorized under Parts 201 or 213 of the Natural Resources and Environmental Protection Act ("NREPA"), or other legal authority;

   ii. Dewatering wells for lawful construction or maintenance activities, provided that appropriate measures are taken to prevent unacceptable human or environmental exposures to hazardous substances and comply with MCL 324.20107a;

   iii. Wells supplying heat pump systems that either operate in a closed loop system or if not, are demonstrated to operate in a manner sufficient to prevent unacceptable human or environmental exposures to hazardous substances and comply with MCL 324.20107a;

   iv. Emergency measures necessary to protect public health, safety, welfare or the environment;

   v. Any existing water supply well that has been demonstrated, on a case-by-case basis and with the written approval of EGLE, to draw water from a formation that is not likely to become contaminated with 1,4-dioxane emanating from the Gelman Property. Such wells shall be monitored for 1,4-dioxane by Defendant at a frequency determined by EGLE; and

   vi. The City of Ann Arbor's Northwest Supply Well, provided that the City of Ann Arbor operates the Northwest Supply Well in a manner that does not prevent its municipal water supply system from complying with all applicable state and federal laws and regulations.

[Insert Prohibition Zone Map]

ATTORNEY GENERAL, et al v GELMAN SCIENCES, INC.

(Washtenaw County Circuit Court No. 88-34734-CE)

ATTACHMENT G

LEGEND

- ⌖ - MONITOR WELL
- ⊕ - EXTRACTION WELL
- △ - ARTESIAN WELL
- ⊡ - FORMER RESIDENTIAL WELL ROUTINELY MONITORED
- ⊸ - INJECTION WELL

- ▬ - PROHIBITION ZONE BOUNDARY
- ╍ - APPROXIMATE EXTRACTION WELL AREA
- ━ - DOWNGRADIENT INVESTIGATION AREA

- ⊗ - APPROXIMATE LOCATION OF WESTERN AREA DELINEATION WELL
- ⦶ - APPROXIMATE LOCATION OF SENTINEL WELL
- ⊘ - APPROXIMATE LOCATION OF PZ BOUNDARY WELL
- ⊖ - APPROXIMATE LOCATION OF EASTERN AREA DELINEATION WELLS

NORTH

0 800 1600
SCALE IN FEET

Hard copy is intended to be 24"x36" when plotted.

FLEIS&VANDENBRINK
DESIGN. BUILD. OPERATE.

GELMAN SCIENCES
Scio Twp., Washtenaw County, Michigan

FOURTH AMENDED CONSENT JUDGEMENT - ATTACHMENT G

PROJECT MGR.	DATE 05/01/20
DRAWN BY	DATE 05/01/20
GEOLOGIST	DATE 05/01/20
CAD FILE	
EDIT	
SCALE	
DRAWING	
PROJECT	806500
SHEET NO.	

ATTORNEY GENERAL, et al v GELMAN SCIENCES, INC.

(Washtenaw County Circuit Court No. 88-34734-CE)

ATTACHMENT H

OPTIONS ARRAY

Pursuant to Section V.A.10 of the Consent Judgment, as amended, Gelman is submitting this Options Array, which sets forth various options for addressing the potential, if unlikely, risks that:

1. Gelman requires more extraction/treatment capacity to maintain compliance with the Eastern Area objectives than the 200 gpm provided by the current infrastructure; and

2. The northern portion of the deep transmission line fails.[1]

Gelman believes that each of the options discussed below is "implementable" within the identified limitations and subject to obtaining the necessary approvals and/or Court-ordered access. Obviously, the necessary approvals and access rights can only be sought if and when there is an actual set of circumstances that gives rise to the need for such approvals/access. Gelman reserves the right to identify additional alternatives if and when such a specific situation arises.

SCENARIO 1 MORE THAN THE CURRENTLY AVAILABLE 200 GALLONS PER MINUTE IS NEEDED TO MEET EASTERN AREA OBJECTIVES

The deep transmission line currently allows Gelman to convey up to 200 gallons per minute (gpm) from the Evergreen Subdivision and Maple Road areas back to the Wagner Road facility for treatment and then disposal via Gelman's permitted surface water discharge. The following alternatives are options for addressing the possibility that Gelman will need to extract more than a total of 200 gpm to meet its Eastern Area cleanup objectives, excluding groundwater extracted from the proposed Parklake Well.

Alternative A: Treatment and Groundwater Injection in Maple Road or Alternative Area

Description. Gelman could utilize a mobile treatment unit similar to that previously used in the Maple Village area along with injection wells to treat and dispose of water. This process was employed previously in the Evergreen and Maple Village areas.

Limitations: This option will take time to implement. Injection locations will need to be identified and necessary permits obtained, infrastructure would need to be installed and, if the existing mobile treatment unit was still in use in connection with the Parklake Well, an additional unit would need to be constructed.

Alternative B: Treatment and Discharge to Ann Arbor Sanitary Sewer System

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[1] Gelman already has in place a redundant near-surface pipeline that could replace the capacity of the Southern transmission line (the portion that begins at the Porter Lot) in the event that part of the transmission line fails.

Description: Discharge of treated water into the sanitary sewer is a possible method of handling additional water beyond the 200 gpm capacity of the deep transmission line. This alternative would involve treatment of the groundwater by a mobile unit and then disposal of the treated groundwater into the City's sanitary sewer. The location of the sewer connection would depend on where the groundwater was extracted and the availability of the necessary City infrastructure.

Limitations: This disposal method would have to be authorized by the City of Ann Arbor. The City Council has previously adopted a resolution that would require Gelman to treat the groundwater to below 3 ppb of 1,4-dioxane before discharging to the sanitary sewer. A mobile unit would utilize ozone to treat 1,4-dioxane contaminated groundwater, which would generate low levels of bromate as a bi-product, particularly if required to treat to such a low level for 1,4-dioxane. Gelman cannot predict how the City would react to a request for such a discharge. In addition, when this discharge option was evaluated in connection with the Unit E Feasibility Analysis, the City informed Gelman that there was insufficient capacity in the sewer system for the high volume of water that would be needed to address that plume. The City would need to confirm what, if any, capacity would exist for this alternative to be feasible. Moreover, costs for this alternative are expected to be high because of the need to operate a mobile treatment system and the cost of sewer fees. This alternative will likely not be implementable due to likely treatment requirements and/or capacity limitations except for low flow and/or temporary situations.

Alternative C Treatment and Discharge to Ann Arbor Storm Sewer

Description: Discharge of treated water into the City's storm sewer is also a possible alternative. This alternative would involve treatment of the groundwater by a mobile treatment unit and then disposal of the treated groundwater into the City's storm sewer. The location of the sewer connection and discharge point would depend on where the groundwater was extracted and the availability of the necessary City infrastructure.

Limitations: The storm sewer system has well-documented capacity limitations. This alternative would require approval from the City of Ann Arbor, the Washtenaw County Drain Commissioner and the State of Michigan, and the installation of the necessary infrastructure to connect to the system. It is likely that this alternative would require flow (discharge) into the storm to be temporally suspended during times when the storm sewer is at or near capacity, such as during storm events. Given the capacity concerns and the governmental approvals that would be needed, this alternative may only be implementable in low flow and/or temporary situations.

Alternative D New Pipeline from Maple Road or Evergreen Area - Treatment at Wagner Road Facility

Description: A new, near-surface, pipeline could be installed to connect the Evergreen Subdivision or Maple Road areas to the Wagner Road facility for treatment. Approximately 600 gpm of treatment capacity would be available to treat water from the Eastern Area (not including groundwater from the Parklake area). It is anticipated that this treatment capacity would be sufficient to accommodate any foreseeable necessary flow from these areas and the pipeline could be sized appropriately. A feasibility study would need to be conducted to determine the best route for the line.

Limitations: This option may be cost effective if additional capacity needs are relatively high (greater than 100 gpm) and the need for the capacity is long term. This option would require right-of-way access from the City and potentially, Scio Township and MDOT or court-ordered access. This option would require significant construction time before it could be implemented.

Future Alternatives

Gelman reserves the right to identify additional alternatives if and when a specific situation requiring capacity beyond that provided by the current infrastructure arises.

SCENARIO 2 NORTH HORIZONTAL TRANSMISSION PIPELINE FAILS

The northern portion of the deep horizontal transmission line is a HDPE pipeline that Gelman inserted into the original northern horizontal well after the original steel transmission pipeline failed in 2008. Gelman has supplied documentation of the HDPE pipeline's 50 year life expectancy. To supplement this information, Gelman has identified the following alternatives, which are options for addressing the possibility that the pipeline fails despite its expected reliability.

Alternative A: Treatment and Groundwater Injection in Maple Road or Alternative Area

Description. Gelman could utilize a mobile treatment unit similar to that previously used in the Maple Village area along with injection wells to treat and dispose of water. This process was employed previously in the Evergreen and Maple Village areas.

Limitations: This option will take time to implement. Injection locations will need to be identified and necessary permits obtained, and infrastructure would need to be installed.

Alternative B: Treatment and Discharge to Ann Arbor Sanitary Sewer System

Description: Discharge of treated water into the sanitary sewer is a possible method of handling additional water beyond the 200 gpm capacity of the deep transmission line. This alternative would involve treatment of the groundwater by a mobile unit and then disposal of the treated groundwater into the City's sanitary sewer. The location of the sewer connection would depend on where the groundwater was extracted and the availability of the necessary City infrastructure.

Limitations: This disposal method would have to be authorized by the City of Ann Arbor. The City Council has previously adopted a resolution that would require Gelman to treat the groundwater to below 3 ppb of 1,4-dioxane before discharging to the sanitary sewer. A mobile unit would utilize ozone to treat 1,4-dioxane contaminated groundwater, which would generate low levels of bromate as a bi-product, particularly if required to treat to such a low level for 1,4-dioxane. Gelman cannot predict how the City would react to a request for such a discharge. In addition, when this discharge option was evaluated in connection with the Unit E Feasibility Analysis, the City informed Gelman that there was insufficient capacity in the sewer system for the high volume of water that would be needed to address that plume. The City would need to confirm what, if any, capacity would exist for this alternative to be feasible. Moreover, costs for this alternative are expected to be high because of the need to operate the mobile treatment system and the cost of sewer fees. This alternative will likely not be implementable due to likely treatment requirements and/or capacity limitations except for low flow and/or temporary situations.

Alternative C Treatment and Discharge to Ann Arbor Storm Sewer

Description: Discharge of treated water into the City's storm sewer is also a possible alternative. This alternative would involve treatment of the groundwater by a mobile treatment unit and then disposal of the treated groundwater into the City's storm sewer. The location of the sewer connection and discharge point would depend on where the groundwater was extracted and the availability of the necessary City infrastructure.

Limitations: The storm sewer system has well-documented capacity limitations. This alternative would require approval from the City of Ann Arbor, the Washtenaw County Drain Commissioner and the State of Michigan, and the installation of the necessary infrastructure to connect to the system. It is likely that this alternative would require flow (discharge) into the storm to be temporally suspended during times when the storm sewer is at or near capacity, such as during storm events. Given the capacity concerns and the governmental approvals that would be needed, this alternative may only be implementable in low flow and/or temporary situations.

Alternative D New Pipeline from Maple Road or Evergreen Area - Treatment at Wagner Road Facility

Description: A new, near-surface, pipeline could be installed to connect the Evergreen Subdivision or Maple Road areas to the Wagner Road facility for treatment. Approximately 600 gpm of treatment capacity would be available to treat water from the Eastern Area (not including groundwater from the Parklake area). It is anticipated that this treatment capacity would be sufficient to accommodate any foreseeable necessary flow from these areas and the pipeline could be sized appropriately. A feasibility study would need to be conducted to determine the best route for the line.

Limitations: This option may be cost effective if additional capacity needs are relatively high (greater than 100 gpm) and the need for the capacity is long term. This option would require right-of-way access from the City and potentially, Scio Township and MDOT or court-ordered access. This option would require significant construction time before it could be implemented.

Future Alternatives

As noted above, Gelman reserves the right to identify additional alternatives if and when a specific situation affecting the availability of the transmission line arises. For example, when the original transmission line failed, the parties determined that it was leaking in an already contaminated portion of the aquifer and agreed that it could continue to operate while repairs were made, with appropriate monitoring. Similar fact-specific alternatives will likely be identified if and when such a contingency arises.

ATTORNEY GENERAL, et al v GELMAN SCIENCES, INC.

(Washtenaw County Circuit Court No. 88-34734-CE)

ATTACHMENT I

Attachment I: Target Treatment Areas
Gelman Sciences
Washtenaw County
Michigan

ATTORNEY GENERAL, et al v GELMAN SCIENCES, INC.

(Washtenaw County Circuit Court No. 88-34734-CE)

ATTACHMENT J

Attachment J: Former Burn Pit Target Treatment Area
Gelman Sciences
Washtenaw County
Michigan

ATTORNEY GENERAL, et al v GELMAN SCIENCES, INC.

(Washtenaw County Circuit Court No. 88-34734-CE)

ATTACHMENT K

BNP PARIBAS
CORPORATE & INVESTMENT BANKING

BNP PARIBAS
TRADE FINANCE OPERATIONS
787 SEVENTH AVENUE
NEW YORK, NY 10019

AUGUST 5, 2014

APPLICANT:
GELMAN SCIENCES INC
600 WAGNER ROAD
ANN ARBOR, MI 48103-9002 USA

BENEFICIARY:
CHIEF, REMEDIATION DIVISION, MICHIGAN DEPARTMENT
OF NATURAL RESOURCES AND ENVIRONMENT, PO BOX
30426, LANSING, MICHIGAN 48909-7926, FEDERAL TAX
IDENTIFICATION NO. 38-6000134 (STATE OF MICHIGAN)
PO BOX 30426, LANSING
MICHIGAN 48909-7926, USA

WE HEREBY AMEND OUR IRREVOCABLE STANDBY LETTER OF CREDIT NO. 04126179 DATED DECEMBER 5, 2013, IN YOUR FAVOR AS FOLLOWS:

1) REPLACE BENEFICIARY NAME AND ADDRESS :
DELETE : BENEFICIARY:
CHIEF, REMEDIATION DIVISION, MICHIGAN DEPARTMENT
OF NATURAL RESOURCES AND ENVIRONMENT, PO BOX
30426, LANSING, MICHIGAN 48909-7926, FEDERAL TAX
IDENTIFICATION NO. 38-6000134 (STATE OF MICHIGAN)
PO BOX 30426, LANSING
MICHIGAN 48909-7926, USA
.
INSERT : BENEFICIARY:
CHIEF, REMEDIATION AND REDEVELOPMENT DIVISION, MICHIGAN
DEPARTMENT OF ENVIRONMENTAL QUALITY
P O BOX 30426
LANSING, MICHIGAN 48909-7926
FEDERAL TAX IDENTIFICATION NO. 38-6000134 (STATE OF MICHIGAN)
.
2) IN SUBJECT HEADING LINE THREE REPLACE SITE ID NUMBER:
DELETE : SITE ID NO. MID005341813
INSERT : SITE ID NO. 81000018
.
3) IN PARAGRAPH 1 REPLACE BENEFICIARY NAME:
DELETE : 'MICHIGAN DEPARTMENT OF NATURAL RESOURCES AND ENVIRONMENT (DEPARTMENT) ON BEHALF OF GELMAN SCIENCES INC.'
INSERT: 'MICHIGAN DEPARTMENT OF ENVIRONMENTAL

Page: 2
Reference No.: 04126179

QUALITY(DEPARTMENT)' ON BEHALF OF GELMAN SCIENCES INC.

.

4) IN PARAGRAPH 2 REPLACE SITE ID NUMBER :
DELETE : MID00534818138
INSERT : 81000018

.

5) REPLACE PARAGRAPH 3 :
DELETE : THE LOC SHALL BE AUTOMATICALLY EXTENDED .... AS EVIDENCED BY THE RETURN CERTIFIED MAIL RECEIPTS.

.

INSERT : THIS LOC IS EFFECTIVE AS OF DECEMBER 5, 2013, AND SHALL EXPIRE ON DECEMBER 5, 2014, BUT SUCH LOC SHALL BE AUTOMATICALLY EXTENDED FOR A PERIOD OF ONE YEAR EACH AND EVERY SUBSEQUENT YEAR UNLESS, NOT LESS THAN ONE HUNDRED AND TWENTY (120) DAYS BEFORE THE EXTENDED EXPIRATION DATE, WE NOTIFY THE DESIGNATED PARTY AND THE DEPARTMENT AUTHORIZED REPRESENTATIVE AS INDICATED ABOVE. WE AGREE THAT THE ONE HUNDRED AND TWENTY (120) DAY PERIOD SHALL BEGIN ON THE DATE WHEN BOTH THE DESIGNATED PARTY AND THE DEPARTMENT AUTHORIZED REPRESENTATIVE HAVE RECEIVED THE NOTICE, AS EVIDENCED BY THE RETURN CERTIFIED MAIL RECEIPTS.

.

6) IN PARAGRAPH 6 READ THE WORD 'UTOMATICALLY' AS ''AUTOMATICALLY''

.

ALL OTHER TERMS AND CONDITIONS REMAIN UNCHANGED.

PLEASE SIGN BELOW TO SIGNIFY YOUR ACCEPTANCE TO THIS AMENDMENT AND FAX RETURN IT TO US TO ATTN: TRADE FINANCE SERVICES AT FAX NO.: (201) 616-7913.

AMENDMENT ACCEPTED:

____
AUTHORIZED SIGNATURE

CERTAIN ADMINISTRATIVE SERVICES FOR BNP PARIBAS MAY BE PROVIDED BY BNP PARIBAS RCC, INC., BNP PARIBAS, THROUGH ITS CANADA BRANCH, OR ANY DIRECT OR INDIRECT MAJORITY OWNED SUBSIDIARY OF BNP PARIBAS.

BNP PARIBAS
BY: BNP PARIBAS RCC, INC., AS AUTHORIZED AGENT

AUTHORIZED SIGNATURE

AUTHORIZED SIGNATURE

```
---------------------  Instance Type and Transmission --------------
 Copy received from SWIFT
 Priority                   : Normal
 Message Output Reference : 1100 140801BNPAUS3NCXXX0109389933
 Correspondent Input Reference : 1600 140801BNPAGB22CXXX5823667961
--------------------------- Message Header -------------------------
 Swift Output : FIN 767 Guar/Stdby Letter Cred Amendnt
 Sender    : BNPAGB22XXX
             BNP PARIBAS LONDON BRANCH
             LONDON GB
 Receiver : BNPAUS3NXXX
             BNP PARIBAS USA- NEW YORK
             NEW YORK US
 MUR : 1919F8213A730000
--------------------------- Message Text ---------------------------
  27: Sequence of Total
      1/1
  20: Transaction Reference Number
      LAD/GTEE/13/1030
  21: Related Reference
      04126179
  23: Further Identification
      REQUEST
  30: Date
      140801
 26E: Number of Amendment
      1
 31C: Date of Issue / Request to Issue
      131204
 77C: Amendment Details
      APPLICANT. GELMAN SCIENCES INC
      BENEFICIARY. CHIEF, REMEDIATION DIVISION, MICHIGAN DEPARTMENT OF
      NATURAL RESOURCES AND ENVIRONMENT
      AMOUNT. USD28,431,846.00
      .
      KINDLY AMEND THE ABOVE-MENTIONED STANDBY LETTER OF CREDIT AS
      FOLLOWS:
      .
      1) REPLACE BENEFICIARY NAME AND ADDRESS :
      DELETE : BENEFICIARY:
      CHIEF, REMEDIATION DIVISION, MICHIGAN DEPARTMENT
      OF NATURAL RESOURCES AND ENVIRONMENT, PO BOX
      30426, LANSING, MICHIGAN 48909-7926, FEDERAL TAX
      IDENTIFICATION NO. 38-6000134 (STATE OF MICHIGAN)
      PO BOX 30426, LANSING
      MICHIGAN 48909-7926, USA
      .
      INSERT : BENEFICIARY:
      CHIEF, REMEDIATION AND REDEVELOPMENT DIVISION, MICHIGAN
      DEPARTMENT OF ENVIRONMENTAL QUALITY
      P O BOX 30426
      LANSING, MICHIGAN 48909-7926
      FEDERAL TAX IDENTIFICATION NO. 38-6000134 (STATE OF MICHIGAN)
      .
      2) IN SUBJECT HEADING LINE THREE REPLACE SITE ID NUMBER :
      DELETE : SITE ID NO. MID005341813
      INSERT : SITE ID NO. 81000018
```

ORIGINAL
SWIFT MESSAGE

COMPLIANCE CHECKED
1st CHECKED Initials DATE 8/5/14
2nd CHECKED Initials DATE 8/5/14

.
3) IN PARAGRAPH 1 REPLACE BENEFICIARY NAME:
DELETE : 'MICHIGAN DEPARTMENT OF NATURAL RESOURCES AND
ENVIRONMENT (DEPARTMENT) ON BEHALF OF GELMAN SCIENCES INC.'
INSERT: 'MICHIGAN DEPARTMENT OF ENVIRONMENTAL
QUALITY(DEPARTMENT)' ON BEHALF OF GELMAN SCIENCES INC.
.
4) IN PARAGRAPH 2 REPLACE SITE ID NUMBER :
DELETE : MID00534818138
INSERT : 81000018
.
5) REPLACE PARAGRAPH 3 :
DELETE : THE LOC SHALL BE AUTOMATICALLY EXTENDED .... AS
EVIDENCED BY THE RETURN CERTIFIED MAIL RECEIPTS.

.
INSERT : THIS LOC IS EFFECTIVE AS OF DECEMBER 5, 2013, AND SHALL EXPIRE ON DECEMBER 5, 2014, BUT SUCH LOC SHALL BE AUTOMATICALLY EXTENDED FOR A PERIOD OF ONE YEAR EACH AND EVERY SUBSEQUENT YEAR UNLESS, NOT LESS THAN ONE HUNDRED AND TWENTY (120) DAYS BEFORE THE EXTENDED EXPIRATION DATE, WE NOTIFY THE DESIGNATED PARTY AND THE DEPARTMENT AUTHORIZED REPRESENTATIVE AS INDICATED ABOVE. WE AGREE THAT THE ONE HUNDRED AND TWENTY (120) DAY PERIOD SHALL BEGIN ON THE DATE WHEN BOTH THE DESIGNATED PARTY AND THE DEPARTMENT AUTHORIZED REPRESENTATIVE HAVE RECEIVED THE NOTICE, AS EVIDENCED BY THE RETURN CERTIFIED MAIL RECEIPTS.
.
6) IN PARAGRAPH 6 READ THE WORD 'UTOMATICALLY' AS
''AUTOMATICALLY''
.
ALL OTHER TERMS AND CONDITIONS REMAIN UNCHANGED.
.
KINDLY DELIVER THE ORIGINAL AMENDMENT BY COURIER TO :
.
PALL CORPORATION
25 HARBOR PARK DRIVE
PORT WASHINGTON
NY 11050
USA
ATTN. JOHN GRUBER, TREASURY DIRECTOR
PHONE. +15168019494
.
PLEASE FORWARD A COPY OF THE AMENDMENT TO BNP PARIBAS,
10 HAREWEOOD AVENUE, LONDON NW1 6AA, ATTN.LOANS ADMIN/GUARANTEES

-------------------------- Message Trailer ------------------------

{CHK:7178F9C913A8}

ATTORNEY GENERAL, et al v GELMAN SCIENCES, INC.

(Washtenaw County Circuit Court No. 88-34734-CE)

ATTACHMENT L

FINANCIAL TEST AND FINANCIAL TEST/CORPORATE GUARANTEE PART 201

**Prior to use contact Mr. Brad Ermisch, Compliance and Enforcement Section, Remediation and Redevelopment Division (RRD), at ermischb@michigan.gov or 517-275-1173 for any questions relating to this document or the attached model document; or you may call the RRD main number at 517-284-5087 for assistance.

This document provides instructions on the use of the Financial Test (FT) or Financial Test/Corporate Guarantee (FT/CG) to fulfill the requirements for financial assurance pursuant to Section 20114d(4)(b) of Part 201, Environmental Remediation, of the Natural Resources and Environmental Protection Act, 1994 PA 451, as amended (NREPA), MCL 324.20101 *et seq*. This document and attached model documents are provided to the public as preliminary guidance as to the content, format, and terms of the Financial Assurance Mechanism and are not intended, nor can they be relied upon to create any substantive or procedural rights by any other party.

Pursuant to Section 20114d of the NREPA, upon completion of remedial actions that satisfy the requirements of Part 201, a person may submit a No Further Action Report (NFA Report) to the Michigan Department of Environment, Great Lakes, and Energy (EGLE). If a postclosure agreement (Agreement) is required as part of the NFA Report, Section 20114d(4)(b) requires financial assurance to pay for monitoring, operation and maintenance, oversight, and other costs determined by EGLE to be necessary to assure the effectiveness and integrity of the remedial action unless the financial assurance is de minimis. The de minimis threshold is $2,500 per year in 2001 dollars. A link to a Consumer Price Index Inflation Calculator is provided to determine if the current annual costs exceed the 2001 dollar value: CPI Inflation Calculator. Section 20101(u) of the NREPA, defines financial assurance as a performance bond, escrow, cash, certificate of deposit, irrevocable letter of credit, corporate guarantee, or other equivalent security, or combination thereof. EGLE has determined that the FT and the FT/CG are acceptable FAMs. The FT allows for the financial strength of a company to be used as a FAM. If a company cannot meet the requirements for the FT and is a subsidiary, it may rely on its parent company's financial strength to meet the FT requirements; however, the parent company must agree to assume responsibility for the FAM on behalf of its subsidiary.

If a person elects to use the FT to meet its financial assurance obligations (Designated Party), the Designated Party must meet the following requirements:

1. Pass the Standard Financial Test using either Alternative I or II (See Appendix A).

2. Provide a letter signed by the Designated Party's chief financial officer (CFO) that is worded in accordance with the language in Appendix B and include the documents referenced therein.

If a person elects to use the FT/CG, the parent company must:

1. Pass the Standard Financial Test using either Alternative I or II (See Appendix A).

2 Provide a letter signed by the CFO of the parent company that is worded in accordance with the language provided in Appendix B and include the documents referenced therein.

3. Submit a Corporate Guarantee in accordance with Appendix D.

Drafting Instructions: Copy and paste the text portion of the model documents onto appropriate letterhead. Drafting notes and examples appear as ***italicized bold font***, insertion directions appear as **[*italicized bold font within bold brackets*]**, and word choices appear as **[regular bold font within bold brackets]**.

--END OF GUIDANCE AND INSTRUCTIONS--

Appendix A
STANDARD FINANANCIAL TEST MODEL

STANDARD FINANCIAL TEST

The figures for the following items marked with an (*) are to be identified as to the source of the information for the company. The preferred source is the independently audited year-end financial statements from the latest fiscal year. Also create a header for this document identifying the purpose and parties represented by the standard financial test. And this test must be renewed thirty days following the close and publication of financial information or an alternative financial assurance document is to be provided to EGLE.

ALTERNATIVE I

1. Sum of the current cost estimates for response activities needed at Michigan facilities, including the cost for operation and maintenance of remedial actions for the next 30-year time period. $___________

2. Sum of the current cost estimates for response activities needed at non-Michigan facilities, including the cost for operation and maintenance of remedial actions. $___________

3. Sum of lines 1 and 2. $___________

*4. Total liabilities [if any portion of the cost estimates for response activities (lines 1 or 2) is included in total liabilities, you may deduct that amount from this line and add that amount to lines 5 and 6]. $___________

*5. Tangible net worth. $___________

*6. Net worth. $___________

*7. Current assets. $___________

*8. Current liabilities. $___________

9. Net working capital [line 7 minus line 8]. $___________

*10. The sum of net income plus depreciation, depletion and amortization. $___________

*11. Total assets in the United States. $___________

*12. Total assets in Michigan, excluding the value of all real property on which response activities are necessary. $___________

*13. Total assets in Michigan, including the value of all real property on which response activities are necessary. $___________

	YES	NO
14. Is line 5 at least $10 million?	___	___
15. Is line 5 at least 6 times line 3?	___	___
16. Is line 9 at least 6 times line 3?	___	___
*17. Are at least 90% of the company's assets located in the United States? If not, complete line 18.	___	___
18. Is line 11 at least 6 times line 3?	___	___
19. Is line 4 divided by line 6 less than 2.0?	___	___
20. Is line 10 divided by line 4 greater than 0.1?	___	___
21. Is line 7 divided by line 8 greater than 1.5?	___	___
*22. Is line 12 at least $50 million?	___	___
23. Is line 13 at least 6 times line 1?	___	___

To “pass” Alternative I of the standard financial test, the company must meet two out of three of the ratios listed in lines 19, 20, and 21; meet the criterion of either line 17 or line 18; meet the criteria listed in lines 14, 15, and 16; and meet the criterion of either line 22 or 23.

ALTERNATIVE II

1. Sum of the current cost estimates for response activities needed at Michigan facilities, including the cost for operation and maintenance of remedial actions for the next 30-year time period. $___________

2. Sum of the current cost estimates for response activities needed at non-Michigan facilities, including the cost for operation and maintenance of remedial actions. $___________

3. Sum of lines 1 and 2. $___________

4. Current bond rating of most recent issuance for this company and name of rating service. ____________

5. Date of issuance of bond. ____________

6. Date of maturity of bond. ____________

*7. Tangible net worth (if any portion of the cost estimates for response activities (lines 1 and 2) is included in “total liabilities” on your financial statements, you may add that portion to this line). $___________

*8. Total assets in the United States. $___________

*9. Total assets in Michigan, excluding the value of all real property on which response activities are necessary. $___________

*10. Total assets in Michigan, including the value of all real property on which response activities are necessary. $___________

	YES	NO
11. Is line 7 at least $10 million?	___	___
12. Is line 7 at least 6 times line 3?	___	___
*13. Are at least 90% of company's assets located in the United States? If not, complete line 14.	___	___
14. Is line 8 at least 6 times line 3?	___	___
*15. Is line 9 at least $50 million?	___	___
16. Is line 10 at least 6 times line 1?	___	___

To “pass” Alternative II of the standard financial test, the company must have a current rating for the most recent bond issuance of AAA, AA, A, or BBB for Standard and Poor’s or Aaa, Aa, A, or Baa for Moody’s; meet the criterion of either line 13 or line 14; meet the criteria listed in lines 11 and 12; and meet the criterion of either line 15 or 16.

[*Insert the following at the end of the Standard Financial Test that you chose to use*]

I hereby certify that the wording of this form is a true copy of the model financial test provided by the Michigan Department of Environment, Great Lakes, and Energy (EGLE), with the exception of any changes made and agreed to by representatives of EGLE and **[*insert name of company*]**.

____
Chief Financial Officer

____
Name of Company

Date: __________________________

Signed and sealed
in the presence of:

____
NOTARY PUBLIC

Notary Public ______________ County
My Commission Expires ___________

Appendix B
LETTER FROM CHIEF FINANCIAL OFFICER
FOR FINANCIAL TEST or FINANCIAL TEST/CORPORATE GUARANTEE MODEL

[***Insert name of Remediation and Redevelopment Division (RRD) Director***], Director
Remediation and Redevelopment Division
Michigan Department of Environment, Great Lakes, and Energy
P.O. Box 30426
Lansing, MI 48909-76115

Dear [***Insert name of RRD Director***]:

I am the chief financial officer of [***insert name of company or name of parent company if Financial Test/Corporate Guarantee (FT/CG)***], [***insert address***].

The [***insert name of company or, for FT/CG, name of subsidiary***] is liable under Part 201, Environmental Remediation, of the Natural Resources and Environmental Protection Act, 1994 PA 451, as amended (NREPA), for the [***insert name of facility***] located at [***insert address of facility***] that is the subject of a postclosure agreement submitted as part of a no further action report to the Michigan Department of Environment, Great Lakes, and Energy (EGLE) on [***insert date of no further action report***]. Pursuant to the postclosure agreement, [***insert name of company***] has agreed to [***briefly describe response activities required by the Order/Decree/Agreement and any other obligations as necessary***] at the [***insert name of facility***].

In order for EGLE to approve implementation of these response activities, EGLE requires that [***insert name of company***] provide financial assurance to assure performance of the necessary and appropriate response activities to protect public health, safety, and welfare, and to assure the effectiveness and integrity of the remedial action at the facility.

For a company that is providing its own financial test, insert the following paragraph

This letter is in support of [***insert name of company***]'s use of the financial test to satisfy the financial assurance requirements of Part 201 for the [***insert name of facility***]**.**

For a parent company that is providing a FT/CG for its subsidiary, insert the following paragraph

This company is the parent corporation of [***insert name of subsidiary that is the beneficiary of the FT/CG***]. This letter is in support of [***insert name of parent company***]'s use of the financial test and financial test/corporate guarantee to satisfy the financial assurance requirements of Part 201 for the [***insert name of facility***].

This company has prepared a Standard Financial Test-Alternative [***insert as appropriate:* I *or* II**] (SFT) using EGLE model SFT and has passed that test as shown in the attached SFT document. The estimated annual cost of response activities to be performed at this facility as reflected in the SFT is [***insert estimated annual cost amount***].

With this letter, I also am submitting the following items to demonstrate to EGLE that [***insert name of company***] meets the requirements for using the [***Insert as appropriate:* financial test *or* financial test and corporate guarantee**] as its financial assurance mechanism:

1. A copy of an independent certified public accountant's report for the latest fiscal year for [***insert as appropriate: name of company or parent company***]. The fiscal year of this firm ends on [***insert date of end of company's fiscal year***].

   NOTE: Please provide a footnote explaining line items in the financial test that deviate from the amounts given in the audited year-end financial statements.

   and

2. A letter from an independent certified public accountant certifying its review of this letter and this company's financial statements. ***See Appendix C***

This company **[*insert as appropriate:* is *or* is not]** required to file Form 10K with the Securities and Exchange Commission for the latest completed fiscal year which ended [***insert date***].

I hereby certify that the wording of this letter is identical to the model letter provided by EGLE, with the exception of any changes that have been made with the concurrence of representatives of EGLE and [***insert as appropriate: name of company or parent company***].

____
Chief Financial Officer

____
[*Name of Company or Parent Company*]

Date: __________________________

Attachments

Signed and sealed
in the presence of:

____
NOTARY PUBLIC

Notary public ____________ County
My commission expires: ___________

Appendix C
FINANCIAL TEST or FINANCIAL TEST/CORPORATE GUARANTEE REPORT OF THE INDEPENDENT CERTIFIED PUBLIC ACCOUNTANT MODEL

[*Insert name of Chief Financial Officer* (CFO)]
[*Insert name and address of Company*]

Dear **[*insert name of CFO*]**:

We have audited, in accordance with generally accepted auditing standards, the financial statements of **[*insert as appropriate: name of company or parent company*]** for its fiscal year ending **[*insert fiscal year end date*]** and have issued our report thereon dated **[*insert date*].**

We have not performed any auditing procedures since that date.

At your request, I have read your letter to the Michigan Department of Environment, Great Lakes, and Energy (EGLE) dated **[*insert date of letter to EGLE*]**, that provided a standard Financial Test and have compared the data in that letter, which are specified as having been derived from the **[*insert name of company*]**'s audited financial statements for its fiscal year ending **[*insert fiscal year end date*]**, to the **[*insert name of company*]**'s financial statements for its most recent fiscal year. In connection with that review, no matters came to my attention that caused me to believe that the specified data should be adjusted or corrected.

This letter is furnished solely for the use of **[*insert name of company*]** and EGLE and is not to be used for any other purpose.

[*Name and address of Accounting Firm*]

Appendix D
CORPORATE GUARANTEE MODEL

CORPORATE GUARANTEE

This Corporate Guarantee (Guarantee) is made this **[*insert date*]** to the State of Michigan by **[*insert name of Parent Company <u>or</u> other guaranteeing entity*]** (Guarantor), a business corporation organized under the laws of the State of **[*insert name of state*]**, **[*insert address*]**, on behalf of our subsidiary **[*insert name of subsidiary company*]**, **[*insert subsidiary business address*]**.

RECITALS

Whereas, Guarantor is the parent corporation of **[*insert name of subsidiary company*]**, is a majority shareholder of **[*insert name of subsidiary company*]**, and will benefit from the operation and activities of **[*insert name of subsidiary company*]**.

Whereas, **[*insert name of subsidiary company*]** is liable pursuant to Part 201, Environmental Remediation, of the Natural Resources and Environmental Protection Act, 1994 PA 451, as amended (NREPA), MCL 324.20101 *et seq.* for the **[*insert name of Facility*]** facility (Facility) located at **[*insert street address, or township, county, and state]*** with Site ID No. **[*insert number*]** which is covered by this Guarantee.

Whereas Section 20114d of the NREPA states that upon completion of remedial actions that satisfy the requirements of Part 201, a person may submit a No Further Action Report (NFA Report) to the Michigan Department of Environment, Great Lakes, and Energy (EGLE).

Whereas Section 20114d(4)(b) requires financial assurance to pay for monitoring, operation and maintenance, oversight and other costs determined by EGLE to be necessary to assure the effectiveness and integrity of the remedial action if a postclosure agreement is required as part of the NFA Report.

Whereas **[*insert name of subsidiary company*]** is required to submit a postclosure agreement as part of their NFA Report.

Whereas Section 20101(u) of the NREPA, defines financial assurance as a performance bond, escrow, cash, certificate of deposit, irrevocable letter of credit, corporate guarantee, or other equivalent security, or combination thereof.

Whereas, EGLE has determined that the Financial Test/Corporate Guarantee (FT/CG) is an acceptable FAM.

Whereas, the Guarantor has met the Financial Test (FT) criteria and provided other financial information to EGLE (Attachment **[*xx*]**) ***NOTE: this attachment must contain the documents included in Appendices A, B, and C*** and EGLE has determined that **[*insert name of subsidiary company*]** may use this Guarantee to fulfill its financial obligations pursuant to Part 201 of the NREPA.

In consideration of the foregoing, it is hereby agreed by and between EGLE and the Guarantor that this Guarantee will provide the required financial assurance for the Facility described above.

I. REPRESENTATIONS AND WARRANTIES OF GUARANTOR

Guarantor hereby represents and warrants as follows:

1.1 Corporate Authority

A. Guarantor is a corporation duly organized, validly existing and in good standing under the laws of the State of **[*insert name of state where* Guarantor *is incorporated*]**. Guarantor has the requisite corporate powers and authority to own its property and assets, to carry on its business as it is now conducting it, and to execute, deliver, and perform this Guarantee. Guarantor is duly qualified to do business in every jurisdiction, to which such qualification is necessary, including the State of Michigan.

B. The execution, delivery, and performance of this Guarantee and the consummation of the transactions herein contemplated have been duly authorized by all requisite corporate action on the part of the Guarantor and will not violate any provision of law, any order of any court or other agency of government, the articles of incorporation or bylaws of Guarantor, or any indenture, agreement or other instrument to which it is a party or by which it or any of its property is bound; and will not conflict with, result in a breach of, or constitute (with due notice and/or lapse of time) a default under any such indenture, agreement or other instrument.

II. GUARANTOR'S BUSINESS COVENANTS

The Guarantor covenants that, during such time as this Guarantee is in effect, it will comply with the following:

2.1 Financial Records - Guarantor will:

A. Maintain a system of accounting, which is established and administered in accordance with generally accepted accounting principles;

B. Keep adequate records and books of account in which true, accurate, and complete entries are made and which reflect all transactions that are required to be reflected by such accounting principles; and

C. Keep accurate and complete records of any property owned by it.

2.2 Corporate Existence and Rights - Guarantor will perform or cause to be performed all things necessary to preserve and keep in full force and effect its existence, rights and franchises, provided that this covenant shall not apply so as to prevent the Guarantor from entering into any transaction whereby all or substantially all of its assets and liabilities (including its obligations in respect of this Guarantee) are acquired and assumed by another corporation, whether by, merger or otherwise, as long as such other successor corporation meets the FT criteria set forth in Section III and assumes the obligations of this Guarantee.

2.3 Compliance with Law - Guarantor will not violate any laws, ordinances or governmental rules and regulations to which it is subject and will not fail to obtain any licenses, permits, franchises or other governmental authorizations that are necessary to the ownership of its property or the conduct of its business, if such violation or failure to obtain might materially and adversely affect Guarantor's ability to perform its obligations under this Guarantee.

III. INFORMATION AS TO GUARANTOR

Guarantor shall provide the following financial and business information to EGLE during the time period that this Guarantee is in effect.

3.1 Financial Information:

A. Except as otherwise provided by Paragraph 3.1.B., within 90 days after the close of each succeeding fiscal year that this Guarantee is in effect, Guarantor shall prepare and submit to EGLE the following:

(1) A letter signed by Guarantor's chief financial officer, which is worded as specified by EGLE, and includes Guarantor's demonstration that it has passed the standard FT using the EGLE model for the FT. ***NOTE: This is Appendix B***

(2) A copy of an independent certified public accountant's report regarding his/her examination of Guarantor's year-end financial statements for the last 5 years.

(3) A letter from an independent certified public accountant to Guarantor which states both of the following: ***NOTE: This letter is Appendix C***

(a) That the independent certified public accountant has compared the data referenced in the letter from the chief financial officer in Paragraph 3.1.A(1) as having been derived from the independently audited, year-end financial statements for the latest fiscal year with the amounts in such financial statements; and

(b) That, in connection with Paragraph 3.1.A(3)(a), no matters came to the attention of the independent certified public accountant that caused the accountant to believe the specified data was incorrect or should be adjusted.

(4) A certificate from the President or a Vice President and the Treasurer or an Assistant Treasurer of Guarantor setting forth that the signers have reviewed the relevant terms of this Guarantee and have made, or caused to be made, under their supervision, a review of the transactions and conditions of the Guarantor from the beginning of the accounting period covered by the financial statements being delivered therewith to the date of the certificate, and that such review has not disclosed the existence during such period of any condition which constitutes an event of noncompliance under this Guarantee. If during such period any such condition or event of noncompliance existed or exists, the certificate shall specify the nature and period of existence thereof and the actions Guarantor has taken or proposes to take with respect thereto.

B. Pursuant to the terms of the postclosure agreement, within 30 days after each succeeding 5-year anniversary date of the end of the fiscal year that the postclosure agreement is in effect, **[*insert name of subsidiary company*]** is required to submit to EGLE and Guarantor an updated cost estimate for implementing the **[*describe the general nature of response activities, including, if appropriate oversight, monitoring and other costs*]** for the next **[*insert* 30-year period*, <u>or</u> if appropriate, other period of time*]**. Within 60 days of Guarantor's receipt of this information from **[*insert name of subsidiary company*]**, Guarantor shall re-evaluate whether it meets the FT criteria set forth in Paragraph 3.1.A(1) and submit the information required in Paragraph 3.1.A(1)-(4) to EGLE.

3.2 Requested Information – In addition to the information specified in Paragraph 3.1.A, EGLE, based on a reasonable belief that the Guarantor may no longer be able to pass the FT specified in Paragraph 3.1.A(1), may require Guarantor, at any time, to submit reports of its financial condition to EGLE. Guarantor shall provide with reasonable promptness to EGLE any other data and information that may reasonably be expected to materially adversely affect the Guarantor's ability to perform its obligations under the Guarantee.

3.3 Notice of Breach of Covenants or Noncompliance Events - Immediately upon becoming aware of the existence of any condition or event that constitutes either a noncompliance with the pertinent requirements of the postclosure agreement or a Breach of any Covenants under this Guarantee (with the exception of breaches or notices of breach that EGLE sends to Guarantor), Guarantor shall provide written notice to EGLE. Such notice shall specify the nature and duration of the condition or event and the actions the Guarantor is taking or proposes to take to address the condition or event.

IV. GUARANTEE OF OBLIGATIONS

4.1 Guarantor hereby irrevocably guarantees the full and prompt performance of all obligations of [***insert name of subsidiary company***] under the postclosure agreement including, without limitation, payment of all amounts including any interest or stipulated penalties, which are or may become due thereunder.

4.2 Guarantor guarantees that in the event [***insert name of subsidiary company***] fails to perform [***describe the general nature of response activities required under the postclosure agreement***] for the Facility in accordance with EGLE approved plans, Guarantor will do so.

4.3 Guarantor guarantees that if, at the end of any fiscal year before termination of this Guarantee, Guarantor fails to meet the FT criteria as set forth in Paragraph 3.1.A(1), Guarantor will send within 90 days, by certified mail, notice to EGLE and [***insert name of subsidiary company***] that it will provide alternate financial assurance, in a FAM acceptable to EGLE, in the name of [***insert name of subsidiary company***].

4.4 If an alternate FAM must be secured by Guarantor, within 30 days of providing the notice required by Paragraph 4.3, Guarantor shall submit for review and approval to EGLE, the necessary forms and documents for implementing the alternate FAM. Such forms and documents shall be in a form acceptable to EGLE and shall include the type of FAM, the amount of funds to be secured, and a procedure for the continued review and approval of that FAM by the parties, if appropriate. Submittals provided to EGLE pursuant to this paragraph shall be reviewed and approved and/or disapproved in accordance with the postclosure agreement. Upon receipt of approval by the Remediation and Redevelopment Division Director, Guarantor shall implement the alternate FAM within 15 days.

4.5 Pursuant to the postclosure agreement, if at any time [***insert name of subsidiary company***] or EGLE identifies the need for additional response activity as provided for in the postclosure agreement**,** [***insert name of subsidiary company***] is required to submit to EGLE for review and approval a proposed plan and schedule for these response activities and is required to provide to EGLE and Guarantor, an estimate of the cost for implementing these response activities. [***insert name of subsidiary company***] is required to submit these items to the designated parties within 30 days of identification of the need for the additional response activities. If requested by EGLE, Guarantor shall then re-evaluate whether it meets the FT criteria as set forth in Paragraph 3.1.A(1) in view of the additional cost that will be incurred to implement these response activities and Guarantor shall submit the FT information to EGLE.

4.6 EGLE, based on a reasonable belief that Guarantor may no longer be able to meet the FT requirements specified in Paragraph 3.1.A(1), may require Guarantor to submit updated FT information to determine whether it can continue to meet the FT requirements. If based on that updated information EGLE determines that the Guarantor no longer meets the requirements for the FT, Guarantor shall provide an alternate FAM in accordance with Paragraphs 4.3 and 4.4 of this Guarantee.

4.7 Guarantor agrees to remain bound under this Guarantee notwithstanding any amendment or modification of:

(1) The response activities or other obligations, including **[*generally describe response activities or obligations, for example: plans for monitoring, operation and maintenance, and oversight*]**; or

(2) Plans for additional response activities that are necessary to protect public health, safety, or welfare, or the environment.

4.8 Guarantor agrees to remain bound under this Guarantee for so long as **[*insert name of subsidiary company*]** must comply with the applicable financial assurance requirements of the postclosure agreement for the Facility.

4.9 Guarantor agrees to notify EGLE by certified mail within 10 days of commencement of a voluntary or involuntary proceeding under Title 11 (Bankruptcy), United States Code that names Guarantor as debtor.

4.10 If **[*insert name of subsidiary company*]** and Guarantor fail at any time to adequately implement the response activities required under the postclosure agreement or any response activities provided in a plan approved by EGLE, EGLE, at its discretion, may choose to implement those response activities that have not been performed or may seek other available remedies as specified by the postclosure agreement If **[*insert name of subsidiary company*]** has not reimbursed EGLE its costs within the 30-day time frame or alternate time frame specified in the postclosure agreement, Guarantor shall reimburse EGLE its costs for implementing those response activities as set forth in the postclosure agreement.

4.11 Guarantor further agrees that it shall irrevocably guarantee performance of the obligations of **[*insert name of subsidiary company*]** under the postclosure agreement whether or not it continues to be the holder, directly or indirectly, of the stock of **[*insert name of subsidiary company*]** and whether or not the Facility, or any part of it, is sold, transferred or otherwise alienated. However, this Guarantee may be assigned to a purchaser of Guarantor's interests in **[*insert name of subsidiary company*]** or to a purchaser of all or substantially all of the assets of **[*insert name of subsidiary company*]**, if the following terms and conditions are met in advance of such transaction:

(1) The purchaser demonstrates to EGLE that it can meet the FT set forth in Paragraph 3.1.A(1);

(2) Guarantor and the purchaser enter into an assumption agreement in which the purchaser agrees to assume all of the obligations set forth in this Guarantee and which sets forth the terms and conditions of the transaction;

(3) EGLE agrees in writing to the assumption agreement; and

(4) The postclosure agreement is modified, in accordance with the applicable procedures therein, to reflect this modification.

Upon compliance with the foregoing requirements of this paragraph, Guarantor shall be discharged from its obligations under this Guarantee.

V. NOTICE TO GUARANTOR/OPPORTUNITY TO CURE

Any obligations of [***insert name of subsidiary company***], which are contained in the postclosure agreement and guaranteed by Guarantor under this Guarantee, shall be enforceable against Guarantor only after EGLE has first made demand of [***insert name of subsidiary company***] for performance of such obligations pursuant to the terms of the postclosure agreement. EGLE demand to [***insert name of subsidiary company***] for performance shall set forth a detailed description of the nature of the violation of the postclosure agreement and the specific performance required to cure the violation. EGLE shall also provide a copy of the demand for performance to the Guarantor. If [***insert name of subsidiary company***] has not complied with EGLE demand for performance within 15 days of receipt of such demand, Guarantor shall either:

(1) Cure the violation within 15 days; or

(2) Commence and diligently pursue the cure and, if the cure cannot be completely performed within 15 days, provide a proposed schedule for approval by EGLE for completion of the cure. Guarantor shall then complete the cure within the time frame approved by EGLE. Under either scenario, within 15 days of completing the cure, Guarantor shall notify EGLE of the date the violation was cured and the actions that were taken to cure the violation.

VI. TERMS OF GUARANTEE

6.1 This Guarantee shall be fully enforceable by EGLE from the effective date of the Guarantee until EGLE [***specify the conditions that must be met for the FAM to be released***] pursuant to the postclosure agreement.

6.2 Except as provided in Paragraph 4.11 of this Guarantee, Guarantor may be excused from its obligations as set forth in this Guarantee only if all of the following conditions are met:

(1) [***insert name of subsidiary company***] is willing and financially able to provide an alternate FAM;

(2) [***insert name of subsidiary company***] submits and EGLE approves an alternate FAM that meets EGLE requirements;

(3) Such a FAM is in place prior to the termination of this Guarantee; and

(4) The postclosure agreement is modified, in accordance with the applicable procedures stated therein, to reflect this modification.

VII. NOTICE

Any notifications required under this Guarantee shall be directed to the following individuals at the addresses specified below, unless any of these individuals, their successors, or their attorneys provide notification of a change to the other party in writing.

As to Guarantor:

[*Insert Guarantor Name*]
[*Title*]
[*insert Address*]

As to EGLE:

[*insert Name of Division Director*], Director
Remediation and Redevelopment Division
Michigan Department of Environment, Great Lakes, and Energy
P.O. Box 30426
Lansing, MI 48909-7926

VIII. REMEDIES

No failure on the part of EGLE to exercise, nor any delay in exercising, any right hereunder shall operate as a waiver hereof. Neither the single or partial exercise of this Guarantee, nor the exercise of any other right, shall operate as a waiver hereof.

IX. GOVERNING LAW/CONSENT TO JURISDICTION

This Guarantee shall be governed by and construed in accordance with the laws of the State of Michigan. For the sole and exclusive purpose of enforcing the terms of this Guarantee, Guarantor consents to jurisdiction over it and the subject matter of this Guarantee in the appropriate state or federal courts within the State of Michigan.

X. SUCCESSORS AND ASSIGNS

This Guarantee shall be binding upon, and shall inure to the benefit of, the successors and assigns of the parties.

XI. INTEGRATION

This Guarantee constitutes the entire obligation of Guarantor insofar as it concerns the postclosure agreement between **[*insert name of subsidiary company*]** and EGLE.

XII. EFFECTIVE DATE

This Guarantee shall become effective on the date that it is executed by the Guarantor and EGLE.

XIII. AUTHORITY

The undersigned representative of Guarantor certifies that he/she is fully authorized to execute and legally bind Guarantor to the obligations undertaken in this Guarantee. The undersigned representative of the State of Michigan certifies that he/she is fully authorized to accept this Guarantee.

EXECUTED THIS _______ day of ______________________, **[*insert year*]**.

[*insert name of Guarantor*] ACCEPTANCE OF GUARANTEE	Michigan Department of Environment, Great Lakes, and Energy
By: ___________________________	By: ___________________________
Name: ___________________________ (type name)	Name: ___________________________ (type name)
Title: ___________________________	Title: Director, Remediation and Redevelopment Division