

2020

PART 115 RULES CHECKLIST
COAL ASH LANDFILL AND COAL ASH IMPOUNDMENTS
HYDROGEOLOGICAL MONITORING PLAN

Facility Name: Monroe Power Plant Bottom Ash Impoundment Date: June 2020
 _____Initials

Report Name: MONPP BAI HMP Report Date June 2020

| ITEM | | Y/N/NA |
|------|---|---|
| 1. | <u>Design and siting</u> ensure groundwater will not exceed: R306(1) | NA |
| | MCLs in 40 CFR Part 257 and Appendix I. (Note: if the design and siting ensure GW will not exceed MCLs identified in appendix I, they will likely ensure that Michigan's cleanup criteria are not exceeded) | NA |
| | Existing concentrations, where these already exceed 40 CFR Part 257 and Appendix I, unless groundwater has greater than 10,000 mg/L TDS. | NA |
| 2. | Design and siting ensure that requirements of Part 31 and its rules will be met. R306(2) | NA |
| 3. | Hydrogeologic monitoring plan for the coal ash landfill or coal ash impoundment includes the following components: | Y |
| | A monitoring well system which complies with R906. R905(1)a | Y, Attachment A |
| | Leachate and SCS monitoring programs as specified in R432, if required. R905(1)b | NA |
| | Surface water monitoring program for surface waters that may receive runoff from the "active work area" (see R101(g)). R905(1)c | NA |
| 4. | Contains the following specific information: R905(2) | Y |
| | All GW sampling locations. R905(2)a | Y, Attachment B Table 2-1 |
| | Sampling constituents/parameters and frequency. R905(2)b | Y, Attachment B, Section 2, Table 2-2 and 2-3 |
| | Sampling and analysis procedures for each parameter including: R905(2)c | Y, Attachment B |
| | Sample collection. | Y, Attachment B Section 2.4 |
| | Sample preservation and shipment. | Y, Attachment B |

| ITEM | | Y/N/NA |
|------|--|--|
| | | Table 2-6 and Section 3.5 |
| | Analytical procedures, including detection limits. | Y, Attachment B Table 2-2 and 2-3 |
| | Chain of custody control. | Y, Attachment B Section 3.5 |
| | Laboratory and field quality assurance and quality control procedures. | Y, Attachment B Sections 3.1 and 3.2 |
| | Procedures for prevention of cross contamination in wells during well installation, purging and sampling. | Y, Attachment B Section 2.5 |
| | Statistical procedures for data evaluation in compliance with R908. | Y, Attachment C |
| 5. | Sufficient number of wells, installed at appropriate locations and depths, to yield groundwater samples from the uppermost aquifer that represent the quality of: R906(1) | Y, Attachment A |
| | Background water quality not affected by leakage from a unit. R906(1)a | Y, Attachment A Section 3.0 |
| | Meets conditions for use of wells other than true upgradient. R906(1)(a)i or ii | Y, Attachment A Section 3.0 |
| | Downgradient groundwater and ensures detection of groundwater contamination in the uppermost aquifer, and other groundwater specified by the Director. R906(1)b | Y, Attachment A Section 3.0 |
| | Meets conditions for downgradient monitor well installation at locations other than the solid waste boundary. | NA |
| | Wells installed at the closest practicable distance from the solid waste boundary. | Y, Attachment A Figure 3 |
| 6. | Meets conditions for a multi-unit groundwater monitoring system instead of separate monitoring systems for each landfill unit when the facility has several discrete units. R906(2) | NA |
| | Monitoring wells not more than 150 meters from the solid waste boundary of each unit, located on land owned by the owner of the unit | NA |

| ITEM | | Y/N/NA |
|------|---|-----------------------------------|
| | R906(2)a | |
| | Sufficient number of wells, installed at appropriate locations and depths, to yield groundwater samples from the uppermost aquifer. R906(2)b | NA |
| | Is as protective of human health and environment as individual monitoring systems for each unit, based on the following: R906(2)b | NA |
| | Number, spacing and orientation of the units. | NA |
| | Hydrogeologic setting. | NA |
| | Site history. | NA |
| | Engineering design of the units. | NA |
| | Type of waste accepted at the units. | NA |
| 7. | Monitoring wells cased in a manner that maintains the integrity of the well borehole. R 906(3) | Y, Attachment A Appendix A |
| 8. | Well casings screened or perforated and packed with gravel or sand, where necessary, to enable the collection of groundwater samples. R906(3) | Y, Attachment A Appendix A |
| 9. | Annular space in each monitoring well sealed to prevent contamination of the samples and groundwater. R906(3) | Y, Attachment A Appendix A |
| 10. | Notified the Director that the design, installation, development, and decommission of any monitoring wells, piezometers, and other measurement, sampling, and analytical devices documentation have been placed in the operating record. R906(4) | Y, Attachment B Section 1.2 |
| 11. | All monitoring wells, piezometers, and other measurement, sampling, and analytical devices designed, operated and maintained to perform to design specifications throughout the life of the monitoring program. R906(5) | Y, Attachment A |
| 12. | Monitoring wells designed to minimize the time necessary to recharge well, given hydraulic conductivity of the aquifer. R906(6) | Y, Attachment A |
| 13. | Number, spacing, and depths of monitoring systems in compliance with the following conditions: R906(7) | Y, Attachment A Section 2.2 |
| | Site-specific technical information that includes thorough characterization of both of the following: R906(7)(a) | Y, Attachment A Section 2.2 |
| | The uppermost aquifer, including all of the following information: R906(7)(a)i | Y, Attachment A Section 2.2 |
| | Aquifer thickness. | Y, |

| ITEM | | Y/N/NA |
|------|--|-------------------------------------|
| | | Attachment A Section 2.2 |
| | Groundwater flow rate. | Y, Attachment A Section 2.2 |
| | Groundwater flow direction including seasonal and temporal fluctuations in groundwater flow. | Y, Attachment A Section 2.2 |
| | Saturated and unsaturated geologic units and fill materials overlying the uppermost aquifer, materials comprising the uppermost aquifer, and materials comprising the confining unit defining the lower boundary of the uppermost aquifer, including all of the following: R906(7)(a)ii | Y, Attachment A Section 2.2 |
| | Thickness. | Y, Attachment A Section 2.2 |
| | Stratigraphy. | Y, Attachment A Section 2.2 |
| | Lithology. | Y, Attachment A Section 2.2 |
| | Hydraulic conductivities. | Y, Attachment A Section 2.2.2 |
| | Porosities. | N |
| | Effective Porosities. | Y, Attachment A Section 2.2.2 |
| | Certified by a Geologist. R906(7)b | Y, Attachment A Section 5.0 |
| | Approved by the Director. Within 14 days of this approval, the owner or operator shall notify the Director that the certification and approval have been placed in the operating record. R906(7)c | N |
| 14. | All wells clearly labeled, properly vented, capped, and locked when not in use. R906(8) | Y, Attachment B Section 1.2.2 |
| 15. | All wells visible throughout the year. R906(8) | Y, Attachment B |

| ITEM | | Y/N/NA |
|------|---|---|
| | | Section 1.2.2 |
| 16. | Owner or operator to notify the Director or designee prior to undertaking well abandonment, plugging, replacement, or repair. R906(9) | Y, Attachment B Section 1.2 |
| 17. | Groundwater monitoring program includes sampling and analysis procedures designed to ensure monitoring results that provide an accurate representation of groundwater quality at the background and downgradient wells installed in compliance with R906. R907(1) | Y, Attachment B |
| 18. | Owner or operator has notified Director that sampling and analysis program documentation has been placed in the operating record. R907(1) | Y, Cover letter |
| 19. | The sampling and analysis program shall include all of the following: | Y, Attachment B |
| | Sample collection. R907(1)a | Y, Attachment B Section 2.4 |
| | Sample preservation and shipment. R907(1)b | Y, Attachment B Table 2-6 and Section 3.5 |
| | Analytical procedures. R907(1)c | Y, Attachment B Table 2-2 and 2-3 |
| | Chain of custody control. R907(1)d | Y, Attachment B Section 3.5 |
| | Quality assurance and quality control. R907(1)e | Y, Attachment B Section 3 |
| 20. | Sampling and analysis programs include sampling and analytical methods appropriate for groundwater sampling and accurately measure hazardous constituents and other monitoring parameters in groundwater samples. R907(2) | Y, Attachment B |
| 21. | Groundwater samples shall not be field filtered. 324.11511a(3)e | Y, Attachment B Section 2.4 |
| 22. | Sampling procedures and frequency are protective of human health and the environment. R907(3) | Y, Cover letter |
| 23. | Analytical methods and practical quantitation limits for groundwater monitoring are approved by the Director. R907(4) | N |
| 24. | Groundwater elevations measured immediately prior to purging each time | Y, |

| ITEM | | Y/N/NA |
|------|---|-------------------------------------|
| | groundwater is sampled. R907(5) | Attachment B Section 2.3 |
| 25. | Owner or operator to determine rate and direction of groundwater flow each time groundwater is sampled. R907(5) | Y, Attachment B Section 2.3 |
| 26. | Facility to measure groundwater elevations within a period of time short enough to avoid temporal variations in groundwater flow which could preclude accurate determination of groundwater flow rate and direction. R907(5) | Y, Attachment B Section 2.3 |
| 27. | Groundwater elevations measured by methods giving precision to 1/8 inch or 0.01 foot, measured from the top of the well reference point using a determined USGS datum point. R907(6) | Y, Attachment B Section 2.3 |
| 28. | Facility has established background water quality in a hydraulically upgradient or background well or wells for each of the monitoring parameters or constituents required in groundwater monitoring program. (Background groundwater quality may be established at wells not located hydraulically upgradient from the unit if the well meets R906(1)(a)). R907(7) | Y, Attachment C Section 3.1 |
| 29. | Number of samples to establish groundwater quality data consistent with statistical procedures determined per R908. The sampling procedures are those specified pursuant to the provisions of the following: R907(8) | Y, Attachment C |
| | For detection monitoring R440 | Y, Attachment C Section 3.1 |
| | For assessment monitoring R441 | Y, Attachment C Section 4.0 |
| | For remedial action R444 | Y, Attachment C Section 4.0 |
| 30. | All samples obtained shall be representative of the site's groundwater quality. R907(9) | Y, Attachment B Section 2.4 |
| | Each well will be purged until dry or until not less than 3 times the amount of water in the well casing has been removed. | Y, Attachment B Section 2.4.1 |
| | Monitoring wells will be sampled immediately after purging where recovery rates allow. | Y, Attachment B Section 2.4.3 |
| | If well pumped dry during purging, samples will be taken within 24 hours. | Y, Attachment B Section 2.4.3 |

| ITEM | | Y/N/NA |
|------|---|---|
| 31. | If nondedicated pumps or mobile sampling equipment is used, facility will use the following procedures to minimize the potential for cross-contamination: R907(10) | Y, Attachment B |
| | Sample wells from upgradient to downgradient, except areas of known contamination will be sampled from least contaminated to most contaminated well. R907(10)a | NA |
| | Each piece of equipment will be thoroughly cleaned and rinsed with distilled water before use in each well. R907(10)b | Y, Attachment B Section 2.5 |
| | Other decontamination procedures approved by the Department. R907(10)c | NA |
| 32 | The owner and operator shall submit all monitoring results to the director or designee not later than 30 days after the end of the calendar quarter. R907(11) | Y, Attachment B Section 4 |
| 33. | The owner and operator of a landfill will sample and analyze groundwater by methods specified in "Standard Methods for the Examination of Water and Wastewater.... Or other methods approved by the director or his or her designee. (we would accept SW-846 methods). 324.11511a(4) | Y, Attachment B Tables 2-2 and 2-3 |
| 34. | Detection monitoring parameter list includes: 324.11511a(3)(c) | Y, Attachment B Table 2-2 |
| | Boron 324.11511a(3)(c)i | Y, Attachment B Table 2-2 |
| | Calcium 324.11511a(3)(c)ii | Y, Attachment B Table 2-2 |
| | Chloride 324.11511a(3)(c)iii | Y, Attachment B Table 2-2 |
| | Fluoride 324.11511a(3)(c)iv | Y, Attachment B Table 2-2 |
| | Iron 324.11511a(3)(c)v | Y, Attachment B Table 2-2 |
| | pH 324.11511a(3)(c)vi | Y, Attachment B Table 2-2 |

| ITEM | | Y/N/NA |
|------|---|---|
| | Sulfate 324.11511a(3)(c)vii | Y, Attachment B Table 2-2 |
| | Total Dissolved Solids 324.11511a(3)(c)viii | Y, Attachment B Table 2-2 |
| 35. | Contains a statistics plan or statistical procedures that meets the requirements of Rule 908. (Use Part 115 Rules Checklist – Landfill Groundwater Monitoring Statistical Procedures). R908 | Y, Attachment C |
| 36. | Detection monitoring is conducted quarterly during the active life and semiannually during the post-closure period, except as provided for in R440(5). R440(1)(a) | Y, Cover letter |
| 37. | Meets conditions for deletion of R452 to R454 parameters. | NA |
| | Parameters and breakdown products are not in leachate for not less than 2 consecutive and historic samplings. R440(4) | NA |
| 38. | Meets conditions for alternative monitoring frequency for R450-451 parameters (at least semiannually) or for R452-454 parameters (at least annually) based on following factors: R440(5) | Y, Cover letter |
| | Lithology of aquifer and unsaturated zone. R440(5)a | NA |
| | Hydraulic conductivity of aquifer and unsaturated zone. R440(5)b | Y, Cover letter and Attachment B Section 3.1 |
| | Groundwater flow rates. R440(5)c | Y, Cover letter and Attachment B Sections 2.2 and 3.1 |
| | Minimum distance from the waste and the closest downgradient well screen, or presence of SCS. R440(5)d | NA |
| | Resource value of aquifer. R440(5)e | NA |
| 39. | First sampling event includes 4 independent samples from each well. Subsequent events include minimum of 1 sample from each well. R440(7) | N, Attachment B Section 4 |
| 40. | In case of statistically significant increase over background: | Y, Attachment C |
| | Place notice in operating record within 14 days. R440(8)a | Y, Attachment C Section 4.0 |
| | Prepare assessment monitoring plan per R441 and a response action | Y, |

| ITEM | | Y/N/NA |
|------|--|-----------------------------------|
| | plan within 45 days. R440(8)b | Attachment C Section 4.0 |
| 41. | If statistically significant increase over background due to other source or is due to an error, has owner: | Y, Attachment C Section 4.0 |
| | Documented a demonstration of this and placed notice in operating record within 30 days. R440(9) | Y, Attachment C Section 4.0 |
| | If a successful demonstration is made, | Y, Attachment C Section 4.0 |
| | Continue detection monitoring. R440(9)(a) | Y, Attachment C Section 4.0 |
| | Determined if the unit remains monitorable R440(9)(b) | Y, Attachment C Section 4.0 |
| | If a successful demonstration is not made, then 15 days after notification by the director, prepare an assessment monitoring plan and a response action plan. R440(10) | Y, Attachment C Section 4.0 |
| 42. | Text in the HMP indicates an assessment monitoring program will be developed if required under R441 <u>or</u> the Assessment Monitoring Program is included with the HMP. (use the assessment monitoring program checklist if the program is provided) <u>or</u> the Assessment Monitoring program has already been approved and is referenced in the HMP. R441 <u>Or</u> a schedule, approved by the department, that leads to compliance by no later than December 28, 2020 has been provided. 324.11511a(3)(f)ii | Y, Attachment C Section 4 |
| 43. | Text in the HMP indicates a response action plan will be developed if required under R442 <u>or</u> the Response Action Plan is included. (use the response action plan checklist if a plan is provided) <u>or</u> the Response Action Plan has already been approved and is referenced in the HMP. R442 <u>Or</u> a schedule, approved by the department, that leads to compliance by no later than December 28, 2020 has been provided. 324.11511a(3)(f)ii | Y, Attachment C Section 4 |
| 44. | Text in the HMP indicates that corrective measures will be assessed if required under R443 <u>or</u> the assessment of corrective measures is included in the HMP <u>or</u> the assessment of corrective measures has already been approved and is referenced in the HMP. R443 <u>Or</u> a schedule, approved by the department, that leads to compliance by no later than December 28, 2020 has been provided. 324.11511a(3)(f)ii | Y, Attachment C Section 4 |
| 45. | Text in the HMP indicates that a remedy will be selected, if required, in compliance with R444 <u>or</u> the remedy selection and remedial action plan is included with the HMP <u>or</u> the remedy selection and remedial action plan has | Y, Attachment C Section 4 |

| ITEM | Y/N/NA |
|--|---------------------------------|
| <p>already been approved and is referenced in the HMP. R444</p> <p><u>Or</u> a schedule, approved by the department, that leads to compliance by no later than December 28, 2020 has been provided. 324.11511a(3)(f)ii</p> | |
| <p>46. Text in the HMP indicates that a remedial action plan will be implemented, if required, in compliance with R445 <u>or</u> the remedial action plan implementation details are included with the HMP <u>or</u> the remedial action plan has already been implemented and is referenced in the HMP. R445</p> <p><u>Or</u> a schedule, approved by the department, that leads to compliance by no later than December 28, 2020 has been provided. 324.11511a(3)(f)ii</p> | Y, Attachment C Section 4 |
| COMMENTS: | |
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| | |
| | |
| | |

June 30, 2020

Mr. Chris Scieszka
Environmental Management & Safety
DTE Electric Company
One Energy Plaza, 410 G.O.
Detroit, Michigan 48226

Subject: Hydrogeological Monitoring Plan for the DTE Electric Company Monroe Power Plant Bottom Ash Impoundment, 3500 East Front Street, Monroe, Michigan

Dear Mr. Scieszka:

On December 28, 2018, the State of Michigan enacted Public Act No. 640 of 2018 to amend Part 115 of the Natural Resources and Environmental Protection Act, PA 451 of 1994, as amended (Part 115). The December 2018 amendments to Part 115 were developed to provide the State of Michigan oversight of coal combustion residual (CCR) impoundments and landfills and to better align existing state solid waste management rules and statutes with the United States Environmental Protection Agency (USEPA) CCR Resource Conservation and Recovery Act (RCRA) Rule, as amended¹ (40 CFR 257 Subpart D) ("CCR Rule"). On August 5, 2016, the USEPA published the CCR Rule companion *Extension of Compliance Deadlines for Certain Inactive Surface Impoundments* to establish the compliance deadlines for CCR units that were inactive prior to April 17, 2018, which applies to the DTE Electric Company (DTE Electric) Monroe Power Plant (MONPP) Bottom Ash Impoundment (BAI) Inactive CCR unit. This alignment between the state and federal programs would ensure compliance with the federal CCR standards through a state-approved permitting program that would be deemed to be "equivalent to" or "as protective as" through an administrative application that would be reviewed and authorized by USEPA.

The DTE Electric MONPP is located in Section 16, Township 7 South, Range 9 East, at 3500 East Front Street, Monroe in Monroe County, Michigan (Figure 1). The MONPP BAI was operated from the mid-1970s through 2015 and is located within the southern portion of the MONPP parcel at latitude 41° 52' 30" North and longitude 83° 20' 70" West. DTE Electric is currently planning to close the MONPP BAI by removing all CCR material from the basin. The design for the closure by removal is ongoing.

Groundwater monitoring activities have been conducted at the MONPP BAI entirely in accordance with the CCR Rule since January 2017 when background monitoring began and has commenced and continued detection monitoring in 2019 through the present.

DTE Electric is in the process of establishing a Part 115 operating license for the MONPP BAI in order to manage closure of the site under Part 115. Revisions to Part 115 through PA 640, in particular Section 11512(a)(1), require an approved Hydrogeologic Monitoring Plan (HMP) that complies with Rules 299.4440 to 299.4445, if applicable, and Rules 299.4905 to 299.4908 of the Part 115 Rules. As part of the license requirements, on behalf of DTE Electric, TRC has prepared this HMP in place of the CCR Rule monitoring program documents to provide a means to comply with applicable monitoring

¹ United States Environmental Protection Agency (USEPA) final rule for the regulation and management of Coal Combustion Residuals (CCR) under the Resource Conservation and Recovery Act (RCRA) published April 17, 2015, as amended.

requirements described in Part 115, as amended, and the CCR Rule. It should be noted that the Michigan statute does not act in lieu of the federal standards until such a time as the USEPA authorizes Michigan's permit program in lieu of the Federal rule.

The components of this HMP have largely been developed in compliance with the CCR Rule in order to document the procedures for the collection and analysis of groundwater data used to monitor groundwater at the MONPP BAI. These existing documents will collectively serve as the updated HMP presented in this letter report with the additional modifications described herein that are necessary to comply with Part 115, as amended.

Groundwater Monitoring System

A groundwater monitoring system has been established under the CCR Rule that also meets the requirements of Part 115 Rule 299.4905(1)(a) that states that an HMP shall include a groundwater monitoring well system that complies with the provisions of Rule 299.4906. The groundwater monitoring system along with a detailed hydrogeological site characterization, geologic cross sections and well construction logs, are presented in the Monitoring Well Installation Report (WIR) prepared by AECOM in October 2017, updated in April 2020, and is included in Attachment A. The WIR also describes the methods and procedures associated with the installation of the monitoring wells, which will also be used for the construction of any future monitoring wells. Michigan Department of Environment, Great Lakes, and Energy (EGLE) approval will be requested prior to any future modifications to the monitoring well network, including installation of additional monitoring wells, replacements of existing wells, or decommissioning/removal of any wells from the monitoring program. A map of the monitoring system is provided in Figure 2.

Groundwater Sampling and Analytical Program

The Groundwater Monitoring and Quality Assurance Project Plan (QAPP) prepared by TRC in April 2020 for the MONPP BAI presents the updated groundwater monitoring program that will be implemented as part of this HMP and is included in Attachment B. The QAPP addresses collection and handling of samples at the site and laboratory analysis in conformance with Rule 299.4907 of the Part 115 Rules.

Groundwater monitoring will be conducted semiannually for the parameters listed in Section 11511a(3)(c) – Detection Monitoring Constituents. This frequency is consistent with the monitoring program established per the CCR Rule and is appropriate considering the hydrogeology of the site as described in Section 2.0 of the WIR in Attachment A and Section 1.2 of the Groundwater Statistical Evaluation Plan (AECOM 2019, updated April 2020) in Attachment C.

Data Evaluation and Reporting

Groundwater data will be evaluated for each constituent included in the groundwater monitoring program using statistical methods that comply with Rule 299.4908 and will be conducted in accordance with the "Statistical Analysis of Groundwater Monitoring Data at RCRA Facilities – Unified Guidance" USEPA, 2009 (Unified Guidance). The Groundwater Statistical Evaluation Plan in Attachment C

describes the statistical data evaluation procedures.

In order to comply with the Part 115 amendments, background will be established for the Section 11511a(3) constituents not already included in the CCR Rule Appendix III (i.e., iron) as detailed in Section 4 of the QAPP (Attachment B) using the statistical methods in Attachment C. Background groundwater monitoring was conducted at the MONPP BAI from January 2017 through February 2019 in accordance with the 2017 Groundwater Monitoring Work Plan, pursuant to the CCR Rule, with the results documented in the Annual Groundwater Monitoring Report prepared by TRC in July 2019 for the MONPP BAI (2019 Annual Report). Data collected to-date and statistical limits established as part of the CCR Rule implementation will be used to implement this HMP.

Routine statistical evaluation will entail the following process:

1. Analytical results for routine sampling events will be compared to the statistical limits established as discussed above in order to determine if a statistically significant increase (SSI) is observed. The statistical comparisons will be performed within 30 days of the end of the calendar quarter in which sampling and analysis was conducted, as specified in Rule 908(6).
2. In the event that a SSI has been determined to occur, DTE Electric will place a notice in the operating record and notify the EGLE in accordance with Rule 299.4440(8)(a).
3. As described in the Groundwater Statistical Evaluation Plan (Attachment C), verification sampling will be performed in order to achieve the site wide false positive rates (SWFPR) recommended in the Unified Guidance. If there is an exceedance of prediction limit for one or more of the parameters, the well(s) of concern will be resampled within 30 days of the completion of the initial statistical analysis. Only constituents that initially exceed their statistical limit (i.e., have no previously recorded SSIs) will be analyzed for verification purposes. If the verification sample remains statistically significant, then statistical significance will be considered, and the 14-day notification will be made. If the verification sample is not statistically significant, then no SSI will be recorded for the monitoring event and the 14-day notice will not be necessary.
4. If a SSI is determined, a 30-day demonstration period will be initiated upon determining the increase to identify if the apparent increase was attributable to error in sampling, analysis, statistical evaluation, impact from an off-site source, or natural variability in groundwater quality in accordance with Rule 299.4440(9). If it is determined that the apparent increase resulted from any of the aforementioned sources, the report will be submitted to the EGLE and routine monitoring will be resumed.
5. If it is determined that the detected increase was not the result of error, natural variability or an off-site source, (e.g. if the results of the second analysis confirm the initial results), an assessment monitoring plan in compliance with Rule 299.4441 and a response action plan in compliance with Rule 299.4442 will be prepared and submitted within 45 days of the SSI determination.

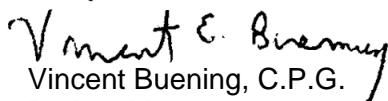
Groundwater sampling will be conducted on a semiannual frequency during the spring and fall. Analytical results and data reports as defined below will be submitted to the director no later than 30 days after the end of the calendar quarter in which the samples were obtained.

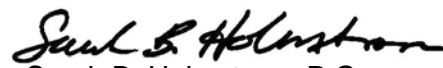
Data reports will include the following:

- Statement of adherence to the approved HMP;
- Description of the sampling event;
- Groundwater contour maps with summary of groundwater flow direction and rates;
- Tables of analytical results from the groundwater monitoring program that summarize the statistical exceedances (if any);
- Discussion of statistical data evaluation;
- Alternate source demonstration(s) (if applicable);
- Laboratory analytical results and chain of custody information;
- Field forms; and
- Signature of certified professional.

Sincerely,

TRC


Vincent Buening, C.P.G.
Project Manager


Sarah B. Holmstrom, P.G.
Project Hydrogeologist

Attachments

Figure 1 – Site Location

Figure 2 – Site Plan with Monitoring Network

Attachment A – Monitoring Well Installation Report (WIR) – AECOM, October 2017, revised June 2020

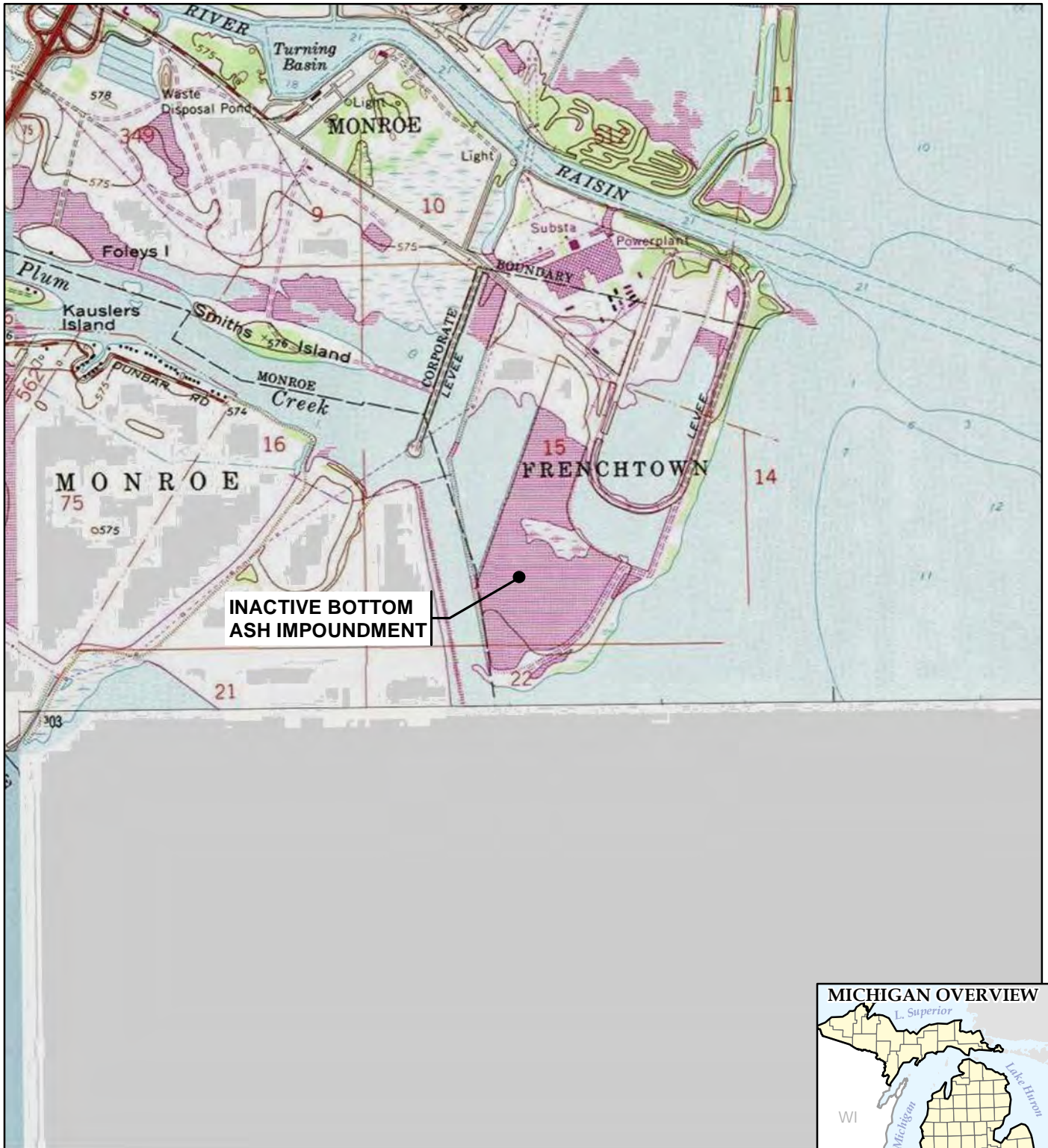
Attachment B – Groundwater Monitoring and Quality Assurance Project Plan – TRC, April 2020

Attachment C – Groundwater Statistical Evaluation Plan – AECOM, April 2019, revised April 2020

cc: Robert Lee, DTE Electric Company

Mr. Chris Scieszka
DTE Electric Company
June 30, 2020

Figures



BASE MAP FROM USGS 7.5 MINUTE TOPOGRAPHIC QUADRANGLE SERIES.



1540 Eisenhower Place
Ann Arbor, MI 48108-3284
Phone: 734.971.7080

TRC - GIS

PROJECT: **DTE ELECTRIC COMPANY
MONROE POWER PLANT BOTTOM ASH IMPOUNDMENT
3500 EAST FRONT STREET
MONROE, MI 48161**

TITLE: **SITE LOCATION MAP**

| | |
|--------------|-------------------|
| DRAWN BY: | R. SUEMNICHT |
| CHECKED BY: | S. HOLMSTROM |
| APPROVED BY: | V. BUENING |
| DATE: | SEPTEMBER 2019 |
| PROJ. NO.: | 320511.0006 |
| FILE: | 320511-001slm.mxd |

FIGURE 1

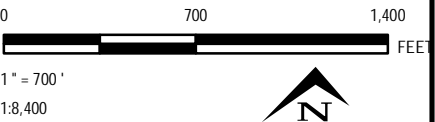


LEGEND

- CCR PROGRAM MONITORING WELL
- INVESTIGATION MONITORING WELL (STATIC WATER LEVELS ONLY)
- UNIT SEPARATION BERM
- APPROXIMATE BOUNDARY OF INACTIVE BOTTOM ASH IMPOUNDMENT
- APPROXIMATE PLANT BOUNDARY

NOTES

1. BASE MAP IMAGERY FROM GOOGLE EARTH PRO & PARTNERS, APRIL 2018.



| | |
|----------|--|
| PROJECT: | DTE ELECTRIC COMPANY MONROE POWER PLANT 3500 EAST FRONT STREET MONROE, MI 48161 |
| TITLE: | INACTIVE BOTTOM ASH IMPOUNDMENT WELL LOCATION MAP |

| | |
|--------------|---------------------|
| DRAWN BY: | S.MAJOR |
| CHECKED BY: | Kelly Cratsenburg |
| APPROVED BY: | Vince Buening |
| DATE: | APRIL 2020 |
| PROJ. NO.: | 370029.0006.0000 |
| FILE: | 370029.0006-003.mxd |
| FIGURE 2 | |

Mr. Chris Scieszka
DTE Electric Company
June 30, 2020

Attachment A

Monitoring Well Installation Report

REVISED MONITORING WELL INSTALLATION REPORT

INACTIVE BOTTOM ASH IMPOUNDMENT DTE Monroe Plant Monroe, Michigan

Prepared for:

DTE Energy
One Energy Plaza
Detroit, MI 48226

April 2019, Revised April 2020

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1.0 INTRODUCTION

At the request of DTE Electric Company (DTE), AECOM Technical Services, Inc. (AECOM) has prepared this revised Well Installation Report so that the documentation of the installation of monitoring wells at the DTE Energy Monroe Power Plant located in Monroe, Michigan (**Figure 1**) better addresses the requirements of new Michigan rules (cited below).

Monitoring wells were installed in the vicinity of the inactive Bottom Ash Impoundment in order to establish a groundwater monitoring system as required by the United States Environmental Protection Agency (USEPA) Final Rule 40 Code of Federal Regulations (CFR), Part 257 (Rule), Section 257.91 Sub-Part (a). The CCR Rule was established to regulate the disposal of Coal Combustion Residuals (CCR) produced by electric generating facilities (USEPA, 2015).

On December 28, 2018, the State of Michigan enacted Public Act No. 640 of 2018, to amend Part 115 of the Natural Resources and Environmental Protection Act of PA 451 of 1994, as amended (Part 115). The Michigan Public Act was established to provide the State of Michigan oversight of CCR impoundments and landfills and to better align existing state solid waste management rules and statutes with the CCR Rule (EGLE, 2018). After passage of Public Act, the design and installation of the groundwater monitoring system was reviewed and found to be in compliance with its requirements.

1.1 Site Location

The DTE Monroe Plant (Monroe Plant) is located in Monroe County Michigan approximately 2 miles east of the city of Monroe. The Monroe Plant was built in the early 1970s and occupies a parcel of land approximately 440 acres in size. The plant buildings, coal pile, and appurtenances associated with power generation reside on the northern (approximately 274 acres) portion of the 440-acre land parcel. The southern portion of the land parcel consists of the inactive Bottom Ash Impoundment area plus the Process Pond area which, together cover approximately 166 acres.

The Monroe Plant is bounded to the east and south by the shoreline of Lake Erie; to the west by neighboring industrial facilities and the plant discharge canal; and to the north by mixed residential/commercial properties as well as Plum Creek, as shown on **Figure 1**. Topography at the Bottom Ash Impoundment area is relatively flat with elevations ranging from 580 down to 572 feet mean sea level (msl), which is close to the mean elevation of Lake Erie.

1.2 Description of the CCR Unit

The Inactive Bottom Ash Impoundment is located to the south of the main Monroe Plant area and encompasses an area approximately 86.4 acres in size (**Figure 1**). The Inactive CCR Impoundment area was constructed in the late 1960s by building a perimeter dike to surround a low area of the adjacent Lake Erie; the area south of the plant was removed from the Waters of the United States by an Act of Congress prior to plant construction. CCR materials have been placed and allowed to drain into the pond from the north end of the pond; these materials currently form a delta that extends about 1/3 of the way into the pond. For purposes of the CCR groundwater study, the Inactive Bottom Ash Impoundment is considered a single CCR unit.

2.0 HYDROGEOLOGY

The following section presents information regarding the site-specific geologic and hydrogeologic conditions based on the findings from field investigation activities.

2.1 Geologic Setting

The Monroe Plant site is located on the eastern side of the Michigan Basin, which is a regional geologic structure in which the bedrock layers have warped downward towards a low spot in west-central Michigan. Accordingly, bedrock layers in the site vicinity are inclined (dip) at a very shallow angle to the west. The bedrock underlying the site is comprised of late Silurian age sedimentary rocks (predominantly dolomites and shales) from the Bass Island Group. The uppermost bedrock in the area tends to be highly weathered and is comprised of a tan, argillaceous dolomite with interbedded dark gray, firm to soft shales. The Bass Island Group is underlain by the middle to late Silurian age Salina Group, which is also comprised of alternating dolomite and shale units as well as anhydrite beds.

The bedrock in the site vicinity is overlain by approximately 40 to 50 feet of unconsolidated deposits of glacial origin. The deposits are comprised of two distinct units: a hard glacial till immediately overlying bedrock and lacustrine (lake bed or lake shore) deposits which overlay the till unit. Various thicknesses of surficial fill materials are present across the entire Monroe Plant and ash impoundment areas.

2.2 Local Hydrogeology

A series of cross-sections was prepared by NTH Consultants, LTD as part of a sitewide study completed in 2014. The locations of these sections are illustrated on **Figure 2**. These sections illustrate the sequence of geologic materials present under the Plant, Bottom Ash Impoundment, and Process Pond areas based on an assemblage of available boring logs. The lowermost unit identified in these areas is the glacial till. The till is comprised of overconsolidated (highly compacted) gray silty to sandy clay with some cobbles and boulders, and ranges from approximately 20 to 50 feet in thickness (**Figures 2a and 2b**). The overlying lacustrine deposits are composed of 10 to 30 feet of fine-grained sand and silt with some soft clay except where there is a thin, discontinuous coarse sand unit at the base of the lacustrine sequence (**Figure 2b**).

Under parts of the Plant, the Inactive Bottom Ash Impoundment, and Process Pond areas, this sand unit ranges in thickness from 5 to 20 feet and yields groundwater. The sand unit thins progressively to the west, having a thickness of approximately 12 feet on the east side of the discharge canal and thinning to less than a few feet within 150 feet to the west of the discharge canal. Further to the west the sand unit is not evident in soil borings for monitoring wells drilled in 2016 around the Fly Ash Basin. This is consistent with the expectation that lake-deposited materials will decrease in thickness with distance away from Lake Erie. Accordingly, it appears that this sand unit is a localized lakeshore beach deposit formed by westward aggradation with rising lake level and subsequently blanketed by finer lacustrine deposits. Groundwater in the sand unit is under semi-confined conditions with groundwater elevations ranging between approximately 572.6 and 575.6 feet above mean sea level (msl).

Lithologic information for each Inactive Bottom Ash Impoundment monitoring well is provided on the monitoring well construction logs included in **Appendix A**. Geologic Cross-sections are presented in **Figures 2a and 2b**.

2.2.1 Uppermost Aquifer System

The following section presents the expectations under the CCR Rule and Part 115 R 299.4101 and R 299.4105 for identifying the uppermost aquifer subject to groundwater monitoring and describes the lithologic unit identified as the uppermost aquifer in the vicinity of the combined footprint of the Inactive Bottom Ash Impoundment at the Monroe Plant.

As described in Part 115 R 299.4906:

“A landfill groundwater monitoring system shall be installed and shall consist of a sufficient number of wells, installed at appropriate locations and depths, to yield groundwater samples from the uppermost aquifer...”

Applicable definitions from Part 115 of PA 451 of 1994, as amended regarding the definition of an aquifer and the uppermost aquifer include the following:

“Aquifer means a geologic formation, group of formations, or portion of a formation that is capable of yielding significant quantities of groundwater to wells or springs.”

“Uppermost aquifer means the geologic formation which is nearest to the natural ground surface and which is an aquifer and includes lower aquifers that are hydraulically interconnected with this aquifer within the facility’s property boundary...”

Based on the hydrogeologic investigation findings, the uppermost aquifer zone occurs in the lower portion of a sequence of lacustrine deposits that is dominated by silty materials near the ground surface or under fill materials, which transitions at depth to a fine-grained sand. The shallow water-bearing zone is semi-confined by the overlying silts, with water levels generally higher than the top of the lacustrine unit. This water-bearing zone overlies a thick, hard glacial till. The glacial till unit acts as an aquitard between the unconsolidated deposits and the deeper, underlying bedrock.

2.2.2 Groundwater Flow and Hydraulic Conductivity

Water level data collected during the baseline groundwater monitoring program were used to construct potentiometric surface maps for the shallow groundwater zone. The data suggest that the direction of groundwater flow within the upper water-bearing zone is generally to the southeast and southwest towards Lake Erie, with an average gradient along the flow direction of approximately 0.00044 foot/foot (roughly 0.45 foot per 1000 feet). These values are within the expected range for the type of aquifer and the hydraulic setting. Potentiometric surface maps from the March 2018 and September 2018 sampling events are included in **Figures 4a and 4b**. As noted above, the aquifer unit thins to the west and the north such that there is no aquifer under areas north of the Inactive Bottom Ash Impoundment. Consequently, there is no representative upgradient or background monitoring position available for the unit. This directly affects the approach to the evaluation of compliance for the monitoring system as noted in the Statistical Methods Certification for this unit.

Hydraulic Conductivity

Aquifer testing (via drawdown and recovery tests using a submersible pump) was completed at monitoring wells MW-1S, MW-3S, MW-7S, and MW-8S. Testing data were evaluated on a well-by-well basis to assist in selecting the appropriate solution via the Aqtesolv™ software platform. Some key assumptions included the following: confined or leaky confined, presence of wellbore storage, and whether individual wells were considered fully or partially penetrating. The test pumping rates were low enough that the potential boundary conditions represented by the physical aquifer limits (to the north and west) were not expected to be detected in the drawdown or recovery data.

The shallow water-bearing zone wells yield groundwater at a relatively high rate. Where the zone has a component of gravel in the fine sand, the wells (MW-1S and MW-7S) produced significantly more water than monitoring wells screened in fine sand with silt (wells MW-3S and MW-8S). Calculated hydraulic conductivity values for the uppermost aquifer are summarized below:

| Well ID | Transmissivity (cm ² /sec) | Hydraulic Conductivity (cm/sec) | Hydraulic Conductivity (m/day) | Hydraulic Conductivity (ft/day) |
|---------|---------------------------------------|---------------------------------|--------------------------------|---------------------------------|
| MW-1S | 10.16 | 0.0423 | 36.5 | 119.8 |
| MW-3S | 0.68 | 0.0035 | 3.02 | 9.90 |
| MW-7S | 42.03 | 0.1274 | 110 | 360.9 |
| MW-8S | 0.57 | 0.0024 | 3.07 | 10.07 |

cm²/sec – centimeters squared per second

cm/sec – centimeters per second

m/day – meters per day

ft/day – feet per day

Horizontal Time of Travel

The horizontal time of travel for the Inactive Bottom Ash Impoundment area was calculated using Darcy Flux calculations and the following input values:

- Hydraulic Gradient (foot/foot) – based on average of dry and wet season potentiometric contours
- Hydraulic Conductivity (feet/day) – based on a median value estimated for the shallow aquifer system
- Effective Porosity (unit less) – based on published values for silty sands

Assuming an effective porosity of 30 percent for silty sand with some gravel, a gradient value of 0.00044 foot/foot (average gradient value of MW-14 to MW-7 and MW-14 to MW-3) with a median conductivity value of 119 feet/day, the horizontal time of travel is estimated to be 0.174 feet/day (or 260 feet/year).

3.0 GROUNDWATER MONITORING SYSTEM INSTALLATION

The CCR groundwater monitoring system well network was installed in two phases. The first phase of activities, conducted between September 19 and October 4, 2016, included the installation of seven (7) shallow and four (4) exploratory, deep (bedrock) monitoring wells in the vicinity of the inactive Bottom Ash Impoundment. Groundwater monitoring was performed over an 8-month period to evaluate the hydrogeology and groundwater chemistry in the vicinity of the inactive Bottom Ash Impoundment. Findings were used to select the location of seven (7) additional monitoring wells to establish the CCR groundwater monitoring system well network. The additional monitoring wells were installed between September 20 and September 26, 2017.

3.1 Borehole Advancement and Well Installation

Each monitoring well was installed by a State of Michigan licensed well driller as directly observed by an AECOM Geologist. Borings were advanced using a rotasonic drill rig and soil cores were collected in continuous sections for examination and lithologic description by the on-site geologist to the terminating depth of each borehole. Photographs of each soil core were collected. In total, 14 boreholes were advanced into the upper water-bearing zone in unconsolidated materials. Upon reaching the target depth, a monitoring well was installed in each borehole. Four (4) separate boreholes were advanced into a water-bearing zone of the bedrock that underlies the unconsolidated materials, but these wells are not included in the monitoring system because there is a strong upward hydraulic gradient between the bedrock and shallow groundwater systems that prevents downward migration of contaminants.

3.2 Well Construction

Each monitoring well was constructed using 2-inch inside diameter polyvinyl chloride (PVC) casing with a 10-foot section of 0.010-inch slotted PVC screen. The annular space (between the borehole wall and well

screen/casing) was backfilled with a clean silica sand pack extending at least 2 feet above the top of the screen. A minimum 2-foot thick bentonite seal was placed on top of the sand pack and each seal was allowed to hydrate for at least 1 hour per manufacturer's specifications. After hydrating the seal, the remaining annular space was filled with a cement/bentonite grout emplaced via tremie method to within approximately 12 inches of the ground surface.

3.3 Well Development

Each monitoring well was developed no sooner than 24-hours after grout emplacement to enhance hydraulic connection between the well and the aquifer and to remove potable water introduced to the subsurface during drilling activities. A submersible pump was used to remove at least five (5) well volumes or until the water was visibly clear of sediments, turbidity was less than 10 nephelometric turbidity units (NTUs), and water quality measurements [temperature, pH, conductivity, and oxidation-reduction potential (ORP)] were stable over at least three (3) well volumes.

3.4 Well Survey

Each monitoring well was surveyed for horizontal location (North American Datum of 1983 or NAD 83) and elevation data (North American Vertical Datum of 1988 or NAVD 88). by a surveyor licensed in the State of Michigan. Top-of-casing and ground surface elevations were recorded to the nearest 0.01 foot.

4.0 CCR GROUNDWATER MONITORING SYSTEM DESCRIPTION

Based on site-specific hydrogeologic information and groundwater flow, 11 shallow monitoring wells were selected as the groundwater monitoring system for the inactive Bottom Ash Impoundment. The number, spacing, and depth of monitoring wells was based on a thorough characterization of the hydrogeologic factors included in Part 115 R 299.4906. As noted in Section 3.1 above, each well was installed into the uppermost water-bearing zone underlying the site. It was determined that, although MW-8S was installed in the fine sand with silt in the uppermost water-bearing zone (similar to MW-3S), potentiometric data and discharge canal dredging information indicates that there is no hydraulic connection between MW-8S and the CCR unit. Groundwater flow potential in the vicinity of MW-8S is generally east toward the CCR unit and, given that the historical dredging depth went below the clay unit and intercepted the uppermost water-bearing zone, there is a vertical flow pathway between the uppermost water-bearing zone and the discharge canal (detail provided in Appendix B). The zone is comprised primarily of sand with varying amounts of silt present between approximately 25 to 35 feet below ground surface (bgs) on site. Each well is equipped with a dedicated bladder pump system and tubing installed for sampling purposes.

Monitoring well locations are shown on **Figure 3**. **Table 1** contains information regarding well locations and construction details. Well lithologic and construction logs are included as **Attachment A**.

5.0 CCR GROUNDWATER MONITORING SYSTEM CERTIFICATION

AECOM ("Consultant") has been retained by DTE Energy to provide certification of the groundwater monitoring system as required under Part 115 R 299.4906(7)(b) and 40 CFR § 257.91(f) of the HAZARDOUS AND SOLID WASTE MANAGEMENT SYSTEM; DISPOSAL OF COAL COMBUSTION RESIDUALS FROM ELECTRIC UTILITIES; FINAL RULE, 80 Fed. Reg. 21302 (Apr. 17, 2015) ("CCR Rule") for the inactive CCR unit identified by DTE Energy at their Monroe Plant located in Monroe, Michigan.

Requirements

Pursuant to Part 115 the owner or operator of an inactive CCR unit must install a groundwater monitoring system that meets the requirements of Part 115 R 299.4906. The groundwater monitoring system must meet the Part 115 performance standard, which requires the system to consist of a sufficient number of

wells, installed at appropriate locations and depths, to yield groundwater samples from the uppermost aquifer that accurately represent the quality of:

- (1) background groundwater that has not been affected by leakage from a CCR unit; and
- (2) groundwater passing the waste boundary of the CCR unit and monitoring all potential contaminant pathways.

The CCR unit identified at the site is the Inactive Bottom Ash Impoundment. The groundwater monitoring system requirement is addressed by a single system consisting of 11 monitoring wells. Information regarding the groundwater monitoring system design and construction has been provided to the qualified professional engineer as required by Part 115 R 299.4906(7)(b) and .

Limitations

The signature of Consultant's authorized representative on this document represents that to the best of Consultant's knowledge, information, and belief in the exercise of its professional judgment, it is Consultant's professional opinion that the aforementioned information is accurate as of the date of such signature. Any opinion or decisions by Consultant are made on the basis of Consultant's experience, qualifications, and professional judgment and are not to be construed as warranties or guaranties. In addition, opinions relating to environmental, geologic, and geotechnical conditions or other estimates are based on available data, and actual conditions may vary from those encountered at the times and locations where data are obtained, despite the use of due care.

6.0 CERTIFICATION

I, Scott Hutsell, being a Registered Professional Engineer, in accordance with the State of Michigan Professional Engineer's Registration program, possessing the technical knowledge and experience to make the specific technical certifications required under Part 115 and 40 Code of Federal Regulations (CFR) Part 257, Subpart D, Standards for the Disposal of Coal Combustion Residuals (CCRs) in Landfills and Surface Impoundments, and being licensed in the state where the CCR unit(s) is located, do hereby certify to the best of my knowledge, information, and belief, that the groundwater monitoring system that is the subject of this certification has been designed and constructed to meet the requirements of R 299.4906 and 40 CFR § 257.91.

Signature: _____

Date: _____

License #: _____

License Renewal Date: _____



TABLE

**TABLE 1
DTE ENERGY
MONROE POWER PLANT
MONITORING WELL CONSTRUCTION SUMMARY**

| Well ID | Easting | Northing | Well Installation Date | TOC Elevation (ft MSL) | Ground Surface Elevation (ft MSL) | Total Depth (ft BTOC) | Bottom Elevation (ft MSL) | Screen Length (feet) | Top of Screen Elevation (ft MSL) | Bottom of Screen Elevation (ft MSL) | Pump Depth (ft BTOC) | Well Casing Material | Well Screen Material and Slot Size | Groundwater Flow Location | Program Use |
|---------|-------------|-----------|------------------------|------------------------|-----------------------------------|-----------------------|---------------------------|----------------------|----------------------------------|-------------------------------------|----------------------|------------------------|---|---------------------------|----------------|
| MW-1S | 13401951.05 | 140176.14 | 9/19/2016 | 582.62 | 579.80 | 43.82 | 538.80 | 10 | 548.80 | 538.80 | 40.74 | 2-inch Schedule 40 PVC | 2-inch Schedule 40 PVC and 0.01-inch slot | Downgradient | Detection |
| MW-2S | 13401077.48 | 139070.06 | 9/19/2016 | 578.85 | 579.20 | 49.65 | 529.20 | 10 | 548.20 | 538.20 | 37.34 | | | | |
| MW-3S | 13399871.43 | 139417.18 | 9/20/2016 | 577.58 | 578.10 | 39.48 | 538.10 | 10 | 548.10 | 538.10 | 35.00 | | | Downgradient | |
| MW-7S | 13399510.36 | 141102.76 | 9/28/2016 | 576.20 | 576.60 | 33.60 | 542.60 | 10 | 552.60 | 542.60 | 29.70 | | | | |
| MW-9 | 13399606.60 | 140623.10 | 9/19/2017 | 579.05 | 576.37 | 37.73 | 541.32 | 10 | 551.37 | 541.37 | 33.00 | | | Downgradient | |
| MW-10 | 13399724.80 | 140207.50 | 9/20/2017 | 577.46 | 577.79 | 36.58 | 540.88 | 10 | 550.79 | 540.79 | 31.50 | | | | |
| MW-11 | 13399991.4 | 138811.7 | 9/20/2017 | 580.58 | 577.84 | 41.90 | 538.68 | 10 | 547.84 | 537.84 | 36.00 | | | | |
| MW-12 | 13400748.3 | 138911.9 | 9/21/2017 | 582.49 | 579.90 | 44.79 | 537.70 | 10 | 547.90 | 537.90 | 39.00 | | | Downgradient | |
| MW-13 | 13401644.6 | 139800.4 | 9/21/2017 | 580.97 | 578.25 | 38.08 | 542.89 | 10 | 553.25 | 543.25 | 33.00 | | | | |
| MW-14 | 13401772.2 | 141406.5 | 9/22/2017 | 580.76 | 577.87 | 42.67 | 538.09 | 10 | 547.87 | 537.87 | 37.50 | | | | |
| MW-15 | 13399419.6 | 141789.1 | 9/26/2017 | 580.80 | 578.11 | 40.88 | 539.92 | 10 | 549.61 | 539.61 | 36.00 | | | | |
| MW-4S | 13401614.14 | 141163.06 | 9/26/2016 | 580.67 | 578.10 | 42.57 | 538.10 | 10 | 551.10 | 541.10 | 35.84 | | | N/A | Potentiometry* |
| MW-5S | 13401176.41 | 142564.92 | 10/4/2016 | 584.50 | 581.70 | 72.80 | 511.70 | 10 | 568.70 | 558.70 | 20.96 | | | | |
| MW-8S | 13397828.28 | 140560.53 | 9/30/2016 | 586.59 | 583.70 | 45.89 | 540.70 | 10 | 550.70 | 540.70 | 42.57 | | | | |
| MW-1D | 13401952.04 | 140178.92 | 9/19/2016 | 582.82 | 579.40 | 83.42 | 499.40 | 10 | 509.40 | 499.40 | N/A | | | | |
| MW-3D | 13399871.16 | 139422.09 | 9/20/2016 | 577.42 | 578.00 | 79.42 | 498.00 | 10 | 509.00 | 499.00 | N/A | | | | |
| MW-7D | 13399510.92 | 141099.21 | 9/28/2016 | 576.17 | 576.70 | 69.47 | 506.70 | 10 | 517.70 | 507.70 | N/A | | | | |
| MW-8D | 13397828.00 | 140561.00 | 9/30/2016 | 586.45 | 583.70 | 72.75 | 513.70 | 10 | 527.20 | 517.20 | N/A | | | | |

TOC - Top of Casing
ft MSL - feet above Mean Sea Level
ft BTOC - feet below top of casing
PVC - Polyvinyl Chloride
* Monitoring wells used for potentiomeric evaluation only

FIGURES



Aerial Source: Esri, DigitalGlobe, GeoEye, Earthstar Geographics, CNES/Airbus DS, USDA, USGS, AEX, Getmapping, Aerogrid, IGN, IGP, swisstopo, and the GIS User Community and Google Earth 2016 Aerial Imagery

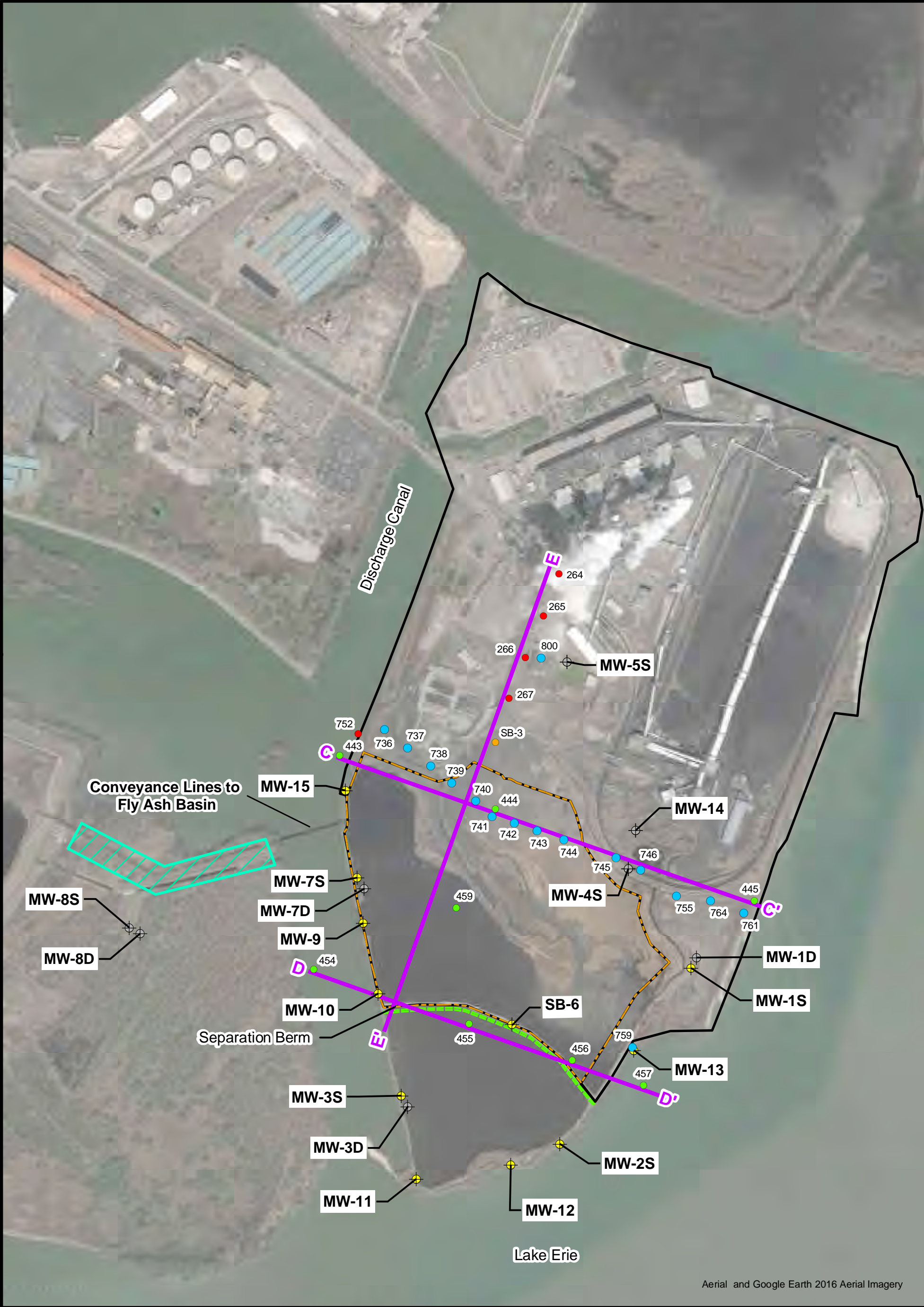
- LEGEND:**
- Unit Separation Berm
 - Approximate Plant Boundary
 - Revised Approximate Boundary of Inactive Bottom Ash Impoundment



Monroe Power Plant

FIGURE 1
SITE VICINITY MAP

| | |
|------------------|-------------------------|
| DATE: 7/22/2016 | SCALE 1 inch = 720 feet |
| CREATED BY: KLP | |
| JOB NO. 60489524 | |



Aerial and Google Earth 2016 Aerial Imagery

LEGEND:

- CCR Program Monitoring Well
- Investigation Monitoring Well (Potentiometry Only)
- Soil Borings (NTH)**
 - Soil Boring pre-1971
 - Soil Boring 1971 and later
 - Soil Boring 1971 and later with ash fill
 - Soil Boring in 2015 by NTH with ash fill
- Unit Separation Berm
- Cross Section Location
- Conveyance Line
- Boring Corridor (No Basal Sand)
- Approximate Plant Boundary
- Revised Approximate Boundary of Inactive Bottom Ash Impoundment

0 700 1,400 Feet



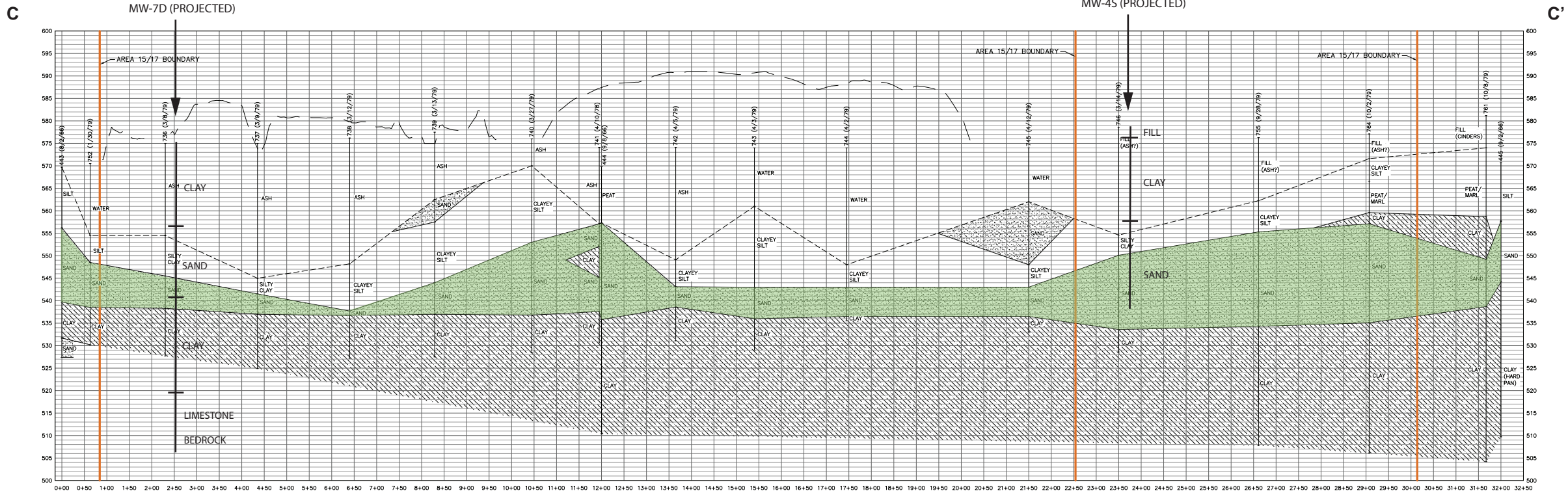
DTE Energy Monroe Power Plant

**FIGURE 2
GEOLOGIC CROSS-SECTION
LOCATION MAP**

| | |
|------------------|-------------------------|
| DATE: 3/21/2019 | SCALE 1 inch = 700 feet |
| JOB NO. 60516675 | |

NORTHWEST

SOUTHEAST



LEGEND

| | |
|--|-------------------|
| | ASH |
| | CLAY |
| | SAND |
| | UPPERMOST AQUIFER |

PRELIMINARY
NOT FOR CONSTRUCTION

| C | | | | B | | | | A | | | | DETROIT EDISON APPROVALS | | DATE | | OTHER DISCIPLINE APPROVALS | | DATE | | DESIGNED BY | | DATE | |
|------------|------------|--|--|------------|------------|--|--|------------|------------|--|--|--------------------------|--|------|--|----------------------------|--|------|--|------------------|--|-----------|--|
| PROJ. ENG. | PROJ. MGR. | | | PROJ. ENG. | PROJ. MGR. | | | PROJ. ENG. | PROJ. MGR. | | | ARCH | | | | ARCH | | | | ACE | | 7/13/2015 | |
| | | | | | | | | | | | | FOS GEN | | | | | | | | DRAWN BY | | 7/13/2015 | |
| | | | | | | | | | | | | TECH ORG | | | | | | | | CWS | | 7/13/2015 | |
| | | | | | | | | | | | | | | | | | | | | CHECKED BY | | 7/13/2015 | |
| | | | | | | | | | | | | FOS GEN | | | | | | | | DRL | | 7/13/2015 | |
| | | | | | | | | | | | | PLANT | | | | | | | | APPROVED BY | | | |
| | | | | | | | | | | | | ENG | | | | | | | | PROJECT ENGINEER | | | |
| | | | | | | | | | | | | | | | | | | | | PROJECT MANAGER | | | |
| | | | | | | | | | | | | DISC | | | | | | | | | | | |
| | | | | | | | | | | | | OPR | | | | | | | | | | | |
| | | | | | | | | | | | | PROJECT | | | | | | | | | | | |
| | | | | | | | | | | | | ENG | | | | | | | | | | | |
| | | | | | | | | | | | | COMP | | | | | | | | | | | |

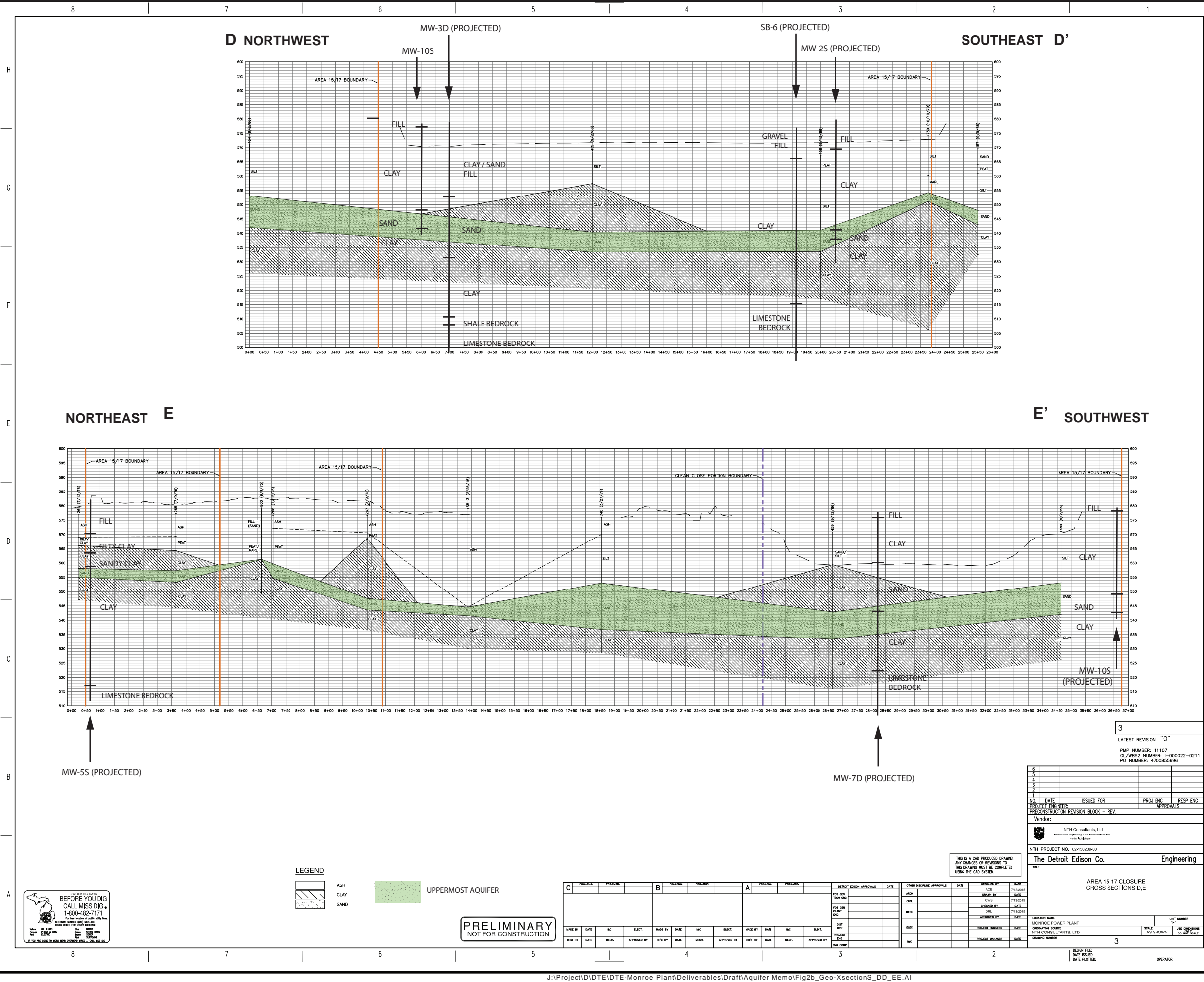
| | | | | | |
|---|------|------------|------------|------------|--|
| 6 | | | | | |
| 5 | | | | | |
| 4 | | | | | |
| 3 | | | | | |
| 2 | | | | | |
| 1 | | | | | |
| NO. | DATE | ISSUED FOR | PROJ. ENG. | RESP. ENG. | |
| PROJECT ENGINEER: APPROVALS | | | | | |
| PRECONSTRUCTION REVISION BLOCK - REV. | | | | | |
| Vendor: | | | | | |
| NTH Consultants, Ltd. Infrastructure Engineering & Environmental Services Brentwood, TN | | | | | |
| NTH PROJECT NO. 62-150239-00 | | | | | |
| The Detroit Edison Co. Engineering | | | | | |
| TITLE | | | | | |
| AREA 15-17 CLOSURE CROSS SECTIONS A,B,C | | | | | |
| LOCATION NAME | | | | | |
| MONROE POWER PLANT | | | | | |
| ORIGINATING SOURCE | | | | | |
| NTH CONSULTANTS, LTD. | | | | | |
| DRAWING NUMBER | | | | | |
| 2 | | | | | |
| DESIGN FILE: | | | | | |
| DATE ISSUED: | | | | | |
| DATE PLOTTED: | | | | | |
| OPERATOR: | | | | | |

Monroe Power Plant

FIGURE 2a
NTH CONSULTANTS, LTD - GEOLOGIC
CROSS-SECTION C - C'

| | | | |
|----------|----------|---------|----------|
| DATE | REV. NO. | DWG. BY | CHKD. BY |
| 03/20/19 | 1 | JMA | WBL |

JOB NO. 60516675



Monroe Power Plant

FIGURE 2b
NTH CONSULTANTS, LTD
GEOLOGIC CROSS-SECTIONS D - D' AND E - E'

| DATE | REV NO. | DWG. BY | CHKD. BY |
|----------|---------|---------|----------|
| 03/20/19 | 1 | JMA | WBL |

JOB NO. 60489524

AECOM



- LEGEND:**
- CCR Program Monitoring Well
 - Investigation Monitoring Well (Potentiometry Only)

- Unit Separation Berm
- Approximate Plant Boundary
- Revised Approximate Boundary of Inactive Bottom Ash Impoundment

0 700 1,400 Feet



Monroe Power Plant

FIGURE 3
INACTIVE BOTTOM ASH IMPOUNDMENT
WELL LOCATION MAP
2018

DATE: 3/20/2019

SCALE 1 inch = 720 feet

JOB NO. 60516675



Aerial and Google Earth 2016 Aerial Imagery

LEGEND:

| | |
|--|---|
| Monitoring Well | Unit Separation Berm |
| Investigation Monitoring Well (Potentiometry Only) | Approximate Plant Boundary |
| Groundwater Contour (Dashed where inferred) | Revised Approximate Boundary of Inactive Bottom Ash Impoundment |

0 700 1,400 Feet

DTE Energy

Monroe Power Plant

FIGURE 4a
EVENT 3 (NO MW-5S)
GROUNDWATER CONTOUR MAP
3/12/2018

| | |
|------------------|-------------------------|
| DATE: 3/20/2019 | SCALE 1 inch = 720 feet |
| JOB NO. 60516675 | |



- LEGEND:
- Monitoring Well
 - Investigation Monitoring Well (Potentiometry Only)
 - Groundwater Contour (Dashed where inferred)

- Unit Separation Berm
- Approximate Plant Boundary
- Revised Approximate Boundary of Inactive Bottom Ash Impoundment

0 700 1,400 Feet



Monroe Power Plant

FIGURE 4b
EVENT 6 (NO MW-5S)
GROUNDWATER CONTOUR MAP
9/24/2018

DATE: 3/20/2019

SCALE 1 inch = 720 feet

JOB NO. 60516675

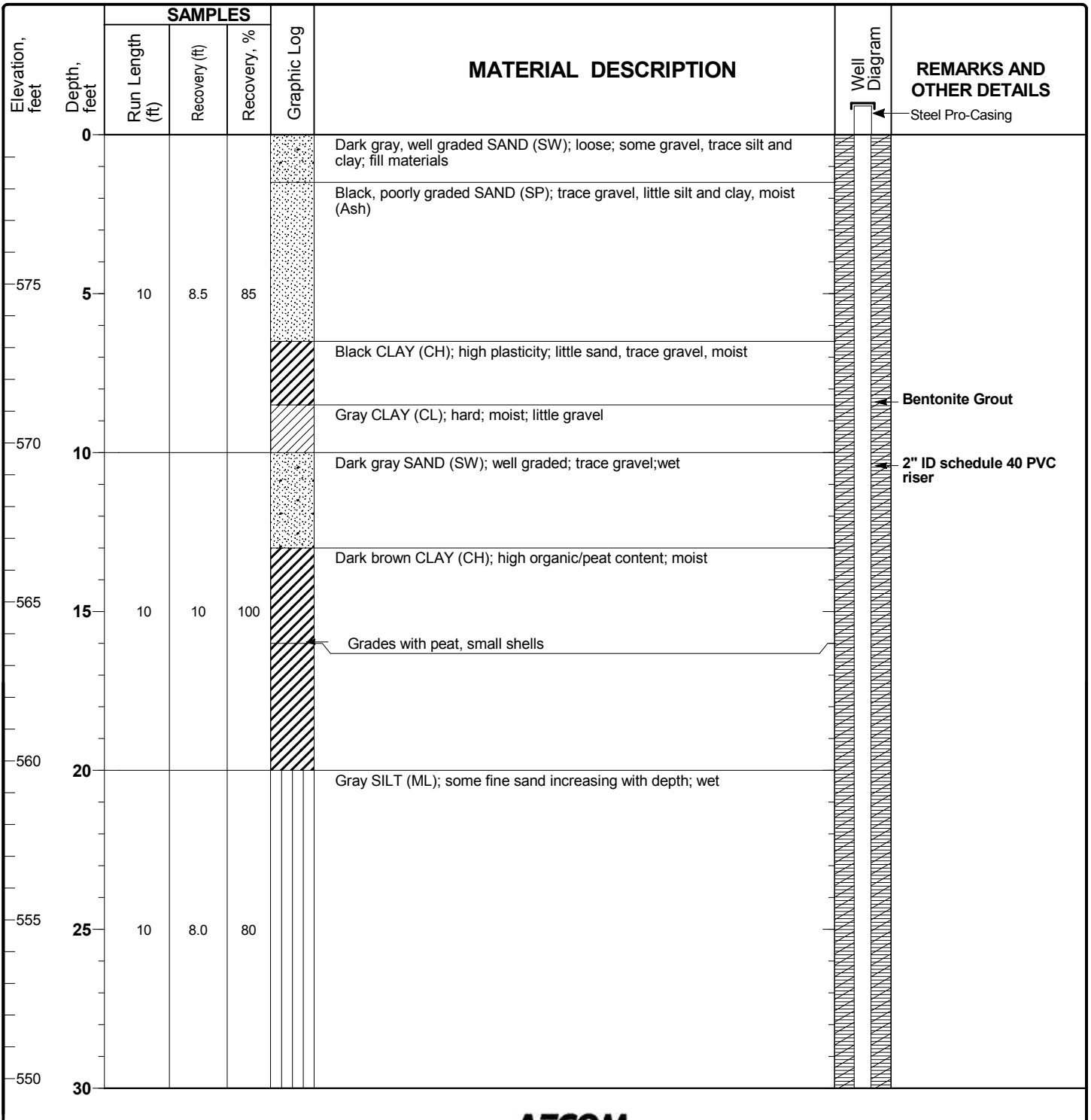
APPENDIX A
Monitoring Well Construction Logs

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-1D**
 Sheet 1 of 3

| | | |
|---|---|---|
| Date(s) Drilled 9/15/16 to 9/19/2016 | Logged By Ron Friend | Checked By M Hawrylak |
| Drilling Method Sonic | Drill Bit Size/Type Sonic 6" | Total Depth of Borehole 80.0 ft |
| Drill Rig Type Mini Sonic | Drilling Contractor Cascade Drilling | Surface Elevation 579.7 ft msl |
| Borehole Backfill Monitoring Well | Sampling Method(s) Sonic Core Barrel - 4" | Top of Casing Elevation 582.60 ft msl |
| Boring Location Inactive Bottom Ash Basin | Groundwater Level(s) Artesian (flowing) [Measurement after development] | |

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:25:06 PM



Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

**Log of
MW-1D**
 Sheet 2 of 3

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:25:06 PM

| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|-------------|--|--|------------------------------|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | | | | |
| | | | | | | Dark gray fine SAND (SP-SM); poorly graded; some silt; wet | | |
| 545 | 35 | 10 | 8.5 | 85 | | | | |
| | | | | | | same as above; decayed wood present | | |
| | | | | | | Gray GRAVEL (GW); rounded; well graded; trace clay; wet | | Bentonite Grout |
| 540 | 40 | | | | | | | 2" ID schedule 40 PVC riser |
| | | | | | | Gray CLAY (CL); glacial till; hard; trace fine sand; moist | | |
| 535 | 45 | 10 | 10 | 100 | | | | |
| 530 | 50 | | | | | | | |
| | | 5 | 5.0 | 100 | | | | |
| | | | | | | Highly weathered LIMESTONE-SHALE (large bedrock inclusion in till) | | |
| 525 | 55 | | | | | | | |
| | | 5 | 5.0 | 100 | | | | |
| | | | | | | Gray CLAY (CL); glacial till; very hard; trace medium sand; dry | | |
| 520 | 60 | | | | | | | |
| | | 5 | 0.5 | 10 | | | | |
| 515 | 65 | | | | | | | |

Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

**Log of
MW-1D**
 Sheet 3 of 3

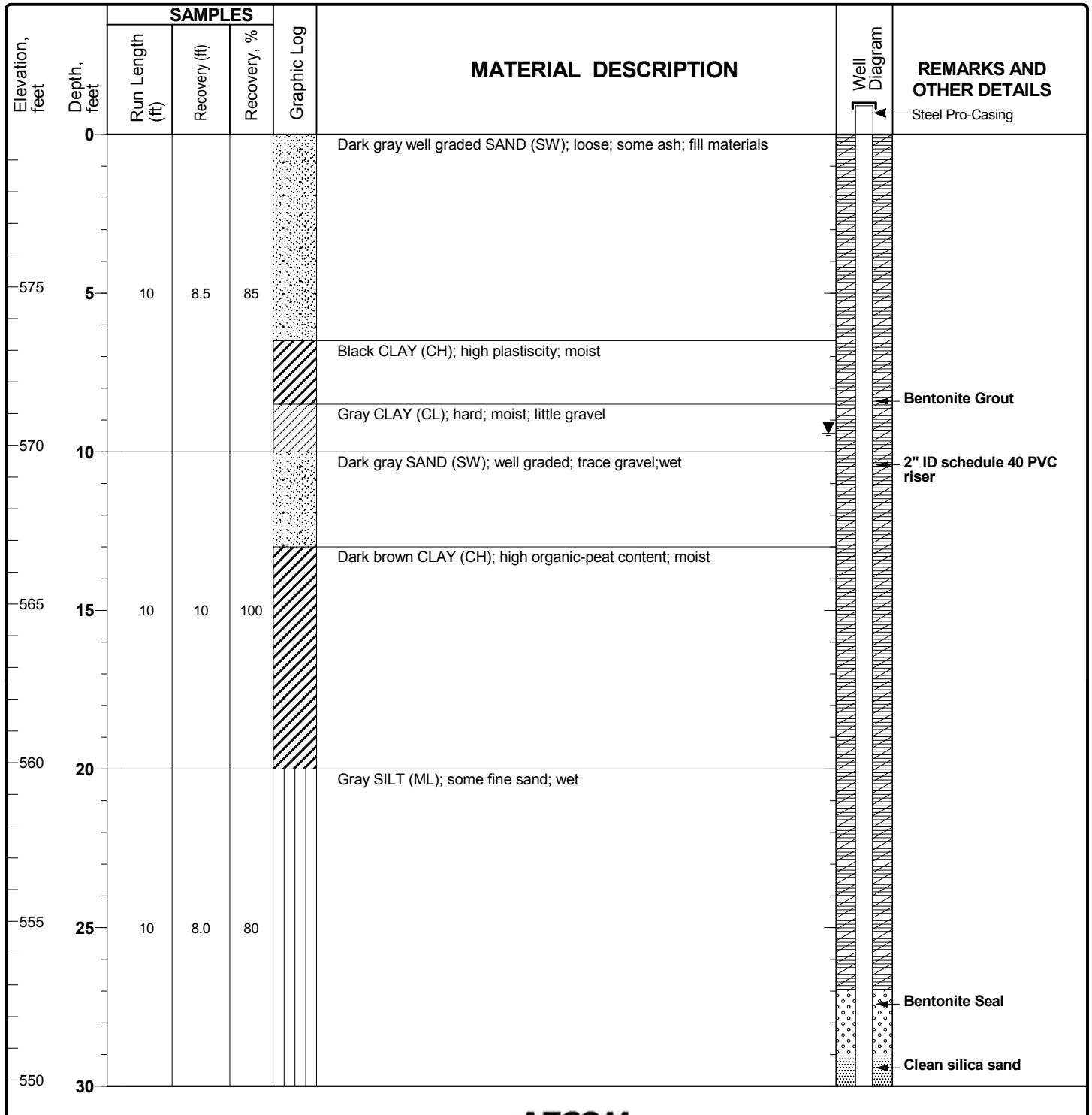
Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:25:07 PM

| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|-------------|---|--|--|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 510 | 70 | 5 | 2.75 | 55 | | | | Bentonite Grout 2" ID schedule 40 PVC riser Bentonite Seal |
| 505 | 75 | 5 | 4.0 | 80 | | LIMESTONE-SHALE (bedrock); gray, highly weathered; wet; some intact 2"-3" | | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| 500 | 80 | 5 | 5.0 | 100 | | LIMESTONE (bedrock); gray, slightly weathered; breaks apart with hammer blows; moist to wet | | Clean silica sand |
| 495 | 85 | | | | | | | End of boring |
| 490 | 90 | | | | | | | |
| 485 | 95 | | | | | | | |
| 480 | 100 | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-1S**
Sheet 1 of 2

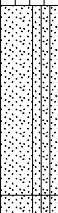
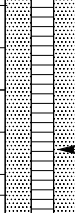

| | | |
|---|---|---|
| Date(s) Drilled 9/15/16 to 9/19/2016 | Logged By Ron Friend | Checked By M Hawrylak |
| Drilling Method Sonic | Drill Bit Size/Type Sonic 6" | Total Depth of Borehole 41.0 ft |
| Drill Rig Type Mini Sonic | Drilling Contractor Cascade Drilling | Surface Elevation 579.8 ft msl |
| Borehole Backfill Monitoring Well | Sampling Method(s) Sonic Core Barrel - 4" | Top of Casing Elevation 582.62 ft msl |
| Boring Location Inactive Bottom Ash Basin | Groundwater Level(s) 9.42' BTOC [Measurement after development] | |



Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

**Log of
MW-1S**
 Sheet 2 of 2

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:25:12 PM

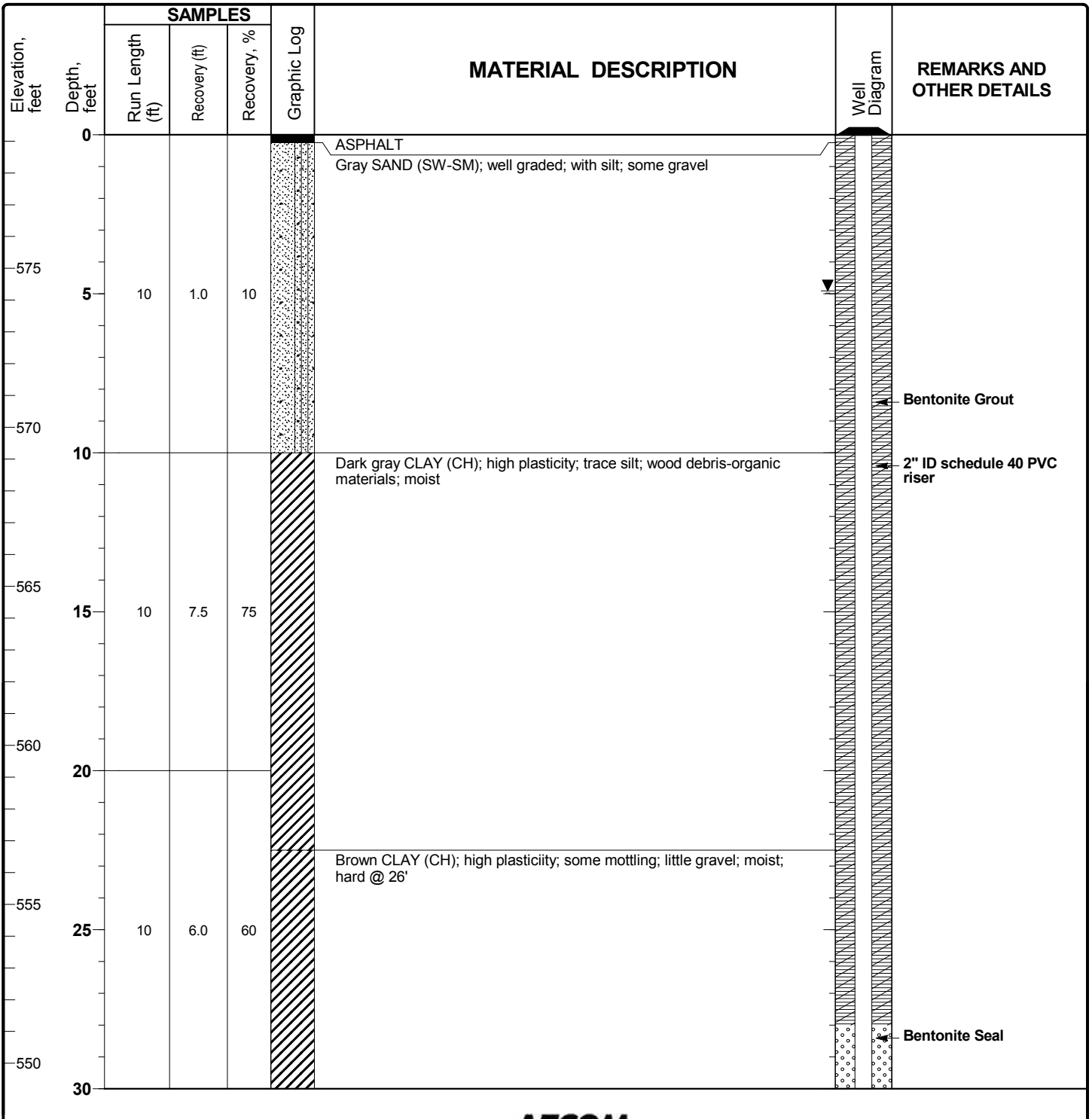
| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|---|--|---|---|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | | | | 2" ID schedule 40 PVC riser |
| | | | | | | | | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| 545 | 35 | 10 | 8.5 | 85 |  | Dark gray fine SAND (SP-SM); poorly graded; some silt; wet |  | Clean silica sand |
| 540 | 40 | | | |  | same as above; decayed wood present Gray GRAVEL (GW); rounded; well graded; trace clay; wet | | |
| 535 | 45 | 10 | 10 | 100 | | | | |
| 530 | 50 | | | | | | | End of boring |
| 525 | 55 | | | | | | | |
| 520 | 60 | | | | | | | |
| 515 | 65 | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-2S**
Sheet 1 of 2

| | | |
|---|---|---|
| Date(s) Drilled 9/19/16 to 9/19/2016 | Logged By Ron Friend | Checked By M Hawrylak |
| Drilling Method Sonic | Drill Bit Size/Type Sonic 6" | Total Depth of Borehole 50.0 ft |
| Drill Rig Type Mini Sonic | Drilling Contractor Cascade Drilling | Surface Elevation 579.2 ft msl |
| Borehole Backfill Monitoring Well | Sampling Method(s) Sonic Core Barrel - 4" | Top of Casing Elevation 578.85 ft msl |
| Boring Location Inactive Bottom Ash Basin | Groundwater Level(s) 4.91' BTOC [Measurement after development] | |

Report: DTE_MONROE; File J:\RESOURCEDISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:25:17 PM



Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-2S**
Sheet 2 of 2

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE GRANVILLE CLONE.GPJ; 10/27/2016 4:25:17 PM

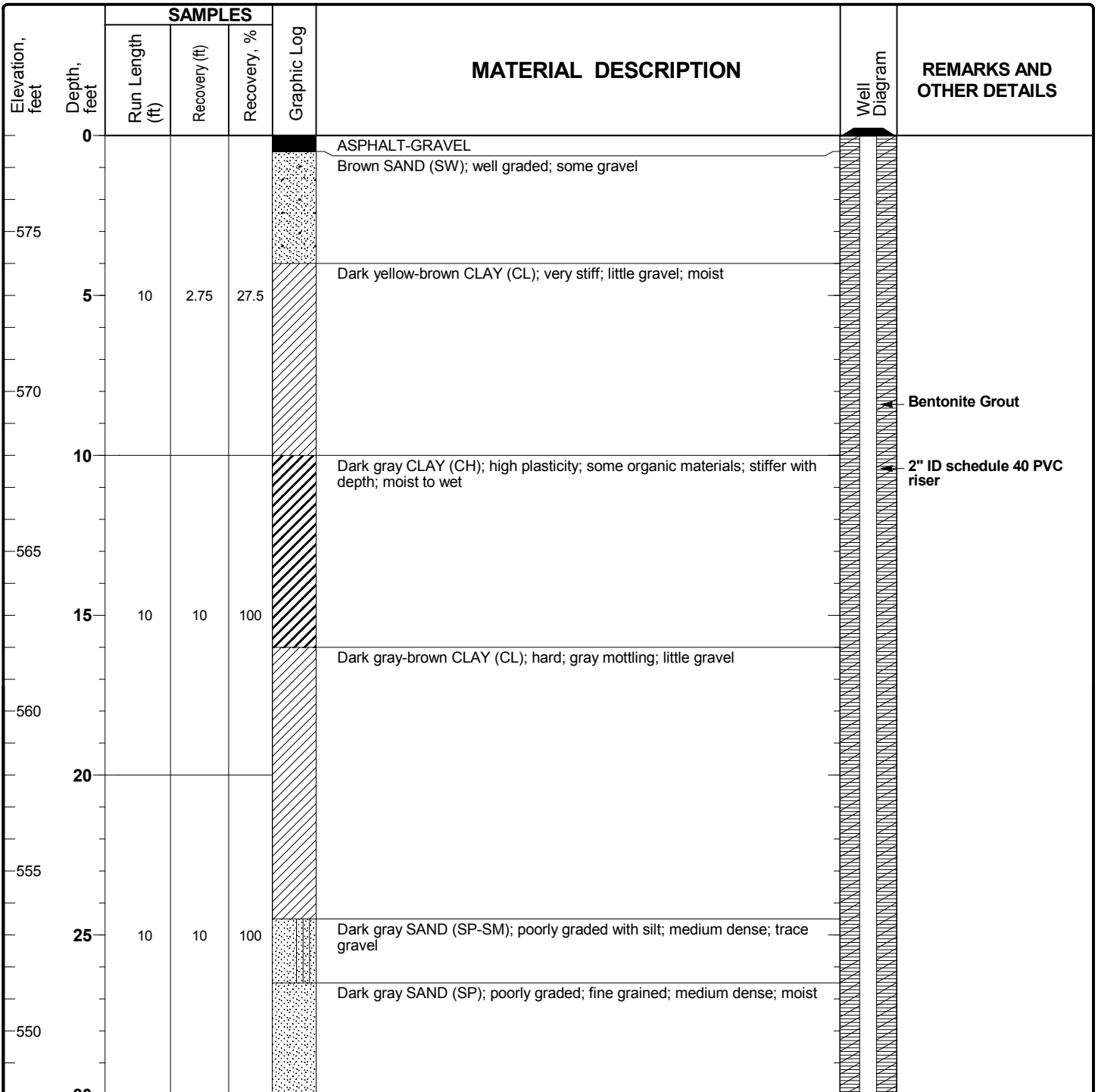
| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|-------------|---|--|---|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | | | | 2" ID schedule 40 PVC riser |
| | | | | | | Dark gray CLAY (CL); trace coarse sand; little gravel; very hard; dry-moist | | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| 545 | 35 | 10 | 8.5 | 85 | | | | Clean silica sand |
| | | | | | | Dark gray SAND (SP-SM); poorly graded; fine grained; some silt; wet | | |
| 540 | 40 | | | | | | | |
| | | | | | | Dark gray SAND (SP); poorly graded; wet | | |
| | | | | | | Dark Gray CLAY (CL); glacial till; very hard, trace gravel and sand; moist | | Bentonite Seal |
| 535 | 45 | 10 | 8.0 | 80 | | | | |
| 530 | 50 | | | | | | | |
| | | | | | | | | End of boring |
| 525 | 55 | | | | | | | |
| 520 | 60 | | | | | | | |
| 515 | 65 | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-3D**
Sheet 1 of 3

| | | |
|---|---|---|
| Date(s) Drilled 9/20/16 to 9/20/2016 | Logged By Ron Friend | Checked By M Hawrylak |
| Drilling Method Sonic | Drill Bit Size/Type Sonic 6" | Total Depth of Borehole 80.0 ft |
| Drill Rig Type Mini Sonic | Drilling Contractor Cascade Drilling | Surface Elevation 578.0 ft msl |
| Borehole Backfill Monitoring Well | Sampling Method(s) Sonic Core Barrel - 4" | Top of Casing Elevation 577.42 ft msl |
| Boring Location Inactive Bottom Ash Basin | Groundwater Level(s) Artesian (flowing) [Measurement after development] | |

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE GRANVILLE CLONE.GPJ; 10/27/2016 4:25:25 PM



Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-3D**
Sheet 2 of 3


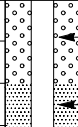





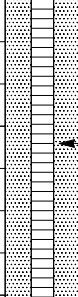

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:25:26 PM

| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|-------------|--|--|------------------------------|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | | | | |
| 545 | | | | | | Dark gray SAND (SP-SM); poorly graded with silt; fine grained; higher silt content at depth; wet | | |
| 35 | | 10 | 9.5 | 95 | | | | |
| 540 | | | | | | Gray SILT (ML); soft; little fine sand; wet | | Bentonite Grout |
| 40 | | | | | | Dark gray CLAY (CL); glacial till; hard; trace gravel and sand; moist | | 2" ID schedule 40 PVC riser |
| 535 | | | | | | | | |
| 45 | | 10 | 5.5 | 55 | | | | |
| 530 | | | | | | | | |
| 50 | | | | | | | | |
| 525 | | | | | | | | |
| 55 | | 10 | 5.5 | 55 | | | | |
| 520 | | | | | | | | |
| 60 | | | | | | | | |
| 515 | | 10 | 3.0 | 30 | | Gray highly weathered LIMESTONE (bedrock inclusion in till); some granite pebbles | | |
| 65 | | | | | | Dark gray CLAY (CL); glacial till; hard; trace gravel; moist | | Bentonite Seal |

Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

**Log of
MW-3D**
 Sheet 3 of 3

Report: DTE_MONROE; File J:\RESOURCE\DISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:25:27 PM

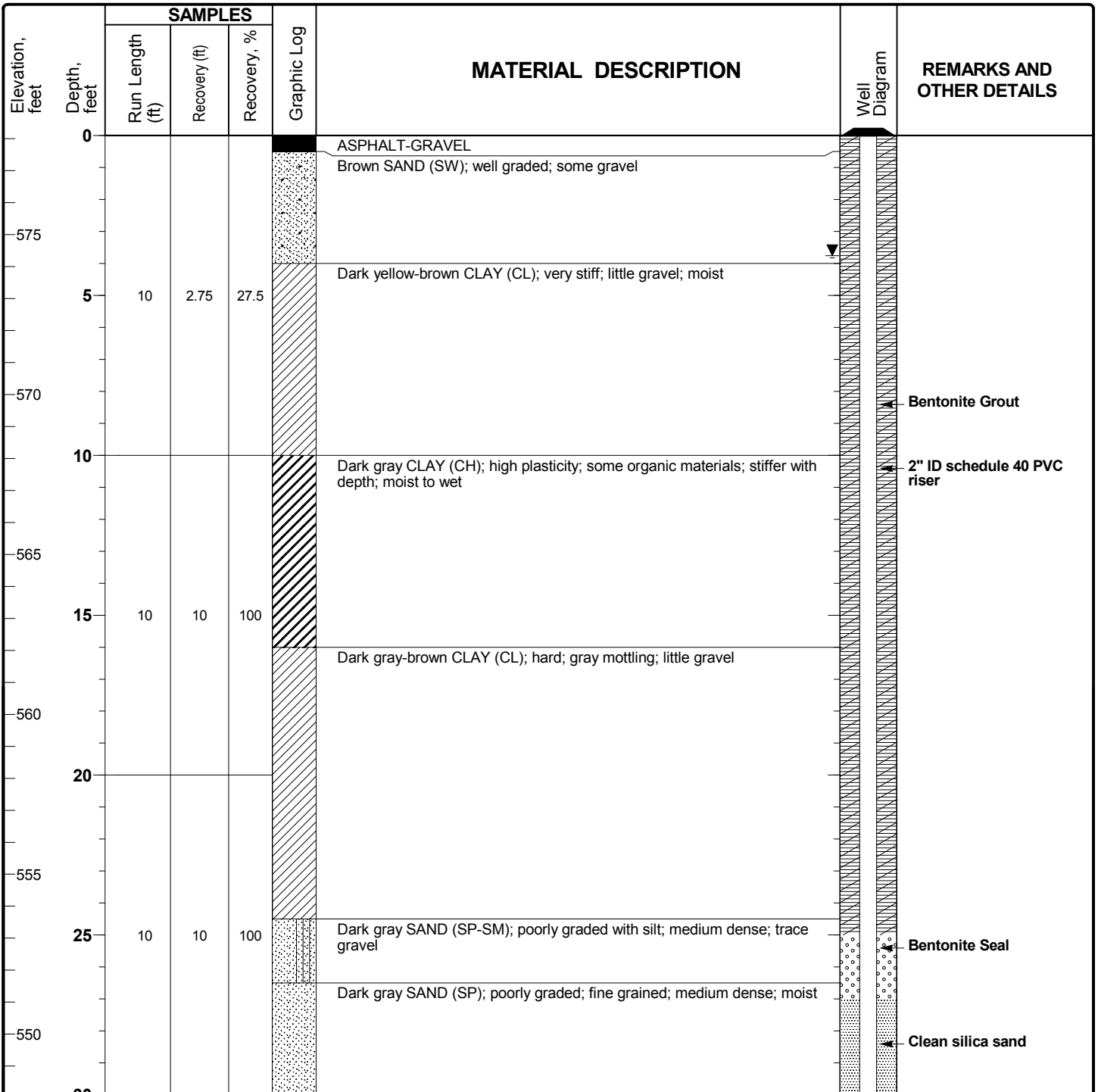
| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|---|---|--|---|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 510 | 70 | 10 | 3.0 | 30 |  | |  | Bentonite Seal |
| | | | | |  | Light gray SH (bedrock); highly weathered; broken from drilling process; moist to wet |  | 2" ID schedule 40 PVC riser |
| 505 | | | | |  | Gray LIMESTONE (bedrock); weathered; some intact sections (< 3" long); moist to wet |  | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| 75 | | 10 | 4.0 | 40 |  | |  | Clean silica sand |
| 500 | 80 | | | |  | Pale brown LIMESTONE (bedrock); highly weathered 1-2" long pieces | | |
| 495 | 85 | | | | | | | End of boring |
| 490 | 90 | | | | | | | |
| 485 | 95 | | | | | | | |
| 480 | | | | | | | | |
| 100 | | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-3S**
Sheet 1 of 2

| | | |
|---|---|---|
| Date(s) Drilled 9/20/16 to 9/20/2016 | Logged By Ron Friend | Checked By M Hawrylak |
| Drilling Method Sonic | Drill Bit Size/Type Sonic 6" | Total Depth of Borehole 40.0 ft |
| Drill Rig Type Mini Sonic | Drilling Contractor Cascade Drilling | Surface Elevation 578.1 ft msl |
| Borehole Backfill Monitoring Well | Sampling Method(s) Sonic Core Barrel - 4" | Top of Casing Elevation 577.58 ft msl |
| Boring Location Inactive Bottom Ash Basin | Groundwater Level(s) 3.76' BTOC [Measurement after development] | |

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:25:31 PM



Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

**Log of
MW-3S**
 Sheet 2 of 2

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:25:32 PM

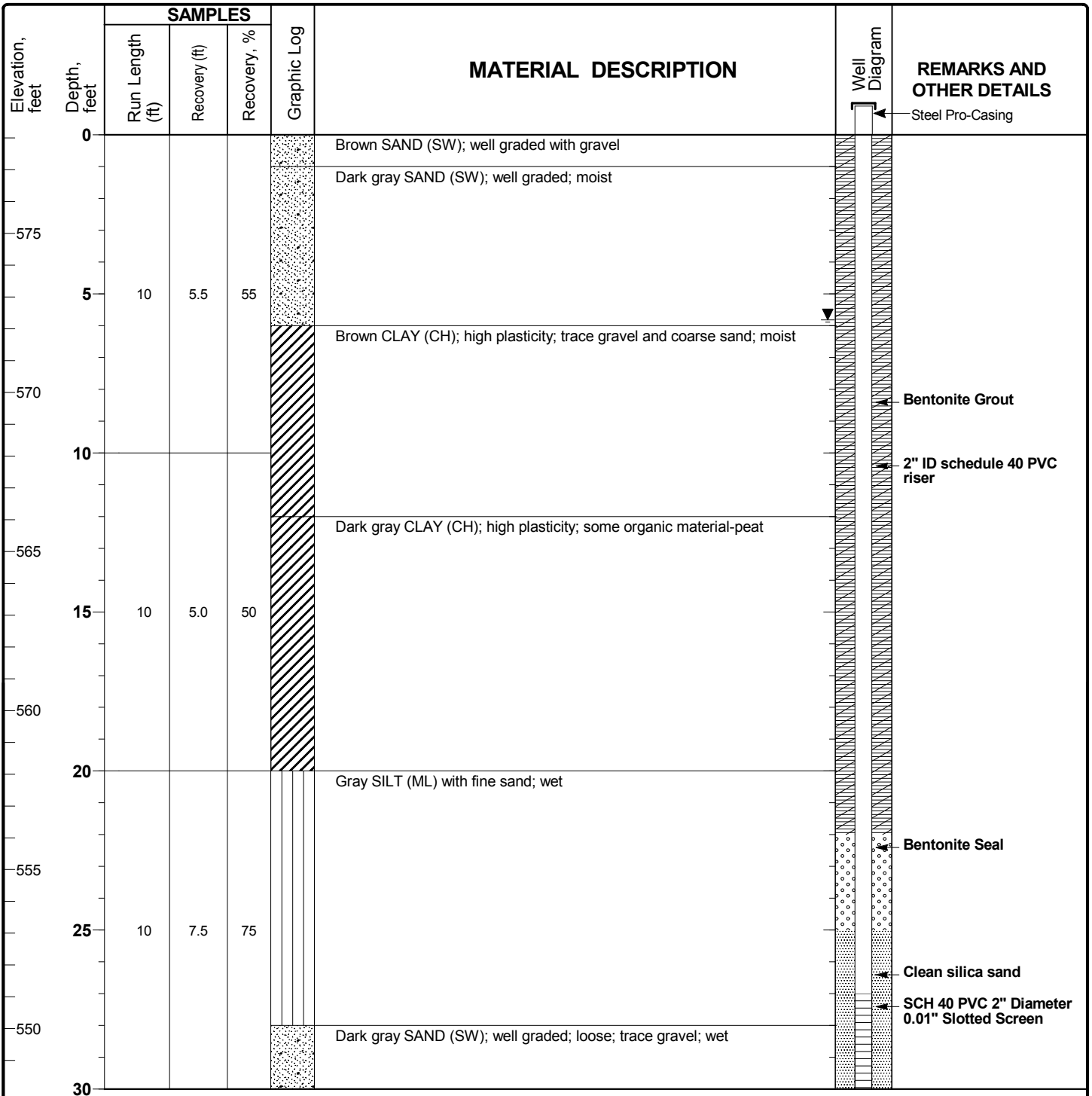
| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|-------------|---|--|--|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | | | | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| 545 | | | | | | Dark gray SAND (SP-SM); poorly graded with silt; fine grained; higher silt content at depth; wet | | 2" ID schedule 40 PVC riser |
| 35 | | 10 | 9.5 | 95 | | | | Clean silica sand |
| 540 | | | | | | Gray SILT (ML); soft; little fine sand; wet | | |
| 40 | | | | | | | | End of boring |
| 535 | | | | | | | | |
| 45 | | 10 | 5.5 | 55 | | | | |
| 530 | | | | | | | | |
| 50 | | | | | | | | |
| 525 | | | | | | | | |
| 55 | | | | | | | | |
| 520 | | | | | | | | |
| 60 | | | | | | | | |
| 515 | | | | | | | | |
| 65 | | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-4S**
Sheet 1 of 2

| | | |
|---|---|---|
| Date(s) Drilled 9/26/16 to 9/26/2016 | Logged By Ron Friend | Checked By M Hawrylak |
| Drilling Method Sonic | Drill Bit Size/Type Sonic 6" | Total Depth of Borehole 40.0 ft |
| Drill Rig Type Mini Sonic | Drilling Contractor Cascade Drilling | Surface Elevation 578.1 ft msl |
| Borehole Backfill Monitoring Well | Sampling Method(s) Sonic Core Barrel - 4" | Top of Casing Elevation 580.67 ft msl |
| Boring Location Inactive Bottom Ash Basin | Groundwater Level(s) 5.82' BTOC [Measurement after development] | |

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE GRANVILLE CLONE.GPJ; 10/27/2016 4:25:36 PM



Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

Log of
MW-4S
Sheet 2 of 2

| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|-------------|--|--|---|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | | | | 2" ID schedule 40 PVC riser |
| 545 | | | | | | | | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| 35 | | 10 | 7.5 | 75 | | Dark gray SILT (ML) with fine sand; wet | | Clean silica sand |
| 540 | | | | | | Gray CLAY (CL); glacial till; hard; trace gravel and coarse sand | | Bentonite Seal |
| 40 | | | | | | | | End of boring |
| 535 | | | | | | | | |
| 45 | | | | | | | | |
| 530 | | | | | | | | |
| 50 | | | | | | | | |
| 525 | | | | | | | | |
| 55 | | | | | | | | |
| 520 | | | | | | | | |
| 60 | | | | | | | | |
| 515 | | | | | | | | |
| 65 | | | | | | | | |

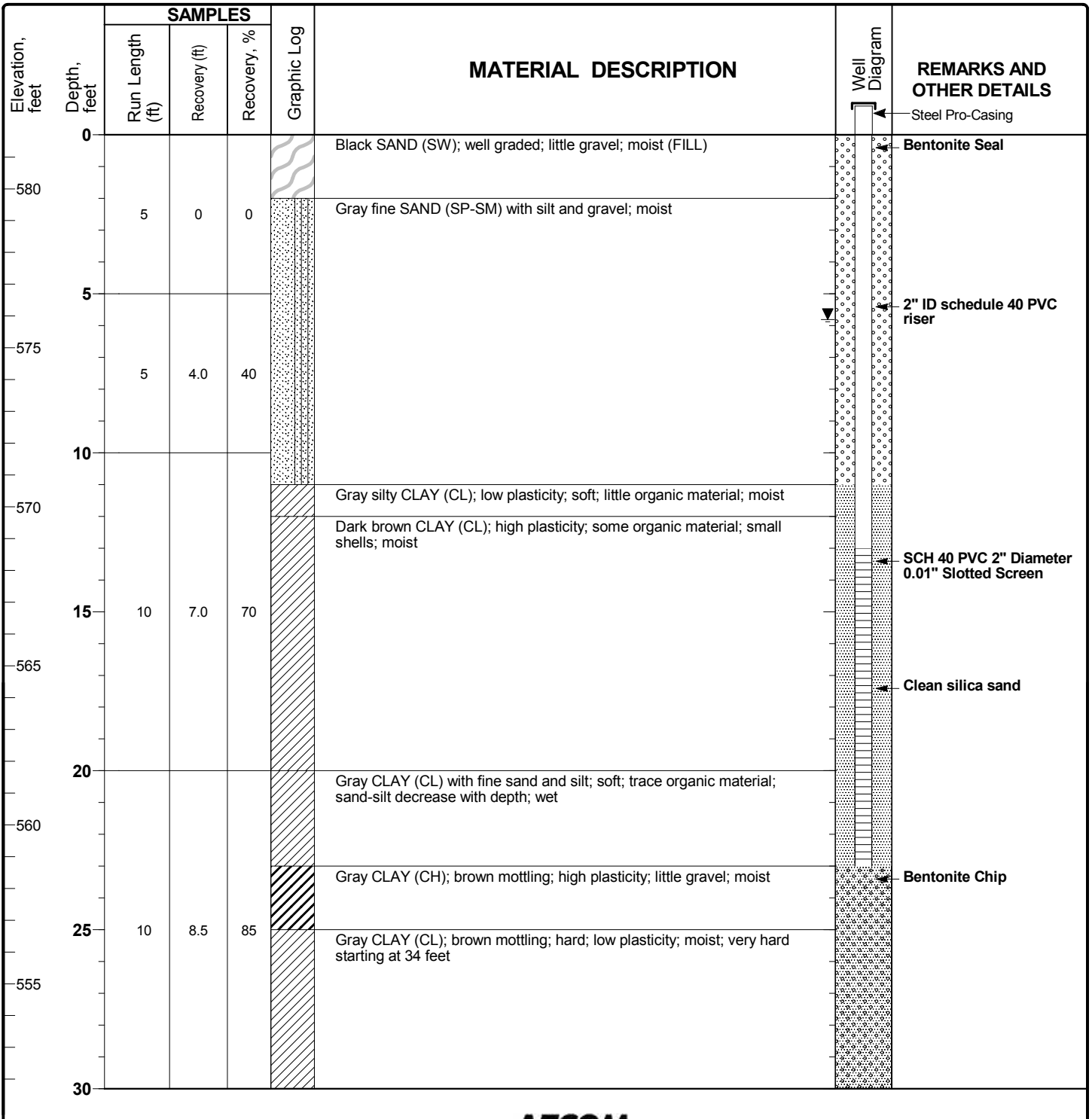
Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE GRANVILLE CLONE.GPJ; 10/27/2016 4:25:37 PM

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-5S**
Sheet 1 of 3

| | | |
|---|---|---|
| Date(s) Drilled 10/4/16 to 10/4/2016 | Logged By Ron Friend | Checked By M Hawrylak |
| Drilling Method Sonic | Drill Bit Size/Type Sonic 6" | Total Depth of Borehole 70.0 ft |
| Drill Rig Type Mini Sonic | Drilling Contractor Cascade Drilling | Surface Elevation 581.7 ft msl |
| Borehole Backfill Monitoring Well | Sampling Method(s) Sonic Core Barrel - 4" | Top of Casing Elevation 584.50 ft msl |
| Boring Location Inactive Bottom Ash Basin | Groundwater Level(s) 5.81' BTOC [Measurement after development] | |

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE GRANVILLE CLONE.GPJ; 10/27/2016 4:25:42 PM


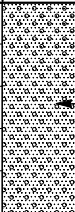


Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

Log of
 MW-5S
 Sheet 2 of 3

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:25:43 PM

| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|-------------|---|--|------------------------------|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | | | | |
| 550 | | | | | | | | |
| 35 | | 10 | 7.5 | 75 | | | | |
| 545 | | | | | | | | |
| 40 | | | | | | | | |
| 540 | | | | | | Gray CLAY (CH); brown mottling; high plasticity; little gravel; moist | | Bentonite Chip |
| 45 | | 10 | 8.5 | 85 | | Gray CLAY (CL); glacial till; very hard; moist to dry | | |
| 535 | | | | | | | | |
| 50 | | | | | | | | |
| 530 | | | | | | | | |
| 55 | | 10 | 5.5 | 55 | | | | |
| 525 | | | | | | | | |
| 60 | | | | | | | | |
| 520 | | 5 | 4.0 | 80 | | | | |
| 65 | | | | | | | | |

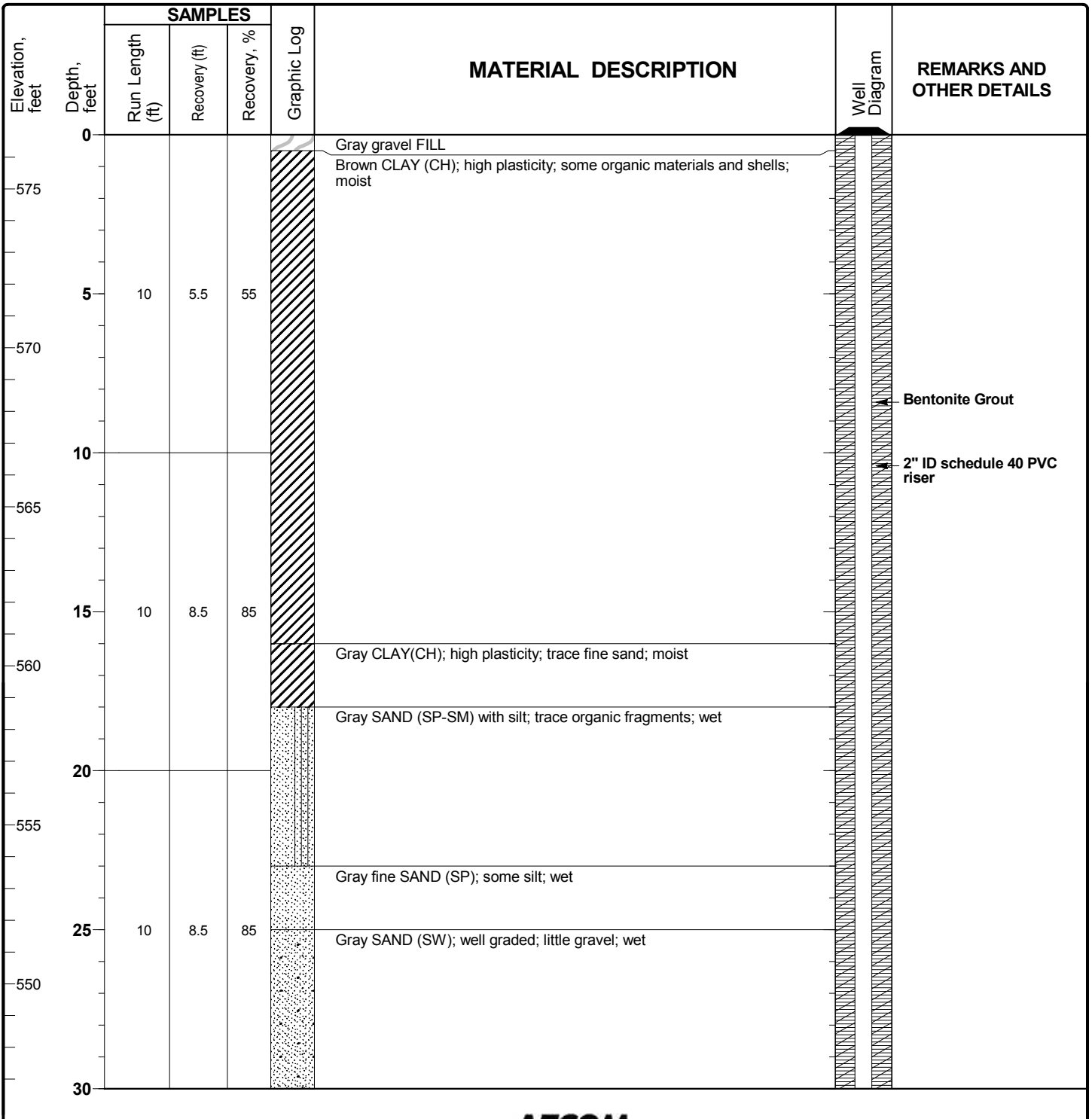
| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|---|---|---|------------------------------|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 515 | | 5 | 4.0 | 80 |  | Gray LIMESTONE (bedrock); highly weathered; dry from drilling methods but zone produces water |  | Bentonite Chip |
| 70 | | | | | | | | End of boring |
| 510 | | | | | | | | |
| 75 | | | | | | | | |
| 505 | | | | | | | | |
| 80 | | | | | | | | |
| 500 | | | | | | | | |
| 85 | | | | | | | | |
| 495 | | | | | | | | |
| 90 | | | | | | | | |
| 490 | | | | | | | | |
| 95 | | | | | | | | |
| 485 | | | | | | | | |
| 100 | | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-7D**
Sheet 1 of 3

| | | | | | |
|-------------------|---------------------------|----------------------|--|-------------------------|---------------|
| Date(s) Drilled | 9/28/16 to 9/28/2016 | Logged By | Ron Friend | Checked By | M Hawrylak |
| Drilling Method | Sonic | Drill Bit Size/Type | Sonic 6" | Total Depth of Borehole | 70.0 ft |
| Drill Rig Type | Mini Sonic | Drilling Contractor | Cascade Drilling | Surface Elevation | 576.7 ft msl |
| Borehole Backfill | Monitoring Well | Sampling Method(s) | Sonic Core Barrel - 4" | Top of Casing Elevation | 576.17 ft msl |
| Boring Location | Inactive Bottom Ash Basin | Groundwater Level(s) | Artesian (flowing) [Measurement after development] | | |

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE GRANVILLE CLONE.GPJ; 10/27/2016 4:25:50 PM



Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-7D**
Sheet 2 of 3

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:25:51 PM

| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|-------------|--|--|---|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | | | | |
| 545 | | | | | | Gray GRAVEL (GW); well graded with sand; wet | | |
| | | | | | | Gray SAND (SW); well graded; little gravel; wet | | |
| 35 | | 10 | 8.5 | 85 | | Dark gray CLAY (CL); glacial till; hard; trace gravel and coarse sand; moist | | |
| 540 | | | | | | | | |
| 40 | | | | | | | | Bentonite Grout |
| 535 | | | | | | | | 2" ID schedule 40 PVC riser |
| 45 | | 10 | 9.5 | 95 | | | | |
| 530 | | | | | | | | |
| 50 | | | | | | | | |
| 525 | | | | | | | | |
| 55 | | 10 | 10 | 100 | | Light gray LIMESTONE (bedrock); highly weathered; some larger pieces; wet | | Bentonite Seal |
| 520 | | | | | | | | |
| 60 | | | | | | Light gray LIMESTONE (bedrock); highly weathered; some intact pieces; poor recovery overall; wet | | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| 515 | | 10 | 2.5 | 25 | | | | |
| 65 | | | | | | | | Clean silica sand |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

Log of
MW-7D
Sheet 3 of 3

| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|-------------|----------------------|--|---|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 510 | | 10 | 2.5 | 25 | | | | 2" ID schedule 40 PVC riser SCH 40 PVC 2" Diameter 0.01" Slotted Screen Clean silica sand Collapse |
| 70 | | | | | | | | End of boring |
| 505 | | | | | | | | |
| 75 | | | | | | | | |
| 500 | | | | | | | | |
| 80 | | | | | | | | |
| 495 | | | | | | | | |
| 85 | | | | | | | | |
| 490 | | | | | | | | |
| 90 | | | | | | | | |
| 485 | | | | | | | | |
| 95 | | | | | | | | |
| 480 | | | | | | | | |
| 100 | | | | | | | | |

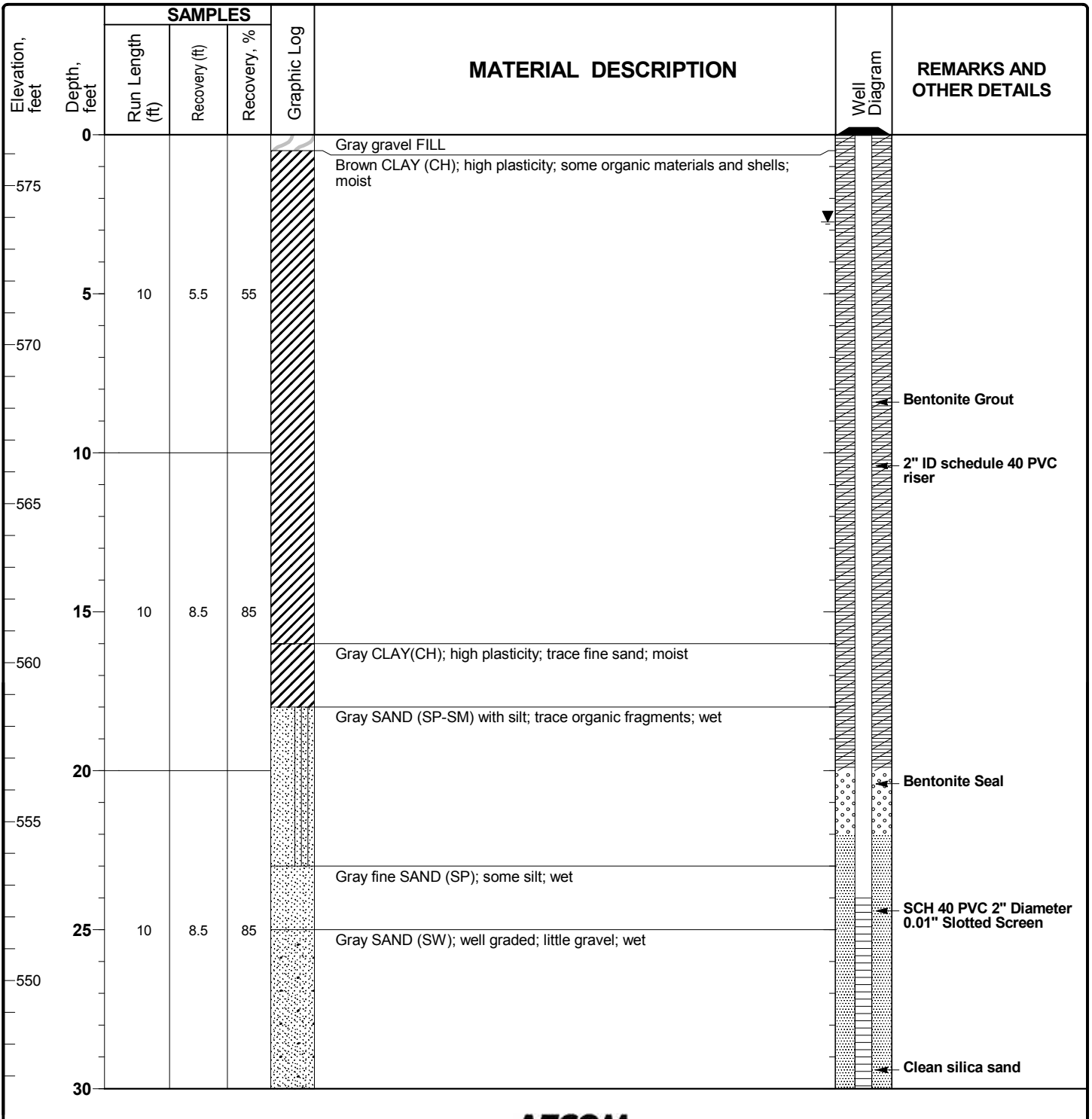
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Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-7S**
Sheet 1 of 2

| | | | | | |
|-------------------|---------------------------|----------------------|--|-------------------------|---------------|
| Date(s) Drilled | 9/28/16 to 9/28/2016 | Logged By | Ron Friend | Checked By | M Hawrylak |
| Drilling Method | Sonic | Drill Bit Size/Type | Sonic 6" | Total Depth of Borehole | 34.0 ft |
| Drill Rig Type | Mini Sonic | Drilling Contractor | Cascade Drilling | Surface Elevation | 576.6 ft msl |
| Borehole Backfill | Monitoring Well | Sampling Method(s) | Sonic Core Barrel - 4" | Top of Casing Elevation | 576.20 ft msl |
| Boring Location | Inactive Bottom Ash Basin | Groundwater Level(s) | 2.74' BTOC [Measurement after development] | | |

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE GRANVILLE CLONE.GPJ; 10/27/2016 4:25:56 PM

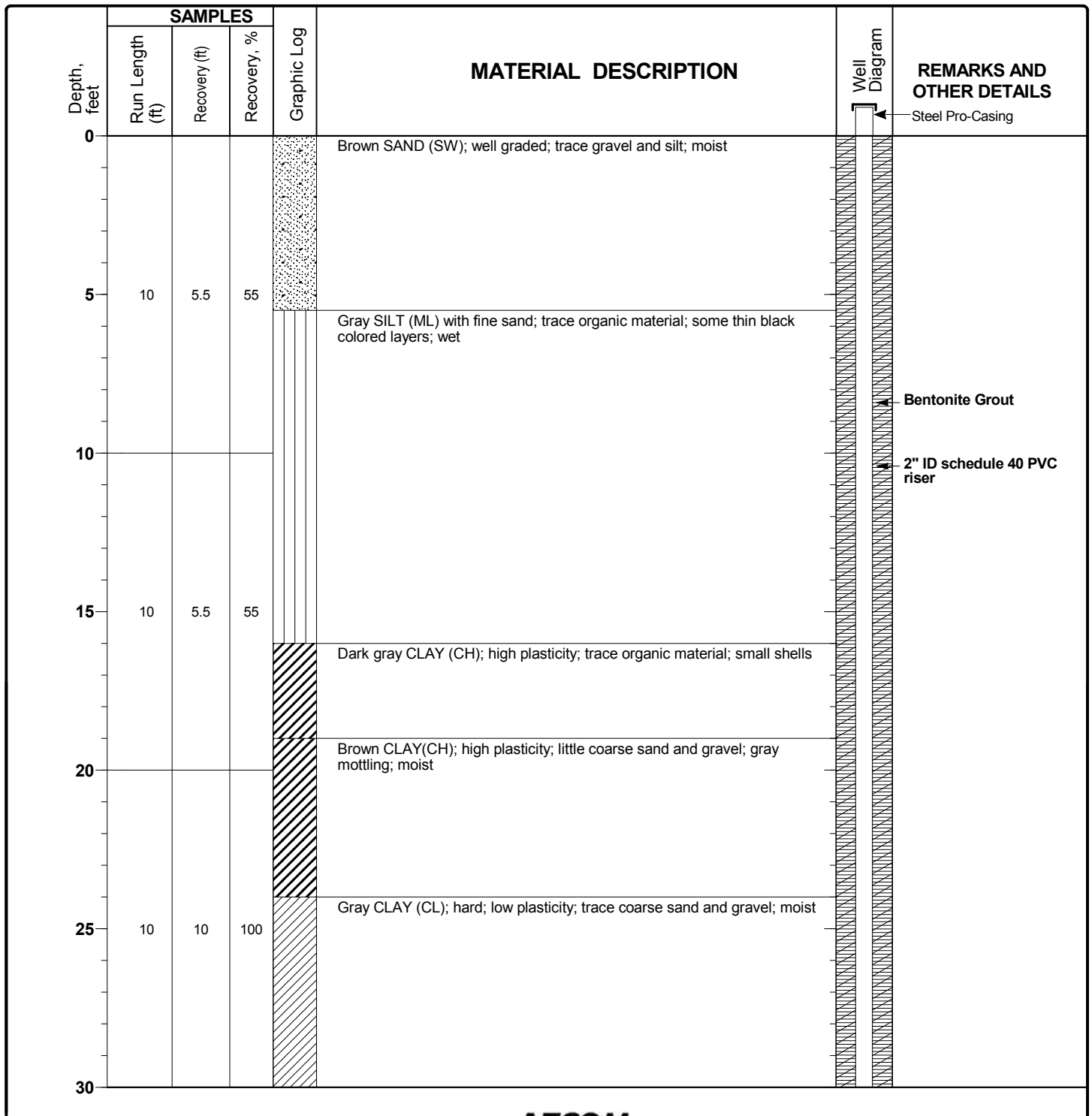


| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|-------------|---|--|---|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | | | | |
| 545 | | 4 | 3.4 | 85 | | Gray GRAVEL (GW); well graded with sand; wet | | 2" ID schedule 40 PVC riser |
| | | | | | | Gray SAND (SW); well graded; little gravel; wet | | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| | | | | | | | | Clean silica sand |
| 35 | | | | | | | | End of boring |
| 540 | | | | | | | | |
| 40 | | | | | | | | |
| 535 | | | | | | | | |
| 45 | | | | | | | | |
| 530 | | | | | | | | |
| 50 | | | | | | | | |
| 525 | | | | | | | | |
| 55 | | | | | | | | |
| 520 | | | | | | | | |
| 60 | | | | | | | | |
| 515 | | | | | | | | |
| 65 | | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-8D**
Sheet 1 of 3

| | | |
|--|---|---|
| Date(s) Drilled 9/29/16 to 9/30/2016 | Logged By Ron Friend | Checked By M Hawrylak |
| Drilling Method Sonic | Drill Bit Size/Type Sonic 6" | Total Depth of Borehole 70.0 ft |
| Drill Rig Type Mini Sonic | Drilling Contractor Cascade Drilling | Surface Elevation ft msl |
| Borehole Backfill Monitoring Well | Sampling Method(s) Sonic Core Barrel - 4" | Top of Casing Elevation ft msl |
| Boring Location Fly Ash Basin | Groundwater Level(s) Artesian (flowing) [Measurement after development] | |



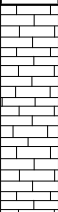
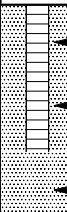
Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

Log of
 MW-8D
 Sheet 2 of 3

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|----------------|--------------------|---------------|-------------|-------------|--|--|---|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | | | |
| | 5 | 5 | 100 | | | | |
| 35 | | | | | Gray fine SAND (SP-SM) with silt; thin silt seams (<1" thick); wet | | |
| | 5 | 2.5 | 50 | | | | Bentonite Grout |
| 40 | | | | | | | 2" ID schedule 40 PVC riser |
| | | | | | Gray CLAY (CL); hard; glacial till; med-low plasticity; little gravel and coarse sand; moist | | |
| 45 | 10 | 8.0 | 80 | | | | |
| 50 | | | | | | | |
| | | | | | Light gray LIMESTONE (bedrock); highly weathered; soft; wet | | Bentonite Seal |
| 55 | 10 | 8.0 | 80 | | | | |
| | | | | | Brown LIMESTONE (bedrock); weathered; wet | | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| 60 | | | | | | | |
| | 10 | 2.0 | 20 | | | | Clean silica sand |
| 65 | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

Log of
MW-8D
Sheet 3 of 3

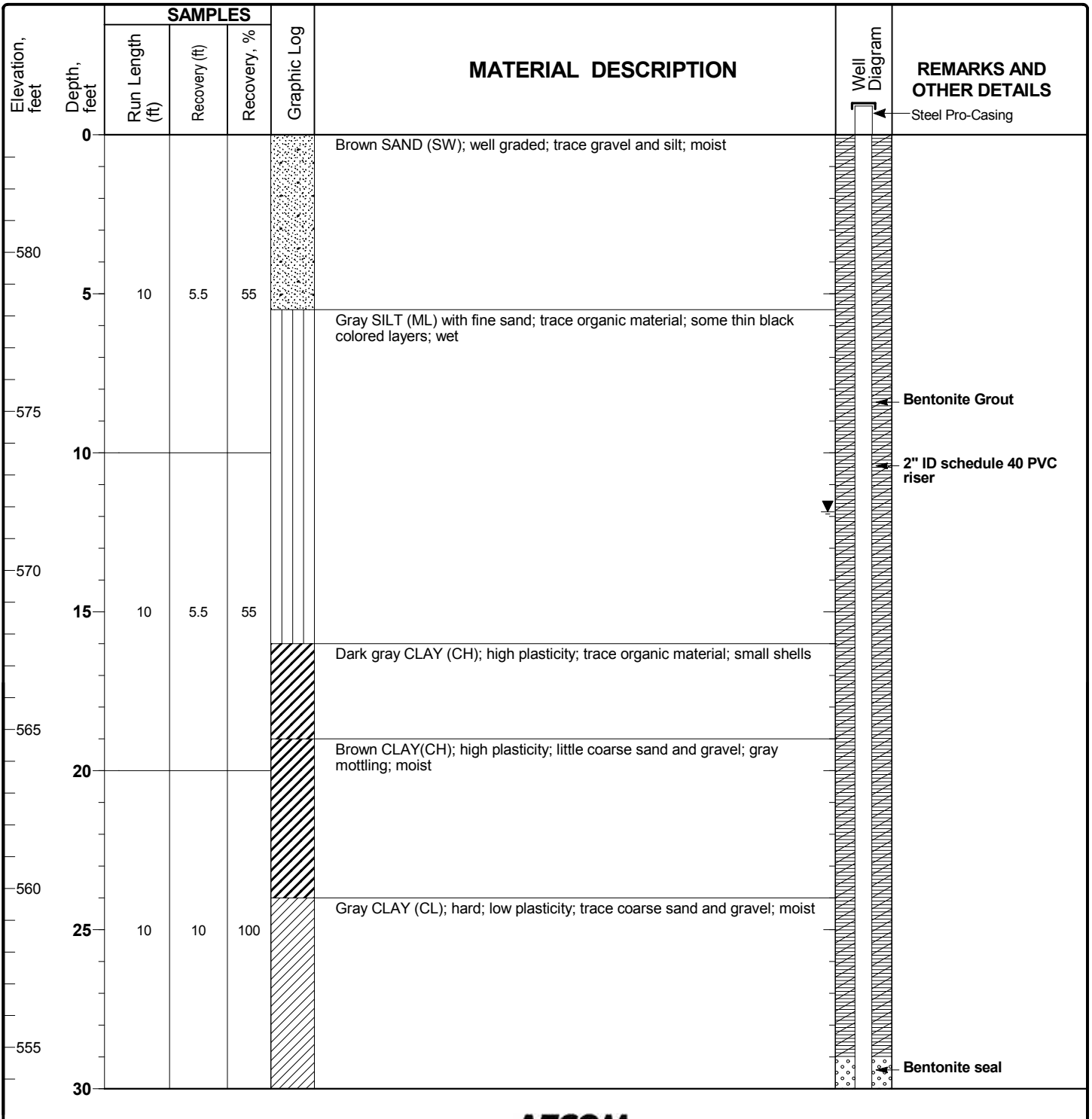
| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|----------------|--------------------|---------------|-------------|---|----------------------|---|---|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 70 | 10 | 2.0 | 20 |  | |  | 2" ID schedule 40 PVC riser SCH 40 PVC 2" Diameter 0.01" Slotted Screen Clean silica sand |
| 75 | | | | | | | End of boring |
| 80 | | | | | | | |
| 85 | | | | | | | |
| 90 | | | | | | | |
| 95 | | | | | | | |
| 100 | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-8S**
Sheet 1 of 2

| | | |
|--|--|---|
| Date(s) Drilled 9/29/16 to 9/30/2016 | Logged By Ron Friend | Checked By M Hawrylak |
| Drilling Method Sonic | Drill Bit Size/Type Sonic 6" | Total Depth of Borehole 43.0 ft |
| Drill Rig Type Mini Sonic | Drilling Contractor Cascade Drilling | Surface Elevation 583.7 ft msl |
| Borehole Backfill Monitoring Well | Sampling Method(s) Sonic Core Barrel - 4" | Top of Casing Elevation 586.59 ft msl |
| Boring Location Fly Ash Basin | Groundwater Level(s) 11.86' BTOC [Measurement after development] | |

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:26:12 PM



Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

**Log of
MW-8S**
 Sheet 2 of 2

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:26:12 PM

| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|-------------|--|--|---|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | | | | Bentonite seal |
| | | 5 | 5 | 100 | | | | 2" ID schedule 40 PVC riser |
| 550 | | | | | | | | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| 35 | | | | | | Gray fine SAND (SP-SM) with silt; thin silt seams (<1" thick); wet | | |
| | | 5 | 2.5 | 50 | | | | Clean silica sand |
| 545 | | | | | | | | |
| 40 | | | | | | | | |
| | | 3 | 2.4 | 80 | | | | |
| 540 | | | | | | | | End of boring |
| 45 | | | | | | | | |
| | | | | | | | | |
| 535 | | | | | | | | |
| 50 | | | | | | | | |
| | | | | | | | | |
| 530 | | | | | | | | |
| 55 | | | | | | | | |
| | | | | | | | | |
| 525 | | | | | | | | |
| 60 | | | | | | | | |
| | | | | | | | | |
| 520 | | | | | | | | |
| 65 | | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524






**Log of
MW-9**
Sheet 1 of 2

| | | |
|---|---|---|
| Date(s) Drilled 9/19/17 to 9/19/2017 | Logged By Ron Friend | Checked By B Finnigan |
| Drilling Method Sonic | Drill Bit Size/Type Sonic 6" | Total Depth of Borehole 40.0 ft |
| Drill Rig Type Sonic | Drilling Contractor Cascade Drilling | Surface Elevation ft msl |
| Borehole Backfill Monitoring Well | Sampling Method(s) Sonic Core Barrel - 4" | Top of Casing Elevation ft msl |
| Boring Location Inactive Bottom Ash Basin | Groundwater Level(s) | |

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | Well Diagram | REMARKS AND OTHER DETAILS |
|-------------|-----------------|---------------|-------------|-------------|---|--------------|---------------------------|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 0 | | | | | Gravelly FILL, dry | | |
| | 2 | 1.5 | 50% | | | | |
| 5 | | | | | Very dark gray high plasticity CLAY with some organics and shell fragments, soft, moist | | |
| | 5 | 5 | 100% | | grades with fine sand | | |
| 10 | | | | | grades with some organic material | | |
| 15 | 10 | 8 | 80% | | grades with more organics | | |
| | | | | | Dark brown clayey PEAT, soft | | |
| 20 | | | | | Gray high plasticity CLAY, soft, moist | | |
| | | | | | Gray sandy SILT, soft, wet | | |
| | | | | | grades with less sand with depth | | |
| 25 | 10 | 9 | 90% | | Gray fine SAND with silt, loose, wet | | |
| | | | | | grades with less silt with depth | | |
| | | | | | Gray poorly graded SAND with trace fines, loose, wet | | |
| 30 | | | | | Gray poorly graded GRAVEL with sand, loose, wet | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-9**
 Sheet 2 of 2

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|----------------|--------------------|---------------|-------------|---|---|---|------------------------------|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | |  | Gray fine grained SAND with trace fines, wet |  | Clean silica sand |
| 35 | 10 | 10 | 100% |  | Gray low plasticity CLAY with little gravel, hard, moist [TILL] |  | Natural Collapse |
| 40 | | | |  | Terminate boring at 40 feet BGS. | | End of boring |
| 45 | | | | | | | |
| 50 | | | | | | | |
| 55 | | | | | | | |
| 60 | | | | | | | |
| 65 | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524



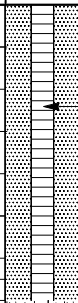





**Log of
MW-10**
Sheet 1 of 2

| | | | | | |
|-------------------|---------------------------|----------------------|------------------------|-------------------------|------------|
| Date(s) Drilled | 9/20/17 to 9/20/2017 | Logged By | Ron Friend | Checked By | B Finnigan |
| Drilling Method | Sonic | Drill Bit Size/Type | Sonic 6" | Total Depth of Borehole | 40.0 ft |
| Drill Rig Type | Sonic | Drilling Contractor | Cascade Drilling | Surface Elevation | ft msl |
| Borehole Backfill | Monitoring Well | Sampling Method(s) | Sonic Core Barrel - 4" | Top of Casing Elevation | ft msl |
| Boring Location | Inactive Bottom Ash Basin | Groundwater Level(s) | | | |

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | Well Diagram | REMARKS AND OTHER DETAILS |
|-------------|-----------------|---------------|-------------|-------------|---|--------------|-----------------------------|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 0 | | | | | Grey poorly graded GRAVEL with clay and sand, dry | | |
| 5 | 5 | 3 | 60% | | Dark yellowish brown low plasticity CLAY with sand, moist grades with gravel grades with less gravel with depth | | |
| 10 | 5 | 3 | 60% | | Very dark grey high plasticity CLAY with trace shell fragments, moist grades with fine sand | | Bentonite Grout |
| 15 | 10 | 9.5 | 95% | | grades with small sand seams, fine sand, 1-2 inches apart grades with little organic material | | 2" ID schedule 40 PVC riser |
| 20 | | | | | | | |
| 25 | 10 | 9.5 | 95% | | Grey silty CLAY with fine sand, very soft, wet Grey high plasticity CLAY, moist | | Bentonite Seal |
| 30 | | | | | | | |

Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

Log of
 MW-10
 Sheet 2 of 2

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|----------------|--------------------|---------------|-------------|---|--|--|---|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | |  | | | |
| | | | |  | Dark grey poorly graded SAND with gravel, wet | |  SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| | | | |  | Dark grey well graded SAND, with little silt, fine grained sand, wet | | |
| 35 | 10 | 9.5 | 95% |  | grades with increasing silt with depth | | |
| | | | |  | Grey SILT with little gravel, hard, moist | | |
| | | | |  | Grey CLAY with little gravel, hard, moist | |  Natural Collapse |
| 40 | | | | | Terminate boring at 40 feet BGS. | | |
| 45 | | | | | | | End of boring |
| 50 | | | | | | | |
| 55 | | | | | | | |
| 60 | | | | | | | |
| 65 | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-11**
Sheet 1 of 2

| | | | | | |
|-------------------|---------------------------|----------------------|------------------------|-------------------------|------------|
| Date(s) Drilled | 9/20/17 to 9/20/2017 | Logged By | Ron Friend | Checked By | B Finnigan |
| Drilling Method | Sonic | Drill Bit Size/Type | Sonic 6" | Total Depth of Borehole | 45.0 ft |
| Drill Rig Type | Sonic | Drilling Contractor | Cascade Drilling | Surface Elevation | ft msl |
| Borehole Backfill | Monitoring Well | Sampling Method(s) | Sonic Core Barrel - 4" | Top of Casing Elevation | ft msl |
| Boring Location | Inactive Bottom Ash Basin | Groundwater Level(s) | | | |

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | Well Diagram | REMARKS AND OTHER DETAILS |
|-------------|-----------------|---------------|-------------|-------------|---|--------------|-----------------------------|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 0 | | | | | Pale brown poorly graded SAND (fill), loose, dry | | |
| | 5 | 2 | 40% | | | | |
| 5 | | | | | grades with gravel | | |
| | 5 | 4 | 80% | | | | |
| 10 | | | | | Dark brown high plasticity CLAY with grey mottling, very stiff, moist | | Bentonite Grout |
| | 5 | 5 | 100% | | Very dark grey high plasticity CLAY with little organics and trace shell fragments, soft, moist | | 2" ID schedule 40 PVC riser |
| 15 | | | | | Grey low plasticity CLAY with brown mottling, little coarse sand and gravel, hard, moist | | |
| | 5 | 5 | 100% | | grades to brown | | |
| 20 | | | | | | | |
| | 5 | 5 | 100% | | grades to grey | | |
| 25 | | | | | Grey CLAY with little gravel and coarse sand, hard, moist-dry | | Bentonite Seal |
| | 5 | 5 | 100% | | | | |
| 30 | | | | | Grey SILT with fine sand, stiff, moist - wet, slow dilatancy | | |

Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

Log of
 MW-11
 Sheet 2 of 2

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|----------------|--------------------|---------------|-------------|-------------|---|--|--|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | | | |
| 35 | 10 | 10 | 100% | | | | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| 40 | | | | | | | |
| 45 | 5 | 5 | 100% | | Grey CLAY (till) with little gravel and coarse sand, hard, friable, moist - dry | | Natural Collapse |
| 45 | | | | | Terminate boring at 45 feet BGS. | | End of boring |
| 50 | | | | | | | |
| 55 | | | | | | | |
| 60 | | | | | | | |
| 65 | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-12**
Sheet 1 of 2

| | | |
|---|---|---|
| Date(s) Drilled 9/21/17 to 9/21/2017 | Logged By Ron Friend | Checked By B Finnigan |
| Drilling Method Sonic | Drill Bit Size/Type Sonic 6" | Total Depth of Borehole 50.0 ft |
| Drill Rig Type Sonic | Drilling Contractor Cascade Drilling | Surface Elevation ft msl |
| Borehole Backfill Monitoring Well | Sampling Method(s) Sonic Core Barrel - 4" | Top of Casing Elevation ft msl |
| Boring Location Inactive Bottom Ash Basin | Groundwater Level(s) | |

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | Well Diagram | REMARKS AND OTHER DETAILS |
|-------------|-----------------|---------------|-------------|-------------|---|--------------|-----------------------------|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 0 | | | | | Pale brown well graded SAND with fined grained sand and trace gravel, dry | | |
| | 5 | 2 | 40% | | moist | | |
| 5 | | | | | grades with trace shell fragments | | |
| | 5 | 3 | 60% | | Dark grey SAND with silt, moist | | Bentonite Grout |
| 10 | | | | | wet | | |
| | | | | | Very dark grey high plasticity CLAY with little organics and trace shell fragments, soft, moist | | 2" ID schedule 40 PVC riser |
| 15 | 10 | 8 | 80% | | Gray high plasticity CLAY with trace coarse sand and gravel, stiff, moist | | |
| | | | | | grades with color change to brown, hard, some grey mottling | | |
| 20 | | | | | | | |
| | 10 | 10 | 100% | | Very dark grey low plasticity CLAY | | Bentonite Seal |
| 25 | | | | | | | |
| 30 | | | | | | | |

Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

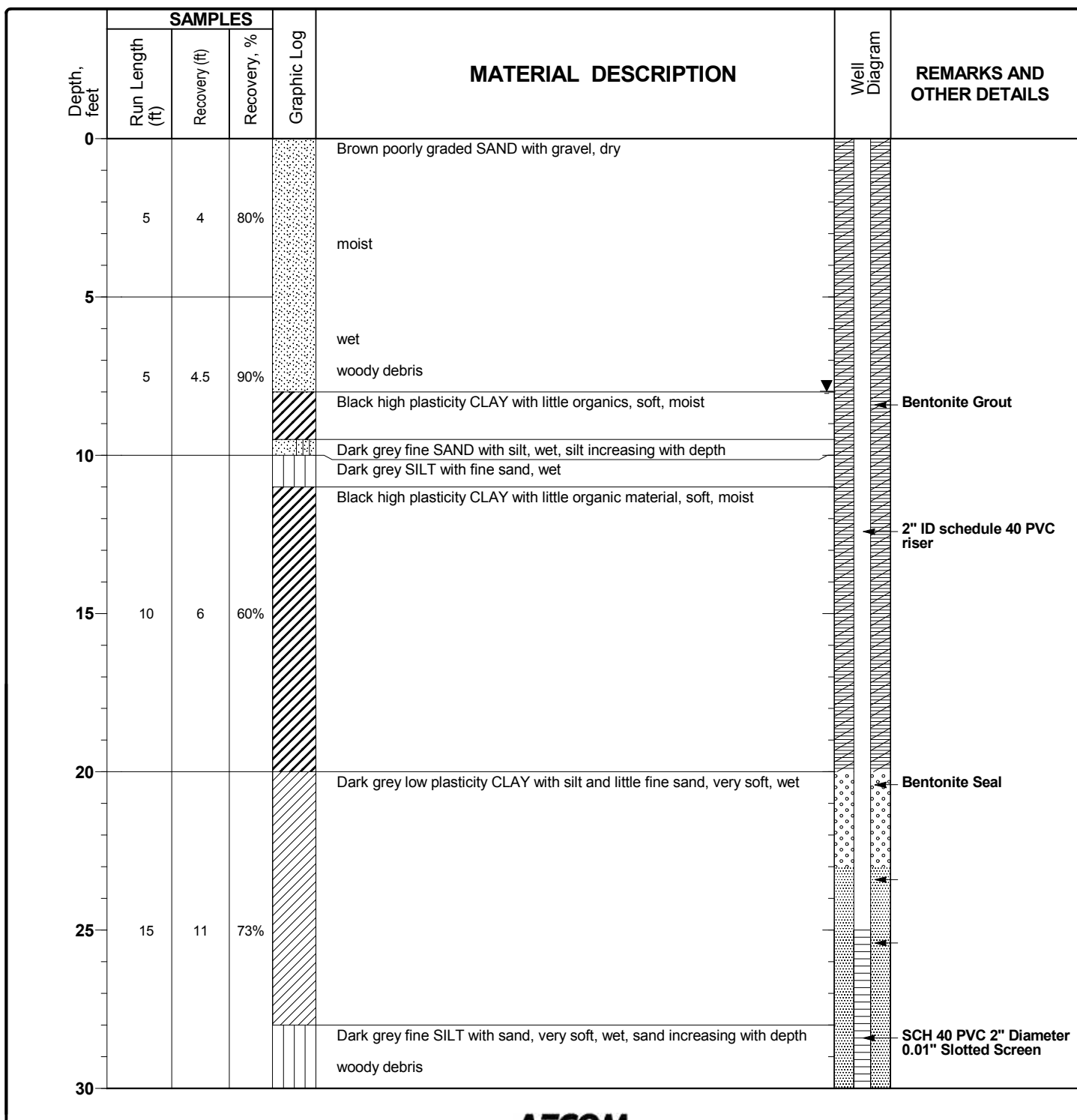
Log of
 MW-12
 Sheet 2 of 2

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | REMARKS AND OTHER DETAILS |
|----------------|--------------------|---------------|-------------|-------------|---|--|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | |
| 30 | | | | | Dark grey fine SILT with sand, stiff, wet-moist | |
| 35 | 10 | 9 | 90% | | Dark grey fine SAND with silt, stiff, wet-moist | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| 40 | | | | | | |
| 45 | 10 | 10 | 100% | | Dark grey low plasticity CLAY (till) with little gravel and coarse sand, hard, moist | Natural Collapse |
| 50 | | | | | | End of boring |
| 55 | | | | | | |
| 60 | | | | | | |
| 65 | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-13**
Sheet 1 of 2

| | | |
|---|---|---|
| Date(s) Drilled 9/21/17 to 9/21/2017 | Logged By Ron Friend | Checked By B Finnigan |
| Drilling Method Sonic | Drill Bit Size/Type Sonic 6" | Total Depth of Borehole 43.5 ft |
| Drill Rig Type Sonic | Drilling Contractor Cascade Drilling | Surface Elevation ft msl |
| Borehole Backfill Monitoring Well | Sampling Method(s) Sonic Core Barrel - 4" | Top of Casing Elevation ft msl |
| Boring Location Inactive Bottom Ash Basin | Groundwater Level(s) | |



Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

Log of
 MW-13
 Sheet 2 of 2

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|----------------|--------------------|---------------|-------------|-------------|---|--|------------------------------|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | Dark grey fine SAND with silt, wet | | |
| | 15 | 11 | 73% | | | | |
| 35 | | | | | Dark grey low plasticity CLAY with little gravel and coarse sand, hard, moist | | |
| | 8.5 | 8.5 | 100% | | | | Natural Collapse |
| 40 | | | | | | | |
| 45 | | | | | Terminate boring at 43.5 feet BGS. | | End of boring |
| 50 | | | | | | | |
| 55 | | | | | | | |
| 60 | | | | | | | |
| 65 | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-14**
Sheet 1 of 2

| | | |
|---|---|---|
| Date(s) Drilled 9/22/17 to 9/22/2017 | Logged By Ron Friend | Checked By B Finnigan |
| Drilling Method Sonic | Drill Bit Size/Type Sonic 6" | Total Depth of Borehole 40.0 ft |
| Drill Rig Type Sonic | Drilling Contractor Cascade Drilling | Surface Elevation ft msl |
| Borehole Backfill Monitoring Well | Sampling Method(s) Sonic Core Barrel - 4" | Top of Casing Elevation ft msl |
| Boring Location Inactive Bottom Ash Basin | Groundwater Level(s) | |

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | Well Diagram | REMARKS AND OTHER DETAILS |
|-------------|-----------------|---------------|-------------|-------------|---|--------------|-----------------------------|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 0 | | | | | Black coal ash FILL with gravel and coal, loose, dry | | |
| 5 | 5 | 4 | 80% | | Black coal ash FILL, coarse, sand-gravel sized, moist | | |
| 10 | 5 | 4.5 | 90% | | Very dark grey high plasticity CLAY, soft, moist | | Bentonite Grout |
| 15 | 10 | 6 | 60% | | grades to coarse sand and gravel, wet | | 2" ID schedule 40 PVC riser |
| 20 | | | | | Very dark grey fine SAND with silt and clay, wet | | |
| 25 | 10 | 0 | 0% | | No recovery | | Bentonite Seal |
| 30 | | | | | | | |

Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

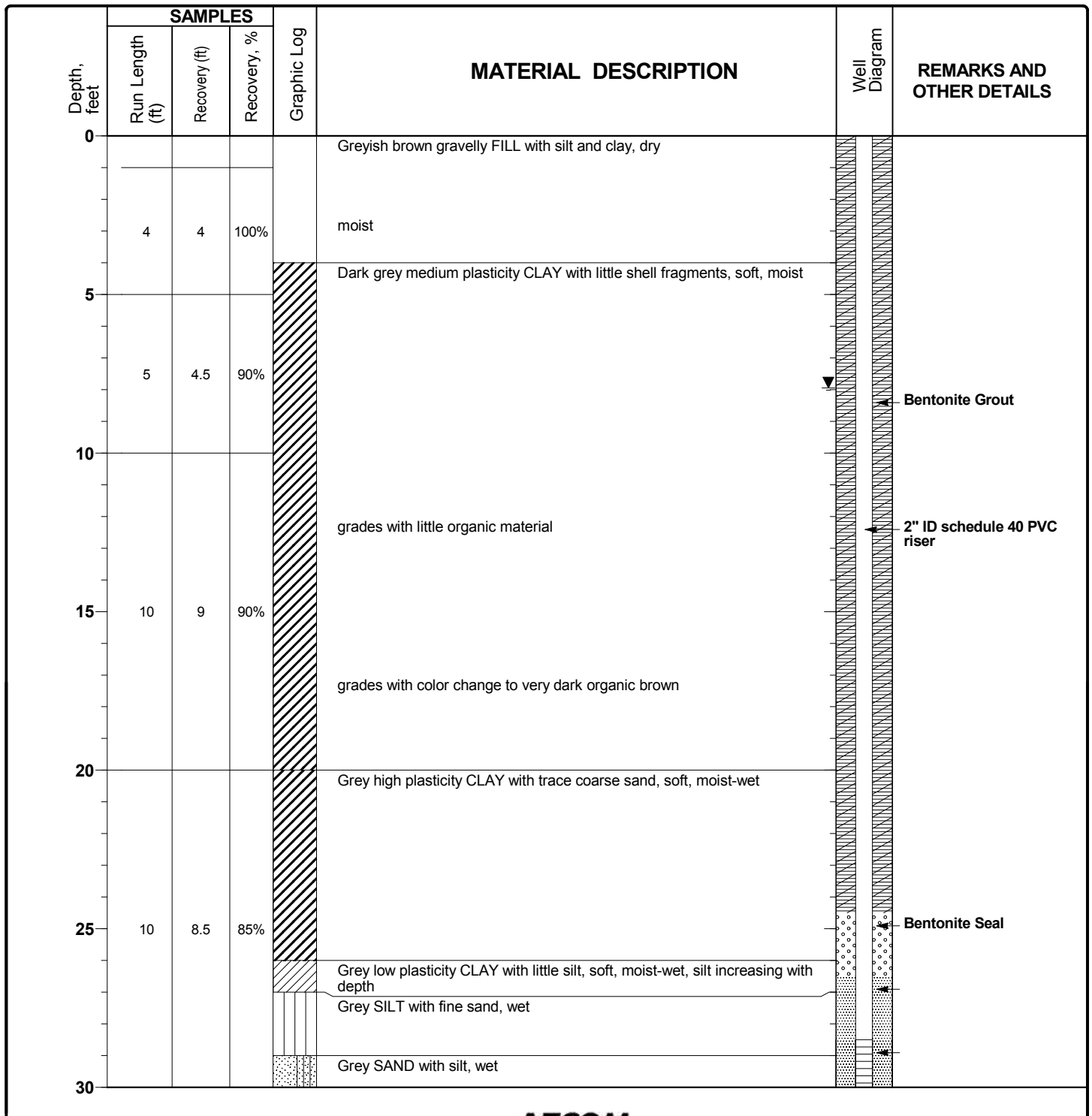
**Log of
MW-14**
 Sheet 2 of 2

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|----------------|--------------------|---------------|-------------|-------------|---|--|--|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | Dark grey CLAY with sand, fine-medium grained sand. wet | | |
| | | | | | Dark grey poorly graded SAND with gravel, coarse grained sand, wet | | |
| 35 | 10 | 9 | 90% | | | | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| | | | | | Dark grey CLAY (till) with little coarse sand and gravel, hard, moist | | |
| 40 | | | | | Terminate boring at 40 feet BGS. | | End of boring |
| 45 | | | | | | | |
| 50 | | | | | | | |
| 55 | | | | | | | |
| 60 | | | | | | | |
| 65 | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-15**
Sheet 1 of 2

| | | | | | |
|-------------------|---------------------------|----------------------|------------------------|-------------------------|------------|
| Date(s) Drilled | 9/26/17 to 9/26/2017 | Logged By | Ron Friend | Checked By | B Finnigan |
| Drilling Method | Sonic | Drill Bit Size/Type | Sonic 6" | Total Depth of Borehole | 45.0 ft |
| Drill Rig Type | Sonic | Drilling Contractor | Cascade Drilling | Surface Elevation | ft msl |
| Borehole Backfill | Monitoring Well | Sampling Method(s) | Sonic Core Barrel - 4" | Top of Casing Elevation | ft msl |
| Boring Location | Inactive Bottom Ash Basin | Groundwater Level(s) | | | |



Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

Log of
 MW-15
 Sheet 2 of 2

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|----------------|--------------------|---------------|-------------|-------------|---|--|--|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | Grey well graded SAND, fine to coarse grained, wet | | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| 35 | 10 | 10 | 100% | | grades with gravel | | |
| | | | | | Grey well graded GRAVEL with coarse sand, wet | | |
| | | | | | Grey well graded SAND with gravel, wet | | |
| 40 | | | | | Grey low plasticity CLAY (till) with little coarse sand and gravel, hard, moist | | Natural Collapse |
| | | | | | | | End of boring |
| 45 | | | | | Terminate boring at 45 feet BGS. | | |
| 50 | | | | | | | |
| 55 | | | | | | | |
| 60 | | | | | | | |
| 65 | | | | | | | |

APPENDIX B

MW-8S Hydraulic Isolation Alternative Source Demonstration

Technical Memorandum

Date: October 14, 2019

To: Christopher P. Scieszka
DTE Electric Company

From: Graham Crockford, TRC
David McKenzie, TRC

Project No.: 320511.0006.0000 Phase 001, Task 001

Subject: Alternate Source Demonstration: 2019 Initial Detection Monitoring Sampling Event
Monroe Power Plant Bottom Ash Impoundment Inactive Coal Combustion Residual Unit

Introduction

On April 17, 2015, the United States Environmental Protection Agency (USEPA) published the final rule for the regulation and management of Coal Combustion Residuals (CCR) under the Resource Conservation and Recovery Act (RCRA) (the CCR Rule), as amended July 30, 2018. The CCR Rule, which became effective on October 19, 2015 (amendment effective August 29, 2018), applies to the DTE Electric Company (DTE Electric) Monroe Power Plant (MONPP) Bottom Ash Impoundment (BAI) Inactive CCR unit. On August 5, 2016, the USEPA published the CCR Rule companion *Extension of Compliance Deadlines for Certain Inactive Surface Impoundments*, which established the compliance deadlines for CCR units that were inactive prior to October 15, 2015.

TRC prepared the 2019 *Annual Groundwater Monitoring Report* (Annual Report) for the MONPP BAI Inactive CCR unit on behalf of DTE Electric in accordance with the requirements of §257.90(e) (TRC, July 2019). The Annual Report included the results of the May 2019 semiannual groundwater monitoring event for the MONPP BAI Inactive CCR unit and the statistical evaluation of the detection monitoring parameters (Appendix III to Part 257 of the CCR Rule) for the MONPP BAI Inactive CCR unit. This event was the initial detection monitoring event performed to comply with §257.94. The monitoring was performed in accordance with the *Groundwater Monitoring Work Plan Coal Combustion Residuals (CCR) Rule – Inactive Bottom Ash Basin DTE Monroe Plant* (Work Plan) (AECOM, September 2017). As part of the statistical evaluation, the data collected during detection monitoring events are evaluated to identify statistically significant increases (SSIs) in detection monitoring parameters to determine if concentrations in detection monitoring well samples exceed background levels. The statistical analysis was performed pursuant to §257.93(f) and (g), and in accordance with the

Technical Memorandum

Groundwater Statistical Evaluation Plan Coal Combustion Residuals (CCR) Rule – Inactive Bottom Ash Impoundment DTE Monroe Plant (Stats Plan) (AECOM, April 2019, Revised August 2019).

The statistical evaluation of the May 2019 Appendix III indicator parameters showed potential SSIs over background for:

- Boron at MW-8S;
- Sulfate at MW-9, MW-10, MW-11; and
- TDS at MW-9 and MW-10.

All other Appendix III constituents were within the statistical background limits. As discussed in the August 2019 Annual Groundwater Monitoring Report (TRC, August 2019), verification resampling was conducted on July 8 and 9, 2019, by TRC personnel for boron at MW-8S, sulfate and TDS at MW-9 and MW-10, and sulfate at MW-11. The verification resampling confirmed only the boron SSI at MW-8S.

In accordance with §257.94(3)(2), DTE Electric may demonstrate that a source other than the CCR unit caused the SSI or that the SSI resulted from error in sampling, analysis, statistical evaluation, or natural variation in groundwater quality. This Alternate Source Demonstration (ASD) has been prepared to evaluate the initial boron SSI identified in the May 2019 detection monitoring event. The results of this ASD show that the SSI at MW-8S is not due to a release from the MONPP BAI Inactive CCR unit.

Background

The MONPP is located in Section 15, Township 7 South, Range 9 East, at 3500 East Front Street, Monroe in Monroe County, Michigan. The site location is shown in Figure 1. The MONPP BAI Inactive CCR unit is located within the southern portion of the MONPP parcel and is bounded by the MONPP facility to the north and northeast, Lake Erie to the southeast and south, and Plum Creek/the discharge canal to the west. The MONPP BAI Inactive CCR unit was operated from the early-1970s through part of 2015.

As presented in the Stats Plan, the bedrock in the site vicinity is overlain by approximately 40 to 50 feet of unconsolidated deposits of glacial origin. The deposits are comprised of two (2) distinct units: a hard glacial till immediately overlying bedrock and lacustrine (lake bed or lake shore) deposits which overlay the till unit. The till is comprised of over consolidated (highly compacted) gray silty to sandy clay with some cobbles and boulders, and ranges from approximately 20 to 50 feet in thickness. The overlying lacustrine deposits are composed of 10 to 30 feet of fine-grained sand and silt with some soft clay except where there is a thin, discontinuous coarse sand unit at the base of the lacustrine sequence.

The detection monitoring well network for the MONPP BAI Inactive CCR unit currently consists of twelve monitoring wells that are screened in the uppermost aquifer. As discussed in the Stats Plan, intrawell statistical methods for the MONPP BAI Inactive CCR unit were selected based on the

Technical Memorandum

geology and hydrogeology at the Site (the variability in the presence of the sand unit aquifer across the site and the strong confined hydraulic pressure in the sand unit aquifer), in addition to other supporting lines of evidence that the aquifer is unaffected by the CCR unit (such as the consistency in concentrations of water quality data). Monitoring wells MW-1S through MW-3S and MW-7S through MW-15 are located around the perimeter of the MONPP BAI and provide data on both background and downgradient groundwater quality that has not been affected by the CCR unit (total of twelve background/downgradient monitoring wells). The monitoring well locations are shown in Figure 2. The *Monitoring Well Installation Report Coal Combustion Residuals (CCR) Rule – Inactive Bottom Ash Impoundment DTE Monroe* (Well Installation Report) (AECOM, April 2019, Revised August 2019) details the groundwater monitoring system.

Alternate Source Demonstration

Verification resampling for boron at MW-8S, sulfate and TDS at MW-9 and MW-10, and sulfate at MW-11 was performed as recommended per the Stats Plan and the *USEPA's Statistical Analysis of Groundwater Monitoring Data at RCRA Facilities, Unified Guidance* (Unified Guidance, USEPA, 2009) to achieve performance standards as specified by §257.93(g) in the CCR rules. The verification resampling confirmed the boron exceedance at MW-8S during the July 2019 verification sampling event (Table 1). The following discussion presents the ASD for the confirmed prediction limit exceedance.

Boron at MW-8S: Based on historical site modifications that changed the underlying lithology beneath the discharge channel, groundwater in the area of monitoring well MW-8S is not hydraulically connected to groundwater in the vicinity of the MONPP BAI Inactive CCR unit. Therefore, concentrations in groundwater at MW-8S are not indicative of a release from the CCR unit.

A deep channel was historically dredged along the current location of the MONPP discharge channel to provide access to the MONPP parcel during the late 1960s/early 1970s based on the historic topographic maps (from 1952 to 1973) and aerial photographs (from 1961 and 1973) provided in Attachment A. As shown on Figure 2, the deep channel extended from the area near East Front Street (adjacent to the main plant building) toward Lake Erie to the south (between MW-8S and the MONPP BAI). Based on current available bathymetry data that was collected on July 24, 2019 using a Lowrance HDS9 sonic sonar unit, the channel was dredged to a depth of approximately 28 feet (to an elevation of approximately 546 feet above sea level per NAVD88) such that the bottom of the deep channel intersects the uppermost aquifer (Attachment B). The portion of the discharge channel south of the main channel of Plum Creek (between MW-8S and the MONPP BAI) has been partially filled with sediment since the MONPP was completed in the 1970s, as the channel was no longer maintained for navigation.

As illustrated on Figures 3 and 4, the upper portion of the uppermost aquifer at MW-7S and MW-9 is at a higher elevation than the bottom of the now partially sediment filled discharge channel. This demonstrates that the sediment fill within the discharge channel intersects the uppermost aquifer, creating a hydraulic connection between the uppermost aquifer and the discharge channel.

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Groundwater and Lake Erie surface water elevation data also support the hydraulic connection between the discharge channel/Lake Erie and the uppermost aquifer. A graphical depiction of the MONPP BAI Inactive CCR unit groundwater elevations at select monitoring wells and surface water elevations in Lake Erie are shown in Figure 5. These data demonstrate that groundwater in the uppermost aquifer is interacting with surface water as shown by the monitoring well groundwater surface elevations rising and lowering concurrently with the Lake Erie surface water elevations.

Groundwater naturally flows horizontally in the downgradient direction (from high potential to low potential) along the path of least resistance toward the closest discharge features, which in this case are Plum Creek, the discharge channel, and Lake Erie. At the point of discharge, vertical groundwater flow gradients are expected as groundwater discharges to surface water. Groundwater potentiometric surface elevation data from MW-7S, MW-9 and MW-8S are consistently higher than the Lake Erie surface elevation recorded on the same date as shown on Figure 5. This demonstrates that the groundwater from the area of MW-8S will flow east and groundwater from the area of MW-7S and MW-9 will flow west toward the discharge channel and discharge into the channel, given that the surface water elevation in the channel is lower and there is a hydraulic connection between the uppermost aquifer and the channel (Figures 3 and 4). As such, groundwater beneath the MONPP BAI cannot physically flow west of the discharge channel to the area of MW-8S.

In addition, clay is present beneath the uppermost aquifer preventing downward vertical migration of groundwater in the area of the discharge channel (Figures 3 and 4). Upward vertical flow potential is observed in groundwater beneath the uppermost aquifer as evidenced by the artesian flowing conditions at MW-7D and MW-8D that are at higher groundwater elevations compared to their shallow counterparts, MW-7S and MW-8S, further demonstrating that vertical flow potential is upward beneath the uppermost aquifer (Figure 5).

Given that groundwater from the area of the MONPP BAI cannot reach monitoring well MW-8S due to the hydraulic separation along the discharge channel, the boron SSI at MW-8S is not indicative of a release from the MONPP BAI Inactive CCR unit.

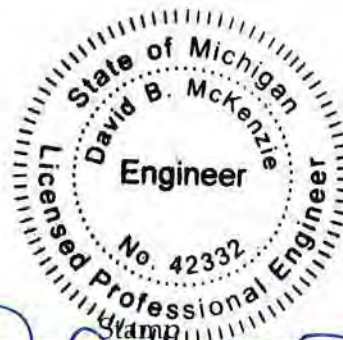
Conclusions and Recommendations

The information provided in this report serves as the ASD for the DTE Electric MONPP BAI Inactive CCR unit, was prepared in accordance with 40 CFR 257.94(e)(2) of the CCR Rule, and demonstrates that the boron SSI determined based on the initial semiannual detection monitoring event performed in 2019 is not due to a release of CCR leachate into the groundwater from the MONPP BAI Inactive CCR unit. Therefore, based on the information provided in this ASD, DTE Electric will continue detection monitoring as per 40 CFR 257.94 at the MONPP BAI Inactive CCR unit removing monitoring well MW-8S from the well network for future detection monitoring since MW-8S is not hydraulically connected to the MONPP BAI Inactive CCR unit.

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Certification Statement

I hereby certify that the alternative source demonstration presented within this document for the MONPP BAI Inactive CCR unit has been prepared to meet the requirements of Title 40 CFR §257.94(e)(2) of the Federal CCR Rule. This document is accurate and has been prepared in accordance with good engineering practices, including the consideration of applicable industry standards, and with the requirements of Title 40 CFR §257.94(e)(2).

| | | |
|--|--------------------------------------|---|
| Name: David B. McKenzie, P.E. | Expiration Date: October 31, 2019 |  |
| Company: TRC Engineers Michigan, Inc. | Date: 10/14/19 | |

Technical Memorandum

References

- TRC Environmental Corporation. July 2019. Annual Groundwater Monitoring Report – DTE Electric Company Monroe Power Plant Bottom Ash Basin Inactive Coal Combustion Residual Unit, 3500 East Front Street, Monroe, Michigan. Prepared for DTE Electric Company.
- AECOM. September 2017. Groundwater Monitoring Work Plan Coal Combustion Residuals (CCR) Rule – Inactive Bottom Ash Basin, DTE Monroe Plant, Monroe, Michigan. Prepared for DTE Electric Company.
- AECOM. April 2019, Revised August 2019. Groundwater Statistical Evaluation Plan Coal Combustion Residuals (CCR) Rule – Inactive Bottom Ash Impoundment, DTE Monroe Plant, Monroe, Michigan. Prepared for DTE Electric Company.
- AECOM. April 2019, Revised August 2019. Monitoring Well Installation Report Coal Combustion Residuals (CCR) Rule – Inactive Bottom Ash Impoundment, DTE Monroe Plant, Monroe, Michigan. Prepared for DTE Electric Company.
- USEPA. 2009. Statistical Analysis of Groundwater Monitoring Data at RCRA facilities, Unified Guidance. Office of Conservation and Recovery. EPA 530/R-09-007.

Attachments

- Table 1 Comparison of Verification Sampling Results to Background Limits
- Figure 1 Site Location Map
- Figure 2 Well Location Map
- Figure 3 Generalized Cross-Section A-A'
- Figure 4 Generalized Cross-Section B-B'
- Figure 5 MW-7S, MW-8S, MW-9, MW-7D, MW-8D, and Lake Erie Ground/Surface Water Elevation Time Series Plot
- Attachment A Historic Topographic Maps and Aerial Photographs
- Attachment B Bottom of Discharge Channel Depth Map

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Table 1

Table 1
Comparison of Verification Sampling Results to Background Limits
Monroe Power Plant Inactive Bottom Ash Impoundment – RCRA CCR Monitoring Program
Monroe, Michigan

| Sample Location: | | MW-8S | | MW-9 | | MW-10 | | MW-11 | |
|------------------------|------|------------|-------|----------|-----|----------|-----|----------|-------|
| Sample Date: | | 7/9/2019 | | 7/8/2019 | | 7/8/2019 | | 7/8/2019 | |
| Constituent | Unit | Data | PL | Data | PL | Data | PL | Data | PL |
| Appendix III | | | | | | | | | |
| Boron | ug/L | 490 | 440 | -- | 640 | -- | 530 | -- | 920 |
| Sulfate | mg/L | -- | 1,600 | 3.6 | 12 | 3.7 | 19 | 1,300 | 1,500 |
| Total Dissolved Solids | mg/L | -- | 2,400 | 800 | 810 | 830 | 840 | -- | 2,100 |

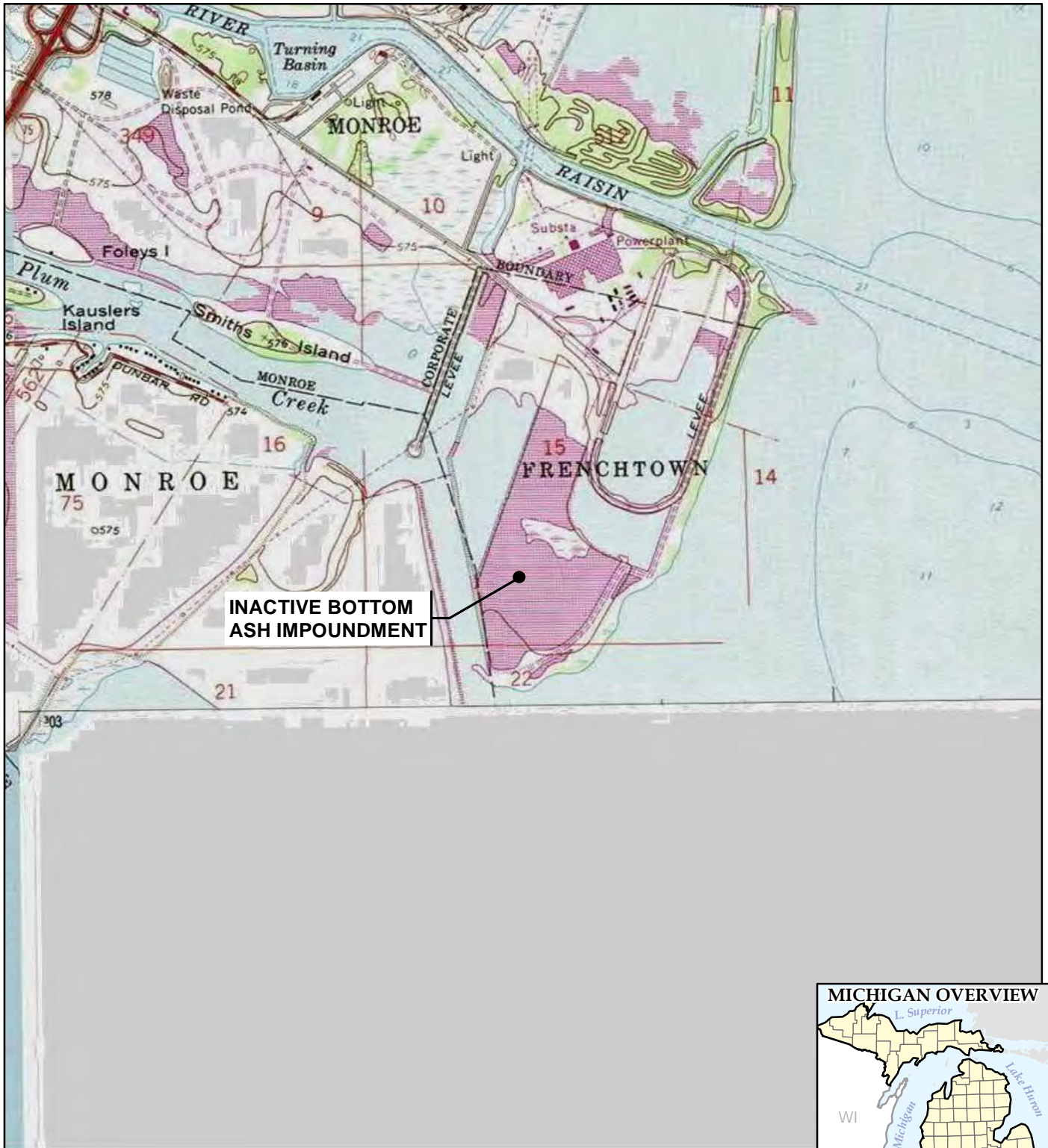
Notes:

-- = not analyzed

| | |
|---------------|--|
| RESULT | Shading and bold font indicates a confirmed exceedance of the Prediction Limit (PL). |
|---------------|--|

Technical Memorandum

Figures



BASE MAP FROM USGS 7.5 MINUTE TOPOGRAPHIC QUADRANGLE SERIES.



1540 Eisenhower Place
Ann Arbor, MI 48108-3284
Phone: 734.971.7080

TRC - GIS

PROJECT: **DTE ELECTRIC COMPANY
MONROE POWER PLANT BOTTOM ASH IMPOUNDMENT
3500 EAST FRONT STREET
MONROE, MI 48161**

TITLE: **SITE LOCATION MAP**

| | |
|--------------|-------------------|
| DRAWN BY: | R. SUEMNICHT |
| CHECKED BY: | S. HOLMSTROM |
| APPROVED BY: | V. BUENING |
| DATE: | SEPTEMBER 2019 |
| PROJ. NO.: | 320511.0006 |
| FILE: | 320511-001slm.mxd |

FIGURE 1



CCR PROGRAM MONITORING WELL

INVESTIGATION MONITORING WELL (STATIC WATER LEVELS ONLY)

UNIT SEPARATION BERM

CROSS SECTION LOCATION

APPROXIMATE BOUNDARY OF INACTIVE BOTTOM ASH IMPOUNDMENT

APPROXIMATE PLANT BOUNDARY

NOTES

1. BASE MAP IMAGERY FROM GOOGLE EARTH PRO & PARTNERS, APRIL 2018.

07001,400

1" = 700'

1:8,400

N

PROJECT:

DTE ELECTRIC COMPANY

MONROE POWER PLANT BOTTOM ASH IMPOUNDMENT

3500 EAST FRONT STREET

MONROE, MI 48161

TITLE:

INACTIVE BOTTOM ASH IMPOUNDMENT

WELL LOCATION MAP

2019

DRAWN BY:

R. SUEMNICHT

CHECKED BY:

S. HOLMSTROM

APPROVED BY:

V. BUENING

DATE:

SEPTEMBER 2019

PROJ. NO.:

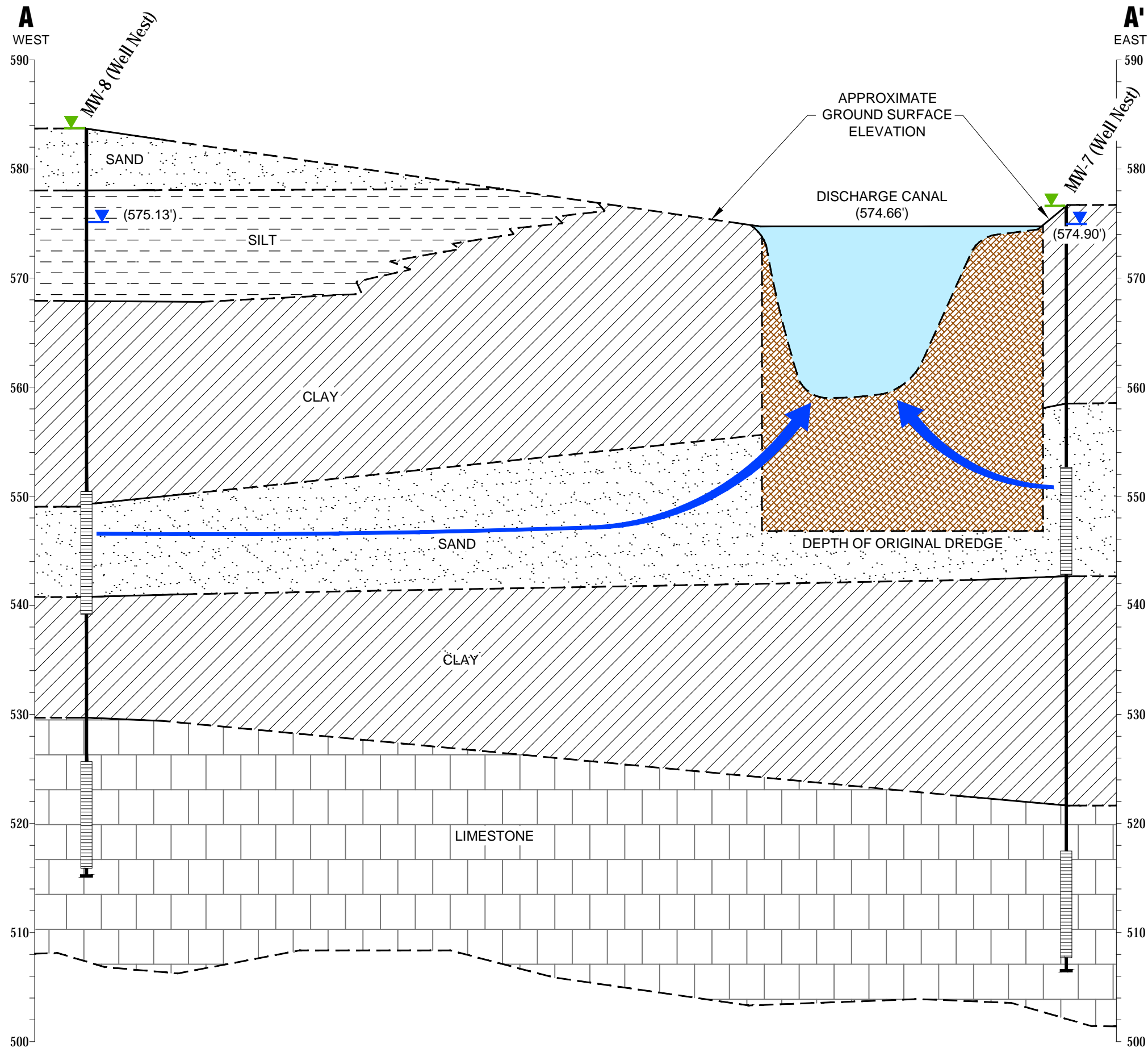
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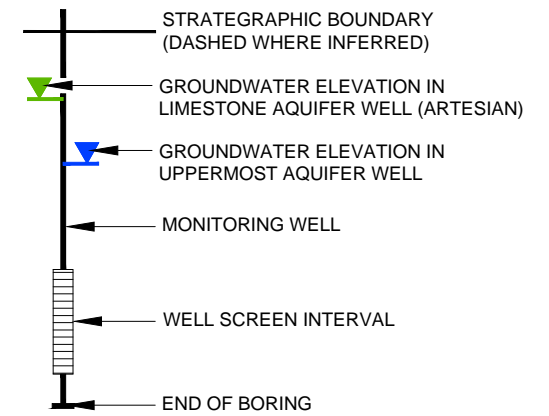
320511-002.mxd

FIGURE 2

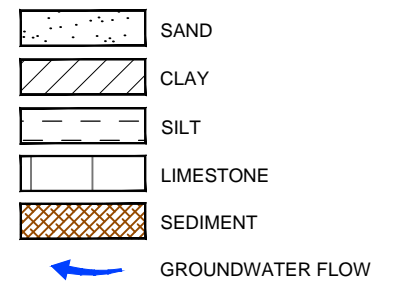
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LEGEND

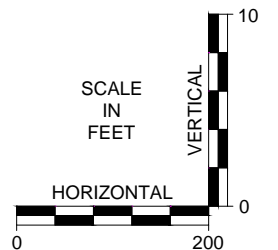


Lithology Key



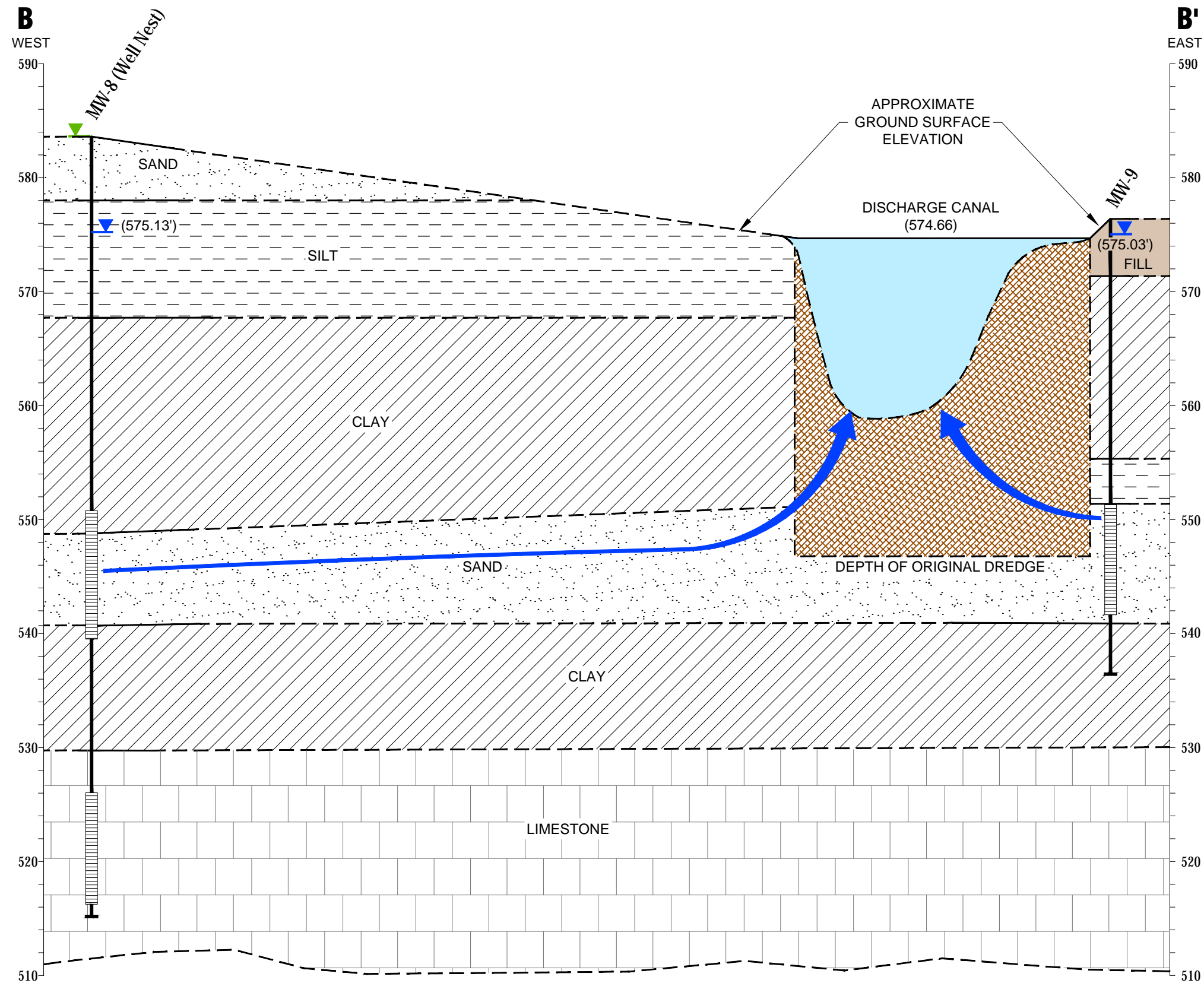
NOTE

- ALL GROUNDWATER ELEVATIONS AND SURFACE WATER ELEVATIONS ARE FROM 5/21/2019 AND ARE IN NAVD88.
- DEPTH OF DISCHARGE CANAL APPROXIMATED FROM BIOBASE MAP - LAKE ERIE 7/24/2019 (PROVIDED IN ATTACHMENT B).



| | | | |
|---|--|---|--|
| PROJECT: | | DTE ELECTRIC COMPANY MONROE POWER PLANT INACTIVE BOTTOM ASH IMPOUNDMENT MONROE, MICHIGAN | |
| TITLE: Generalized Cross-Section A-A' | | | |
| DRAWN BY: D.STEHLE | | PROJ NO.: 320511.0006.01 | |
| CHECKED BY: M.BREHOB | | FIGURE 3 | |
| APPROVED BY: - | | | |
| DATE: SEPTEMBER 2019 | | | |
| | | 1540 Eisenhower Place Ann Arbor, MI 48108 Phone: 734.971.7080 www.trccompanies.com | |
| FILE NO.: | | 320511.0006.01.03-04 ASD.dwg | |

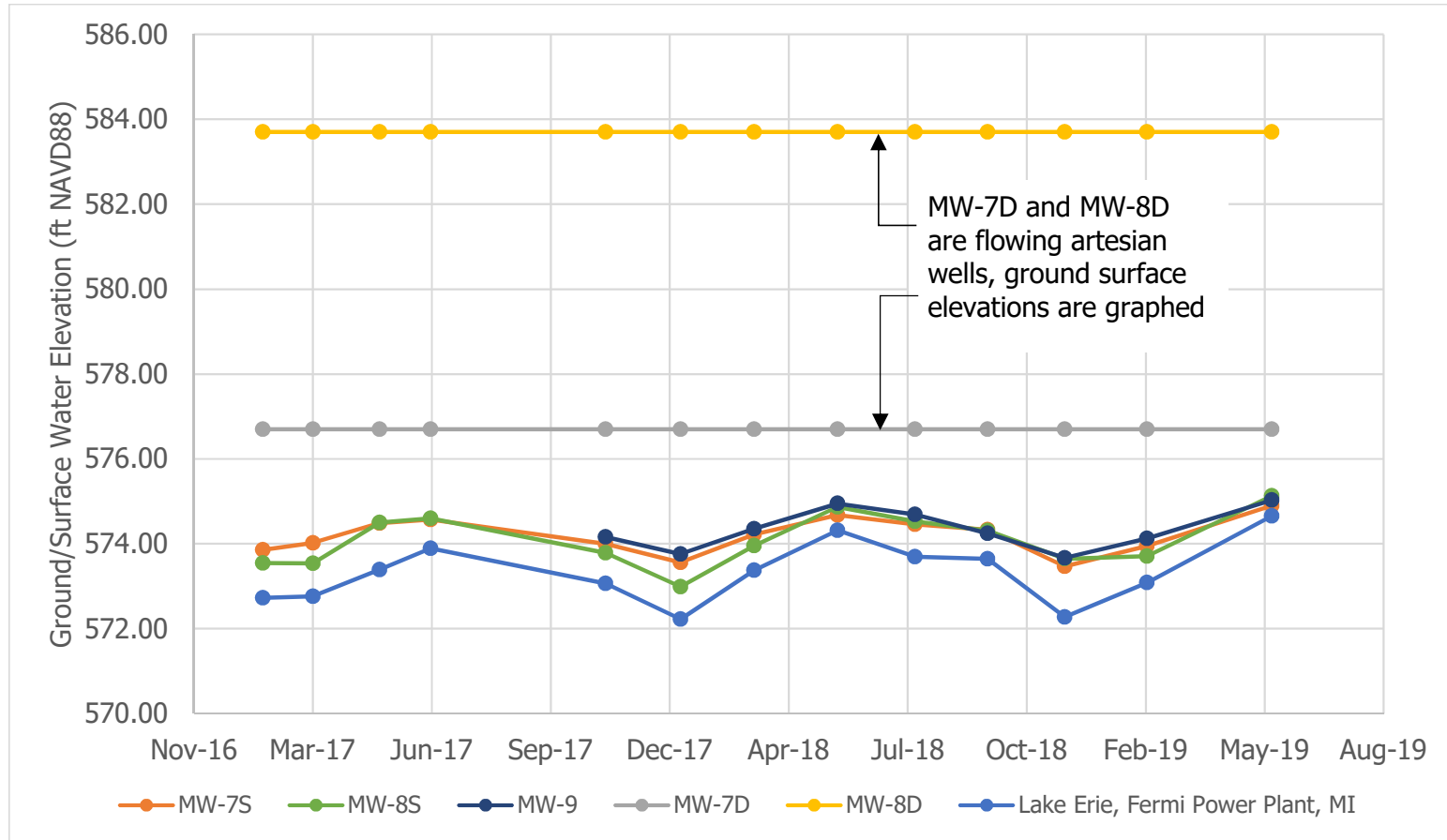
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| | | | |
|---|--|---|--|
| PROJECT: | | DTE ELECTRIC COMPANY MONROE POWER PLANT INACTIVE BOTTOM ASH IMPOUNDMENT MONROE, MICHIGAN | |
| TITLE: Generalized Cross-Section B-B' | | | |
| DRAWN BY: D.STEHLE | | PROJ NO.: 320511.0006.01 | |
| CHECKED BY: M.BREHOB | | FIGURE 4 | |
| APPROVED BY: - | | | |
| DATE: SEPTEMBER 2019 | | | |
| | | 1540 Eisenhower Place Ann Arbor, MI 48108 Phone: 734.971.7080 www.trccompanies.com | |
| FILE NO.: | | 320511.0006.01.03-04 ASD.dwg | |

Figure 5

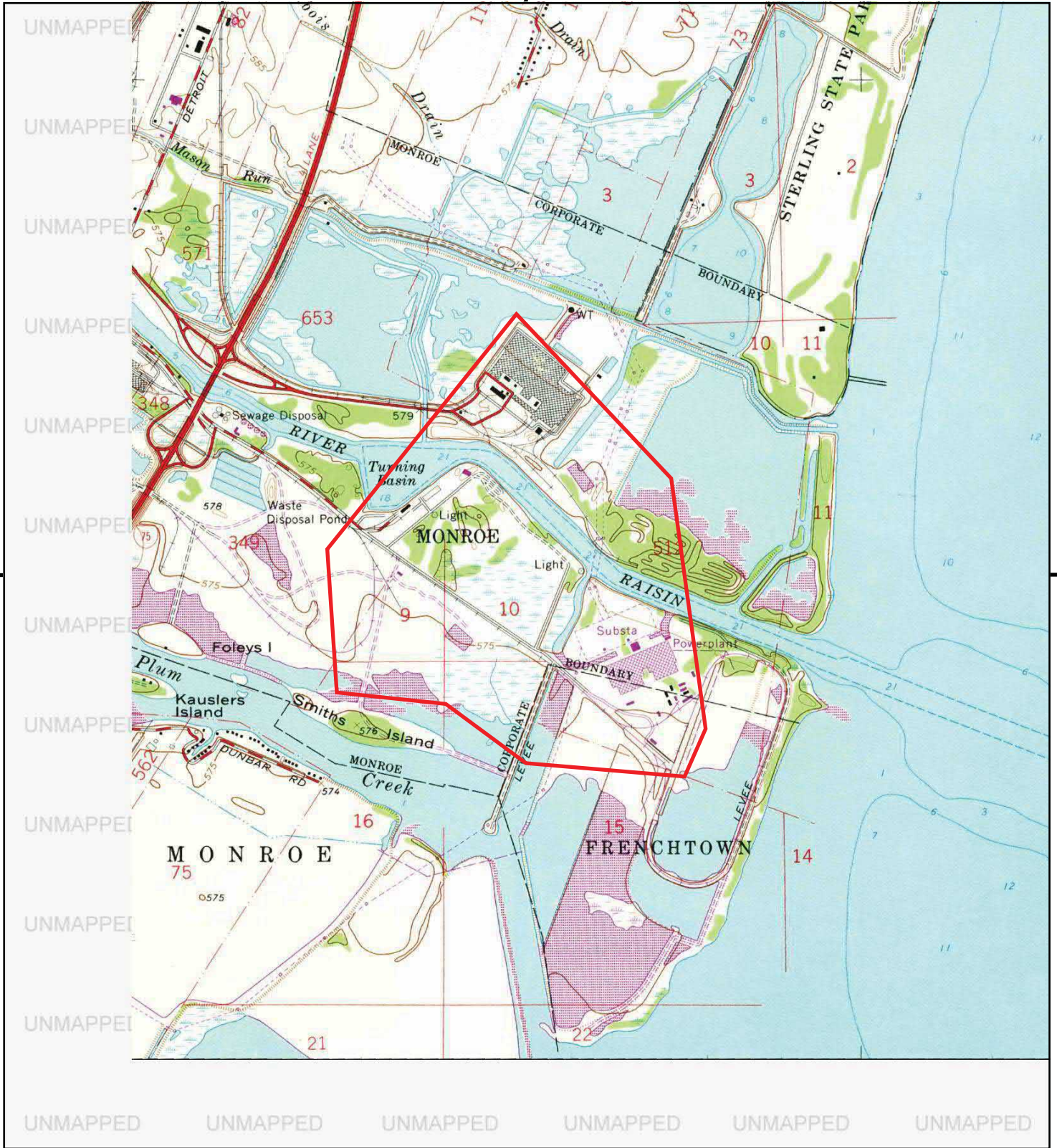
MW-7S, MW-8S, MW-9, MW-7D, MW-8D, and Lake Erie Ground/Surface Water Elevation Time Series Plot
Monroe Power Plant Inactive Bottom Ash Impoundment – RCRA CCR Monitoring Program



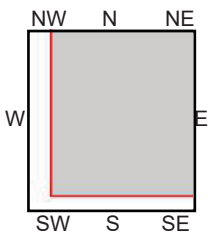
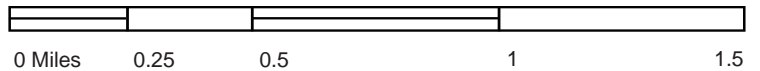
Technical Memorandum

Attachment A

Historic Topographic Maps and Aerial Photographs



This report includes information from the following map sheet(s).

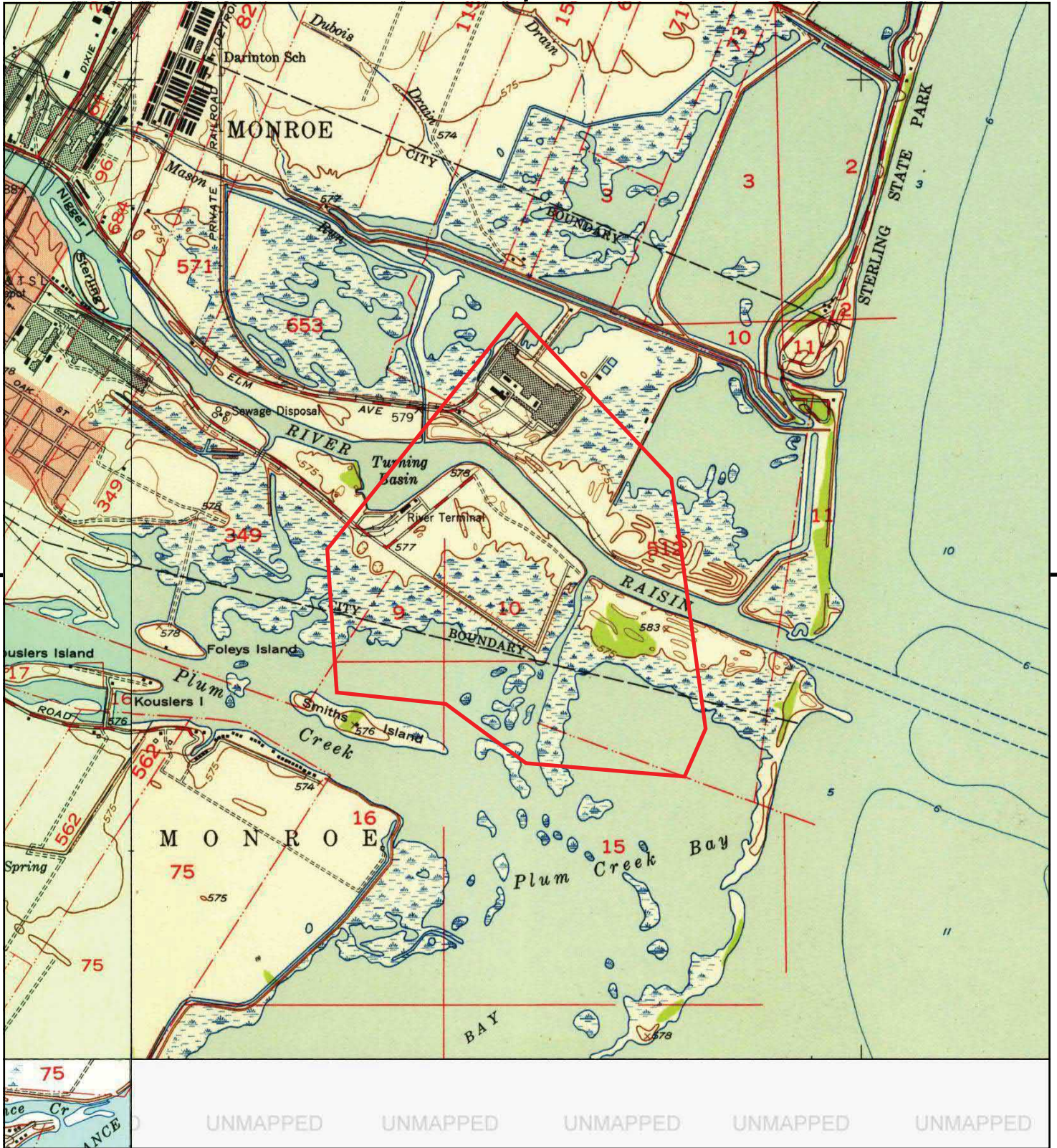


TP, Stony Point, 1973, 7.5-minute

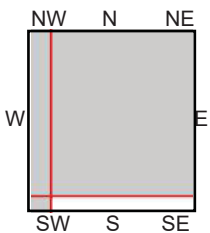
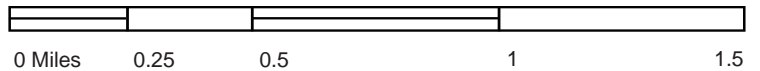
SITE NAME: Port of Monroe/Gerdau Ameristeel
ADDRESS: 3000 E. Front Street
Monroe, MI 48161
CLIENT: AECOM







This report includes information from the following map sheet(s).



TP, Stony Point, 1952, 7.5-minute
SW, Erie, 1952, 7.5-minute
NW, Monroe, 1952, 7.5-minute

SITE NAME: Port of Monroe/Gerdau Ameristeel
ADDRESS: 3000 E. Front Street
Monroe, MI 48161
CLIENT: AECOM





INQUIRY #: 4694578.9

YEAR: 1973

— = 600'



-61



INQUIRY # 4694578.9

YEAR: 1961

— = 500'



Technical Memorandum

Attachment B

Bottom of Discharge Channel Depth Map



Lowrance HDS9 Sonic Sonar unit used to collect bathymetric data from Plum Creek and Discharge Channel.
BioBase Mapping Software used to generate figure.

Mr. Chris Scieszka
DTE Electric Company
June 30, 2020

Attachment B

Groundwater Monitoring and Quality Assurance Project Plan



Groundwater Monitoring and Quality Assurance Project Plan

**DTE Electric Company Monroe Power Plant
Bottom Ash Impoundment**

**3500 East Front Street
Monroe, Michigan**

June 2020

Prepared For:

DTE Electric Company

Prepared By:

TRC

1540 Eisenhower Place
Ann Arbor, Michigan 48108

A handwritten signature in black ink that reads "Kelly C. Cratsenburg".

Kelly C. Cratsenburg, C.P.G.
Project Hydrogeologist

A handwritten signature in black ink that reads "Vincent E. Buening".

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Senior Project Manager

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APPENDICES

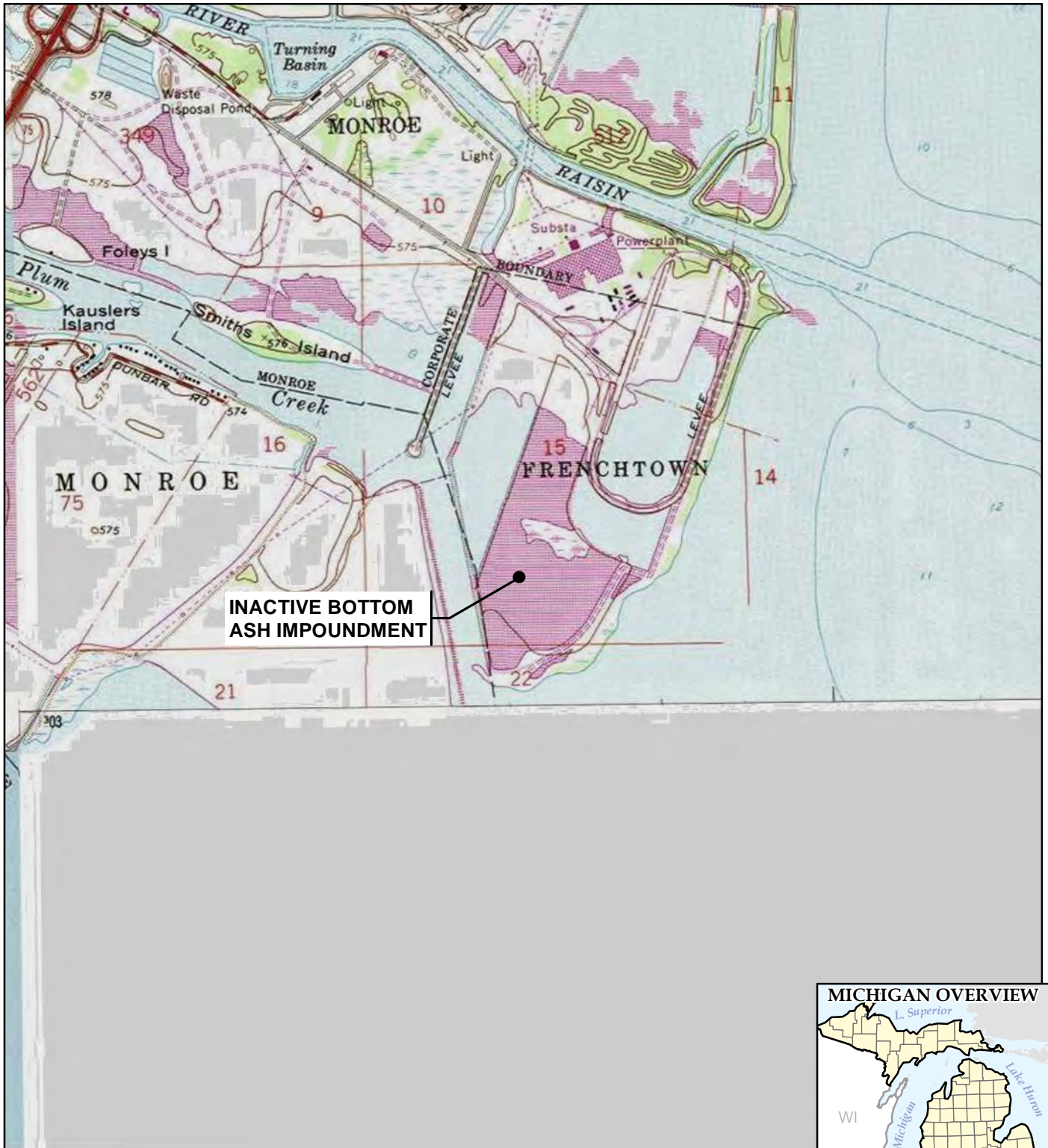
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1.0 Introduction

DTE Electric Company (DTE Electric) is a utility provider that owns and operates Monroe Power Plant (MONPP), a coal combustion power plant located at 3500 East Front Street, in Monroe, Michigan. A site location map is included as Figure 1. As part of power plant operations, there is a bottom ash impoundment (BAI) on the southern portion of the power plant property that had historically been used to manage coal combustion residuals (CCR) from the MONPP from the mid-1970s through 2015 (hereinafter “the MONPP BAI”). This BAI qualifies as a CCR storage unit and, as such, is required to be monitored under the United States Environmental Protection Agency (USEPA) final rule for the regulation and management of Coal Combustion Residuals under the Resource Conservation and Recovery Act (RCRA) published April 17, 2015, as amended (40 CFR 257 Subpart D) (herein after “the CCR Rule”), and Michigan Part 115 of the Natural Resources and Environmental Protection Act PA 451 of 1994, as amended (Part 115). On August 5, 2016, the USEPA published the CCR Rule companion *Extension of Compliance Deadlines for Certain Inactive Surface Impoundments* to establish the compliance deadlines for CCR units that were inactive prior to April 17, 2018, which applies to the MONPP BAI.

TRC, on behalf of DTE Electric, has prepared this Groundwater Monitoring and Quality Assurance Project Plan (QAPP) to comply with R 299.4907 of the Part 115 Solid Waste Management Rules. Given that Part 115 covers implementation of the CCR Rule, by following the monitoring program within this QAPP, monitoring requirements of the CCR Rule are also being met. The purpose of the groundwater monitoring program is to determine the current impact, if any, the MONPP BAI may have on the underlying uppermost aquifer system. This will be done by collecting and analyzing groundwater samples that represent the quality of the groundwater that has not been affected by past or present operations at the MONPP BAI and that represent the quality of groundwater passing directly downgradient of the limits of the storage unit.

The sampling and analysis plan and quality assurance procedures contained in this QAPP will be referred to during all groundwater sampling events required to be performed under Part 115 and the CCR Rule.



BASE MAP FROM USGS 7.5 MINUTE TOPOGRAPHIC QUADRANGLE SERIES.



1540 Eisenhower Place
Ann Arbor, MI 48108-3284
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TRC - GIS

PROJECT: **DTE ELECTRIC COMPANY
MONROE POWER PLANT BOTTOM ASH IMPOUNDMENT
3500 EAST FRONT STREET
MONROE, MI 48161**

TITLE: **SITE LOCATION MAP**

| | |
|--------------|-------------------|
| DRAWN BY: | R. SUEMNICHT |
| CHECKED BY: | S. HOLMSTROM |
| APPROVED BY: | V. BUENING |
| DATE: | SEPTEMBER 2019 |
| PROJ. NO.: | 320511.0006 |
| FILE: | 320511-001slm.mxd |

FIGURE 1

2.0 Groundwater Sampling and Analysis Plan

2.1 Objective

The objective of the groundwater sampling and analysis plan is to comply with the requirements of Part 115 by providing consistent sampling and analysis procedures that are protective of human health and the environment and designed to ensure monitoring results that accurately represent groundwater quality throughout the monitoring system. Reasonable attempts will be made to collect samples and analyze them in accordance with these procedures; however, if unforeseen circumstances prevent the collection and/or analysis of groundwater samples in accordance with this plan, the circumstances and result of those circumstances will be fully described in the monitoring report that includes the data.

2.2 Sampling Parameters and Frequency

The monitoring well network is shown on Figure 2 and the monitoring program is summarized on Table 2-1. Monitoring well construction diagrams are included in Appendix A. The constituents for the groundwater monitoring program include constituents listed in Section 11511a. (3)(c) and Section 11519b. (2) of Part 115 and are listed respectively in Tables 2-2 and 2-3 of this plan. Background concentrations of the CCR Rule Appendix III (Table 2-2) and Appendix IV (Table 2-3) groundwater parameters were established from January 2017 through February 2019 by sampling each monitoring well listed in Table 2-1 a minimum of eight times. The detection monitoring program was initiated on July 16, 2019. Background will be established for groundwater detection monitoring constituents not already included in the CCR Rule Appendix III (i.e., iron) throughout eight sampling events per R 299.4440(7).

The detection monitoring program consists of semiannual sampling for the detection monitoring constituents (Table 2-2) at the monitoring wells listed in Table 2-1.

Background and detection monitoring groundwater data will be reported to the laboratory reporting limits (RLs) shown on Tables 2-2 and 2-3.



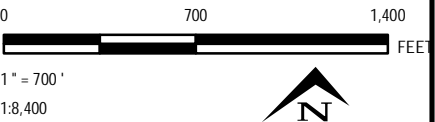
LEGEND

- CCR PROGRAM MONITORING WELL
- INVESTIGATION MONITORING WELL (STATIC WATER LEVELS ONLY)
- UNIT SEPARATION BERM

- APPROXIMATE BOUNDARY OF INACTIVE BOTTOM ASH IMPOUNDMENT
- APPROXIMATE PLANT BOUNDARY

NOTES

- BASE MAP IMAGERY FROM GOOGLE EARTH PRO & PARTNERS, APRIL 2018.



| | |
|----------|--|
| PROJECT: | DTE ELECTRIC COMPANY MONROE POWER PLANT 3500 EAST FRONT STREET MONROE, MI 48161 |
| TITLE: | INACTIVE BOTTOM ASH IMPOUNDMENT WELL LOCATION MAP |

| | |
|--------------|---------------------|
| DRAWN BY: | S.MAJOR |
| CHECKED BY: | Kelly Cratsenburg |
| APPROVED BY: | Vince Buening |
| DATE: | APRIL 2020 |
| PROJ. NO.: | 370029.0006.0000 |
| FILE: | 370029.0006-003.mxd |
| FIGURE 2 | |

Table 2-1
Summary of Groundwater Monitoring Program
DTE Electric Company – Monroe Power Plant Bottom Ash Impoundment

| Well Location | Static Water Level Monitoring | HMP Sampling / Analysis Program | Sampling Frequency | Northing | Easting | TOC Elevation (ft) | Date Installed | Geologic Unit of Screen Interval | Well Construction | Screen Interval Depth (ft BGS) | | | Screen Interval Elevation (ft) | | |
|---------------|-------------------------------|---------------------------------|--------------------|-----------|-------------|--------------------|----------------|----------------------------------|-------------------|--------------------------------|----|-------|--------------------------------|----|--------|
| MW-1S | √ | √ | Semi-Annual | 140176.14 | 13401951.05 | 582.62 | 9/19/2016 | Silt, Sand, and Gravel | 2" PVC, 10 Slot | 33.82 | to | 43.82 | 538.80 | to | 548.80 |
| MW-2S | √ | √ | Semi-Annual | 139070.06 | 13401077.48 | 578.85 | 9/19/2016 | Sand and Clay | 2" PVC, 10 Slot | 30.65 | to | 40.65 | 538.20 | to | 548.20 |
| MW-3S | √ | √ | Semi-Annual | 139417.18 | 13399871.43 | 577.58 | 9/20/2016 | Silt and Sand | 2" PVC, 10 Slot | 29.48 | to | 39.48 | 538.10 | to | 548.10 |
| MW-7S | √ | √ | Semi-Annual | 141102.76 | 13399510.36 | 576.20 | 9/28/2016 | Sand and Gravel | 2" PVC, 10 Slot | 23.60 | to | 33.60 | 542.60 | to | 552.60 |
| MW-9 | √ | √ | Semi-Annual | 140623.10 | 13399606.60 | 579.05 | 9/19/2017 | Sand and Gravel | 2" PVC, 10 Slot | 27.68 | to | 37.68 | 541.37 | to | 551.37 |
| MW-10 | √ | √ | Semi-Annual | 140207.50 | 13399724.80 | 577.46 | 9/20/2017 | Sand and Clay | 2" PVC, 10 Slot | 26.67 | to | 36.67 | 540.79 | to | 550.79 |
| MW-11 | √ | √ | Semi-Annual | 138811.70 | 13399991.40 | 580.58 | 9/20/2017 | Silt | 2" PVC, 10 Slot | 32.74 | to | 42.74 | 537.84 | to | 547.84 |
| MW-12 | √ | √ | Semi-Annual | 138911.90 | 13400748.30 | 582.49 | 9/21/2017 | Silt and Sand | 2" PVC, 10 Slot | 34.59 | to | 44.59 | 537.90 | to | 547.90 |
| MW-13 | √ | √ | Semi-Annual | 139800.40 | 13401644.60 | 580.97 | 9/21/2017 | Clay, Silt, and Sand | 2" PVC, 10 Slot | 27.72 | to | 37.72 | 543.25 | to | 553.25 |
| MW-14 | √ | √ | Semi-Annual | 141406.50 | 13401772.20 | 580.76 | 9/22/2017 | Sand and Clay | 2" PVC, 10 Slot | 32.89 | to | 42.89 | 537.87 | to | 547.87 |
| MW-15 | √ | √ | Semi-Annual | 141789.10 | 13399419.60 | 580.80 | 9/26/2017 | Sand and Gravel | 2" PVC, 10 Slot | 31.19 | to | 41.19 | 539.61 | to | 549.61 |
| | | | | | | | | | | | | | | | |
| MW-1D | √ | | | 140178.92 | 13401952.04 | 582.82 | 9/19/2016 | Limestone Bedrock | 2" PVC, 10 Slot | 73.42 | to | 83.42 | 499.40 | to | 509.40 |
| MW-3D | √ | | | 139422.09 | 13399871.16 | 577.42 | 9/20/2016 | Limestone Bedrock | 2" PVC, 10 Slot | 68.42 | to | 78.42 | 499.00 | to | 509.00 |
| MW-4S | √ | | | 141163.06 | 13401614.14 | 580.67 | 9/26/2016 | Silt and Sand | 2" PVC, 10 Slot | 29.57 | to | 39.57 | 541.10 | to | 551.10 |
| MW-5S | √ | | | 142564.92 | 13401176.41 | 584.50 | 10/4/2016 | Clay | 2" PVC, 10 Slot | 15.80 | to | 25.80 | 558.70 | to | 568.70 |
| MW-7D | √ | | | 141099.21 | 13399510.92 | 576.17 | 9/28/2016 | Limestone Bedrock | 2" PVC, 10 Slot | 58.47 | to | 68.47 | 507.70 | to | 517.70 |
| MW-8S | √ | | | 140560.53 | 13397828.28 | 586.59 | 9/30/2016 | Sand and Clay | 2" PVC, 10 Slot | 35.89 | to | 45.89 | 540.70 | to | 550.70 |
| MW-8D | √ | | | 140561.00 | 13397828.00 | 586.45 | 9/30/2016 | Limestone Bedrock | 2" PVC, 10 Slot | 59.25 | to | 69.25 | 517.20 | to | 527.20 |

Notes:

Survey completed by AECOM, 2016-2017

Elevation in feet relative to North American Vertical Datum 1988 (NAVD 88).

TOC: Top of well casing.

ft BTOC: Feet below top of well casing.

ft BGS: Feet below ground surface.

NM = Not measured

NR = Not recorded

Table 2-2
Groundwater Monitoring Parameters – Detection Monitoring Constituents
DTE Electric Company – Monroe Power Plant Bottom Ash Impoundment

| Part 115 Amendments - Public Act No. 640 of 2018 | | | | |
|--|--|-------|-----------------|-----------------------------------|
| Section 11511a(3)(c) - Detection Monitoring Constituents | | | | |
| Parameter | Monitored Under 40 CFR 257 Subpart D | Units | Reporting Limit | Method |
| Field Parameters | | | | |
| pH | √ | S.U. | NA | Field |
| Wet Chemistry | | | | |
| Chloride | √ | mg/L | 1.0 | EPA 325.3/SM4500- CL/9056A_28D |
| Fluoride | √ | mg/L | 0.050 | EPA 325.3/SM4500- CL/9056A_28D |
| Sulfate | √ | mg/L | 1.0 | EPA 375.2/ 9056A_28D |
| Total Dissolved Solids | √ | mg/L | 10.0 | EPA 160.1/SM2540C |
| Metals | | | | |
| Boron | √ | ug/L | 100 | 6010/6020 |
| Calcium | √ | ug/L | 1,000 | 6010/6020 |
| Iron | | ug/L | 100 | 6010/6020 |

ug/L = micrograms per liter

mg/L = milligrams per liter

NA = Not Applicable

EPA = United States Environmental Protection Agency

S.U. = Standard Unit

Table 2-3
Groundwater Monitoring Parameters – Assessment Monitoring Constituents
DTE Electric Company – Monroe Power Plant Bottom Ash Impoundment

| Part 115 Amendments - Public Act No. 640 of 2018 | | | | |
|--|--------------------------------------|-------|-----------------|-------------------|
| Section 11519b(2) - Assessment Monitoring Constituents | | | | |
| Parameter | Monitored Under 40 CFR 257 Subpart D | Units | Reporting Limit | Method |
| Radium | | | | |
| Radium-226 | √ | pCi/L | 5 | 903.0 |
| Radium-228 | √ | pCi/L | 5 | 904.0 |
| Combined Radium 226 and 228 | √ | pCi/L | 5 | Ra226_Ra228 |
| Metals | | | | |
| Antimony | √ | ug/L | 2 | 6020 |
| Arsenic | √ | ug/L | 5 | 6020 |
| Barium | √ | ug/L | 5 | 6010/6020 |
| Beryllium | √ | ug/L | 1 | 6020 |
| Cadmium | √ | ug/L | 1 | 6020 |
| Chromium | √ | ug/L | 2 | 6020 |
| Cobalt | √ | ug/L | 1 | 6020 |
| Copper | | ug/L | 2 | 6010/6020 |
| Lead | √ | ug/L | 1 | 6020 |
| Lithium | √ | ug/L | 8 | 6010/6020 |
| Mercury | √ | ug/L | 0.2 | 245.1/7470A/7471A |
| Molybdenum | √ | ug/L | 5 | 6020 |
| Nickel | | ug/L | 2 | 6010/6020 |
| Selenium | √ | ug/L | 5 | 6020 |
| Silver | | ug/L | 1 | 6020 |
| Thallium | √ | ug/L | 1 | 6020 |
| Vanadium | | ug/L | 5 | 6010/6020 |
| Zinc | | ug/L | 20 | 6010/6020 |

pCi/L = picocurie per liter
ug/L = micrograms per liter

2.3 Water Level Measurements

Groundwater elevations will be measured throughout the monitoring system during each sampling event, prior to purging for the collection of groundwater samples. Measurements will be taken within a single 24-hour period. Data from monitoring wells screened in the uppermost aquifer will be used to determine the direction of groundwater flow in that zone, and potentiometric surface maps will be prepared following each sampling event.

Groundwater level measurements will be made using a surveyed reference point established on the well casing. The reference point will consist of an indelible mark on the well casing on the highest point, or the north side of the well casing.

A battery-operated water level indicator will be utilized as the primary device for water level measurements. The indicator is a self-contained instrument equipped with a cable and sensor that activates a buzzer and a light when it comes in contact with the water. The depth to water is read from permanent 0.01-foot increment markings on the cable. Depth to water is measured to the nearest 0.01 foot.

Depth to bottom in each well will be determined based on well construction logs. If depth to bottom measurements are necessary, the measurements will be collected after groundwater sampling has been completed such that the depth to bottom measurement does not cause disturbance within the well prior to sampling.

Water level measurements will be used to evaluate the rate and direction of groundwater flow during each sampling event.

2.4 Groundwater Sample Collection

Groundwater sample collection will consist of the following steps:

1. Note the condition of the wells on the Well Inspection Form (included in Appendix B) and implement corrective action for any deficiencies as applicable.
2. Measure and record the water level measurement.
3. Conduct well purging as described below with a dedicated bladder pump and tubing.
4. Collect samples for laboratory and field analyses as described below.

2.4.1 Purging Procedures

All monitoring wells will be purged with a dedicated bladder pump using low-stress purging methods except for MW-7S, which will be sampled using a peristaltic pump and dedicated tubing. Field measurements (e.g., temperature, pH, specific conductance, turbidity) for the purposes of stabilization will begin after drawdown of the water level in the well has stabilized. Oxidation reduction potential (ORP) and dissolved oxygen (DO) measurements may also be collected but will not be used to determine stabilization.

The following procedures will be used for all monitoring wells sampled:

- For monitoring wells with dedicated bladder pumps, connect the control box and air supply to the dedicated bladder pump head connectors.
- For monitoring wells sampled using a peristaltic pump (MW-7S), connect silicon tubing to the polyethylene tubing, then place the silicon tubing in the peristaltic pumps tubing dock.
- Connect the pump discharge tubing to the flow-through cell containing pre-calibrated instruments.
- Start the pump at low speed and slowly increase the rate until discharge occurs. Check water level. Adjust pumping rate until there is little or no water level drawdown, maintaining a flow rate within 0.1 to 0.5 liters per minute. Try to match pumping rate used during previous sampling event(s). Check drawdown every 1 to 5 minutes to ensure drawdown does not exceed more than approximately 0.3 feet over three consecutive 3 to 5-minute interval readings. If flow is reduced to lowest possible rate (~0.1 liters per minute) and drawdown continues, continue with purging at lowest rate until parameters stabilize or the water level reaches the top of the well screen.
- Once an appropriate purge rate is achieved, and the total volume of the pump system and/or tubing has been evacuated (Table 2-4), continue purging until stabilization is achieved (after all field parameters have equilibrated for at least 3 consecutive readings). Readings will be taken in 3 to 5-minute intervals such that the flow of water through the cell is replaced between measurements. The water is considered stable when the following conditions have been achieved: pH change is +/- 0.1 S.U., conductivity change is +/- 10 percent, temperature change is +/- 0.5°C, and turbidity change is +/- 10 percent (or value is less than or equal to 5 Nephelometric Turbidity Units [NTUs]) for three successive 3 to 5-minute intervals. Stabilization requirements are also noted on the Water Sample Log in Appendix B. Groundwater in the well will be considered representative when both drawdown and groundwater quality indicator parameters have stabilized.
- If the water level does not stabilize at the lowest possible pumping rate, and the well is essentially being dewatered during purging, the well should be sampled as soon as the water level has recovered sufficiently to collect the volume needed for all anticipated samples. The project manager or technical coordinator will need to make the decision when samples should be collected, and the reasons recorded on the purge form and/or fieldbook.
- If water level is stable and field parameters will still not stabilize, the field technician will contact the project manager or technical coordinator who will need to make the decision when samples should be collected, and the reasons recorded on the purge form and/or fieldbook.
- In addition to providing basic groundwater quality characterization information, each groundwater sample may also be field-analyzed for ORP and DO using the flow-through cell
- After stability is reached, disconnect the flow-through cell from the pump outlet line and fill all of the sampling bottles for the appropriate parameters in the appropriate manner as described below
- Well purging information will be recorded on the sampling form(s) included in Appendix B.

Table 2-4
Bladder/Peristaltic Pump System Specifications
DTE Electric Company – Monroe Power Plant Bottom Ash Impoundment

| Monitoring Well | Purge Method | Bladder Pump Tubing Length (ft) | Peristaltic Pump Tubing Length (ft) ¹ | Total Tubing Length (cm) | Tubing Inner Radius (cm) | Tubing Volume (mL) | Tubing Volume (L) | Bladder Pump Volume (L) | Tubing and Bladder Pump Total Volume (L) |
|-----------------|------------------------|---------------------------------|--|--------------------------|--------------------------|--------------------|-------------------|-------------------------|--|
| MW-1S | Dedicated Bladder Pump | 37.2 | -- | 1,135 | 0.318 | 359 | 0.4 | 0.4 | 0.8 |
| MW-2S | Dedicated Bladder Pump | 33.8 | -- | 1,031 | 0.318 | 327 | 0.3 | 0.4 | 0.7 |
| MW-3S | Dedicated Bladder Pump | 31.5 | -- | 960 | 0.318 | 304 | 0.3 | 0.4 | 0.7 |
| MW-7S | Peristaltic Pump | -- | 35.6 | 1,085 | 0.216 | 159 | 0.2 | 0.4 | 0.6 |
| MW-9 | Dedicated Bladder Pump | 29.5 | -- | 899 | 0.318 | 285 | 0.3 | 0.4 | 0.7 |
| MW-10 | Dedicated Bladder Pump | 28.0 | -- | 853 | 0.318 | 270 | 0.3 | 0.4 | 0.7 |
| MW-11 | Dedicated Bladder Pump | 32.5 | -- | 991 | 0.318 | 314 | 0.3 | 0.4 | 0.7 |
| MW-12 | Dedicated Bladder Pump | 35.5 | -- | 1,082 | 0.318 | 343 | 0.3 | 0.4 | 0.7 |
| MW-13 | Dedicated Bladder Pump | 29.5 | -- | 899 | 0.318 | 285 | 0.3 | 0.4 | 0.7 |
| MW-14 | Dedicated Bladder Pump | 34.0 | -- | 1,036 | 0.318 | 328 | 0.3 | 0.4 | 0.7 |
| MW-15 | Dedicated Bladder Pump | 32.5 | -- | 991 | 0.318 | 314 | 0.3 | 0.4 | 0.7 |

¹ Calculated using the total depth of the well, minus 1 foot for the amount the tubing is from the bottom of the well, plus 3 feet for the amount of tubing above the top of casing

For wells MW-1S - MW-3S, the tubing length was determined by using the pump depth information referenced in AECOM WIR Table 1, Monitoring Well Construction Summary, and subtracting 3.5 feet for the length of the pump.

For wells MW-9 - MW-15, the bladder pump tubing length was referenced from AECOM Well Wizard Specification Sheet

2.4.2 Field Measurements

Equipment used in the field will be operated and calibrated in accordance with manufacturer's specifications. The flow-through cell used for temperature, conductivity, ORP, DO, and pH will be a YSI 556 model, or equivalent. A Lamotte 2020 or equivalent will be used to measure turbidity. Field parameter results and notes will be recorded on field forms and will be used to note stabilization data (Appendix B). Further explanation of documentation is contained below in Section 2.6. Calibration procedures for field instruments are included in Section 3.1.

2.4.2.1 Measurement of Temperature

Temperature will be measured in a flow-through cell to $\pm 0.01^{\circ}\text{C}$ accuracy. The flow-through cell will be connected directly to the pump discharge. If a flow-through cell cannot be used, a portable temperature probe will be utilized. When using the portable temperature probe, a water sample will be collected directly from the pump discharge. The temperature probe will immediately be inserted into the cup, and temperature will be measured and recorded.

2.4.2.2 Measurement of Specific Conductance

A flow-through cell with a temperature-compensating specific conductance meter, YSI 556 or equivalent, will be used to measure the specific conductance of the groundwater sample. Alternatively, if a flow-through cell cannot be utilized, a water sample will be collected and placed in a disposable cup. A portable specific conductance probe will immediately be inserted into the cup and the specific conductivity will be measured and recorded.

2.4.2.3 Measurement of pH

A flow-through cell with a pH probe will be used to measure the pH of the groundwater sample. Alternatively, if a flow-through cell cannot be utilized, a water sample will be collected and placed in a disposable cup. A portable pH probe will immediately be inserted into the cup, and the pH will be measured. The measurements will be recorded to the nearest 0.01 pH standard unit.

2.4.2.4 Measurement of Turbidity

During purging, a portion of a groundwater sample will be analyzed for turbidity using a portable nephelometric turbidity meter (Lamotte 2020, or equivalent) as follows:

1. Collect sample in clean, unscratched vial. If small scratches are present, apply a thin film of silicon oil with a lint-free cloth, as recommended by the manufacturer. Fill the vial to its neck, avoiding the creation of bubbles, and place the cap on the vial. If large scratches are present, discard the vial.
2. Wipe the vial dry using a lint free cloth, place in the sample well, cover and take the reading. If condensation develops, allow the sample to come to ambient temperature, then take the reading after agitating the vial to re-suspend any clay, silt, or sand particles that may have settled.

Measurement of Dissolved Oxygen (DO)

A flow through cell with a dissolved oxygen probe will be used to measure dissolved oxygen. Alternatively, if a flow-through cell cannot be utilized, a water sample will be collected and placed in a disposable cup. A portable DO probe will be immediately inserted into the cup and the DO will be measured. Measurement will be made to the nearest 0.01 milligrams per liter.

Measurement of Oxidation Reduction Potential (ORP)

A flow through cell with an ORP probe will be used to measure ORP. Alternatively, if a flow-through cell cannot be utilized, a water sample will be collected and placed in a disposable cup. A portable ORP probe will be immediately inserted into the cup and the ORP will be measured. Measurement will be made to the nearest 0.1 millivolts.

2.4.3 Sampling Procedures

Immediately after the well has reached stabilization, samples will be collected. If the well is pumped dry, it will be sampled when a sufficient volume of water has re-entered the well. All sampling will be performed within 24 hours of purging unless a well does not yield sufficient groundwater. The list of parameters and analytical methods are provided in Tables 2-1 and 2-2.

Procedures for the sampling of the monitoring wells are as follows:

1. Verify that sufficient pre-cleaned vials and pre-cleaned bottles are available for each sampling location and that each is properly labeled.
2. Samplers will use nitrile or other appropriate gloves as specified in the site-specific health and safety plan before handling sampling containers.
3. Immediately fill the sample bottle by allowing the water stream to strike the inner wall of the bottle to minimize formation of air bubbles. Do not rinse the sample bottle. Fill the sample bottle with minimal splashing. Bottle size and preservative information is outlined in Table 2-5.
4. Fill the bottles in the following order:
 - Total recoverable metals (unfiltered);
 - Chloride, fluoride, sulfate, pH, and TDS; and
 - Radium 226 and 228.
5. Collect blind duplicate samples at the rate of one each per every 10 samples or part thereof. When collecting the duplicate sample, split aliquots of sample into the primary and duplicate sample containers used for each analytical parameter until both sample containers are full.

See Table 2-5 for requirements for containers, filtering, preservatives, and holding times for sample collection. The sample containers will be pre-cleaned by the manufacturer and tested to meet or exceed the analyte specifications provided in USEPA guidance documents. Any chemicals necessary for sample preservation will be provided by the laboratory.

2.5 Equipment Decontamination

Between sampling each well, the following procedures will be employed to decontaminate the equipment.

2.5.1 Water Level Indicator

1. Rinse probe with soapy (phosphate-free soap) water.
2. Rinse with deionized water (ASTM Type II).
3. Air-dry.

2.5.2 Turbidity Meter, Conductivity Probe, pH Meter, and Thermometer

1. Rinse with deionized water.

2.5.3 Flow Through Cell

The flow through cell will be decontaminated by washing with Alconox soap and potable water, followed by a potable water rinse. More rigorous decontamination is not needed, since samples will be collected by disconnecting the inflow hose from the flow through cell and filling sample containers directly. Sample collection in this manner ensures that the sample material does not contact the flow through cell.

2.5.4 Documentation

- Field forms/logs (raw data)
- Chain of Custody (COC)
- Sample labels

2.6 Field Records

Daily field activities will be recorded in a bound notebook. The sample collection notebook will contain equipped with forms for general notes (including weather conditions and a daily summary of the work performed), meter calibration logs, low-flow groundwater sampling stabilization log, groundwater sample logs, and water level data forms. Entries will include the name, make, and model (or equivalent) of equipment utilized for field water level, temperature, conductivity, pH, and turbidity measurements. The make and model of pump(s) dedicated to each well will also be noted. Example field forms are located in Appendix B. Any deviations from normal operating conditions will be noted on the stabilization form or water sample log.

Entries in the field notebook and sample collection and measurement forms will be legibly written and will provide a clear record of field activities. Entries will be made in waterproof ink, in language that is objective and factual. Errors will be indicated by drawing a single line through the text, such that the text in error remains legible. Errors addressed in this manner will be initialed and dated by the person making the correction. The person taking notes in the fieldbook will sign and date each page.

Table 2-5
Groundwater Sample Containers and Holding Times
DTE Electric Company – Monroe Power Plant Bottom Ash Impoundment

| Parameters | METHOD | CONTAINER | PRESERVATION | HOLDING TIME |
|--|-------------------------------|----------------|---------------------------|--------------|
| Chloride, Fluoride | EPA 325.3/SM4500-CL/9056A_28D | 500 mL Plastic | 4 degrees C unpreserved | 28 days |
| Sulfate | EPA 375.2/9056A_28D | | | 28 days |
| Total Dissolved Solids | EPA 160.1/SM2540C | | | 7 days |
| pH | Field Measurement | None | None | In Field |
| Total Recoverable Metals (unfiltered) | SW846 6010/6020 | 500 mL Plastic | HNO ₃ to pH <2 | 6 months |
| Total Mercury (unfiltered) | 245.1/7470A/7471A | | | 28 days |
| Radium 226 | GFPC 903.0 | 1 L Plastic | HNO ₃ to pH <2 | 6 months |
| Radium 228 | GFPC 904.0 | 1 L Plastic | | |

Note:

No samples require field filtration.

3.0 Quality Assurance/Quality Control Plan

3.1 Field Quality Assurance/Quality Control (QA/QC)

The following steps will be taken so that the analytical data gathered in the field are both valid and unbiased:

- Field technicians are trained in the use of each piece of equipment.
- Preventive maintenance programs are carried out on a scheduled basis.
- Spare components are taken into the field in case of equipment failure or damage.
- Instruments are calibrated on a daily basis and rechecked at various times daily.
- Standard forms are used to document field activities (see Appendix B).
- Readings and calibrations are documented.
- Daily QC checks of field notes are performed.

The accuracy, sensitivity, and precision of the field analytical measurements (e.g., pH, temperature, turbidity, DO, ORP, and specific conductance) are dependent upon the specifications for the instruments used, as well as the QC techniques employed during their use. These techniques are outlined below.

Temperature

Field measurements of samples will be recorded to the nearest 0.01°C as it moves through the flow-through cell, or immediately after the sample is removed from the well, if a flow-through cell is not utilized.

Each field temperature probe will be visually checked before each field trip and daily while in use to determine that it is not cracked and that there are no air spaces or bubbles in the probe. Damaged or suspect equipment will be replaced before use.

Specific Conductance

Each meter will be inspected for physical damage before each field trip and daily while in the field. Calibration of the meter will be completed at the beginning of each day and additionally as needed, in accordance with the instrument's operation manual. In case of an apparent discrepancy in a specific conductance measurement, the electrode will be checked with the standard solution and recalibrated. The sample will then be reanalyzed. Results will be expressed in micromhos/centimeter ($\mu\text{mhos/cm}$), with the temperature automatically corrected to 25°C, and the value reported to the nearest 1 unit.

pH

The meter will be checked before each field trip and daily while in the field for any mechanical or electrical failures, weak batteries, and cracked or fouled electrodes. The meter will be

calibrated once daily using standard buffer solutions of known pH values (e.g., 4, 7, and 10) as described in the instrument's operation manual. In case of an apparent discrepancy in a pH measurement, the electrode will be checked with pH 7.0 buffer and rebuffed to the closest reference buffer. The sample will then be reanalyzed. Results will be expressed to the nearest 0.01 standardized unit (SU).

Turbidity

Each meter will be inspected for physical damage before each field trip and daily while in the field. The accuracy of the instrument will be checked daily against a known NTU standard. If turbidity readings exceed the range of the calibrated standard, the meter will be re-calibrated for the next highest standard, and the sample will be reanalyzed. Calibration results should agree within ± 5 percent and be recorded in the field forms. Results will be expressed in NTUs and recorded to ± 0.01 NTU. Negative NTU readings indicate that air is entrained within the sample and requires resampling and/or recalibration of the meter.

DO

Each meter will be inspected for physical damage before each field trip and daily while in the field. Calibration of the meter will be completed at the beginning of each day and in accordance with the instrument's operation manual. In case of an apparent discrepancy in a DO measurement, the electrode will be checked with a water-saturated air measurement and recalibrated. The sample will then be reanalyzed. Results will be expressed in milligrams per liter (mg/L) and the value reported to the nearest 0.01 unit.

ORP

Each meter will be inspected for physical damage before each field trip and daily while in the field. Calibration of the meter will be completed at the beginning of each day and in accordance with the instrument's operation manual. In case of an apparent discrepancy in an ORP measurement, the electrode will be checked with the standard solution and recalibrated. The sample will then be reanalyzed. Results will be expressed in millivolts (mV) and the value reported to the nearest 0.1 unit.

3.2 Laboratory Analytical Procedures and QA/QC

The current groundwater quality monitoring program parameters, and analytical procedures, are provided in Tables 2-2, and 2-3. Holding time and preservation requirements for the various parameters are listed in Table 2-5.

Test America Laboratories, Inc. has been contracted to perform the sampling analysis, a copy of the laboratory's QA/QC plan is included on CD in Appendix C. The laboratory's QA/QC Plan will meet the quality assurance and quality control procedures given in SW-846 Test Methods for Evaluating Solid Waste. Any changes to the laboratory selected to perform the groundwater analyses will be documented in the annual monitoring report.

The laboratory will perform at least the following QC checks (summarized in Table 3-1):

- Spikes
 - Laboratory control samples, one per analytical batch of up to 20 samples; and
 - Matrix spikes/Matrix Spike Duplicates (MSs/MSDs), one per analytical batch of up to 20 samples, where applicable.
- Blanks (method/preparation) for each analytical batch
- Calibration standards
- Inorganics: initial calibrations and calibration verifications at a frequency of every 10 samples or, as specified in the analytical method

The following QC deliverables will be included with each laboratory data package:

- Laboratory preparation blanks
- Laboratory control samples (LCS) results
- MS/MSD spike recovery and relative percent difference (RPD) results
- Laboratory duplicate results (at least 1 in 20) for inorganic parameters where MS/MSD is not appropriate
- COC for all samples
- Sample Receipt Form that indicates cooler receipt temperatures
- Case narrative for all samples/analyses signed by the Laboratory Manager or QA Manager
- Statement of Quality Assurance signed by the Laboratory Manager or QA Manager

Table 3-1
Laboratory Quality Control Samples
DTE Electric Company – Monroe Power Plant Bottom Ash Impoundment

| Total Recoverable Metals | Anions | Radium | Total Dissolved Solids | pH |
|---|--|--|--|------------------------------------|
| Laboratory method / preparation blanks | Laboratory method / preparation blanks | Laboratory method / preparation blanks | Laboratory method / preparation blanks | -- |
| Laboratory control sample (LCS) results | LCS results | LCS results | LCS results | LCS results |
| MS/MSD recoveries (1 in 20) | MS/MSD recoveries (1 in 20) | -- | -- | -- |
| Duplicate sample results (1 in 20) | -- | Duplicate sample results (1 in 20) | Duplicate sample results (1 in 20) | Duplicate sample results (1 in 20) |

Notes:

Chain-of-Custody for all samples
Case Narratives for all samples/analyses

3.3 Data Quality Objectives

The data quality objective (DQO) for this Groundwater Monitoring Plan is to generate data from the analytical laboratory that meet the requirements set forth in the CCR Rule and Part 115.

3.4 Quality Assurance Objectives

The overall QA objective is to ensure that the data are of known and acceptable quality. The data must be sufficiently precise and accurate to be used for the purposes indicated in this Groundwater Monitoring Plan.

To achieve the overall DQOs, proper sample handling and analysis, and data-handling procedures will be followed. This document describes the specific objectives for analytical accuracy, precision, sensitivity, completeness, representativeness, and comparability.

3.4.1 Accuracy, Precision, and Sensitivity of Analysis

The fundamental QA objective, with respect to accuracy, precision, and sensitivity of laboratory analytical data, is to achieve the QC acceptance criteria of the analytical protocols. The definitions of the above-mentioned parameters are provided as follows:

3.4.1.1 Accuracy

Accuracy is the proximity of a measurement to the true value. Both field and analytical accuracy will be monitored through initial and continuing calibration of instruments. In addition, the data from the matrix spikes, and blanks will be used to assess the accuracy of the laboratory analytical data.

The accuracy of laboratory results will be assessed for compliance with the method-specific QC criteria using the analytical results of method/preparation blank, and matrix spike/matrix spike duplicate samples.

3.4.1.2 Precision

Precision is the level of agreement among multiple measurements of the same parameters. The goal is to maintain an acceptable level of analytical precision. Checks for analytical precision include the analysis of MS/MSDs, laboratory duplicates, and field duplicates.

The precision of laboratory analysis will be assessed by comparing the analytical results between MSs/MSDs, and laboratory duplicate analyses. The relative percent difference (%RPD) will be calculated for each pair of duplicate analyses.

3.4.1.3 Sensitivity

Method Detection Limits (MDLs) and Practical Quantitation Limits (PQLs) are measures of the analytical procedure's sensitivity for the detection and quantitation of analytes, respectively.

The achievement of MDLs depends on instrument sensitivity and matrix effects. Therefore, it is

important to monitor the instrument sensitivity to ensure the quality of the data through constant instrument performance. The instrument sensitivity will be monitored through the analysis of a method blank, a calibration check sample, and laboratory control samples, etc. The laboratory performs periodic MDL studies for each analyte.

Data will be reported to the laboratory reporting limits presented in Tables 2-2 and 2-3.

3.4.2 Completeness, Representativeness, and Comparability

3.4.2.1 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared with the amount that was expected to be obtained under normal conditions. Following completion of the analytical testing, the percent completeness will be calculated by the following equation:

$$\text{completeness (\%)} = \frac{(\text{number of valid measurements})}{(\text{total number of measurements})} \times 100$$

It is expected that the laboratory will provide data meeting QC acceptance criteria for 95 percent or more of all samples tested. The completeness of laboratory and field requirements is 90 percent or better.

3.4.2.2 Representativeness

Representativeness expresses the degree to which the data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness is a qualitative parameter that is dependent upon the proper design of the sampling program and the proper laboratory protocol. Representativeness will be satisfied by ensuring that the Sampling and Analysis Plan is followed, proper sampling techniques are used, and proper analytical procedures are followed. Representativeness will be assessed by the analysis of selected field duplicate samples.

3.4.2.3 Comparability

Comparability expresses the confidence with which one data set can be compared with another. The extent to which existing and planned analytical data will be comparable depends upon the similarity of sampling and analytical methods. The procedures used to obtain the planned analytical data, as documented in this document are expected to provide comparable data.

Field analysis QA/QC has been described above. Any deviations in the procedures from this plan and any difficulties encountered while sampling will be documented in the field. All field data sheets are also checked by qualified staff upon their return to the office.

Project-specific field QC checks are summarized in Table 3-1 and include the following:

- Blind field duplicates

Blind field duplicate groundwater samples are collected at the rate of one for every 10 samples or fraction thereof. No field equipment blanks will be collected because all purging and sampling will be performed with dedicated equipment.

The handling and processing of all samples and QA/QC procedures by the laboratory are described in detail in the laboratory QA/QC Manual and the laboratory Standard Operating Procedures (SOPs).

3.5 Chain-of-Custody Procedures

The possession of samples must be traceable from the time of collection through the use of COC procedures. Specific COC forms must accompany all sample shipping containers to document the transfer of the shipping containers and samples from the field to the laboratory receiving the samples for analysis. Chain-of-custody procedures are as follows.

Field Chain-of-Custody Procedures

- The field sampler is personally responsible for the care and the custody of the samples until they are transferred or properly dispatched. As few people as possible will handle the samples.
- Label bottles with sample identification, time, date and sampler initials.
- Complete sample labels for each sample using waterproof ink, unless prohibited by weather conditions. For example, a field note entry would explain that a pencil was used to fill out the sample tag because the ball-point pen would not function in freezing weather.
- The site coordinator/project manager will review field activities to ensure proper custody procedures were followed during the fieldwork.

Transfer of Custody of Shipment Procedures

The possession of samples must be traceable from the time of collection through the use of Chain-of-Custody Record procedures. Specific Chain-of-Custody forms will accompany sample-shipping containers to document the transfer of the shipping containers and samples from the field to the laboratory receiving the samples for analysis. The procedures are as follows:

- Prepare sample containers with pre-applied labels by the laboratory (see example in Appendix B), with Chain-of-Custody seals on the shipping containers.
- Properly identify and label each sample in the field with indelible, waterproof ink.
- Complete the Chain-of-Custody forms in the field, indicating the sample identification, the containers filled, the sampling date, the sampling time, the sample collector's name, and the sample preservation method, if applicable. This information will also be noted on the field logs.

- Repack the shipping containers with samples, Chain-of-Custody forms, and ice packs. Each set of sample containers to be shipped together in a single shipping container is assigned a Chain-of-Custody form, which travels with the shipping container.
- Seal and ship containers to the appropriate laboratory. Common carriers or intermediate individuals shall be identified on the Chain-of-Custody form, and copies of bills-of-lading will be retained.
- Receive and check the shipping containers in the laboratory for broken seals or damaged sample containers. If no problems are noted, the samples are logged into the laboratory, and the Chain-of-Custody form is completed. The person relinquishing the samples to the facility or agency should request the representative's signature acknowledging sample receipt. If the representative is unavailable or refuses, this is noted in the "Received By" space.
- Include copies of the Chain-of-Custody form with the analytical data.
- Return unused sample containers to the laboratory with the Chain-of-Custody forms.

While filling out the Chain-of-Custody form, it is important to write legibly. Errors are to be corrected by first drawing a single line through the incorrect information and then entering the correct information. All corrections are to be initialed and dated by the person making the correction.

Completed Chain-of-Custody forms will be placed in a plastic bag, sealed, and taped to the inside cover of the shipping container. After icing the samples, the coolers will be sealed, dated, and shipped to the laboratory using an overnight delivery service.

If a Chain-of-Custody form is lost in shipment, a written statement will be prepared by the person who collected the samples listing the samples that were recorded on the lost form and describing when and how the samples were collected. The statement should include information such as field notes regarding the sample. This statement is submitted to the TRC project manager for further action, as necessary.

Final Evidence Files Custody Procedures

TRC is the custodian of the evidence files and maintains the contents of the evidence files, including all relevant records, reports, logs, field notebooks, pictures, subcontractor reports, and data reviews. The laboratory custody procedures followed should be as dictated by the contract laboratory QC Manual.

3.6 Field Data Validation Procedure

Procedures to validate field data will primarily include checking for transcription errors and reviewing field forms. This task is the responsibility of the Field Coordinator. The data reviewer will review field notes and field Chain-of-Custody Records to determine if procedures have been followed.

3.7 Groundwater Quality Data Validation Procedure

DTE Electric will perform data validation. The designated data validator(s) will conduct a review

of the spike, duplicate, and blank results provided by the laboratory as well as verify that the sample holding times were met. Additional QC information set forth in the following bullets will be reviewed to check for appropriate matrix performance using the specified analytical methods.

The procedures used to evaluate data may include the following items:

- Checking technical holding times for inorganic and organic analyses, as applicable.
- Reviewing data for blanks, surrogate spikes, matrix spikes/matrix spike duplicates (organics), laboratory duplicates (inorganic), and laboratory control samples.
- Reviewing internal standard areas and retention times (RT), as appropriate.
- Determining field precision from blind field duplicate data.
- Checking the completeness of the data package to determine if samples and analyses required by the QAPP were processed, if the procedures specified in the QAPP were implemented, and if deliverables specified in the QAPP are included.
- Identifying any questionable data and data omissions and interacting with the laboratory to correct data deficiencies. This is the responsibility of the data reviewer.
- Deciding whether to repeat sample collection and analyses based on the extent of the deficiencies and their importance in the overall context of the project. This is the responsibility of the TRC project manager.
- Assessing the usability of results against the DQOs. This is the responsibility of the data reviewer.

The data usability report will address the following items:

- The usability of the data if QC results indicate that there are potential problems with all or some of the data.
- Potential sample contamination as a result of blank contributions.

4.0 Establishing Background Quality

Background groundwater quality in the uppermost aquifer will be established at the site by analyzing groundwater collected from the HMP program monitoring wells listed in Table 2-1. If site conditions change, the well(s) included in the network/background database may be revised, and, if necessary, a new well location will be proposed for EGLE approval, as appropriate based on site data. The background dataset for each monitoring well will include a minimum of eight rounds of data.

Background groundwater monitoring was conducted for constituents in Appendix III and Appendix IV of the CCR Rule at the monitoring wells shown in Table 2-1 from January 2017 through February 2019. Background will be established for the Section 11511a. (3)(c) constituents not already included in the CCR Rule Appendix III (i.e., iron) throughout eight sampling events. Following the establishment of background groundwater quality, monitoring wells will be sampled semiannually as part of the detection monitoring program. Analytical results and data reports as defined below will be submitted to the director no later than 30 days after the end of the calendar quarter in which the samples were obtained.

All data collected from the wells in accordance with CCR Rule and Part 115 will be documented in the operating record in accordance with the recordkeeping requirements specific in 40 CFR 257.105(h) and, as necessary, made available on the CCR Web site in accordance with 40 CFR 257.107, as referenced by Section 11519a. (2)(b) and (c) of Part 115.

4.1 Data Evaluation Procedures

In accordance with the CCR Rule and Part 115, DTE Electric will determine whether or not there is a statistically significant increase from the background data set for each of the detection monitoring constituents after completing the first round of sampling subsequent to completing the background data collection. After the eighth background sample has been obtained, the background dataset will be evaluated using the statistical procedures summarized in the statistical data evaluation plan developed for this site. Per R 299.4908, the statistical test will be by one of the following methods:

- A Parametric Analysis of Variance followed by a multiple comparisons test to identify statistically significant evidence of contamination. This will include estimation and testing of the contrasts between each monitoring well's mean and the background mean levels for the applicable indicator parameter.
- An Analysis of Variance based on ranks followed by a multiple comparison test to identify statistically significant evidence of contamination. This will include estimation and testing of the contrasts between each monitoring well's median and the background median levels for the applicable indicator parameter.
- A Tolerance or Prediction Interval Test in which an interval for each indicator parameter is established from the distribution of the pooled background data set, and the level of each parameter in each monitoring well is compared with the Upper Tolerance Limit or Prediction Limit.
- A control chart approach that gives control limits for each indicator parameter.

- Another suitable statistical method selected from applicable tests that meet the performance standards set forth in R 299.4908 (1)(e) and subpart 257.93(g) of the CCR Rule.

The statistical method selected is detailed in the Groundwater Statistical Evaluation Plan for the MONPP BAI and complies with the performance standards outlined in R 299.4908 and the CCR Rule, subpart 257.93(g).

For data collected from background wells the following shall be adhered to:

- If RLs are increased due to laboratory interference during the period of background data collection and a nondetect is reported, the value will not be included in the background data set. The well will be re-sampled and analyzed in accordance with an alternate method (ICP-MS).
- If data quality review results in any anomaly or potential error, the value is subject to be excluded from the background data set and possibly re-sampled to confirm or disconfirm the anomalous result.

Appendix A

Soil Boring Logs and Well Construction Diagrams

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-1D**
Sheet 1 of 3

| | | |
|---|---|---|
| Date(s) Drilled 9/15/16 to 9/19/2016 | Logged By Ron Friend | Checked By M Hawrylak |
| Drilling Method Sonic | Drill Bit Size/Type Sonic 6" | Total Depth of Borehole 80.0 ft |
| Drill Rig Type Mini Sonic | Drilling Contractor Cascade Drilling | Surface Elevation 579.7 ft msl |
| Borehole Backfill Monitoring Well | Sampling Method(s) Sonic Core Barrel - 4" | Top of Casing Elevation 582.60 ft msl |
| Boring Location Inactive Bottom Ash Basin | Groundwater Level(s) Artesian (flowing) [Measurement after development] | |

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:25:06 PM

| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | Well Diagram | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|-------------|---|--------------|-----------------------------|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 0 | | | | | | Dark gray, well graded SAND (SW); loose; some gravel, trace silt and clay; fill materials | | Steel Pro-Casing |
| | | | | | | Black, poorly graded SAND (SP); trace gravel, little silt and clay, moist (Ash) | | |
| 575 | 5 | 10 | 8.5 | 85 | | Black CLAY (CH); high plasticity; little sand, trace gravel, moist | | |
| | | | | | | Gray CLAY (CL); hard; moist; little gravel | | Bentonite Grout |
| 570 | 10 | | | | | Dark gray SAND (SW); well graded; trace gravel; wet | | 2" ID schedule 40 PVC riser |
| | | | | | | Dark brown CLAY (CH); high organic/peat content; moist | | |
| 565 | 15 | 10 | 10 | 100 | | Grades with peat, small shells | | |
| | | | | | | Gray SILT (ML); some fine sand increasing with depth; wet | | |
| 560 | 20 | | | | | | | |
| 555 | 25 | 10 | 8.0 | 80 | | | | |
| 550 | 30 | | | | | | | |

Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

Log of
 MW-1D
 Sheet 2 of 3

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:25:06 PM

| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|-------------|--|--|------------------------------|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | | | | |
| | | | | | | Dark gray fine SAND (SP-SM); poorly graded; some silt; wet | | |
| 545 | 35 | 10 | 8.5 | 85 | | | | |
| | | | | | | same as above; decayed wood present | | |
| | | | | | | Gray GRAVEL (GW); rounded; well graded; trace clay; wet | | Bentonite Grout |
| 540 | 40 | | | | | | | 2" ID schedule 40 PVC riser |
| | | | | | | Gray CLAY (CL); glacial till; hard; trace fine sand; moist | | |
| 535 | 45 | 10 | 10 | 100 | | | | |
| 530 | 50 | | | | | | | |
| | | 5 | 5.0 | 100 | | | | |
| | | | | | | Highly weathered LIMESTONE-SHALE (large bedrock inclusion in till) | | |
| 525 | 55 | | | | | | | |
| | | 5 | 5.0 | 100 | | | | |
| | | | | | | Gray CLAY (CL); glacial till; very hard; trace medium sand; dry | | |
| 520 | 60 | | | | | | | |
| | | 5 | 0.5 | 10 | | | | |
| 515 | 65 | | | | | | | |

Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

**Log of
MW-1D**
 Sheet 3 of 3

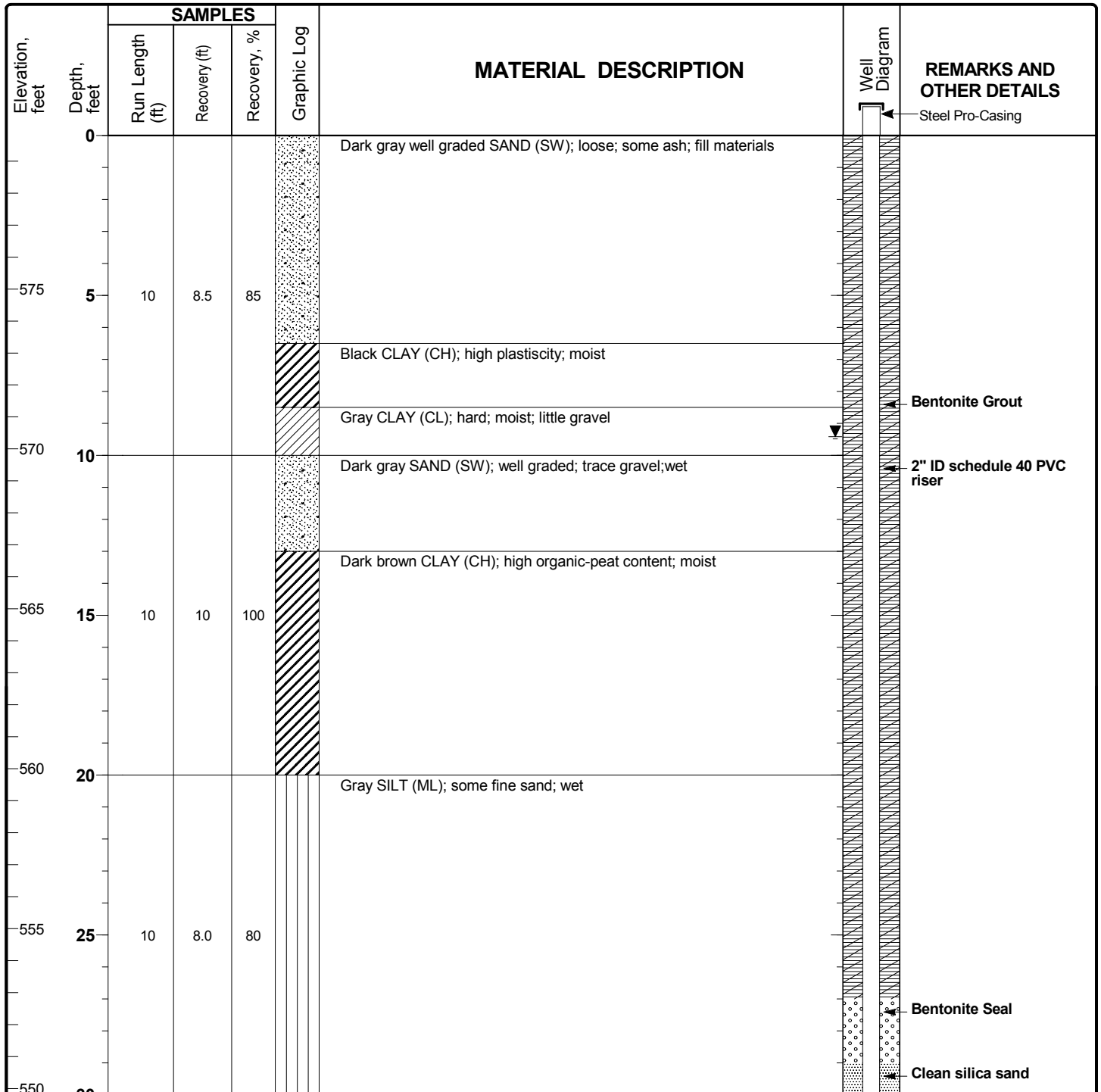
Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:25:07 PM

| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|-------------|---|--|--|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 510 | 70 | 5 | 2.75 | 55 | | | | Bentonite Grout 2" ID schedule 40 PVC riser Bentonite Seal |
| 505 | 75 | 5 | 4.0 | 80 | | LIMESTONE-SHALE (bedrock); gray, highly weathered; wet; some intact 2"-3" | | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| 500 | 80 | 5 | 5.0 | 100 | | LIMESTONE (bedrock); gray, slightly weathered; breaks apart with hammer blows; moist to wet | | Clean silica sand |
| 495 | 85 | | | | | | | End of boring |
| 490 | 90 | | | | | | | |
| 485 | 95 | | | | | | | |
| 480 | 100 | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-1S**
Sheet 1 of 2

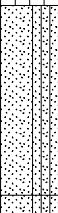
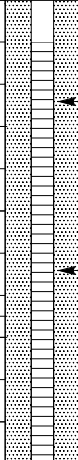

| | | |
|---|---|---|
| Date(s) Drilled 9/15/16 to 9/19/2016 | Logged By Ron Friend | Checked By M Hawrylak |
| Drilling Method Sonic | Drill Bit Size/Type Sonic 6" | Total Depth of Borehole 41.0 ft |
| Drill Rig Type Mini Sonic | Drilling Contractor Cascade Drilling | Surface Elevation 579.8 ft msl |
| Borehole Backfill Monitoring Well | Sampling Method(s) Sonic Core Barrel - 4" | Top of Casing Elevation 582.62 ft msl |
| Boring Location Inactive Bottom Ash Basin | Groundwater Level(s) 9.42' BTOC [Measurement after development] | |



Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

**Log of
MW-1S**
 Sheet 2 of 2

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:25:12 PM

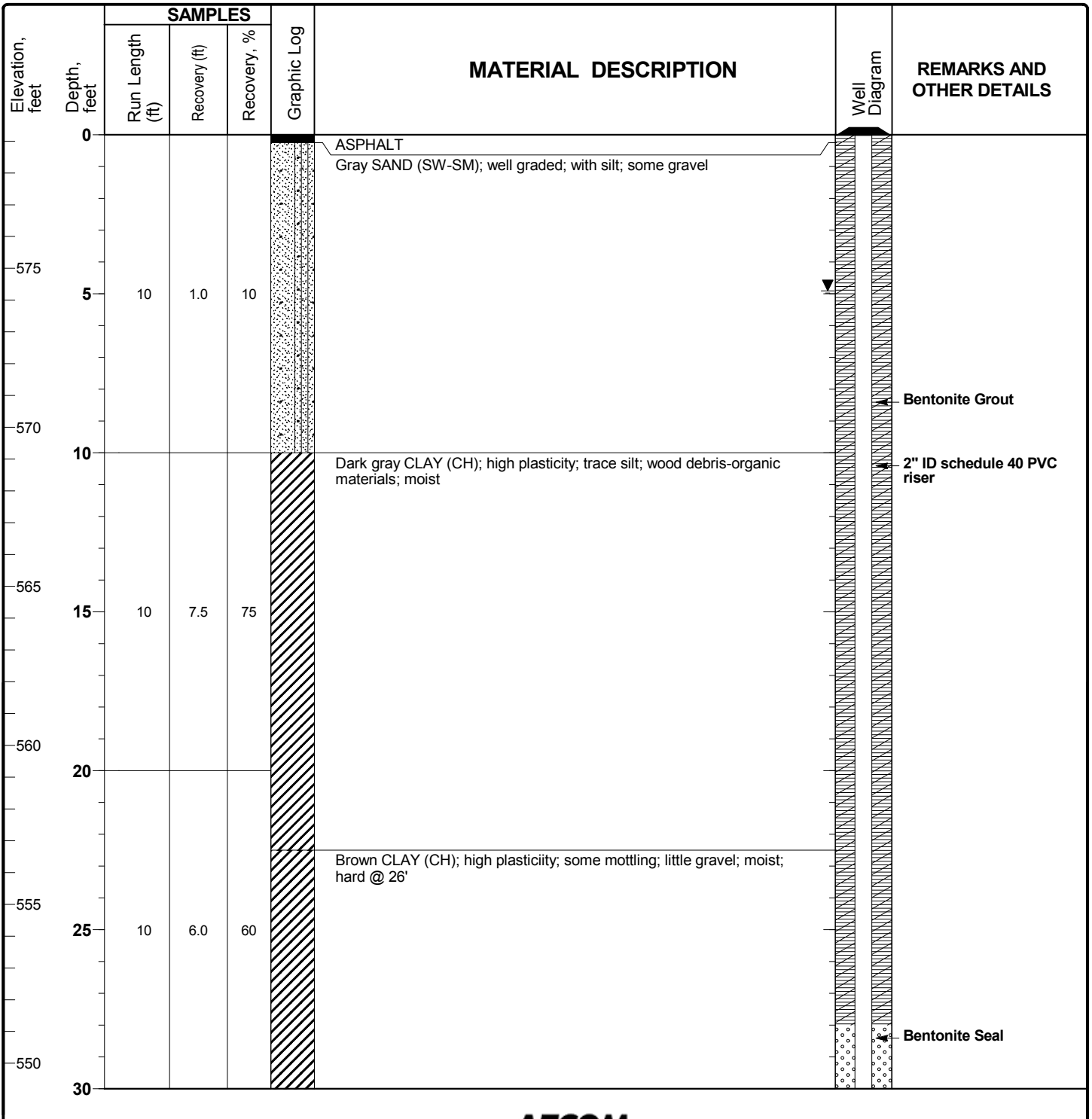
| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|---|--|---|---|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | | | | 2" ID schedule 40 PVC riser |
| | | | | | | | | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| 545 | 35 | 10 | 8.5 | 85 |  | Dark gray fine SAND (SP-SM); poorly graded; some silt; wet |  | Clean silica sand |
| 540 | 40 | | | |  | same as above; decayed wood present Gray GRAVEL (GW); rounded; well graded; trace clay; wet | | |
| 535 | 45 | 10 | 10 | 100 | | | | |
| 530 | 50 | | | | | | | |
| 525 | 55 | | | | | | | |
| 520 | 60 | | | | | | | |
| 515 | 65 | | | | | | | End of boring |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-2S**
Sheet 1 of 2

| | | |
|---|---|---|
| Date(s) Drilled 9/19/16 to 9/19/2016 | Logged By Ron Friend | Checked By M Hawrylak |
| Drilling Method Sonic | Drill Bit Size/Type Sonic 6" | Total Depth of Borehole 50.0 ft |
| Drill Rig Type Mini Sonic | Drilling Contractor Cascade Drilling | Surface Elevation 579.2 ft msl |
| Borehole Backfill Monitoring Well | Sampling Method(s) Sonic Core Barrel - 4" | Top of Casing Elevation 578.85 ft msl |
| Boring Location Inactive Bottom Ash Basin | Groundwater Level(s) 4.91' BTOC [Measurement after development] | |

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:25:17 PM



Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

**Log of
MW-2S**
 Sheet 2 of 2

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE GRANVILLE CLONE.GPJ; 10/27/2016 4:25:17 PM

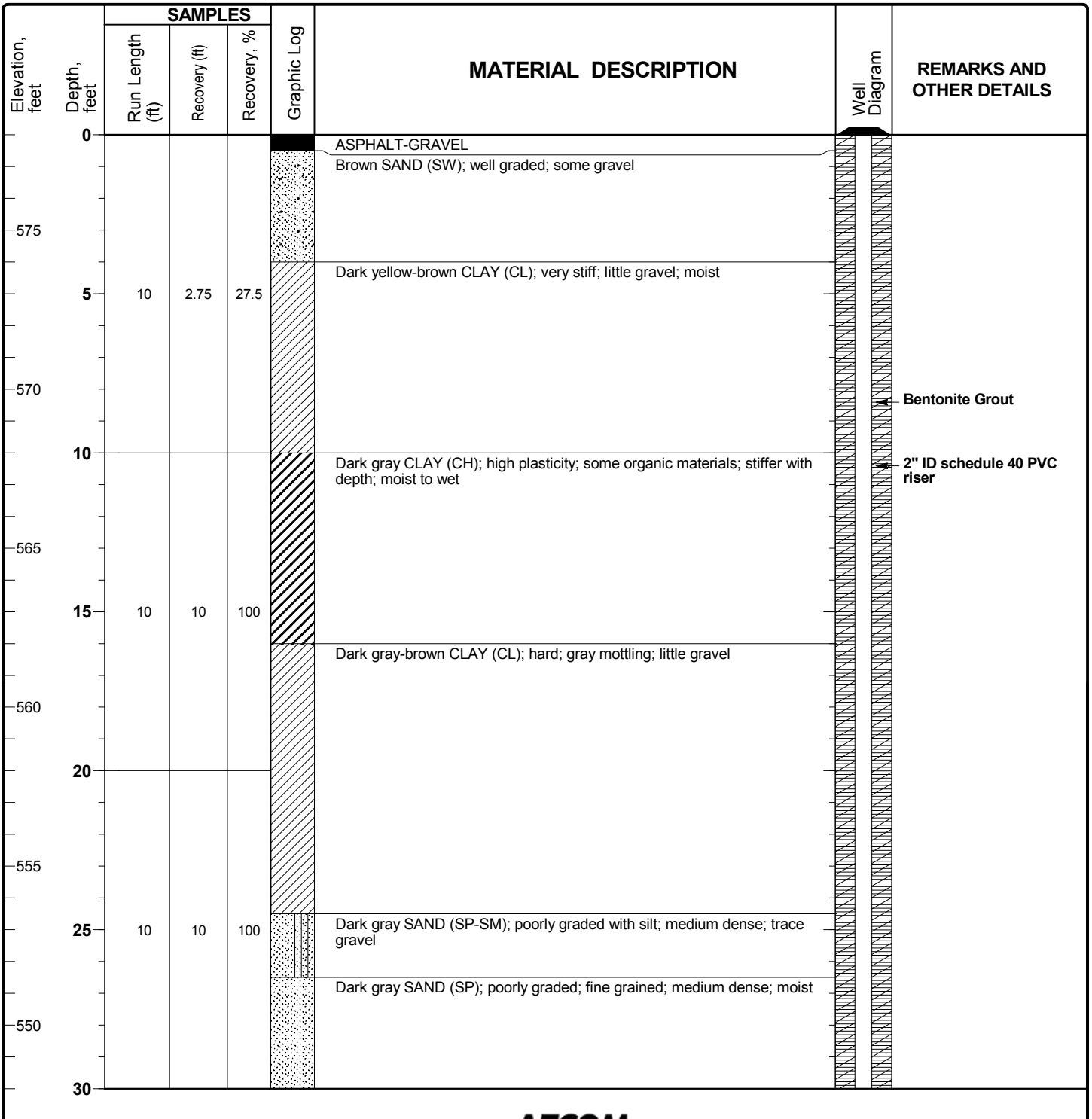
| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|-------------|---|--|---|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | | | | 2" ID schedule 40 PVC riser |
| | | | | | | Dark gray CLAY (CL); trace coarse sand; little gravel; very hard; dry-moist | | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| 545 | 35 | 10 | 8.5 | 85 | | | | Clean silica sand |
| | | | | | | Dark gray SAND (SP-SM); poorly graded; fine grained; some silt; wet | | |
| 540 | 40 | | | | | | | |
| | | | | | | Dark gray SAND (SP); poorly graded; wet | | |
| | | | | | | Dark Gray CLAY (CL); glacial till; very hard, trace gravel and sand; moist | | Bentonite Seal |
| 535 | 45 | 10 | 8.0 | 80 | | | | |
| 530 | 50 | | | | | | | |
| | | | | | | | | End of boring |
| 525 | 55 | | | | | | | |
| 520 | 60 | | | | | | | |
| 515 | 65 | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-3D**
Sheet 1 of 3

| | | |
|---|---|---|
| Date(s) Drilled 9/20/16 to 9/20/2016 | Logged By Ron Friend | Checked By M Hawrylak |
| Drilling Method Sonic | Drill Bit Size/Type Sonic 6" | Total Depth of Borehole 80.0 ft |
| Drill Rig Type Mini Sonic | Drilling Contractor Cascade Drilling | Surface Elevation 578.0 ft msl |
| Borehole Backfill Monitoring Well | Sampling Method(s) Sonic Core Barrel - 4" | Top of Casing Elevation 577.42 ft msl |
| Boring Location Inactive Bottom Ash Basin | Groundwater Level(s) Artesian (flowing) [Measurement after development] | |

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:25:25 PM



Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

**Log of
MW-3D**
 Sheet 2 of 3


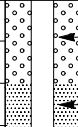



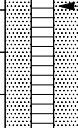


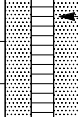



Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE_CLONE.GPJ; 10/27/2016 4:25:26 PM

| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|-------------|--|--|------------------------------|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | | | | |
| 545 | | | | | | Dark gray SAND (SP-SM); poorly graded with silt; fine grained; higher silt content at depth; wet | | |
| 35 | | 10 | 9.5 | 95 | | | | |
| 540 | | | | | | Gray SILT (ML); soft; little fine sand; wet | | Bentonite Grout |
| 40 | | | | | | Dark gray CLAY (CL); glacial till; hard; trace gravel and sand; moist | | 2" ID schedule 40 PVC riser |
| 535 | | | | | | | | |
| 45 | | 10 | 5.5 | 55 | | | | |
| 530 | | | | | | | | |
| 50 | | | | | | | | |
| 525 | | | | | | | | |
| 55 | | 10 | 5.5 | 55 | | | | |
| 520 | | | | | | | | |
| 60 | | | | | | | | |
| 515 | | 10 | 3.0 | 30 | | Gray highly weathered LIMESTONE (bedrock inclusion in till); some granite pebbles | | |
| 65 | | | | | | Dark gray CLAY (CL); glacial till; hard; trace gravel; moist | | Bentonite Seal |

Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

**Log of
MW-3D**
 Sheet 3 of 3

Report: DTE_MONROE; File J:\RESOURCE\DISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:25:27 PM

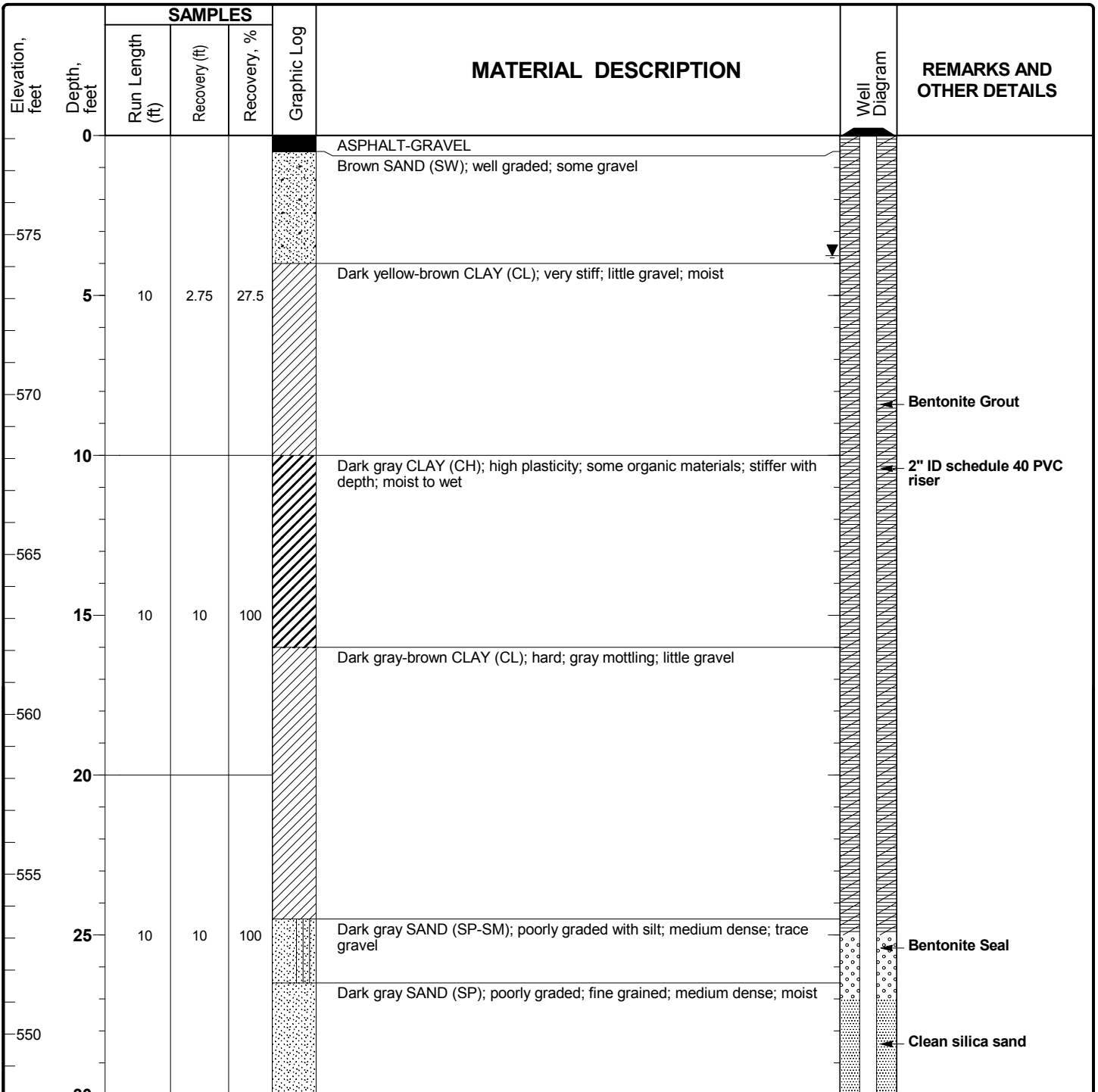
| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|---|---|---|---|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| | | 10 | 3.0 | 30 |  | |  | Bentonite Seal |
| 510 | | | | |  | Light gray SH (bedrock); highly weathered; broken from drilling process; moist to wet |  | 2" ID schedule 40 PVC riser |
| | 70 | | | |  | Gray LIMESTONE (bedrock); weathered; some intact sections (< 3" long); moist to wet |  | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| 505 | | | | |  | | | |
| | 75 | 10 | 4.0 | 40 |  | |  | Clean silica sand |
| 500 | | | | |  | | | |
| | 80 | | | |  | Pale brown LIMESTONE (bedrock); highly weathered 1-2" long pieces |  | End of boring |
| 495 | | | | | | | | |
| | 85 | | | | | | | |
| 490 | | | | | | | | |
| | 90 | | | | | | | |
| 485 | | | | | | | | |
| | 95 | | | | | | | |
| 480 | | | | | | | | |
| 100 | | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-3S**
Sheet 1 of 2

| | | |
|---|---|---|
| Date(s) Drilled 9/20/16 to 9/20/2016 | Logged By Ron Friend | Checked By M Hawrylak |
| Drilling Method Sonic | Drill Bit Size/Type Sonic 6" | Total Depth of Borehole 40.0 ft |
| Drill Rig Type Mini Sonic | Drilling Contractor Cascade Drilling | Surface Elevation 578.1 ft msl |
| Borehole Backfill Monitoring Well | Sampling Method(s) Sonic Core Barrel - 4" | Top of Casing Elevation 577.58 ft msl |
| Boring Location Inactive Bottom Ash Basin | Groundwater Level(s) 3.76' BTOC [Measurement after development] | |

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:25:31 PM



Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

Log of
MW-3S
Sheet 2 of 2

| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|-------------|---|--|--|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | | | | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| 545 | | | | | | Dark gray SAND (SP-SM); poorly graded with silt; fine grained; higher silt content at depth; wet | | 2" ID schedule 40 PVC riser |
| 35 | | 10 | 9.5 | 95 | | | | Clean silica sand |
| 540 | | | | | | Gray SILT (ML); soft; little fine sand; wet | | |
| 40 | | | | | | | | End of boring |
| 535 | | | | | | | | |
| 45 | | 10 | 5.5 | 55 | | | | |
| 530 | | | | | | | | |
| 50 | | | | | | | | |
| 525 | | | | | | | | |
| 55 | | | | | | | | |
| 520 | | | | | | | | |
| 60 | | | | | | | | |
| 515 | | | | | | | | |
| 65 | | | | | | | | |

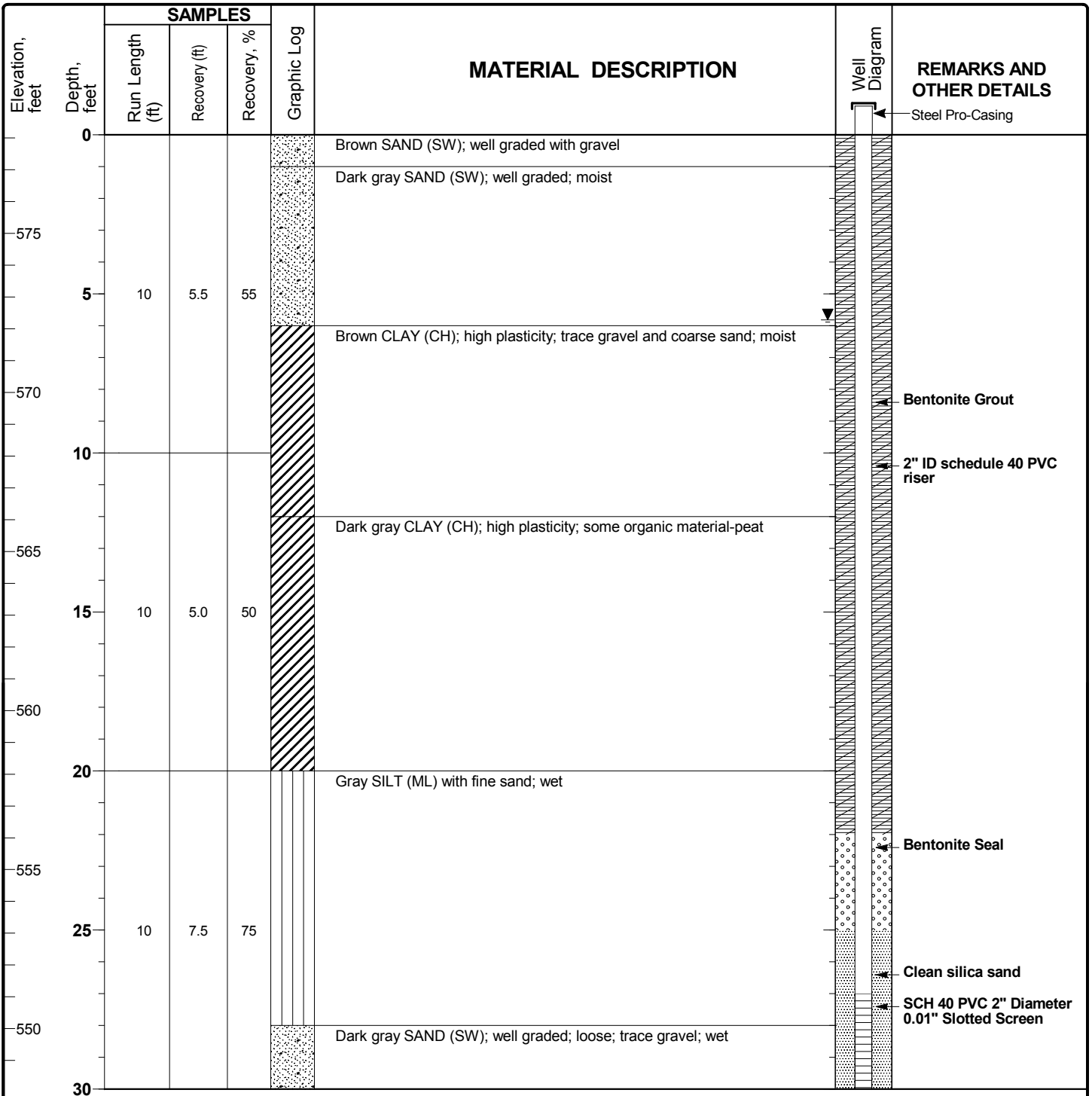
Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:25:32 PM

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-4S**
Sheet 1 of 2

| | | |
|---|---|---|
| Date(s) Drilled 9/26/16 to 9/26/2016 | Logged By Ron Friend | Checked By M Hawrylak |
| Drilling Method Sonic | Drill Bit Size/Type Sonic 6" | Total Depth of Borehole 40.0 ft |
| Drill Rig Type Mini Sonic | Drilling Contractor Cascade Drilling | Surface Elevation 578.1 ft msl |
| Borehole Backfill Monitoring Well | Sampling Method(s) Sonic Core Barrel - 4" | Top of Casing Elevation 580.67 ft msl |
| Boring Location Inactive Bottom Ash Basin | Groundwater Level(s) 5.82' BTOC [Measurement after development] | |

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE GRANVILLE CLONE.GPJ; 10/27/2016 4:25:36 PM



Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

**Log of
MW-4S**
 Sheet 2 of 2

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:25:37 PM

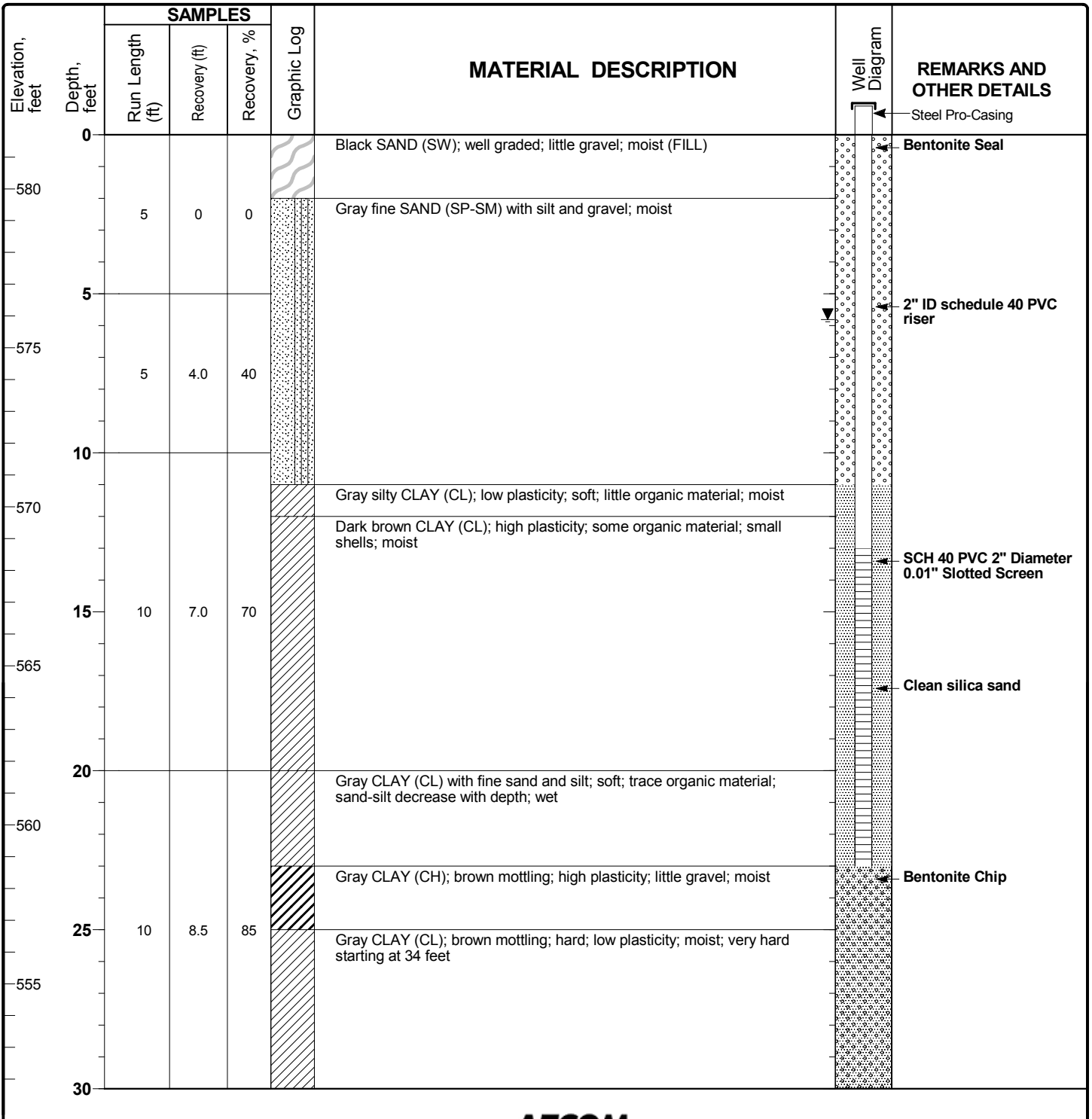
| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|-------------|--|--|---|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | | | | 2" ID schedule 40 PVC riser |
| 545 | | | | | | | | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| 35 | | 10 | 7.5 | 75 | | Dark gray SILT (ML) with fine sand; wet | | Clean silica sand |
| 540 | | | | | | Gray CLAY (CL); glacial till; hard; trace gravel and coarse sand | | Bentonite Seal |
| 40 | | | | | | | | End of boring |
| 535 | | | | | | | | |
| 45 | | | | | | | | |
| 530 | | | | | | | | |
| 50 | | | | | | | | |
| 525 | | | | | | | | |
| 55 | | | | | | | | |
| 520 | | | | | | | | |
| 60 | | | | | | | | |
| 515 | | | | | | | | |
| 65 | | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-5S**
Sheet 1 of 3

| | | |
|---|---|---|
| Date(s) Drilled 10/4/16 to 10/4/2016 | Logged By Ron Friend | Checked By M Hawrylak |
| Drilling Method Sonic | Drill Bit Size/Type Sonic 6" | Total Depth of Borehole 70.0 ft |
| Drill Rig Type Mini Sonic | Drilling Contractor Cascade Drilling | Surface Elevation 581.7 ft msl |
| Borehole Backfill Monitoring Well | Sampling Method(s) Sonic Core Barrel - 4" | Top of Casing Elevation 584.50 ft msl |
| Boring Location Inactive Bottom Ash Basin | Groundwater Level(s) 5.81' BTOC [Measurement after development] | |

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE GRANVILLE CLONE.GPJ; 10/27/2016 4:25:42 PM



Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524



Log of
MW-5S
Sheet 2 of 3

| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|-------------|---|--|------------------------------|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | | | | |
| 550 | | | | | | | | |
| 35 | | 10 | 7.5 | 75 | | | | |
| 545 | | | | | | | | |
| 40 | | | | | | | | |
| 540 | | | | | | Gray CLAY (CH); brown mottling; high plasticity; little gravel; moist | | Bentonite Chip |
| 45 | | 10 | 8.5 | 85 | | Gray CLAY (CL); glacial till; very hard; moist to dry | | |
| 535 | | | | | | | | |
| 50 | | | | | | | | |
| 530 | | | | | | | | |
| 55 | | 10 | 5.5 | 55 | | | | |
| 525 | | | | | | | | |
| 60 | | | | | | | | |
| 520 | | 5 | 4.0 | 80 | | | | |
| 65 | | | | | | | | |

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\W\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:25:43 PM

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

Log of
MW-5S
Sheet 3 of 3

| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|---|---|---|------------------------------|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 515 | | 5 | 4.0 | 80 |  | Gray LIMESTONE (bedrock); highly weathered; dry from drilling methods but zone produces water |  | Bentonite Chip |
| 70 | | | | | | | | End of boring |
| 510 | | | | | | | | |
| 75 | | | | | | | | |
| 505 | | | | | | | | |
| 80 | | | | | | | | |
| 500 | | | | | | | | |
| 85 | | | | | | | | |
| 495 | | | | | | | | |
| 90 | | | | | | | | |
| 490 | | | | | | | | |
| 95 | | | | | | | | |
| 485 | | | | | | | | |
| 100 | | | | | | | | |

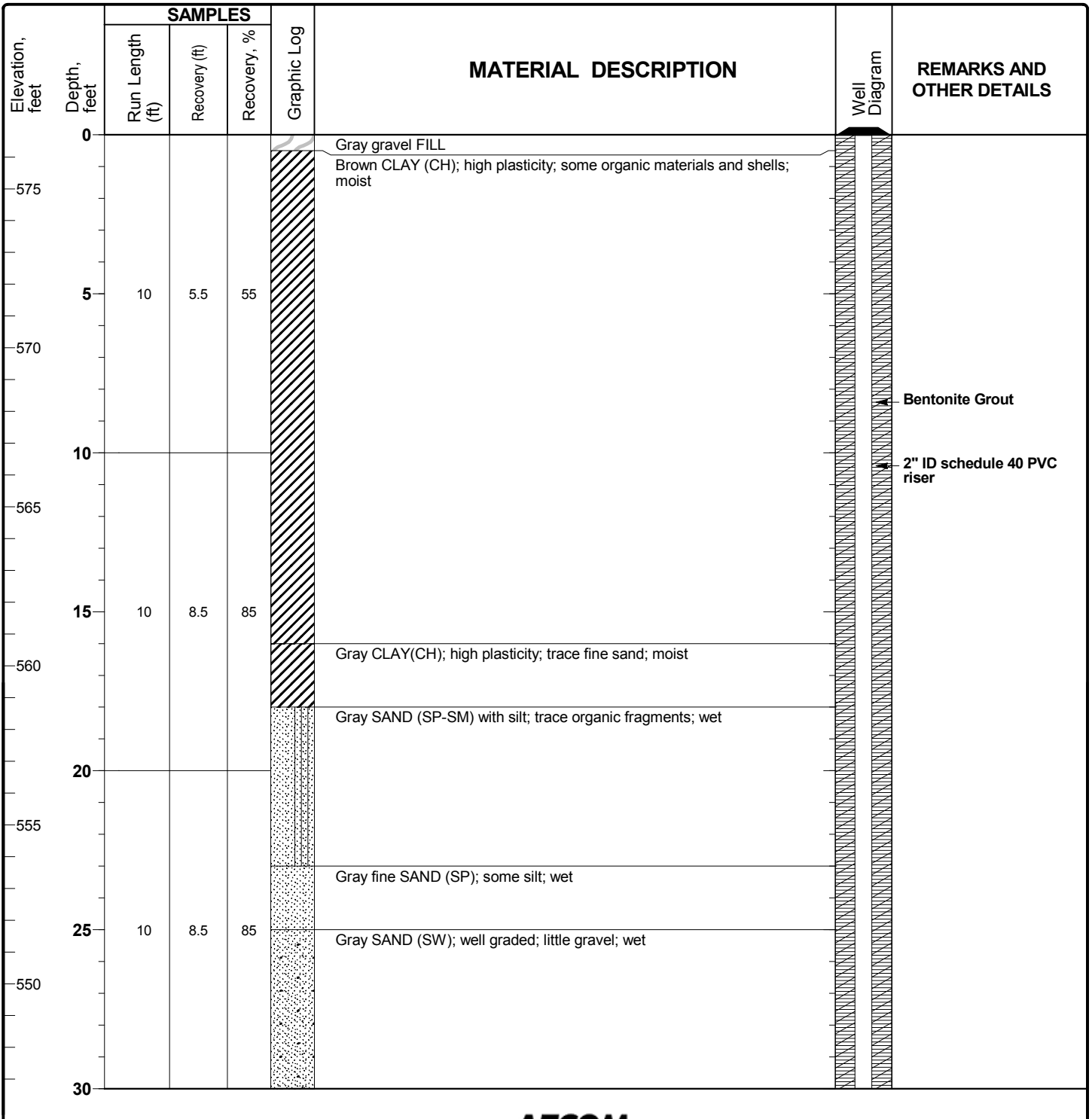
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Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-7D**
 Sheet 1 of 3

| | | | | | |
|-------------------|---------------------------|----------------------|--|-------------------------|---------------|
| Date(s) Drilled | 9/28/16 to 9/28/2016 | Logged By | Ron Friend | Checked By | M Hawrylak |
| Drilling Method | Sonic | Drill Bit Size/Type | Sonic 6" | Total Depth of Borehole | 70.0 ft |
| Drill Rig Type | Mini Sonic | Drilling Contractor | Cascade Drilling | Surface Elevation | 576.7 ft msl |
| Borehole Backfill | Monitoring Well | Sampling Method(s) | Sonic Core Barrel - 4" | Top of Casing Elevation | 576.17 ft msl |
| Boring Location | Inactive Bottom Ash Basin | Groundwater Level(s) | Artesian (flowing) [Measurement after development] | | |

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTM\MONROE GRANVILLE CLONE.GPJ; 10/27/2016 4:25:50 PM

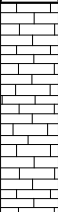
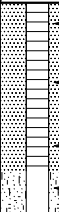


Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-7D**
Sheet 2 of 3

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:25:51 PM

| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|-------------|--|--|---|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | | | | |
| 545 | | | | | | Gray GRAVEL (GW); well graded with sand; wet | | |
| | | | | | | Gray SAND (SW); well graded; little gravel; wet | | |
| 35 | 10 | 8.5 | 85 | | | Dark gray CLAY (CL); glacial till; hard; trace gravel and coarse sand; moist | | |
| 540 | | | | | | | | |
| 40 | | | | | | | | Bentonite Grout |
| 535 | | | | | | | | 2" ID schedule 40 PVC riser |
| 45 | 10 | 9.5 | 95 | | | | | |
| 530 | | | | | | | | |
| 50 | | | | | | | | |
| 525 | | | | | | | | |
| 55 | 10 | 10 | 100 | | | Light gray LIMESTONE (bedrock); highly weathered; some larger pieces; wet | | Bentonite Seal |
| 520 | | | | | | | | |
| 60 | | | | | | Light gray LIMESTONE (bedrock); highly weathered; some intact pieces; poor recovery overall; wet | | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| 515 | 10 | 2.5 | 25 | | | | | |
| 65 | | | | | | | | Clean silica sand |

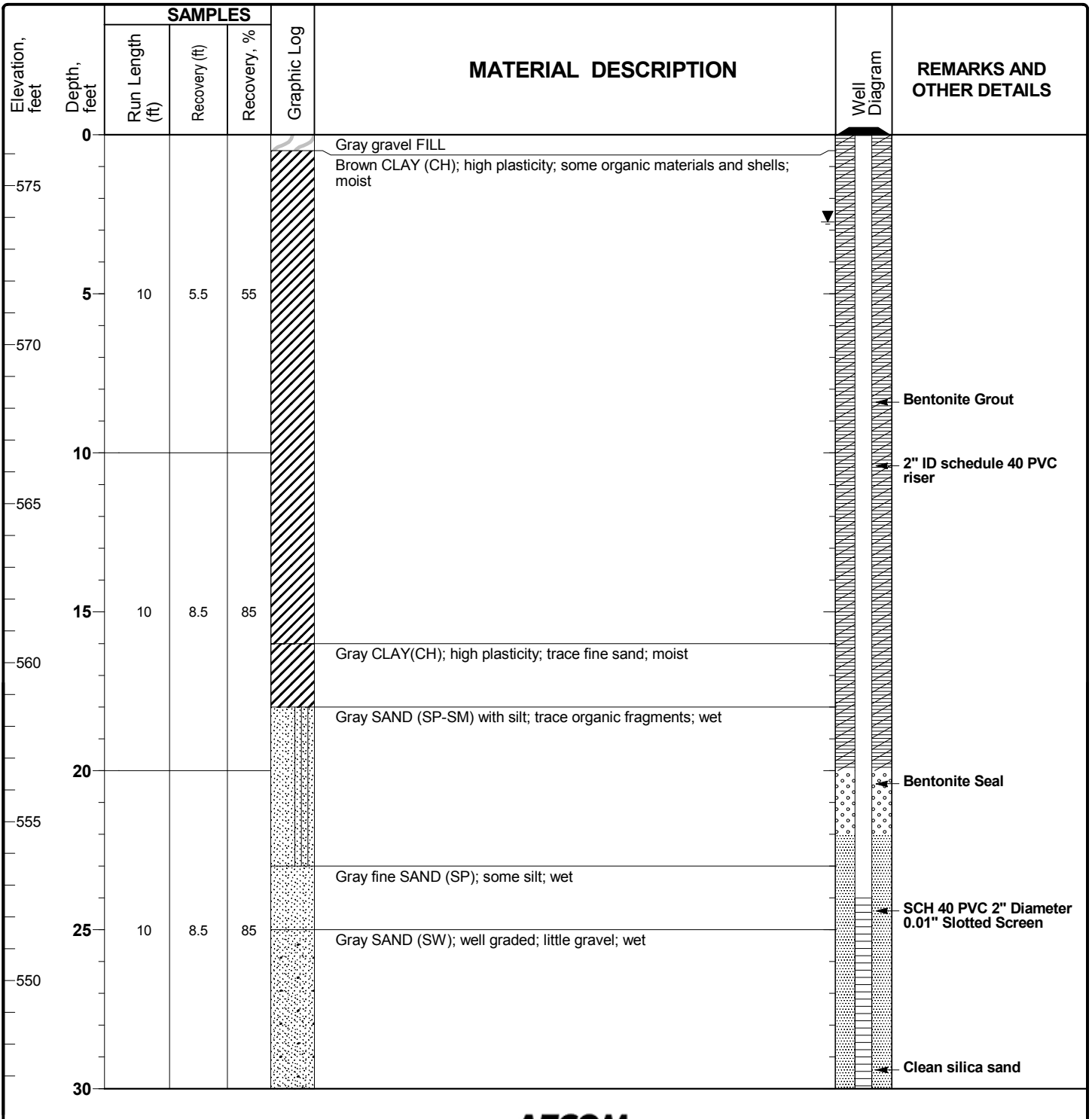
| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|---|----------------------|---|---|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 510 | | 10 | 2.5 | 25 |  | |  | 2" ID schedule 40 PVC riser SCH 40 PVC 2" Diameter 0.01" Slotted Screen Clean silica sand Collapse |
| 70 | | | | | | | | End of boring |
| 505 | | | | | | | | |
| 75 | | | | | | | | |
| 500 | | | | | | | | |
| 80 | | | | | | | | |
| 495 | | | | | | | | |
| 85 | | | | | | | | |
| 490 | | | | | | | | |
| 90 | | | | | | | | |
| 485 | | | | | | | | |
| 95 | | | | | | | | |
| 480 | | | | | | | | |
| 100 | | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-7S**
Sheet 1 of 2

| | | | | | |
|-------------------|---------------------------|----------------------|--|-------------------------|---------------|
| Date(s) Drilled | 9/28/16 to 9/28/2016 | Logged By | Ron Friend | Checked By | M Hawrylak |
| Drilling Method | Sonic | Drill Bit Size/Type | Sonic 6" | Total Depth of Borehole | 34.0 ft |
| Drill Rig Type | Mini Sonic | Drilling Contractor | Cascade Drilling | Surface Elevation | 576.6 ft msl |
| Borehole Backfill | Monitoring Well | Sampling Method(s) | Sonic Core Barrel - 4" | Top of Casing Elevation | 576.20 ft msl |
| Boring Location | Inactive Bottom Ash Basin | Groundwater Level(s) | 2.74' BTOC [Measurement after development] | | |

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE GRANVILLE CLONE.GPJ; 10/27/2016 4:25:56 PM



Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

Log of
MW-7S
Sheet 2 of 2

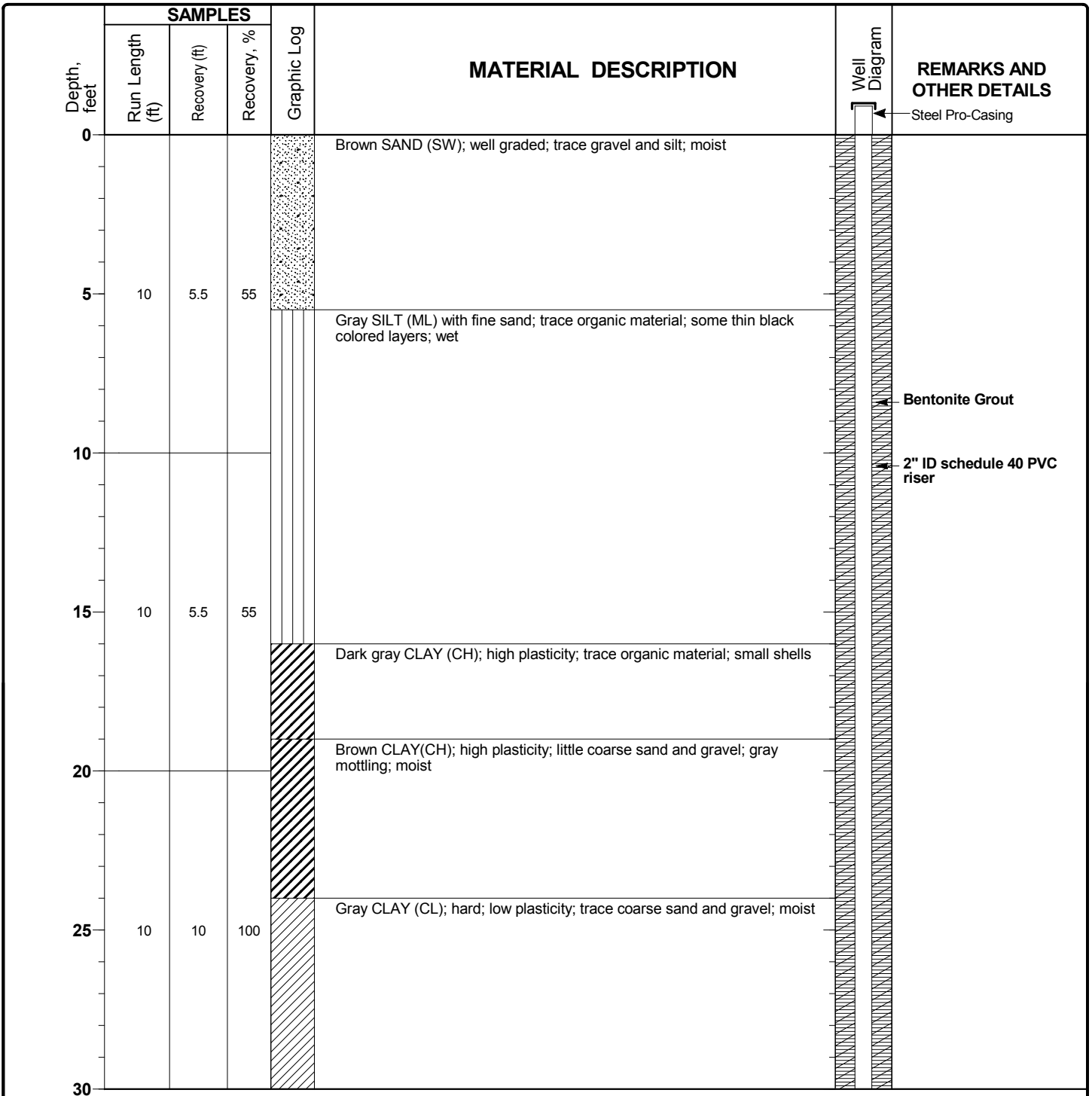
| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|-------------|---|--|---|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | | | | |
| 545 | | 4 | 3.4 | 85 | | Gray GRAVEL (GW); well graded with sand; wet | | 2" ID schedule 40 PVC riser |
| | | | | | | Gray SAND (SW); well graded; little gravel; wet | | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| | | | | | | | | Clean silica sand |
| 35 | | | | | | | | End of boring |
| 540 | | | | | | | | |
| 40 | | | | | | | | |
| 535 | | | | | | | | |
| 45 | | | | | | | | |
| 530 | | | | | | | | |
| 50 | | | | | | | | |
| 525 | | | | | | | | |
| 55 | | | | | | | | |
| 520 | | | | | | | | |
| 60 | | | | | | | | |
| 515 | | | | | | | | |
| 65 | | | | | | | | |

Report: DTE_MONROE; File J:\RESOURCE\DISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:25:57 PM

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-8D**
Sheet 1 of 3

| | | | | | |
|-------------------|----------------------|----------------------|--|-------------------------|------------|
| Date(s) Drilled | 9/29/16 to 9/30/2016 | Logged By | Ron Friend | Checked By | M Hawrylak |
| Drilling Method | Sonic | Drill Bit Size/Type | Sonic 6" | Total Depth of Borehole | 70.0 ft |
| Drill Rig Type | Mini Sonic | Drilling Contractor | Cascade Drilling | Surface Elevation | ft msl |
| Borehole Backfill | Monitoring Well | Sampling Method(s) | Sonic Core Barrel - 4" | Top of Casing Elevation | ft msl |
| Boring Location | Fly Ash Basin | Groundwater Level(s) | Artesian (flowing) [Measurement after development] | | |



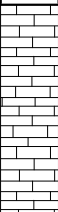
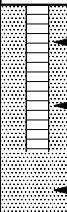
Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

Log of
 MW-8D
 Sheet 2 of 3

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|----------------|--------------------|---------------|-------------|-------------|--|--|---|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | | | |
| | 5 | 5 | 100 | | | | |
| 35 | | | | | Gray fine SAND (SP-SM) with silt; thin silt seams (<1" thick); wet | | |
| | 5 | 2.5 | 50 | | | | Bentonite Grout |
| 40 | | | | | | | 2" ID schedule 40 PVC riser |
| | | | | | Gray CLAY (CL); hard; glacial till; med-low plasticity; little gravel and coarse sand; moist | | |
| 45 | 10 | 8.0 | 80 | | | | |
| 50 | | | | | | | |
| | | | | | Light gray LIMESTONE (bedrock); highly weathered; soft; wet | | Bentonite Seal |
| 55 | 10 | 8.0 | 80 | | | | |
| | | | | | Brown LIMESTONE (bedrock); weathered; wet | | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| 60 | | | | | | | |
| | 10 | 2.0 | 20 | | | | Clean silica sand |
| 65 | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

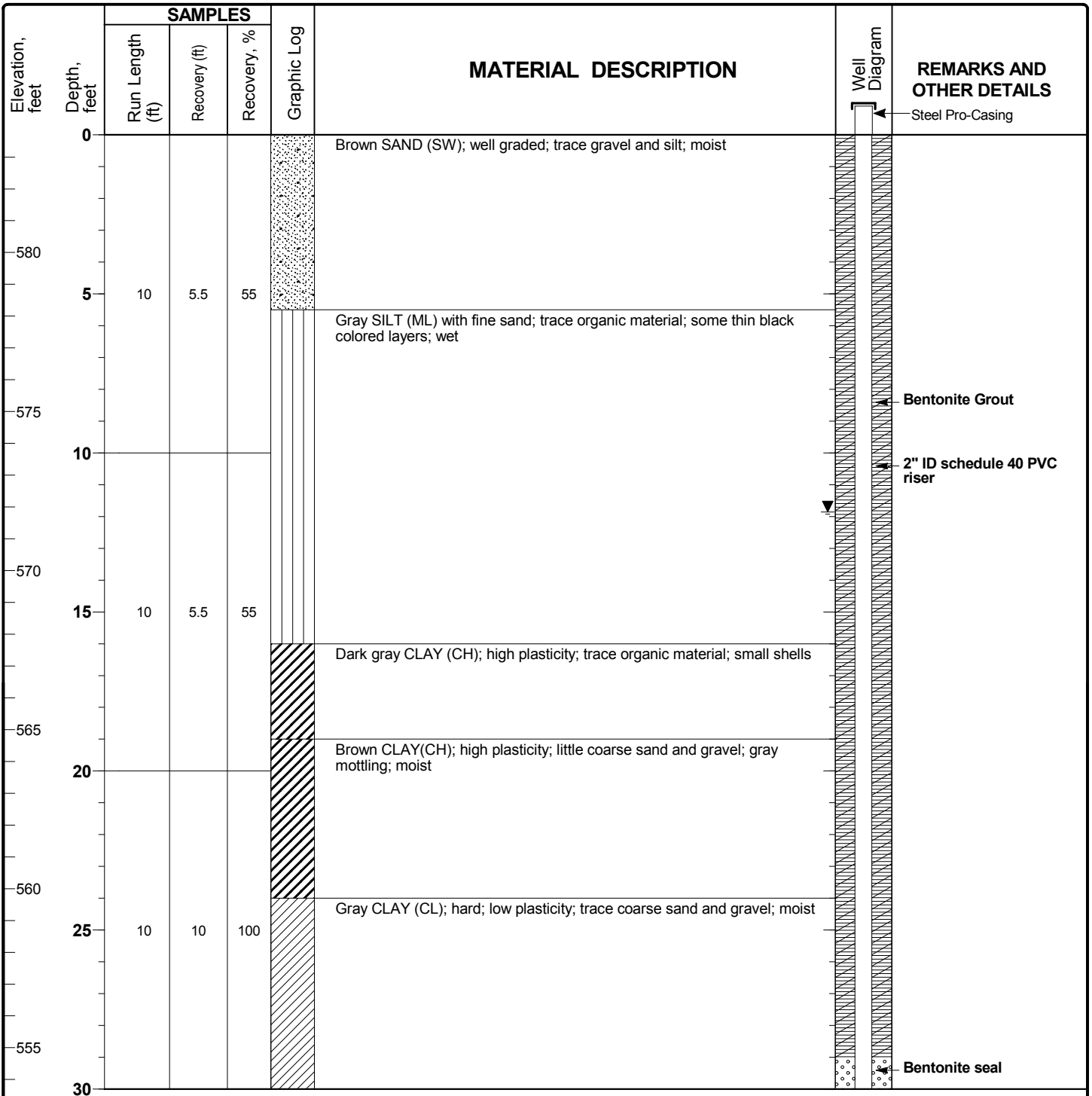
Log of
MW-8D
Sheet 3 of 3

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|----------------|--------------------|---------------|-------------|---|----------------------|---|---|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 70 | 10 | 2.0 | 20 |  | |  | 2" ID schedule 40 PVC riser SCH 40 PVC 2" Diameter 0.01" Slotted Screen Clean silica sand |
| 75 | | | | | | | End of boring |
| 80 | | | | | | | |
| 85 | | | | | | | |
| 90 | | | | | | | |
| 95 | | | | | | | |
| 100 | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-8S**
Sheet 1 of 2

| | | |
|--|--|---|
| Date(s) Drilled 9/29/16 to 9/30/2016 | Logged By Ron Friend | Checked By M Hawrylak |
| Drilling Method Sonic | Drill Bit Size/Type Sonic 6" | Total Depth of Borehole 43.0 ft |
| Drill Rig Type Mini Sonic | Drilling Contractor Cascade Drilling | Surface Elevation 583.7 ft msl |
| Borehole Backfill Monitoring Well | Sampling Method(s) Sonic Core Barrel - 4" | Top of Casing Elevation 586.59 ft msl |
| Boring Location Fly Ash Basin | Groundwater Level(s) 11.86' BTOC [Measurement after development] | |



Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

**Log of
MW-8S**
 Sheet 2 of 2

Report: DTE_MONROE; File J:\RESOURCES\DISCIPLINES\GINT\PROJECTS\DTE\MONROE_GRANVILLE CLONE.GPJ; 10/27/2016 4:26:12 PM

| Elevation, feet | Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|--------------------|----------------|--------------------|---------------|-------------|-------------|--|--|---|
| | | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | | | | Bentonite seal |
| | | 5 | 5 | 100 | | | | 2" ID schedule 40 PVC riser |
| 550 | | | | | | | | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| 35 | | | | | | Gray fine SAND (SP-SM) with silt; thin silt seams (<1" thick); wet | | |
| | | 5 | 2.5 | 50 | | | | Clean silica sand |
| 545 | | | | | | | | |
| 40 | | | | | | | | |
| | | 3 | 2.4 | 80 | | | | |
| 540 | | | | | | | | End of boring |
| 45 | | | | | | | | |
| | | | | | | | | |
| 535 | | | | | | | | |
| 50 | | | | | | | | |
| | | | | | | | | |
| 530 | | | | | | | | |
| 55 | | | | | | | | |
| | | | | | | | | |
| 525 | | | | | | | | |
| 60 | | | | | | | | |
| | | | | | | | | |
| 520 | | | | | | | | |
| 65 | | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524






**Log of
MW-9**
Sheet 1 of 2

| | | | | | |
|-------------------|---------------------------|----------------------|------------------------|-------------------------|------------|
| Date(s) Drilled | 9/19/17 to 9/19/2017 | Logged By | Ron Friend | Checked By | B Finnigan |
| Drilling Method | Sonic | Drill Bit Size/Type | Sonic 6" | Total Depth of Borehole | 40.0 ft |
| Drill Rig Type | Sonic | Drilling Contractor | Cascade Drilling | Surface Elevation | ft msl |
| Borehole Backfill | Monitoring Well | Sampling Method(s) | Sonic Core Barrel - 4" | Top of Casing Elevation | ft msl |
| Boring Location | Inactive Bottom Ash Basin | Groundwater Level(s) | | | |

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | Well Diagram | REMARKS AND OTHER DETAILS |
|-------------|-----------------|---------------|-------------|-------------|---|--------------|---------------------------|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 0 | | | | | Gravelly FILL, dry | | |
| | 2 | 1.5 | 50% | | | | |
| 5 | | | | | Very dark gray high plasticity CLAY with some organics and shell fragments, soft, moist | | |
| | 5 | 5 | 100% | | grades with fine sand | | |
| 10 | | | | | grades with some organic material | | |
| 15 | 10 | 8 | 80% | | grades with more organics | | |
| | | | | | Dark brown clayey PEAT, soft | | |
| 20 | | | | | Gray high plasticity CLAY, soft, moist | | |
| | | | | | Gray sandy SILT, soft, wet | | |
| | | | | | grades with less sand with depth | | |
| 25 | 10 | 9 | 90% | | Gray fine SAND with silt, loose, wet | | |
| | | | | | grades with less silt with depth | | |
| | | | | | Gray poorly graded SAND with trace fines, loose, wet | | |
| 30 | | | | | Gray poorly graded GRAVEL with sand, loose, wet | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-9**
 Sheet 2 of 2

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|----------------|--------------------|---------------|-------------|---|---|---|------------------------------|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | |  | Gray fine grained SAND with trace fines, wet |  | Clean silica sand |
| 35 | 10 | 10 | 100% |  | Gray low plasticity CLAY with little gravel, hard, moist [TILL] |  | Natural Collapse |
| 40 | | | |  | Terminate boring at 40 feet BGS. | | End of boring |
| 45 | | | | | | | |
| 50 | | | | | | | |
| 55 | | | | | | | |
| 60 | | | | | | | |
| 65 | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524



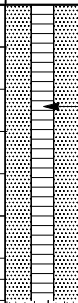





**Log of
MW-10**
Sheet 1 of 2

| | | | | | |
|-------------------|---------------------------|----------------------|------------------------|-------------------------|------------|
| Date(s) Drilled | 9/20/17 to 9/20/2017 | Logged By | Ron Friend | Checked By | B Finnigan |
| Drilling Method | Sonic | Drill Bit Size/Type | Sonic 6" | Total Depth of Borehole | 40.0 ft |
| Drill Rig Type | Sonic | Drilling Contractor | Cascade Drilling | Surface Elevation | ft msl |
| Borehole Backfill | Monitoring Well | Sampling Method(s) | Sonic Core Barrel - 4" | Top of Casing Elevation | ft msl |
| Boring Location | Inactive Bottom Ash Basin | Groundwater Level(s) | | | |

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | Well Diagram | REMARKS AND OTHER DETAILS |
|-------------|-----------------|---------------|-------------|-------------|---|--------------|-----------------------------|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 0 | | | | | Grey poorly graded GRAVEL with clay and sand, dry | | |
| 5 | 5 | 3 | 60% | | Dark yellowish brown low plasticity CLAY with sand, moist grades with gravel grades with less gravel with depth | | |
| 10 | 5 | 3 | 60% | | Very dark grey high plasticity CLAY with trace shell fragments, moist grades with fine sand | | Bentonite Grout |
| 15 | 10 | 9.5 | 95% | | grades with small sand seams, fine sand, 1-2 inches apart grades with little organic material | | 2" ID schedule 40 PVC riser |
| 20 | | | | | | | |
| 25 | 10 | 9.5 | 95% | | Grey silty CLAY with fine sand, very soft, wet Grey high plasticity CLAY, moist | | Bentonite Seal |
| 30 | | | | | | | |

Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

Log of
 MW-10
 Sheet 2 of 2

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|----------------|--------------------|---------------|-------------|---|--|--|---|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | |  | | | |
| | | | |  | Dark grey poorly graded SAND with gravel, wet | |  SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| | | | |  | Dark grey well graded SAND, with little silt, fine grained sand, wet | | |
| 35 | 10 | 9.5 | 95% |  | grades with increasing silt with depth | | |
| | | | |  | Grey SILT with little gravel, hard, moist | | |
| | | | |  | Grey CLAY with little gravel, hard, moist | |  Natural Collapse |
| 40 | | | | | Terminate boring at 40 feet BGS. | | |
| 45 | | | | | | | End of boring |
| 50 | | | | | | | |
| 55 | | | | | | | |
| 60 | | | | | | | |
| 65 | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-11**
Sheet 1 of 2

| | | |
|---|---|---|
| Date(s) Drilled 9/20/17 to 9/20/2017 | Logged By Ron Friend | Checked By B Finnigan |
| Drilling Method Sonic | Drill Bit Size/Type Sonic 6" | Total Depth of Borehole 45.0 ft |
| Drill Rig Type Sonic | Drilling Contractor Cascade Drilling | Surface Elevation ft msl |
| Borehole Backfill Monitoring Well | Sampling Method(s) Sonic Core Barrel - 4" | Top of Casing Elevation ft msl |
| Boring Location Inactive Bottom Ash Basin | Groundwater Level(s) | |

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | Well Diagram | REMARKS AND OTHER DETAILS |
|-------------|-----------------|---------------|-------------|-------------|---|--------------|-----------------------------|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 0 | | | | | Pale brown poorly graded SAND (fill), loose, dry | | |
| | 5 | 2 | 40% | | | | |
| 5 | | | | | grades with gravel | | |
| | 5 | 4 | 80% | | | | |
| 10 | | | | | Dark brown high plasticity CLAY with grey mottling, very stiff, moist | | Bentonite Grout |
| | 5 | 5 | 100% | | Very dark grey high plasticity CLAY with little organics and trace shell fragments, soft, moist | | 2" ID schedule 40 PVC riser |
| 15 | | | | | Grey low plasticity CLAY with brown mottling, little coarse sand and gravel, hard, moist | | |
| | 5 | 5 | 100% | | grades to brown | | |
| 20 | | | | | | | |
| | 5 | 5 | 100% | | grades to grey | | |
| 25 | | | | | Grey CLAY with little gravel and coarse sand, hard, moist-dry | | Bentonite Seal |
| | 5 | 5 | 100% | | | | |
| 30 | | | | | Grey SILT with fine sand, stiff, moist - wet, slow dilatancy | | |

Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

Log of
 MW-11
 Sheet 2 of 2

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|----------------|--------------------|---------------|-------------|-------------|---|--|--|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | | | |
| 35 | 10 | 10 | 100% | | | | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| 40 | 5 | 5 | 100% | | Grey CLAY (till) with little gravel and coarse sand, hard, friable, moist - dry | | Natural Collapse |
| 45 | | | | | Terminate boring at 45 feet BGS. | | End of boring |
| 50 | | | | | | | |
| 55 | | | | | | | |
| 60 | | | | | | | |
| 65 | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-12**
Sheet 1 of 2

| | | |
|---|---|---|
| Date(s) Drilled 9/21/17 to 9/21/2017 | Logged By Ron Friend | Checked By B Finnigan |
| Drilling Method Sonic | Drill Bit Size/Type Sonic 6" | Total Depth of Borehole 50.0 ft |
| Drill Rig Type Sonic | Drilling Contractor Cascade Drilling | Surface Elevation ft msl |
| Borehole Backfill Monitoring Well | Sampling Method(s) Sonic Core Barrel - 4" | Top of Casing Elevation ft msl |
| Boring Location Inactive Bottom Ash Basin | Groundwater Level(s) | |

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | Well Diagram | REMARKS AND OTHER DETAILS |
|-------------|-----------------|---------------|-------------|-------------|---|--------------|-----------------------------|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 0 | | | | | Pale brown well graded SAND with fined grained sand and trace gravel, dry | | |
| | 5 | 2 | 40% | | moist | | |
| 5 | | | | | grades with trace shell fragments | | |
| | 5 | 3 | 60% | | Dark grey SAND with silt, moist | | Bentonite Grout |
| 10 | | | | | wet | | |
| | | | | | Very dark grey high plasticity CLAY with little organics and trace shell fragments, soft, moist | | 2" ID schedule 40 PVC riser |
| 15 | 10 | 8 | 80% | | Gray high plasticity CLAY with trace coarse sand and gravel, stiff, moist | | |
| | | | | | grades with color change to brown, hard, some grey mottling | | |
| 20 | | | | | | | |
| | 10 | 10 | 100% | | Very dark grey low plasticity CLAY | | Bentonite Seal |
| 25 | | | | | | | |
| 30 | | | | | | | |

Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

Log of
 MW-12
 Sheet 2 of 2

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | REMARKS AND OTHER DETAILS |
|----------------|--------------------|---------------|-------------|-------------|---|--|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | |
| 30 | | | | | Dark grey fine SILT with sand, stiff, wet-moist | |
| 35 | 10 | 9 | 90% | | Dark grey fine SAND with silt, stiff, wet-moist | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| 40 | | | | | | |
| 45 | 10 | 10 | 100% | | Dark grey low plasticity CLAY (till) with little gravel and coarse sand, hard, moist | Natural Collapse |
| 50 | | | | | | End of boring |
| 55 | | | | | | |
| 60 | | | | | | |
| 65 | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-13**
Sheet 1 of 2

| | | |
|---|---|---|
| Date(s) Drilled 9/21/17 to 9/21/2017 | Logged By Ron Friend | Checked By B Finnigan |
| Drilling Method Sonic | Drill Bit Size/Type Sonic 6" | Total Depth of Borehole 43.5 ft |
| Drill Rig Type Sonic | Drilling Contractor Cascade Drilling | Surface Elevation ft msl |
| Borehole Backfill Monitoring Well | Sampling Method(s) Sonic Core Barrel - 4" | Top of Casing Elevation ft msl |
| Boring Location Inactive Bottom Ash Basin | Groundwater Level(s) | |

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | Well Diagram | REMARKS AND OTHER DETAILS |
|-------------|-----------------|---------------|-------------|-------------|--|--------------|---|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 0 | | | | | Brown poorly graded SAND with gravel, dry | | |
| 5 | 5 | 4 | 80% | | moist | | |
| 5 | | | | | wet | | |
| 5 | 5 | 4.5 | 90% | | woody debris | | |
| 10 | | | | | Black high plasticity CLAY with little organics, soft, moist | | Bentonite Grout |
| 10 | | | | | Dark grey fine SAND with silt, wet, silt increasing with depth | | |
| 10 | | | | | Dark grey SILT with fine sand, wet | | |
| 15 | 10 | 6 | 60% | | Black high plasticity CLAY with little organic material, soft, moist | | 2" ID schedule 40 PVC riser |
| 20 | | | | | Dark grey low plasticity CLAY with silt and little fine sand, very soft, wet | | Bentonite Seal |
| 25 | 15 | 11 | 73% | | | | |
| 30 | | | | | Dark grey fine SILT with sand, very soft, wet, sand increasing with depth | | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| 30 | | | | | woody debris | | |

Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

Log of
 MW-13
 Sheet 2 of 2

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | REMARKS AND OTHER DETAILS |
|----------------|--------------------|---------------|-------------|-------------|---|------------------------------|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | |
| 30 | | | | | Dark grey fine SAND with silt, wet | |
| | 15 | 11 | 73% | | | |
| 35 | | | | | Dark grey low plasticity CLAY with little gravel and coarse sand, hard, moist | |
| | 8.5 | 8.5 | 100% | | | Natural Collapse |
| 40 | | | | | | |
| 45 | | | | | Terminate boring at 43.5 feet BGS. | End of boring |
| 50 | | | | | | |
| 55 | | | | | | |
| 60 | | | | | | |
| 65 | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-14**
Sheet 1 of 2

| | | |
|---|---|---|
| Date(s) Drilled 9/22/17 to 9/22/2017 | Logged By Ron Friend | Checked By B Finnigan |
| Drilling Method Sonic | Drill Bit Size/Type Sonic 6" | Total Depth of Borehole 40.0 ft |
| Drill Rig Type Sonic | Drilling Contractor Cascade Drilling | Surface Elevation ft msl |
| Borehole Backfill Monitoring Well | Sampling Method(s) Sonic Core Barrel - 4" | Top of Casing Elevation ft msl |
| Boring Location Inactive Bottom Ash Basin | Groundwater Level(s) | |

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | Well Diagram | REMARKS AND OTHER DETAILS |
|-------------|-----------------|---------------|-------------|-------------|---|--------------|-----------------------------|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 0 | | | | | Black coal ash FILL with gravel and coal, loose, dry | | |
| 5 | 5 | 4 | 80% | | Black coal ash FILL, coarse, sand-gravel sized, moist | | |
| 10 | 5 | 4.5 | 90% | | Very dark grey high plasticity CLAY, soft, moist | | Bentonite Grout |
| 15 | 10 | 6 | 60% | | grades to coarse sand and gravel, wet | | 2" ID schedule 40 PVC riser |
| 20 | | | | | Very dark grey fine SAND with silt and clay, wet | | |
| 25 | 10 | 0 | 0% | | No recovery | | Bentonite Seal |
| 30 | | | | | | | |

Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

**Log of
MW-14**
 Sheet 2 of 2

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|----------------|--------------------|---------------|-------------|-------------|---|--|--|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | Dark grey CLAY with sand, fine-medium grained sand. wet | | |
| | | | | | Dark grey poorly graded SAND with gravel, coarse grained sand, wet | | |
| 35 | 10 | 9 | 90% | | | | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| | | | | | Dark grey CLAY (till) with little coarse sand and gravel, hard, moist | | |
| 40 | | | | | Terminate boring at 40 feet BGS. | | End of boring |
| 45 | | | | | | | |
| 50 | | | | | | | |
| 55 | | | | | | | |
| 60 | | | | | | | |
| 65 | | | | | | | |

Project: DTE Monroe Plant
Project Location: Monroe, Michigan
Project Number: 60489524

**Log of
MW-15**
Sheet 1 of 2

| | | | | | |
|-------------------|---------------------------|----------------------|------------------------|-------------------------|------------|
| Date(s) Drilled | 9/26/17 to 9/26/2017 | Logged By | Ron Friend | Checked By | B Finnigan |
| Drilling Method | Sonic | Drill Bit Size/Type | Sonic 6" | Total Depth of Borehole | 45.0 ft |
| Drill Rig Type | Sonic | Drilling Contractor | Cascade Drilling | Surface Elevation | ft msl |
| Borehole Backfill | Monitoring Well | Sampling Method(s) | Sonic Core Barrel - 4" | Top of Casing Elevation | ft msl |
| Boring Location | Inactive Bottom Ash Basin | Groundwater Level(s) | | | |

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | Well Diagram | REMARKS AND OTHER DETAILS |
|-------------|-----------------|---------------|-------------|-------------|--|--------------|---------------------------|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 0 | | | | | Greyish brown gravelly FILL with silt and clay, dry | | |
| | 4 | 4 | 100% | | moist | | |
| 5 | | | | | Dark grey medium plasticity CLAY with little shell fragments, soft, moist | | |
| | 5 | 4.5 | 90% | | | | |
| 10 | | | | | grades with little organic material | | |
| 15 | 10 | 9 | 90% | | grades with color change to very dark organic brown | | |
| 20 | | | | | Grey high plasticity CLAY with trace coarse sand, soft, moist-wet | | |
| 25 | 10 | 8.5 | 85% | | Grey low plasticity CLAY with little silt, soft, moist-wet, silt increasing with depth | | |
| | | | | | Grey SILT with fine sand, wet | | |
| 30 | | | | | Grey SAND with silt, wet | | |

Project: DTE Monroe Plant
 Project Location: Monroe, Michigan
 Project Number: 60489524

Log of
 MW-15
 Sheet 2 of 2

| Depth, feet | SAMPLES | | | Graphic Log | MATERIAL DESCRIPTION | | REMARKS AND OTHER DETAILS |
|----------------|--------------------|---------------|-------------|-------------|---|--|--|
| | Run Length (ft) | Recovery (ft) | Recovery, % | | | | |
| 30 | | | | | Grey well graded SAND, fine to coarse grained, wet | | SCH 40 PVC 2" Diameter 0.01" Slotted Screen |
| 35 | 10 | 10 | 100% | | grades with gravel | | |
| | | | | | Grey well graded GRAVEL with coarse sand, wet | | |
| | | | | | Grey well graded SAND with gravel, wet | | |
| 40 | | | | | Grey low plasticity CLAY (till) with little coarse sand and gravel, hard, moist | | Natural Collapse |
| | | | | | | | End of boring |
| 45 | | | | | Terminate boring at 45 feet BGS. | | |
| 50 | | | | | | | |
| 55 | | | | | | | |
| 60 | | | | | | | |
| 65 | | | | | | | |

Appendix B

Example Field Forms



PROJECT NAME: _____

PROJECT NUMBER: _____

PROJECT MANAGER: _____

SITE LOCATION: _____

DATES OF FIELDWORK: _____

PURPOSE OF FIELDWORK: _____

WORK PERFORMED BY: _____

SIGNED

DATE

CHECKED BY

DATE



GENERAL NOTES

| | | |
|-----------------|---------|---------------|
| PROJECT NAME: | DATE: | TIME ARRIVED: |
| PROJECT NUMBER: | AUTHOR: | TIME LEFT: |

| WEATHER | | |
|---------------------------|-----------------|-------------------|
| TEMPERATURE: _____ °F | WIND: _____ MPH | VISIBILITY: _____ |
| WORK / SAMPLING PERFORMED | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |

| PROBLEMS ENCOUNTERED | CORRECTIVE ACTION TAKEN |
|----------------------|-------------------------|
| | |
| | |
| | |
| | |

| COMMUNICATION | | |
|---------------|--------------|--------------------|
| NAME | REPRESENTING | SUBJECT / COMMENTS |
| | | |
| | | |
| | | |
| | | |

| INVESTIGATION DERIVED WASTE SUMMARY | | |
|-------------------------------------|----------|----------|
| WASTE MATRIX | QUANTITY | COMMENTS |
| | | |
| | | |
| | | |
| | | |

SIGNED _____ DATE _____

CHECKED BY _____ DATE _____



EQUIPMENT SUMMARY

| | |
|---------------|---------------|
| PROJECT NAME: | SAMPLER NAME: |
| PROJECT NO.: | |

WATER LEVEL MEASUREMENTS COLLECTED WITH:

NAME AND MODEL OF INSTRUMENT

SERIAL NUMBER (IF APPLICABLE)

PRODUCT LEVEL MEASUREMENTS COLLECTED WITH:

NA

NAME AND MODEL OF INSTRUMENT

SERIAL NUMBER (IF APPLICABLE)

DEPTH TO BOTTOM OF WELL MEASUREMENTS COLLECTED WITH:

NA

NAME AND MODEL OF INSTRUMENT

SERIAL NUMBER (IF APPLICABLE)

PURGING METHOD

NAME AND MODEL OF PUMP OR TYPE OF BAILER

SERIAL NUMBER (IF APPLICABLE)

SAMPLING METHOD

NAME AND MODEL OF PUMP OR TYPE OF BAILER

SERIAL NUMBER (IF APPLICABLE)

NAME AND MODEL OF FILTRATION DEVICE

FILTER TYPE AND SIZE

☐ LOW-FLOW SAMPLING EVENT

TUBING TYPE

PURGE WATER DISPOSAL METHOD

☐ GROUND

☐ DRUM

☐ POTW

☐ POLYTANK

☐ OTHER _____

DECONTAMINATION AND FIELD BLANK WATER SOURCE

STORE BOUGHT

STORE BOUGHT

POTABLE WATER SOURCE

DI WATER SOURCE

SIGNED

DATE

CHECKED BY

DATE



WATER QUALITY METER CALIBRATION LOG

| | | |
|---------------|-----------|----------|
| PROJECT NAME: | MODEL: | SAMPLER: |
| PROJECT NO.: | SERIAL #: | DATE: |

PH CALIBRATION CHECK

| pH 7 (LOT #): (EXP. DATE): | pH 4 / 10 (LOT #): (EXP. DATE): | CAL. RANGE | TIME |
|----------------------------------|---------------------------------------|---------------------------------------|------|
| POST-CAL. READING / STANDARD | POST-CAL. READING / STANDARD | | |
| / | / | <input type="checkbox"/> WITHIN RANGE | |
| / | / | <input type="checkbox"/> WITHIN RANGE | |
| / | / | <input type="checkbox"/> WITHIN RANGE | |
| / | / | <input type="checkbox"/> WITHIN RANGE | |

SPECIFIC CONDUCTIVITY CALIBRATION CHECK

| CAL. READING (LOT #): (EXP. DATE): | TEMPERATURE (°CELSIUS) | CAL. RANGE | TIME |
|--|---------------------------|---------------------------------------|------|
| POST-CAL. READING / STANDARD | | | |
| / | | <input type="checkbox"/> WITHIN RANGE | |
| / | | <input type="checkbox"/> WITHIN RANGE | |
| / | | <input type="checkbox"/> WITHIN RANGE | |
| / | | <input type="checkbox"/> WITHIN RANGE | |

ORP CALIBRATION CHECK

| CAL. READING (LOT #): (EXP. DATE): | TEMPERATURE (°CELSIUS) | CAL. RANGE | TIME |
|--|---------------------------|---------------------------------------|------|
| POST-CAL. READING / STANDARD | | | |
| / | | <input type="checkbox"/> WITHIN RANGE | |
| / | | <input type="checkbox"/> WITHIN RANGE | |
| / | | <input type="checkbox"/> WITHIN RANGE | |
| / | | <input type="checkbox"/> WITHIN RANGE | |

D.O. CALIBRATION CHECK

| CAL. READING (LOT #): (EXP. DATE): | TEMPERATURE (°CELSIUS) | CAL. RANGE | TIME |
|--|---------------------------|---------------------------------------|------|
| POST-CAL. READING / SATURATED AIR | | | |
| / | | <input type="checkbox"/> WITHIN RANGE | |
| / | | <input type="checkbox"/> WITHIN RANGE | |
| / | | <input type="checkbox"/> WITHIN RANGE | |
| / | | <input type="checkbox"/> WITHIN RANGE | |

TURBIDITY CALIBRATION CHECK

| CALIBRATION READING (NTU) | | CAL. RANGE | TIME |
|------------------------------|------------------------------|---------------------------------------|------|
| (LOT #): (EXP. DATE): | (LOT #): (EXP. DATE): | | |
| POST-CAL. READING / STANDARD | POST-CAL. READING / STANDARD | | |
| / | / | <input type="checkbox"/> WITHIN RANGE | |
| / | / | <input type="checkbox"/> WITHIN RANGE | |
| / | / | <input type="checkbox"/> WITHIN RANGE | |
| / | / | <input type="checkbox"/> WITHIN RANGE | |

COMMENTS

| <input type="checkbox"/> AUTOCAL SOLUTION | <input type="checkbox"/> STANDARD SOLUTION (S) |
|---|--|
| (LOT #): (EXP. DATE): | LIST LOT NUMBERS AND EXPIRATION DATES UNDER CALIBRATION CHECK |
| CALIBRATED PARAMETERS | CALIBRATION RANGES ⁽¹⁾ |
| <input type="checkbox"/> pH | pH: +/- 0.2 S.U. |
| <input type="checkbox"/> COND | COND: +/- 1% OF CAL. STANDARD |
| <input type="checkbox"/> ORP | ORP: +/- 25 mV |
| <input type="checkbox"/> D.O. | D.O.: VARIES |
| <input type="checkbox"/> TURB | TURB: +/- 5% OF CAL. STANDARD |
| <input type="checkbox"/> _____ | ⁽¹⁾ CALIBRATION RANGES ARE SPECIFIC TO THE MODEL OF THE WATER QUALITY METER |
| <input type="checkbox"/> _____ | |

NOTES

| |
|--|
| |
| |
| |
| |

| PROBLEMS ENCOUNTERED | CORRECTIVE ACTIONS |
|----------------------|--------------------|
| | |
| | |
| | |

SIGNED

DATE

CHECKED BY

DATE

[illegible]

SIGNED _____ DATE _____

| | |
|---------|------|
| CHECKED | DATE |
|---------|------|



WATER SAMPLE LOG

| | | | | |
|-----------------|----------|-------|---------|-------|
| PROJECT NAME: | PREPARED | | CHECKED | |
| PROJECT NUMBER: | BY: | DATE: | BY: | DATE: |

| | | | | | | |
|----------------|------------------------------|-----------------------------|---|---|-----------------------------------|--------------------------------|
| SAMPLE ID: | | | WELL DIAMETER: <input type="checkbox"/> 2" <input type="checkbox"/> 4" <input type="checkbox"/> 6" <input type="checkbox"/> OTHER | | | |
| WELL MATERIAL: | <input type="checkbox"/> PVC | <input type="checkbox"/> SS | <input type="checkbox"/> IRON | <input type="checkbox"/> GALVANIZED STEEL | <input type="checkbox"/> OTHER | |
| SAMPLE TYPE: | <input type="checkbox"/> GW | <input type="checkbox"/> WW | <input type="checkbox"/> SW | <input type="checkbox"/> DI | <input type="checkbox"/> LEACHATE | <input type="checkbox"/> OTHER |

| | | | | | | | | | |
|---|--|--|-------|--|---|--|------------------------------|--------------|--|
| PURGING | | TIME: | DATE: | | SAMPLE | | TIME: | DATE: | |
| PURGE METHOD: | | <input type="checkbox"/> PUMP <input type="checkbox"/> BAILER | | | PH: _____ SU | | CONDUCTIVITY: _____ umhos/cm | | |
| | | | | | ORP: _____ mV | | DO: _____ mg/L | | |
| DEPTH TO WATER: _____ T/ PVC | | | | | TURBIDITY: _____ NTU | | | | |
| DEPTH TO BOTTOM: _____ T/ PVC | | | | | <input type="checkbox"/> NONE <input type="checkbox"/> SLIGHT <input type="checkbox"/> MODERATE <input type="checkbox"/> VERY | | | | |
| WELL VOLUME: _____ <input type="checkbox"/> LITERS <input type="checkbox"/> GALLONS | | | | | TEMPERATURE: _____ °C | | | OTHER: _____ | |
| VOLUME REMOVED: _____ <input type="checkbox"/> LITERS <input type="checkbox"/> GALLONS | | | | | COLOR: _____ | | | ODOR: _____ | |
| COLOR: _____ ODOR: _____ | | | | | FILTRATE (0.45 um) <input type="checkbox"/> YES <input type="checkbox"/> NO | | | | |
| TURBIDITY | | | | | FILTRATE COLOR: _____ | | FILTRATE ODOR: _____ | | |
| <input type="checkbox"/> NONE <input type="checkbox"/> SLIGHT <input type="checkbox"/> MODERATE <input type="checkbox"/> VERY | | | | | QC SAMPLE: <input type="checkbox"/> MS/MSD <input type="checkbox"/> DUP- _____ | | | | |
| DISPOSAL METHOD: <input type="checkbox"/> GROUND <input type="checkbox"/> DRUM <input type="checkbox"/> OTHER | | | | | COMMENTS: | | | | |

[illegible]

NOTE: STABILIZATION TEST IS COMPLETE WHEN 3 SUCCESSIVE READINGS ARE WITHIN THE FOLLOWING LIMITS:

pH: +/- 0.1 COND.: +/- 10 % ORP: +/- 10 % D.O.: +/- 10 % TURB: +/- 10 % or <= 5 TEMP.: +/- 0.5°C

| BOTTLES FILLED | | PRESERVATIVE CODES A - NONE B - HNO3 C - H2SO4 D - NaOH E - HCL F - _____ | | | | | | | | | | | | | |
|----------------|------|---|--------------|--------------------------|---|--------------------------|------|------|--------------|----------|--|--------------------------|---|-------------------------------------|---|
| NUMBER | SIZE | TYPE | PRESERVATIVE | FILTERED | | NUMBER | SIZE | TYPE | PRESERVATIVE | FILTERED | | | | | |
| | | | | <input type="checkbox"/> | Y | <input type="checkbox"/> | N | | | | | <input type="checkbox"/> | Y | <input type="checkbox"/> | N |
| | | | | <input type="checkbox"/> | Y | <input type="checkbox"/> | N | | | | | <input type="checkbox"/> | Y | <input checked="" type="checkbox"/> | N |
| | | | | <input type="checkbox"/> | Y | <input type="checkbox"/> | N | | | | | <input type="checkbox"/> | Y | <input type="checkbox"/> | N |
| | | | | <input type="checkbox"/> | Y | <input type="checkbox"/> | N | | | | | <input type="checkbox"/> | Y | <input type="checkbox"/> | N |
| | | | | <input type="checkbox"/> | Y | <input type="checkbox"/> | N | | | | | <input type="checkbox"/> | Y | <input type="checkbox"/> | N |

| | | |
|------------------------|---------------------|-----------------------|
| SHIPPING METHOD: _____ | DATE SHIPPED: _____ | AIRBILL NUMBER: _____ |
| COC NUMBER: _____ | SIGNATURE: _____ | DATE SIGNED: _____ |



WATER SAMPLE LOG

(CONTINUED FROM PREVIOUS PAGE)

| | | | | |
|-----------------|----------|-------|---------|-------|
| PROJECT NAME: | PREPARED | | CHECKED | |
| PROJECT NUMBER: | BY: | DATE: | BY: | DATE: |

SAMPLE ID: _____

[illegible]

SIGNATURE: _____

DATE SIGNED: _____

4101 Shuffel Street NW

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Regulatory Program: ☐ DW ☐ NPDES ☐ RCRA ☐ Other:

TestAmerica Laboratories, Inc.

[illegible]



MONITORING WELL DECOMMISSIONING LOG

| | | | |
|----------------------|-------|----------------------------|-----------------------|
| PROJECT NAME: | | MONITORING WELL ID: | |
| PROJECT NUMBER: | DATE: | LOCATION: | LOCATION COORDINATES: |
| OBSERVED BY: | | | N: |
| DRILLING CONTRACTOR: | | | E: |
| CREW CHIEF: | | TOP OF CASING ELEV.: _____ | SURFACE ELEV.: _____ |

| | | | | |
|----------------------------|-----------------------------------|--------------------------------------|------------------------------------|---|
| PROTECTIVE COVER TYPE: | <input type="checkbox"/> STICK-UP | <input type="checkbox"/> FLUSH MOUNT | <input type="checkbox"/> TRAF. BOX | <input type="checkbox"/> OTHER _____ |
| PROTECTIVE COVER DIAMETER: | <input type="checkbox"/> 4" | <input type="checkbox"/> 8" | <input type="checkbox"/> 9" | <input type="checkbox"/> 10" |
| WELL MATERIAL: | <input type="checkbox"/> PVC | <input type="checkbox"/> SS | <input type="checkbox"/> IRON | <input type="checkbox"/> GALVANIZED STEEL |
| WELL CASING DIAMETER: | <input type="checkbox"/> 1" | <input type="checkbox"/> 2" | <input type="checkbox"/> 4" | <input type="checkbox"/> 6" |
| WELL SCREEN MATERIAL: | <input type="checkbox"/> PVC | <input type="checkbox"/> SS | <input type="checkbox"/> IRON | <input type="checkbox"/> GALVANIZED STEEL |
| WELL SCREEN LENGTH: | <input type="checkbox"/> 5-FT | <input type="checkbox"/> 10-FT | <input type="checkbox"/> UNKNOWN | <input type="checkbox"/> OTHER _____ |
| WELL SCREEN SLOT SIZE: | <input type="checkbox"/> 0.01" | <input type="checkbox"/> 0.02" | <input type="checkbox"/> UNKNOWN | <input type="checkbox"/> OTHER _____ |
| | | | | DTW: _____ T/ PVC |
| | | | | DTB: _____ T/ PVC |

| |
|----------------------------|
| DECOMMISSIONING PROCEDURE: |
| NOTES: |

| | |
|--|--------|
| GROUTING PROCEDURE: | NOTES: |
| GROUT TYPE: | |
| GROUT MIX: | |
| GROUT INTERVAL: _____ FT-BGS TO _____ FT-BGS | |
| BENTONITE SEAL: | |
| SEAL INTERVAL: _____ FT-BGS TO _____ FT-BGS | |

| |
|----------------------|
| ADDITIONAL COMMENTS: |
|----------------------|

SIGNED

DATE

CHECKED

DATE

Appendix C

Laboratory QAP (on CD)

**Test America Laboratories' QA/QC Plan
is provided on the enclosed CD**

Quality Assurance Manual

TestAmerica Canton

4101 Shuffel Street NW

North Canton, OH 44720

Phone: 330-497-9396

Fax: 330-497-0772

www.testamericainc.com

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1. TITLE PAGE:

Quality Assurance Manual

Approval Signatures



Laboratory Director – Daniel Pittman

07/15/14

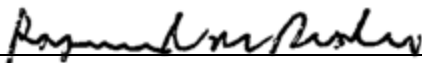
Date



Quality Assurance Manager – Dee Shepperd

07/15/14

Date



Technical Director – Raymond Riden

07/16/14

Date

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SOPs AND POLICIES REFERRED TO IN THE QA MANUAL

| SOP/Policy Reference | Title |
|----------------------|--|
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| CW-L-P-004 | Ethics Policy |
| CA-L-P-002 | Contract Compliance Policy |
| CW-L-S-002 | Internal Investigation of Potential Data Discrepancies and Determination for Data Recall |
| CA-L-S-002 | Subcontracting Procedures |
| CA-Q-S-001 | Solvent and Acid Lot Testing and Approval |
| CA-Q-S-002 | Acceptable Manual Integration Practices |
| CA-Q-S-004 | Method Compliance & Data Authenticity Audits |
| CA-Q-S-006 | Detection Limits |
| CA-Q-S-008 | Management Systems Review |
| CA-T-P-001 | Qualified Products List |
| CW-E-M-001 | Corporate Environmental Health & Safety Manual |
| CW-F-P-002 | Company-wide Authorization Matrix |
| CW-F-P-004 | Procurement and Contracts Policy |
| CW-F-S-007 | Capital Expenditure, Controlled Purchase Requests, and Fixed Asset Capitalization |
| | |
| CW-Q-S-001 | Corporate Document Control and Archiving |
| CW-Q-S-002 | Writing a Standard Operating Procedure (SOP) |
| CA-Q-M-002 | Corporate Quality Management Plan |
| NC-QA-015 | Equipment Monitoring and Thermometer Calibration |
| NC-QA-018 | Statistical Evaluation of Data and Development of Control Charts |
| NC-QA-019 | Records Information Management |
| NC-QA-027 | Preparation and Management of Standard Operating Procedures (SOPs) |
| NC-QA-028 | Employee Orientation and Training |
| NC-QA-029 | Nonconformance and Corrective Action System |
| NC-QA-030 | Document Control |
| NC-SC-005 | Sample Receiving and Sample Control |
| NC-SC-006 | Sample Procurement Protocol |
| CA-Q-T-005 | Laboratory Documentation |
| NC-QA-021 | Evaluation of Method Detection Limits for Chemical Tests |
| NC-QA-031 | Internal Audits |

3. INTRODUCTION, SCOPE, AND APPLICABILITY

- 3.1. Introduction and Compliance References
- 3.2. TestAmerica Canton's Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organizational objectives and functional responsibilities for achieving TestAmerica's data quality goals. The laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.
- 3.3. The QA Manual has been prepared to assure compliance with the NELAC Institute (TNI) Standard, dated 2009, Volume 1, Modules 2 and 4, ISO/IEC Guide 17025:2005(E), and DoD QSM 4.2 (will transition to QSM 5.0 in 2015). In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Quality Management Plan, CA-Q-M-002, (CQMP) and the various accreditation and certification programs listed in Appendix 4. The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations. The relevant NELAC section is included in the heading of each QAM section.
- 3.4. The QA Manual has been prepared to be consistent with the requirements of the following documents:
 - 3.4.1. EPA 600/4-79-019, Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EPA, March 1979.
 - 3.4.2. Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.
 - 3.4.3. U.S. Department of Defense, (DoD)/Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, Version 4.2, October 2010 (transitioning in 2015 to QSM 5.0, July 2013).
 - 3.4.4. APHA, Standard Methods for the Examination of Water and Wastewater, 18th Edition, 19th, 20th, 21st, and on-line Editions.
 - 3.4.5. Statement of Work for Inorganics & Organics Analysis, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.
 - 3.4.6. Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
 - 3.4.7. Toxic Substances Control Act (TSCA).

3.5. Terms and Definitions

3.5.1. A Quality Assurance Program is a company-wide system designed to ensure data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

3.5.2. Refer to Appendix 3 for the Glossary/Acronyms.

3.6. Scope / Fields of Testing

3.6.1. The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among effluent water, groundwater, hazardous waste, sludge, wipes, and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical, physical and biological parameters. The Program also contains guidelines on maintaining documentation of analytical processes, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all analytical requests are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

3.6.2. The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in Appendix 2 . The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet these requirements. All methods performed by the laboratory shall meet or exceed these criteria, as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual and the referenced methods. In these cases, the laboratory must abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director, the Quality Assurance (QA) Manager, and the Technical Director. In some cases, QAPPs and DQOs may specify less stringent requirements. The Technical Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

3.6.3. Specific requirements delineated in project plans may supersede general quality requirements described in this manual. Ohio VAP requirements are listed throughout the document.

3.7. Management of the Manual

3.7.1. Review Process

3.7.1.1. The template on which this manual is based is reviewed annually by Corporate Quality Management personnel to assure it remains in compliance with Section 3.1. This manual itself is reviewed annually by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager must review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates must be reviewed by the senior laboratory management staff (Laboratory Director, Technical Director, Operations Manager, and QA Manager). The laboratory updates and approves such changes according to our Document Control SOP (NC-QA-030) and Updating Procedures SOP (NC-QA-027).

4. MANAGEMENT REQUIREMENTS

4.1. Overview

4.1.1. TestAmerica Canton is a local operating unit of TestAmerica Laboratories, Inc. The organizational structure, responsibilities, and authorities of the corporate staff of TestAmerica Laboratories, Inc. are presented in the CQMP. The laboratory has day-to-day independent operational authority overseen by corporate officers (e.g., Chief Executive Officer (CEO), Executive VP Operations, Corporate Quality, and EH&S Director, etc.). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate and TestAmerica North Canton is presented in Figure 4-1. Employee names are provided to demonstrate range and size of departments however the actual staff members may vary over time. The most current Organization Chart may be obtained from Quality Assurance Manager or Laboratory Director.

4.2. Roles and Responsibilities

4.2.1. In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions briefly define each role in its relationship to the Quality Assurance Program. More extensive job descriptions are maintained by laboratory management.

4.3. Additional Requirements for Laboratories

4.3.1. The responsibility for quality resides with every employee of the laboratory. All employees have access to the QAM, are trained to this manual, and are responsible for knowing the content of this manual and upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. Role descriptions for Corporate personnel are defined in the CQMP. This manual is specific to the operations of TestAmerica's Canton laboratory.

4.4. Canton Laboratory Key Personnel

| Name | Position |
|-----------------|--|
| Rusty Vicinie | VP of Operations, Central |
| Daniel Pittman | Laboratory Director |
| Raymond Risdien | Technical Director |
| Carolynne Roach | Operations Manager |
| Dee Shepperd | Quality Assurance Manager |
| Rebecca Strait | Client Relations Manager |
| Steve Jackson | Regional Safety Director, Waste Management Supervisor |
| Chris Coast | Extractions Group Leader |
| Will Cordell | Field Analytical Group Leader |
| Olguita Colon | GC Volatile/Semivolatiles Group Leader |
| Tom Hula | GC/MS Semivolatiles Group Leader |
| Lucas Grossman | General Chemistry Group Leader |
| Darren Miller | Maintenance |
| Aaron Martin | Metals Group Leader |
| Patrick O'Meara | Project Management Group Leader |
| Ann Maddux | Sample Control Group Leader |
| Lance Hershman | Shipping Group Leader |

4.5. Quality Assurance (QA) Manager or Designee

- 4.5.1. The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system.
- 4.5.2. The QA Manager reports directly to the Laboratory Director, and has access to Corporate QA for advice and resources. This position is able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence. Corporate QA may be used as a resource in dealing with regulatory requirements, certifications, and other quality assurance related items. The QA Manager directs the activities of the QA officers to accomplish specific responsibilities, which include, but are not limited to:
 - 4.5.2.1. Serves as the focal point for QA/QC in the laboratory.
 - 4.5.2.2. Having functions independent from laboratory operations for which he/she has quality assurance oversight.
 - 4.5.2.3. Maintaining and updating the QA Manual.
 - 4.5.2.4. Monitoring and evaluating laboratory certifications, scheduling proficiency testing (PT) samples.
 - 4.5.2.5. Monitoring and communicating to management, regulatory changes that may affect the laboratory.
 - 4.5.2.6. Training and advising the laboratory staff on quality assurance/quality control (QA/QC) procedures that are pertinent to their daily activities.
 - 4.5.2.7. Having documented training and/or experience in QA/QC procedures and the laboratory's Quality System.
 - 4.5.2.8. Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
 - 4.5.2.9. Arranging for or conducting internal audits on quality systems and the technical operation.
 - 4.5.2.10. Maintaining records of all ethics-related training, including the type and proof of attendance.
 - 4.5.2.11. Maintaining, improving, and evaluating the corrective action database and the corrective and preventive action systems.

- 4.5.2.12. Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QA Manual or laboratory SOPs shall be investigated following procedures outlined in Section 12; and if deemed necessary, may be temporarily suspended during the investigation.
- 4.5.2.13. Objectively monitoring standards of performance in QC and QA without outside (e.g., managerial) influence.
- 4.5.2.14. Coordinating of document control of SOPs, MDL, control limits, and miscellaneous forms and information.
- 4.5.2.15. Reviewing a percentage of all final data reports for internal consistency. Review of Chain of Custody (COC), correspondence with the analytical request, batch QC status, completeness of any corrective action statements, 5% of calculations, format, holding time, reasonableness of results and completeness of the project file contents.
- 4.5.2.16. Reviewing external audit reports and data validation requests.
- 4.5.2.17. Following up with data and laboratory audits to ensure client QAPP requirements are met.
- 4.5.2.18. Establishing reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or Corporate QA.
- 4.5.2.19. Developing suggestions and recommendations to improve quality systems.
- 4.5.2.20. Researching current state and federal requirements and guidelines.
- 4.5.2.21. Captaining the QA team to enable communication and to distribute duties and responsibilities.
- 4.5.2.22. Ensuring communication and monitoring standards of performance to ensure systems are in place to produce the level of quality as defined in this document.
- 4.5.2.23. Evaluating the thoroughness and effectiveness of training.
- 4.5.2.24. Assuring compliance with ISO 17025.
- 4.5.2.25. Assuring compliance with DoD ELAP.

4.6. Technical Director & Department Group Leader

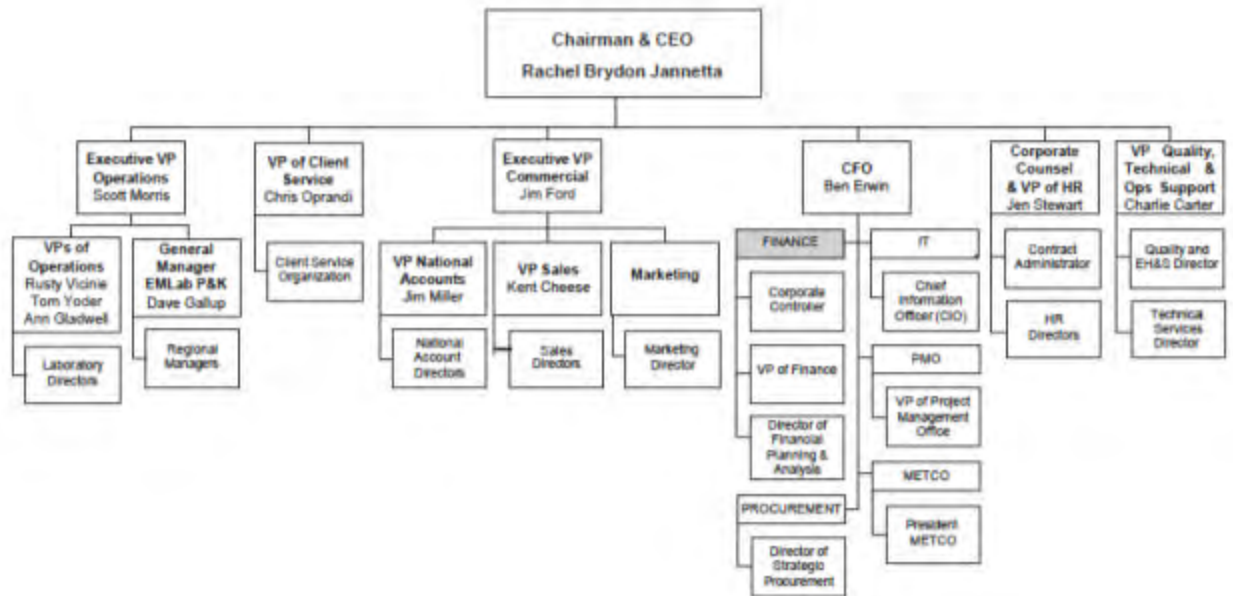
- 4.6.1.1. The Technical Director reports directly to the Laboratory Director. The Technical Director along with the Laboratory Director, the QA Manager, the Operations Manager, and each Department Group Leader is accountable for compliance with the ISO 17025 Standard. The Technical Director works with QA and Department Group Leaders to solve day-to-day technical issues, provide technical training and guidance to laboratory staff, project managers, and clients, and assists with method development and validation.
- 4.6.1.2. The Department Group Leaders report to the Operations Manager. The Group Leaders maintain overall responsibilities for a defined portion of the laboratory. These responsibilities include but are not limited to:
- 4.6.1.3. Day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results. Working with the QA Manager to coordinate preparation of test method SOPs and perform subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples and/or requirements.
- 4.6.1.4. Monitoring the validity of the analyses performed and data generated in the laboratory.
- 4.6.1.5. Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a continuing, scheduled basis. Training includes instruction on calculations, instrumentation, troubleshooting, and preventive maintenance.
- 4.6.1.6. Enhancing efficiency and improving quality through technical advances and improved laboratory information management system (LIMS) utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- 4.6.1.7. Working with the QA Manager in scheduling all QA/QC-related requirements for compliance, e.g. MDLs, etc.
- 4.6.1.8. Captains department personnel to communicate quality, technical, personnel and instrumental issues for a consistent team approach.
- 4.6.1.9. Compliance with ISO 17025 (where applicable).
- 4.6.1.10. Compliance with DoD ELAP (where applicable).

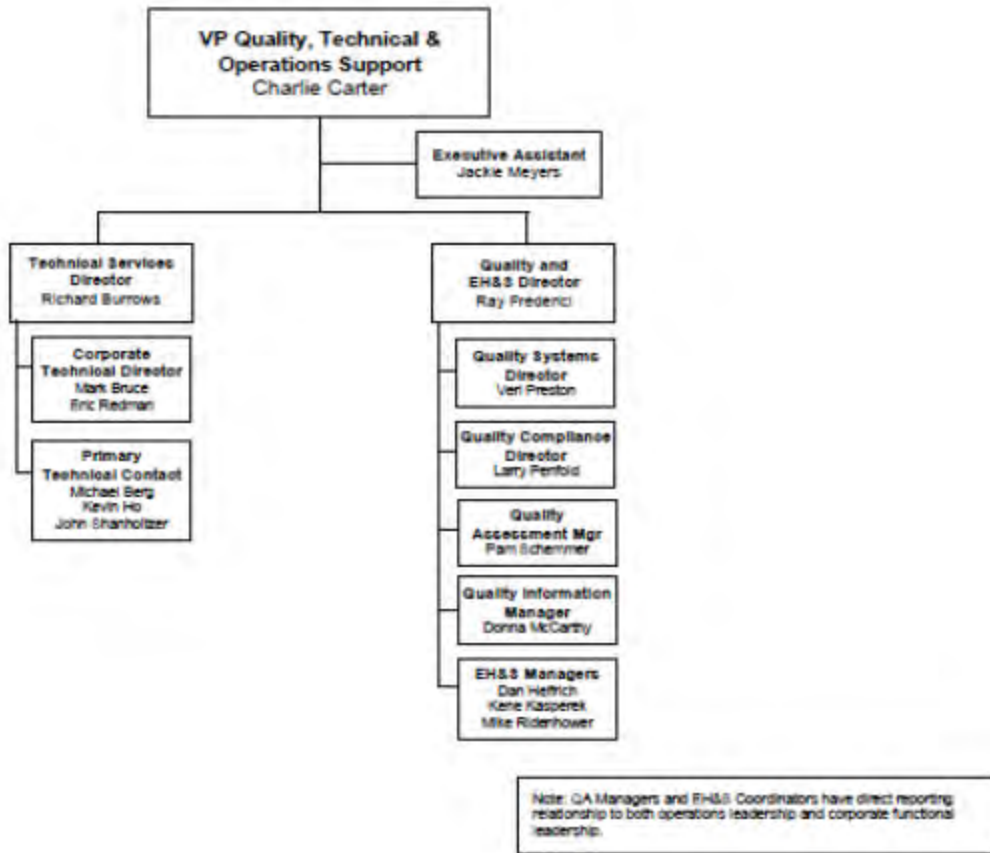
4.6.2. Deputies

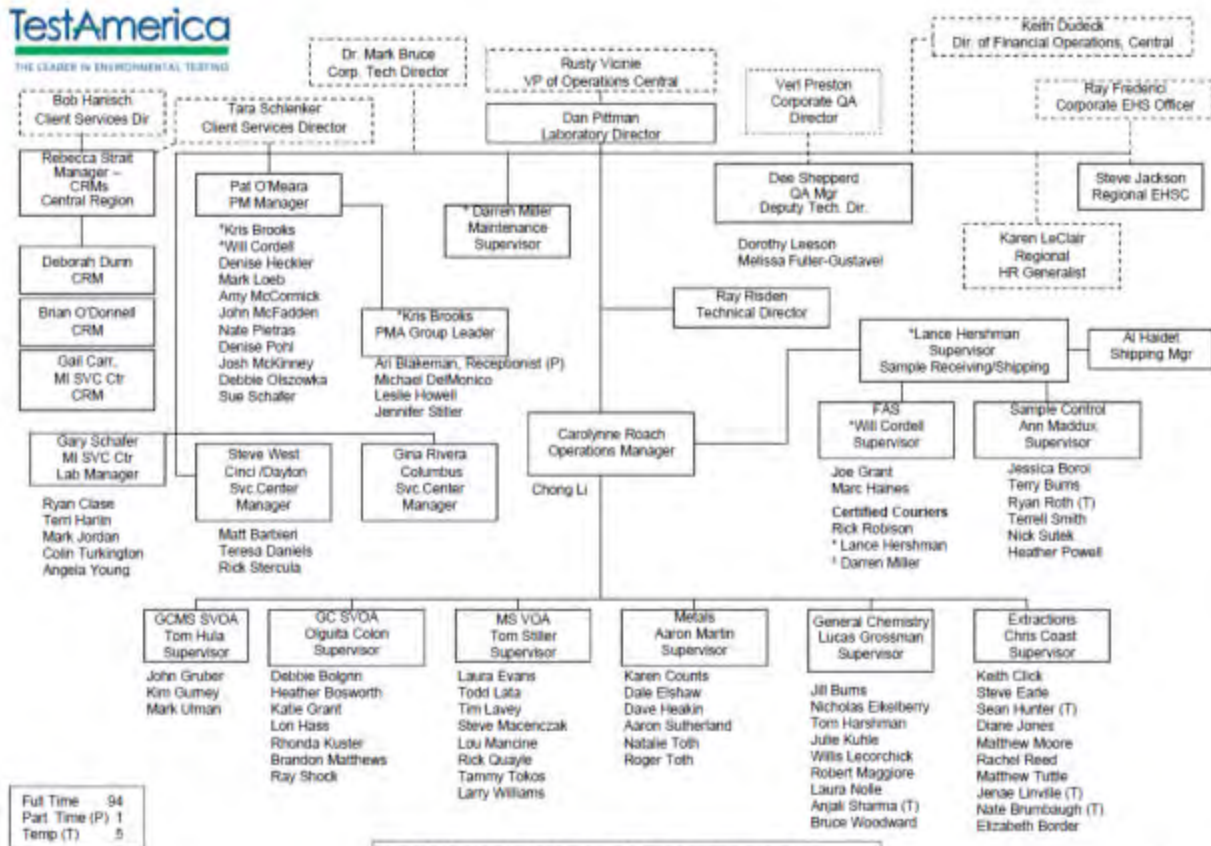
4.6.2.1. The following table defines who assumes the responsibilities of key personnel in their absence:

| Key Personnel | Deputy |
|---------------------------|--|
| Laboratory Director | Technical Director QA Manager |
| Quality Assurance Manager | Laboratory Director Quality Assurance Coordinator |
| Technical Director | Operations Manager Quality Assurance Manager |
| EHS Coordinator | Technical Director Operations Manager |

Figure 4-1. Corporate and Laboratory Organization Charts







Full Time 94
 Part Time (P) 1
 Temp (T) 5

* Listed under multiple groups

Note: QA Manager, EH&S Manager, CSO team members have a direct reporting relationship to both operations leadership and corporate functional leadership. Regional staff assigned to lab are included in headcount. Corporate operations & finance staff are excluded from lab headcount.

Effective: 6-17-14

5. QUALITY SYSTEM

5.1. Quality Policy Statement

5.2. It is TestAmerica's policy to:

- 5.2.1. Provide data of know quality to its clients by adhering to approved methodologies, regulatory requirements, and the QA/QC protocols.
- 5.2.2. Effectively manage all aspects of the laboratory and business operations by the highest ethical standards.
- 5.2.3. Continually improve systems and provide support to quality improvement efforts in laboratory, administrative, and managerial activities.
TestAmerica recognizes that the implementation of a QA program requires management's commitment and support as well as the involvement of the entire staff.
- 5.2.4. Provide clients with the highest level of professionalism and the best service practices in the industry.
- 5.2.5. Comply with the ISO/IEC 17025:2005(E) International Standard, the 2009 TNI Standard, and to continually improve the effectiveness of the management system.
- 5.2.6. Every staff member at the laboratory plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

5.3. Ethics and Data Integrity

- 5.3.1. TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The elements of the TestAmerica Ethics and Data Integrity Program include:
- 5.3.2. An Ethics Policy (Corporate Policy CW-L-P-004) and Employee Ethics Statements (Appendix 1)
- 5.3.3. Ethics and Compliance Officers (ECOs)
- 5.3.4. A training program
- 5.3.5. Self-governance through disciplinary action for violations
- 5.3.6. A confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct (Corporate SOP CW-L-S-002)

- 5.3.7. Procedures and guidance for recalling data if necessary (Corporate SOP CW-L-S-002)
- 5.3.8. Effective external and internal monitoring system that includes procedures for internal audits (Section 16)
- 5.3.9. Production of results which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).
- 5.3.10. Presenting services in a confidential, honest, and forthright manner.
- 5.3.11. Providing employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry.
- 5.3.12. Operating our facilities in a manner that protects the environment and the health and safety of employees and the public.
- 5.3.13. Obeying all pertinent federal, state, and local laws and regulations and encourage other members of our industry to do the same.
- 5.3.14. Educating clients as to the extent and kinds of services available.
- 5.3.15. Asserting competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- 5.3.16. Promoting the status of environmental laboratories, their employees, and the value of services rendered by them.
- 5.4. Quality System Documentation
 - 5.4.1. The laboratory's Quality System is communicated through a variety of documents
 - 5.4.1.1. Quality Assurance Manual – Each laboratory has a lab-specific Quality Assurance Manual.
 - 5.4.1.2. Corporate SOPs and Policies - Corporate SOPs and Policies are developed for use by all relevant laboratories. They are incorporated into the laboratory's normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
 - 5.4.1.3. Work Instructions - A subset of procedural steps, tasks, or forms associated with an operation of a management system, e.g., checklists, preformatted bench sheets, forms.
 - 5.4.1.4. Laboratory SOPs – General and technical
 - 5.4.1.5. Laboratory QA/QC Policy Memorandums

5.5. Order of Precedence

5.5.1. In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

5.5.1.1. Corporate Quality Management Plan (CQMP)

5.5.1.2. Corporate SOPs and Policies

5.5.1.3. Laboratory QA/QC Policy Memorandum

5.5.1.4. Laboratory Quality Assurance Manual (QA Manual)

5.5.1.5. Laboratory SOPs and Policies

5.5.1.6. Other: Work Instructions (WI), memos, flow charts, etc.

Note: The laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the CQMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's QA Manager shall take precedence over the CQMP in those cases.

5.5.2. Any regulatory requirements (e.g.; Ohio VAP, CT RCP, etc) provided in the laboratory specific documents (i.e., QA Manual and SOPs) take precedence over any policies provided in corporate documents.

5.6. QA/QC Objectives for the Measurement of Data

5.6.1. Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. QA is generally understood to be more comprehensive than Q C. QA can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

5.6.2. QC is generally understood to be limited to the analyses of samples and to be synonymous with the term "analytical quality control". QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

5.6.3. Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives (DQOs) in order to ensure that analytical services closely correspond to client needs. The client is responsible for developing the QAPP. In order to ensure the ability of the laboratory to

meet the DQOs specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory must provide support to the client for developing the sections of the QAPP that concern laboratory activities.

5.6.4. Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity, and sensitivity (PARCCSS). Equations to derive relevant QC objectives can be found in the method specific SOPs.

5.6.5. Precision

5.6.5.1. The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) and/or matrixspike duplicate(MSD)samples.

5.6.6. Accuracy

5.6.6.1. The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet DQOs of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptable recovery centered on the mean recovery.

5.6.7. Representativeness

5.6.7.1. The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference (RPD) between separately procured, but otherwise identical, samples or sample aliquots.

5.6.7.2. The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

5.6.8. Comparability

- 5.6.8.1. The comparability objective is to provide analytical data for which the accuracy, precision, representativeness, and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the same laboratory over time.
- 5.6.8.2. The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision, and reporting limits with those of other laboratories.

5.6.9. Completeness

- 5.6.9.1. The completeness objective for data is 90% (or as specified by a particular project) expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability must be defined in a QAPP, project scope, or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

5.6.10. Selectivity

- 5.6.10.1. Selectivity is defined as the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), inter-element corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc.

5.6.11. Sensitivity

- 5.6.11.1. Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (Method Detection Limit [MDL]) or quantified (Reporting Limit [RL]).

5.7. Criteria for Quality Indicators

5.7.1. The laboratory maintains Quality Control Limits in LIMS that summarize the precision and accuracy acceptability limits for performed analyses. These summaries include an effective date, are updated each time new limits are generated, and are managed by the laboratory's QA Department. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where U.S. EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits are contained in NC-QA-018 Statistical Evaluation of Data and Development of Control Charts and in Section 24).

5.8. Statistical Quality Control

5.8.1. Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory (dated and approved by the Group Leader and QA Manager) and entered into LIMS. An archive of all limits used within the laboratory is maintained in the LIMS. If a method defines the QC limits, the method limits are used.

5.8.2. If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in Section 25. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

5.8.3. Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. If one or more QC values are outside of limits, the analyst then evaluates whether the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

5.9. QC Charts

5.9.1. The laboratory's procedures for the creation of control charts are described in laboratory SOP No. NC-QA-018, "Statistical Evaluation of Data and Development of Control Charts." Control charts are created from data stored in the LIMS. The charts are evaluated by QA or technical staff to determine if limits need to be updated or to assess the need for corrective actions to improve method performance.

5.9.2. Control charts are used to develop control limits, trouble-shoot analytical problems, and, in conjunction with the non-conformance system, to monitor for trends. Program-specific data analysis requirements for

control charts are followed as required for data generated under those programs. These additional requirements shall be documented in a QAPP.

5.10. Quality System Metrics

- 5.10.1. In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 16). These metrics are used to drive continuous improvement in the laboratory's Quality System.

6. DOCUMENT CONTROL

6.1. Overview

- 6.1.1. The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled at each laboratory Facility:
- 6.1.1.1. Laboratory Quality Assurance Manual
 - 6.1.1.2. Laboratory Standard Operating Procedures (SOP)
 - 6.1.1.3. Laboratory Policies
 - 6.1.1.4. Work Instructions and Forms
 - 6.1.1.5. Laboratory spreadsheets used for calibration and analysis
 - 6.1.1.6. Corporate Policies and Procedures distributed outside the intranet
- 6.1.2. Corporate Quality posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers, and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the company intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving Corporate documents is found in Corporate SOP CW-Q-S-001, Corporate Document Control and Archiving. The laboratory's internal document control procedure is defined in SOP NC-QA-030, "Document Control" and SOP NC-QA-027, "Preparation and Management of Standard Operating Procedures."
- 6.1.3. The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. The laboratory also maintains instrument manuals (hard or electronic copies).

These documents are maintained on the public drive in a document control master database.

- 6.1.4. The QA department maintains control of supporting records such as audit reports and responses, logbooks, standard logs, Ethics and QA training files, MDL studies, PT studies, certifications and related correspondence, and corrective action reports. Raw analytical data, consisting of bound logbooks, instrument printouts, any other notes, technical training files, magnetic media, electronic data, and final reports are retained electronically by each analytical section, the QA department, or on the company servers.

6.2. Document Approval and Issue

- 6.2.1. The pertinent elements of a document control system for each document include a unique document title and number, pagination, the total number of pages of the item, the effective date, revision number, and the laboratory name and facility. The QA Department is responsible for the maintenance of this system.
- 6.2.2. Controlled documents are authorized by the QA Department and members of management. In order to develop a new document, a staff member submits a draft to the QA Department for comments, changes, and approval before use. Upon approval, QA personnel add the identifying version information to the document and retain that document as the official document on file. The document is then provided to all applicable operational units (may include electronic access). Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution (see SOP NC-QA-027 for more information).
- 6.2.3. The QA Department maintains a list of the official versions of controlled documents in the document control database.
- 6.2.4. Quality System Policies and Procedures must be reviewed at a minimum of every 24 months, and revised as appropriate. For procedures associated with DoD and Ohio VAP project work, applicable SOPs and Policies are reviewed every 12 months. Changes to documents occur when a procedural change warrants.

6.3. Procedures for Document Control Policy

- 6.3.1. For changes to the QA Manual, refer to SOPs NC-QA-019 and CW-Q-S-001. Uncontrolled copies must not be used within the laboratory. Previous revisions are stored electronically by the QA Department on the public server in the QAQC folder for the applicable revision. The current revision is located in the public controlled document folder accessible to all employees.

- 6.3.2. For changes to SOPs, refer to Corporate SOP CW-Q-S-002, Writing a Standard Operating Procedure (SOP), and SOP NC-QA-027, Preparation and Management of Standard Operating Procedures. The SOP identified above also defines the process of changes to SOPs.
- 6.3.3. Forms, worksheets, work instructions, electronic spreadsheets, logbooks, and information are identified and organized by the QA department in accordance with the procedures specified in laboratory SOP NC-QA-027.
- 6.4. Obsolete Documents
 - 6.4.1. All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, hard copies of obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived in accordance with SOP NC-QA-027.

7. SERVICE TO THE CLIENT

- 7.1. Overview
 - 7.1.1. The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.
 - 7.1.2. A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.
 - 7.1.3. All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, turnaround time, sensitivity (detection and reporting levels), accuracy, and precision requirements (Recovery [%R] and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential

subcontract laboratories must be certified, as required, for all proposed tests.

- 7.1.4. The laboratory must determine if it has the necessary physical, personnel, and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time must be checked for feasibility.
- 7.1.5. Electronic or hard-copy deliverable requirements are evaluated against the laboratory's capacity for production of the documentation.
- 7.1.6. If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this must be documented and discussed with the client prior to contract approval (refer to Section 8 for Subcontracting Procedures).
- 7.1.7. The laboratory informs the client of the results of the review and whether any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily is indicated. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.
- 7.1.8. All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.
- 7.1.9. The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.
- 7.2. Review Sequence and Key Personnel
 - 7.2.1. Appropriate personnel must review the work request at each stage of evaluation.
 - 7.2.2. For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.
 - 7.2.3. For new, complex or large projects, the opportunity is forwarded to a Customer Service Manager (CSM) for review. The CSM contacts the appropriate Sales Executive (National Account Manager, Key Account

Executive, Regional Account Executive, and/or Program Manager) to determine which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, reporting specifications, and available capacity to perform the work. The contract review process is outlined in TestAmerica's Corporate SOP CA-L-P-002, Contract Compliance Policy.

- 7.2.4. This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, based on scope of contract, to evaluate all of the requirements shown above (not necessarily in this order):
 - 7.2.4.1. Contract Administrator
 - 7.2.4.2. Laboratory Client Service Manager
 - 7.2.4.3. Laboratory Project Manager
 - 7.2.4.4. Laboratory and/or Corporate Technical Director
 - 7.2.4.5. Laboratory and/or Corporate Information Technology Managers/Directors
 - 7.2.4.6. Regional and/or National Account representatives
 - 7.2.4.7. Laboratory and/or Corporate Quality Assurance Managers
 - 7.2.4.8. Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
 - 7.2.4.9. The Laboratory Director reviews the formal laboratory quote, and makes final acceptance for their facility.
 - 7.2.4.10. Based on the level of discount extended for the project, approval of the VP of Operations or Sales Director may also be required.
 - 7.2.4.11. The Sales Director, Contract Administrator, Account Executive, or Proposal Coordinator then submits the final proposal to the client.
 - 7.2.4.12. In the event that one of the above personnel is not available to review the contract, his or her backup will fulfill the review requirements.
 - 7.2.4.13. The Contracts Department (or their designee) maintains copies of all signed contracts. The Laboratory Director also maintains an electronic copy of any contract signed at the local level.

7.3. Documentation

- 7.3.1. Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. Documents are reviewed by the Laboratory Director and stored on the laboratory's public drive.
- 7.3.2. The contract must be distributed to and maintained by the Corporate Contracts Department and the applicable Account Executive. A copy of the contract must be filed electronically by the Laboratory Director. Quotes must be archived electronically in the laboratory quote module in TALs or in the public shared drive if an off-TALs quote is submitted.
- 7.3.3. Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps email records or a phone log of conversations with the client.
- 7.3.4. Project-Specific Quality Planning
 - 7.3.4.1. Communication of contract-specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, the laboratory assigns a PM to each client. The PM is the first point of contact for the client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements.
 - 7.3.4.2. PM's are the primary client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.
 - 7.3.4.3. Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

- 7.3.4.4. During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes, e.g., use of a non-standard method or modification of a method, and approvals must be documented prior to implementation. Documentation pertains to any document, e.g., letter, e-mail, variance, contract addendum, which has been signed by both parties.
- 7.3.4.5. Such changes are also communicated to the laboratory. Project-specific changes made after samples are in-house are communicated through Change Information Notification emails
- 7.3.4.6. Programmatic and/or method changes are communicated via email transmittal and/or in meetings with the applicable Operations Managers. If the modification includes use of a non-standard method, or significant modification of a method, documentation of the modification is made in the case narrative of the applicable data report(s).
- 7.3.4.7. The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

7.4. Special Services

- 7.4.1. The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 15 and 25).

Note: ISO/IEC 17025:2005(E) states that a laboratory "shall afford clients or their representatives' cooperation to clarify the client's request". This topic is discussed in Section 7 of the ISO standard.

- 7.4.2. The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:
- 7.4.3. Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- 7.4.4. Assist client-specified third-party data validators as specified in the client's contract.
- 7.4.5. Supplemental information pertaining to the analysis of their samples.
Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

7.5. Client Communication

- 7.5.1. Customer Service Managers (CSMs) and Project Managers (PMs) are the primary communication link to the clients. They must inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project Management must maintain ongoing client communication throughout the entire client project.
- 7.5.2. The Technical Director, Operation Manager, QA Manager or Group Leaders are available to discuss any technical questions or concerns the client may have.

7.6. Reporting

- 7.6.1. The laboratory works with our clients to produce any special communication reports required by the contract.

7.7. Client Surveys

- 7.7.1. The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service. TestAmerica Sales and Marketing teams periodically develop lab and client-specific surveys to assess client satisfaction.

8. SUBCONTRACTING OF TESTS

8.1. Overview

- 8.1.1. For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the TestAmerica Laboratories. The phrase “work sharing” refers to internal transfers of samples between the TestAmerica Laboratories. The term “outsourcing” refers to the act of subcontracting tests to external laboratories or laboratories within the TestAmerica network.
- 8.1.2. When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and meet the same commitments we have made to the client. Refer to TestAmerica’s Corporate SOPs on Subcontracting Procedures (CA-L-S-002).
- 8.1.3. When outsourcing analytical services, the laboratory must assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in TNI ISO/IEC 17025:2005(E) and/or the client’s Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client’s analytical program are transmitted to the subcontractor and

agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation must be placed with an appropriately accredited laboratory. In all cases, TNI accredited as well as non-TNI, the laboratory performing the subcontracted work must be identified in the final report.

- 8.1.4. For DoD projects, the subcontractor laboratories used must have an established and documented laboratory quality system that complies with DoD QSM requirements. The subcontractor laboratories are evaluated following the procedures outlined below and as seen in Figure 8-1. The subcontractor laboratory must receive project-specific approval from the DoD client before any samples are analyzed.
- 8.1.5. The QSM has five specific requirements for subcontracting:
 - 8.1.5.1. Subcontractor laboratories must have an established laboratory quality system that complies with the QSM.
 - 8.1.5.2. Subcontractor laboratories must be approved by the specific DoD component laboratory approval process (outlined in the QSM).
 - 8.1.5.3. Subcontractor laboratories must demonstrate the ability to generate acceptable results from the analysis of PT samples, subject to availability, using each applicable method, in the specified matrix, and provide appropriate documentation to the DoD client.
 - 8.1.5.4. Subcontractor laboratories must receive project-specific approval from the DoD client before any samples are analyzed.
 - 8.1.5.5. Subcontractor laboratories are subject to project-specific, on-site assessments by the DoD client or their designated representatives.
- 8.1.6. PMs or Client Service Managers (CSM) or Account Executives (AE) (or others as defined by the lab) for the Export Lab (TestAmerica laboratory that transfers samples to another laboratory) are responsible for obtaining client approval prior to subcontracting samples to another laboratory) are responsible for obtaining client approval prior to outsourcing any samples. The laboratory must advise the client of a subcontract or work sharing arrangement in writing and, when possible, approval from the client must be retained in the project folder.

Note: In addition to the client, some regulating agencies (e.g., USDA) or contracts (e.g., certain USACE projects) may require notification prior to placing such work.

8.2. Qualifying and Monitoring Subcontractors

- 8.2.1. Whenever a PM or CSM becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:
 - 8.2.1.1. The first priority is to attempt to place the work in a qualified TestAmerica laboratory
 - 8.2.1.2. Firms specified by the client for the task. (Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be as simple as placing a copy of an e-mail from the client in the project folder.)
 - 8.2.1.3. Firms listed as pre-qualified and currently under a subcontract with TestAmerica. A listing of all approved subcontracting laboratories is available on the TestAmerica intranet site. Supporting documentation is maintained by Corporate offices and by the TestAmerica laboratory originally requesting approval of the subcontract lab. Verify necessary accreditation, where applicable (e.g., on the subcontractors TNI, A2LA accreditation, or State Certification).
 - 8.2.1.4. Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses
 - 8.2.1.5. TNI or A2LA-accredited laboratories
- 8.2.2. In addition, the firm must hold the appropriate certification to perform the work required
- 8.2.3. All TestAmerica Laboratories are pre-qualified for work sharing, provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. Refer to Corporate SOP CA-C-S-001, "Work Sharing Process."
- 8.2.4. When the potential subcontract laboratory has not been previously approved, CRMs or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director. The Laboratory Director requests that the QA Manager begin the process of approving the subcontract laboratory as outlined in Corporate SOP CA-L-S-002, Subcontracting Procedures. The client must provide acknowledgement that the samples can be sent to

that facility. (An e-mail is sufficient documentation; or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented.)

- 8.2.5. Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to the Corporate Quality Information Manager for review. Once all documents are reviewed for completeness, the Corporate QI Manager will forward the documents to the Purchasing Manager for formal signature and contractive with the laboratory. The approved vendor will be added to the subcontractor list on the intranet site, and the Finance Group is concurrently notified for J.D.Edwards.
- 8.2.6. The client must assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are to meet minimal standards. TestAmerica does not certify laboratories. The subcontractor is on our approved list, and can only be recommended to the extent that we would use them.
- 8.2.7. The status and performance of qualified subcontractors must be monitored periodically by the Corporate Contracts and/or Quality Departments. Any problems identified must be brought to the attention of TestAmerica's Corporate Finance or Corporate Quality personnel.
- 8.2.8. Complaints must be investigated. Documentation of the complaint, investigation, and corrective action must be maintained in the subcontractor file on the intranet site. Complaints are posted using the Vendor Performance Report.
- 8.2.9. Information must be updated on the intranet when new information is received from the subcontracted laboratories.
- 8.2.10. Subcontractors in good standing must be retained on the intranet listing. The QA Manager must notify all TestAmerica laboratories, Corporate Quality, and Corporate Contracts if any laboratory requires removal from the intranet site. This notification must be posted on the intranet site and e-mailed to all Laboratory Directors, QA Managers, and Sales Personnel.

8.3. Oversight and Reporting

- 8.3.1. The CRM or PM must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which reflect the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and the Legal & Contracts Director can tailor the document or assist with negotiations, if needed. The CRM or PM responsible for the project must advise and obtain client consent to the subcontract as appropriate, and

provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

- 8.3.2. Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented and retained in the project folder. For TestAmerica Laboratories, certifications can be viewed on the company's TotalAccess Database.
- 8.3.3. The Sample Control Department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.
- 8.3.4. All subcontracted samples must be accompanied by a TestAmerica Chain of Custody (COC). A copy of the original COC sent by the client must also be included with all samples workshared within TestAmerica. Client COCs are only forwarded to external subcontractors when samples are shipped directly from the project site to the subcontractor lab. Under routine circumstances, client COCs are not provided to external subcontractors.
- 8.3.5. Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.
- 8.3.6. Non-TNI accredited work must be identified in the subcontractor's report as non-TNI accredited work. If TNI accreditation is not required for the project, the report does not need to include this information.
- 8.3.7. Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratory EDD, i.e., imported, the report must explicitly indicate the specific lab that produced the data and identify the specific methods and samples.

Note: The results submitted by a TestAmerica work-sharing laboratory may be transferred electronically and the results reported by the TestAmerica work-sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

8.4. Contingency Planning

- 8.4.1. The Laboratory Director may waive the full qualification of a subcontractor process temporarily to meet emergency needs; however, this decision and justification must be documented in the project files, and the "Purchase Order Terms and Conditions for Subcontracted Laboratory Services" must be sent with the samples and Chain-of-Custody. In the

event this provision is utilized, the laboratory (e.g., QA Manager) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation requirements will still be applicable, but the subcontractor need not have signed a subcontract with TestAmerica at this time. The comprehensive approval process must then be initiated within 30 calendar days of subcontracting.

9. PURCHASING SERVICES AND SUPPLIES

9.1. Overview

- 9.1.1. Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet laboratory demand on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with TestAmerica's Corporate Controlled Purchases Procedure, SOP CW-F-S-007.
- 9.1.2. Contracts must be signed in accordance with TestAmerica's Corporate Authorization Matrix Policy, Policy CW-F-P-002. Request for Proposals (RFP's) must be issued when more information is required from the potential vendors than just price. Process details are available in TestAmerica's Corporate Procurement and Contracts Policy (Policy CW-F-P-004). RFP's allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

9.2. Glassware

- 9.2.1. Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass must be used where possible. For safety purposes, thick-wall glassware must be used where available.

9.3. Reagents, Standards & Supplies

- 9.3.1. Purchasing guidelines for equipment and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with TestAmerica's Corporate SOP on Solvent and Acid Lot Testing and Approval, SOP CA-Q-S-001.

9.4. Purchasing

9.4.1. Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP. The analyst may check the item out of the on-site consignment system that contains items approved for laboratory use. If the item is not in consignment, the analyst must provide the master item number, item description, package size, catalogue page number, and the quantity needed. If an item being ordered is not the exact item requested, approval must be obtained from the Operations Manager or Group Leader prior to placing the order. The purchasing manager places the order.

9.5. Receiving

9.5.1. It is the responsibility of the Warehouse Manager to receive the shipment. It is the responsibility of the analyst who ordered the materials to document the date materials were received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. Safety Data Sheets (SDSs) are kept on a backup disc located in the Wet Chemistry bullpen and available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

9.6. Specifications

- 9.6.1. Methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, analytical reagent grade will be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent. Specifications are listed in SOP NC-QA-017, Reagents and Standards.
- 9.6.2. Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory must contact the manufacturer to determine an expiration date.
- 9.6.3. The laboratory assumes a five-year expiration date on inorganic dry chemicals and solvents, unless noted otherwise by the manufacturer, or by the reference source method. Chemicals/solvents must not be used past the manufacturer's or SOP's expiration date unless "verified" (refer to Item 3 listed below).

- 9.6.4. An expiration date cannot be extended if the dry chemical/solvent is discolored or appears otherwise physically degraded, the dry chemical/solvent must be discarded.
- 9.6.5. Expiration dates can be extended if the dry chemical/solvent is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Method Blanks, LCS, etc.).
- 9.6.6. If the dry chemical/solvent is used for the preparation of standards, the expiration dates can be extended six months if the dry chemical/solvent is compared to an unexpired independent source in performing the method and the performance of the dry chemical/solvent is found to be satisfactory. The comparison must show that the dry chemical/solvent meets CCV limits. The comparison studies are maintained in the Reagent module of LIMS for each laboratory group.
- 9.6.7. Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.
- 9.6.8. Compressed gases in use are checked for pressure and secure positioning daily. To prevent a tank from going to dryness, or introducing potential impurities, the pressure should be closely watched as it decreases to approximately 15% of the original reading, at which point it should be replaced. For example, a standard sized laboratory gas cylinder containing 3,000 psig of gas should be replaced when it drops to approximately 500 psig. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.
- 9.6.9. Water used in the preparation of standards or reagents must have a conductivity of less than 1 $\mu\text{mho/cm}$ (or specific resistivity of greater than 1.0 mega ohm/cm) at 25°C. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Operations Manager and appropriate Technical Manager must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.
- 9.6.10. The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified "clean" by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.
- 9.6.11. Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.
- 9.6.12. Purchased bottle ware used for sampling must be certified clean, and the certificates must be maintained. If uncertified sampling bottle ware is

purchased, all lots must be verified clean prior to use. This verification must be maintained.

9.7. Storage

9.7.1. Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Storage conditions are per the Corporate Environmental Health & Safety Manual (Corporate Document CW-E-M-001) and method SOPs or manufacturer instructions.

9.8. Purchase Of Equipment/Instruments/Software

9.8.1. When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or group leader makes a supply request to the Operations Manager and/or the Laboratory Director. If they agree with the request the procedures outlined in TestAmerica's Corporate Policy CA-T-P-001, Qualified Products List, are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed, and Purchasing places the order.

9.8.2. Upon receipt of a new or used piece of equipment, an identification name is assigned, such as HP-20, and added to the equipment list described in Section 21 that is maintained by the QA Department, and I.T. must be notified so they can synchronize the instrument for backups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated followed by MDLs, and other relevant criteria (refer to Section 20). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department or QA Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained at the bench. All equipment manuals are also recorded in the QA department document tracking system.

9.9. Services

9.9.1. Service to analytical instruments (except analytical balances) is performed on an as-needed basis. Routine preventative maintenance is discussed in Section 20. The need for service is determined by analysts and/or Department Managers. The service providers that perform the services are approved by the Department Managers or Operations Manager.

9.10. Suppliers

9.10.1. TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Procurement and Contracts Policy (Policy CW-F-P-004). The level of control used in the selection

process is dependent on the anticipated spending amount and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers /vendors that have been approved for use.

- 9.10.2. Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.
- 9.10.3. Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report (CW-F-WI-009).
- 9.10.4. The Corporate Purchasing Group must work through the appropriate channels to gather the information required to clearly identify the problem and must contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.
- 9.10.5. As deemed appropriate, the Vendor Performance Reports must be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors
- 9.10.6. The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the JD Edwards purchasing system.

9.11. New Vendor Procedure

- 9.11.1. TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form.
- 9.11.2. New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department, Technical Services Director, and/or the Laboratory Director are consulted with vendor and product selection that have an impact on quality.

10. COMPLAINTS

10.1. OVERVIEW

- 10.1.1. The laboratory considers an effective client complaint handling process to be of significant business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that enables our operations to continually improve processes and improving client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.
 - 10.1.2. A client complaint is any expression of dissatisfaction with any aspect of our business services, (e.g., communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.
 - 10.1.3. The laboratory has procedures for addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.
 - 10.1.4. The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.
 - 10.1.5. The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented following SOPs NC-QA-029, Nonconformance and Corrective Action System, and CA-C-S-002, Complaint Handling and Service Recovery.
- 10.2. External Complaints
- 10.2.1. An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to CA-C-S-002, Complaint Handling and Service Recovery.
 - 10.2.2. Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints must be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.
 - 10.2.3. The general steps in the complaint handling process are:
 - 10.2.3.1. Receiving and Documenting Complaints
 - 10.2.3.2. Complaint Investigation and Service Recovery

10.2.3.3. Process Improvement

10.2.4. The laboratory must inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

10.2.5. Single event complaints are documented for tracking and trend analysis and initiate a non-conformance notification/memo (NCM). QA is notified and tracks the NCMs for identification of trends or systematic issues. A high-level or repeat complaint will initiate the corrective action process and will be documented with a formal Corrective Action Report (CAR). All client complaints are tracked in the corrective action worksheet maintained by the QA department.

10.3. Internal Complaints

10.3.1. Internal complaints include, but are not limited to errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and must follow the procedures outlined in Section 12. In addition, Corporate Management, Sales and Marketing, and Information Technology (IT) may initiate a complaint by contacting the laboratory or through the Corrective Action system described in Section 12.

10.3.2. All audit findings (internal and external) will initiate the CA process, are documented with a CAR, and are tracked in the QA CA tracking workbook.

10.4. Management Review

10.4.1. The number and nature of client complaints is reported by the QA Manager to the laboratory and QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 16)

11. CONTROL OF NON-CONFORMING WORK

11.1. OVERVIEW

11.1.1. When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies, and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a Corrective Action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the Corrective Action plan could include a more in depth investigation and a possible suspension of an

analytical method. In all cases, the actions taken are documented using the laboratory's Corrective Action system (refer to Section 12).

- 11.1.2. Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the supervisor for resolution. The supervisor may elect to discuss it with the Technical Director or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratory's corrective action system described in Section 12. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.
- 11.1.3. Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 19. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Operations Manager and QA Manager, documented and included in the project folder. Deviations must also be noted on the final report with a statement that the compound is not reported in compliance with TNI (or the analytical method) requirements and the reason. Data being reported to a non-TNI state would need to note the change made to how the method is normally run.
- 11.1.4. Note: The laboratory must implement Corrective Action procedures to resolve the deviation and limit qualification of the final results. The laboratory is not permitted to deviate from its VAP approved SOP if it intends to attest under affidavit that the "results" are VAP certified. When all Corrective Actions listed in the SOP have been exhausted, it may be necessary to use technical judgment in which case the decision process and rationale will be presented in the final report and/or affidavit and the data will be noted as 'not VAP certified' on the affidavit.

11.2. Responsibilities And Authorities

- 11.2.1. TestAmerica's Corporate SOP entitled Internal Investigation of Potential Data Discrepancies and Determination for Data Recall (SOP CW-L-S-002) outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or violations of the TestAmerica's data integrity policies as well as the policies and procedures related to the determination of the potential need to recall data.

- 11.2.2. Under certain circumstances the Laboratory Director, Operations Manager, Project Manager, or a member of the QA team may exceptionally authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client must be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's Corrective Action procedures described in Section 12. This information may also need to be documented in logbooks and/or data review as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.
 - 11.2.3. Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility Senior Management within 24 hours. The Senior Management staff is comprised of the Laboratory Director, QA Manager, Customer Service Manager, Operations Manager, I.T. Manager, H.R. Manager, PM Manager, and Technical Director. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an Ethics and Compliance Officer (ECO), Director of Quality and Client Advocacy, and the laboratory's Corporate Quality Director within 24 hours of discovery.
 - 11.2.4. Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.
 - 11.2.5. The Laboratory Director, QA Manager, ECOs, Corporate Quality Director, Executive VP of Operations, and the Corporate Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.
- 11.3. Evaluation Of Significance And Actions Taken
- 11.3.1. For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.
 - 11.3.2. TestAmerica's Corporate Data Investigation and Recall Procedure (SOPCW-L-S-002) distinguishes between situations when it would be appropriate for laboratory management to make the decision on the need for client notification (written or verbal) and data recall (report revision) and when the decision must be made with the assistance of the ECOs and Corporate Management. Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/Corrective Action reporting in lieu of the data recall determination form contained in TestAmerica Corporate SOPCW-L-S-002.

11.4. Prevention Of Nonconforming Work

11.4.1. If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's Corrective Action system. Periodically, as defined by the laboratory's preventive action schedule (monthly), the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's Corrective Action process may be followed.

11.5. Method Suspension/Restriction (Stop Work Procedures)

11.5.1. In some cases it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 11.2, Paragraph 5.

11.5.2. Prior to suspension/restriction, confidentiality must be respected, and the problem with the required corrective and preventive action must be stated in writing and presented to the Laboratory Director.

11.5.3. The Laboratory Director must arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting must be held to confirm that there is a problem, that suspension/restriction of the method is required and must be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target, or test fully back on line.

11.5.4. The QA Manager must also initiate a Corrective Action report as described in Section 12 if one has not already been started. A copy of any meeting notes and agreed-upon steps should be faxed or e-mailed by the laboratory to the appropriate General Manager and member of Corporate QA. This fax/e-mail acts as notification of the incident.

11.5.5. After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the Internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction, i.e., Project Management, Log-in, etc. Clients must NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

11.5.6. Within 72 hours, the QA Manager must determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Technical Director, QA Manager, Group Leader) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project

Management and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval is given by final signature on the completed Corrective Action report.

12. CORRECTIVE ACTION

12.1. Overview

12.1.1. A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the Corrective Action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Nonconformance Memos (NCM) are used to document excursions for SOPs, control limits, holding times, etc. A Corrective Action report is used to document and communicate actions taken to investigate, correct, and prevent recurrence of a more significant problem. All incidents are documented and tracked in the QA corrective action database. A brief summary of the system is described below, for more detail refer to SOP NC-QA-029.

12.2. General

12.2.1. Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, PT performance, client complaints, staff observation, etc.

12.2.2. The purpose of a Corrective Action system is to:

12.2.2.1. Identify non-conformance events and assign responsibility(s) for investigating.

12.2.2.2. Resolve non-conformance events and assign responsibility for any required corrective action.

12.2.2.3. Identify systematic problems before they become serious.

12.2.2.4. Identify and track client complaints and provide resolution

12.2.2.5. Improve systems and/or processes

12.3. Non-Conformance Memo (NCM)

12.3.1. An NCM is used to document the following types of one-off corrective actions:

12.3.1.1. Deviations from an established procedure or SOP

12.3.1.2. QC outside of limits (non-matrix related)

12.3.1.3. Isolated reporting / calculation errors

12.3.1.4. Client Complaints

12.3.1.5. Discrepancies in materials / goods received vs. manufacturer packing slips

12.4. Corrective Action Report (CAR)

12.4.1. A CAR is used to document the following types of investigations and resulting corrective actions:

12.4.1.1. Questionable trends that are found in the review of NCMs.

12.4.1.2. Issues found while reviewing NCMs that warrant further investigation.

12.4.1.3. Internal and external audit findings

12.4.1.4. Failed or unacceptable PT results.

12.4.1.5. Corrective actions that cross multiple departments in the laboratory.

12.4.1.6. Systematic reporting / calculation errors

12.4.1.7. Client complaints

12.4.1.8. Data recall investigations

12.4.1.9. Identified poor process or method performance trends

12.4.1.10. Excessive revised reports

12.4.2. This will provide background documentation to enable root cause analysis and preventive action.

12.5. Closed Loop Corrective Action Process

12.5.1. Any employee in the company can initiate a Corrective Action. There are four main components to a closed-loop Corrective Action process once an issue has been identified--Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

12.6. Root Cause Analysis

- 12.6.1. Upon discovery of a non-conformance event, the event must be defined and documented. An NCM or CA must be initiated, someone is assigned to investigate the issue, and the event is investigated for cause. Table 12-1 provides some general guidelines on determining responsibility for assessment. SOP NC-QA-029, Nonconformance and Corrective Action System, establishes procedures for the identification and documentation of nonconformances and corrective actions and the steps taken to investigate and respond as a result of these events.
- 12.6.2. The root cause analysis step is the key to the process as a long-term corrective action cannot be determined until the root cause is determined.
- 12.6.3. Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness.
- 12.6.4. Systematically analyze and document the Root Causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the Root Cause data from these incidents to identify root causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.
- 12.6.5. Identify the one event associated with problem and ask why this event occurred. Brainstorm the root causes of failures; for example, by asking why events occurred or conditions existed; and then why the cause occurred five consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.
- 12.6.6. Root cause analysis does not mean the investigation is over. Look at technique, or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed, and continue to plague the laboratory or operation.
- 12.6.7. If the root cause is not readily obvious, the Group Leader, Technical Director, Lab Director, QA Manager, or designee is consulted. A team may be assigned to investigate and will collaborate on the resolution of the problem.

12.7. Selection and Implementation of Corrective Actions

- 12.7.1. Where corrective action is needed, the laboratory must identify potential corrective actions. The action(s) most likely to eliminate the problem and

prevent recurrence are selected and implemented. Responsibility for implementation is assigned.

12.7.2. Corrective actions must be, to a degree, appropriate to the magnitude of the problem identified through the cause analysis.

12.7.3. Whatever corrective action is determined to be appropriate, the laboratory must document and implement the changes. The NCM or CAR is used for this documentation. NCMs are tracked in the laboratory LIMS NCM module. Corrective Actions are tracked in the QA department CA tracking workbook.

12.8. Monitoring of the Corrective Actions

12.8.1. The Group Leader or Technical Director and QA Manager is responsible to ensure the corrective action taken was effective.

12.8.2. Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. The Technical Director are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.

12.8.3. Each corrective action is recorded in the QA corrective action database for tracking to completion.

12.8.4. Each NCM is recorded in TALS and available for tracking purposes and a summary report of all NCMs can be reviewed to evaluate whether an on-going problem may exist by assessing trending.

12.8.5. The QA Manager reviews monthly NCMs for trends. Highlights are included in the QA monthly report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.

12.8.6. Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

12.9. Follow-up Audits

12.9.1. Follow-up audits may be initiated by the QA Manager and must be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.

12.9.2. These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered. (Also refer to Section 15.2.4, Special Audits.)

12.10. Technical Corrective Actions

- 12.10.1. In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 11 for information regarding the control of non-conforming work). The documentation of these procedures is through the use of an NCM.
- 12.10.2. Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs.
- 12.10.3. Table 12-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions, and QA Manual Sections 19 and 20. The QA Manager reviews all corrective actions monthly, at a minimum, and highlights are included in the QA monthly report.
- 12.10.4. To the extent possible, samples must be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data must be reported with an appropriate data qualifier and/or the deficiency must be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by a written NCM and appropriate corrective action (e.g., re-analysis) is taken and documented.

12.11. Basic Corrections

- 12.11.1. When mistakes occur in records, each mistake must be crossed-out with a single line [not obliterated (e.g. no White-Out)], and the correct value entered alongside. All such corrections must be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original "uncorrected" file must be maintained intact and a second "corrected" file is created.
- 12.11.2. This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.
- 12.11.3. When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) must also be documented.

Table 12-1: General Corrective Action Procedures

Inorganic Laboratory Quality Control Samples

| Analysis | *QC Sample | Method | NPDES (1) | Method | RCRA (SW846) (2) |
|------------|--|---|--|--------|------------------|
| Alkalinity | Method Blank (MB) | 310.1 2320B | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank | -- | NA |
| | Laboratory Control Sample (LCS) | 310.1 2320B | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples | -- | NA |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 310.1 2320B | Total alkalinity: 1 per batch of 20 samples | -- | NA |
| | Duplicate (DU) | 310.1 2320B | For carbonate, bicarbonate, hydroxide, alkalinity, and total alkalinity by SM2320B <u>Frequency:</u> 1 per batch of 10 samples <u>Criteria</u> 310.1: ? 20 % RPD(3) <u>Criteria</u> 2320B: ? 25 % RPD(3) <u>Corrective Action:</u> Flag data outside of limit. | -- | NA |
| Ammonia | Method Blank (MB) | 350.2 350.3 SM4500 NH3-C and D | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method | -- | NA |

| Analysis | *QC Sample | Method | NPDES (1) | Method | RCRA (SW846) (2) |
|---------------|--|--|---|--------|------------------|
| | | | blank | | |
| | Laboratory Control Sample (LCS) | 350.2 350.3 SM4500 NH3-C and D | Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within control limits, rerun all associated samples | -- | NA |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 350.2 350.3 SM4500 NH3-C and D | Frequency: 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit | -- | NA |
| | Duplicate (DU) | 350.2 350.3 SM4500 NH3-C and D | N/A | — | N/A |
| Ammonia (TKN) | Method Blank (MB) | 351.3 SM4500 N- Org C / SM4500NH3-C | Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank | — | N/A |
| | Laboratory Control Sample (LCS) | 351.3 SM4500 N- Org C / SM4500NH3-C | Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples | — | N/A |
| | Matrix Spike/Matrix | 351.3 | Frequency: 1 per 20 samples, minimum of | — | N/A |

| Analysis | *QC Sample | Method | NPDES (1) | Method | RCRA (SW846) (2) |
|--|--|--|---|--------|---|
| | Spike Duplicate (MS/MSD) | SM4500 N- Org C / SM4500NH3- C | one per batch of samples processed <u>Criteria:</u> Must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit | | |
| | Duplicate (DU) | 351.3 SM4500 N- Org C / SM4500NH3- C | N/A | — | N/A |
| BOD | Method Blank (MB) | 405.1 SM5210B | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit. <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank | — | N/A |
| | Laboratory Control Sample (LCS) | 405.1 SM5210B | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples. <u>Criteria:</u> Percent recovery must be within laboratory control limits. <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples | — | N/A |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 405.1 SM5210B | N/A | — | N/A |
| | Duplicate (DU) | 405.1 SM5210B | N/A | — | N/A |
| Anions : Bromide Chloride Fluoride Sulfate Nitrate Nitrite Ortho-phos | Method Blank (MB) | 300.0 (4) | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit | 9056A | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration |

| Analysis | *QC Sample | Method | NPDES (1) | Method | RCRA (SW846) (2) |
|----------|--|------------------|---|--------|---|
| | | | <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank | | must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank |
| | Laboratory Control Sample (LCS) | 300.0 (4) | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within control limits, rerun all associated samples | 9056A | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within control limits, rerun all associated samples |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 300.0 (4) | <u>Frequency:</u> 1 per 10 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit | 9056A | <u>Frequency:</u> 1 per 10 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit |
| | Duplicate (DU) | 300.0 (4) | N/A | 9056A | N/A |
| COD | Method Blank (MB) | 410.4 SM5220D | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank. | — | N/A |
| | Laboratory Control Sample | 410.4 SM5220D | <u>Frequency:</u> 1 with each batch of samples processed not to | — | N/A |

| Analysis | *QC Sample | Method | NPDES (1) | Method | RCRA (SW846) (2) |
|----------|--|-------------------|---|--------|---|
| | (LCS) | | exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples | | |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 410.4 SM5220D | <u>Frequency:</u> 1 per 10 samples, minimum of one per batch of samples processed <u>Criteria:</u> Must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit | — | N/A |
| | Duplicate (DU) | 410.4 SM5220D | N/A | — | N/A |
| Chloride | Method Blank (MB) | 325.2 SM4500 Cl-E | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank | 9251 | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank |
| | Laboratory Control Sample (LCS) | 325.2 SM4500 Cl-E | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within control limits, rerun all associated samples | 9251 | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within control limits, rerun all associated samples |
| | Matrix Spike/Matrix Spike | 325.2 SM4500 Cl-E | <u>Frequency:</u> 1 per 10 samples, minimum of one per batch of | 9251 | <u>Frequency:</u> 1 per 10 samples, minimum of one |

| Analysis | *QC Sample | Method | NPDES (1) | Method | RCRA (SW846) (2) |
|--------------------|--|----------------------|---|----------------|--|
| | Duplicate (MS/MSD) | | samples processed <u>Criteria:</u> Percent recovery must be within laboratory <u>Control limits</u> Flag data outside of limit | | per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit |
| | Duplicate (DU) | 325.2 SM4500 CI-E | N/A | 9251 | N/A |
| Chlorine, Residual | Method Blank (MB) | 330.5 SM4500 CI-G | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank | — | N/A |
| | Laboratory Control Sample (LCS) | 330.5 SM4500 CI-G | N/A | — | N/A |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 330.5 SM4500 CI-G | N/A | — | N/A |
| | Duplicate (DU) | 330.5 SM4500 CI-G | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> =20 % RPD(3) <u>Corrective Action:</u> Flag data outside of limit. | — | N/A |
| Chromium (Cr+6) | Method Blank (MB) | 3500 Cr-B | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank unless the method blank is above | 7196A 3060A | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with |

| Analysis | *QC Sample | Method | NPDES (1) | Method | RCRA (SW846) (2) |
|------------------------|--|------------------|--|----------------|---|
| | | | RL, and samples are ND. | | unacceptable method blank unless the method blank is above RL, and samples are ND. |
| | Laboratory Control Sample (LCS) | 3500 Cr-B | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples | 7196A 3060A | <u>Frequency:</u> 1 soluble and 1 insoluble with each batch of solid samples, 1 with each batch of water samples processed not to exceed 20 samples prepped <u>Criteria:</u> percent recovery for water must be within $\pm 15\%$ and for solids must be within $\pm 20\%$ <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 3500 Cr-B | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Must be within laboratory QC limits <u>Corrective Action:</u> Flag data outside of limit | 7196A 3060A | <u>Frequency:</u> 1 with each batch of water samples processed not to exceed 20 samples <u>Criteria:</u> Advisory limits are 75% - 125% recovery <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike <i>The Method of Standard Addition is used for solid samples in lieu of a Matrix Spike.</i> |
| | Duplicate (DU) | 3500 Cr-B | N/A | 7196A 3060A | N/A |
| Conductivity, Specific | Method Blank (MB) | 120.1 SM2510B | N/A | 9050A | N/A |
| | Laboratory Control Sample (LCS) | 120.1 SM2510B | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples | 9050A | <u>Frequency:</u> 1 with each batch of samples processed not to |

| Analysis | *QC Sample | Method | NPDES (1) | Method | RCRA (SW846) (2) |
|---------------------------------|--|---------------|--|--------|--|
| | | | Criteria: Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples | | exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 120.1 SM2510B | N/A | 9050A | N/A |
| | Duplicate (DU) | 120.1 SM2510B | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 10 samples <u>Criteria:</u> =20 % RPD(3) <u>Corrective Action:</u> Flag data outside of limit. | 9050A | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 10 samples <u>Criteria:</u> =20 % RPD(3) <u>Corrective Action:</u> Flag data outside of limit. |
| Cyanide (Weak Acid Dissociable) | Method Blank (MB) | SM4500 CN-I | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank | — | N/A |
| | Laboratory Control Sample (LCS) | SM4500 CN-I | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples | — | N/A |
| | Matrix | SM4500 CN-I | <u>Frequency:</u> 1 per 20 | — | N/A |

| Analysis | *QC Sample | Method | NPDES (1) | Method | RCRA (SW846) (2) |
|--------------------|--|----------------------|--|----------------|--|
| | Spike/Matrix Spike Duplicate (MS/MSD) | | samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit | | |
| | Duplicate (DU) | SM4500 CN-I | N/A | — | N/A |
| Cyanide (Amenable) | Method Blank (MB) | 335.1 SM4500 CN-G | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank | 9012A 9012B | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank |
| | Laboratory Control Sample (LCS) | 335.1 SM4500 CN-G | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples | 9012A 9012B | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 335.1 SM4500 CN-G | <u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit | 9012A 9012B | <u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> |

| Analysis | *QC Sample | Method | NPDES (1) | Method | RCRA (SW846) (2) |
|-----------------|--|---|---|----------------|---|
| | | | | | Flag data outside of limit |
| | Duplicate (DU) | 335.1 SM4500 CN-G | N/A | 9012A 9012B | N/A |
| Cyanide (Total) | Method Blank (MB) | 335.2 335.4 SM4500 CN-E 335.2-CLP-M (Ohio VAP) | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank unless the method blank is above RL, and samples are ND. | 9012A 9012B | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank unless the method blank is above RL, and samples are ND. |
| | Laboratory Control Sample (LCS) | 335.2 335.4 SM4500 CN-E 335.2-CLP-M (Ohio VAP) | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples | 9012A 9012B | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 335.2 335.4 SM4500 CN-E 335.2-CLP-M (Ohio VAP) | <u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit | 9012A 9012B | <u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit |

| Analysis | *QC Sample | Method | NPDES (1) | Method | RCRA (SW846) (2) |
|----------------------|--|---|---|----------------|---|
| | Duplicate (DU) | 335.2 335.4 SM4500 CN-E 335.2-CLP-M (Ohio VAP) | N/A | 9012A 9012B | N/A |
| Dissolve Oxygen (DO) | Method Blank (MB) | 360.1 SM4500 O-G | N/A | — | N/A |
| | Laboratory Control Sample (LCS) | 360.1 SM4500 O-G | N/A | — | N/A |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 360.1 SM4500 O-G | N/A | — | N/A |
| | Duplicate (DU) | 360.1 SM4500 O-G | N/A | — | N/A |
| Flashpoint | Method Blank (MB) | | N/A | 1010 1010A | N/A |
| | Laboratory Control Sample (LCS) | | N/A | 1010 1010A | N/A |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | | N/A | 1010 1010A | N/A |
| | Duplicate (DU) | | Frequency: 1 per 20 samples per matrix, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit | 1010 1010A | Frequency: 1 per 20 samples per matrix, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit |
| Fluoride (ISE) | Method Blank (MB) | 340.2 SM4500 F-C | Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with | — | N/A |

| Analysis | *QC Sample | Method | NPDES (1) | Method | RCRA (SW846) (2) |
|----------|--|-----------------------|--|--------|------------------|
| | | | unacceptable method blank | | |
| | Laboratory Control Sample (LCS) | 340.2 SM4500 F-C | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples | — | N/A |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 340.2 SM4500 F-C | <u>Frequency:</u> 1 per 20 samples by <u>Criteria:</u> Must be within laboratory QC limits <u>Corrective Action:</u> Flag data outside of limit | — | N/A |
| | Duplicate (DU) | 340.2 SM4500 F-C | N/A | — | N/A |
| Hardness | Method Blank (MB) | 130.2 SM2340B SM2340C | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank | — | N/A |
| | Laboratory Control Sample (LCS) | 130.2 SM2340B SM2340C | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples | — | N/A |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 130.2 SM2340B SM2340C | <u>Method 130.2:</u> 1 per 20 samples <u>Method 2340B:</u> <u>Frequency, Criteria, and Corrective Action:</u> See ICP Metals Method 200.7 | — | N/A |

| Analysis | *QC Sample | Method | NPDES (1) | Method | RCRA (SW846) (2) |
|---------------------------|--|-----------------------------|---|----------------|---|
| | | | Requirements | | |
| | Duplicate (DU) | 130.2 SM2340B SM2340C | Frequency: One with every 10 samples. | — | N/A |
| Iron (Ferrous and Ferric) | Method Blank (MB) | SM3500 Fe-B | Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank | — | N/A |
| | Laboratory Control Sample (LCS) | SM3500 Fe-B | Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples | — | N/A |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | SM3500 Fe-B | Frequency: 1 every 20 samples Criteria: Must be within laboratory QC limits Corrective Action: Flag associated data outside of limit | — | N/A |
| | Duplicate (DU) | SM3500 Fe-B | N/A | — | N/A |
| Paint Filter | Method Blank (MB) | — | N/A | 9095A 9095B | N/A |
| | Laboratory Control Sample (LCS) | — | N/A | 9095A 9095B | N/A |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | — | N/A | 9095A 9095B | N/A |
| | Duplicate (DU) | — | N/A | 9095A 9095B | Frequency: Two per batch of 20 samples. |
| pH | Method Blank (MB) | 150.1 SM4500 H+B | N/A | 9040B 9040C | N/A |

| Analysis | *QC Sample | Method | NPDES (1) | Method | RCRA (SW846) (2) |
|-----------|--|---------------------|--|--|--|
| | | | | 9045C 9045D 9041 | |
| | Laboratory Control Sample (LCS) | 150.1 SM4500 H+B | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples | 9040B 9040C 9045C 9045D 9041 | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 150.1 SM4500 H+B | N/A | 9040B 9040C 9045C 9045D 9041 | N/A |
| | Duplicate (DU) | 150.1 SM4500 H+B | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 10 samples per matrix <u>Criteria:</u> =20 % RPD(3) limit <u>Corrective Action:</u> Flag data outside of limit. | 9040B 9040C 9045C 9045D 9041 | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 10 samples per matrix <u>Criteria:</u> = 20 % RPD(3) limit <u>Corrective Action:</u> Flag data outside of limit. |
| Phenolics | Method Blank (MB) | 420.1 | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank | 9065 | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank |
| | Laboratory Control | 420.1 | <u>Frequency:</u> 1 with each batch of samples | 9065 | <u>Frequency:</u> 1 with each batch of |

| Analysis | *QC Sample | Method | NPDES (1) | Method | RCRA (SW846) (2) |
|------------------------------|--|------------------|--|--------|--|
| | Sample (LCS) | | processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples | | samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 420.1 | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data associated with unacceptable matrix spike | 9065 | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data associated with unacceptable matrix spike |
| | Duplicate (DU) | 420.1 | N/A | 9065 | N/A |
| Phosphorus (Total and Ortho) | Method Blank (MB) | 365.1 SM4500 P-E | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank | — | N/A |
| | Laboratory Control Sample (LCS) | 365.1 SM4500 P-E | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory | — | N/A |

| Analysis | *QC Sample | Method | NPDES (1) | Method | RCRA (SW846) (2) |
|-----------------|--|--|--|--------|------------------|
| | | | control limits, rerun all associated samples | | |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 365.1 SM4500 P-E | <u>Frequency:</u> 1 per 20 samples <u>Criteria:</u> Must be within laboratory QC limits <u>Corrective Action:</u> Flag data outside of limit | — | N/A |
| | Duplicate (DU) | 365.1 SM4500 P-E | N/A | — | N/A |
| Solids in Water | Method Blank (MB) | 160.1 160.2 160.3 SM2540B SM2540C SM2540D | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> If analyte level in method blank is \pm RL for the analyte of interest in the sample, all associated samples with reportable levels of analyte are re-prepared and re-analyzed. | — | N/A |
| | Laboratory Control Sample (LCS) | 160.1 160.2 160.3 SM2540B SM2540C SM2540D | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, re-prepare and rerun all associated samples | — | N/A |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 160.1 160.2 160.3 SM2540B SM2540C SM2540D | N/A | — | N/A |
| | Duplicate (DU) | 160.1 160.2 160.3 SM2540B SM2540C SM2540D | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 10 samples <u>Criteria:</u> Sample results should agree within 20%. | — | N/A |
| | | | | | |

| Analysis | *QC Sample | Method | NPDES (1) | Method | RCRA (SW846) (2) |
|---------------------------|--|---------------|---|--------|------------------|
| Solids (Settleable) | Method Blank (MB) | 160.5 SM2540F | Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: If analyte level in method blank is \pm RL for the analyte of interest in the sample, all associated samples with reportable levels of analyte are re-prepared and re-analyzed. | — | N/A |
| | Laboratory Control Sample (LCS) | 160.5 SM2540F | Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, re-prepare and rerun all associated samples | — | N/A |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 160.5 SM2540F | N/A | — | N/A |
| | Duplicate (DU) | 160.5 SM2540F | N/A | — | N/A |
| Solids (Percent Moisture) | Method Blank (MB) | 160.3 (mod) | N/A | — | N/A |
| | Laboratory Control Sample (LCS) | 160.3 (mod) | N/A | — | N/A |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 160.3 (mod) | N/A | — | N/A |
| | Duplicate (DU) | 160.3 (mod) | Frequency: 1 with each batch of samples processed not to exceed 10 samples Criteria: Sample results | — | N/A |

| Analysis | *QC Sample | Method | NPDES (1) | Method | RCRA (SW846) (2) |
|----------------------------|--|----------------------|--|---------------|---|
| | | | should agree within 20%. | | |
| Sulfate (Turbidimetric) | Method Blank (MB) | 375.4 | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank | 9038 | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank |
| | Laboratory Control Sample (LCS) | 375.4 | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples | 9038 | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within $\pm 15\%$ <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples |
| | Matrix Spike/Matrix Duplicate (MS/MSD) | 375.4 | <u>Frequency:</u> 1 per 10 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit | 9038 | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 10 samples <u>Criteria:</u> Limits are 75% - 125% recovery <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike |
| | Duplicate (DU) | 375.4 | N/A | 9038 | N/A |
| Sulfide | Method Blank (MB) | 376.1 SM4500 S2-F | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration | 9030B 9034 | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 |

| Analysis | *QC Sample | Method | NPDES (1) | Method | RCRA (SW846) (2) |
|----------------------------|--|-------------------|--|--------------------------|--|
| | | | must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank | | samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank |
| | Laboratory Control Sample (LCS) | 376.1 SM4500 S2-F | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples | 9030B 9034 | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 376.1 SM4500 S2-F | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit | 9030B 9034 | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit |
| | Duplicate (DU) | 376.1 SM4500 S2-F | N/A | 9030B 9034 | N/A |
| Total Organic Carbon (TOC) | Method Blank (MB) | 415.1 SM5310C | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method | 9060 9060A Walkley Black | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples. <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples |

| Analysis | *QC Sample | Method | NPDES (1) | Method | RCRA (SW846) (2) |
|-----------|--|---------------|--|--------------------------|--|
| | | | blank | | associated with unacceptable method blank |
| | Laboratory Control Sample (LCS) | 415.1 SM5310C | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples | 9060 9060A Walkley Black | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples Method 9060 requires and LCS every 15 samples. <u>Criteria:</u> percent recovery must be within laboratory control limits <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 415.1 SM5310C | <u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit | 9060 9060A Walkley Black | <u>Frequency:</u> (Water matrix only) 1 with each batch of samples processed not to exceed 20 samples. Method 9060 requires a matrix spike every 10 samples. <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Reanalyze if sample remaining. If not, flag data associated with unacceptable Matrix Spike |
| | Duplicate (DU) | 415.1 SM5310C | N/A | 9060 9060A Walkley Black | <u>Frequency:</u> (Solid matrix only) One for every 10 samples. <u>Criteria:</u> = 20% RPD between sample results. <u>Corrective Action:</u> Flag data with unacceptable RPD |
| Turbidity | Method Blank | 180.1 | <u>Frequency:</u> 1 with each | — | N/A |

| Analysis | *QC Sample | Method | NPDES (1) | Method | RCRA (SW846) (2) |
|-------------------|--|----------------|--|----------------|--|
| | (MB) | | batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank | | |
| | Laboratory Control Sample (LCS) | 180.1 | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples | — | N/A |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 180.1 | N/A | — | N/A |
| | Duplicate (DU) | 180.1 | <u>Frequency:</u> 1 per 10 samples <u>Criteria:</u> Must be within laboratory QC limits <u>Corrective Action:</u> Flag data outside of limit | — | N/A |
| Specific Gravity | Method Blank (MB) | SM2710 F | N/A | — | N/A |
| | Laboratory Control Sample (LCS) | SM2710 F | N/A | — | N/A |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | SM2710 F | N/A | — | N/A |
| | Duplicate (DU) | SM2710 F | <u>Frequency:</u> 1 per 20 samples <u>Criteria:</u> Must be within laboratory QC limits <u>Corrective Action:</u> Flag data outside of limit | — | N/A |
| Mercury by CVAA & | Method Blank (MB) | 245.1 1631E | <u>Frequency:</u> 1 with each batch of samples | 7470A 7471A | <u>Frequency:</u> 1 with each batch of |

| Analysis | *QC Sample | Method | NPDES (1) | Method | RCRA (SW846) (2) |
|----------|--|----------------|---|-------------------------|--|
| CVAF | | | processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank, unless the method blank is above RL, and samples are ND. | 7471B | samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank, unless the method blank is above RL, and samples are ND. |
| | Laboratory Control Sample (LCS) | 245.1 1631E | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> For 245.1 percent recovery of analyte must be within ± 20 %. For 1631E the percent recovery is ± 23 % <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS, unless samples are ND, results are reported. | 7470A 7471A 7471B | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery of analyte must be within ± 20 % <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS samples are ND, results are reported. Exception: If samples are ND, results are reported. |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 245.1 1631E | <u>Frequency:</u> with each batch of samples processed not to exceed 20 samples. 1631E frequency is 1 in 10 samples, 71-125% <u>Criteria:</u> For Method 245.1 recovery should be within 70-130 % <u>Corrective Action:</u> Flag data associated with unacceptable MS. | 7470A 7471A 7471B | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> For Method 7470A, recovery should be within 75-125 %. For Methods 7471A and 7471B, a criterion is 70-130%. <u>Corrective Action:</u> |

| Analysis | *QC Sample | Method | NPDES (1) | Method | RCRA (SW846) (2) |
|-------------------------|---------------------------------|----------------|--|---------------------------------|--|
| | | | | | Flag data associated with unacceptable MS. |
| | Duplicate (DU) | 245.1 1631E | N/A | 7470A 7471A 7471B | N/A |
| Metals (ICP and ICP/MS) | Method Blank (MB) | 200.7 200.8 | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit. Concentration less than reporting with the exception of lab common contaminants. Sample results <RL are also valid. <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank unless the method blank is above RL, and samples are ND. | 6010B 6010C 6020 6020A | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit. Concentration less than reporting with the exception of lab common contaminants. Sample results <RL are also valid. <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank unless the method blank is above RL, and samples are ND. |
| | Laboratory Control Sample (LCS) | 200.7 200.8 | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery of analyte must be \pm 85-115%. If LCS is biased high and samples are <RL, the results are valid. <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS If samples are ND, results are reported. | 6010B 6010C 6020 6020A | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery of analyte must be \pm 20 %. If LCS is biased high and samples are <RL, the results are valid. <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS If samples are ND, results are |

| Analysis | *QC Sample | Method | NPDES (1) | Method | RCRA (SW846) (2) |
|----------|--|----------------|---|---------------------------------|---|
| | | | | | reported. |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 200.7 200.8 | Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Limits for percent recovery are 70-130%, RPD(3) must be within 20% <u>Corrective Action:</u> Flag data associated with unacceptable matrix spike | 6010B 6010C 6020 6020A | Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Limits for percent recovery must be within laboratory limits. RPD(3) must be within 20% <u>Corrective Action:</u> Flag data associated with unacceptable matrix spike |
| | Duplicate (DU) | 200.7 200.8 | N/A | 6010B 6010C 6020 6020A | N/A |
| | Serial Dilution (SD) | 200.7 200.8 | Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> 10% difference. 10% difference only applied if sample results are >50 times MDL. <u>Corrective Action:</u> Flag data associated with unacceptable serial dilution | 6010B 6010C 6020 6020A | Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> 10% difference. 10% difference only applied if sample results are >50 times MDL. <u>Corrective Action:</u> Flag data associated with unacceptable serial dilution |
| | Post Digestion Spike (PDS) | 200.7 200.8 | Frequency: When dilution test fails to meet criteria. <u>Criteria:</u> Recovery must be within 75 – 125%. <u>Corrective Action:</u> Flag results for matrix interference. | 6010B 6010C 6020 6020A | Frequency: When dilution test fails to meet criteria. <u>Criteria:</u> Recovery must be within 75 – 125%. <u>Corrective Action:</u> Flag results for matrix interference. |

Footnotes

1. *National Pollutant Discharge Elimination System*

2. *Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996), Update IV (2007).*
3. *RPD-Relative Percent Difference*
4. *Method not listed in 40 CFR Part 136. Method 300.0 is a proposed 40CFR method. Specific state and/or region approval is required for NPDES.*

Organic Laboratory Quality Control Samples

| Analysis | *QC Sample | Method | NPDES 1 | Method | RCRA (SW846) 2 |
|------------|--|--------|---------|--------|--|
| Herbicides | Method Blank (MB) | -- | NA | 8151A | Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable method blank |
| | Laboratory Control Sample (LCS) | -- | NA | 8151A | Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: Percent recovery for each analyte must be within laboratory control limits Corrective Action: Re-extract and reanalyze all samples associated with unacceptable LCS |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | -- | NA | 8151A | Frequency: 1 with each extraction batch of samples not to exceed 20 samples Criteria: percent recovery for each analyte should be within laboratory |

| Analysis | *QC Sample | Method | NPDES 1 | Method | RCRA (SW846) 2 |
|---------------------|---------------------------------|--------|--|---------------------------------|--|
| | | | | | control limits Corrective Action: Flag data associated with unacceptable matrix spike sample |
| | Duplicate (DU) | -- | NA | 8151A | N/A |
| | Surrogates (Surr) | -- | NA | 8151A | Surrogates spiked into method blank and all samples (QC included) Method Blank Criteria and LCS: All surrogates must fall within laboratory established control limits before sample analysis may proceed. Sample Criteria: Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria |
| | Internal Standards (IS) | -- | NA | 8151A | Optional |
| Pesticides and PCBs | Method Blank (MB) | 608 | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank | 8081A 8081B 8082 8082A | <u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Re-prepare and reanalyze all samples associated with unacceptable method blank |
| | Laboratory Control Sample (LCS) | 608 | <u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery must be within control limits given in method for each analyte | 8081A 8081B 8082 8082A | <u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery must be within control limits given in method for each analyte <u>Corrective Action:</u> |

| Analysis | *QC Sample | Method | NPDES 1 | Method | RCRA (SW846) 2 |
|--|--|--------|--|---------------------------------|--|
| | | | <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS | | Rerun all samples associated with unacceptable LCS |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 608 | <u>Frequency:</u> 1 per 10 samples from each site or 1 per month, whichever is more frequent <u>Criteria:</u> percent recovery for each analyte should be within advisory limits given in method <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike | 8081A 8081B 8082 8082A | <u>Frequency:</u> 1 per 10 samples from each site or 1 per month, whichever is more frequent <u>Criteria:</u> percent recovery for each analyte should be within advisory limits given in method <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike |
| | Duplicate (DU) | 608 | N/A | 8081A 8081B 8082 8082A | N/A |
| | Surrogates (Surr) | 608 | <u>Frequency:</u> Surrogates spiked into method blank and all samples (QC included) <u>Method Blank Criteria and LCS:</u> Results must fall within laboratory established control limits <u>Sample Criteria:</u> Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria | 8081A 8081B 8082 8082A | <u>Frequency:</u> Surrogates spiked into method blank and all samples (QC included) <u>Method Blank Criteria and LCS:</u> Results must fall within laboratory established control limits <u>Sample Criteria:</u> Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria |
| Petroleum Hydrocarbons (Inorganics: HEM/SGT HEM) | Method Blank (MB) | 1664A | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples | — | N/A |

| Analysis | *QC Sample | Method | NPDES 1 | Method | RCRA (SW846) 2 |
|---------------|--|--------|---|----------------|---|
| | | | associated with unacceptable method blank | | |
| | Laboratory Control Sample (LCS) | 1664A | Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery is specified by the method. 78-114%, 11% RPD for HEM and 64-132%, 28% RPD for SGT HEM. Corrective Action: If not within laboratory control limits, rerun all associated samples | — | N/A |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 1664A | Frequency: 1 with every 10 samples per site Criteria: Percent recovery is specified by the method, 78-114%, 11% RPD for HEM and 64-132%, 28% RPD for SGT HEM Corrective Action: Flag data associated with unacceptable Matrix Spike | — | N/A |
| | Duplicate (DU) | 1664A | N/A | — | N/A |
| Semivolatiles | Method Blank (MB) | 625 | Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit. Corrective Action: Rerun all samples associated with unacceptable | 8270C 8270D | Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit. Corrective Action: Rerun all samples associated with unacceptable |

| Analysis | *QC Sample | Method | NPDES 1 | Method | RCRA (SW846) 2 |
|----------|--|--------|--|----------------|---|
| | | | method blank | | method blank |
| | Laboratory Control Sample (LCS) | 625 | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples. <u>Criteria:</u> Percent recovery must be within laboratory control limits. <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples | 8270C 8270D | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples. <u>Criteria:</u> Percent recovery must be within laboratory control limits. <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 625 | <u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within advisory limits given in method <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike | 8270C 8270D | <u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within advisory limits given in method <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike |
| | Duplicate (DU) | 625 | N/A | 8270C 8270D | N/A |
| | Surrogates (Surr) | 625 | <u>Frequency:</u> Surrogates spiked into method blank and all samples (QC included) <u>Method Blank and LCS Criteria:</u> All surrogates must be in control before sample analysis may proceed. One surrogate per fraction may exceed control limits if greater than 10% recovery. <u>Sample Criteria:</u> | 8270C 8270D | <u>Frequency:</u> Surrogates spiked into method blank and all samples (QC included) <u>Method Blank and LCS Criteria:</u> All surrogates must be in control before sample analysis may proceed. One surrogate per fraction may exceed control limits if greater than 10% recovery. <u>Sample Criteria:</u> Re-extract samples or flag sample data not |

| Analysis | *QC Sample | Method | NPDES 1 | Method | RCRA (SW846) 2 |
|--------------------|---------------------------------|--------|---|-------------------------|--|
| | | | Re-extract samples or flag sample data not meeting surrogate criteria | | meeting surrogate criteria |
| | Internal Standards (IS) | 625 | <u>Frequency:</u> Internal standards spiked into method blank and all samples (QC included) <u>Criteria:</u> All internal standard recoveries must be within laboratory control limits <u>Corrective Action:</u> Flag sample data not meeting internal standard recovery requirements | 8270C 8270D | <u>Frequency:</u> Internal Standards are added to all samples (QC samples included). <u>Criteria:</u> area of daily standard must be within 50% to 200% of the response in the mid-level of the initial calibration standard. The retention time (RT) for any internal standard (IS) in the continuing calibration must not exceed ± 0.5 minutes from mid-level initial calibration standard IS RT. |
| Volatiles by GC/MS | Method Blank (MB) | 624 | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank | 8260A 8260B 8260C | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank |
| | Laboratory Control Sample (LCS) | 624 | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within control limits, rerun all | 8260A 8260B 8260C | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within control limits, rerun all associated samples |

| Analysis | *QC Sample | Method | NPDES 1 | Method | RCRA (SW846) 2 |
|----------------|--|--------|--|-------------------------|--|
| | | | associated samples | | |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 624 | Frequency: 1 per 20 samples, minimum of one per batch of samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit | 8260A 8260B 8260C | Frequency: 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit |
| | Duplicate (DU) | 624 | N/A | 8260A 8260B 8260C | N/A |
| | Surrogates (Surr) | 624 | <u>Frequency:</u> Surrogates are spiked into all samples (including all QC samples) <u>Criteria:</u> All surrogates must meet criteria <u>Corrective Action:</u> Re-extract and re-analyze samples or flag sample data not meeting surrogate criteria. | 8260A 8260B 8260C | <u>Frequency:</u> Surrogates are spiked into all samples (including all QC samples) <u>Criteria:</u> All surrogates must meet criteria <u>Corrective Action:</u> Re-extract and re-analyze samples or flag sample data not meeting surrogate criteria. |
| | Internal Standards (IS) | 624 | Frequency: Internal standards spiked into method blank and all samples (QC included) <u>Criteria:</u> All internal standard recoveries must be within laboratory control limits <u>Corrective Action:</u> Flag sample data not meeting internal standard recovery requirements | 8260A 8260B 8260C | Frequency: Internal standards spiked into method blank and all samples (QC included) <u>Criteria:</u> All internal standard recoveries must be within laboratory control limits <u>Corrective Action:</u> Flag sample data not meeting internal standard recovery requirements |
| Methyl Mercury | Method Blank (MB) | 1630 | <u>Frequency:</u> 1 with each batch of samples | — | N/A |

| Analysis | *QC Sample | Method | NPDES 1 | Method | RCRA (SW846) 2 |
|----------|--|--------|---|--------|----------------|
| | | | processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank. | | |
| | Laboratory Control Sample (LCS) | 1630 | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples | — | N/A |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | 1630 | <u>Frequency:</u> 1 per 10 samples, minimum of one per batch of samples processed <u>Criteria:</u> Must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit | — | N/A |
| | Duplicate (DU) | 1630 | N/A | — | N/A |
| | Surrogates (Surr) | 1630 | <u>Frequency:</u> Surrogates are spiked into all samples (including all QC samples) <u>Criteria:</u> All surrogates must meet criteria <u>Corrective Action:</u> Re-extract and re-analyze samples | — | N/A |

| Analysis | *QC Sample | Method | NPDES 1 | Method | RCRA (SW846) 2 |
|---|--|--------|---|-------------------------|---|
| | | | or flag sample data not meeting surrogate criteria. | | |
| Formaldehyde | Method Blank (MB) | — | N/A | 8315A | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable method blank |
| | Laboratory Control Sample (LCS) | — | N/A | 8315A | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within control limits, rerun all associated samples |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | — | N/A | 8315A | <u>Frequency:</u> 1 per 10 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit |
| | Duplicate (DU) | — | N/A | 8315A | N/A |
| Diesel Range Organics (DRO) and Gasoline Range Organics (GRO) | Method Blank (MB) | — | N/A | 8015B 8015C 8015D | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples |

| Analysis | *QC Sample | Method | NPDES 1 | Method | RCRA (SW846) 2 |
|----------------|--|--------|---------|-------------------------|--|
| | | | | | associated with unacceptable method blank |
| | Laboratory Control Sample (LCS) | — | N/A | 8015B 8015C 8015D | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery should be within advisory limits given in the method. <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | — | N/A | 8015B 8015C 8015D | <u>Frequency:</u> 1 per 20 samples. <u>Criteria:</u> percent recovery for each analyte should be within laboratory limits. <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike |
| | Surrogates (Surr) | — | N/A | 8015B 8015C 8015D | <u>Frequency:</u> Surrogates are spiked into all samples (including all QC samples) <u>Criteria:</u> All surrogates must meet criteria <u>Corrective Action:</u> Re-extract and re-analyze samples or flag sample data not meeting surrogate criteria. |
| Aromatic Acids | Method Blank (MB) | — | N/A | Client Derived | <u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable |

| Analysis | *QC Sample | Method | NPDES 1 | Method | RCRA (SW846) 2 |
|----------|--|--------|---------|----------------|---|
| | | | | | method blank unless the method blank is above RL, and samples are ND. |
| | Laboratory Control Sample (LCS) | — | N/A | Client Derived | <u>Frequency:</u> 1 soluble and 1 insoluble with each batch of solid samples, 1 with each batch of water samples processed not to exceed 20 samples prepped <u>Criteria:</u> Percent recovery for analytes should be within laboratory accepted limits <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS |
| | Matrix Spike/Matrix Spike Duplicate (MS/MSD) | — | N/A | Client Derived | <u>Frequency:</u> 1 with each batch of water samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery for analytes should be within laboratory accepted limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike |
| | Duplicate (DU) | -- | N/A | Client Derived | N/A |

* For the Ohio EPA Voluntary Action Program (VAP), please refer to the SOPs for the acceptable criteria, corrective actions, and exceptions.

Footnotes

1. *National Pollutant Discharge Elimination System*
2. *Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), Final Update III (December 1996), and Final Update IV (2007)*

13. PREVENTIVE ACTION / IMPROVEMENT

13.1. OVERVIEW

- 13.1.1. The laboratory's preventive action programs improve or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive and continuous process of improvement activities that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure the preventive action process is in place, and that relevant information on actions is submitted for management review.
- 13.1.2. Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, customer service and client satisfaction can be improved through continuous improvements to laboratory systems.
- 13.1.3. Opportunities for improvement may be discovered during management reviews, the monthly QA Metrics Report, evaluation of internal or external audits, results and evaluation of proficiency testing (PT) performance, data analysis and review processing operations, client complaints, staff observation, etc.
- 13.1.4. The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, Ethics training, etc. These metrics are used in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.
- 13.1.5. The laboratory's corrective action process (Section 12) is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action provides a valuable mechanism for identifying preventive action opportunities.
- 13.1.6. The following elements are part of a preventive action system:
 - 13.1.6.1. Identification of an opportunity for preventive action.
 - 13.1.6.2. Process for the preventive action.
 - 13.1.6.3. Define the measurements of the effectiveness of the process once undertaken.
 - 13.1.6.4. Execution of the preventive action.
 - 13.1.6.5. Evaluation of the plan using the defined measurements.

13.1.6.6. Verification of the effectiveness of the preventive action.

13.1.6.7. Close-out by documenting any permanent changes to the Quality System as a result of the Preventive Action. Documentation of Preventive Action is incorporated into the monthly QA reports, corrective action process, and management review.

13.1.7. Any Preventive Actions undertaken or attempted must be taken into account during the Annual Management Systems Review (Section 16). A highly detailed report is not required; however, a summary of success and failure within the preventive action program is sufficient to provide management with a measurement for evaluation.

13.2. Management Of Change

13.2.1. The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The laboratory has a graded approach for managing change based based on the Management Systems Review.

14. CONTROL OF RECORDS

14.1. The laboratory maintains a records management system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued.

14.2. Overview

14.2.1. The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 14-1. Quality records are maintained by the QA Department which is backed up as part of the regular network backup. Records are of two types--either electronic or hard-copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by the Records Manager.

Table 14-1. Records Index (1)

| | |
|-----------------|-----------------|
| Record Types 1: | Retention Time: |
|-----------------|-----------------|

| | Record Types 1: | Retention Time: |
|------------------------|---|--|
| Technical Records | <ul style="list-style-type: none"> - Raw Data - Logbooks² - Standards - Certificates - Analytical Records - MDLs/IDLs/DOCs - Lab Reports | 5 Years from analytical report issue* |
| Official Documents | <ul style="list-style-type: none"> - Quality Assurance Manual (QAM) - Work Instructions - Policies - SOPs - Policy Memorandums - Manuals | 5 Years from document retirement date* |
| QA Records | <ul style="list-style-type: none"> - Internal & External Audits/Responses - Certifications - Corrective/Preventive Actions - Management Reviews - Method & Software Validation / Verification Data - Data Investigation | 5 Years from archival* Data Investigation: 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation) |
| Project Records | <ul style="list-style-type: none"> - Sample Receipt & COC Documentation - Contracts and Amendments - Correspondence - QAPP - SAP - Telephone Logbooks - Lab Reports | 5 Years from analytical report issue* |
| Administrative Records | Finance and Accounting | 10 years |
| | EH&S Manual, Permits | 7 years |
| | Disposal Records | Indefinitely |
| | Employee Handbook | Indefinitely |
| | Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics) | Refer to HR Manual |
| | Administrative Policies Technical Training Records | 7 years |

1. Record Types encompass hardcopy and electronic records.
2. Examples of logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).

* Exceptions listed in Table 14-2.

14.2.2. All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility that provides a suitable environment to prevent damage or deterioration and to prevent loss. All records must be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records and electronic

or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

14.2.3. Access to the data is limited to laboratory and company employees, and shall be documented with an access log. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

14.2.4. For raw data and project records, record retention must be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 14-2 have lengthier retention requirements and are subject to the requirements in Section 14.1.3.

14.3. Programs with Longer Retention Requirements

14.3.1. Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Note: For the Ohio VAP program the laboratory is required to notify Ohio EPA of its intent to dispose of any records.

Table 14-2. Special Record Retention Requirements

| Program | Retention Requirement |
|---|--|
| Ohio – Drinking Water | 5 years (project records) 10 years – radio chemistry (project records) |
| Michigan Department of Environmental Quality – all environmental data | 10 years |
| OSHA - 40 CFR Part 1910 | 30 years |
| TSCA - 40 CFR Part 792 | 10 years after publication of final test rule or negotiated test agreement and others as negotiated. |
| Ohio Voluntary Action Program | 10 years |

Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

- 14.3.2. The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hardcopy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, refer to Section 19.14.1 for more information.
- 14.3.3. The record-keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data. (Records stored off site should be accessible within two days of a request for such records). The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This must include inter-laboratory transfers of samples and/or extracts.
- 14.3.4. The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory copy of the Chain-of-Custody is stored with the invoice and the Work Order sheet generated by LIMS. The Chain-of-Custody would indicate the name of the sampler. If any sampling notes are provided with a Work Order, they are kept with this package.
- 14.3.5. All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.
- 14.3.6. The record-keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes, e.g., set format for naming electronic files, set format for what is included with a given analytical data set. SOP NC-QA-019, Records Information Management, outlines this procedure. Instrument data is stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. Run logs are maintained for each instrument or method; each day's run long or instrument sequence is stored with the data to aid in reconstructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is entered into LIMS for each method as required.
- 14.3.7. Changes to hardcopy records must follow the procedures outlined in Sections 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.
- 14.3.8. The reason for a signature or initials on a document is clearly indicated in the records such as "Sampled by," "Prepared by," "Reviewed by", or "Analyzed by".

- 14.3.9. All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.
- 14.3.10. Hard-copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure no data is lost, and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard-copy which was scanned.
- 14.3.11. Also refer to Section 19.14.1, "Computer and Electronic Data Related Requirements".

14.4. Technical And Analytical Records

- 14.4.1. The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement (refer to Section 15.1). The records for each analysis must contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records must include the identity of laboratory personnel responsible for the sampling, performance of each analysis and reviewing results.
- 14.4.2. Observations, data, and calculations are recorded in real-time at the time they are made and are identifiable to the specific task.
- 14.4.3. Changes to hardcopy records must follow the procedures outlined in Sections 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails. The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

14.5. Laboratory sample ID code

- 14.5.1. Date of analysis. Time of analysis is also required if the holding time is 72 hours or less, or when time-critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation.
- 14.5.2. Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available. Instrument logs may be in electronic format.
- 14.5.3. Analysis type
- 14.5.4. All manual calculations and manual integrations

- 14.5.5. Analyst or operator initials/signature
- 14.5.6. Sample preparation, including cleanup, separation protocols, incubation periods, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents
- 14.5.7. Test results
- 14.5.8. Standard and reagent origin, ID codes, and dates of receipt, preparation, and use
- 14.5.9. Calibration criteria, frequency, and acceptance criteria
- 14.5.10. Data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions
- 14.5.11. Quality control protocols and assessment
- 14.5.12. Electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries
- 14.5.13. Method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats.
- 14.5.14. All logbooks used during receipt, preparation, storage, analysis, and reporting of samples or monitoring of support equipment shall undergo a documented supervisory or peer review on a monthly basis.
- 14.6. Laboratory Support Activities
 - 14.6.1. In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):
 - 14.6.2. All original raw data, whether hard-copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records)
 - 14.6.3. A written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value
 - 14.6.4. Copies of final reports
 - 14.6.5. Archived SOPs
 - 14.6.6. Correspondence relating to laboratory activities for a specific project

- 14.6.7. All Corrective Action reports, audits and audit responses
- 14.6.8. Proficiency test results and raw data
- 14.6.9. Results of data review, verification, and cross-checking procedures
- 14.7. Sample Handling Records
 - 14.7.1. Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include, but are not limited to, records pertaining to:
 - 14.7.2. Sample preservation including appropriateness of sample container and compliance with holding time requirement
 - 14.7.3. Sample identification, receipt, acceptance or rejection and login
 - 14.7.4. Sample storage and tracking including shipping receipts, sample transmittal / COC forms
 - 14.7.5. Procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.
- 14.8. Administrative Records
 - 14.8.1. The laboratory also maintains the administrative records in either electronic or hard-copy form Refer to Table 14-1.
- 14.9. Records Management, Storage, And Disposal
 - 14.9.1. All records (including those pertaining to test equipment), certificates, and reports are safely stored, held secure, and in confidence to the client. Certification-related records are available to the accrediting body upon request.
 - 14.9.2. All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.
 - 14.9.3. Records that are stored or generated by computers or personal computers have hardcopy, write-protected backup copies, or an electronic audit trail controlling access.
 - 14.9.4. The laboratory has a record management system for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage, and reporting. Laboratory notebooks are issued on a per analysis basis, and are numbered sequentially.

14.10. Transfer Of Ownership

- 14.10.1. In the event the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous five years of such action.

14.11. Records Disposal

- 14.11.1. Records are removed from the archive and destroyed after five years, unless otherwise specified by a client or regulatory requirement. On a project-specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration (refer to Tables 14-1 and 14-2).
- 14.11.2. Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.
- 14.11.3. If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required.

15. AUDITS

15.1. Internal Audits

- 15.1.1. Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and when requested to corporate management.
- 15.1.2. Audits are conducted and documented as described in TestAmerica Corporate SOP CW-Q-S-003 on performing Internal Auditing. The types and frequency of routine internal audits are described in Table 15-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 15-1. Types of Internal Audits and Frequency

| Description | Performed by | Frequency |
|------------------------|--|--|
| Quality Systems Audits | QA Department, QA approved designee, or Corporate QA | All areas of the laboratory annually |
| Method Audits | Joint responsibility: QA Manager or designee with assistance by the Technical Director or designee (refer to CA-Q-S-004) | Method audits frequency: 50% of methods annually 100% of methods annually (DoD Labs) |
| QA Technical Audits | Joint responsibility: QA manager or designee Technical Manager or Designee (Refer to CW-Q-S-003) | Technical Audits Frequency: 50% of methods annually |
| SOP Method Compliance | Joint responsibility: QA Manager or Designee D) Technical Manager or Designee (Refer to CW-Q-S-003) | SOP Compliance Review Frequency Every 2 years 100% of SOPs annually (DoD Labs) |
| Special | QA Department or Designee | Surveillance or spot checks performed as needed, e.g., to confirm corrective actions from other audits |
| Performance Testing | Analysts with QA oversight | Two successful per year for each TNI field of testing or as dictated by regulatory requirements |

15.2. Annual Quality Systems Audit

15.2.1. An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, TestAmerica's Data Integrity and Ethics Policies, TNI quality systems, client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to, data review, quality controls, preventive action, and corrective action. The completeness of earlier corrective action is assessed for effectiveness and sustainability. The audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

Note: Part of the quality systems audit relates to regulatory compliance. An assessment of the laboratory's compliance to regulatory requirements

is performed by Corporate QA through monthly management reports, review of client and regulatory concerns, and also through periodic on-site evaluations.

15.3. QA Technical Audits

15.3.1. QA technical audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, electronic audit Miner programs (e.g., Chrom AuditMiner) are used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits must include all methods within a two-year period.

15.4. SOP Method Compliance

15.4.1. Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs must be assessed by the Technical Director and the QA department at least every two years. (Annually for methods and administrative SOPs related to DoD programs.) The work of each newly hired analyst is assessed within three months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products must be performed within three months of completing the documented training.

15.5. Special Audits

15.5.1. Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue. Special audits will also be performed when new methods and/or instrumentation is implemented.

15.6. Performance Testing

15.6.1. The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies—non potable water and soil.

15.6.2. It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in

agreement with any decisions made to treat a PT sample differently due to some special circumstance.

- 15.6.3. Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

15.7. External Audits

- 15.7.1. External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory group leaders are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit. When requested, a copy of the audit report and the laboratory's Corrective Action plan must be forwarded to Corporate Quality.
- 15.7.2. The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

15.8. Confidential Business Information (CBI) Considerations

- 15.8.1. During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2009 TNI standards.

15.9. Audit Findings

- 15.9.1. Audit findings are documented using the Corrective Action process and spreadsheet. The laboratory's Corrective Action responses for both types of audits may include action plans that could not be completed within a

predefined timeframe. In these instances, a completion date must be set and agreed to by Operations management and the QA Manager.

15.9.2. Developing and implementing Corrective Action to findings is the responsibility of the Department Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. When requested, a copy of the audit report and the laboratory's Corrective Action plan must be forwarded to Corporate Quality.

15.9.3. If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory must take timely corrective action, and must notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

15.9.4. Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24 hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

16. MANAGEMENT REVIEWS

16.1. Quality Assurance Report

16.1.1. A comprehensive QA Report must be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director and Corporate Quality Director, as well as the VP of Operations. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, VP of Operations, or Corporate QA may request that additional information be added to the report.

16.1.2. On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Directors prepare a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and General Managers.

16.2. Annual Management Review

16.2.1. The Senior Lab Management Team (Laboratory Director, Technical Director, Operations Manager, QA Manager, HR Supervisor, PM Manager) conducts a review annually of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or

improvements. It will also provide a platform for defining goals, objectives, and action items that feed into the laboratory planning system. Corporate Operations and Corporate QA personnel may be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the LIMS. The laboratory must summarize any critical findings that cannot be solved by the lab, and report them to Corporate IT.

- 16.2.2. The Management Systems Review (Corporate SOP CW-Q-S-004 and Work Instruction CW-Q-WI-003) uses information generated during the preceding year to assess the “big picture” by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective; therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

16.2.2.1. Matters arising from the previous annual review

16.2.2.2. Prior Monthly QA Reports issues

16.2.2.3. Laboratory QA Metrics

16.2.2.4. Review of report reissue requests

16.2.2.5. Review of client feedback and complaints

16.2.2.6. Issues arising from any prior management or staff meetings

16.2.2.7. Minutes from prior Senior Lab Management Team meetings.
Issues that may be raised from these meetings include:

16.2.2.7.1. Adequacy of staff, equipment and facility resources

16.2.2.7.2. Adequacy of policies and procedures

16.2.2.7.3. Future plans for resources and testing capability and capacity

16.2.2.8. The annual internal double blind PT program sample performance (if performed)

16.2.2.9. Compliance to the Ethics Policy and Data Integrity Plan, including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity

16.2.2.10. A management system review report is generated by the QA Manager and management. The report is distributed to the

appropriate VP of Operations, and Corporate Quality Director.
The report includes, but is not limited to:

- 16.2.2.11. The date of the review and the names and titles of participants
- 16.2.2.12. A reference to the existing data quality related documents and topics that were reviewed
- 16.2.2.13. Quality system or operational changes or improvements that will be made as a result of the review, e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)
- 16.2.2.14. Changes to the quality systems requiring update to the laboratory QA Manual must be included in the next revision of the QA Manual.

16.3. Potential Integrity Related Managerial Reviews

- 16.3.1. Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. TestAmerica's Corporate Data Investigation/ Recall SOP CW-L-S-002 must be followed. All investigations that result in finding inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.
- 16.3.2. TestAmerica's CEO, Executive VP of Operations, VP of Client & Technical Services, VPs of Operations and Quality Directors receive a monthly report from the Corporate Quality and EHS Director summarizing any current data integrity or data recall investigations. The VPs of Operations are also made aware of progress on these issues for their specific labs.

17. PERSONNEL

- 17.1. The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1.
- 17.2. All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training must have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff must be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

- 17.3. The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.
- 17.4. All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.
- 17.5. Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training must be relevant to the present and anticipated responsibilities of the lab staff.
- 17.6. The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance with the laboratory's quality system.
- 17.7. Education And Experience Requirements For Technical Personnel
- 17.7.1. The laboratory makes every effort to hire analytical staff that posses a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. There are competent analysts and technicians in the industry who have not earned a college degree. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.
- 17.7.2. The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are located on the TestAmerica intranet "Human Resources" web-page (also see Section 4 for position descriptions/responsibilities).
- 17.7.3. Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance or quantitation techniques, etc. are also considered
- 17.7.4. As a general rule for analytical staff:

| Specialty | Education | Experience |
|-----------|-----------|------------|
|-----------|-----------|------------|

| Specialty | Education | Experience |
|--|--|---|
| Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses | H.S. Diploma | On the job training (OJT) |
| CVAA, FLAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC | A college degree in an applied science or 2 years of college and at least one year of college chemistry | Or 2 years prior analytical experience is required |
| ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS | A college degree in an applied science or 2 years of college chemistry | Or 5 years of prior analytical experience |
| Spectra Interpretation | A college degree in an applied science or 2 years of college chemistry | And 2 years relevant experience. Or 5 years of prior analytical experience |
| Group Leaders – General | Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience | And 2 years experience in environmental analysis of representative analytes for which they will oversee |
| Group Leader – Wet Chem only (no advanced instrumentation) | Associate degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry | And 2 years relevant experience |

17.7.5. When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Department Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

17.8. Training

17.8.1. The laboratory is committed to furthering the professional and technical development of employees at all levels.

17.8.2. Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

| Required Training | Time Frame* | Employee Type |
|--|--|-------------------|
| New Hire Orientation | Immediately | All |
| Environmental Health & Safety Orientation | Prior to lab work | All |
| Environmental Health & Safety Orientation Follow-up Test | 30-60 days after hire | All |
| Environmental Health & Safety Training | Refer to EH&S Manual | All |
| Ethics – New Hires | 1 week of hire | All |
| Ethics - Comprehensive | 90 days of hire | All |
| Data Integrity | 30 days of hire | Technical and PMs |
| Quality Assurance | 90 days of hire | All |
| Ethics – Comprehensive Refresher | Annually | All |
| Initial Demonstration of Capability (DOC) | Prior to unsupervised method performance | Technical |

17.8.3. The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to “Demonstration of Capability” in Section 19.

17.8.4. The training of technical staff is kept up to date by:

- 17.8.4.1. Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual, and SOPs, and any work instructions involving their area of responsibility. This documentation is updated as the various documents are revised.
- 17.8.4.2. Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics are maintained in the employee’s training file.
- 17.8.4.3. Documentation of proficiency (refer to Section 19)
- 17.8.4.4. An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training
- 17.8.4.5. A Confidentiality Agreement signed by each staff member at the time of employment
- 17.8.4.6. Human Resources maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct, e.g., ethics. This

information is maintained in the employee's secured personnel file.

17.8.5. Evidence of successful training could include such items as:

17.8.5.1. Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.

17.8.5.2. Analysts' knowledge of the QA Manual for quality issues

17.8.5.3. Analysts following SOPs, i.e., practice matches SOPs

17.8.5.4. Analysts regularly communicate to group leaders and QA if SOPs need revision rather than waiting for auditors to find problems.

17.8.6. Further details of the laboratory's analyst training program are described in the Laboratory Training SOP NC-QA-028, Employee Orientation and Training.

17.9. Data Integrity And Ethics Training Program

17.9.1. Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within one week of hire followed by technical data integrity training within 30 days, comprehensive training within 90 days, and annual refresher for all employees. Senior management at each facility performs the Ethics training for their staff.

17.9.2. In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times, TestAmerica has established a Corporate Ethics Policy (CW-L-P-004) and an Ethics Statement. All initial and annual training is documented by employee signature on the signed Ethics Statement/Agreement demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

17.9.3. Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts; for that reason, TestAmerica has a zero tolerance approach to such violations.

- 17.9.3.1. Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:
 - 17.9.3.2. Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting
 - 17.9.3.3. Ethics Policy
 - 17.9.3.4. How and when to report ethical/data integrity issues. Confidential reporting.
 - 17.9.3.5. Record keeping
 - 17.9.3.6. Discussion regarding data integrity procedures
 - 17.9.3.7. Specific examples of breaches of ethical behavior--peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion
 - 17.9.3.8. Internal monitoring. Investigations and data recalls
 - 17.9.3.9. Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution
 - 17.9.3.10. Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient
- 17.9.4. Additionally, a Data Integrity Hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

18. ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

- 18.1. The laboratory is a 54,440 sq. ft. secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.
- 18.2. The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc. OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity-controlled), access, and safety equipment are met or exceeded.
- 18.3. Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area

is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

- 18.4. The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, and administrative functions.

- 18.5. Environment

- 18.5.1. Laboratory accommodation, test areas, energy sources, and lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

- 18.5.2. The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

- 18.5.3. The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity, voltage, temperature, and vibration levels in the laboratory. A 225KVA UPS is installed in the main electrical bus to provide at least 15 minutes of backup power in the event of a power failure. This unit also provides voltage and frequency control of lab and office power. A spike/surge arrestor is installed to protect against power surge/sag and lightning strikes. A 30 KW natural gas-fueled backup generator is installed to provide power to the I.T. area in the event of a power failure. Additionally, this generator provides power to two walk-in sample storage coolers and several other smaller sample storage coolers. Smaller portable generators are available to provide "spot power" where needed in the event of a power failure.

- 18.5.4. When any of the method or regulatory required environmental conditions change to an extent that they may adversely affect test results, analytical testing must be discontinued until the environmental conditions are returned to the required levels.

- 18.5.5. Environmental conditions of the offsite facility housing the computer network and LIMS are regulated to protect against raw data loss.

- 18.6. Work Areas

- 18.6.1. There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- 18.6.2. Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.
- 18.6.3. Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory.
- 18.6.4. Access to, and use of, all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.
 - 18.6.4.1. Access and entryways to the laboratory
 - 18.6.4.2. Sample receipt areas
 - 18.6.4.3. Sample storage areas
 - 18.6.4.4. Chemical and waste storage areas
 - 18.6.4.5. Data handling and storage areas
 - 18.6.4.6. Sample processing areas
 - 18.6.4.7. Sample analysis areas

18.7. Floor Plan

- 18.7.1. A floor plan can be found in Appendix 1.

18.8. Building Security

- 18.8.1. Building keys and keybadges are distributed to employees as necessary.
- 18.8.2. Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into the laboratory, the visitor is provided with any necessary personal protection equipment. The Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed.
- 18.8.3. Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.
- 18.8.4. Signs are posted in the laboratory designating employee only areas - "Authorized employees beyond this point".

19. TEST METHODS AND METHOD VALIDATION

- 19.1. The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage, and preparation of samples; and, where appropriate, an estimation of the measurement of uncertainty, as well as statistical techniques for analysis of environmental data.
- 19.2. Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.
- 19.3. Standard Operating Procedures (Sops)
 - 19.3.1. The laboratory maintains SOPs that accurately reflect all of the laboratory procedures such as assessing data integrity, taking corrective action, handling customer complaints, as well as all analytical methods and sampling procedures. The method SOPs are derived from promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory.
 - 19.3.2. All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
 - 19.3.3. Procedures for writing an SOP are included in TestAmerica's Corporate SOP CW-Q-S-002 entitled Writing a Standard Operating Procedure, or the Canton laboratory SOP NC-QA-027, Preparation and Management of Standard Operating Procedures.
 - 19.3.4. SOPs are reviewed at a minimum of every two years (annually for DoD SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.
- 19.4. Laboratory Methods Manual
 - 19.4.1. For each test method, the laboratory must have available the published referenced method(s) as well as the laboratory developed SOP(s).

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory must demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

19.4.2. The laboratory maintains an SOP Index/Listing for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

19.5. Selection Of Methods

19.5.1. Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services, e.g., special matrices, non-routine compound lists, etc., the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

19.5.2. Sources of Methods

19.5.3. Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods must be used.

19.5.4. When clients do not specify the method to be used or specific methods are not available, the methods that are used must be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

19.5.5. The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

19.5.5.1. Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM); Non-polar Material) by Extraction and Gravimetry, EPA-821-R-98-002, February 1999

19.5.5.2. Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. Revised as of July 1, 1995, Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)

- 19.5.5.3. Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.
- 19.5.5.4. Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.
- 19.5.5.5. Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.
- 19.5.5.6. Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th edition/ on-line edition Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.
- 19.5.5.7. Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996, Final Update IV, January 2008.
- 19.5.5.8. Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.
- 19.5.5.9. Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261
- 19.5.6. The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, client requirements, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.
- 19.5.7. Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.
- 19.5.8. The laboratory must inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it must be documented.
- 19.6. Demonstration of Capability
 - 19.6.1. Before the laboratory may institute a new method and begin reporting results, the laboratory must confirm that it can properly perform the method. In general, this demonstration does not test the performance of

the method in real world samples, but in an applicable and available clean matrix.

- 19.6.2. A demonstration of capability is performed (SOP NC-QA-028, Employee Orientation and Training) whenever there is a change in instrument type (e.g., new instrumentation), method, or personnel (e.g., analyst has not performed the test within the last 12 months).
- 19.6.3. The initial demonstration of capability (IDOC) must be thoroughly documented and approved by the department group leader and QA Manager prior to an analyst independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures for analyst training documentation.
- 19.6.4. Before the laboratory can analyze client samples by an analytical method, there must be an approved SOP in place, a demonstration of satisfactory analyst performance must be completed, and an MDL study (where applicable) must be performed. There may be other additional requirements stated within the published method or regulations (i.e., retention time window study for GC methods like 8081).
- Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Analyst IDOC/CDOC).
- 19.6.5. If the client states that the information is not for regulatory purposes, and is intended to screen for the presence of the analyte the result may be reported as long as the following criteria are met:
- 19.6.6. A low-level standard containing the non-routine analyte at the RL must be analyzed to verify the laboratory's (and method) capability to detect the analyte at the RL.
- 19.6.7. If the client states that a quantitative result is required, a multi-point calibration must be analyzed, and ICV/CCV criteria must be met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- 19.6.8. The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve (low standard at or below the QL) and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).

Note: For Ohio VAP work, the term Reporting Limit will be used.

19.6.9. The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted as "Reporting Limit based on the low standard of the calibration curve".

19.7. Initial Demonstration of Capability (IDOC) Procedures

19.7.1. At least four aliquots must be prepared (including any applicable clean-up procedures) in the same fashion, and following all of the same procedures, as client samples, and analyzed according to the test method (either concurrently or over a period of days).

19.7.2. Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest. Refer to SOP NC-QA-028, Employee Orientation and Training, for details on this procedure.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

19.7.3. A certification statement (see Figure 19-1 as an example) must be used to document the completion of each IDOC. A copy of the certification is archived in the analyst's training folder.

19.8. Laboratory-Developed Methods And Non-Standard Methods

19.8.1. Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must agree to the use of the non-standard method.

19.9. Validation Of Methods

19.9.1. Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

19.9.2. All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are suitable for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

19.9.3. Method Validation and Verification Activities for All New Methods

19.9.3.1. While method validation can take various courses, the following activities can be required as part of method validation. Method

validation records are designated QC records and are archived accordingly.

19.9.4. Determination of Method Selectivity

19.9.4.1. Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices. In some cases, to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

19.9.5. Determination of Method Sensitivity

19.9.5.1. Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed.

19.9.6. Relationship of Limit of Detection (LOD) to the Limit of Quantitation (LOQ)

19.9.6.1. An important characteristic of expression of sensitivity is the difference in the LOD and the LOQ. The LOD is the minimum level at which the presence of an analyte can be reliably determined. The LOQ is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and accuracy. For most instrumental measurement systems, there is a region where estimated is generated around the LOD (both above and below the estimated MDL or LOD) and below the LOQ. In this range, detection of an analyte may be confirmed, but quantification of the analyte is unreliable with unknown accuracy and precision. When an analyte is detected below the LOQ, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the presence of the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data are to be reported in this range, it must be done so with a qualification that denotes the estimated/uncertain nature of the result.

19.9.7. Determination of Interferences

19.9.7.1. A determination that the method is free from interferences in a blank matrix is performed.

19.9.8. Determination of Range

19.9.8.1. Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a

curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or LOQ cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of precision and accuracy.

19.9.9. Determination of Accuracy and Precision

19.9.9.1. Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

19.9.10. Documentation of Method

19.9.10.1. The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment or Amendment, describing the specific differences in the new method is acceptable in place of a separate SOP.

19.9.11. Continued Demonstration of Method Performance

19.9.11.1. Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch-specific QC samples such as LCS, method blanks, or PT samples.

19.10. Method Detection Limits (MDL)/ Limits Of Detection (LOD)

19.10.1. Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B, or alternatively by other technically valid practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements (refer to Section 19.7.10). Generally, the analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL. To allow for some flexibility, this low level standard may be analyzed every batch or every week or some other frequency rather

than doing the study all at once. In addition, a larger number of data points may be used if the appropriate t-value multiplier is used.

- 19.10.2. Refer to the Corporate SOP CA-Q-S-006 or the laboratory's SOP NC-QA-021 for details on the laboratory MDL process.

Note: For Ohio VAP projects, the MDL procedure must also comply with OAC Rule 3745-300-01(A)(78).

19.11. Instrument Detection Limits (IDL)

- 19.11.1. The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in the demonstration of instrument performance in other areas.
- 19.11.2. IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either by using seven replicate spike analyses, like MDL but without sample preparation, or by the analysis of ten instrument blanks and calculating three times the absolute value of the standard deviation.
- 19.11.3. If IDL is > than the MDL, it may be used as the reported MDL.

19.12. Verification Of Detection And Reporting Limits

- 19.12.1. Once the MDL is determined, it must be verified on each instrument used for the given method. TestAmerica defines the DoD QSM Detection Limit (DL) as being equal to the MDL. TestAmerica also defines the DoD QSM Limit of Detection (LOD) as being equal to the lowest concentration standard that successfully verifies the MDL, also referred to as the MDLV standard. MDL and MDLV standards are extracted/digested and analyzed through the entire analytical process. The MDL and MDLV determinations do not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDLV standard is not successful, then the laboratory will redevelop their MDL or perform and pass two consecutive MDLVs at a higher concentration and set the LOD at the higher concentration. Initial and quarterly verification is required for all methods listed in the laboratory's DoD ELAP Scope of Accreditation. Refer to the laboratory SOP NC-QA-021 or Corporate CA-Q-S-006 for further details.
- 19.12.2. The laboratory quantitation limit is equivalent to the DoD Limit of Quantitation (LOQ), which is at a concentration equal to or greater than the lowest non-zero calibration standard. The DoD QSM requires the laboratory to perform an initial characterization of the accuracy and precision at the LOQ and to perform quarterly LOQ verifications thereafter. If the quarterly verification results are not consistently within the three-standard deviation confidence limits established initially, then the accuracy and precision will be reevaluated and clients contacted for

any on-going projects. For DoD projects, TestAmerica makes a distinction between the Reporting Limit (RL) and the LOQ. The RL is a level at or above the LOQ that is used for specific project reporting purposes, as agreed to between the laboratory and the client. The RL cannot be lower than the LOQ concentration, but it may be higher.

19.13. Retention Time Windows

- 19.13.1. Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept in each department. Complete details are available in the laboratory SOPs.

19.14. Evaluation Of Selectivity

- 19.14.1. The laboratory evaluates selectivity by following the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, atomic absorption, or fluorescence profiles.

19.15. Estimation Of Uncertainty Of Measurement

- 19.15.1. Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurement" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty," the range within which the value of the measurement is believed to lie within at least a 95% confidence level with the coverage factor $k=2$.
- 19.15.2. Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be

Gaussian in distribution, and reducible by increasing the number of measurements.

- 19.15.3. The minimum uncertainty associated with results generated by the laboratory within a specified concentration range can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.
- 19.15.4. To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent uncertainties at approximately the 99% confidence level with a coverage factor of $k = 3$. As an example, for a reported result of 1.0 mg/L with an LCS recovery range of 50 to 150%, the estimated uncertainty in the result would be 1.0 ± 0.5 mg/l.
- 19.15.5. In the case where a well-recognized test method specifies limits to the values of major sources of uncertainty of measurement, e.g., 524.2, 525, etc., and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

19.16. Sample Reanalysis Guidelines

- 19.16.1. Because there is a certain level of uncertainty with any analytical measurement, a sample re-preparation (where appropriate) and subsequent analysis (hereafter referred to as 'reanalysis') may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample non-homogeneity, analyte precipitation or other loss over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. Client specific Contractual Terms & Conditions for reanalysis protocols may supersede the following items.
- 19.16.2. Homogenous samples: If a re-analysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within + 1 reporting limit for samples < 5x the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.
- 19.16.3. If the re-analysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze

the sample a third time for confirmation, if sufficient sample is available. The three results are then compared to determine the most reliable/usable result(s).

19.16.4. Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.

19.16.5. Due to the potential for increased variability, reanalysis may not be applicable to Non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Group leader, if unsure.

19.17. Control Of Data

19.17.1. The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated.

19.18. Computer and Electronic Data Related Requirements

19.18.1. The three basic objectives of our computer security procedures and policies are shown below. The laboratory is currently running the TALS LIMS which is an in-house developed LIMS system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes Microsoft SQL, which is a relational database platform. It is referred to as Database for the remainder of this section.

19.19. Maintain the Database Integrity

19.19.1. Assurance is made that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

19.19.2. LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.

19.19.3. Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use. Cells containing calculations must be lock-protected and controlled.

19.19.4. Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails, and controlled access.

19.20. Ensure Information Availability

19.20.1. Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

19.21. Maintain Confidentiality

- 19.21.1. Data confidentiality is ensured through physical access controls, such as password protection or website access approval, when electronically transmitting data.

19.22. Data Reduction

- 19.22.1. The complexity of the data reduction depends on the analytical method and the number of discrete operations involved, e.g., extractions, dilutions, instrument readings, and concentrations. The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.
- 19.22.2. For manual data entry, e.g., General Chemistry, the data is reduced by the analyst and then verified by peer review once uploaded into LIMS. The review checklists are signed by both the analyst and reviewer to confirm the accuracy of the manual entry(s).
- 19.22.3. Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP CA-Q-S-002, Acceptable Manual Integration Practices.
- 19.22.4. Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's specification; otherwise, it must not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.
- 19.22.5. All raw data must be retained. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). The person who performed each task (if multiple people were involved) in the preparation and analysis must be easily identifiable in the documentation.
- 19.22.6. In general, analyte results are reported in milligrams per liter (mg/L) or micrograms per liter ($\mu\text{g/L}$) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram ($\mu\text{g/kg}$) for solids. The units "mg/L" and "mg/kg" are the same as "parts per million (ppm)". The units " $\mu\text{g/L}$ " and " $\mu\text{g/kg}$ " are the same as "parts per billion (ppb)." For values greater than 10,000 mg/L, results may be reported in percent, i.e., 10,000 mg/l = 1%. Units appropriate for us are defined in each laboratory SOP.
- 19.22.7. For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS System, the raw results and dilution factors are entered directly into LIMS by the analyst, and

the software calculates the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte.

- 19.22.8. The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or unconfirmed compounds. The analyst reviews what has been entered into LIMS to check for errors.

19.23. Logbook / Worksheet Use Guidelines

- 19.23.1. Logbooks and worksheets are filled out in 'real time' and have enough information on them to trace the events of the applicable analysis/task (e.g., calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, traceable calculations, etc.). Logbooks and worksheets can also be in electronic format.
- 19.23.2. Corrections are made following the procedures outlined in Section 12.
- 19.23.3. Logbooks are controlled by the QA Department. A record is maintained of all logbooks in the lab.
- 19.23.4. Unused portions of pages must be "Z"ed out, signed and dated.
- 19.23.5. Worksheets are created with the approval of the QA Department at the facility. The QA Department controls all worksheets following the procedures in Section 6.

19.24. Data Recording Procedures

- 19.24.1. To ensure data integrity, all documentation of data and records generated or used during the process of data generation must be performed in compliance with Section 3 of this document and Policy CA-Q-T-005, Laboratory Documentation.

19.25. Data Review and Verification Procedures

- 19.25.1. Data review procedures comprise a set of computerized and manual checks applied at appropriate levels of the measurement process. Data review begins with the reduction or processing of data and continues through verification of the data and the reporting of analytical results. Calculations are checked from the raw data to the final value prior to reporting results for each group of samples. Data reduction can be performed by the analyst who obtained the data or by another analyst. Data verification starts with the analyst who performs a 100% review of the data to ensure the work was done correctly the first time. Data verification continues with review by a second reviewer who verifies that

data reduction has been correctly performed and that the analytical results correspond to the data acquired and processed.

19.26. Data Reduction and Initial Verification

- 19.26.1. Data reduction and initial verification may be performed by more than one analyst depending upon the analytical method employed. The preparation and analytical data may be reviewed independently by different analysts. In these instances, each item may not be applicable to the subset of the data verified or an item may be applicable in both instances. It is the responsibility of the analyst to ensure that the verification of data in his or her area is complete. The data reduction and initial verification process must ensure that:
- 19.26.2. Sample preparation information is correct and complete including documentation of standard identification, solvent lot numbers, sample amounts, etc.
- 19.26.3. Analysis information is correct and complete including proper identification of analysis output (charts, chromatograms, mass spectra, etc.)
- 19.26.4. Analytical results are correct and complete including calculation or verification of instrument calibration, QC results, and qualitative and quantitative sample results with appropriate qualifiers
- 19.26.5. The appropriate SOPs have been followed and are identified in the project and/or laboratory records
- 19.26.6. Proper documentation procedures have been followed
- 19.26.7. All non-conformances have been documented
- 19.26.8. Special sample preparation and analytical requirements have been met.
- 19.26.9. The data generated have been reported with the appropriate number of significant figures as defined by the analytical method in the LIMS or otherwise specified by the client.
- 19.26.10. In general, data will be processed by an analyst in one of the following ways:
- 19.26.11. Manual computation of results directly on the data sheet or on calculation pages attached to the data sheets
- 19.26.12. Input of raw data for computer processing

19.27. Direct acquisition and processing of raw data by a computer.

- 19.27.1. If data are manually processed by an analyst, all steps in the computation must be provided including equations used and the source

of input parameters such as response factors (RFs), dilution factors, and calibration constants. If calculations are not performed directly on the data sheet, they may be attached to the data sheets.

- 19.27.2. Manual integrations are sometimes necessary to correct misintegrations by an automatic data system software program, but must only be performed when necessary. Further discussion of manual integrations and the required documentation is given in Policy CA-Q-S-002, Acceptable Manual Integration Practices.
- 19.27.3. For data that are input by an analyst and processed using a computer, a copy of the input must be kept and uniquely identified with the project number and other information as needed. The samples analyzed must be clearly identified.
- 19.27.4. If data are directly acquired from instrumentation or a test procedure and processed, or immediately entered into LIMS, the analyst must verify that the following are correct:
 - 19.27.4.1. Project and sample numbers
 - 19.27.4.2. Calibration constants and RFs
 - 19.27.4.3. Units
 - 19.27.4.4. Numerical values used for reporting limits.
- 19.27.5. Analysis-specific calculations for methods are provided in SOPs. In cases where computers perform the calculations, software must be validated or verified, as described in Section 6.0 of this document, before it is used to process data.
- 19.27.6. The data reduction is documented, signed and dated by the analyst completing the process. Initial verification of the data reduction by the same analyst is documented on a data review checklist, signed and dated by the analyst.

19.28. Data Verification

- 19.28.1. Following the completion of the initial verification by the analyst performing the data reduction, a systematic check of the data that has been fully reduced and checked through Level 1 review is performed by an experienced peer, group leader, or designee. This Level 2 check is performed to ensure that Level 1 review has been completed correctly and thoroughly. The second level reviewer examines the data signed by the analyst. Any exceptions noted by the analyst must be reviewed. Included in this review is an assessment of the acceptability of the data with respect to:
 - 19.28.1.1. Adherence of the procedure used to the requested analytical method SOP

- 19.28.1.2. Correct interpretation of chromatograms, mass spectra, etc.
- 19.28.1.3. Correctness of numerical input when computer programs are used (checked randomly)
- 19.28.1.4. Correct identification and quantitation of constituents with appropriate qualifiers
- 19.28.1.5. Numerical correctness of calculations and formulas (checked randomly)
- 19.28.1.6. Acceptability of QC data (100% review)
- 19.28.1.7. Documentation that instruments were operating according to method specifications (calibrations, performance checks, etc.)
- 19.28.1.8. Documentation of dilution factors, standard concentrations, etc.
- 19.28.1.9. Sample holding time assessment.
- 19.28.2. This review also serves as verification that the process the analyst has followed is correct in regard to the following:
- 19.28.3. The analytical procedure follows the methods and client-specific instructions.
- 19.28.4. Nonconforming events have been addressed by corrective action as defined on a nonconformance memo
- 19.28.5. Valid interpretations have been made during the examination of the data and the review comments of the initial reviewer are correct
- 19.28.6. The package contains all of the necessary documentation for data review and report production and results are reported in a manner consistent with the method used for preparation of data reports.
- 19.28.7. The specific items covered in the second stage of data verification may vary according to the analytical method, but this review of the data must be documented by signing the same checklist.
- 19.29. Completeness Verification
 - 19.29.1. A third-level review is performed by the project management staff. This review is required before results are submitted to clients. This review serves to verify the completeness of the data report and to ensure that project requirements are met for the analyses performed. The items to be reviewed are:
 - 19.29.2. Analysis results are present for every sample in the analytical batch, reporting group, or sample delivery group (SDG)

- 19.29.3. Every parameter or target compound requested is reported with either a value or reporting limit
- 19.29.4. All nonconformances, including holding time violations and data evaluation statements that impact the data quality are accompanied by clearly expressed comments from the laboratory
- 19.29.5. The final report contains all the supporting documentation required by the project, and is in either the standard TestAmerica format or in the client-required format.
- 19.29.6. Implement checks to monitor the quality of laboratory results using correlation of results for different parameters of a sample (for example, does the TOC results justify the concentration of organic compounds found by GC/MS.)
- 19.29.7. A narrative to accompany the final report must be finalized by the PM. This narrative must include relevant comments collected during the earlier reviews.
- 19.29.8. The Quality Assurance Department performs data reviews per SOP CA-Q-S-004, Internal Auditing. For DoD work, 10% of all reports must undergo an internal data review.

19.30. Manual Integrations

- 19.30.1. Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods, and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using TestAmerica's Corporate SOP (CA-Q-S-002).
- 19.30.2. The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved, or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.
- 19.30.3. Analysts must not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been

unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principals and policy and is grounds for immediate termination.

- 19.30.4. Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.
- 19.30.5. All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale “after” chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale “before” chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate-approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Figure 19-1. Example - Demonstration of Capability Documentation

GC Analyst Demonstration of Capability

TestAmerica Canton

Analyst:

DOC Run Date:

Preparation Method(s):

| | | | | |
|--|---|---|---|---|
| 8151 Herbicide SOP: NC-GC-038 | WI DRO SOP: NC-GC-013 | 8315 Formaldehyde SOP: NC-GC-035 | WI GRO Prep/Analysis SOP: NC-GC-031 | 8082/608 PCBs SOP: NC-GC-007/NC-GC-038 |
| 8081/608 Pesticides SOP: NC-GC-038 | 8015 DRO SOP NC-GC-043 | 8015 GRO Prep/Analysis SOP: NC-GC-025 | Aromatic Acids Analysis (solid and water), solid prep SOP: NC-GC-036 | RSK-175 SOP: NC-GC-032 |
| 1630 MeHg Prep/Analysis SOP: NC-GC-039 | 8011 Prep/Analysis SOP: NC-GC-040 | | | |

Matrix: ? Water ? Solid

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method with the specifications in the cited SOP, which is in use at the facility for the analysis of samples under the laboratory's Quality Assurance Plan, has completed the Demonstration of Capability (DOC).
2. The test method(s) was performed by the analyst identified on this certificate.
3. The data associated with the demonstration of capability are true, accurate, complete, and self-explanatory.
4. All raw data to reconstruct and validate these analyses have been retained at the facility.
5. The associated information is organized and available for review.

Department Supervisor

Signature

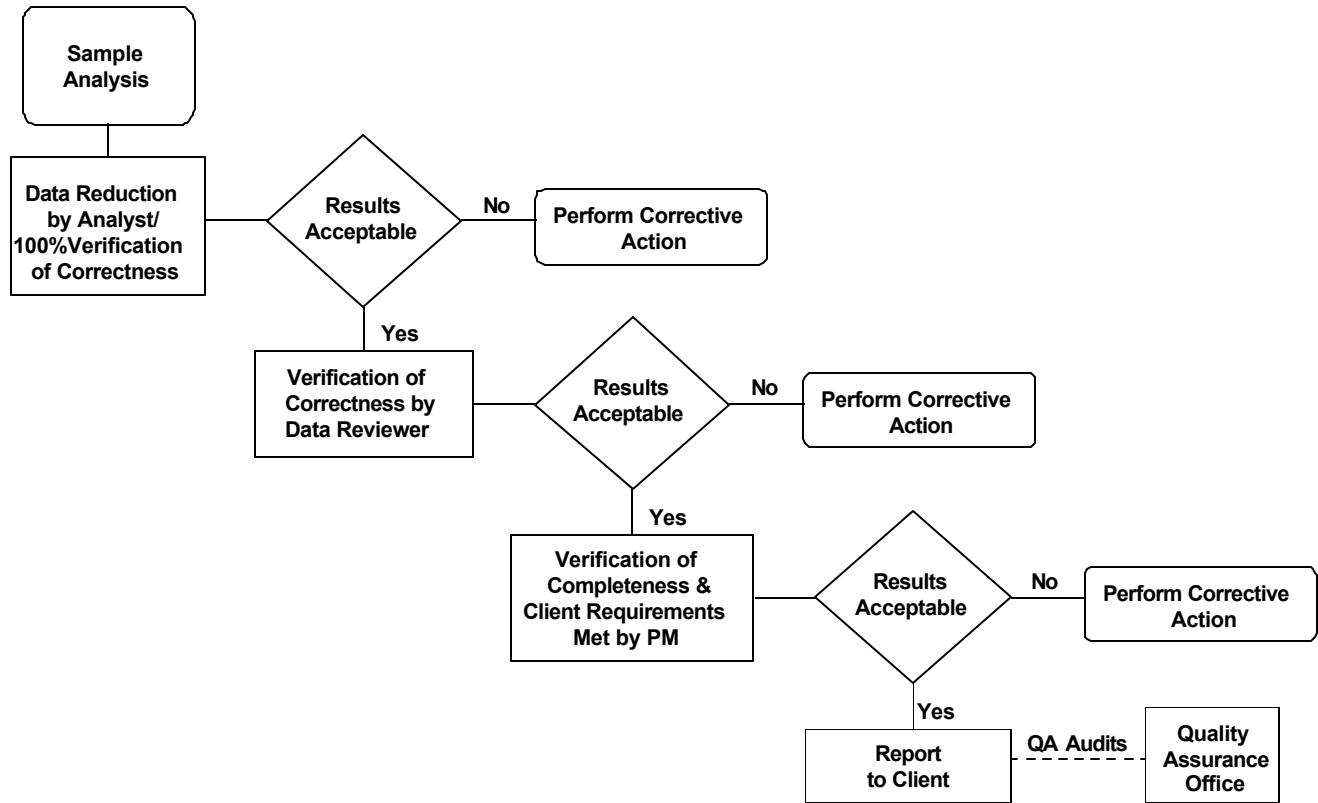
Date

Quality Assurance Officer

Signature

Date

Figure 19-2. Work Flow



20. EQUIPMENT AND CALIBRATIONS

- 20.1. The laboratory purchases technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency, and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to ensure that it meets its intended requirements. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory equipment and instrumentation is presented in Table 20-1.
- 20.2. Equipment is only operated by authorized and trained personnel. Manufacturers' instructions for equipment use are readily accessible to all appropriate laboratory personnel on the laboratory intranet.
- 20.3. Preventive Maintenance
 - 20.3.1. The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.
 - 20.3.2. Routine preventive maintenance procedures and frequency, such as lubrication, cleaning, and replacements, are performed according to the procedures outlined in the manufacturer's manual. Qualified personnel also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.
 - 20.3.3. Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Group Leader to ensure instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures are also outlined in analytical SOPs or instrument manuals. (Note: For some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear which instrument is associated with an entry.)
 - 20.3.4. Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs must be kept for all major pieces of equipment. Instrument Maintenance Logbooks may also be used to specify instrument parameters.
 - 20.3.5. Documentation must include all major maintenance activities such as contracted preventive maintenance and service, upgrades, and in-house

activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning, and adjustments.

20.3.6. Each entry in the instrument log includes the Analyst's initials, date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control, e.g., CCV run on 'date' was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records. A return to service date must be documented in the logbook.

20.3.7. When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled-in page must be signed across the page entered and the logbook, so it is clear that a page is missing if only half a signature is found in the logbook. At a minimum, if an instrument is sent out for service or transferred to another facility it must be recalibrated upon installation and the laboratory MDL must be verified (using an MDLV) prior to return to laboratory operation.

20.4. Instrument Repair

20.4.1. If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has been shown to be defective or outside of specified limits) it must be taken out of operation and tagged as out of service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory must examine whether this defect had any effect on previous analyses.

20.5. Equipment Malfunction

20.5.1. In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor, manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Backup instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the backup is not available and the analysis cannot be carried out within the needed timeframe, the samples must be subcontracted.

20.6. Instrument Transfer or Send-Out

20.6.1. If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study) prior to return to lab operations.

20.7. Support Equipment

20.7.1. This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to balances, ovens, refrigerators, freezers, incubators, water baths, field sampling devices, temperature measuring devices, dispensing devices, if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document method performance.

20.8. Weights and Balances

20.8.1. The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

20.8.2. Each balance is checked prior to initial serviceable use with at least two certified ASTM Type 1 weights spanning its range of use (weights that have been calibrated to ASTM Type 1 weights may also be used for daily verification). ASTM Type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every five years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM Type 1 weights).

20.8.3. All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards and the error term inherent in the calibration.

20.8.4. All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file. Reference SOP NC-QA-015, Equipment Monitoring and Thermometer Calibration. A list of balances is in Table 21.2.

20.9. pH, Conductivity, and Turbidity Meters

20.9.1. The pH meters used in the laboratory are accurate to + 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

20.9.2. Conductivity meters are also calibrated before each use with a known standard to demonstrate that the meters do not exceed an error of 1% or one umhos/cm.

20.9.3. Turbidity meters are also calibrated before each use. All of this information is documented in logs.

20.9.4. Consult pH, Conductivity, and Turbidity SOPs for further information.

20.10. Thermometers

- 20.10.1. All thermometers are calibrated on an annual basis with a NIST-traceable thermometer at temperatures bracketing the range of use. IR thermometers, digital probes, thermocouples, refrigerator thermometers (not NIST-Traceable), and freezer thermometers (not NIST –Traceable) are calibrated quarterly. IR Thermometers should be calibrated over the full range of use, including ambient, iced (4 degrees) and frozen (0 to -5 degrees), per the Drinking Water Manual.
- 20.10.2. The mercury/digital NIST thermometer is recalibrated every two to five years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.
- 20.10.3. All of this information is documented in logsheets. Monitoring of method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method-specific logsheets. More information on this subject can be found in SOP NC-QA-015, Equipment Monitoring and Thermometer Calibration.

20.11. Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators

- 20.11.1. The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day (seven days a week for DOD labs).
- 20.11.2. Ovens, waterbaths and incubators are monitored on days of use.
- 20.11.3. All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.
- 20.11.4. Sample storage refrigerator temperatures are kept between or $4 \pm 2^\circ\text{C}$.
- 20.11.5. Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.
- 20.11.6. All of this information is documented in Daily Temperature Logsheets posted on each unit or saved electronically if an electronic monitoring system (such as Temp Guard) is used.

20.12. Autopipettors, Dilutors, and Syringes

- 20.12.1. Mechanical volumetric dispensing devices including burettes (except Class A glassware and glass microliter syringes) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.
- 20.12.2. Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an "Accuracy and Precision Statement of Conformance" from Hamilton attesting established accuracy.
- 20.12.3. The laboratory maintains a sufficient inventory of autopipettors, and dilutors of differing capacities that fulfill all method requirements.
- 20.12.4. These devices are given unique identification numbers, and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.
- 20.12.5. Any device not regularly verified cannot be used for any quantitative measurements.

20.13. Field Sampling Devices (ISCO Autosamplers)

- 20.13.1. Each autosampler (ISCO) is assigned a unique identification number in order to keep track of the calibration. This number is recorded on the sampling documentation in a logbook.
- 20.13.2. The autosampler is calibrated semi-annually by setting the sample volume to 100ml and recording the volume received. The results are filed in a logbook/binder. The autosampler is programmed to run three cycles, and each of the three cycles is measured into a beaker to verify 100 ml are received.
- 20.13.3. If the RSD (Relative Standard Deviation) between the three cycles is greater than 20%, the procedure is repeated. If the result is still greater than 20%, the following options may be employed:
 - 20.13.3.1. The unit is taken out of service.
 - 20.13.3.2. The unit is used to pull composite samples only over a 24-hour period.
 - 20.13.3.3. The results of this check are kept in a logbook/binder.

20.14. Instrument Calibrations

- 20.14.1. Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

- 20.14.2. Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)
- 20.14.3. Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method, or program.
- 20.14.4. If the initial calibration results are outside of the acceptance criteria, action is performed and any affected samples are re-analyzed, if possible. If re-analysis is not possible, any data associated with an unacceptable initial calibration must be reported with appropriate data qualifiers (refer to Section 12). All sample analyses reported for Ohio VAP certified data must be associated with a valid calibration.

Note: Instruments are calibrated initially and as needed after that and at least annually.

20.15. Calibration Standards

- 20.15.1. Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of three calibration points (exception being ICP and ICP/MS methods) will be used.
- 20.15.2. Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.
- 20.15.3. The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).
- 20.15.4. The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to at least the same number of significant figures used to report the data) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exception to these rules is ICP methods or other methods where the referenced method does not specify two or more standards.
- 20.15.5. All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or

vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.16. Calibration Verification

20.16.1. The calibration relationship established during the initial calibration must be verified initially (with a second source ICV) and at least daily (with a CCV) as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 TNI Standard. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models.

20.16.2. Note: The process of calibration verification referred to is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

20.16.3. All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met, i.e., RPD, per 2009 TNI Standard EL-V1M4 Section 1.7.2.

20.16.4. All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Note: If an internal standard calibration is being used (e.g., most GCMS methods), then bracketing standards are not required. Only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

20.16.5. Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample or standard that can be injected within 12 hours of the beginning of the shift.

20.16.6. A continuing calibration verification (CCV) standard must be repeated at the beginning and, for methods that have quantitation by external

calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements (see specific SOPs). Most Inorganic methods require the CCV to be analyzed after every 10 samples or injections including matrix or batch QC samples.

Note: If an internal standard calibration is being used, then bracketing standards are not required. Only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

- 20.16.7. If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed and documented, the laboratory shall demonstrate acceptable instrument / method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.
- 20.16.8. Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with unacceptable calibration verification may be fully useable under the following special conditions and reported based upon discussion and approval of the client.
- 20.16.9. When acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported with a footnote or case narrative explaining the high bias. Otherwise, the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated, and accepted; or
- 20.16.10. When the acceptance criteria for the CCV are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated, and accepted.
- 20.16.11. Samples reported by the two conditions identified above will be appropriately flagged.

20.17. Verification of Linear Calibrations

- 20.17.1. Calibration verification for linear calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the laboratory method SOPs.) Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

- 20.17.2. Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.
- 20.17.3. When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.
- 20.17.4. When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. For Ohio VAP samples, results may not be reported when calibration verifications are exceeded low.

20.18. Tentatively Identified Compounds (TICs) – GC/MS Analysis

- 20.18.1. 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. TICs cannot be reported as “VAP certified” data for Ohio VAP projects.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

- 20.18.2. 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.

20.19. GC/MS Tuning

- 20.19.1. Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.
- 20.19.2. Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Table 20-1. Laboratory Equipment and Instrumentation

| Instrument Type | Manufacturer/ID | Model/Serial Number | Year into Service |
|-----------------------------|------------------------|--|-------------------|
| GC/MS Volatiles Instrument | Hewlett-Packard (UX2) | 5971A-5890, S/N US00029070 (screening) | 1992 |
| | Hewlett-Packard (HP6) | 5973-6890, S/N US00005571 (screening) | 1998 |
| | Hewlett-Packard (UX7) | 5973-6890, S/N US00010937 (screening) | 1998 |
| | Hewlett-Packard (UX8) | 5973-6890, S/N US00027773 | 1999 |
| | Hewlett-Packard (UX9) | 5973-6890, S/N US00028329 | 2000 |
| | Hewlett-Packard (UX10) | 5973-6890, S/N US00032072 | 2000 |
| | Agilent (UX11) | 5973-6890, S/N US00038093 | 2000 |
| | Agilent (UX12) | 5973-6890, S/N US10202133 | 2002 |
| | Agilent (UX14) | 5973-6890, S/N CN10340027 | 2003 |
| | Agilent (UX15) | 5973-6890, S/N CN10515062 | 2005 |
| | Agilent (UX16) | 5975-6890, S/N CN10539065 | 2005 |
| | Agilent (UX17) | 5975-7890, S/N US10831043 | 2012 |
| | Agilent (UX18) | 5973-6890, S/N US00020913 | 2013 |
| GC/MS Volatiles Autosampler | OI Analytical (UX2) | 4552, S/N 12019(screening) | 1999 |
| | OI Analytical (HP6) | 4552, S/N 12258 , 12151(screening) | 1998 |
| | OI Analytical (UX7) | 4552, S/N 13154 (screening) | 1998 |
| | OI Analytical (UX8) | 4552, S/N 13089 | 1999 |
| | OI Analytical (UX9) | 4552, S/N 13667 | 2000 |
| | OI Analytical (UX10) | 4552, S/N 12058 | 2000 |
| | OI Analytical (UX11) | 4552, S/N 13408 | 2000 |
| | OI Analytical (UX12) | 4552, S/N 12075 | 2002 |
| | OI Analytical (UX14) | 4552, S/N 14092 | 2003 |
| | OI Analytical (UX15) | 4552, S/N 14368 | 2005 |
| | OI Analytical (UX16) | 4552, S/N 14519 | 2005 |
| | OI Analytical (UX17) | 4552, S/N US12160002 | 2012 |

| Instrument Type | Manufacturer/ID | Model/Serial Number | Year into Service |
|---|----------------------|--|-------------------|
| | OI Analytical (UX18) | 4552, S/N 14519 | 2013 |
| GC/MS Volatiles Purge and Trap | OI Analytical (UX2) | 4560, S/N N251460461 (screening) | 1999 |
| | OI Analytical (HP6) | Encon (screening) | 1998 |
| | OI Analytical (UX7) | 4560, S/N K822460889 (screening) | 2004 |
| | OI Analytical (UX8) | 4560, S/N B444466152P | 2004 |
| | OI Analytical (UX9) | 4560, S/N M946460832 | 2000 |
| | OI Analytical (UX10) | 4660, S/N BETA6 | 2003 |
| | OI Analytical (UX11) | 4560 S/N K811460270 | 2000 |
| | OI Analytical (UX12) | 4560, S/N NM041460393 | 2002 |
| | OI Analytical (UX14) | 4660 S/N D829466914P | 2008 |
| | OI Analytical (UX15) | 4660, S/N C511466149P | 2005 |
| | OI Analytical (UX16) | 4660, S/N D539446261P | 2005 |
| | OI Analytical (UX17) | 4660, S/N H224466292P | 2012 |
| | OI Analytical (UX18) | 4560, S/N N213460621 | 2013 |
| GC/MS Semivolatiles Instrument | Hewlett-Packard HP7 | 5973-6890, S/N US71190756- US00009247 | 1998 |
| | Hewlett-Packard HP9 | 5973-6890, S/N US91422379- US72020889 | 2000 |
| | Agilent HP10 | 5973-6890, S/N US33220074- CN10340002 | 2003 |
| | Agilent A4AG2 | 5975C-7890, S/N US71235692- CN10721110 | 2007 |
| GC Volatiles (GCV) Analyzer | Agilent (A) | 6890 FID, S/N US10402056 | 2004 |
| | Hewlett-Packard (O) | 6890 PID/FID, S/N US00007206 | 1997 |
| | Hewlett-Packard (Y) | 6890N PID/FID, S/N US10337062 | 2003 |
| | Agilent (Z) | 6890 EPC & PDD/FID, S/N 10205072 | 2000 |
| GCV Autosampler | OI Analytical (O) | Archon, S/N 13196 | 2000 |
| | OI Analytical (Y) | 4552, S/N 14045 | 1998 |
| | EST (A) | Archer 8100 SN 14280 | 2013 |
| | Agilent (Z) | 7694 S/N IT21111663 | 2000 |
| GCV Purge and Trap | OI Analytical (O) | 4560 S/N N336460661 | 2000 |
| | Tekmar (A) | 3000 S/N 93104002 | 1998 |
| | Tekmar (Y) | 3000 S/N 97155002 | 1993 |
| GC Semivolatiles (GCS) MeHg Analyzer | Agilent N | 7890 Atomic Fluorescence, S/N CN10820009 (MeHg) | 2008 |
| | Tekran (MHg) | 2700 S/N 025 | |
| GCS MeHg Autosampler | Tekran (MHg) | AIM3300 S/N 5143A 26273 | |
| | EST (N) | Centurion (MeHg) S/N CENT249041408 | 2008 |
| | Tekmar (N) | Stratum (MeHg) S/N US08141001 | 2008 |
| | Tekmar (NOT IN USE) | Stratum (MeHg) S/N US08140004 | 2008 |
| GCS MeHg Detector | PS Analytical | Model 10.750 (MeHg) | 2008 |

| Instrument Type | Manufacturer/ID | Model/Serial Number | Year into Service |
|--------------------------|------------------------------|---|-------------------|
| GCS Instruments | Hewlett-Packard (P1) | 6890 EPC & Dual ECD Y-Splitter S/N US00023208 | 1998 |
| | Hewlett-Packard (P2) | 6890 EPC & Dual ECD Y-Splitter S/N US00023512 | 1998 |
| | Hewlett-Packard (P3) | 6890 EPC & Dual ECD Y-Splitter S/N US00023674 | 1998 |
| | Hewlett-Packard (P4) | 6890 EPC & Dual ECD Y-Splitter S/N US00029531 | 1999 |
| | Hewlett-Packard (P5) | 6890 EPC & Dual ECD S/N US00029508 | 2010 |
| | Hewlett-Packard (P6) | 6890 EPC & Dual FID S/N US00032848 | 2000 |
| | Agilent (P9) | 6890N EPC & Dual ECD Y-Splitter S/N US10205045 | 2005 |
| | Agilent (P10) | 6890 EPC & Dual ECD Y-Splitter S/N US10151110 | 1999 |
| | Agilent (P11) | 6890N EPC & Dual ECD Y-Splitter S/N CN10517088 | 2004 |
| | Agilent (P12) | 6890N EPC & Dual ECD Y-Splitter S/N CN10512025 | 2005 |
| | Agilent (P13) | 6890N EPC & Dual ECD Y-Splitter S/N CN10435032 | 2004 |
| | Agilent (P14) | 7890 EPC & Dual FID S/N CN 10281044 | 2010 |
| | Agilent (P15) | 6890N EPC & Dual ECD Y-Splitter S/N CN10427010 | 2012 |
| GCS HPLC | Hewlett-Packard (L2) | HPLC 1100, S/N US82404153 | 1998 |
| Extractions Sonicator | Misonix | 3000 (self-tuning), S/N R1044 | 2005 |
| | Fisher | Ultrasonic Processor FB-705 S/N 80587G-14 | 2014 |
| Extractions pH Meter | Mettler Toledo | SevenEasy pH (self-calibrating) S/N 1228295055 | 2008 |
| | Denver Instrument (spare) | UB-5 S/N UB-5093011 | 2004 |
| Metals ICP | Thermo (I12) | ICAP 6500 Duo Trace Analyzer, S/N ICP 20101711 | 2014 |
| | Thermo (I9) | ICAP 6500 Duo Trace Analyzer, S/N ICP 20102403 | 2010 |
| Metals ICP/MS | Thermo (I11) | Series 2, S/N 01952C | 2013 |
| | Agilent (I10) | 7700x S/N JP12452145 | 2013 |
| Metals Mercury | Leeman (CVAA) (H1) | PS200 II, S/N HG9031 | 1999 |
| | Leeman (CVAA) (H4) | Hydra AA , S/N 6011 | 2006 |
| Metals Low Level Mercury | Leeman (CVAF) (H6) | Hydra AF Gold+, Install # 64264 | 2005 |
| | Leeman (CVAF) (H7) | Hydra AF Gold, Install #64547 | 2011 |
| WC Autotitrator | Man-Tech (Steve) | PC – Titrate, S/N MS-9K8-217 | 2001 |
| WC Block Digester | Andrews (Moe) | 110-40-EZ | 1999 |

| Instrument Type | Manufacturer/ID | Model/Serial Number | Year into Service |
|---------------------------|----------------------------|--|-------------------|
| | Andrews (Larry) | 110-40-PA | 1999 |
| | Andrews (Curly) | 110-40-PA | 1999 |
| | Lachat (Carol) | BD-46 TKN, S/N 00000993 | 2010 |
| | Lachat (Mike) | BD-46, S/N 1800-910 | 2014 |
| WC BOD | Mantech (Bugsy) | BOD, S/N MT-113-207 | 2014 |
| WC Conductivity | ManTech (Arnie) | 4310, S/N 1613 | 1989 |
| WC Cyanide | LabCrest MidiDist | PRG-2520-BL, S/N 1000-99-01 | 1999 |
| WC Discrete Analyzer | Kone (Barney) | Konelab 200, Z1718383 | 2001 |
| | Kone (Sauron) | Konelab 250, A2120021 | 2005 |
| | Systea (Maggie) | EasyChem Plus, S/N 07004 | 2013 |
| WC Dissolved Oxygen Meter | YSI | YSI 5100, 13D 100737 | 2014 |
| WC Flashpoint | Herzog (Whitey) | HFP 339, S/N 073390084 | 2007 |
| WC Ion Chromatograph | Dionex (Cecilia) | ICS 1500, S/N 03100737 | 2014 |
| | Dionex (Simon) | DX-120, S/N 98110093 | 1999 |
| | Dionex (Veronica) | ICS 2100, S/N 12031443 | 2012 |
| WC pH Meter | Orion pH Meter (Randolph) | Star A211, S/N X02404 | 2012 |
| | Orion (Ammonia ISE) (Dave) | 520A, S/N 48029 | 1996 |
| WC TOC | OI Analytical (Sparky) | 1010 TOC Analyzer, S/N K503710931 | 2005 |
| WC EOX | Thermo Electron (Brian) | 1200, S/N 2005.0234 | 2005 |
| WC Turbidimeter | HF Scientific (Jack) | Micro 100, S/N 200705143 | 2001 |
| WC UV/VIS | Genesys (Bert) | Spectronic 20, S/N 3SGL078016 | 1998 |
| | Genesys (Ernie) | Spectronic 20, S/N 3SGL226006 (Model 4001/4) | 2008 |
| WC Sulfide | Westco EasyDist | | 2008 |

Table 20-2. Schedule of Routine Maintenance

(Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations. Refer to the analytical SOP for frequency and criteria)

20.20. Instrument Maintenance Schedule

| ION CHROMATOGRAPH | | | |
|---|--------------------------|----------------------------|--|
| As Needed | Daily | Weekly | Monthly |
| Clean micro-membrane suppressor when decreases in sensitivity are observed. | Check plumbing/leaks | Check pump heads for leaks | Check all air and liquid lines for discoloration and crimping, if indicated. |
| Check fuses when power problems occur. | Check gases | Check filter (inlet) | Check/change bed supports guard and analytical columns, if indicated. |
| Reactivate or change column when peak shape and resolution deteriorate or when retention time shortening indicates that exchange sites have become deactivated. | Check pump pressure | | |
| De-gas pump head when flow is erratic. | Check conductivity meter | | |

| HIGH PRESSURE LIQUID CHROMATOGRAPH | |
|---|--|
| Daily | As Needed |
| Check level of solution in reservoirs. If adding, verify that solvent is from the same source. If changing, rinse gas and delivery lines to prevent contamination of the new solvent. | Replace columns when peak shape and resolution indicate that chromatographic performance of column is below method requirements. |
| Check gas supply. | Oil autosampler slides when sample does not advance. |
| Flush with an appropriate solvent to remove all bubbles. | Rinse flow cell with 1N nitric acid if sensitivity low. |
| Pre-filter all samples. | Change pump seals when flow becomes inconsistent. |
| | Repack front end of column Back-flush column. |

ICP AND ICP/MS

| Daily | Monthly or As Needed | Semi-Annually | Annually |
|---|--|--|---|
| Check vacuum pump gage. (<10 millitorr) | Clean plasma torch assembly to remove accumulated deposits | Change vacuum pump oil | Notify manufacturer service engineer for scheduled preventive maintenance service |
| Check cooling water supply system is full and drain bottle is not full. Also drain tubing is clear, tight fitting, and has few bends. | Clean nebulizer and drain chamber; keep free flowing to maintain optimum performance | Replace coolant water filter (may require more or less frequently depending on quality of water) | |
| Check nebulizer is not clogged | Clean filters on back of power unit to remove dust | | |
| Check capillary tubing is clean and in good condition | Replace when needed: - peristaltic pump tubing - sample capillary tubing - autosampler sipper probe | | |
| Check peristaltic pump windings are secure | - Check yttrium position - Check O-rings - Clean/lubricate pump rollers | | |
| Check high voltage switch is on | | | |
| Check torch, glassware, aerosol injector tube, and bonnet are clean | | | |

CVAS AND CVAFS

| Daily | As Needed | Annually |
|--------------------------------|----------------------|----------------|
| Change drying tube | Change pump tubing | Change Hg lamp |
| Check pump tubing/drain tubing | Check/change Hg lamp | |
| Check gas pressure | Clean optical cell | |
| Check aperture reading | Lubricate pump | |
| Check tubing | | |

GAS CHROMATOGRAPH

| Daily * | As Needed |
|---|---|
| Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures. | <p>Replace front portion of column packing or break off front portion of capillary columns. Replace column if this fails to restore column performance, or when column performance (e.g., peak tailing, poor resolution, high backgrounds, etc.) indicates it is required.</p> <p>Quarterly FID: clean detector, only as needed—not quarterly/or semi-annually.</p> |
| Check temperatures of injectors and detectors. Verify temperature programs by RT shift. | Change glass wool plug in injection port and/or replace injection port liner when front portion of column packing is changed or front portion of capillary column is removed. |
| Clean injector port weekly for TPH for 8015B, when breakdown fails; otherwise, when RT shift or bad samples run. | <p>Annually FID: replace flame tip, only as needed.</p> <p>Only as needed: ECD--detector cleaning and re-foiling, whenever loss of sensitivity, erratic response, or failing resolution is observed</p> |
| Check baseline level during analysis of run—not maintenance. | Perform gas purity check (if high baseline indicates that impure carrier gas may be in use). |
| <p>Watched weekly: check reactor temperature of electrolytic conductivity detector.</p> <p>Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks, when analyzing pesticides; part of analysis—not maintenance.</p> <p>Clip column leader when chromatography looks bad—not daily.</p> | <p>Replace or repair flow controller if constant gas flow cannot be maintained.</p> <p>Replace fuse.</p> <p>Reactivate external carrier gas dryers.</p> <p>Detectors: clean when baseline indicates contamination or when response is low. FID: clean/replace jet, replace ignitor. ECD: follow manufacturer's suggested maintenance schedule.</p> <p>Reactivate flow controller filter dryers when presence of moisture is suspected.</p> <p>HP 7673 Autosampler: replace syringe, fill wash bottle, dispose of waste bottle contents.</p> |

*No daily maintenance done on any instrument/method. Weekly change IPL on TPH instrument. Everything else is on an “as needed” basis.

MASS SPECTROMETER

| Daily | Weekly | As Needed | Quarterly | Annually |
|---|---|--|--|---|
| Check for sufficient gas supply. Check for correct column flow and/or inlet pressure. | Check mass calibration (PFTBA or FC-43) | Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed between maintenance. | Check ion source and analyzer (clean, replace parts as needed) | Replace the exhaust filters on the mechanical rough pump every 1-2 years. |
| Check temperatures of injector, detector. Verify temperature programs. | | Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels. | Check vacuum, relays, gas pressures and flows | |
| Check inlets, septa | | Clean Source, including all ceramics and lenses - the source cleaning is indicated by a variety of symptoms including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination. | Change oil in the mechanical rough pump. | |
| Check baseline level | | Repair/replace jet separator. | | |
| Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds. | | Replace filaments when both filaments burn out or performance indicates need for replacement. | | |

ANALYTICAL/TOP LOADING BALANCES

| Daily | Annually |
|---|---------------------------------------|
| Check using Class 1-verified weights once daily or before use Clean pan and weighing compartment | Manufacturer cleaning and calibration |

REFRIGERATORS/WALK-IN COOLERS

| Daily | As Needed |
|---------------------------------|---|
| Temperatures checked and logged | Refrigerant system and electronics serviced |

OVENS

| Daily | As Needed |
|---------------------------------|----------------------|
| Temperatures checked and logged | Electronics serviced |

SPECIFIC DIGITAL ION ANALYZER

| Daily | As Needed |
|--|----------------------|
| Daily when used: Calibrate with check standards Inspect electrode daily, clean as needed Inspect electrode proper levels of filling solutions daily; fill as needed Clean probe after each use | Electronics serviced |

TURBIDIMETER

| Daily | Monthly | As Needed |
|--|--------------------------|----------------------|
| Daily when used: Adjust linearity on varying levels of NTU standards. Standardize with NTU standards Inspect cells | Clean instrument housing | Electronics serviced |

DISSOLVED OXYGEN METER

| Daily | As Needed |
|--|--|
| Daily when used: Calibrate with saturated air Check probe membrane for deterioration Clean and replace membrane with HCl solution | Electronics serviced Clean and replace membrane with HCl solution |

CONDUCTANCE METER

| Daily | As Needed |
|---|----------------------|
| Daily when used: Check probe and cables Inspect conductivity cell | Electronics serviced |

CHEMICAL OXYGEN DEMAND (COD) REACTOR 1

| Daily | As Needed |
|--|----------------------|
| Daily when used: Calibrate with check standards | Electronics serviced |

SPECTROPHOTOMETER

| As Needed | Daily | Monthly | Annually |
|---|-----------------------------|---------------|--|
| Dust the lamp and front of the front lens | Check the zero % adjustment | Clean windows | Check instrument manual |
| | Clean sample compartment | | Perform wavelength calibration |
| | Clean cuvettes | | Replace lamp annually or when erratic response is observed |
| | | | Clean and align optics |

pH METER

| As Needed | Daily |
|----------------------------|---|
| Clean electrode | Inspect electrode. Verify electrodes are properly connected and filled |
| Refill reference electrode | Inspect electrode proper levels of filling solutions. Make sure electrode is stored in buffer |

TOTAL ORGANIC CARBON ANALYZER

| Daily | As Needed | Weekly | Monthly |
|---|--|--|---|
| Check: Oxygen supply Persulfate supply Acid supply Carrier gas flow rate (~ 150 cc/min) IR millivolts for stability (after 30 min. warm-up) Reagent reservoirs | Check injection port septum after 50-200 runs Tube end-fitting connections after 100 hours or use Indicating drying tube NDIR zero, after 100 hours of use Sample pump, after 2000 hours for use Digestion vessel/condensation chamber, after 2000 hours of use Permeation tube, after 2000 hours of use NDIR cell, after 2000 hours of use Change pump tubing | Check liquid-flow- rate-pump-tubing conditions on autosampler Check injection port septum | Clean digestion vessel Clean condenser column Do the leak test |

Digestion Block

| |
|---|
| Annually |
| Check temperature with NIST thermometer |

Flash Point Tester

| |
|---|
| Daily |
| Check tubing Clean sample cup each use |
| Check gas |
| Clean flash assembly |
| Check stirrer |

Table 20-3. Preventive Maintenance Procedures

(Note: Refer to the analytical SOP for frequency and criteria.)

SUMMARY OF INORGANIC METHOD CALIBRATIONS

| Analysis | Calibration | NPDES 1 | | RCRA (SW846) 2 | |
|--|-------------|----------------|---|----------------|-------------|
| | | Method | Requirement | Method | Requirement |
| Alkalinity, Bicarbonate, Carbonate | Initial | 310.1 2320B | 2 point calibration of pH meter ± 0.05 pH units of true value | -- | N/A |
| | Continuing | 310.1 2320B | One buffer check ± 0.05 pH units of true value Everyone 10 samples | -- | N/A |
| | Ending | 310.1 2320B | N/A | -- | N/A |
| Ammonia | Initial | 350.1 | 6 levels including blank, "r" 3 ≥ 0.995 | -- | N/A |
| | Continuing | 350.1 | One level or LCS every 10 samples ± 10% of true value | -- | N/A |
| | Ending | 350.1 | One level or LCS every 10 samples ± 10% of true value | -- | N/A |

| Analysis | Calibration | NPDES 1 | | RCRA (SW846) 2 | |
|--|-------------|------------------|---|----------------|-------------|
| | | Method | Requirement | Method | Requirement |
| Biochemical Oxygen Demand (BOD) | Initial | 405.1 SM5210B | a. Winkler titration: Iodometric with standard thiosulfate b. Membrane electrode: Read in air and in water with zero dissolved oxygen | -- | N/A |
| | Continuing | 405.1 SM5210B | N/A | -- | N/A |
| | Ending | 405.1 SM5210B | N/A | -- | N/A |

| Analysis | Calibration | NPDES 1 | | RCRA (SW846) 2 | |
|--|-------------|------------------|---|----------------|--|
| | | Method | Requirement | Method | Requirement |
| Anions, Bromide, Chloride, Fluoride, Sulfate, Nitrite, Nitrate, O- Phos | Initial | 300.0A | 5 levels plus a blank, "r" $3 \geq 0.995$ | 9056A | 5 levels plus a blank, "r" $3 \geq 0.995$ |
| | Continuing | 300.0A | Level every 10 samples $\pm 10\%$ of true value | 9056A | N/A |
| | Ending | 300.0A | N/A | 9056A | N/A |
| Chemical Oxygen Demand (COD) | Initial | 410.4 SM5220D | 5 levels plus a blank "r" $3 \geq 0.995$ | -- | N/A |
| | Continuing | 410.4 SM5220D | One level every 10 samples $\pm 10\%$ of true value | -- | N/A |
| | Ending | 410.4 SM5220D | One level $\pm 10\%$ of true value | -- | N/A |

| Analysis | Calibration | NPDES 1 | | RCRA (SW846) 2 | |
|------------------|-------------|----------------------|---|----------------|---|
| | | Method | Requirement | Method | Requirement |
| Chloride | Initial | 325.2 SM4500 Cl-E | 5 levels plus blank "r" $3 \geq 0.995$ | 9251 | 5 levels plus blank "r" $3 \geq 0.995$ |
| | Continuing | 325.2 SM4500 Cl-E | One level every 10 samples $\pm 10\%$ of true value | 9251 | One level every 10 samples, $\pm 10\%$ of true value |
| | Ending | 325.2 SM4500 Cl-E | One level every 10 samples $\pm 10\%$ of true value | 9251 | Method 9056 : N/A Method 9252: One level $\pm 10\%$ of true value |
| Chromium Cr+6 | Initial | 3500 Cr-B | 3 levels plus blank | 7196A | 5 levels plus blank "r" $3 \geq 0.995$ |
| | Continuing | 3500 Cr-B | One level every 10 samples $\pm 10\%$ of true value | 7196A | One level every 10 samples $\pm 15\%$ |

| Analysis | Calibration | NPDES 1 | | RCRA (SW846) 2 | |
|-----------------------|-------------|---------------------|----------------------------------|----------------|--------------------|
| | | Method | Requirement | Method | Requirement |
| | Ending | 3500 Cr-B | One level ± 10% of true value | 7196A | One level ± 15% |
| Chlorine, Residual | Initial | 330.5 SM4500CL-G | N/A | -- | N/A |
| | Continuing | 330.5 SM4500CL-G | N/A | -- | N/A |
| | Ending | 330.5 SM4500CL-G | N/A | -- | N/A |

| Analysis | Calibration | NPDES 1 | | RCRA (SW846) 2 | |
|-----------------------|-------------|---|---|----------------|--|
| | | Method | Requirement | Method | Requirement |
| Conductivity | Initial | 120.1 SM2510B | Standard KCl solution | 9050A | One level to determine cell constant |
| | Continuing | 120.1 SM2510B | N/A | 9050A | N/A |
| | Ending | 120.1 SM2510B | N/A | 9050A | N/A |
| Cyanide (Amenable) | Initial | 335.1 SM4500CN-G | 6 levels plus blank "r" 3 ≥ 0.995 | 9012A, B | 6 levels plus blank "r" 3 ≥ 0.995 |
| | Continuing | 335.1 SM4500CN-G | One level every 10 samples ± 10% of true | 9012A, B | One mid-level every 10 samples ± 15% of true value |
| | Ending | 335.1 SM4500CN-G | One level ± 10 % of true value | 9012A, B | ± 15% of true value |
| Cyanide (Total) | Initial | 335.2 335.4 SM4500CN-E 335.2-CLP-M (Ohio VAP) | 6 levels plus blank "r" 3 ≥ 0.995 | 9012A, B | 6 levels plus blank "r" 3 ≥ 0.995 |
| | Continuing | 335.2 335.4 SM4500CN-E 335.2-CLP-M (Ohio VAP) | One mid-level every 10 samples ± 10 % of true value | 9012A, B | One mid-level every 10 samples ± 15% of true value |
| | Ending | 335.2 335.4 SM4500CN-E 335.2-CLP-M (Ohio VAP) | One mid-level ± 10 % of true value | 9012A, B | ± 15% of true value |

| Analysis | Calibration | NPDES 1 | | RCRA (SW846) 2 | |
|------------------------------------|-------------|--------------|---|----------------|---|
| | | Method | Requirement | Method | Requirement |
| Cyanide (Weak Acid Dissociable) | Initial | SM 4500 CN-I | 6 levels plus blank "r" $3 \geq 0.995$ | | |
| | Continuing | SM 4500 CN-I | One mid-level every 10 samples $\pm 10\%$ of true value | | |
| | Ending | SM 4500 CN-I | One mid-level $\pm 10\%$ of true value | | |
| Flashpoint | Initial | -- | N/A | 1010, 1010A | p-Xylene reference standard must have flashpoint of 81oF ± 2 oF |
| | Continuing | -- | N/A | 1010, 1010A | N/A |
| | Ending | -- | N/A | 1010, 1010A | N/A |

| Analysis | Calibration | NPDES 1 | | RCRA (SW846) 2 | |
|----------------|-------------|---------------------------------|---|----------------|-------------|
| | | Method | Requirement | Method | Requirement |
| Fluoride | Initial | 340.2 SM 4500 F-C | 5 levels "r" $3 \geq 0.995$ | | |
| | Continuing | 340.2 SM 4500 F-C | One mid-level every 10 samples $\pm 10\%$ of true value | | |
| | Ending | 340.2 SM 4500 F-C | One mid-level $\pm 10\%$ of true value | | |
| Hardness | Initial | 130.2 SM 2340B SM2340C | Method 130.2: Standardize titrant Method 2340B: See ICP Metals 200.7 | -- | N/A |
| | Continuing | 130.2 SM2340B SM2340C | Method 130.2: N/A Method 2340B: See ICP Metals 200.7 | -- | N/A |
| | Ending | 130.2 SM2340B SM2340C | Method 130.2: N/A Method 2340B: See ICP Metals 200.7 | -- | N/A |
| Iron (Ferrous) | Initial | SM3500- Fe B | 3 levels plus a blank, "r" $3 \geq 0.995$ | - | N/A |

| Analysis | Calibration | NPDES 1 | | RCRA (SW846) 2 | |
|----------|-------------|-------------|---|----------------|-------------|
| | | Method | Requirement | Method | Requirement |
| | Continuing | SM3500-Fe B | One mid-level every 10 samples ± 10% of true value | - | N/A |
| | Ending | SM3500-Fe B | One mid-level ± 10% of true value | - | N/A |

| Analysis | Calibration | NPDES 1 | | RCRA (SW846) 2 | |
|---|-------------|--------------------|---|----------------------------------|--|
| | | Method | Requirement | Method | Requirement |
| Phosphorus (Total and Ortho-phosphate) | Initial | 365.1 SM4500P-E | 5 levels plus a blank | -- | N/A |
| | Continuing | 365.1 SM4500P-E | One level for every 10 samples. ±10% of true value | -- | N/A |
| | Ending | 365.1 SM4500P-E | ±10% of true value | -- | N/A |
| pH | Initial | 150.1 SM4500H-B | 2 level calibration that bracket the expected pH of the sample ± 0.05 pH units of true value | 9040B 9040C 9041A 9045C | 2 point calibration ± 0.05 pH units of true value |
| | Continuing | 150.1 SM4500H-B | One buffer check every 10 samples ± 0.05 pH units true value | 9040B 9040C 9041A 9045C | N/A |
| | Other | 150.1 SM4500H-B | Third point check | 9040B 9040C 9041A 9045C | Third point check |
| | Ending | 150.1 SM4500H-B | One buffer check ± 0.05 pH units of true value | 9040B 9040C 9041A 9045C | N/A |

| Analysis | Calibration | NPDES 1 | | RCRA (SW846) 2 | |
|-------------------|-------------|------------------|--|----------------|--|
| | | Method | Requirement | Method | Requirement |
| Phenolics | Initial | 420.1 | 5 levels plus a blank "r" $3 \geq 0.995$ | 9065 | 5 levels plus a blank "r" $3 \geq 0.995$ |
| | Continuing | 420.1 | One mid-level every 10 samples $\pm 10\%$ true value | 9065 | One mid-level $\pm 10\%$ true value |
| | Ending | 420.1 | One mid-level $\pm 10\%$ true value | 9065 | One mid-level $\pm 10\%$ true value |
| Settleable Solids | Initial | 160.5 SM2540F | N/A | -- | N/A |
| | Continuing | 160.5 SM2540F | N/A | -- | N/A |
| | Ending | 160.5 SM2540F | N/A | -- | N/A |
| Sulfate | Initial | 375.4 | Method 375.4: 3 levels plus blank "r" $3 \geq 0.995$ | 9038 | 3 levels plus a blank for every hour of continuous sample analysis. |

| Analysis | Calibration | NPDES 1 | | RCRA (SW846) 2 | |
|------------------------------|-------------|-------------------------|--|----------------|---|
| | | Method | Requirement | Method | Requirement |
| Sulfate (Cont'd) | Continuing | 375.4 | One level every 3 or 4 samples ± 10% of true value | 9038 | Independent-prepared check standard every 15 samples |
| | Ending | 375.4 | ± 10% of true value | 9038 | N/A |
| Sulfide | Initial | 376.1 SM4500S 2-F | This is a titration method. Therefore, calibrations are not applicable. | 9030B/ 9034 | This is a colorimetric titration. Therefore, calibration is not applicable. |
| | Continuing | 376.1 SM4500S 2-F | N/A | 9030B/ 9034 | This is a colorimetric titration. Therefore, calibration is not applicable. |
| | Ending | 376.1 SM4500S 2-F | N/A | 9030B/ 9034 | This is a colorimetric titration. Therefore, calibration is not applicable. |
| Total Dissolved Solids | Initial | 160.1 SM2540C | This is a gravimetric determination. Calibrate balance prior to analysis | -- | N/A |
| | Continuing | 160.1 SM2540C | | -- | N/A |
| | Ending | 160.1 SM2540C | | -- | N/A |

| Analysis | Calibration | NPDES 1 | | RCRA (SW846) 2 | |
|-----------------------------------|-------------|----------------------|--|--------------------------|---|
| | | Method | Requirement | Method | Requirement |
| Total Kjeldahl Nitrogen (TKN) | Initial | 351.3 SM4500NH3-C | Method 351.3: Titrimetric: Standardize titrant Colorimetric: 7 levels plus blank | -- | N/A |
| | Continuing | 351.3 SM4500NH3-C | Method 351.3: N/A | -- | N/A |
| | Ending | 351.3 SM4500NH3-C | Method 351.3: N/A | -- | N/A |
| Total Organic Carbon (TOC) | Initial | 415.1 SM5310C | 3 levels plus blank | 9060 Walkley Black | 3 levels plus blank "r" $3 \geq 0.995$ |
| | Continuing | 415.1 SM5310C | 1 mid-level every 10 samples $\pm 10\%$ of true value | 9060 Walkley Black | 1 mid-level every 10 samples $\pm 15\%$ of true value |
| | Ending | 415.1 SM5310C | $\pm 10\%$ of true value | 9060 Walkley Black | $\pm 15\%$ of true value |
| Extractable Organic Halides (EOX) | Initial | | | 9023 | Daily instrument calibration standard and blank in duplicate $\pm 10\%$ of true value (calibration standard) Verify with independently- prepared check standard –ICV $\pm 10\%$ |

| Analysis | Calibration | NPDES 1 | | RCRA (SW846) 2 | |
|--|-------------|---------------|---|----------------|-----------------------------|
| | | Method | Requirement | Method | Requirement |
| Extractable Organic Halides (EOX) (cont'd) | Continuing | | | 9023 | CCV \pm 10% of true value |
| | Ending | | | 9023 | CCV \pm 10% of true value |
| Total Solids | Initial | 160.3 | This is a gravimetric determination. Calibrate balance before use. | -- | N/A |
| | Continuing | 160.3 | | -- | N/A |
| | Ending | 160.3 | | -- | N/A |
| Total Suspended Solids (Nonfilterable) | Initial | 160.2 SM2540D | This is a gravimetric determination. Calibrate balance before use. | -- | N/A |
| | Continuing | 160.2 SM2540D | | -- | N/A |
| | Ending | 160.2 SM2540D | | -- | N/A |
| Turbidity | Initial | 180.1 | Minimum of 1 level in each instrument range. Follow manufacturer's instructions | -- | N/A |
| | Continuing | 180.1 | \pm 10% of true value | -- | N/A |
| | Ending | 180.1 | \pm 10% of true value | -- | N/A |

| Analysis | Calibration | NPDES 1 | | RCRA (SW846) 2 | |
|--------------------------------------|-------------|---------|---|----------------|--|
| | | Method | Requirement | Method | Requirement |
| ICP & ICP/MS Metals (excludes Hg) | Initial | 200.7 | One level and blank. ICV RSD <3% from replicate - daily | 6010B 6010C | One level and blank. ICV RSD <5% from replicate - daily |
| | Initial | 200.8 | One level and blank | 6020 6020A | One level and blank |
| | Continuing | 200.7 | Every 10 samples ±10% of true value CCV RSD < 5% from replicate | 6010B 6010C | Mid-level calibration standard Every 10 samples ± 10% of true value CCV RSD < 5% from replicate |
| | Continuing | 200.8 | N/A | 6020 6020A | N/A |
| | Ending | 200.7 | ±10% of true value CCV RSD < 5% from replicate | 6010B 6010C | Mid-level calibration standard ± 10% of true value CCV RSD < 5% from replicate |
| | Ending | 200.8 | N/A | 6020 6020A | N/A |
| | Other | 200.7 | ICSA, ICSAB: Analyze at beginning of run. For ICSA, AB criteria see SOP Semi-Annually: ICP interelement correction factors Instrument detection limits | 6010B 6010C | ICSA, ICSAB: Analyze at beginning of run. For ICSA, AB criteria see SOP Semi-Annually: ICP interelement correction factors Instrument detection limits |
| | Other | 200.8 | N/A | 6020 6020A | N/A |

| Analysis | Calibration | NPDES 1 | | RCRA (SW846) 2 | |
|-------------------------------|-------------|-----------------|---|-------------------------|--|
| | | Method | Requirement | Method | Requirement |
| Mercury by CVAA & CVAFS | Initial | 245.1 1631E | 5 levels plus blank ICV $\pm 10\%$ of true value "r" ≥ 0.995 | 7470A 7471A 7471B | 5 levels plus blank ICV $\pm 10\%$ of true value "r" ≥ 0.995 |
| | Continuing | 245.1* 1631E | Daily or every 10 samples, whichever is more frequent $\pm 20\%$ of true value | 7470A 7471A 7471B | Every 10 samples $\pm 20\%$ of true value |
| | Ending | 245.1 1631E | $\pm 20\%$ of true value | 7470A 7471A 7471B | $\pm 20\%$ of original prepared standard |
| | Other | 245.1 1631E | Annually: MDL | 7470A 7471A 7471B | Annually: MDL |

* 245.1 continuing – Initial CCV $\pm 5\%$ of true value

Footnotes

- 1 *National Pollutant Discharge Elimination System.*
- 2 *Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December, 1996).*
- 3 "r" = correlation coefficient.

SUMMARY OF ORGANIC METHOD CALIBRATIONS

| Analysis | Calibration | NPDES 1 | | RCRA (SW846) 2 | |
|---------------------|-------------|---------|-------------|----------------|--|
| | | Method | Requirement | Method | Requirement |
| Herbicides by GC | Initial | | | 8151A | Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed. |
| | Continuing | | | 8151A | Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported. |
| | Ending | | | 8151A | Mid-level calibration standard. % D < 15% of predicted response for any analyte quantitated and reported. |

| Analysis | Calibration | NPDES 1 | | RCRA (SW846) 2 | |
|---|-------------|---------|---|---------------------------------|--|
| | | Method | Requirement | Method | Requirement |
| Pesticides/ PCBs by GC | Initial | 608 | Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed | 8081A 8081B 8082 8082A | Minimum of 5 levels. If % RSD < 20%, use avg RF. Otherwise, calibration curve employed. (See SOP NC-GC-038) |
| | Continuing | 608 | One or more calibration standards analyzed daily. % D \pm 15% of predicted response | 8081A 8081B 8082 8082A | Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported. |
| | Ending | 608 | N/A | 8081A 8081B 8082 8082A | Mid-level calibration standard. % D < 15% of predicted response for any analyte quantitated and reported. |
| | Other | 608 | N/A | 8081A 8081B 8082 8082A | N/A |
| Petroleum Hydrocarbons /Oil and Grease | Initial | 1664A | Calibrate analytical balance at 2 mg and 1000 mg Calibration must be \pm 10% at 2 mg and \pm 0.5% at 1000 mg or recalibrate balance | | |
| | Continuing | 1664A | N/A | | |
| | Ending | 1664A | N/A | | |

| Analysis | Calibration | NPDES 1 | | RCRA (SW846) 2 | |
|---------------|-------------|---------|---|----------------|---|
| | | Method | Requirement | Method | Requirement |
| Semivolatiles | Initial | 625 | Minimum of 3 levels, lowest near but above MDL. If % RSD \leq 35%, use avg RF. Otherwise calibration curve employed. | 8270C 8270D | Minimum of 5 levels, % RSD for RF for CCCs(4) < 30% SPCCs(5): RF > 0.050 |
| | Continuing | 625 | One level every 24 ours. Acceptance criteria are found in the method and SOP. | 8270C 8270D | Mid-level standard every 12 hours (after tuning) %D for CCCs(4) < 20 % between RF from standard and avg RF from initial SPCCs(5): RF > 0.050. |
| | Ending | 625 | N/A | 8270C 8270D | N/A |
| | Other | 625 | DFTPP(7) tuning every 24 hours before standard or sample runs. | 8270C 8270D | DFTPP(7) tuning at the beginning of every 12 hour shift. |

| Analysis | Calibration | NPDES 1 | | RCRA (SW846) 2 | |
|-----------|-------------|---------|--|----------------|---|
| | | Method | Requirement | Method | Requirement |
| Volatiles | Initial | 624 | Minimum of 3 levels, lowest near but above MDL. If % RSD \leq 35%, use avg RF. Otherwise, calibration curve employed. | 8260B 8260C | Minimum of 5 levels, %RSD for RF for CCCs4 < 30.0% SPCCs5:RF \geq 0.300 for Chlorobenzene and 1,1,2,2-tetrachloroethane, Chloromethane and 1,1-dichloroethane, and RF > 0.100 for Bromoform |
| | Continuing | 624 | 1 level every 24 hours Acceptance criteria are found in the method and SOP | 8260B 8260C | Mid-level standard every 12 hours (after tuning) %Drift for CCCs(4) < 20.0% between RF from standard and avg RF from initial SPCCs(5): RF \geq 0.300 for Chlorobenzene and 1,1,2,2-tetrachloroethane, Chloromethane and 1,1-dichloroethane, and RF > 0.100 for Bromoform |
| | Ending | 624 | N/A | 8260B 8260C | N/A |
| | Other | 624 | BFB(6)tuning at the beginning of every 24 hour shift. | 8260B 8260C | BFB(6)tuning at the beginning of every 12 hour shift. |

Footnotes:

- 1 National Pollutant Discharge Elimination System.
- 2 Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).
- 3 TCDD - 2,3,7,8-Tetrachlorodibenzo-p-dioxin.
- 4 CCC - Continuing Calibration Compounds.
- 5 SPCC - System Performance Check Compound.
- 6 BFB – Bromofluorobenzene.
- 7 DFTPP – Decafluorotriphenylphosphine.
- 8 Footnote deleted.
- 9 Method not listed in 40 CFR Part 136.

21. MEASUREMENT TRACEABILITY

21.1. Traceability of measurements must be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard must be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices (refer to Section 20.3). With the exception of Class A glassware and glass microliter syringes, quarterly accuracy checks are performed for all mechanical volumetric devices. Microsyringes are verified at least semi-annually or disposed of after six months of use. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A glassware and glass microliter syringes should be routinely inspected for chips, acid etching, or deformity (e.g., bent needle). If the Class A glassware or syringe are suspect, the accuracy of the glassware must be assessed prior to use. Actions to correct or segregate ancillary equipment that does not meet required specifications are identified in the calibration and maintenance section of SOPs and maintenance logbooks for the specific equipment.

21.2. NIST-Traceable Weights and Thermometers

21.2.1. Reference standards of measurement must be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

21.2.2. For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), APLAC (Asia-Pacific Laboratory Accreditation Cooperation), or another accreditation organization that is a signatory to a MRA (Mutual Recognition Arrangement) of one or more of the following cooperations – ILAC (International Laboratory Accreditation Cooperation) or APLAC (Asia-Pacific Laboratory Accreditation Cooperation). A calibration certificate and scope of accreditation is kept on file at the laboratory. Refer to Section 21 for calibration of weights and thermometers.

21.2.3. An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

21.3. Reference Standards / Materials

21.3.1. Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by ISO Guide 34 and ISO/IEC Guide 17025, with an accompanying Certificate of Analysis that documents the following information:

21.3.1.1. Manufacturer

21.3.1.2. Analytes or parameters calibrated

21.3.1.3. Identification or lot number

21.3.1.4. Calibration method

21.3.1.5. Concentration with associated uncertainties

21.3.1.6. Purity

21.3.2. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. (Refer to Section 9 for additional information on purchasing). The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

21.3.3. All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor-certified different lot is acceptable for use as a second source. For unique situations, where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g., calibration checks, laboratory control samples).

21.3.4. All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to The Corporate Environmental Health & Safety Manual (CW-E-M-001) or laboratory SOPs. For safety requirements,

please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

21.3.5. Standards and reference materials must not be used after their expiration dates unless their reliability is verified by the laboratory and their use is approved by the Quality Assurance Manager. The laboratory must have documented contingency procedures for re-verifying expired standards. Some regulatory programs, such as Ohio VAP, prohibit the use of re-verified standards.

21.4. Documentation And Labeling Of Standards, Reagents, And Reference Materials

21.4.1. Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to companywide purchase. Refer to TestAmerica's Corporate SOP CA-Q-S-001, Solvent and Acid Lot Testing and Approval.

21.4.2. All manufacturer or vendor-supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained in each group. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection.

21.4.3. Commercial materials purchased for preparation of calibration solutions, spike solutions, etc., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96%, a correction must be made to concentrations applied to solutions prepared from the stock commercial material. Blended gas standard cylinders use a nominal concentration if the certified value is within +/-15%, otherwise the certified values are used for the canister gas concentrations.

21.4.4. All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS system, and are assigned a unique identification number. The following information is typically recorded in the electronic database within the LIMS:

21.4.4.1. Standard ID

21.4.4.2. Description of Standard

21.4.4.3. Department

21.4.4.4. Preparer's name

- 21.4.4.5. Final volume and number of vials prepared
- 21.4.4.6. Solvent type and lot number
- 21.4.4.7. Preparation date
- 21.4.4.8. Expiration date
- 21.4.4.9. Standard source type (stock or daughter)
- 21.4.4.10. Standard type (spike, surrogate, other)
- 21.4.4.11. Parent standard ID (if applicable)
- 21.4.4.12. Parent standard analyte concentration (if applicable)
- 21.4.4.13. Parent standard amount used (if applicable)
- 21.4.4.14. Component analytes
- 21.4.4.15. Final concentration of each analyte
- 21.4.4.16. Comment box (text field)
- 21.4.5. Records are maintained electronically in each group for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date, and preparer's name or initials. Preparation procedures are provided in the Method SOPs.
- 21.4.6. All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:
 - 21.4.6.1. Expiration date (include prep date for reagents)
 - 21.4.6.2. Standard ID (from LIMS)
 - 21.4.6.3. Special health/safety warnings, if applicable
- 21.4.7. Records must also be maintained of the date of receipt for commercially purchased items or date of preparation for laboratory prepared items. Special health/safety warnings must also be available to the analyst. This information is maintained in the analytical SOP.
- 21.4.8. In addition, the following information may be helpful:
 - 21.4.8.1. Date of receipt for commercially purchased items or date of preparation for laboratory prepared items
 - 21.4.8.2. Date opened (for multi-use containers, if applicable)

- 21.4.8.3. Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- 21.4.8.4. Recommended storage conditions
- 21.4.8.5. Concentration (if applicable)
- 21.4.8.6. Initials of analyst preparing standard or opening container
- 21.4.9. All containers of prepared reagents must include an expiration date, and an ID number to trace back to preparation.
- 21.4.10. Procedures for preparation of reagents can be found in the Method SOPs.
- 21.4.11. Standard ID numbers must be traceable through associated logbooks, worksheets, and preparation and batch records.
- 21.4.12. All reagents and standards must be stored in accordance to the following priority:
 - 21.4.12.1. With the manufacturer's recommendations
 - 21.4.12.2. With requirements in the specific analytical methods as specified in the laboratory SOP

22. SAMPLING

- 22.1. The laboratory provides sampling services. Sampling procedures are described in SOP NC-SC-006, Sample Procurement Protocol.
- 22.2. Sampling Containers
 - 22.2.1. The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Certificates of cleanliness provided by the supplier are maintained at the laboratory. Alternatively, the certificates are available from the vendor electronically and available to the laboratory online.
- 22.3. Preservatives
 - 22.3.1. Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:
 - 22.3.1.1. Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent

22.3.1.2. Methanol – Purge and Trap grade

22.3.1.3. Nitric Acid – Instra-Analyzed or equivalent

22.3.1.4. Sodium Bisulfate – ACS Grade or equivalent

22.3.1.5. Sodium Hydroxide – Instra-Analyzed or equivalent

22.3.1.6. Sulfuric Acid – Instra-Analyzed or equivalent

22.3.1.7. Sodium Thiosulfate – ACS Grade or equivalent

22.4. Definition Of Holding Time

22.4.1. The date and time of sampling documented on the Chain-of-Custody (COC) form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in “days” (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in “hours” (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. The first day of holding time ends 24 hours after sampling. Holding times for analysis include any necessary re-analysis. However, there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of holding time length.

22.5. Sampling Containers, Preservation Requirements, Holding Times

22.5.1. The preservation and holding time criteria specified in the following tables are derived from the source documents for the methods. If method-required holding times (refer to Tables 23-1 to 23-7 and in SOPs) or preservation requirements are not met, the reports must be qualified using a flag, footnote, or case narrative. As soon as possible, or “ASAP”, is an EPA designation for tests for which rapid analysis is advised; but for which neither EPA nor the laboratory have a basis for a holding time.

22.6. Sample Aliquots / Subsampling

22.6.1. Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample needs consideration when sub-sampling for sample preparation. It is the laboratory’s responsibility to take a representative sub-sample or aliquot of the sample provided for analysis. In that regard the following guidelines apply to analysts:

- 22.6.2. Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.
- 22.6.3. Guidelines on taking sample aliquots and sub-sampling are located in each analytical SOP.
- 22.6.4. Tables 23-1 to 23-7 detail holding times, preservation and container requirements, and sample volumes for NPDES methods. The sample volumes are intended to be a minimal amount to perform the method. The containers used may be of larger size.

Please note: The holding times are program specific and different programs may have different holding times for equivalent methods, e.g., there are differences in holding times for many organic analytes between RCRA and NPDES.

Table 22-1. Inorganic Sample Containers, Preservatives, and Holding Times

| Analytical Parameters | Matrix | Minimum Sample Size 1 | NPDES 2, 3, 7 | | RCRA (SW846) 3, 4 | |
|---|--------|-----------------------|----------------------------------|---|-------------------|--|
| | | | Method | Requirements | Method | Requirements |
| | | | | | | |
| Alkalinity, Bicarbonate, Carbonate | Water | 100 mL | 310.1 SM2320B | 250 mL plastic or glass. Cool to 4°C, 14 days | --- | N/A |
| | Solid | N/A | --- | N/A | --- | N/A |
| | Waste | N/A | --- | N/A | --- | N/A |
| Ammonia | Water | 100 mL | 350.2 SM4500NH3-C SM4500NH3-B | 500 mL plastic or glass. Cool to 4°C H ₂ SO ₄ to pH < 2, 28 days | --- | N/A |
| | Solid | N/A | --- | N/A | --- | N/A |
| | Waste | N/A | --- | N/A | --- | N/A |
| | | | | | | |
| Biochemical Oxygen Demand (BOD), Carbonaceous | Water | 1000 mL | 405.1 SM5210B | 1000 mL plastic or glass. Cool to 4°C, 48 hours | --- | N/A |
| | Solid | N/A | --- | N/A | --- | N/A |
| | Waste | N/A | --- | N/A | --- | N/A |
| Anions, Bromide, Chloride, Fluoride, Sulfate, | Water | 50 mL | 300.0A7 | 250 mL plastic or glass. No preservative required, 28 days | 9056A | Cool to 4°C. Analyze ASAP after collection |
| | Solid | N/A | --- | N/A | --- | N/A |
| | Waste | N/A | --- | N/A | --- | N/A |
| Anions, Nitrate, Nitrite, ortho-Phosphate | Water | 50 mL | 300.0A 7 | 250 mL plastic or glass. Cool to 4°C, 48 hours. | 9056A | Cool to 4°C. Analyze within 48 hours of collection |
| | Solid | N/A | --- | N/A | --- | N/A |
| | Waste | N/A | --- | N/A | --- | N/A |
| | | | | | | |
| Chemical Oxygen Demand (COD) | Water | 100 mL | 410.4 5220D | 250 mL glass or plastic. Cool to 4°C, H ₂ SO ₄ to pH < 2, 28 days | --- | N/A |
| | Solid | N/A | --- | N/A | --- | N/A |
| | Waste | N/A | --- | N/A | --- | N/A |

| Analytical Parameters | Matrix | Minimum Sample Size 1 | NPDES 2, 3, 7 | | RCRA (SW846) 3, 4 | |
|-----------------------|--------|-----------------------|--------------------|--|-------------------|--|
| | | | Method | Requirements | Method | Requirements |
| Chloride | Water | 50 mL | 325.2 SM 4500-Cl-E | 250 mL plastic or glass. No preservative required, 28 days | 9251 | Method 9251: 250ml plastic or glass, no preservative required, 28 days |
| | Solid | N/A | --- | N/A | --- | N/A |
| | Waste | N/A | --- | N/A | --- | N/A |
| Chlorine, Residual | Water | 100 mL | 330.5 SM 4500 Cl-G | 250 mL glass or plastic. Cool to 4°C, analyze immediately | --- | N/A |
| | Solid | N/A | --- | N/A | --- | N/A |
| | Waste | N/A | --- | N/A | --- | N/A |
| Chromium (Cr+6) | Water | 100 mL | 3500 Cr-B | Method 3500 Cr-D: 200 mL quartz, TFE, or polypropylene HNO3 to pH <2. Cool to 4°C. Analyze ASAP after collection | 7196A | 200 mL plastic or glass. Cool to 4°C, 24 hours |
| | Solid | 20 g | --- | N/A | 7196A 3060A | 250 mL plastic or glass, 30 days to digestion, 168 hours after digestion |
| | Waste | N/A | --- | N/A | --- | N/A |
| Conductivity | Water | 100 mL | 120.1 SM2510B | 200 mL glass or plastic. Cool to 4°C, 28 days | 9050A | 200 mL glass or plastic. Cool to 4°C, 28 days |
| | Solid | N/A | --- | N/A | --- | N/A |
| | Waste | N/A | --- | N/A | --- | N/A |

| Analytical Parameters | Matrix | Minimum Sample Size 1 | NPDES 2, 3, 7 | | RCRA (SW846) 3, 4 | |
|---------------------------|--------|-----------------------|--|---|-------------------|--|
| | | | Method | Requirements | Method | Requirements |
| Cyanide (Amenable) | Water | 250 mL | 335.1 SM4500CN-G | 1 liter plastic or glass, NaOH to pH >12 Cool to 4°C, 14 days unless sulfide is present. Then maximum holding time is 24 hours. | 9012A, B | 1 liter plastic or glass, NaOH to pH >12 Cool to 4°C, 14 days |
| | Solid | 50g | --- | N/A | 9012A, B | Not Specified |
| | Waste | 50g | --- | N/A | 9012A, B | Not Specified |
| Cyanide (Total) | Water | 1L | 335.2 335.3 335.4 (7) SM4500CN-E 335.2-CLP-M | 1 liter plastic or glass, NaOH to pH >12 Cool to 4°C, 14 days unless sulfide is present. Then maximum holding time is 24 hours. | 9012A, B | 1 liter plastic or glass, NaOH to pH >12 Cool to 4°C, 14 days. |
| | Solid | 50g | -- | N/A | 9012A, B | 8 or 16 oz glass Teflon-lined lids, Cool to 4°C, 14 days |
| | Waste | 50g | -- | N/A | 9012A, B | 8 or 16 oz glass Teflon-lined lids, Cool to 4°C |
| Flashpoint (Ignitability) | Liquid | 100 mL | --- | N/A | 1010, 1010A | No requirements, 250 mL amber glass. Cool to 4°C recommended |
| | Solid | 100 g | -- | N/A | --- | N/A |
| | Waste | 100 mL | -- | N/A | --- | N/A |
| Fluoride | Water | 300 mL | 340.2 SM 4500 F-C | 500 mL plastic. No preservation required, 28 days. | | |
| | Solid | N/A | --- | N/A | --- | N/A |
| | Waste | N/A | --- | N/A | --- | N/A |

| Analytical Parameters | Matrix | Minimum Sample Size 1 | NPDES 2, 3, 7 | | RCRA (SW846) 3, 4 | |
|---------------------------------------|--------|-----------------------|--------------------|--|-------------------|--------------|
| | | | Method | Requirements | Method | Requirements |
| Hardness (Total) | Water | 50 mL | 130.2 SM2340C | 250 mL glass or plastic, HNO ₃ to pH < 2, 6 months | --- | N/A |
| | Solid | N/A | --- | N/A | --- | N/A |
| | Waste | N/A | --- | N/A | --- | N/A |
| Iron (Ferrous) Ortho-phosphate | Water | 100 mL | 3500-Fe B | 1 liter glass or polyethylene containe. This test should be performed in the field. | - | N/A |
| | Solid | N/A | - | N/A | - | N/A |
| | Waste | N/A | - | N/A | - | N/A |
| | Water | 50 mL | 365.1 SM4500P-E | 100 mL plastic or glass. Filter on site. Cool to 4°C, 48 hours | - | N/A |
| | Solid | N/A | --- | N/A | --- | N/A |
| | Waste | N/A | --- | N/A | --- | N/A |

| Analytical Parameters | Matrix | Minimum Sample Size 1 | NPDES 2, 3, 7 | | RCRA (SW846) 3, 4 | |
|-----------------------|--------|-----------------------|--------------------|---|-------------------|--|
| | | | Method | Requirements | Method | Requirements |
| pH | Water | 50 mL | 150.1 SM4500H-B | 100 mL plastic or glass. Analyze immediately. This test should be performed in the field. | 9040B, C | 100 mL plastic or glass. Analyze immediately. This test should be performed in the field.(8) |
| | Solid | N/A | --- | N/A | 9045C, C | 4 oz glass or plastic. Cool to 4°C. Analyze as soon as possible.8 |
| | Waste | N/A | --- | N/A | 9045C, C | 4 oz glass or plastic, Cool to 4°C. Analyze as soon as possible.8 |
| Phenolics | Water | 100 mL | 420.1 | 500 mL glass, Cool to 4°C, H ₂ SO ₄ to pH < 2, 28 days | 9065 | 1 liter glass recommended, Cool to 4°C, H ₂ SO ₄ to pH < 4, 28 days |
| | Solid | N/A | --- | N/A | --- | N/A |

| Analytical Parameters | Matrix | Minimum Sample Size 1 | NPDES 2, 3, 7 | | RCRA (SW846) 3, 4 | |
|-----------------------|--------|-----------------------|---------------|--------------|-------------------|---------------|
| | | | Method | Requirements | Method | Requirements |
| | Waste | N/A | --- | N/A | 9065 | Not Specified |

| Analytical Parameters | Matrix | Minimum Sample Size 1 | NPDES 2, 3, 7 | | RCRA (SW846) 3, 4 | |
|-----------------------|--------|-----------------------|-----------------|---|-------------------|---|
| | | | Method | Requirements | Method | Requirements |
| Phosphorus (Total) | Water | 100 mL | 365.1 SM4500P-E | 100 mL plastic or glass, H2SO4 to pH < 2, 28 days | --- | N/A |
| | Solid | N/A | --- | N/A | --- | N/A |
| | Waste | N/A | --- | N/A | --- | N/A |
| Settleable Solids | Water | 1000 mL | 160.5 SM2540F | 1000 mL plastic or glass. Cool to 4°C, 48 hours | --- | N/A |
| | Solid | N/A | --- | N/A | --- | N/A |
| | Waste | N/A | --- | N/A | --- | N/A |
| Sulfate (SO4) | Water | 50 mL | 375.4 | 100 mL plastic or glass. Cool to 4°C, 28 days | 9038 | 200 mL plastic or glass, Cool to 4°C, 28 days |
| | Solid | N/A | --- | N/A | --- | N/A |
| | Waste | 100 mL | --- | N/A | 9038 | 200 mL plastic or glass. Cool to 4°C, 28 days |

| Analytical Parameters | Matrix | Minimum Sample Size 1 | NPDES 2, 3, 7 | | RCRA (SW846) 3, 4 | |
|-------------------------------------|--------|-----------------------|--------------------------|---|-------------------------|---|
| | | | Method | Requirements | Method | Requirements |
| Sulfide | Water | 250 mL | 376.1 SM 4500 S2-F | 500 mL plastic or glass. Cool to 4°C, Add 2 mL zinc acetate plus NaOH to pH > 9, 7 days | 9030A 9030B/ 9034 | 500 mL plastic, No headspace. Cool to 4°C. Add 4 drops of 2N zinc acetate per 100 mL of sample, adjust the pH to > 9 with 6 N NaOH solution, 7 days |
| | Solid | 50 g | --- | N/A | 9030A 9030B/ 9034 | Cool to 4°C. Fill surface of solid with 2N Zinc acetate until moistened. Store headspace-free |
| | Waste | 50 g | --- | N/A | 9030A 9030B/ 9034 | Cool to 4°C. Fill surface of solid with 2N Zinc acetate until moistened. Store headspace-free |
| Total Dissolved Solids (Filterable) | Water | 100 mL | 160.1 SM2540C | 250 mL plastic or glass. Cool to 4°C, 7 days | --- | N/A |
| | Solid | N/A | --- | N/A | --- | N/A |
| | Waste | N/A | --- | N/A | --- | N/A |

| Analytical Parameters | Matrix | Minimum Sample Size 1 | NPDES 2, 3, 7 | | RCRA (SW846) 3, 4 | |
|---|--------|-----------------------|------------------------|--|-------------------|---|
| | | | Method | Requirements | Method | Requirements |
| Total Kjeldahl Nitrogen (TKN) | Water | 100 mL | 351.3 SM 4500-NH3-C | 500 mL plastic or glass. Cool to 4°C, H2SO4 to pH < 2, 28 days | --- | N/A |
| | Solid | N/A | --- | N/A | --- | N/A |
| | Waste | N/A | --- | N/A | --- | N/A |
| Total Organic Carbon (TOC) | Water | 100 mL | 415.1 SM5310C | 100 mL plastic or glass. Cool to 4°C, H2SO4 to pH < 2, 28 days | 9060, 9060A | 100 mL glass or 40 mL VOA vials, Cool to 4°C, H2SO4 or HCl to pH < 2, 28 days |
| | Solid | N/A | --- | N/A | Walkley-Black | Not Specified Cool to 4°C, 28 days |
| | Waste | N/A | --- | N/A | Walkley-Black | Not Specified Cool to 4°C, 28 days |
| Extractable Organic Halides (EOX) | Solid | 100 mL | | | 9023 (EOX) | 500 mL amber glass, Teflon®-lined lid. Cool to 4°C no headspace, 28 days |
| Total Solids | Water | 100 mL | 160.3 | 250 mL plastic or glass. Cool to 4°C, 7 days | --- | N/A |
| | Solid | N/A | --- | N/A | --- | N/A |
| | Waste | N/A | --- | N/A | --- | N/A |
| Total Suspended Solids (Non-filterable) | Water | 100 mL | 160.2 | 250 mL plastic or glass. Cool, 4°C, 7 days | --- | N/A |
| | Solid | N/A | --- | N/A | --- | N/A |
| | Waste | N/A | --- | N/A | --- | N/A |

| Analytical Parameters | Matrix | Minimum Sample Size 1 | NPDES 2, 3, 7 | | RCRA (SW846) 3, 4 | |
|------------------------|--------|-----------------------|----------------|--|---------------------------------|---|
| | | | Method | Requirements | Method | Requirements |
| Turbidity | Water | 50 mL | 180.1 | 250 mL plastic or glass. Cool, 4°C, 48 hours | --- | N/A |
| | Solid | N/A | --- | N/A | --- | N/A |
| | Waste | N/A | --- | N/A | --- | N/A |
| Metals (excludes Hg) | Water | 100 mL | 200 series | 1 liter glass or polyethylene container, HNO ₃ to pH < 2, 6 months | 6010B 6010C 6020 6020A | 1 liter glass or polyethylene container, HNO ₃ to pH < 2, 6 months |
| | Solid | 200 g | 200 series | 2, 8, or 16 oz glass or polyethylene container storage at 4 °C | 6010B 6010C 6020 6020A | 8 or 16 oz glass or polyethylene container, storage at 4°C, 6 months |
| | Waste | 200 g | 200 series | N/A | 6010B 6010C 6020 6020A | 8 or 16 oz glass or polyethylene container, storage at 4°C, 6 months |
| Mercury (CVAA) (CVAFS) | Water | 100 mL | 245.1 1631E | 250 mL glass or polyethylene container, HNO ₃ to pH < 2, 28 days | 7470A | 1 liter glass or polyethylene container, HNO ₃ to pH < 2, 28 days |
| | Solid | 200 g | -- | 2, 8, or 16 oz glass or polyethylene container. Cool to 4°C, 28 days. Not applicable for Method 1631E. | 7471A 7471B | 8 or 16 oz glass or polyethylene container. Cool to 4°C, 28 days (CORP-MT-0007) |
| | Waste | 200 g | -- | N/A | 7471A 7471B | 8 or 16 oz glass or polyethylene container. Cool, 4°C, 28 days (CORP-MT-0007) |

Footnotes

- 1 Minimum sample size indicates sample amount needed for a single analysis. Matrix spikes or duplicates will require an additional sample amount of at least this amount for each additional QC sample aliquot required.
- 2 National Pollutant Discharge Elimination System - MCAWW, March 1983.
- 3 Holding times are calculated from date of collection. Holding Times are determined based on date of collection to preparation/analysis.
- 4 Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA, (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).
- 5 Solid matrix type includes soil, sediment, sludge and other solid materials not classified as waste.

- 6 *Samples to be analyzed for cyanide should be field-tested for residual chlorine. If residual chlorine is detected, ascorbic acid should be added.*
- 7 *Method not listed in 40 CFR Part 136.*
- 8 *If not done in the field (ASAP) per the method and requested by client, analyze in lab within 48 hours.*

Table 22-2. Organic Sample Containers, Preservatives, and Holding Times

| Analytical Parameters | Matrix | Minimum Sample Size 1 | NPDES 2, 3 | | RCRA (SW846) 3, 4 | |
|-----------------------|--------|-----------------------|------------|--------------|-------------------|--|
| | | | Method | Requirements | Method 6 | Requirements |
| Herbicides | Water | 1L | | | 8151A | 1 liter amber glass with Teflon®-lined lid. If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool to 4°C. Extraction, 7 days. Analysis, 40 days of the start of extraction. |
| | Solid | 50 g | | | 8151A | 4 or 8 oz glass widemouth with Teflon®-lined lid. Cool to 4 °C. Extraction, 14 days. Analysis, 40 days of the start of the extraction. |
| | Waste | 50 g | | | 8151A | 4 or 8 oz glass widemouth with Teflon®-lined lid. Cool to 4 °C. Extraction, 14 days. Analysis, 40 days of the start of the extraction. |

| Analytical Parameters | Matrix | Minimum Sample Size 1 | NPDES 2, 3 | | RCRA (SW846) 3, 4 | |
|-----------------------|--------|-----------------------|------------|---|---------------------------------|---|
| | | | Method | Requirements | Method 6 | Requirements |
| Pesticides/PCBs | Water | 1L | 608 | 1 liter amber glass with Teflon®-lined lid, Adjust pH to 5-9 if extraction not to be done within 72 hours of sampling. Add sodium thiosulfate if residual chlorine present and aldrin is being determined. Cool, 4°C. Extraction, 1 year. Analysis, 40 days after extraction. | 8081A 8081B 8082 8082A | 1 liter amber glass with Teflon®-lined lid, If residual chlorine present, add 3 mL 10% sodium thiosulfate per gallon. Cool, 4°C. Extraction, 7 days (1 year for 8082A). Analysis, 40 days of the start of the extraction. |
| | Solid | 50 g | --- | N/A | 8081A 8081B 8082 8082A | 4 or 8 oz glass wide mouth with Teflon®-lined lid. Cool, 4°C. Extraction, 14 days (1 year for 8082A). Analysis, 40 days of the start of the extraction. |
| | Waste | 50 g | --- | N/A | 8081A 8081B 8082 8082A | 4 or 8 oz glass wide mouth with Teflon®-lined lid. Cool, 4°C. Extraction, 14 days (1 year for 8082A). Analysis, 40 days of the start of the extraction. |

| Analytical Parameters | Matrix | Minimum Sample Size 1 | NPDES 2, 3 | | RCRA (SW846) 3, 4 | |
|-----------------------|--------|-----------------------|------------|---|-------------------|--|
| | | | Method | Requirements | Method 6 | Requirements |
| Oil and Grease | Water | 1 L | 1664A(7) | 1 liter glass, Cool, 4°C HCl or H2SO4 to pH <2 28 days | | |
| | Solid | 30 g | 1664A(7) | 8 or 16 oz. Wide mouth glass jar, Cool, 4°C, 28 days | | |
| | Waste | --- | --- | N/A | | |
| Semivolatiles | Water | 1L | 625 | 1 liter amber glass with Teflon®-lined lid. Cool, 4°C. Extraction, 7 days. Analysis, 40 days. | 8270C 8270D | 1 liter amber glass with Teflon®-lined lid, If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool, 4°C. Extraction, 7 days. Analysis, within 40 days of extraction. |
| | Solid | 50 g | --- | N/A | 8270C 8270D | 8 or 16 oz glass wide mouth with Teflon-lined lid. Cool, 4°C. Extraction, 14 days. Analysis, within 40 days of extraction. |
| | Waste | 50 g | --- | N/A | 8270C 8270D | 8 or 16 oz glass wide mouth with Teflon®-lined lid. Cool, 4°C. Extraction, 14 days. Analysis, within 40 days of extraction. |

| Analytical Parameters | Matrix | Minimum Sample Size 1 | NPDES 2, 3 | | RCRA (SW846) 3, 4 | |
|-----------------------|--------|-----------------------|------------|---|-------------------|--|
| | | | Method | Requirements | Method 6 | Requirements |
| Volatile Organics | Water | 40 mL | 624 | 40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C. Add sodium thiosulfate if residual chlorine, 7 days with pH > 2, 14 days with pH ≤ 28. | 8260B 8260C | 40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C. Add sodium thiosulfate if residual chlorine, 1:1 HCl to pH ≤ 2, 14 days with pH ≤ 29. |
| | Solid5 | 5 g or 25 g | -- | N/A | 8260B 8260C | 4 or 8 oz. glass with Teflon®-lined lid. Cool to 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hrs. of sampling. Maximum holding time for EnCore™ sampler is 48 hrs. (before the sample is added to methanol or sodium bisulfate). Cool to 4°C(12) |
| | Waste | 5 g or 25 g | -- | N/A | 8260B 8260C | 4 or 8 oz. glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hrs of sampling. Maximum holding time for Encore™ sampler is 48 hrs. (before sample is added to methanol or sodium bisulfate). Cool to 4°C12 |

Footnotes

- 1 *Minimum sample size indicates sample amount needed for a single analysis. Matrix spikes or duplicates will require an additional sample amount of at least this amount for each additional QC sample aliquot required.*
- 2 *National Pollutant Discharge Elimination System - 40 CFR Part 136, Appendix A.*
- 3 *Holding times are calculated from the date of collection.*
- 4 *Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).*
- 5 *Solid matrix type includes soil, sediment, sludge or other solids not classified as waste.*
- 6 *Only one determination method is listed when separate methods are required for preparation and analysis.*
- 7 *Method 1664 was promulgated by the EPA with an effective date of June 14, 1999.*
- 8 *For acrolein and acrylonitrile the pH should be adjusted to 4-5. This pH adjustment is not required if acrolein is not measured. Samples requiring analysis of acrolein that received no pH adjustment must be analyzed within three days of sampling.*
- 9 *For acrolein and acrylonitrile the pH should be adjusted to 4-5.*
- 10 *Method not listed in 40 CFR Part 136.*
- 11 *Should only be used in the presence of residual chlorine.*
- 12 *Depending on regulatory programs, EnCore[®] samplers may be preserved for up to 14 days from sampling by freezing at -5 to -12°C until analysis. Alternatively the EnCore[®] sample may be transferred to a 40-ml VOA vial and preserved by freezing at -5 to -12°C until analysis. Some regulatory agencies may require 4 or 8 oz glass with Teflon[®]-lined lid, Cool 4°C, 14 days. This technique is not recommended, but will be supported where required. (Preservation and holding times are subject to client specifications.)*

Table 22-3. Sample Containers, Preservatives, and Holding Times for TCLP1 and SPLP2

| Analytical Parameters | Matrix | Minimum Sample Size | TCLP Method 1311 and SPLP Method 1312 Requirements | |
|-------------------------|--------------------|---------------------|--|---|
| | | | From Field Collection to TCLP/SPLP Extraction | From TCLP/SPLP Extraction to Analysis |
| Mercury | Liquid Solid Waste | 1L | 1L glass, Cool, 4°C, 28 days | Glass or polyethylene 28 days |
| Metals (except mercury) | Liquid Solid Waste | 1L | 1L glass, Cool, 4°C, 180 days | Glass or polyethylene 180 days |
| Semivolatiles | Liquid Solid Waste | 1L | 1L glass, Cool 4°C, 14 days | 1L glass Extraction of leachate within 7 days of TCLP extraction, Analyze extract within 40 days |
| Volatiles | Liquid Solid Waste | 6 oz | 4 oz glass, Cool 4°C, 14 days | 40 mL glass, 14 days |

Footnotes

TCLP = Toxicity Characteristic Leaching Procedure
SPLP = Synthetic Precipitation Leaching Procedure

23. HANDLING OF SAMPLES

23.1. Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.2. Chain of Custody (COC)

23.2.1. The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the Sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 23-1.

23.3. Field Documentation

23.3.1. The information the sampler needs to provide at the time of sampling on the container label is:

23.3.1.1. Sample identification

23.3.1.2. Date and time

23.3.1.3. Preservative

23.3.2. During the sampling process, the COC form is completed and must be legible (see Figure 23-1). This form includes information such as:

23.3.2.1. Client name, address, phone number and fax number (if available)

23.3.2.2. Project name and/or number

23.3.2.3. The sample identification

23.3.2.4. Date, time, and location of sampling

23.3.2.5. Sample collectors name

23.3.2.6. The matrix description

23.3.2.7. The container description

23.3.2.8. The total number of each type of container

23.3.2.9. Preservatives used

23.3.2.10. Analysis requested

23.3.2.11. Requested turnaround time (TAT)

23.3.2.12. Any special instructions

23.3.2.13. Purchase Order number or billing information (e.g. quote number) if available

23.3.2.14. The date and time that each person received or relinquished the sample(s), including their signed name.

23.3.3. When the sampling personnel deliver the samples directly to TestAmerica personnel, the samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's Field technician until the samples are delivered to the laboratory personnel. The sample collector must assure each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the Sample Control personnel at the laboratory or to a TestAmerica courier. When sampling personnel deliver the samples through a common carrier (FedEx, UPS), the COC relinquished date/time is completed by the Field personnel; and samples are released to the carrier. Samples are only considered to be received by lab when personnel at the fixed laboratory facility have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The COC is stored with project information and the report.

23.4. Legal / Evidentiary Chain-of-Custody

23.4.1. The lab does not accept samples that require legal Chain-of-Custody.

23.5. Sample Receipt

23.5.1. Samples are received at the laboratory by designated Sample Receiving personnel, and a unique laboratory project identification number is assigned. Each sample container must be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking, and storage procedures are summarized in the following sections. SOP NC-SC-005, Sample Receiving and Sample Control, describes the laboratory's sample receipt procedure.

23.5.2. Per State and/or Federal Regulation, the client is responsible to ensure that samples are shipped in accordance with DOT/IATA requirements, and that radioactive materials may only be delivered to licensed facilities. Any samples containing (or suspected to contain) Source, Byproduct, or special Nuclear Material, as defined by 20 CFR should be delivered directly to facilities licensed to handle such radioactive material. Natural material or ores containing naturally occurring radionuclides may be delivered to any TestAmerica facility or courier as long as the activity concentration of the material does not exceed 270 pCi/g alpha or 2700 pCi/g beta (49CFRPart173).

23.6. Laboratory Receipt

23.6.1. Samples must be received and logged in at TestAmerica by a designated sample custodian or other properly trained associate. Upon sample receipt, the sample custodian shall, as appropriate:

23.6.1.1. Wear appropriate personal protective equipment. At a minimum, this consists of cut-resistant gloves, a lab coat, and safety glasses

23.6.1.2. Examine the shipping containers to verify that the custody tape is intact

23.6.1.3. Examine all sample containers for damage

23.6.1.4. Open shipping containers in adequately ventilated areas to assure worker safety

23.6.1.5. Determine if the temperature required by the requested testing program has been maintained during shipment. Document the shipping container temperature on the Cooler Receipt Form

23.6.1.6. Compare samples received against those listed on the COC

23.6.1.7. Verify that sample holding times have not been exceeded

23.6.1.8. Examine all shipping records for accuracy and completeness

23.6.1.9. Determine sample pH (if required for the scheduled analysis) (except VOA and TOX samples) and record on the Cooler Receipt Form (CRF)

23.6.1.10. Sign and date the COC immediately (only after shipment is accepted) and attach the waybill

23.6.1.11. Note any problems associated with the coolers and samples on the cooler receipt form and notify the PM who in turn notifies the client

23.6.1.12. Attach durable (water-resistant) laboratory sample container labels with unique laboratory identification number and test

23.6.1.13. Place the samples in proper laboratory storage.

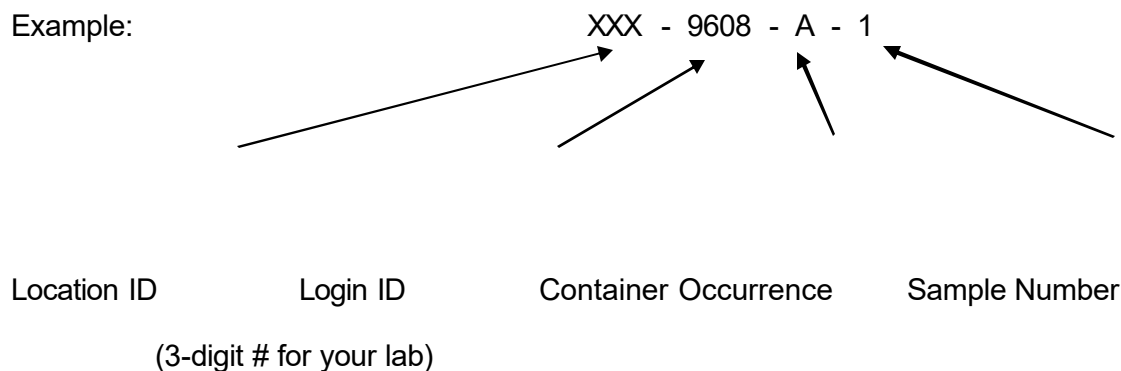
23.6.2. A Cooler Receipt Form (CRF) or an equivalent form/system is generated by sample control during the sample log-in process to document anomalies identified upon the receipt of samples in the laboratory. These anomalies are outside of laboratory control and do not require corrective actions to be taken within the laboratory. The affected client must be notified by the PM or designee of all issues generated for their samples. The PM is responsible for resolving with the client how to proceed with the samples and documenting the decision to proceed with the analysis of compromised samples. Issues must be resolved prior to sample preparation and analysis. The completed CRF must be stored in the project file. An example CRF is shown in Figure 24-4. The report narrative must include an explanation of sample receiving related anomalies.

23.7. Unique Sample Identification

23.7.1. All samples that are processed through the laboratory receive a unique sample identification to ensure that there can be no confusion regarding the identity of such samples at any time. This system includes identification for all samples, subsamples and subsequent extracts and/or digestates.

23.7.2. The laboratory assigns a unique identification (e.g., Sample ID) code to each sample container received at the laboratory. This Primary ID is made up of the following information (consisting of 4 components):

Example:



23.7.3. The above example states that TestAmerica <location> Laboratory (Location XXX). Login ID is 9608 (unique to a particular client/job occurrence). The container code indicates it is the first container ("A") of Sample #1.

23.7.4. If the primary container goes through a prep step that creates a “new” container, then the new container is considered secondary and gets another ID. An example of this being a client sample in a 1-Liter amber bottle is sent through a Liquid/Liquid Extraction and an extraction vial is created from this step. The vial would be a SECONDARY container. The secondary ID has 5 components.

Example: XXX - 9608 - A - 1 - A ← Secondary Container Occurrence

23.7.5. Example: 220-9608-A-1-A, would indicate the PRIMARY container listed above that went through a step that created the 1st occurrence of a Secondary container.

23.7.6. With this system, a client sample can literally be tracked throughout the laboratory in every step from receipt to disposal.

23.8. Sample Acceptance Policy

23.8.1. The laboratory has a written sample acceptance policy outlined in SOP NC-SC-005, Sample Receiving and Sample Control, that clearly outlines the circumstances under which samples must be accepted or rejected. These include:

23.8.1.1. A COC filled out completely

23.8.1.2. Samples must be properly labeled

23.8.1.3. Proper sample containers with adequate volume for the analysis and necessary QC

23.8.1.4. Samples must be preserved according to the requirements of the requested analytical method

23.8.1.5. Sample holding times must be adhered to

23.8.1.6. All samples submitted for water/solid Volatile Organic analyses must have a Trip Blank submitted at the same time

23.8.2. The Project Manager must be notified if any sample is received in damaged condition.

23.8.3. Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined.

23.8.4. Once sample acceptance is verified, the samples are logged into LIMS according to SOP NC-SC-005.

23.9. Sample Storage

23.9.1. In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators, freezers, or protected locations suitable for the sample matrix. Metals samples may be unrefrigerated. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards, or materials that may create contamination.

23.9.2. To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed every week.

23.9.3. Analysts and technicians retrieve the sample container allocated to their analysis from the designated refrigerator and place them on carts, analyze the sample, and return the remaining sample or empty container to the refrigerator from which it originally came. All unused portions of samples, including empty sample containers, are returned to the secure sample control area. All samples are kept in the refrigerators for a minimum of 30 days after report generation, which meets or exceeds most sample holding times. After this time period, the samples are removed from the refrigerator shelves and prepared for disposal. Special arrangements may be made to store samples for longer periods of time.

23.9.4. Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

23.10. Hazardous Samples And Foreign Soils

23.10.1. All samples per SOP are treated as hazardous. If any extra or known hazards are present in the sample, the sample is flagged and precautions / instructions are put in the comments. Hazardous samples are segregated out, and go into the waste stream profile for the nature of the hazard. All soils--foreign and domestic--go to a USDA approved incinerator. See SOP NC-SC-019 Procedure of Acceptance and Handling of USDA Regulated Domestic and Foreign Soil for further information.

23.11. Sample Shipping

23.11.1. In the event the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet

maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses. The Chain-of-Custody form is signed by the Sample Control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper Chain-of-Custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they are not inadvertently omitting a key part of regulatory compliance testing.

23.12. Sample Disposal

- 23.12.1. Samples should be retained for a minimum of 30 days after the project report is sent; however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist--the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP NC-SC-005, Sample Receiving and Sample Control). All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than two months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work. Waste disposal complies with all federal and state laws and regulations.
- 23.12.2. If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), and names of individuals who conducted the arrangements and physically completed the task. Sample labels are destroyed through the disposal method, e.g., samples are incinerated. A Waste Manifest is completed.

Figure 23-1. Example: Chain of Custody (COC)

[illegible]

Figure 23-2.

Example: Custody Seal

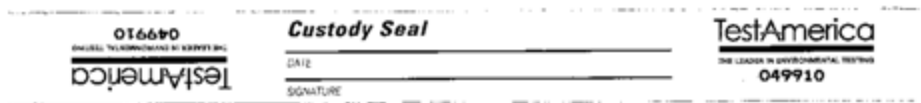


Figure 23-3. Example: Internal Chain of Custody (COC)

TestAmerica Laboratories, Inc.

Sample Control Record

Client:

Lot Number:

Case Number/SDG:

Storage Location:

| Laboratory Sample ID | Transferred By | Date | Entered | Removed | Reason |
|----------------------|----------------|------|---------|---------|--------|
| | | | | | |

Figure 23-4. Example: Cooler Receipt Form

| | | | | | |
|---|--------------------------|--------------------------------|----------|---------------------------------|--|
| TestAmerica North Canton Sample Receipt Form/Narrative | | | | Login # : _____ | |
| Client _____ | | Site Name _____ | | By: _____ | |
| Cooler Received on _____ | | Opened on _____ | | (Signature) _____ | |
| FedEx: 1st Grd Exp _____ | | UPS FAS Stetson _____ | | Client Drop Off _____ | |
| TestAmerica Courier _____ | | TestAmerica _____ | | Other _____ | |
| TestAmerica Cooler # _____ | | Foam Box _____ | | Client Cooler _____ | |
| Box _____ | | Other _____ | | _____ | |
| Packing material used: Bubble Wrap _____ | | Foam _____ | | Plastic Bag _____ | |
| None _____ | | Other _____ | | _____ | |
| COOLANT: Wet Ice _____ | | Blue Ice _____ | | Dry Ice _____ | |
| Water _____ | | None _____ | | _____ | |
| 1. Cooler temperature upon receipt | | | | | |
| IR GUN# 1 (CF -2°C) | | Observed Sample Temp. _____ °C | | Corrected Sample Temp. _____ °C | |
| IR GUN# 4G (CF -1°C) | | Observed Sample Temp. _____ °C | | Corrected Sample Temp. _____ °C | |
| IR GUN# 5G (CF -1°C) | | Observed Sample Temp. _____ °C | | Corrected Sample Temp. _____ °C | |
| IR GUN# 6Y (CF -2°C) | | Observed Sample Temp. _____ °C | | Corrected Sample Temp. _____ °C | |
| 2. Were custody seals on the outside of the cooler(s)? If Yes Yes No | | | | | |
| -Were custody seals on the outside of the cooler(s) signed & dated? Yes No NA | | | | | |
| -Were custody seals on the bottle(s)? Yes No | | | | | |
| 3. Shippers' packing slip attached to the cooler(s)? Yes No | | | | | |
| 4. Did custody papers accompany the sample(s)? Yes No | | | | | |
| 5. Were the custody papers relinquished & signed in the appropriate place? Yes No | | | | | |
| 6. Did all bottles arrive in good condition (Unbroken)? Yes No | | | | | |
| 7. Could all bottle labels be reconciled with the COC? Yes No | | | | | |
| 8. Were correct bottle(s) used for the test(s) indicated? Yes No | | | | | |
| 9. Sufficient quantity received to perform indicated analyses? Yes No | | | | | |
| 10. Were sample(s) at the correct pH upon receipt? Yes No NA | | | | | |
| 11. Were VOAs on the COC? Yes No | | | | | |
| 12. Were air bubbles >6 mm in any VOA vials? Yes No NA | | | | | |
| 13. Was a trip blank present in the cooler(s)? Yes No | | | | | |
| Contacted PM _____ Date _____ by _____ via Verbal Voice Mail Other | | | | | |
| Concerning _____ | | | | | |
| 14. CHAIN OF CUSTODY & SAMPLE DISCREPANCIES | | | | | |
| 15. SAMPLE CONDITION | | | | | |
| Sample(s) _____ were received after the recommended holding time had expired. | | | | | |
| Sample(s) _____ were received in a broken container. | | | | | |
| Sample(s) _____ were received with bubble >6 mm in diameter. (Notify PM) | | | | | |
| 16. SAMPLE PRESERVATION | | | | | |
| Sample(s) _____ were further preserved in Sample Receiving to meet recommended pH level(s). Nitric Acid Lot# 110410-HNO ₃ ; Sulfuric Acid Lot# 041911-H ₂ SO ₄ ; Sodium Hydroxide Lot# 121809 -NaOH; Hydrochloric Acid Lot# 041911-HCl; Sodium Hydroxide and Zinc Acetate Lot# 100108-(CH ₃ COO) ₂ ZN/NaOH. What time was preservative added to sample(s)? _____ | | | | | |
| Client ID | pH | Date | Initials | | |
| | | | | | |
| | | | | | |
| | | | | | |
| Cooler # | Observed Sample Temp. °C | Corrected Sample Temp. °C | IR # | Coolant | |
| | | | | | |
| | | | | | |
| | | | | | |

24. ASSURING THE QUALITY OF TEST RESULTS

24.1. In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g., Method Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. Quality control samples are to be treated in the exact same manner as the associated field samples being tested. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

24.2. Controls

24.2.1. Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

24.3. Negative Controls

Table 24-1. Example – Negative Controls

| Control Type | Details |
|--------------------|---|
| Method Blanks (MB) | are used to assess preparation and analysis for possible contamination during the preparation and processing steps. |
| | The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples. |
| | The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. |
| | The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.). |
| | Re-analyze or quality-associated sample results when the concentration of a targeted analyte in the method blank is at, or above, the reporting limit as established by the method or by regulation, AND is greater than 1/20 of the amount measured in the sample. |

Table 24-1. Example – Negative Controls

| Control Type | Details |
|--------------------|--|
| Calibration Blanks | are prepared and analyzed along with calibration standards where applicable or injected at specified frequencies throughout an analytical sequence. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve. These blanks may be termed Initial Calibration Blanks (ICB) or Continuing Calibration Blanks (CCB), |
| Instrument Blanks | are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content. |
| Trip Blanks 1 | are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses (or as specified in the client's project plan). Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples. |
| Field Blanks 1 | are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER) |
| Equipment Blanks 1 | are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (TNI) |
| Holding Blanks | also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory |

24.3.1. When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

24.3.2. Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

24.4. Positive Controls

24.4.1. Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon:

24.4.2. Method Performance [Laboratory Control Sample (LCS) or Blank Spike (BS)], which entails both the preparation and measurement steps

24.4.3. Matrix Effects (Matrix Spike (MS) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed.

24.4.4. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch.

Note that frequency of control samples vary with specific regulatory, methodology, and project- specific criteria. Complete details on method control samples are as listed in each analytical SOP.

24.5. Method Performance Control - Laboratory Control Sample (LCS)

24.5.1. The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

24.5.2. The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard is reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

24.5.3. Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g., solid matrix LCS for metals, TDS, etc.).

24.5.4. The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally one for each batch of sample--not to exceed 20 environmental samples.

24.5.5. If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable, e.g., no spike of pH. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608),

the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

24.5.6. For methods that have 1-10 target analytes, spike all components.

24.5.7. For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.

24.5.8. For methods with more than 20 target analytes, spike at least 16 components.

24.5.9. Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.

24.5.10. Exception: Due to analyte incompatibility between the various PCB Aroclors, Aroclors 1016 and 1260 are used for spiking as they cover the range of all of the Aroclors. Specific Aroclors may be used by request on a project-specific basis.

24.6. Sample Matrix Controls

Table 24-2 Sample Matrix Control

| Control Type | Details | |
|--------------------|-------------------|---|
| Matrix Spikes (MS) | Use | To assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used; |
| | Typical Frequency | At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects. If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details |
| | Description | Essentially, a sample fortified with a known amount of the test analyte(s). |
| Surrogate | Use | Measures method performance to sample matrix (organics only). |
| | Typical Frequency | Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the control limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery. |
| | Description | Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples. |

Table 24-2 Sample Matrix Control

| Control Type | Details | |
|-------------------------|--------------------------------|---|
| Duplicates ² | Use | For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure. |
| | Typical Frequency ¹ | Duplicate samples are usually analyzed with methods that do not require matrix spike analysis. |
| | Description | Performed by analyzing two aliquots of the same field sample independently or an additional LCS. |
| Internal Standards | Use | Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements. |
| | Typical Frequency ¹ | All organic and ICP methods as required by the analytical method. |
| | Description | Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance. |

¹ See the specific analytical SOP for type and frequency of sample matrix control samples.

² LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

24.7. Control Limits

24.7.1. As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project-specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes, and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

Note: For Ohio VAP the laboratory must implement Corrective Action procedures to resolve the deviation and limit qualification of the final results. The laboratory is not permitted to deviate from its VAP approved SOP if it intends to attest under affidavit that the "results" are VAP certified. When all corrective actions listed in the SOP have been exhausted, it may be necessary to use technical judgment in which case the decision process and rationale will be presented in the final report

and/or affidavit and the data will be noted as 'not VAP certified' on the affidavit.

- 24.7.2. Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.
- 24.7.3. Laboratory-generated Percent Recovery acceptance (control) limits are generally established by taking +3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).
- 24.7.4. Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV), (unless the analytical method specifies a tighter limit).
- 24.7.5. In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.
- 24.7.6. The lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for Benzidine will be 5%, and the analyte must be detectable and identifiable.
- 24.7.7. The maximum acceptable recovery limit will be 200%.
- 24.7.8. The maximum acceptable RPD limit will be 30% for organic methods and 20% for inorganic methods. The minimum RPD limit is 10%.
- 24.7.9. If either the high or low end of the control limit changes by < 10% from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no affect on laboratory ability to meet the existing limits.
- 24.7.10. The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits. Refer to NC-QA-018, Statistical Evaluation of Data and Development of Control Charts, for details.

24.7.11. An LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the control limits may be determined as out of control and should be re-analyzed if possible. If re-analysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal Corrective Action process (see Section 12) is also initiated if an LCS exceeds the control limits. Sample results may be qualified and reported without re-analysis if:

24.7.12. The analyte results are below the reporting limit and the LCS is above the upper control limit.

24.7.13. If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

Note: For Ohio VAP the laboratory must implement Corrective Action procedures to resolve the deviation and limit qualification of the final results. The laboratory is not permitted to deviate from its VAP approved SOP if it intends to attest under affidavit that the "results" are VAP certified. When all corrective actions listed in the SOP have been exhausted, it may be necessary to use technical judgment in which case the decision process and rationale will be presented in the final report and/or affidavit and the data will be noted as 'not VAP certified' on the affidavit.

24.7.14. Or, Department Of Defense (DOD) work, there are an allowable number of Marginal Exceedances (ME):

| | |
|------------------|-------------------------------------|
| <11 analytes | 0 marginal exceedances are allowed. |
| 11 – 30 Analytes | 1 marginal exceedance is allowed |
| 31-50 Analytes | 2 marginal exceedances are allowed |
| 51-70 Analytes | 3 marginal exceedances are allowed |
| 71-90 Analytes | 4 marginal exceedances are allowed |
| > 90 Analytes | 5 marginal exceedances are allowed |

24.7.15. Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit ().

Note: Use of Marginal Exceedances is not permitted for Ohio VAP.

- 24.7.16. Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken. The laboratory has a system to monitor marginal exceedances to ensure that they are random.
- 24.7.17. Though marginal exceedances may be allowed, the data must still be qualified to indicate it is outside of the normal limits.
- 24.7.18. If the MS/MSDs do not meet control limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and re-analyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.
- 24.7.19. If a surrogate standard falls outside the control limits, and if there is not obvious chromatographic matrix interference, re-analyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the re-analysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client).

Note: A more detailed discussion of acceptance criteria and corrective action can be found in the laboratory's method SOPs and in Section 12.

24.8. Additional Procedures To Assure Quality Control

- 24.8.1. The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21), and use of PT samples (see Section 15).
- 24.8.2. A discussion regarding MDLs, Limit of Detection (LOD), and Limit of Quantitation (LOQ) can be found in Section 19.
- 24.8.3. Use of formulae to reduce data is discussed in the method SOPs and in Section 20.
- 24.8.4. Selection of appropriate reagents and standards is included in Sections 9 and 21.
- 24.8.5. A discussion on selectivity of the test is included in Section 5.
- 24.8.6. Constant and consistent test conditions are discussed in Section 18.
- 24.8.7. The laboratory sample acceptance policy is included in Section 23.

25. REPORTING RESULTS

- 25.1. The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is a conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory must work with the client during project setup to develop an acceptable solution. Refer to Section 7.
- 25.2. A variety of report formats are available to meet specific needs.
- 25.3. In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client.
- 25.4. Review of reported data is included in Section 19.
- 25.5. Test Reports
 - 25.5.1. Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed, reviewed, and signed by the appropriate Project Manager. At a minimum, the standard laboratory report shall contain the following information:
 - 25.5.1.1. A report title with a "Sample Result" header.
 - 25.5.1.2. Each report cover page printed, which includes the laboratory name, address, and telephone number.
 - 25.5.1.3. A unique identification of the report (e.g., Work Order number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.
 - 25.5.1.4. Page numbers of report are represented at the bottom of each page. The report is sequentially paginated. The final page of the report is labeled as "End of Report".
 - 25.5.1.5. A copy of the Chain-of-Custody (COC).
 - 25.5.1.6. Any COCs involved with subcontracting are included.
 - 25.5.1.7. Any additional addenda to the report must be treated in a similar fashion so it is a recognizable part of the report and cannot

accidentally get separated from the report (e.g., Sampling information).

- 25.5.1.8. The name and address of client and a project name/number, if applicable.
- 25.5.1.9. Client project manager or other contact
- 25.5.1.10. Description and unambiguous identification of the tested sample(s) including the client identification code.
- 25.5.1.11. Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis.
- 25.5.1.12. Date reported or date of revision, if applicable
- 25.5.1.13. Method of analysis including method code (EPA, Standard Methods, etc)
- 25.5.1.14. Certification Summary report, where required, will document that unless otherwise noted, all analytes tested and reported by the laboratory were covered by the noted certifications.
- 25.5.1.15. Reporting limit
- 25.5.1.16. Method detection limits (if requested)
- 25.5.1.17. Definition of data qualifiers and reporting acronyms, e.g., ND
- 25.5.1.18. Sample results
- 25.5.1.19. QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits
- 25.5.1.20. Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (refer to Section 25.2.4 – Item 3, regarding additional addenda).
- 25.5.1.21. A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.
- 25.5.1.22. A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.
- 25.5.1.23. A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Lab Director.

- 25.5.1.24. When TNI accreditation is required, the lab must certify that the test results meet all requirements of TNI or provide reasons and/or justification if they do not.
- 25.5.1.25. The laboratory includes a cover page.
- 25.5.1.26. Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.
- 25.5.1.27. When soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.
- 25.5.1.28. Appropriate laboratory certification number for the state of origin of the sample, if applicable.
- 25.5.2. If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report, e.g., partial report, or how your lab identifies it. A complete report must be sent once all of the work has been completed.
- 25.5.3. Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.
- 25.5.4. A clear statement notifying the client that non-accredited tests were performed and directing the client to the laboratory’s accreditation certificates of approval shall be provided when non-accredited tests are included in the report.

Note: Refer to the Corporate SOP on Electronic Reporting and Signature Policy CA-L-P-002 for details on internally applying electronic signatures of approval.
- 25.5.5. Reports for Ohio VAP work require a VAP affidavit be completed and included with the report. One affidavit can be provided for multiple reports for a project.

Note: For additional information on Ohio VAP affidavits refer to OAC Rule 3745-300-04 and OAC Rule 3745-300-13(N), effective March 1, 2009.

25.6. Reporting Level or Report Type

- 25.6.1. The laboratory offers two levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

25.6.2. Level I is a report with the features described in Section 25.2 above.

25.6.3. Level II is a Level I report plus summary information, including results for the method blank reported to the laboratory MDL, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.

25.6.4. Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.

25.6.5. Level IV is the same as Level III with the addition of all raw supporting data. In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Procedures used to ensure client confidentiality are outlined in Section 25.7.

25.7. Electronic Data Deliverables (EDDs)

25.7.1. EDDs are routinely offered as part of TestAmerica services. TestAmerica Canton offers a variety of EDD formats including (but not limited to) ADR, EQuIS, GISKey, Region 5, NJHAZsite, and a wide variety of client specific multi-file, Excel and flat file formats.

25.7.2. EDD specifications are submitted to the IT Department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

25.7.3. EDDs must be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

25.8. Supplemental Information For Test

25.8.1. The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report.

- 25.8.2. 25.4.1 Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.
 - 25.8.3. 25.4.2 Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet TNI sample acceptance requirements such as improper container, holding time, or temperature.
 - 25.8.4. 25.4.3 Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.
 - 25.8.5. 25.4.4 Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response must be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.
 - 25.8.6. When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.
- 25.9. Environmental Testing Obtained From Subcontractors
- 25.9.1. If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the Corporate SOP CA-L-S-002, Subcontracting.
 - 25.9.2. Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of the TestAmerica network are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.
- 25.10. Client Confidentiality
- 25.10.1. In situations involving the transmission of environmental test results by telephone, facsimile, or other electronic means, client confidentiality must be maintained.
 - 25.10.2. TestAmerica will not intentionally divulge to any person (other than the client or any other person designated by the client in writing) any information regarding the services provided by TestAmerica or any

information disclosed to TestAmerica by the client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Note: This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

25.10.3. Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

25.10.4. "Confidentiality Notice: The information contained in this message is intended only for the use of the addressee, and may be confidential and/or privileged. If the reader of this message is not the intended recipient, or the employee or agent responsible to deliver it to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify the sender immediately."

25.11. Format Of Reports

25.11.1. The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.12. Amendments To Test Reports

25.12.1. Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

25.12.2. When the report is re-issued, a notation of "report reissue" is placed on the cover/signature page of the report or at the top of the narrative page with a brief explanation of reason for the reissue and a reference back to the 1st final report generated.

25.13. Policies On Client Requests For Amendments

25.13.1. Policy on Data Omissions or Reporting Limit Increases

25.13.2. Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

25.13.2.1. Laboratory error

25.13.2.2. Sample identification is indeterminate (confusion between COC and sample labels).

25.13.2.3. An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.

25.13.2.4. Incorrect limits reported based on regulatory requirements

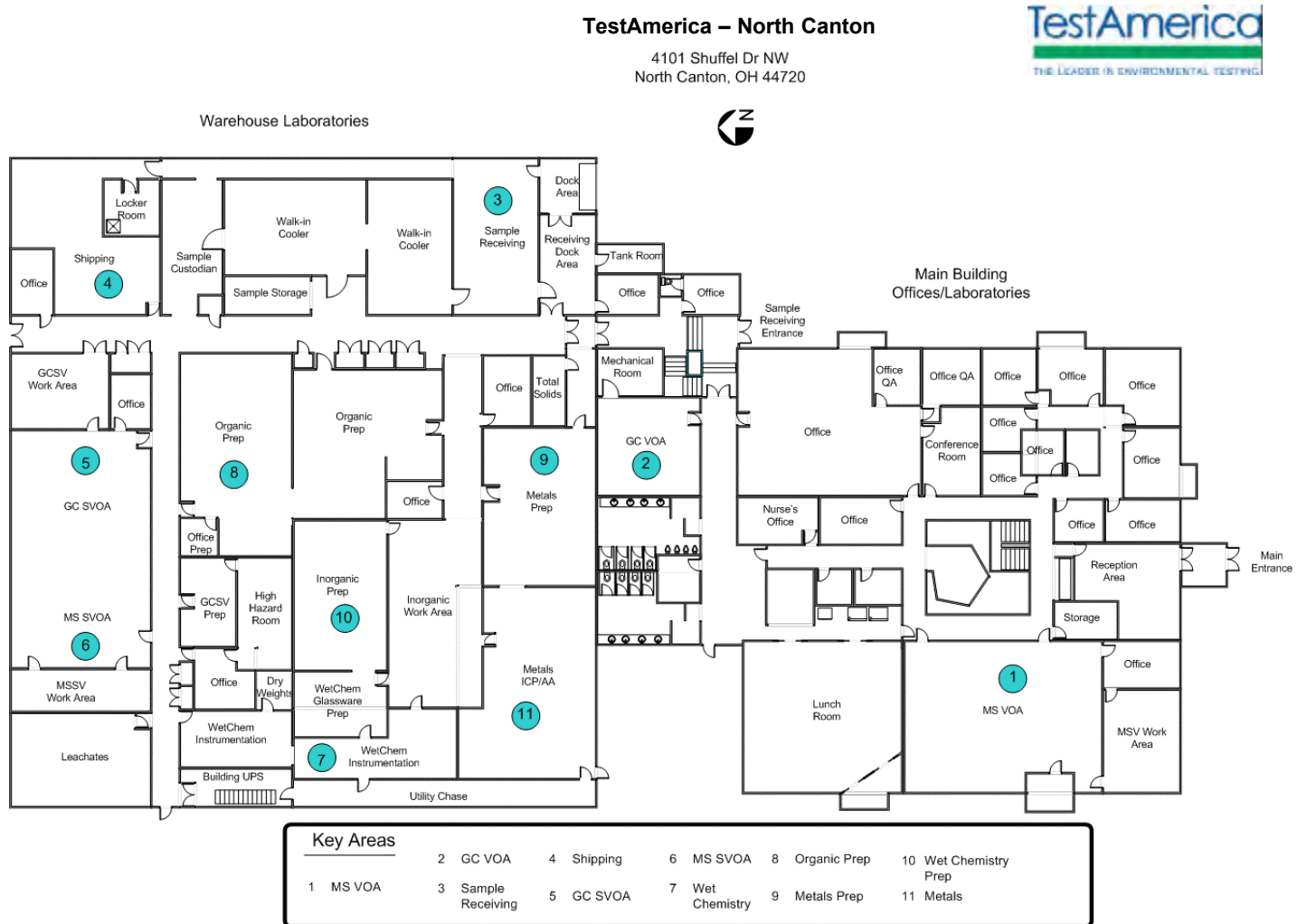
25.13.2.5. The requested change has absolutely no possible impact on the interpretation of the analytical results and there is no possibility of the change being interpreted as misrepresentation by anyone inside or outside of our company.

25.14. Multiple Reports

25.14.1. TestAmerica does not issue multiple reports for the same work order where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

Appendix 1.

Laboratory Floor Plan



Appendix 2. Laboratory Method Listing

Wet Chemistry Methods 1

| Analytical Parameters | Matrix | Fields of Testing | | |
|---|--------|-------------------|------------------------|--------------|
| | | CWA | RCRA (SW846) | Other |
| Alkalinity, Bicarbonate, Carbonate | Water | 310.1. 2 | | SM 2320 B |
| | | | -- | -- |
| Biochemical Oxygen Demand, Carbonaceous | Water | EPA 405.1 | -- | SM 5210 B |
| Anions, Bromide, Chloride, Fluoride, Sulfate, Nitrite, Nitrate, ortho-phosphate Chemical Oxygen Demand | Water | EPA 300.0 | EPA 9056A | -- |
| | Waste | EPA 300.0 | EPA 9056A | -- |
| | Solid | EPA 300.0 (M) | EPA 9056A | -- |
| | Water | EPA 410.4 | -- | SM 5220D |
| | Waste | EPA 410.4 | -- | -- |
| Chloride | Water | EPA 325.22 | EPA 9251 | SM 4500 Cl-E |
| | | | | |
| | Solid | | | -- |
| Chromium, Hexavalent | Water | EPA 3500-Cr-B | EPA 7196A | SM 3500-Cr-B |
| | Waste | EPA 3500-Cr-B | EPA 7196A | SM 3500-Cr-B |
| | Solid | -- | EPA 3060A EPA 7196A | -- |

1 Any matrix not listed is not applicable for the associated method

2 Removed from 40CFR

| Analytical Parameters | Matrix | Fields of Testing | | | |
|---------------------------------------|--------|-------------------|-------------------------|-----------------|--|
| | | | CWA | RCRA (SW846) | Other |
| Specific Conductance | Water | | EPA 120.1 | EPA 9050A | SM 2510B |
| | Waste | | EPA 120.1 | EPA 9050A | -- |
| | Solid | | -- | | -- |
| Chlorine, Residual | Water | | EPA 330.52 | -- | SM 4500 CL-G |
| Cyanide (Amenable) | Water | | EPA 335.12 | EPA 9012A, B | SM 4500 CN-G |
| | Solid | | -- | EPA 9012A, B | -- |
| Cyanide (Total) | Water | | EPA 335.4 | EPA 9012A, B | SM 4500-CN E 335.2-CLP-M (Ohio VAP) |
| | Waste | | -- | EPA 9012A, B | -- |
| | Solid | | -- | EPA 9012A, B | 335.2-CLP-M (Ohio VAP) |
| Cyanide (Weak and Dissociable) (Free) | Water | | | -- | SM 4500-CN I |
| Dissolved Oxygen | Water | | 360.12 | -- | SM 4500 O-G |
| Flash Point | Waste | | -- | EPA 1010, 1010A | |
| | Solid | | -- | EPA 1010, 1010A | |
| Fluoride | Water | | EPA 340.22 | | SM 4500 F-C, ISE |
| | Waste | | EPA 340.2 (M) 2 | | -- |
| | Solid | | | | -- |
| Iron, Ferrous & Ferric | Water | | | -- | SM 3500 FE D |
| Hardness | Water | | EPA 130.22 | -- | SM 2340B SM 2340C |
| Moisture | Solid | | --- | EPA 160.3 (M) | --- |
| Nitrogen, Ammonia | Water | | EPA 350.3 EPA 350.22 | -- | SM 4500 NH3-C(Titration) SM 4500 NH3-D(ISE) |
| | Waste | | EPA 350.3 EPA 350.22 | -- | |
| | Solid | | EPA 350.3 EPA 350.22 | -- | |

| Analytical Parameters | Matrix | Fields of Testing | | | |
|--|--------|-------------------|------------|--------------------------|---------------|
| | | | CWA | RCRA (SW846) | Other |
| Total Kjeldahl Nitrogen (TKN) | Water | | EPA 351.3 | -- | SM 4500 NH3-C |
| | Waste | | EPA 351.3 | -- | -- |
| | Solid | | EPA 351.3 | -- | -- |
| Oil and Grease (Hexane Extractable Material) | Water | | EPA 1664A | | -- |
| | Waste | | EPA 1664A | | -- |
| | Solid | | -- | | -- |
| Ortho-phosphate o-PO4 | Water | | EPA 365.1 | | SM 4500 P-E |
| | Waste | | | | -- |
| | Solid | | | | -- |
| pH | Water | | EPA 150.12 | EPA 9040B EPA 9040C | SM 4500 H+-B |
| | Waste | | | EPA 9045C, C EPA 9041 | SM 4500 H+-B |
| | Solid | | --- | EPA 9045C, D | -- |
| Paint Filter | Water | | -- | EPA 9095A | -- |
| Phenolics | Water | | EPA 420.1 | -- | -- |
| | Waste | | -- | EPA 9065 | -- |
| | Solid | | -- | EPA 9065 | -- |
| Phosphorus (Total) | Water | | EPA 365.1 | -- | SM 4500 P-E |
| | Waste | | EPA 365.1 | -- | -- |
| | Solid | | EPA 365.1 | -- | -- |
| Sulfate (SO4) | Water | | EPA 375.42 | EPA 9038 | -- |
| | Waste | | EPA 375.42 | EPA 9038 | -- |
| | Solid | | | | -- |

| Analytical Parameters | Matrix | Fields of Testing | | | |
|---|-------------|-------------------|---------------------|------------|---------------------|
| | | | CWA | RCRA | Other |
| Sulfide | Water | | EPA 376.12 | 9030B/9034 | SM 4500 S2-E |
| Total Organic Carbon (TOC) Total Petroleum | Water | | EPA 415.12 | EPA 9060 | SM 5310 C |
| | Waste | | -- | EPA 9060 | -- |
| | Solid Water | | | | Walkley-Black -- |
| Hydrocarbons | | | | | |
| | Waste | | EPA 1664A (SGT-HEM) | | -- |
| | Solid | | -- | | -- |
| Total Solids | Water | | EPA 160.3 | -- | -- |
| | Waste | | EPA 160.3 | -- | -- |
| | Solid | | EPA 160.3 (M) | -- | -- |
| Total Dissolved Solids | Water | | EPA 160.1 | -- | SM2540C |
| Total Suspended Solids | Water | | EPA 160.2 | --- | SM2540D |
| | | | | | |
| Settleable Solids | Water | | EPA 160.5 | -- | SM2540F |
| Turbidity | Water | | EPA 180.1 | -- | -- |
| Specific Gravity | Water | | | | SM 2710F |

Methods for Mercury by Cold Vapor Atomic Absorption

| Analytical Parameters | Matrix | Fields of Testing | | |
|-----------------------|---------------|-------------------|------------------|-------|
| | | CWA | RCRA (SW846) | Other |
| Mercury (CVAA) | Water | EPA 245.1 | EPA 7470A | -- |
| | TCLP Leachate | -- | EPA 7470A | -- |
| | Waste | -- | EPA 7471A, 7471B | -- |
| | Solid | | EPA 7471A, 7471B | -- |

Methods for Mercury by Cold Vapor Atomic Fluorescence

| Analytical Parameters | Matrix | Fields of Testing | | |
|----------------------------|--------|-------------------|--------------|-----------|
| | | CWA | RCRA (SW846) | Other |
| Mercury, Low Level (CVAFS) | Water | -- | -- | EPA 1631E |
| | Solid | -- | -- | EPA 1631E |

Methods for Metals by ICP and ICPMS

| Analytical Parameters | Matrix | Fields of Testing | | |
|--------------------------|--------|-------------------|------------------|-------|
| | | CWA | RCRA (SW846) | Other |
| Metals by ICP analysis | Water | EPA 200.7 | EPA 6010B, 6010C | --- |
| | Waste | --- | EPA 6010B, 6010C | --- |
| | Solid | EPA 200.7 | EPA 6010B, 6010C | --- |
| Metals by ICPMS analysis | Water | EPA 200.8 | EPA 6020, 6020A | --- |
| | Waste | --- | EPA 6020, 6020A | --- |

| Analytical Parameters | Matrix | Fields of Testing | | |
|-----------------------|--------|-------------------|-----------------|-------|
| | | CWA | RCRA (SW846) | Other |
| | Solid | EPA 200.8 | EPA 6020, 6020A | --- |

Metals Sample Preparation Methods

| Analytical Parameters | Matrix | Fields of Testing | | |
|--|---------------|-------------------|------------------------|-----------|
| | | CWA | RCRA (SW846) | Other |
| Toxicity Characteristic Leaching Procedure (TCLP)/ SPLP Extraction | Water | --- | EPA 1311 EPA 1312 | --- |
| | Waste | --- | EPA 1311 EPA 1312 | --- |
| | Solid | --- | EPA 1311 EPA 1312 | --- |
| ICP Metals | Water | EPA 200.7 | EPA 3005A EPA 3010A | --- |
| | TCLP Leachate | --- | EPA 3010A | --- |
| | Waste | --- | EPA 3050B | --- |
| | Solid | --- | EPA 3050B | --- |
| ICPMS Metals | Water | EPA 200.8 | EPA 3010A | --- |
| | TCLP | --- | EPA 3010A | --- |
| | Waste | --- | EPA 3050B | --- |
| | Solid | --- | EPA 3050B | --- |
| CVAA Mercury | Water | EPA 245.1 | EPA 7470A | --- |
| | TCLP Leachate | --- | EPA 7470A | --- |
| | Waste | --- | EPA 7471A EPA 7471B | --- |
| | Solid | --- | EPA 7471A EPA 7471B | --- |
| CVAFS Mercury Low Level | Water | --- | --- | EPA 1631E |
| | Solid | --- | --- | EPA 1631E |

Organic Sample Preparation Methods

| Analytical Parameters | Matrix | Fields of Testing | | | |
|------------------------|---------------|-------------------|---------|---|-------|
| | | | CWA | RCRA (SW846) | Other |
| Volatiles by GC/MS | Water | | EPA 624 | EPA 5030B EPA 5030C | --- |
| | Waste | | --- | EPA 5030B EPA 5030C EPA 5035 | --- |
| | Solid | | --- | EPA 5035 EPA 5035A | --- |
| Semivolatiles by GC/MS | Water | | EPA 625 | EPA 3510C EPA 3520C | --- |
| | TCLP Leachate | | --- | EPA 3510C EPA 3520C | --- |
| | Waste | | --- | EPA 3550B EPA 3550C EPA 3540C EPA 3580A | --- |
| | Solid | | --- | EPA 3550B EPA 3550C EPA 3540C | --- |
| Pesticides/PCBs by GC | Water | | EPA 608 | EPA 3510C EPA 3520C | --- |
| | TCLP Leachate | | --- | EPA 3510C EPA 3520C | --- |
| | Waste | | --- | EPA 3550B EPA 3550C EPA 3540C EPA 3546 (PCB only) EPA 3580A | --- |
| | Solid | | --- | EPA 3550B EPA 3550C EPA 3540C | --- |

| Analytical Parameters | Matrix | Fields of Testing | | | |
|---|---------------|-------------------|-----|---|--------|
| | | | CWA | RCRA (SW846) | Other |
| Herbicides by GC | Water | | | EPA 8151A | --- |
| | Waste | | --- | EPA 8151A | --- |
| | Solid | | --- | EPA 8151A | --- |
| Total Petroleum Hydrocarbons (Gasoline Range) by GC | Water | | --- | EPA 5030B EPA 5030C | WI GRO |
| | Waste | | --- | EPA 5030B EPA 5030C EPA 5035 EPA 5035A | WI GRO |
| | Solid | | --- | EPA 5035 EPA 5035A | WI GRO |
| Total Petroleum Hydrocarbons (Diesel Range) by GC | Water | | --- | EPA 3510C EPA 3520C | WI DRO |
| | TCLP Leachate | | --- | EPA 3510C EPA 3520C | --- |
| | Waste | | --- | EPA 3550B EPA 3550C EPA 3580A | WI DRO |
| | Solid | | --- | EPA 3550B EPA 3550C | WI DRO |

Organic Methods of Analysis

| Analytical Parameters | Matrix | Fields of Testing | | | |
|------------------------|---------------|-------------------|---------|---|-------|
| | | | CWA | RCRA (SW846) | Other |
| Volatiles by GC/MS | Water | | EPA 624 | EPA 8260B EPA 8260C | --- |
| | Waste | | --- | EPA 8260B EPA 8260C | --- |
| | Solid | | --- | EPA 8260B EPA 8260C | --- |
| Semivolatiles by GC/MS | Water | | EPA 625 | EPA 8270C EPA 8270D | |
| | Waste | | --- | EPA 8270C EPA 8270D | --- |
| | Solid | | --- | EPA 8270C EPA 8270D | --- |
| Pesticides/PCBs by GC | Water | | EPA 608 | Pesticides 8081A, 8081B PCBs 8082, 8082A | --- |
| | TCLP Leachate | | --- | Pesticides 8081A, 8081B PCBs 8082, 8082A | --- |
| | Waste | | --- | Pesticides 8081A, 8081B PCBs 8082, 8082A | --- |
| | Solid | | --- | Pesticides 8081A, 8081B PCBs 8082, 8082A | --- |

| Analytical Parameters | Matrix | Fields of Testing | | | |
|--|---------------|-------------------|----------|-------------------------------|--------|
| | | | CWA | RCRA (SW846) | Other |
| Phenoxyacid Herbicides by GC | Water | | --- | EPA 8151A | --- |
| | TCLP Leachate | | --- | EPA 8151A | --- |
| | Waste | | --- | EPA 8151A | --- |
| | Solid | | --- | EPA 8151A | --- |
| Gasoline Range Organics by GC | Water | | --- | EPA 8015B (M) EPA 8015C, D | WI GRO |
| | Waste | | --- | EPA 8015B (M) EPA 8015C, D | --- |
| | Solid | | --- | EPA 8015B (M) EPA 8015C, D | WI GRO |
| Total Petroleum Hydrocarbons (Diesel Range) by GC/FID Dissolved Gases RSK-175 | Water | | --- | EPA 8015B (M) EPA 8015C, D | WI DRO |
| | Waste | | --- | EPA 8015B (M) EPA 8015C, D | --- |
| | Water | | --- | --- | SOP |
| Formaldehyde Carbonyl Compounds | Water | | --- | EPA 8315A | --- |
| | Solid | | --- | EPA 8315A | --- |
| Aromatic Acids | Water | | --- | --- | SOP |
| | Solid | | --- | --- | SOP |
| Methyl Mercury | Water | | EPA 1630 | --- | --- |
| | Solid | | EPA 1630 | --- | --- |

Appendix 3. Glossary/Acronyms

Glossary

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQ)

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Analyst: The designated individual who performs the “hands-on” analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (TNI)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

Batch: Environmental samples which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed 20 samples. (TNI)

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample’s true value). (TNI)

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQ)

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (TNI)

1) In calibration of support equipment the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).

2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve: The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

Calibration Standard: A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM): A reference material accompanied by a certificate having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute.

Chain-of-Custody: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes the number and types of containers, the mode of collection, the collector, time of collection, preservation, and requested analyses. (TNI)

Compromised Samples: Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified.

Confidential Business Information (CBI): Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. TNI and its representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:

Second column confirmation

Alternate wavelength

Derivatization

Mass spectral interpretation

Alternative detectors or

Additional cleanup procedures

Conformance: An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Correction: Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis. No significant action is taken to change behavior, process or procedure.

Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria).

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors and collation into a more useable form. (TNI)

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQ)

Duplicate Analyses: The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Equipment Blank: Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.

External Standard Calibration: Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Field Blank: Blank prepared in the field by filling a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Holding Times: The maximum time that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (TNI)

Internal Standard Calibration: Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank: A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Detection Limit (IDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is + 100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. An LCS must be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples must be used to determine batch acceptance.

Least Squares Regression (1st Order Curve): The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit(s) of Detection (LOD) (a.k.a., Method Detection Limit [MDL]): A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. (TNI)

LOD Verification (a.k.a., MDL Verification): A processed QC sample in the matrix of interest, spiked with the analyte at no more than 3X the LOD for single analyte tests and 4X the LOD for multiple analyte tests and processed through the entire analytical procedure.

Limit(s) of Quantitation (LOQ) [a.k.a., Reporting Limit]: The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. (TNI)

(QS) Matrix: The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions must be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.

Drinking Water: any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-aqueous Liquid: any organic liquid with .15% settleable solids.

Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples must be grouped according to origin.

Solids: includes soils, sediments, sludges, and other matrices with .15% settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.

Air and Emissions: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device.
(TNI)

Matrix Spike (spiked sample or fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike is prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Method Detection Limit: The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero

and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Non-conformance: An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Performance Audit: The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (TNI)

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (TNI)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (TNI)

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within specified acceptance criteria. (TNI)

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is of the type of quality needed and expected by the client. (TNI)

Quality Assurance [Project] Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control: The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality. (TNI)

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. (TNI)

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (TNI)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC activities. (TNI)

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (TNI)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Material: A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

Reference Method: A method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (NELAC)

Reference Standard: A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.0-8)

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Second Order Polynomial Curve (Quadratic): The 2nd order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2nd order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. (TNI)

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

Spike: A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies. (TNI)

Standard Operating Procedures (SOPs): A written document which details the method of an operation, analysis, or action with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks. (TNI)

Storage Blank: A blank matrix stored with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes. Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and must be reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit): A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Manager: A member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of accreditation and reporting of results

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

Trip Blank: A blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Uncertainty: A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

Acronyms

| | |
|--------|---|
| ASTM | American Society for Testing & Materials |
| CAR | Corrective Action Report |
| CBI | Confidential Business Information |
| CCB | Continuing Calibration Blank |
| CCV | Continuing Calibration Verification |
| CF | Calibration Factor |
| CFR | Code of Federal Regulations |
| COC | Chain of Custody |
| CQMP | Corporate Quality Management Plan |
| CSM | Customer Service Manager |
| DOC | Demonstration of Capability |
| DoD | Department of Defense |
| DQO | Data Quality Objectives |
| DUP | Duplicate |
| ECO | Ethics and Compliance Officer |
| EDD | Electronic Data Deliverable |
| EHS | Environment, Health and Safety |
| EPA | Environmental Protection Agency |
| GC | Gas Chromatography |
| GC/MS | Gas Chromatography/Mass Spectrometry |
| HPLC | High Performance Liquid Chromatography |
| ICP | Inductively Coupled Plasma Atomic Emission Spectroscopy |
| ICP/MS | ICP/Mass Spectrometry |
| ICB | Initial Calibration Blank |
| ICV | Initial Calibration Verification |
| IDL | Instrument Detection Limit |
| IEC | International Electrotechnical Commission |
| IS | Internal Standard |
| ISO | International Organization for Standardization |
| LCS | Laboratory Control Sample |
| LCSD | Laboratory Control Sample Duplicate |
| LOD | Limit of Detection |
| LOQ | Limit of Quantitation |
| LIMS | Laboratory Information Management System |
| MDL | Method Detection Limit |
| MDLCK | MDL Check Standard |
| MDLV | MDL Verification Check Standard |
| MRL | Method Reporting Limit Check Standard |
| MS | Matrix Spike |
| MSD | Matrix Spike Duplicate |
| MSDS | Material Safety Data Sheet |
| NCM | Nonconformance Memo |
| NELAP | National Environmental Laboratory Accreditation Program |
| NIST | National Institute of Standards and Technology |
| NPDES | National Pollutant Discharge Elimination System |
| OVAP | Ohio Voluntary Action Program |
| PM | Project Manager |
| PT | Performance Testing |
| TIC | Tentatively Identified Compound |
| TNI | The NELAC Institute |

| | |
|-------|---|
| QAM | Quality Assurance Manual |
| QA/QC | Quality Assurance / Quality Control |
| QAPP | Quality Assurance Project Plan |
| RCRA | Resource Conservation and Recovery Act |
| RF | Response Factor |
| RFP | Request for Proposal |
| RPD | Relative Percent Difference |
| RSD | Relative Standard Deviation |
| SAP | Sampling and Analysis Plan |
| SD | Standard Deviation |
| SOP | Standard Operating Procedure |
| SPLP | SPLP = Synthetic Precipitation Leaching Procedure |
| TAT | Turn-Around Time |
| TCLP | Toxicity Characteristic Leaching Procedure |
| TSCA | Toxic Substances Control Act |
| USACE | United States Army Corps of Engineers |
| USDA | United States Department of Agriculture |
| VOA | Volatiles |

Appendix 4. Laboratory Certifications, Accreditations, Validations

TestAmerica North Canton maintains certifications, accreditations, certifications, and approvals with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

| Organization | Certificate Number | Organization | Certificate Number |
|---|--------------------|-----------------------------|--------------------|
| California | 01144CA | Nevada | OH-00048208A |
| Connecticut | PH-0590 | New Jersey | OH001 |
| Florida | E87225 | New York | 10975 |
| Georgia | --- | OVAP | CL0024 |
| Illinois | 001298 | Pennsylvania | 68-00340 |
| Kansas | E-10336 | USDA (Dept. of Agriculture) | P330-08-00123 |
| Kentucky Underground Storage Tank Program | 0058 | Washington | C971 |
| Minnesota | 039-999-348 | West Virginia | 210 |
| DoD – LAB | L2315 | Wisconsin | 999518190 |
| Texas | T104704517-13-2 | Virginia | 2857 |

The certificates and accredited parameter lists are available for each State/Program at www.testamericainc.com under Analytical Services Search – Certifications.

Quality Assurance Manual

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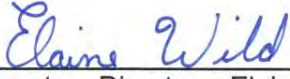





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Title Page:

Quality Assurance Manual Approval Signatures

| | |
|--|------------------------------------|
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|  _____ Quality Manager – Tony L. Byrd | <u>05-23-2016</u> _____ Date |
|  _____ Technical Director – Terry Romanko | <u>5/23/16</u> _____ Date |
|  _____ ES&H Manager – Michael Ridenhower | <u>5/23/16</u> _____ Date |
|  _____ Lab Operations Manager - Aaron Dickson | <u>5/23/16</u> _____ Date |
|  _____ Manager, (Radiochemistry) – Rachel Mueller Manager of PM – Rhonda Ridenhower | <u>5-23-16</u> _____ Date |
|  _____ Technical Manager, (Radiochemistry) – Sarah Bernsen | <u>05/23/2016</u> _____ Date |
|  _____ Technical Manager, (Inorganics) – Kristen Ely | <u>5/23/16</u> _____ Date |
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|  _____ Manager of PM – Rhonda Ridenhower Manager, (Radiochemistry) – Rachel Mueller | <u>5-23-16</u> _____ Date |

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REFERENCED CORPORATE SOPs AND POLICIES

| SOP / Policy Reference | Title |
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| CA-I-P-002 | Electronic Reporting and Signature Policy |
| CA-L-P-002 | Contract Compliance Policy |
| CA-L-S-002 | Subcontracting Procedures |
| CA-Q-M-002 | Corporate Quality Management Plan |
| CA-Q-S-001 | Solvent and Acid Lot Testing and Approval |
| CA-Q-S-002 | Acceptable Manual Integration Practices |
| CA-Q-S-006 | Detection Limits |
| CA-Q-S-009 | Root Cause Analysis |
| CA-T-P-001 | Qualified Products List |
| CW-E-M-001 | Corporate Environmental Health & Safety Manual |
| CW-F-P-002 | Company-Wide Authorization Matrix |
| CW-F-P-004 | Procurement and Contracts Policy |
| CW-F-S-007 | Capital Expenditure, Controlled Purchase Requests and Fixed Asset Capitalization |
| CW-L-P-004 | Ethics Policy |
| CW-L-S-002 | Internal Investigation |
| CW-Q-S-001 | Corporate Document Control and Archiving |
| CW-Q-S-002 | Writing a Standard Operating Procedure (SOPs) |
| CW-Q-S-003 | Internal Auditing |
| CW-Q-S-004 | Management Systems Review |
| CW-Q-S-005 | Data Recall Process |
| CA-C-S-001 | Work Sharing Process |

REFERENCED LABORATORY SOPs

TestAmerica St. Louis Standard Operating Procedures are listed in [Appendix 5](#).

SECTION 3. INTRODUCTION, SCOPE AND APPLICABILITY

3.1 Introduction and Compliance References

TestAmerica St. Louis's Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica's data quality goals. The laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with U.S. Department of Energy Quality Systems for Analytical Services (QSAS, current revision), U.S. Department of Defense Quality Systems Manual for Environmental Laboratories (QSM, current version), The NELAC Institute (TNI) Standard, dated 2009, Volume 1 Modules 2 and 4, and ISO/IEC Guide 17025:2005(E). In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Quality Management Plan (CQMP) and the various accreditation and certification programs listed in [Appendix 3](#). The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations.

The QAM has been prepared to be consistent with the requirements of the following documents:

- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories*, EPA, March 1979.
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846)*, Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008, Final Update V, August 2015.
- U.S. Department of Defense/Department of Energy, *Quality Systems Manual*, Version 5.0, July 2013.
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- APHA, *Standard Methods for the Examination of Water and Wastewater*, 18th Edition, 19th, 20th and 21st, and on-line Editions.
- U.S. Department of Energy Order 414.1B, *Quality Assurance*, Approved April 29, 2004.
- U.S. Department of Energy Order 414.1C, *Quality Assurance*, June 17, 2005.
- U.S. Department of Energy Order 414.1D, *Quality Assurance*, April, 25, 2011.
- U.S. Department of Energy, *Quality Systems for Analytical Services*, Revision 2.9, January 2012.
- Nuclear Regulatory Commission (NRC) Quality Assurance Requirements.
- Federal Register 10CFR 50 Appendix B
- Toxic Substances Control Act (TSCA).
- ASME NQA-1-2000 Quality Assurance Requirements for Nuclear Facility Applications (for nuclear safety related activities)
- ASME NQA-1-1994 Quality Assurance Requirements for Nuclear Facility Applications (for nuclear safety related activities)
- Federal Register 10CFR21 and 10CFR50.55e

3.2 Terms and Definitions

A Quality Assurance Program is a company-wide system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization. Refer to [Appendix 4](#) for the Glossary/Acronyms.

3.3 Scope / Fields of Testing

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among air, drinking water, effluent water, groundwater, hazardous waste, sludge and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical and physical parameters. The Program also contains guidelines on maintaining documentation of analytical processes, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all analytical requests are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found on the www.testamericainc.com web site. The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet these requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director, Technical Directors and the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

3.4 Management of the Manual

3.4.1 Review Process

The template on which this manual is based is reviewed annually by Corporate Quality Management Personnel to assure that it remains in compliance with Section 3.1. This manual itself is reviewed **annually** by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to SOP ST-QA-0035, "Preparation and Management of Standard Operating Procedures".

SECTION 4. MANAGEMENT REQUIREMENTS

4.1 Overview

TestAmerica St. Louis is a local operating unit of TestAmerica Laboratories, Inc. The organizational structure, responsibilities and authorities of the corporate staff of TestAmerica Laboratories, Inc. are presented in the CQMP. The laboratory has day-to-day independent operational authority overseen by corporate officers (e.g., President and Chief Executive Officer (CEO), Chief Operating Officer (COO), Executive Vice President (VP) Operations, Corporate Quality, etc.). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate & TestAmerica St. Louis is presented in Figure 4-1.

4.2 Roles and Responsibilities

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions briefly define each role in its relationship to the Quality Assurance Program. More extensive job descriptions are maintained by laboratory management.

4.2.1 Additional Requirements for Laboratories

The responsibility for quality resides with every employee of the laboratory. All employees have access to the QAM, are trained to this manual, and are responsible for upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. Role descriptions for corporate personnel are defined in the CQMP. This manual is specific to the operations of TestAmerica's St. Louis laboratory.

4.2.2 Laboratory Director (LD) or Designee

The St. Louis Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to

his/her respective General Manager (GM). The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

Specific Responsibilities include, but are not limited to:

- The Laboratory Director is responsible for maintaining positive operating margin to the company at the laboratory level and for meeting and exceeding the annual budget.
- Ensures that personnel are free from commercial, financial and other undue pressures which might adversely affect their quality of work
- Supervise all laboratory personnel and provide guidance and direction as needed.
- Ensure that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.
- Responsible for ensuring compliance and integration of facility operation with corporate and regulatory policies and procedures.
- Ensures that appropriate corrective actions are taken to address issues identified by external and internal audits.
- The laboratory Director has signatory authority for the QAM, policies, SOPs and contracts (as defined by TestAmerica policy).

4.2.3 Quality Assurance (QA) Manager or Designee

The QA Manager has responsibility and authority to ensure the continuous implementation, maintenance and improvement of the quality system.

The QA Manager reports directly to the Laboratory Director and has access to Corporate QA for advice and resources. This position is able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence. Corporate QA may be used as a resource in dealing with regulatory requirements, certifications and other quality assurance related items. The QA Manager directs the activities of the QA officers to accomplish specific responsibilities, which include, but are not limited to:

- Serves as the focal point for QA/QC in the laboratory.
- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.
- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Have documented training and/or experience in QA/QC procedures and the laboratory's Quality System.

- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arranging for or conducting internal audits on quality systems and the technical operation.
- The laboratory QA Manager will maintain records of all ethics-related training, including the type and proof of attendance.
- Maintain, improve, and evaluate the corrective action database and the corrective and preventive action systems.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs shall be investigated following procedures outlined in Section 12 and if deemed necessary the procedures may be temporarily suspended during the investigation.
- Objectively monitor standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Review a percentage of all final data reports for internal consistency. Review of Chain of Custody (COC), correspondence with the analytical request, batch QC status, completeness of any corrective action statements, 5% of calculations, format, holding time, sensibility and completeness of the project file contents.
- Review of external audit reports and data validation requests.
- Follow-up with audits to ensure client QAPP requirements are met.
- Establishment of reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or Corporate QA.
- Development of suggestions and recommendations to improve quality systems.
- Research of current state and federal requirements and guidelines.
- Captains the QA team to enable communication and to distribute duties and responsibilities.
- Ensuring Communication & monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Has final authority to accept or reject data and to stop work in progress in the event that procedures or practices compromise the validity and integrity of the analytical data.
- Evaluation of the thoroughness and effectiveness of training.
- **Compliance with ISO 17025** (where applicable)
- Providing Quality Systems training to all new personnel and ensuring that all personnel understand their contributions to the quality system.
- Evaluate the effectiveness of training.
- Has signatory authority over the QAM, SOPs and policies pertaining to QA/QC
- Compliance with the NELAC Standards (where applicable)
- Compliance with the QSM (where applicable)

4.2.4 Technical Manager or Designee

The Technical Manager(s) report(s) directly to the Laboratory Director. He/she is accountable for all analyses and analysts under their experienced supervision and for compliance with the ISO 17025 Standard. The scope of responsibility ranges from the new-hire process and existing technology through the ongoing training and development programs for existing analysts and new instrumentation. Specific responsibilities include, but are not limited to:

- Exercises day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results. Coordinating, writing, and reviewing preparation of all test methods, i.e. SOPs, with regard to quality, integrity, regulatory and optimum and efficient production techniques, and subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples. He/she insures that the SOPs are properly managed and adhered to at the bench. He/she develops standard costing of SOPs to include supplies, labor, overhead, and capacity (design vs. demonstrated versus first-run yield) utilization.
- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.
- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting all new business contracts, insuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.
- Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting and preventive maintenance.
- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Coordinating sample management from "cradle to grave," insuring that no time is lost in locating samples.
- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc.
- Captains department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.
- Coordinates audit responses with the QA Manager.

- Responsible for ensuring compliance with the NELAC Standards
- Compliance with ISO 17025 (where applicable)
- Compliance with the QSM (where applicable)

4.2.5 Technical Director

The Technical Director(s) report(s) directly to the Laboratory Director. The scope of responsibility ranges from the new hire process and existing technology through the on going training and development programs for existing analysts and second and third generation instrumentation.

Specific responsibilities include:

- Assists in coordinating, writing and reviewing SOPs.
- May assist in the review of proposals
- Solves day to day technical issues, provides technical training and guidance to staff, project managers, and clients.
- Investigates technical issues identified by QA, and directs evaluation of new methods.
- Responsible for ensuring compliance with the NELAC Standards
- Compliance with ISO 17025 (where applicable)
- Compliance with the QSM (where applicable)

4.2.6 Manager of Project Management/Customer Service Manager

In addition to filling the requirements of Project Manager for key accounts, he/she fulfills supervisory duties and responsibilities. As Manager, he supervises the Project Management staff, sets standards for and monitors productivity, manages the assignment of accounts and the daily workload and tracks and maintains information for various revenue reports. With the QA Manager, he determines acceptable corrective actions for the nonconformance occurring within his group, develops and reviews standard operating procedures for the group.

Additional responsibilities include:

- Has signatory authority for final reports.
- Training of the Project Management staff
- Notify supervisors of incoming projects and sample delivery schedules
- Coordinate requests for sample containers and sample pick-up/deliveries

4.2.7 Project Manager

- Coordinates and manages customers' projects through all phases of laboratory operations, ensuring fulfillment of TestAmerica's commitment to client requirements, error-free work, and on-time delivery.

- Responsible to ensure that clients get timely responses to status inquiries, resolutions to problems and the agreed upon deliverables
- Discusses with clients any project related problems, resolves service issues and coordinates technical details with the lab staff
- Responsible for staff familiarization with specific quotes, sample log-in review and final report accuracy and completeness
- Maintains communications with clients and Account Executives and serves as a liaison between clients and laboratory operations to meet client's needs.
- Works closely with business unit personnel to manage quotations and change orders for existing scopes of work.
- Generates narratives outlining project observations, QC excursions, and laboratory comments.
- Has signatory authority for final reports.

4.2.8 Department Manager/Supervisor

The Department Manager/Supervisor is responsible for the overall operations of a specific laboratory area.

These responsibilities include but are not limited to:

- Meeting client satisfaction goals, managing the human resources within the department, and ensuring health and safety and quality assurance plan compliance.
- Serves as a technical resource to department employees, as well as Project Managers, sales personnel, and clients.
- Make recommendations to laboratory management in regard to process improvements.
- Ensure analysts in their department adhere to applicable SOPs and the QAM.

4.2.9 Chemist/Analyst

- Laboratory analysts are responsible for the generation of data by preparing and analyzing samples according to written SOPs and client requirements.
- They are responsible for understanding the requirements in the QAM and the SOPs associated with their specific function.
- Perform the initial technical review of sample preparation information, calculations, qualitative identifications and raw data with the authority to stop, accept, or reject data based on compliance with self-defined QC criteria.
- The laboratory analyst also provides prompt documentation and notification to the Group Leader of problems or anomalies detected.
- Monitor, calibrate, and maintain standard laboratory equipment such as refrigerators, ovens, water systems, and pipettes, and instrumentation, as necessary.

4.2.10 Environmental Health and Safety Coordinator

- The Environmental Health and Safety Coordinator is responsible for administering the EH&S program that provides a safe, healthy working environment for all employees and the environment.
- Monitors all areas for unsafe conditions, acts, and potential hazards. Enforces environmental, health, and safety policies and procedures. Maintains regulatory compliance with local, state, and federal laws.
- Makes safety and health recommendations to laboratory management in conjunction with the facility safety committee.
- Develops and maintains the facility's health and safety and waste disposal procedures.
- Conduct ongoing, necessary safety training and conduct new employee safety orientation.
- Assist in developing and maintaining the Chemical Hygiene/Safety Manual.
- Administer dispersal of all Material Safety Data Sheet (MSDS) information.
- Perform regular chemical hygiene and housekeeping instruction.
- Give instruction on proper labeling and practice.
- Serve as chairman of the laboratory safety committee.
- Provide and train personnel on protective equipment.
- Oversee the inspection and maintenance of general safety equipment – fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed.
- Supervise and schedule fire drills and emergency evacuation drills.
- Determine what initial and subsequent exposure monitoring, if necessary to determine potential employee exposure to chemicals used in the laboratory.
- When determined necessary, conduct exposure monitoring assessments.
- Determine when a complaint of possible over-exposure is “reasonable” and should be referred for medical consultation.
- Assist in the internal and external coordination of the medical consultation/monitoring program conducted by TestAmerica's medical consultants.

4.2.11 Radiation Safety Officer (RSO)

- Under the direction of the Laboratory Director, implements the radiation protection program that, as a minimum, provides compliance with pertinent regulatory requirements, license provisions, and the Radiation Protection Program.
- Maintains direct access to the Laboratory Director on matters relating to radiological protection.
- Maintains sufficient organizational independence to review and evaluate activities involving the use of radioactive materials.
- Provides Authorized Users and radiation workers with the instruments, protective devices, dosimetry, training, and other items needed to perform their work in accordance with the radiological protection program elements.

- Maintains original copies of all St. Louis licenses/permits, including attachments and amendments, for radioactive materials.
- Directs program to monitor and control radioactive materials throughout the laboratory
- Conducts radiation safety training
- Maintains inventory of standards, tracers, and radiological samples
- Manages segregated area for storing radioactive and mixed wastes

4.3 Deputies

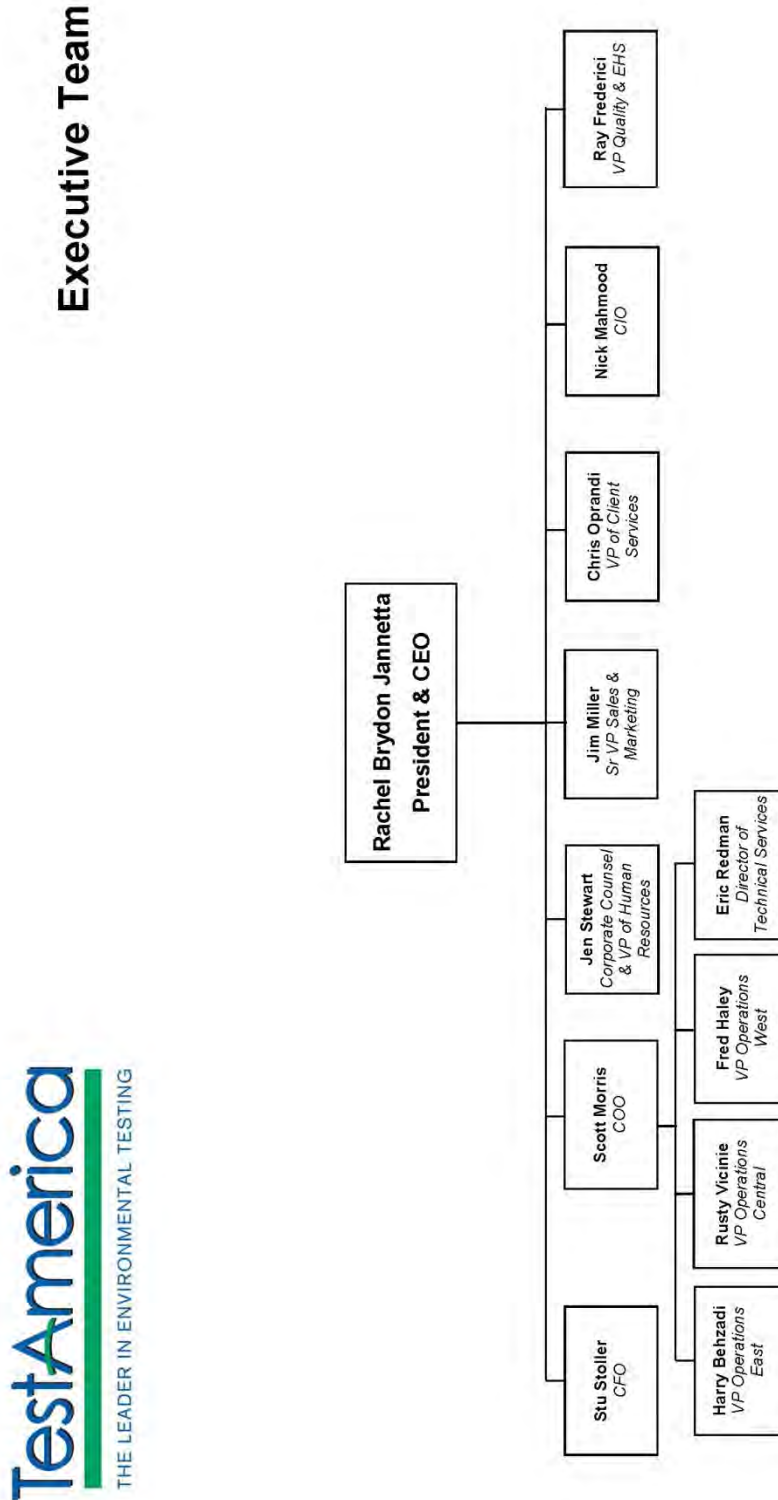
The following table defines who assumes the responsibilities of key personnel in their absence:

| Key Personnel | Deputy |
|--|---|
| Elaine Wild* Laboratory Director | Aaron Dickson Lab Operations Manager |
| Tony Byrd Quality Manager | Marti Ward Quality Assurance Specialist |
| Kristen Ely* Inorganics Technical Manager | Cory Buffington [Metals Deputy] Metals Analyst Jacob Boyd [Wet Chem Deputy] Wet Chem Group Lead |
| Sarah Bernsen* Radiochemistry Prep Technical Manager Rachel Muller Radiochemistry Analyst Technical Manager | Rachel Muller [Count Room Deputy] Radiochemistry Analyst Manager Sarah Bernsen [Prep Deputy] Radiochemistry Prep Manager |
| Michael Ridenhower EHS Coordinator | Terry Romanko* Technical Director |
| Michael Ridenhower Radiation Safety Officer | Terry Romanko* Technical Director |
| Rhonda Ridenhower Manager of Project Management | Jayna Awalt Project Manager |
| Aaron Dickson* Extractable Organics Technical Manager | Dennis Konopka Lab Operations Manager |
| Andrew Buettner* Volatile Organics Technical Manager | Gary Bonkoski Volatile Organics Analyst |

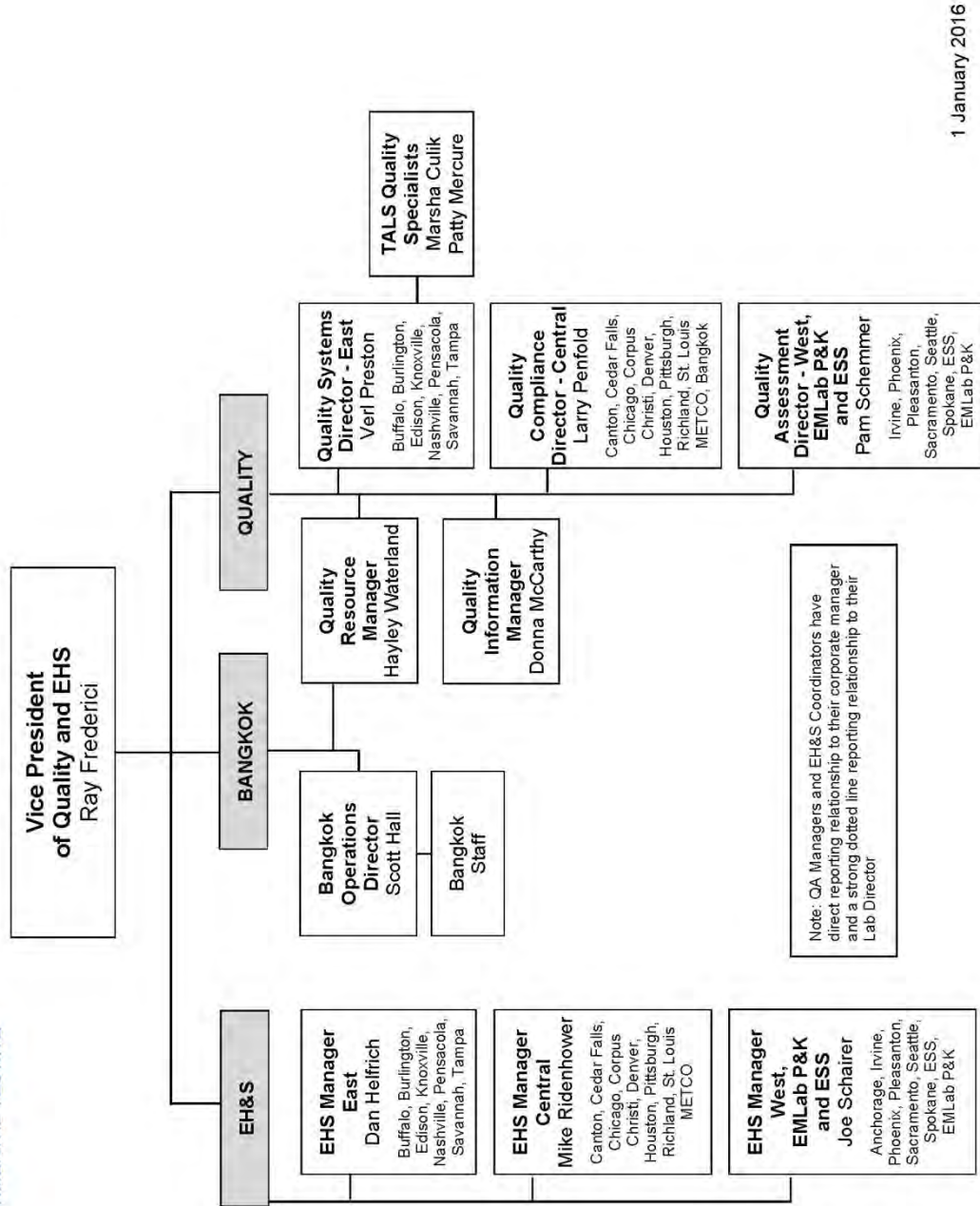
In the event that key Technical Managers are absent for a period exceeding 15 consecutive calendar days, the deputy will temporarily perform the absentee's functions. If the absence exceeds thirty-five consecutive calendar days, the primary accreditation body shall be notified in writing.

Technical Managers are designated with an asterisk (*).

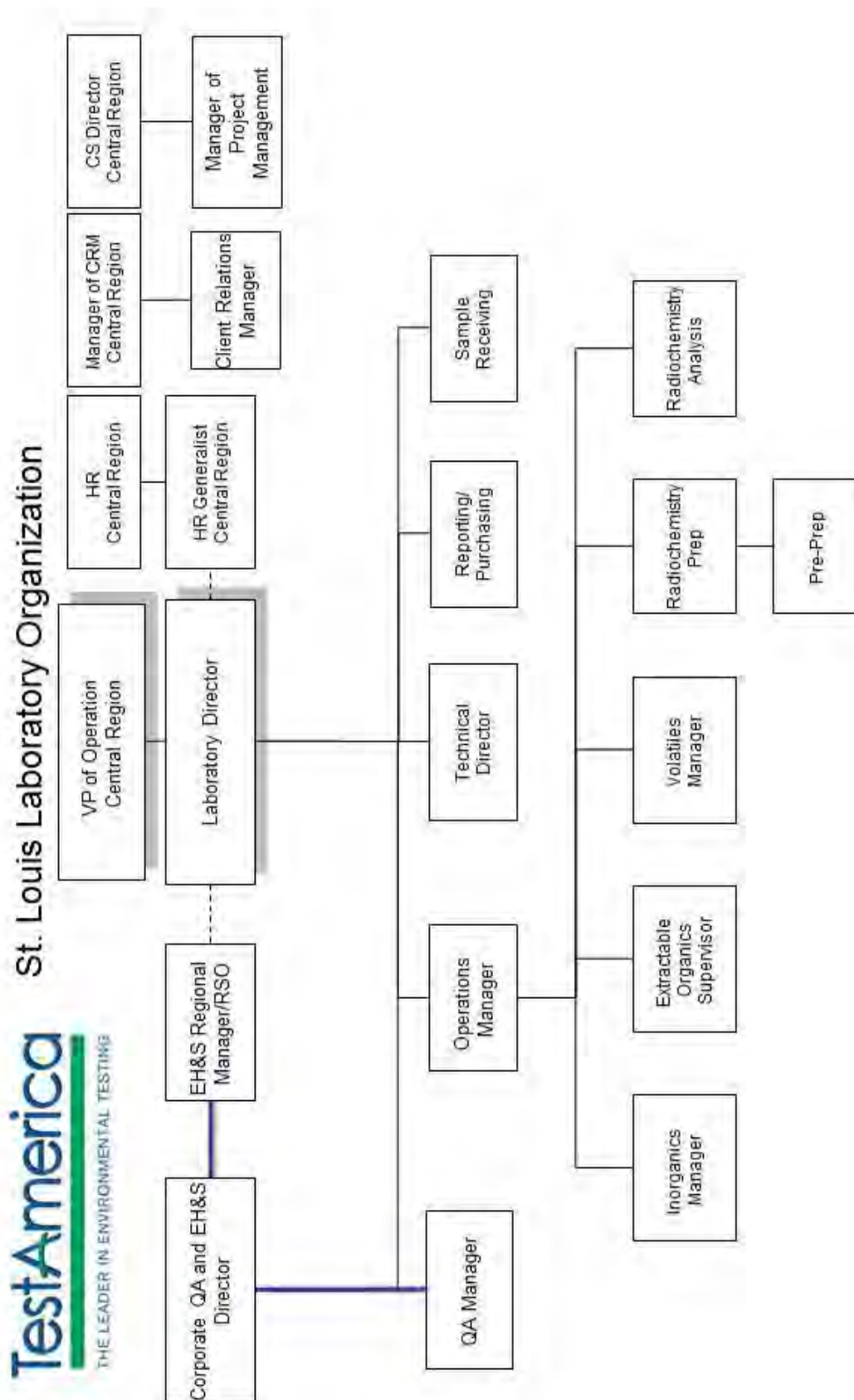
Figure 4-1. Corporate and Laboratory Organization Charts



Quality, EHS, Bangkok



1 January 2016



Note: QA Manager and EH&S Manager have a direct reporting relationship to both operations leadership and corporate functional leadership.

SECTION 5. QUALITY SYSTEM

5.1 Quality Policy Statement

It is TestAmerica's Policy to:

- ❖ Provide data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols.
- ❖ Effectively manage all aspects of the laboratory and business operations by the highest ethical standards.
- ❖ Continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. TestAmerica recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.
- ❖ Provide clients with the highest level of professionalism and the best service practices in the industry.
- ❖ To comply with the ISO/IEC 17025:2005(E) International Standard, the 2009 TNI Standard and to continually improve the effectiveness of the management system.
- ❖ TestAmerica St. Louis' policy includes compliance with the Department of Defense QSM and the Department of Energy QSAS.

Every staff member at the laboratory plays an integral part in quality assurance and is held responsible and accountable for familiarizing themselves with the quality program documentation and implementing those policies and procedures to ensure the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

5.2 Ethics and Data Integrity

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The elements of TestAmerica's Ethics and Data Integrity Program include:

- An Ethics Policy (Corporate Policy No. CW-L-P-004) and Employee Ethics Statements.
- Ethics and Compliance Officers (ECOs).
- A Training Program.
- Self-governance through disciplinary action for violations.
- A Confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (Corporate SOP No. CW-L-S-002)
- Procedures and guidance for recalling data if necessary (Corporate SOP No. CW-Q-S-005).

- Effective external and internal monitoring system that includes procedures for internal audits (Section 15).
- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.
- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

5.3 Quality System Documentation

The laboratory's Quality System is communicated through a variety of documents.

- Quality Assurance Manual – Each laboratory has a lab-specific quality assurance manual.
- Corporate SOPs and Policies – Corporate SOPs and Policies are developed for use by all relevant laboratories. They are incorporated into the laboratory's normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- Work Instructions – A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).
- Laboratory SOPs – General and Technical
- Laboratory QA/QC Policy Memorandums
- Laboratory Waste Management Plan
- Laboratory Radiation Safety Program

5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Corporate Quality Management Plan (CQMP)
- Corporate SOPs and Policies
- Laboratory QA/QC Policy Memorandum
- Laboratory Quality Assurance Manual (QAM)
- Laboratory SOPs and Policies

- Other (Work Instructions (WI), memos, flow charts, etc.)

Note: The laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the CQMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's QAM shall take precedence over the CQMP in those cases.

5.4 QA/QC Objectives for the Measurement of Data

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term "*analytical quality control*". QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. The client is responsible for developing the QAPP. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS).

5.4.1 Precision

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

5.4.2 Accuracy

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

5.4.3 Representativeness

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

5.4.4 Comparability

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories.

5.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

5.4.6 Selectivity

Selectivity is defined as: The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc..

5.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (Method Detection Limit/Minimum Detectable Activity/Detection Limit) or quantified (Reporting Limit/Limit of Quantitation).

5.5 Criteria for Quality Indicators

The laboratory maintains quality limits reference data through the LIMS containing the precision and accuracy acceptability limits for performed analyses. This data is managed by the laboratory's QA department using the Control Chart app in LIMS. Printed and/or electronic copies of method specific QC limits are available upon request. Unless otherwise noted, limits are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits are contained in SOP ST-QA-0014 and Section 24.

5.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory (dated and approved by the QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in Section 24. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

5.6.1 QC Charts

As the QC limits are calculated, QC charts are generated to show warning and control limits for the purpose of evaluating trends. The QA Manager evaluates these to determine if adjustments need to be made or for corrective actions to methods. All findings are documented and kept on file. See SOP ST-QA-0014 "Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts".

5.7 Quality System Metrics

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 16). These metrics are used to drive continuous improvement in the laboratory's Quality System.

SECTION 6. DOCUMENT CONTROL

6.1 Overview

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

Corporate Quality posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving Corporate documents is found in Corporate SOP No. CW-Q-S-001, Corporate Document Control and Archiving. The laboratory's internal document control procedure is defined in SOP No. ST-QA-0023, "Control of Records".

The laboratory QA Department also maintains access (controls) to various references and document sources integral to the operation of the laboratory. This includes reference methods, regulations and instrument manuals (hard or electronic copies).

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, validation requests and corrective action reports. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports.

6.2 Document Approval and Issue

The pertinent elements of a document control system for each document include a unique document title and number, pagination, the total number of pages of the item or an 'end of document' page, the effective date, revision number and the laboratory's name. The QA personnel are responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department and other management. In order to develop a new document, a technical manager submits a draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version

information to the document and retain that document as the official document on file. That document is then provided to all applicable operational units (may include electronic access). Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed at a minimum of every two years. When related to DoD (Department of Defense) work, the review will be done annually. Revisions are made as appropriate. Changes to documents occur when a procedural change warrants.

6.3 Procedures for Document Control Policy

For changes to the QA Manual, refer to SOP No. ST-QA-0035, "Preparation and Management of Standard Operating Procedures". Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. Electronic copies are stored on the Public server in the QA folder.

For changes to SOPs, refer to SOP No. CW-Q-S-002, "Writing a Standard Operating Procedure SOP" and laboratory SOP No. ST-QA-0035, "Preparation and Management of Standard Operating Procedures".

Forms, worksheets, work instructions and information are organized electronically by department in the QA folder on the network server. There is an index. Hard copies are kept in QA files. In order to develop a new form, worksheet or work instruction, the user submits a draft to the QA Department and technical manager for suggestions, approval and validation (where required) before use. Upon approval, QA personnel add the identifying control information to the document. That document is then provided to all applicable operational units (may include electronic access). Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

6.4 Obsolete Documents

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived as described in Section 14.

SECTION 7. SERVICE TO THE CLIENT

7.1 Overview

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and

does not necessarily “fit” into a standard laboratory service or product. It is the laboratory’s intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab’s capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and the laboratory’s capability to meet those requirements. Alternate test methods that are capable of meeting the clients’ requirements may be proposed by the lab. A review of the lab’s capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client’s requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory’s test methods are suitable to achieve these requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory’s equipment and personnel. As part of the review, the proposed turnaround time will be checked for feasibility.

Electronic or hard copy deliverable requirements are evaluated against the laboratory’s capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 8 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client’s requirements and the laboratory’s capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

7.2 Review Sequence and Key Personnel

Appropriate personnel will review the work request at each stage of evaluation. SOP ST-PM-0001, “Project Setup and Quote”, outlines the process at the TestAmerica St. Louis laboratory.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

For new, complex or large projects, the proposed contract is given to the Sales Directors, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in TestAmerica's Corporate SOP No. CA-L-P-002, Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below):

- Legal & Contracts Director
- General Manager
- The Laboratory Project Management Manager
- Laboratory and/or Corporate Technical Managers / Directors
- Laboratory and/or Corporate Information Technology Managers/Directors
- Account Executives
- Laboratory and/or Corporate Quality
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
- The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The Sales Director, Legal Contracts Director, Account Executive or local customer Service Manager or Project Manager then submits the final proposal to the client. In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

The Legal & Contracts Director maintains copies of all signed contracts. A copy is kept in the Project Management directory on the network server.

7.3 Documentation

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes

The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Account Executive. A copy of the contract and formal quote will be filed with the laboratory PM and the Laboratory Director.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps a phone log or e-mail chain of conversations with the client.

7.3.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, the laboratory assigns a PM to each client. It is the Project Manager's responsibility to ensure that project-specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements.

Project Manager's are the primary client contact and they ensure resources are available to meet project requirements. Although Project Manager's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources is sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, a "Client Requirement Memo" may be associated with each sample lot as a reminder of special sample receipt instructions and analytical requirements.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation may include letters, e-mails, variances and/or contract addendum.

Such changes are also communicated to the laboratory during production meetings. Such changes are updated to the Client Requirement Memo and are introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the PM or the individual laboratory Technical Manager. After the modification is implemented into the laboratory process, documentation of the modification is made in the case narrative of the data report(s).

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

7.4 Special Services

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all

client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 15 and 25).

Note: ISO 17025 states that a laboratory “shall afford clients or their representatives cooperation to clarify the client’s request”.

The laboratory’s standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assist client-specified third party data validators as specified in the client’s contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

7.5 Client Communication

Project managers are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Technical Managers/Directors are available to discuss any technical questions or concerns that the client may have.

7.6 Reporting

The laboratory works with our clients to produce any special communication reports required by the contract.

7.7 Client Surveys

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service. TestAmerica’s Sales and Marketing teams periodically develops lab and client specific surveys to assess client satisfaction.

SECTION 8. SUBCONTRACTING OF TESTS

8.1 Overview

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the TestAmerica laboratories. The phrase “work sharing” refers to internal transfers of samples between the TestAmerica laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities,

capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to TestAmerica's Corporate SOPs on Subcontracting Procedures (CA-L-S-002) and the Work Sharing Process (CA-C-S-001).

When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in NELAC/ISO 17025 and/or the client's Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-NELAC accreditation work where required.

For Department of Defense/Department of Energy projects the subcontractor and/or Work Share laboratories used must have an established and documented laboratory quality system that complies with DoD QSM/DOE QSAS requirements. The subcontractor and/or Work Share laboratories are evaluated following the procedures outlined below. The subcontractor and/or Work Share laboratory must receive project-specific approval from the DoD/DOE client before any samples are analyzed.

The DoD QSM requirements for subcontracting:

1. Subcontractor laboratories must have an established laboratory quality system that complies with the QSM.
2. Subcontractor laboratories must be accredited by DoD or its designated representatives.
3. Subcontractor laboratories must receive project-specific approval from the DoD client before any samples are analyzed.
4. Subcontractor laboratories are subject to project-specific, on-site assessments by the DoD client or their designated representatives.

The DOE QSAS has the following requirements for subcontracting:

"The laboratory shall not use any sub-tier laboratories or subclients (including those possessing the same or similar corporate name) for performance of work under this specification without written approval from the Procurement Representative. The laboratory using the sub-tier laboratory or sub-client shall document and is responsible for ensuring that such sub-client meets all of the requirements of this specification, including being available for client inspections and audits.

Some clients may not allow any subcontracting to third party (sub-tier) laboratories. If this is the case, then this will be specifically noted in the site-specific contracts via Contracts, Task Orders, Laboratory Delivery Orders, etc."

Project Managers (PM), Customer Service Managers (CSM), or Account Executives (AE) for the Export Lab are responsible for obtaining client approval prior to outsourcing any samples. The laboratory will advise the client of a subcontract or work sharing arrangement in writing and when possible approval from the client shall be retained in the project folder.

Note: In addition to the client, some regulating agencies (e.g. USDA), such as the DoD and DOE, require notification prior to placing such work.

8.2 Qualifying and Monitoring Subcontractors

Whenever a PM or Account Executive (AE) or Customer Service Manager (CSM) becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- The first priority is to attempt to place the work in a qualified TestAmerica laboratory;
- Firms specified by the client for the task (Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be as simple as placing a copy of an e-mail from the client in the project folder);
- Firms listed as pre-qualified and currently under a subcontract with TestAmerica: A listing of all approved subcontracting laboratories is available on the TestAmerica intranet site. Supporting documentation is maintained by corporate offices and by the TestAmerica laboratory originally requesting approval of the subcontract lab. Verify necessary accreditation, where applicable, (e.g., on the subcontractors, A2LA accreditation or State Certification).
- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses;
- NELAC accreditation laboratories.
- In addition, the firm must hold the appropriate certification to perform the work required.

With the exception of DoD and DOE programs noted above, all TestAmerica laboratories are pre-qualified for work sharing provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. (Corporate SOP No. CA-C-S-001, Work Sharing Process).

When the potential sub-contract laboratory has not been previously approved, Account Executives or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director. The Laboratory Director requests that the QA Manager begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CA-L-S-002, Subcontracting Procedures. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented).

8.2.1 Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to Corporate Contracts for formal contracting with the laboratory. They will add the lab to the approved list on the intranet site and notify the finance group for JD Edwards.

8.2.2 The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the

intranet site are known to meet minimal standards. TestAmerica does not certify laboratories. The subcontractor is on our approved list and can only be recommended to the extent that we would use them.

8.2.3 The status and performance of qualified subcontractors will be monitored periodically by the Corporate Contracts and/or Corporate Quality Departments. Any problems identified will be brought to the attention of TestAmerica's Corporate Finance or Corporate Quality personnel.

- Complaints shall be investigated. Documentation of the complaint, investigation and corrective action will be maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report.
- Information shall be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing will be retained on the intranet listing. CSO personnel will notify all TestAmerica laboratories, Corporate Quality and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all CSO personnel Laboratory Directors, QA Managers and Sales Personnel.

8.3 Oversight and Reporting

The PM must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which flow down the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and the Legal & Contracts Director can tailor the document or assist with negotiations, if needed. The PM (or EDS, AEs or CSM, etc.) responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented within the project records. For TestAmerica laboratories, certifications can be viewed on the company's TotalAccess Database.

The Sample Control department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

All subcontracted samples must be accompanied by a TestAmerica Chain of Custody (COC). A copy of the original COC sent by the client must be available in TALS for all samples workshared within TestAmerica. Client CoCs are only forwarded to external subcontractors when samples are shipped directly from the project site to the subcontractor lab. Under routine circumstances, client CoCs are not provided to external subcontractors.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-NELAC accreditation work must be identified in the subcontractor's report as appropriate. If NELAC accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratories EDD (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples.

Note: The results submitted by a TestAmerica work sharing laboratory may be transferred electronically and the results reported by the TestAmerica work sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

8.4 Contingency Planning

With the exception of DoD and DOE programs, the Laboratory Director may waive the full qualification of a subcontractor process temporarily to meet emergency needs; however, this decision & justification must be documented in the project files, and the 'Purchase Order Terms And Conditions For Subcontracted Laboratory Services' must be sent with the samples and Chain-of-Custody. In the event this provision is utilized, the laboratory (e.g., PM) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation requirements will still be applicable, but the subcontractor need not have signed a subcontract with TestAmerica at this time. The comprehensive approval process must then be initiated within 30 calendar days of subcontracting.

SECTION 9. PURCHASING SERVICES AND SUPPLIES

9.1 Overview

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with TestAmerica's Corporate Controlled Purchases Procedure, SOP No. CW-F-S-007.

Contracts will be signed in accordance with TestAmerica's Corporate Authorization Matrix Policy, Policy No. CW-F-P-002. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. Process details are available in TestAmerica's Corporate Procurement and Contracts Policy (Policy No. CW-F-P-004). RFP's allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

9.2 Glassware

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

9.3 Reagents, Standards & Supplies

Purchasing guidelines for equipment, consumables and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with TestAmerica's Corporate SOP on Solvent & Acid Lot Testing & Approval, SOP No. CA-Q-S-001, laboratory SOP ST-QA-0037, "Procurement of Quality Related Items" and ST-QA0002, "Standard and Reagent Preparation". Approval information for the solvents and acids tested under SOP CA-Q-S-001 is stored on the TestAmerica SharePoint, under Solvent Approvals. A master list of all tested materials, as well as the certificates of analysis for the materials, is stored in the same location.

9.3.1 Purchasing

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOPs.

The procedure for purchasing/ordering quality related items can be found in the laboratory SOP ST-QA-0037, "Procurement of Quality Related Items".

9.3.2 Receiving

It is the responsibility of the purchasing manager to receive the shipment. It is the responsibility of the analyst who ordered the materials to document the date materials were received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. This is documented through the addition of the received date and initials to the information present on the daily order log.

The purchasing manager verifies the lot numbers of received solvents and acids against the pre-approval lists. If a received material is listed as unapproved, or is not listed, it is sequestered and returned to the vendor. Alternatively, the laboratory may test the material for the intended use, and if it is acceptable, document the approval on the approval list. Records of any testing performed locally are maintained on the shared "public" folder on the computer network.

Materials may not be released for use in the laboratory until they have been inspected, verified as suitable for use, and the inspection/verification has been documented.

Safety Data Sheets (SDS) are available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

9.3.3 Specifications

Methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, analytical reagent grade will be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals and solvents unless noted otherwise by the manufacturer or by the reference source method. Chemicals/solvents should not be used past the manufacturer's or SOPs expiration date.

- An expiration date **cannot** be extended if the dry chemical/solvent is discolored or appears otherwise physically degraded, the dry chemical/solvent must be discarded.
- Radiochemical standards can be re-verified and a new expiration date applied. See SOP ST-QA-0002, "Standard and Reagent Preparation".

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. To prevent a tank from going to dryness, or introducing potential impurities, the pressure should be closely watched as it decreases to approximately 15% of the original reading, at which point it should be replaced. For example, a standard sized laboratory gas cylinder containing 3000 psig of gas should be replaced when it drops to approximately 500 psig. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a specific conductivity of less than 1- $\mu\text{mho/cm}$ (or specific resistivity of greater than 1.0 megohm-cm) at 25°C. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and appropriate Technical-Managers must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified "clean" by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased bottleware used for sampling must be certified clean and the certificates must be maintained. If uncertified sampling bottleware is purchased, all lots must be verified clean prior to use. This verification must be maintained.

Records of manufacturer's certification and traceability statements are maintained in electronic files on the network server. These records include date of receipt, lot number (when applicable), and expiration date (when applicable).

9.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Standards and reference materials are stored separately from samples. Radiochemical standards are stored in a controlled access cabinet. Storage conditions are per the Corporate Environmental Health & Safety Manual (Corp. Doc. No. CW-E-M-001) and method SOPs or manufacturer instructions.

9.4 Purchase of Equipment / Instruments / Software

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Laboratory Director. If they agree with the request, the procedures outlined in TestAmerica's Corporate Policy No. CA-T-P-001, Qualified Products List, is followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order.

Upon receipt of a new or used piece of equipment, an identification name is assigned and added to the equipment list. IT must also be notified so that they can synchronize the instrument for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 19). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department or QA Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is accessible to the laboratory.

9.5 Services

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 20. The need for service is determined by analysts and/or Technical Managers. The service providers that perform the services are approved by the Technical Manager.

Analytical balances are serviced and calibrated annually in accordance with SOP ST-QA-0005,. The calibration and maintenance services are performed on-site, and the balances are returned to use immediately following successful calibration. When the calibration certificates are received (usually within two weeks of the service), they are reviewed, and documentation of the review is filed with the certificates. If the calibration was unsuccessful, the balance is immediately removed from service and segregated pending either further maintenance or disposal.

Calibration services for support equipment such as thermometers, weight sets, autopipettors, etc, are obtained from vendors with current and valid ISO 17025 accreditation for calibration of the specific piece of equipment. Prior to utilizing the vendor's services, the vendor's accreditation status is verified. Once the equipment has been calibrated, the calibration certificates are reviewed by the QA department, and documentation of the review is filed with the calibration certificates. The equipment is then returned to service within the laboratory

9.6 Suppliers

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Corporate Finance documents on Vendor Selection (SOP No. CW-F-S-018) and Procurement & Contracts Policy (Policy No. CW-F-P-004). The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers/vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on

packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report.

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc. As deemed appropriate, the Vendor Performance Reports will be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the J.D. Edwards purchasing system.

9.6.1 New Vendor Procedure

TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form.

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Technical Director are consulted with vendor and product selection that have an impact on quality.

Figure 9-1.
Electronic Order Form

The screenshot shows the 'STL St. Louis - Online Order Form' application window. It features a menu bar (File, Edit, View, Insert, Format, Records, Tools, Window, Help) and a toolbar with various icons. The main window has tabs for 'Order Form', 'Catalog', 'Vendors', 'Notification', 'Reports', and 'Pending Approval'. The 'Order Form' tab is active, displaying a 'Locate Order' dropdown and buttons for 'New Order', 'Submit Order', 'View Order', 'New Item', and 'New Vendor'. Below these are 'Order Information' fields: 'Department', 'Ordered By', 'Supervisor', 'Vendor', 'Order Number', 'Date Needed', 'Date Submitted', and a 'Reason for Rush' dropdown. There is also a 'Notes' text area. A 'Purchasing Only' section includes 'J.D. Edwards Order #' and 'PO Number' fields. At the bottom, there are buttons for 'Admin Access', 'Export Order...', 'Refresh Data Sources', and a version string 'OOF version 1.15, updated 12/12/2006'. A status bar at the very bottom shows 'Form View' and 'Click here to begin'.

The screenshot shows the 'Add New Order' dialog box. It contains 'Order Information' fields: 'Ordered By' (a dropdown menu), 'Date Needed' (a date field with '11/21/2007'), 'Vendor' (a dropdown menu), and 'Reason for Rush' (a dropdown menu with 'N/A'). There is also a 'Rush' checkbox. At the bottom, there are 'Add Order' and 'Cancel' buttons.

SECTION 10. COMPLAINTS

10.1 Overview

The laboratory considers an effective client complaint handling processes to be of significant business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services (e.g., communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented in the laboratory's Validation Database.

10.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to SOP ST-QA-0036 "Non-conformance Memorandum (NCM)/Validation Request and Corrective Action Processes".

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and Documenting Complaints
- Complaint Investigation and Service Recovery
- Process Improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

10.3 Internal Complaints

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 12. In addition, Corporate Management, Sales and Marketing and IT may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 12.

10.4 Management Review

The number and nature of client complaints is reported by the QA Manager to the laboratory and QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 16).

SECTION 11. CONTROL OF NON-CONFORMING WORK

11.1 Overview

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 12).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the supervisor for resolution. The supervisor may elect to discuss it with the QA Manager or Technical Director or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratories corrective action system described in Section 12. This information can then be supplied to the client in the case narrative sent with the report.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 19. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Technical Manager Director and QA Manager, documented and included in the project folder. Deviations **must** also be noted on the final report with a statement that the compound is not reported in compliance with NELAC (or the analytical method) requirements and the reason. Data being reported to a non- NELAC state would need to note the change made to how the method is normally run.

11.2 Responsibilities and Authorities

Under certain circumstances, the Laboratory Director, a Technical Manager, or a member of the QA team may authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc.. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures. This information may also be documented in logbooks and/or data review checklists as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility Senior Management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, the QA Manager, and the Technical Managers. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an ECO (e.g., the VP-QA/EHS) and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, ECOs, VP of Operations and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

11.3 Evaluation of Significance and Actions Taken

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

Corporate SOP entitled Data Recalls (CW-Q-S-005) is the procedure to be followed when it is discovered that erroneous or biased data may have been reported to clients or regulatory agencies.

Corporate SOP entitled Internal Investigations (CW-L-S-002) is the procedure to be followed for investigation and correction of situations involved alleged incidents of misconduct or violation of the company's ethics policy.

Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in TestAmerica's Corporate SOP No. CW-Q-S-005.

11.4 Prevention of NonConforming Work

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. Monthly the QA Department evaluates

non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may need to be followed.

11.5 Method Suspension / Restriction (Stop Work Procedures)

In some cases, it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 11.2, Paragraph 5.

Prior to suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases, that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line.

The QA Manager will also initiate a corrective action report as described in Section 12 if one has not already been started. A copy of any meeting notes and agreed upon steps should be faxed or e-mailed by the laboratory to the appropriate General Manager and member of Corporate QA. This fax/e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (e.g., Project Management, Log-in, etc...). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Technical Manager, Technical Director, QA Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval is given by final signature on the completed corrective action report.

SECTION 12. CORRECTIVE ACTION

12.1 Overview

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using Non-Conformance Memos (NCM) and Validation Requests (refer to SOP ST-QA-0036).

For DOE, DoD and other programs where required, the client will be informed of proposed corrective actions.

12.2 General

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc...

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility(s) for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.
- Identify and track client complaints and provide resolution.

12.2.1 Non-Conformance Memo (NCM) - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non-matrix related)
- Isolated reporting / calculation errors
- Discrepancies in materials / goods received vs. manufacturer packing slips.
- Questionable trends that are found in the review of NCMs.
- Issues found while reviewing NCMs that warrant further investigation.
- Internal and external audit findings
- Systematic reporting / calculation errors
- Client complaints
- Data recall investigations
- Identified poor process or method performance trends
- Excessive revised reports

Health and Safety violations are documented in the EH&S Quarterly Inspection Reports

This will provide background documentation to enable root cause analysis and preventive action.

12.3 Closed Loop Corrective Action Process

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

12.3.1 Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. An NCM or Validation Request must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 12-1 provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Technical Manager, Laboratory Director, or QA Manager (or QA designee) is consulted.

12.3.2 Selection and Implementation of Corrective Actions

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The NCM or Validation Request is used for this documentation.

12.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness. Corporate SOP Root Cause Analysis (No. CA-Q-S-009) describes the procedure.

Systematically analyze and document the root causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the Root Cause data from these incidents to

identify Root Causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with problem and ask why this event occurred. Brainstorm the root causes of failures; for example, by asking why events occurred or conditions existed; and then why the cause occurred 5 consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

Root cause analysis does not mean the investigation is over. Look at technique, or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed, and continue to plague the laboratory or operation.

12.3.4 Monitoring of the Corrective Actions

- The Technical Manager and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Technical Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each NCM and Validation Request is entered into a database for tracking purposes and a monthly summary of all corrective actions may be printed out for review to aid in ensuring that the corrective actions have taken effect.
- The QA Manager reviews monthly NCMs and Validation Requests for trends. Highlights are included in the QA monthly report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- TestAmerica laboratories began using the Incident/Corrective Action Tracker (iCAT) database developed by the company in 2015. (Previously, a local database served this purpose.) An incident is an event triggering the need for one or more corrective actions as distinct from a corrective action, a potential deficiency stemming from an incident that requires investigation and possibly fixing. The database is independent of TALS, available to all local and corporate managers, and capable of notifying and tracking multiple corrective actions per event, dates, and personnel. iCAT allows associated document upload, categorization (such as, external/internal audit, client service concerns, data quality issues, proficiency testing, etc.), and trend analysis.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

12.3.5 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.

- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

(Also refer to Section 15.1.4, Special Audits.)

12.4 Technical Corrective Actions

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 11). The documentation of these procedures is through the use of an NCM or Validation Request.

Table 12-1 includes *examples* of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs. The laboratory may also maintain Work Instructions on these items that are available upon request.

Table 12-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions, QAM Sections 19 and 20. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented.

12.5 Basic Corrections

When mistakes occur in records, each mistake shall be crossed-out and not obliterated (e.g. no white-out), and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original “uncorrected” file must be maintained intact and a second “corrected” file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

Table 12-1. Example – General Corrective Action Procedures

| QC Activity (Individual Responsible for Initiation/Assessment) | Acceptance Criteria | Recommended Corrective Action |
|--|--|---|
| Initial Instrument Blank (Analyst) | - Instrument response < RL. | - Prepare another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc... |
| Initial Calibration Standards (Analyst, Technical Manager(s)) | - Correlation coefficient > 0.99 or standard concentration value. - % Recovery within acceptance range. - See details in Method SOP. | - Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument. |
| Independent Calibration Verification (Second Source) (Analyst, Technical Manager(s)) | - % Recovery within control limits. | - Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument. |
| Continuing Calibration Standards (Analyst, Data Reviewer) | % Recovery within control limits documented in QC Browser database | - reanalyze standard -if still unacceptable, recalibrate and rerun affected samples |
| Matrix Spike / Matrix Spike Duplicate (MS/MSD) (Analyst, Data Reviewer) | - % Recovery within limits documented in the LIMS | - If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. - If the LCS is within acceptable limits the batch is acceptable. - The results of the duplicates, matrix spikes and the LCS are reported with the data set. - For matrix spike or duplicate results outside criteria the data for that sample shall be reported with qualifiers. |

| QC Activity (Individual Responsible for Initiation/Assessment) | Acceptance Criteria | Recommended Corrective Action |
|--|--|---|
| Laboratory Control Sample (LCS) (Analyst, Data Reviewer) | - % Recovery within limits specified in the LIMS | - Batch must be re-prepared and re-analyzed. This includes any allowable marginal exceedance. When not using marginal exceedances, the following exceptions apply: 1) when the acceptance criteria for the positive control are exceeded high (i.e., high bias) and there are associated samples that are non-detects, then those non-detects may be reported with data qualifying codes; 2) when the acceptance criteria for the positive control are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level with data qualifying codes. Note: If there is insufficient sample or the holding time cannot be met, contact client and report with flags. |
| Surrogates (Analyst, Data Reviewer) | - % Recovery within limits of method or within three standard deviations of the historical mean. | - Individual sample must be repeated. Place comment in LIMS. - Surrogate results outside criteria shall be reported with qualifiers. |
| Method Blank (MB) (Analyst, Data Reviewer) | < Reporting Limit ¹ | - Reanalyze blank. - If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results. - Qualify the result(s) if the concentration of a targeted analyte in the MB is at or above the reporting limit AND is > 1/10 of the amount measured in the sample. |
| Proficiency Testing (PT) Samples (QA Manager, Technical Manager(s)) | - Criteria supplied by PT Supplier. | - Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected. |
| Internal / External Audits (QA Manager, Technical Manager(s) Laboratory Director) | - Defined in Quality System documentation such as SOPs, QAM, etc... | - Non-conformances must be investigated through Validation system and necessary corrections must be made. |

| QC Activity (Individual Responsible for Initiation/Assessment) | Acceptance Criteria | Recommended Corrective Action |
|---|---|---|
| Reporting / Calculation Errors (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Technical Managers, QA Manager, Corporate QA, Corporate Management) | - SOP CW-Q-S-005, Data Recall | - Corrective action is determined by type of error. Follow the procedures in SOP CW-L-S-002. |
| Client Complaints (Project Managers, Lab Director/Manager, Sales and Marketing) | - | - Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow-up must be performed on the reasons the address was incorrect (e.g., database needs to be updated). |
| QA Monthly Report (Refer to Section 16 for an example) (QA Manager, Lab Director/Manager, Technical Manager(s)) | - QAM, SOPs. | - Corrective action is determined by the type of issue. For example, NCMs and Validations for the month are reviewed and possible trends are investigated. |
| Health and Safety Violation (Safety Officer, Lab Director/Manager, Technical Manager(s)) | - Environmental Health and Safety (EHS) Manual. | - Non-conformance is investigated and corrected |

Note:

1. Except as noted below for certain compounds, the method blank should be below the detection limit. Concentrations up to five times the reporting limit will be allowed for the ubiquitous laboratory and reagent contaminants: methylene chloride, toluene, acetone, 2-butanone and phthalates provided they appear in similar levels in the reagent blank and samples. This allowance presumes that the detection limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur.

SECTION 13. PREVENTIVE ACTION / IMPROVEMENT

13.1 Overview

The laboratory's preventive action programs improve or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive and continuous process of improvement activities that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, customer service and client satisfaction can be improved through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered through any of the following:

- review of the monthly QA Metrics Report,
- trending NCMs,
- review of control charts and QC results,
- trending proficiency testing (PT) results,
- performance of management system reviews,
- trending client complaints,
- review of processing operations, or
- staff observations.

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc... These metrics are used in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

The laboratory's corrective action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action provides a valuable mechanism for identifying preventive action opportunities.

13.1.1 The following elements are part of a preventive action system:

- Identification of an opportunity for preventive action.
- Process for the preventive action.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action.
- Evaluation of the plan using the defined measurements.

- Verification of the effectiveness of the preventive action.
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action. Documentation of Preventive Action is incorporated into the monthly QA reports, corrective action process and management review.

13.1.2 Any Preventive Actions undertaken or attempted shall be taken into account during the annual Management Systems Review (Section 16). A highly detailed report is not required; however, a summary of successes and failures within the preventive action program is sufficient to provide management with a measurement for evaluation.

13.2 Management of Change

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of changes covered under this system include: Facility Changes, Major Accreditation Changes, Addition or Deletion to Division's Capabilities or Instrumentation, Key Personnel Changes, Laboratory Information Management System (LIMS) changes.

TestAmerica St. Louis uses a series of spreadsheets and/or databases to track changes to major capabilities (e.g. equipment, accreditations, etc.). An equipment list is maintained by the QA department. Accreditations are maintained via the OASIS Total Access program on the TestAmerica intranet site.

SECTION 14. CONTROL OF RECORDS

The laboratory maintains a records management system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued.

14.1 Overview

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 14-1. Quality records are maintained by the QA department electronically, which are backed up as part of the regular laboratory backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by the Data Reporting Group (raw data, analytical records, lab reports) and the QA Department (logbooks, standards, certificates, Quality documents).

Table 14-1. Record Index¹

| | <u>Record Types¹:</u> | <u>Retention Time:</u> |
|-------------------------------|---|---|
| Technical Records | <ul style="list-style-type: none"> - Raw Data - Logbooks² - Standards - Certificates - Analytical Records - MDLs/IDLs/DOCs - Lab Reports | 5 Years from analytical report issue* |
| Official Documents | <ul style="list-style-type: none"> - Quality Assurance Manual (QAM) - Work Instructions - Policies - SOPs - Policy Memorandums - Manuals | 5 Years from document retirement date* |
| QA Records | <ul style="list-style-type: none"> - Internal & External Audits/Responses - Certifications - Corrective/Preventive Actions - Management Reviews - Method & Software Validation / Verification Data - Data Investigation | 5 Years from archival* <u>Data Investigation:</u> 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation) |
| Project Records | <ul style="list-style-type: none"> - Sample Receipt & COC Documents - Contracts and Amendments - Correspondence - QAPP - SAP - Telephone Logbooks - Lab Reports | 5 Years from analytical report issue* |
| Administrative Records | Finance and Accounting | 10 years |

| | <u>Record Types</u> ¹ : | <u>Retention Time:</u> |
|--|--|-------------------------------|
| | EH&S Manual, Permits | 7 years |
| | Disposal Records | Indefinitely |
| | Employee Handbook | Indefinitely |
| | Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics) | Refer to HR Manual |
| | Administrative Policies Technical Training Records | 7 years |

¹ Record Types encompass hardcopy and electronic records.

² Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).

* Exceptions listed in Table 14-2.

14.1.1 All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or an offsite location that provides a suitable environment to prevent damage or deterioration and to prevent loss. All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees and shall be documented with an access log. Whether on-site or off-site storage is used, logs are maintained in each storage box to note removal and return of records. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 14-2 have lengthier retention requirements and are subject to the requirements in Section 14.1.2

14.1.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data. For projects/programs that require a retention time longer than five years, the Project Manager informs the Reporting Group of the extended storage requirement. The Data Reporting Group tracks these requirements.

Table 14-2. Example: Special Record Retention Requirements

| Program | ¹Retention Requirement |
|---|---|
| Drinking Water – All States | 5 years (project records) 10 years - Radiochemistry (project records) |
| Drinking Water Lead and Copper Rule | 12 years (project records) |
| Commonwealth of MA – All environmental data 310 CMR 42.14 | 10 years |
| FIFRA – 40 CFR Part 160 | Retain for life of research or marketing permit for pesticides regulated by EPA |
| Housing and Urban Development (HUD) Environmental Lead Testing | 10 years |
| Alaska | 10 years |
| Louisiana – All | 10 years |
| Michigan Department of Environmental Quality – all environmental data | 10 years |
| Navy Facilities Engineering Service Center (NFESC) | 10 years |
| NY Potable Water NYCRR Part 55-2 | 10 years |
| Ohio VAP | 10 years and State contacted prior to disposal |
| TSCA - 40 CFR Part 792 | 10 years after publication of final test rule or negotiated test agreement |

¹Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

14.1.3 The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, refer to Section 19.15.1 for more information.

14.1.4 The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data. The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the COC is stored with the laboratory report. The chain of custody would indicate the name of the sampler. A log of names, initials and signatures for all individuals responsible for signing or initialing laboratory records is maintained in the Human Resources Department. If any sampling notes are provided with a work order, they are kept with the laboratory report.
- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.

- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set). Instrument data is stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. Run logs are maintained for each instrument or method; a copy of each day's run long or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in the Reagent Log in the LIMS and relevant printouts can be included in the data packages as needed.
- Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.
- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".
- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.
- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned.
- Also refer to Section 19.15.1 'Computer and Electronic Data Related Requirements'.

14.2 Technical and Analytical Records

14.2.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for the performance of each analysis and reviewing results.

14.2.2 Observations, data and calculations are recorded real-time and are identifiable to the specific task.

14.2.3 Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails. The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- laboratory sample ID code;
- Date of analysis; Time of Analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times,

incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a bench sheet.

- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs or posted on the instrument.
- analysis type;
- all manual calculations and manual integrations;
- analyst's or operator's initials/signature;
- sample preparation including cleanup, separation protocols, incubation periods or subculture, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;
- test results;
- standard and reagent origin, receipt, preparation, and use;
- calibration criteria, frequency and acceptance criteria;
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;
- quality control protocols and assessment;
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- Method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats.

14.3 Laboratory Support Activities

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
- copies of final reports;
- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

14.3.1 Sample Handling Records

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.
- Chain of Custody protocols required by DOE and DoD

14.4 Administrative Records

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 14-1.

14.5 Records Management, Storage and Disposal

All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access.

The laboratory has a record management system (a.k.a., document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are numbered sequentially. Within each logbook, pages are sequentially numbered. All data are recorded sequentially within a series of sequential notebooks. Bench sheets are filed sequentially. Standards are maintained in the Reagents Log Program in LIMS. Records are considered archived when moved off-site or are so labeled. Dual storage of these records is maintained by the IT Department during its daily and weekly back-ups of the laboratory network. These back-up tapes are stored off-site.

14.5.1 Transfer of Ownership

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer

agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

14.5.2 Records Disposal

Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration. (Refer to Tables 14-1 and 14-2).

Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

If a third party Records Management Company is hired to dispose of records, a "Certificate of Destruction" is required.

SECTION 15. AUDITS

15.1 Internal Audits

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and, when requested, to corporate management.

Audits are conducted and documented as described in the TestAmerica Corporate SOP on performing Internal Auditing, SOP No. CA-Q-S-003. The types and frequency of routine internal audits are described in Table 15-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 15-1. Types of Internal Audits and Frequency

| Description | Performed by | Frequency |
|--------------------------------------|---|--|
| Quality Systems Audits | QA Department, QA approved designee, or Corporate QA | All areas of the laboratory annually |
| Method Audits QA Technical Audits | Joint responsibility: a) QA Manager or designee b) Technical Manager or Designee (Refer to CA-Q-S-003) | Methods Audits Frequency: 50% of methods annually |

| Description | Performed by | Frequency |
|-----------------------|---|--|
| SOP Method Compliance | Joint responsibility: c) QA Manager or designee d) Technical Manager or Designee (Refer to CA-Q-S-003) | SOP Compliance Review Frequency: • Every 2 years • 100% of SOPs annually (DoD Labs) |
| Special | QA Department or Designee | Surveillance or spot checks performed as needed, e.g., to confirm corrective actions from other audits. |
| Performance Testing | Analysts with QA oversight | Two successful per year for each TNI field of testing or as dictated by applicable regulatory requirements |

15.1.1 Audit Planning/Reporting

An audit plan is developed to identify the scope of the audit, the time frame, the personnel involved, the activities to be included, reference documents (i.e. Methods, SOPs, Checklists, and Client Requirement Memos) and persons to be notified of results. The audit team is selected prior to the audit. The size of the team is dependent on the scope of the audit. The lead auditor organizes and directs the audit. The audit report is issued to the appropriate departments by the lead auditor in hardcopy or electronically. The audit report is signed or otherwise endorsed by the Lead Auditor. The report describes the scope of the audit, identified auditors and persons contacted, summarizes results and describes all non-conformances found.

15.1.2 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, TestAmerica's Data Integrity and Ethics Policies, TNI quality systems, client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed for effectiveness & sustainability. The audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

15.1.3 QA Technical Audits

QA technical audits assess data authenticity and analyst integrity. These audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, electronic audit miner programs (e.g., Chrom AuditMiner) are used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period. All analysts should be reviewed over the course of a two year period through at least one QA Technical Audit

15.1.4 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Technical Manager or qualified designee at least every two years. It is also recommended that the work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products will be performed within 3 months of completing the documented training.

15.1.5 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

15.1.6 Performance Testing

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Drinking Water, Non-potable Water, Soil and Radiochemistry.

It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

15.2 External Audits

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

15.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as “a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment.” When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as “trade secret”, “proprietary” or “company confidential”. Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2009 TNI standards.

15.3 Audit Findings

Audit findings are documented using the corrective action process and database. The laboratory’s corrective action responses for both types of audits may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must be set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Technical Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory’s test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24-hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

SECTION 16. MANAGEMENT REVIEWS

16.1 Quality Assurance Report

A comprehensive QA Report shall be prepared each month by the laboratory’s QA Department and forwarded to the Laboratory Director, their Quality Director as well as the General Manager. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, General Manager or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Directors prepare a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and General Managers.

16.2 Annual Management Review

The senior lab management team (Laboratory Director, Technical Director, Technical Managers, QA Manager, EH&S Manager and Radiation Safety Officer) conducts a review annually of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining goals, objectives and action items that feed into the laboratory planning system. Corporate Operations and Corporate QA personnel may be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that is related to the LIMS. The laboratory will summarize any critical findings that cannot be solved by the lab and report them to Corporate IT.

This management systems review (Corporate SOP No. CW-Q-S-004 & Work Instruction No. CW-Q-WI-003) uses information generated during the preceding year to assess the “big picture” by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective; therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports issues.
- Laboratory QA Metrics
- Internal and External audit outcomes & corrective actions
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.
- Minutes from prior senior lab management meetings. Issues that may be raised from these meetings include:
 - Adequacy of staff, equipment and facility resources.
 - Adequacy of policies and procedures.
 - Future plans for resources and testing capability and capacity.
 - Changes in the volume and type of work
- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.
- Laboratory health and safety issues
- Radioactive materials management issues

A report is generated by the QA Manager and management. The report is distributed to the appropriate General Manager and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes].

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual. Quality system changes and improvements are incorporated into the laboratory's yearly goals.

16.3 Potential Integrity Related Managerial Reviews

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. TestAmerica's Corporate Internal Investigations SOP shall be followed (SOP No. CW-L-S-002). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica's President and CEO, Executive VP of Operations, VP of Client & Technical Services, VPs of Operations and Quality Directors receive a monthly report from the VP-QA/EHS summarizing any current data integrity or data recall investigations. The VPs of Operations are also made aware of progress on these issues for their specific labs.

SECTION 17. PERSONNEL

17.1 Overview

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

Management is responsible for authorizing specific personnel to perform specific tests (i.e. environmental testing, issue reports, interpret data, operate equipment).

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

The laboratory ensures that all personnel, including part time, temporary, contracted and administrative personnel, are trained in basic laboratory QA and safety programs.

Personnel dealing with sample receipt, radioactive waste management and materials shipping are trained in waste management, shipping and handling, and hazardous and/or radioactive materials control as appropriate.

17.2 Education and Experience Requirements for Technical Personnel

Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are located on the TestAmerica intranet site's Human Resources web-page (Also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, colony counting, aseptic or quantitation techniques, etc., are also considered).

As a general rule for analytical staff:

| Specialty | Education | Experience |
|--|---|--|
| Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses | H.S. Diploma | On the job training (OJT) |
| CVAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC | A college degree in an applied science or 2 years of college and at least 1 year of college chemistry | Or 2 years prior analytical experience is required |

| Specialty | Education | Experience |
|---|--|---|
| ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS | A college degree in an applied science or 2 years of college chemistry | or 5 years of prior analytical experience |
| Spectra Interpretation | A college degree in an applied science or 2 years of college chemistry | And 2 years relevant experience Or 5 years of prior analytical experience |
| Technical Managers – <u>General</u> | Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience | And 2 years experience in environmental analysis of representative analytes for which they will oversee |
| Technical Managers – <u>Wet Chem</u> only (no advanced instrumentation) | Associates degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry | And 2 years relevant experience |

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewers or Technical Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

17.3 Training

The laboratory is committed to furthering the professional and technical development of employees at all levels. See the laboratory SOP ST-QA-0044 Training for additional information.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

| Required Training | Time Frame | Employee Type |
|-------------------------------|-------------------|-------------------|
| Environmental Health & Safety | Prior to lab work | All |
| Ethics – New Hires | 1 week of hire | All |
| Ethics – Comprehensive | 90 days of hire | All |
| Data Integrity | 30 days of hire | Technical and PMs |
| Quality Assurance | 90 days of hire | All |

| Required Training | Time Frame | Employee Type |
|---|--|---------------|
| Ethics – Comprehensive Refresher | Annually | All |
| Computer Security Awareness | Annually | All |
| Initial Demonstration of Capability (DOC) | Prior to unsupervised method performance | Technical |

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to “Demonstration of Capability” in Section 19.

The following documentation must be on file at the laboratory for each employee:

- Ethics Training documentation
- Signed Ethics agreement
- Signed Confidentiality agreement
- TNI statement of qualification
- Copy of degree, if applicable
- New Employee Orientation checklist
- Safety Orientation checklist

In addition to items listed above, the following documentation is also included in the employee training record:

- Department training checklist
- Demonstration of Capability (IDOC/DOC)
- Manual Integration training, if applicable
- Annual evidence of continuing DOC (may be successful analysis of a blind sample on the specific test method, or a similar method or four successful LCS analyses.
- Specialty training as applicable

The training of technical staff is kept up to date by:

- Each employee must have documentation filed with the QA department that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics is maintained in their training file.
- Documentation of proficiency (refer to Section 19).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.

- Human Resources maintain documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics violations). This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analyst's knowledge to refer to QA Manual for quality issues.
- Analysts following SOPs, i.e., practice match SOPs.
- Analysts regularly communicate to supervisors and QA if SOPs need revision, rather than waiting for auditors to find problems.

17.4 Data Integrity and Ethics Training Program

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within 1 week of hire followed by technical data integrity training within 30 days, comprehensive training within 90 days, and quarterly refreshers for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times; TestAmerica has established a Corporate Ethics Policy (Policy No. CW-L-P-004) and an Ethics Statement. All initial training is documented by signature on the signed Ethics Statement demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity. The Ethics Statement is re-signed annually.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts, and for that reason, TestAmerica has a Zero Tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.

- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

SECTION 18. ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

18.1 Overview

The laboratory is a 52,000 ft² secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc., OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, radiological sample analysis, and administrative functions.

18.2 Environment

Laboratory accommodation, test areas, energy sources and lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

18.3 Work Areas

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.
- Separate high and low level radiochemical preparation areas

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory. Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

18.4 Floor Plan

A floor plan can be found in [Appendix 2](#).

18.5 Building Security

Building keys are distributed to management as necessary. The Human Resources Manager maintains a list of all employees who have been issued keys. Electronic “swipe” cards are issued to all laboratory employees.

All visitors to the laboratory enter through the main entrance and sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed. Visitors (with the exception of company employees) are given a visitor's badge and are escorted by laboratory personnel at all times. Vendors may be issued badges which state that escorts are not required. Visitors and vendors must sign out before leaving the premises.

Entry via the warehouse dock area is permitted for client sample delivery or material supply delivery, without Visitor Log sign-in. The Sample Control Department is responsible for the proper escorting of these visitors.

Vendors issued electronic swipe cards are not required to sign in or out. Visitors from other TestAmerica facilities, while required to sign the Visitor's log, may not require visitor badges.

At the laboratory's discretion, visitors may be asked to show photo identification.

SECTION 19. TEST METHODS AND METHOD VALIDATION

19.1 Overview

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

19.2 Standard Operating Procedures (SOPS)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.

- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP entitled 'Writing a Standard Operating Procedure', No. CW-Q-S-002 and the laboratory's SOP ST-QA-0035, "Preparation and Management of Standard Operating Procedures".
- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water and DoD SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.
- A listing of TestAmerica St. Louis' SOPs is included in [appendix 6](#).

19.3 Laboratory Methods Manual

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

19.4 Selection of Methods

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- Prescribed Procedures for Measurement of Radioactivity in Drinking Water, EPA-600/4-80-032, August 1980.
- Eastern Environmental Radiation Facility Radiochemistry Procedures Manual, EPA, PB84-215581, June 1984.
- HASL-300 28th Edition, Environmental Measurements Laboratory (EML), 1997.
- Method 1664, Revision A: N-Hexane Extractable Material (HEM: Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM): Non-polar Material by Extraction and Gravimetry, EPA-821-R-98-002, February 1999
- Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. Revised as of July 1, 1995, Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)
- Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.
- Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.
- Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.
- Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods).
- Standard Methods for the Examination of Water and Wastewater, 18th/19th/20th/ on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.
- Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008, Final Update V, August 2015.
- Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.
- Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly perform the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

A demonstration of capability is performed whenever there is a change in instrument type (e.g., new instrumentation), method or personnel.

The initial demonstration of capability must be thoroughly documented and approved by the Technical Manager and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures.

The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

For tasks where spiking is not possible (prep techniques including but not limited to compositing, drying and grinding, sub-sampling) the initial demonstration of capability is documented in the analysts training record by the analyst and supervisor signing off on the relevant SOP on the department training checklist. The yearly review and the analyst's acknowledgement of revisions to the SOP serve as the continuing demonstration of capability.

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted.

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

19.4.3.1 The spiking standard used must be prepared independently from those used in instrument calibration.

19.4.3.2 The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP.

19.4.3.3 At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).

19.4.3.4 Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.

19.4.3.5 When it is not possible to determine the mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the Method SOP.

19.4.3.6 Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

19.4.3.7 When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:

- Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 19.4.3.3 above.
- Beginning with 19.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, may confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 19.4.3.1 above.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

A certification statement (see Figure 19-1) shall be used to document the completion of each initial and continuing demonstration of capability. A copy of the certification is archived in the analyst's training folder.

19.5 Laboratory Developed Methods and Non-Standard Methods

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

19.6 Validation of Methods

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The validation process may include one, or a combination of the following: calibration using known reference standards, comparison of results achieved with other methods, PT samples, etc. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

19.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

19.6.1.1 Determination of Method Selectivity

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

19.6.1.2 Determination of Method Sensitivity

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed.

19.6.1.3 Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

19.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 Determination of Range

Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

19.6.1.7 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in a SOP, a SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

19.6.1.8 Continued Demonstration of Method Performance

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

19.7 Method Detection Limits (MDL) / Limits of Detection (LOD)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value can be differentiated from blanks. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements. Generally, the analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL.

Refer to the Corporate SOP No. CA-Q-S-006 or the laboratory's SOP No. ST-QA-0016 "MDL/IDL, LOD/LOQ Determination", for details on the laboratory's MDL process.

19.8 Minimum Detectable Activity (MDA)/Minimum Detectable Concentration (MDC)

For radiochemical analyses, the MDA/MDC is determined based on normal factors and conditions which influence measurement. The MDA/MDC is used to evaluate the capability of a method relative to the required RLs. Sample size, count duration, tracer recovery, detector background and detector efficiency all contribute to determining the sample's MDA/MDC.

The Minimum Detectable Concentration (MDC) for a radionuclide by radiochemical measurement is determined from the blank/background variability associated with the appropriate detector, the detector efficiency, sample aliquot size and chemical yield. The background variability is proportional to the sample count time.

NOTE: The background variability is based on the analytical test and derived by: 1) using sample specific parameters, or 2) process blank specific parameters, or 3) by averaging the multiple MDCs derived in 1 or 2.

Matrix material is used whenever possible and is of a similar composition as the client samples.

The MDC is calculated for individual samples (depending on counting technique) using the formulas provided in [Appendix 4](#). The MDC is expected to be less than the client required detection limit. Cesium-137 is the MDC analyte of interest for gamma evaluation.

If the sample MDC is greater than the client required detection limit (CRDL) or reporting limit (RL), the Data Reviewer shall examine the sample volume/weight, counting time, tracer yield and/or other relevant factors. The Data Reviewer shall decide the corrective action which may include reanalysis, recounting or data acceptance and document per laboratory procedure.

19.9 Instrument Detection Limits (IDL)

The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like the MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 times the absolute value of the standard deviation.

If IDL is > than the MDL, it may be used as the reported MDL.

19.10 Verification of Detection and Reporting Limits

Once the MDL is determined, it must be verified on each instrument used for the given method. TestAmerica defines the DoD QSM Detection Limit (DL) as being equal to the MDL. TestAmerica also defines the DoD QSM Limit of Detection (LOD) as being equal to the lowest concentration standard that successfully verifies the MDL, also referred to as the MDLV standard. MDL and MDLV standards are extracted/digested and analyzed through the entire analytical process. The MDL and MDLV determinations do not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDLV

standard is not successful, then the laboratory will redevelop their MDL or perform and pass two consecutive MDLVs at a higher concentration and set the LOD at the higher concentration. Initial and quarterly verification is required for all methods listed in the laboratory's DoD ELAP Scope of Accreditation. Refer to the laboratory SOP ST-QA-0016, "MDL/IDL, LOD/LOQ Determination", for further details.

The laboratory quantitation limit is equivalent to the DoD Limit of Quantitation (LOQ), which is at a concentration equal to or greater than the lowest non-zero calibration standard. The DoD QSM requires the laboratory to perform an initial characterization of the bias and precision at the LOQ and quarterly LOQ verifications thereafter. If the quarterly verification results are not consistent with three-standard deviation confidence limits established initially, then the bias and precision will be reevaluated and clients contacted for any on-going projects where required. For DoD projects, TestAmerica makes a distinction between the Reporting Limit (RL) and the LOQ. The RL is a level at or above the LOQ that is used for specific project reporting purposes, as agreed to between the laboratory and the client. The RL cannot be lower than the LOQ concentration, but may be higher.

19.11 Retention Time Windows

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specific in the reference method, each analyte will have a specific time of elution from the column to the detector. This is known as the analytes retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept with the files associated with an instrument for later quantitation of the analytes. Complete details are available in the laboratory SOPs.

19.12 Evaluation of Selectivity

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, spectrochemical, fluorescence profiles, co-precipitation evaluations and specific electrode response factors.

19.13 Estimation of Uncertainty of Measurement

19.13.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as human factors, adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty": the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor $k=2$.

19.13.2 Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

19.13.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

19.13.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent uncertainties at approximately the 99% confidence level with a coverage factor of $k = 3$. As an example, for a reported result of 1.0 mg/L with a LCS recovery range of 50 to 150%, the estimated uncertainty in the result would be 1.0 ± 0.5 mg/L. This approach may be used for chemical analyses. For radiochemical uncertainty determination, see the calculations in [Appendix 4](#).

19.13.5 In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g., 524.2, 525, etc.) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

19.14 Sample Reanalysis Guidelines

Because there is a certain level of uncertainty with any analytical measurement, a sample re-preparation (where appropriate) and subsequent analysis (hereafter referred to as 'reanalysis') may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. (Client specific Contractual Terms & Conditions for reanalysis protocols may supersede the following items).

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within ± 1 reporting limit for samples $\leq 5x$ the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.
- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.

- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.
- Due to the potential for increased variability, reanalysis may not be applicable to Non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Area Supervisor or Laboratory Director if unsure.

19.15 Control of Data

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

19.15.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. More detail is outlined in laboratory SOPs ST-IS-0001 "Software Change Management", ST-IS-0002, "Software Testing, Verification and Validation", and ST-IS-0003, "Information Systems". The laboratory is currently running TALS which is a custom in-house developed laboratory information management system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes an industry standard relational database platform. It is referred to as Database for the remainder of this section.

19.15.1.1 Maintain the Database Integrity: Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use. Cells containing calculations must be lock-protected and controlled.
- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails and controlled access.

19.15.1.2 Ensure Information Availability: Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, and secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

19.15.1.3 Maintain Confidentiality: Ensure data confidentiality through physical access controls such as password protection or website access approval.

19.15.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The

analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to updating the data in LIMS. The spreadsheets, or any other type of applicable documents, are signed by both the analyst and second level reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP No. CA-Q-S-002, *Acceptable Manual Integration Practices* and the laboratory SOP ST-QA-0040, "Manual Integration Procedure".

Analytical results are reduced to the appropriate concentration units as specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

- 19.15.2.1** All raw data must be retained in the reporting departments archive files. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (i.e. month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.
- 19.15.2.2** In general, concentration results are reported in milligrams per liter (mg/L) or picocuries per liter (pCi/L) or micrograms per liter (µg/L) for liquids and milligrams per kilogram (mg/kg), micrograms per kilogram (µg/kg) or picocuries per gram (pCi/g) for solids. For values greater than 10,000 mg/L, results can be reported in percent, i.e., 10,000 mg/L = 1%.
- 19.15.2.3** In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant figures. In general, results are reported to 2 significant figures on the final report.
- 19.15.2.4** For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS System, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte.
- 19.15.2.5** The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst reviews what has been entered to check for errors. If printed, the printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. Where possible, the data file is stored in a monthly folder

on the instrument computer; periodically, this file is transferred to the server and, eventually, to a tape file. For instruments without the capability of file storage the data is scanned to a pdf file and archived.

19.15.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 12.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Logbooks have sequentially numbered pages.
- Unused portions of pages must be "Z'd" out, signed and dated.
- Worksheets are created with the approval of the QA Manager or Technical Manager at the facility. The QA Department controls all worksheets following the procedures in Section 6.

19.15.4 Review / Verification Procedures

Data review procedures are out lined in SOP ST-PM-0004, "Data Review, Verification and Reporting" to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP discussing Manual Integrations to ensure the authenticity of the data (ST-QA-0040). The general review concepts are discussed below, more specific information can be found in the SOPs.

19.15.4.1 The data review process at the laboratory starts at the Sample Control level. Sample Control personnel review chain-of-custody forms and input the sample information and required analyses into LIMS. The Sample Control Supervisor, or designee, reviews the transcription of the chain-of-custody forms and the inputted information. The Project Managers perform final review of the chain-of-custody forms and inputted information.

19.15.4.2 The next level of data review occurs with the Analysts. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The Analysts transfer the data into the LIMS and add/review data qualifiers if applicable. To ensure data compliance, a different analyst performs a second level of review. Second level review is accomplished by checking reported results against raw data and evaluating the results for accuracy. During the second level review, blank runs, QA/QC check results, initial and continuing calibration results, laboratory control samples, sample data, qualifiers and spike information are evaluated. Where calibration is not required on a daily basis, secondary review of the initial calibration results may be conducted at the time of calibration. One hundred percent of all manual integrations are reviewed. The review is documented on the chromatogram by the analyst responsible for the integration and on the Second Review Checklist by the peer reviewer. Manual integrations are also periodically

electronically reviewed utilizing auditing software to help ensure compliance to ethics and manual integration policies. Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range

19.15.4.3 Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Assurance Director/Manager, Technical Manager, or Supervisor for further investigation. Corrective action is initiated whenever necessary.

19.15.4.4 The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is created for the client.

19.15.4.5 As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that chemical relationships are evaluated, COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met.

19.15.4.6 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report. When complete, the report is sent out to the client.

19.15.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using TestAmerica's Corporate SOP (CA-Q-S-002) as the guideline for our internal SOP No. ST-QA-0040, entitled "Manual Integration Procedure".

- 19.15.5.1** The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.
- 19.15.5.2** Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principals and policy and is grounds for immediate termination.
- 19.15.5.3** Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.
- 19.15.5.4** All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale “after” chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale “before” chromatograms are also required for all manual integrations done on samples, calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc. unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Figure 19-1. Example - Demonstration of Capability Documentation
Analyst Demonstration of Capability

TestAmerica St. Louis

Analyst Name

MDD/YYYY

Preparation Method(s):

Analytical Method(s):

Matrix: Solid/Water/Waste, etc...

Method Description:

Preparation SOP No: ST-XX-####

Analytical SOP No: ST-XX-####

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method with the specifications in the cited SOP, which is in use at this facility for the analysis of samples under the laboratory's Quality Assurance Plan, has completed the Demonstration of Capability (DOC).
2. The test method(s) was performed by the analyst identified on this certificate.
3. A copy of test method(s) and laboratory SOPs are available for all personnel on-site.
These documents have been reviewed by the analyst as part of this DOC.
4. The data associated with the demonstration of capability are true, accurate, complete and self-explanatory.
5. All raw data necessary to reconstruct and validate these analyses have been retained at the facility. The associated information is organized and available for review.

Analyst

Signature

Date

Dept Supervisor

Signature

Date

QA Manager

Signature

Date

SECTION 20. EQUIPMENT and CALIBRATIONS

20.1 Overview

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturer's instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 Preventive Maintenance

The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

Routine preventive maintenance procedures and frequency, such as cleaning and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Technical Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures maybe/are also outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

- Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.
- Each entry in the instrument log includes the Analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on 'date' was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.

- When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. Folder pockets are used in some logbooks to store service receipts.

If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it shall be taken out of operation and tagged as out-of-service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses. The instrument is “tagged-out” by the analyst who observed the issue, the department manager or the QA department. A non-conformance memo, or some other “tag”, is posted on the affected instrument.

In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back-up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study or MDL verification sample) prior to return to lab operations.

20.3 Support Equipment

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, field sampling devices, temperature measuring devices, thermal/pressure sample preparation devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains “calibration only” ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file.

Refer to SOP ST-QA-0005, "Calibration and Verification Procedures for Thermometers, Balances, Weights and Pipettes," for detailed information.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to ± 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

20.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer. IR thermometers, digital probes and thermocouples are calibrated quarterly.

- If the temperature measuring device is used over a range of 10°C or less, then a single point verification within the range of use is acceptable;
- If the temperature measuring device is used over a range of greater than 10°C, then the verification must bracket the range of use.

The NIST thermometers are recalibrated every five years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks or filed in QA records. Monitoring of method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method-specific logbooks. More information on this subject can be found in the SOP ST-QA-0005.

20.3.4 Refrigerators/Freezer Units, Water baths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day. (Sample storage is monitored 7 days a week for units storing DOE and/or DoD samples).

Ovens, water baths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between $> 0^{\circ}\text{C}$ and $\leq 6^{\circ}\text{C}$; freezers are kept below 10°C .

Specific temperature settings/ranges for other refrigerators, ovens water baths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware and Glass microliter syringes) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

For those dispensers that are not used for analytical measurements, a label is applied to the device stating that it is non-critical. Any device not regularly verified cannot be used for any quantitative measurements.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an "Accuracy and Precision Statement of Conformance" from Hamilton attesting established accuracy.

20.4 Instrument Calibrations

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 12).

Note: Instruments are calibrated initially and as needed after that and at least annually.

20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of 3 calibration points will be used.

Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to at least the same number of significant figures used to report the data) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exceptions to these rules ICP and ICPMS methods which define the working range with periodic linear dynamic range studies, rather than through the range of concentrations of daily calibration standards. This also does not apply to radiochemical methods.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst at a different time or a different preparation would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.1.1 Calibration Verification (Organic/Inorganic)

The calibration relationship established during the initial calibration must be verified initially and at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 TNI Standard. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications may use the same source standards as the calibration curve.

Note: The process of calibration verification referred to here is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met, i.e., RPD, per 2009 TNI Standard.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Note: If an internal standard calibration is being used then bracketing calibration verification standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12 hours of the beginning of the shift.

A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements see specific SOPs. Most Inorganic methods require the CCV to be analyzed after every 10 samples or injections, including matrix or batch QC samples.

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed and documented, the laboratory shall demonstrate acceptable instrument / method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with unacceptable calibration verification may be fully useable under the following special conditions and reported based upon discussion and approval of the client:

- a). when the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported with a case narrative explaining the high bias. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or

- b). when the acceptance criteria for the CCV are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted.

Samples reported by the 2 conditions identified above will be appropriately flagged.

20.4.1.2 Verification of Linear and Non-Linear Calibrations

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in [Appendix 4](#)). Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.
- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

20.4.2 Radiochemical Calibrations

20.4.2.1 CALIBRATION STANDARDS

Shelf life for stock radioactive standards shall not exceed 5 half-lives. Shelf life for stock solutions prepared in the laboratory from salts, metals or dilution from a parent solution shall be no greater than one year, unless stated otherwise on the calibration certificate from the manufacturer. Standards in the form of a soil, sealed sources, filter, plated sources and sealed epoxy Marinelli beakers do not always have an expiration date. After the 1 year shelf life of the stock solution has expired, it must be re-verified.

If the standard is not re-verified, the standard shall be removed or clearly designated as acceptable for qualitative purposes only.

The expiration date of the secondary standard shall not exceed the expiration date of the primary standard.

The accuracy of calibration standards is checked by comparison with a calibration verification standard from a second source. In cases where a second standard source is not available, a source from a different vendor is acceptable. All cases where this requirement cannot be met shall be documented with a nonconformance memo.

When a traceable standard is not available to use for calibration or verification activities, a non-traceable standard may be used if written client approval is obtained (when required).

Calibration standards are prepared using the appropriate procedures.

For each analyte of interest, prepare calibration standards at the minimum number of concentrations as stated in the analytical methods.

Standards for instrument calibration are obtained from a variety of sources. All radioactive standards are traceable to NIST whenever possible. Dilution standards are prepared from stock standards purchased from commercial suppliers. A standard log is maintained, containing concentration/activity, date of receipt, date of standard preparation, any dilutions made, lot number, supplier, type of solvent and a unique code number to identify the standard.

The frequency of calibration can be found in the laboratory's radiochemical methods and [Table 20-4](#).

20.4.3 RADIOCHEMICAL CONTINUING INSTRUMENT CALIBRATION, VERIFICATION and RADIOCHEMICAL BACKGROUND MEASUREMENT

Performance checks shall be performed using appropriate check sources and monitored to ensure that the instruments are running properly and that detector response has not significantly changed. Background measurements are made according to the schedule on Table 20-4 and monitored to ensure that the laboratory maintains its capability to meet required data quality objectives.

20.4.4 RADIOCHEMICAL INSTRUMENT CONTAMINATION MONITORING

The laboratory radiochemical instrumentation SOPs specify the requirements for monitoring radiochemical instrumentation. The SOP specifies the monitoring frequencies and criteria for initiating corrective action.

20.5 Tentatively Identified Compounds (TIC) – GC/MS Analysis

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.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as

a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

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. See SOPs ST-MS-0001 and ST-MS-0002 for guidelines on making tentative identifications and reporting TICs.

20.6GC/MS Tuning

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Table 20-1. Example: Instrumentation List

| Equipment/ Instrument | Manufacturer | Model Number | Serial Number | Year(s) Put into Service | Condition When Received |
|-----------------------------|-----------------|--------------|---------------|-----------------------------|-------------------------------|
| GC/MS – “G” GC System | Hewlett Packard | 5890 | 2807A11075 | 1987 | NEW |
| GC/MS – “G” Concentrator | Tekmar | LSC3000 | 98175006 | 1992 | NEW |
| GC/MS – “G” Autosampler | Varian | Archon | 13540 | 2001 | NEW |
| GC/MS – “F” | Hewlett Packard | 5973 | DE00020247 | 1998 | NEW |
| GC/MS – “F” GC System | Hewlett Packard | 6890 | US80221392 | 1998 | NEW |
| GC/MS – “F” Concentrator | IO | Eclipse 4660 | D530466888P | 2002 | NEW |
| GC/MS – “F” Autosampler | Varian | Archon | 14613 | 2001 | NEW |
| GC/MS – “L” | Hewlett Packard | 5973 | CN10339019 | 2004 | NEW |
| GC/MS – “L” Concentrator | Teledyne Tekmar | Velocity XPT | US03346007 | 2004 | NEW |
| GC/MS – “L” Autosampler | Teledyne Tekmar | SOLATek 72 | US03349002 | 2004 | NEW |
| GC/MS – “M” | Hewlett Packard | 5973 | CN10412013 | 2004 | NEW |
| GC/MS – “M” Concentrator | Teledyne Tekmar | Velocity XPT | US0412001 | 2004 | NEW |
| GC/MS – “M” Autosampler | Teledyne Tekmar | SOLATek 72 | US04119003 | 2004 | NEW |
| GC/MS – “N” | Hewlett Packard | 5973 | CN10512032 | 2005 | NEW |
| GC/MS – “N” GC System | Hewlett Packard | 6890 | US44621325 | 2005 | NEW |

| Equipment/ Instrument | Manufacturer | Model Number | Serial Number | Year(s) Put into Service | Condition When Received |
|--|---------------------|--------------------|---------------|-----------------------------|-------------------------------|
| GC/MS – “N” Concentrator | Tekmar/Dohrman | Velocity XPT | US03247002 | 2009 | Used |
| GC/MS – “N” Autosampler | Teledyne Teckmar | Solatek 72 | US03100004 | 2009 | Used |
| GC/MS – “K” | Hewlett Packard | 5973 | US81221525 | 1998 | NEW |
| GC/MS – “K” GC System | Hewlett Packard | 6890 | US00022347 | 1998 | NEW |
| GC/MS – “K” Series Injector | Hewlett Packard | 7683 | CN31530345 | 1998 | NEW |
| GC/MS – “K” Autosampler | Hewlett Packard | G2614A | US83501656 | 1998 | NEW |
| GC/MS – “J” | Hewlett Packard | 5973 | US80321385 | 1998 | NEW |
| GC/MS – “J” GC System | Hewlett Packard | 6890 | US00021127 | 1998 | NEW |
| GC/MS – “J” Series Injector | Hewlett Packard | 7683 | US81801195 | 1998 | NEW |
| GC/MS – “J” Autosampler | Hewlett Packard | G2614A | US80600251 | 1998 | NEW |
| GC/MS – “I” | Hewlett Packard | 5973 | CN10514049 | 2005 | NEW |
| GC/MS – “I” GC System | Hewlett Packard | G2579A | US44621455 | 2005 | NEW |
| GC/MS – “I” Series Injector | Hewlett Packard | 7683 | CN51224243 | 2005 | NEW |
| GC/MS – “I” Autosampler | Hewlett Packard | G2614A | CN42229061 | 2005 | NEW |
| GC/MS – “X” | Agilent | 5973 | US10461280 | 2008 | NEW |
| GC/MS – “X” GC System | Agilent | 6890N | US10144027 | 2008 | NEW |
| GC/MS – “X” Series Injector | Tekmar | 7683 | US01330017 | 2008 | NEW |
| GC/MS – “X” Autosampler | IO | G2614A | 1411 | 2008 | NEW |
| GC/MS – “Y” | Hewlett Packard | 5970 | 3449A02079 | 2009 | Used |
| GC/MS – “Y” GC System | Hewlett Packard | 5890 | 3336A57239 | 2009 | Used |
| GC/MS – “Y” Concentrator | Tekmar | Tekmar 3000 | 93300001 | 2009 | NEW |
| GC/MS – “Y” Autosampler | Varian | Archon | 12541 | 2009 | Used |
| GC/MS – “Z” | Hewlett Packard | 5973 | US80230105 | 2010 | Refurbished |
| GC/MS – “Z” GC System | Hewlett Packard | 6890 | US00009101 | 2010 | Refurbished |
| GC/MS – “Z” Concentrator | IO | Eclipse 4660 | E002466503P | 2010 | NEW |
| GC/MS – “Z” Autosampler | Varian | Archon | MS1003W019 | 2010 | NEW |
| LC/MS/MS – “R” Mass Spectrometer | Waters | Quattro Premier XE | VAB461 | 2006 | NEW |

| Equipment/ Instrument | Manufacturer | Model Number | Serial Number | Year(s) Put into Service | Condition When Received |
|---|-----------------|--------------------------------------|---------------|-----------------------------|-------------------------------|
| LC/MS/MS – “R” Liquid Chromatograph | Waters | Acquity PDA Detector | L05UPD807N | 2006 | NEW |
| LC/MS/MS – “R” Liquid Chromatograph | Waters | Acquity Sample Manager | 60UPS056M | 2006 | NEW |
| LC/MS/MS – “R” Liquid Chromatograph | Waters | Acquity Binary Solvent Man. | C06UPB008M | 2006 | NEW |
| LC/MS/MS – “T” Mass Spectrometer | Micromass | Ultima | VB280 | 2008 | NEW |
| LC/MS/MS – “T” HPLC – “Q” ALS Therm | Hewlett Packard | G1330A | DE13201124 | 1999 | NEW |
| LC/MS/MS – “T” HPLC – “Q” Quat Pump | Hewlett Packard | G1311A | DE14916965 | 1999 | NEW |
| LC/MS/MS – “X” Liquid Chromatograph | Waters | Xevo | VBA453 | 2010 | NEW |
| LC/MS/MS – “X” Liquid Chromatograph | Waters | Acquity Sample Manager | H07UPB932M | 2010 | NEW |
| LC/MS/MS – “X” Liquid Chromatograph | Waters | Acquity Binary Solvent Manager | H07UPa802M | 2010 | NEW |
| GC – “L” | Hewlett Packard | 5890 | 2413A04451 | 1987 | NEW |
| GC – “L” Autosampler | Varian | Archon | 160098 | 2000 | NEW |
| GC – “L” Concentrator | Tekmar | LSC3000 | 93300001 | 1997 | NEW |
| GC – “K” | Agilent | 6890 | US00039258 | 2000 | NEW |
| GC – “K” Autosampler | Agilent | 7683 | US04709936 | 2000 | NEW |
| GC – “E” | Hewlett Packard | 6890 | US00011425 | 2000 | NEW |
| GC – “E” Autosampler | Hewlett Packard | 6890 | US71701354 | 2000 | NEW |
| GC – “M” | Agilent | 6890 | US10328036 | 2003 | NEW |
| GC – “M” Autosampler | Agilent | 7683 | CN32624339 | 2003 | NEW |
| GC – “O” | Agilent | 6890 | CN10422045 | 2004 | NEW |
| GC – “O” Autosampler | Agilent | 7683 | CN51132513 | 2004 | NEW |
| GC – “P” | Agilent | 6890N | CN10510018 | 2005 | NEW |
| GC – “P” Autosampler | Agilent | 7683 | CN51532846 | 2005 | NEW |
| GC – “V” | Agilent | 6890 | US00008573 | 2009 | USED |

| Equipment/ Instrument | Manufacturer | Model Number | Serial Number | Year(s) Put into Service | Condition When Received |
|---|-----------------|---------------|---------------|-----------------------------|-------------------------------|
| GC – “V” (Auto Sampler) | Agilent | G1530A | US8090377 | 2009 | USED |
| HPLC – “N” | Hewlett Packard | G1329A | DE91603153 | 1999 | NEW |
| HPLC – “N” ALS Therm | Hewlett Packard | G1330A | DE82203165 | 1999 | NEW |
| HPLC – “N” COLCOM | Hewlett Packard | G1316A | DE91609858 | 1999 | NEW |
| HPLC – “N” DAD | Hewlett Packard | G1315A | DE91605478 | 1999 | NEW |
| HPLC – “N” Degasser | Hewlett Packard | G1322A | JP73016399 | 1999 | NEW |
| HPLC – “N” Quat Pump | Hewlett Packard | G1311A | DE91605960 | 1999 | NEW |
| HPLC – “N” FLD | Hewlett Packard | G1321A | DE92001122 | 1999 | NEW |
| HPLC LCE (DAD) | Agilent | G1315D | DE64255811 | 2010 | USED |
| HPLC LCE (COL) | Agilent | G1316A | DE63065337 | 2010 | USED |
| HPLC LCE (Auto Sampler) | Agilent | G1329A | DE64764168 | 2010 | USED |
| HPLC LCE (Pump) | Agilent | G1311A | DE62962744 | 2010 | USED |
| GPC-1 | O-I Analytical | Autoprep 2000 | E427330254 | 2011 | NEW |
| ICP-MS – “6100” | Perkin Elmer | ELAN 6100 | 0859907 | 1999 | NEW |
| ICP-MS – “6100” Autosampler | Perkin Elmer | AS-91 | 4123 | 1999 | NEW |
| ICP-MS – “7500” | Agilent | 7500CX | JP82802890 | 2009 | NEW |
| ICP-MS – “7700” | Agilent | 7700 | JP10110271 | 2011 | NEW |
| ICP-MS – “9000” | Perkin Elmer | ELAN 9000 | P1000302 | 2013 | USED |
| ICP – “6500 Duel View” | Thermo Fisher | 6000 Series | 20105013 | 2011 | NEW |
| CVAA | Leeman Labs | Hydra AA 2 | 0035 | 2011 | NEW |
| IC – “S” Chromatography Oven | Dionex | LC30 | 98070139 | 2008 | NEW |
| IC – “S” Conductivity Detector | Dionex | CD20 | 99070231 | 2008 | NEW |
| IC – “S” Gradient Pump | Dionex | GP50 | 99070382 | 2008 | NEW |
| IC – “S” Autosampler | Dionex | AS40 | 00090205 | 2008 | NEW |
| IC – “2500” Chromatography Oven | Dionex | LC25 | 03120540 | 2004 | NEW |
| IC – “2500” Conductivity Detector | Dionex | CD25 | 03120540 | 2004 | NEW |
| IC – “2500” Gradient Pump | Dionex | GP50 | 03120633 | 2004 | NEW |
| IC – “2500” Autosampler | Dionex | AS40 | 07020461 | 2004 | NEW |

| Equipment/ Instrument | Manufacturer | Model Number | Serial Number | Year(s) Put into Service | Condition When Received |
|---|------------------------|-----------------|--|-----------------------------|-------------------------------|
| IC – “1500” Ion Chromatography System | Dionex | ICS-1500 | 03080236 | 2008 | NEW |
| IC – “1500” Autosampler | Dionex | ASM-3 | 920937 | 2008 | NEW |
| TOC | Shimadzu | TOC-5050A | 36501107 | 1999 | NEW |
| TOX | Mitsubishi | 100 TOX | A7M00017 | 1999 | NEW |
| TOC | Shimadzu | TOC-VCPN | H51404635090 | 2010 | NEW |
| Solid Sample Module | Shimadzu | SSM-5000A | H52504700582NK | 2010 | NEW |
| Discrete Analyzer | Systea | Easy Chem-Plus | 0901262 | 2010 | NEW |
| UV Spec 1 | Thermospectroni c | Genysis | 3SGF211001 | 2003 | NEW |
| UV Spec 2 | Thermospectroni c | Genysis | 3SGR172002 | 2013 | NEW |
| UV Spec | Shimadzu | UV-2401PC | A1083 (320053LP) | 2013 | USED |
| TRAACS – “1” | Technicon | Traacs 800 | 0103011 | 1988 | NEW |
| BOD | Man-Tech Associates | 04-227 | 270D3XB245 | 2003 | NEW |
| Ignitability Apparatus: Open Cup | Fisher | D-92 | 906N0014 | 1998 | NEW |
| Ignitability Apparatus: Closed Cup | Fisher | 162 | 1149 | 1992 | NEW |
| Multimeter | Thermo | 5 Star | B15814 | 2009 | NEW |
| Multimeter | Thermo | 5 Star | 015748 | 2009 | NEW |
| Alpha Spectrometer – “AV1 - AV24” “AV43 - AV122” “AV123 - AV226” “AV227 – AV247” | Ortec | Multi-Component | Multiple* | 1987-2011 | NEW |
| Gamma Spectrometer Intrinsic Germanium Detector “GE1 - GE10” “GE11 – GE19” | Tennelec / Ortec | Multi-Component | Multiple* | 1991-2011 | NEW |
| GFPC – “Protean” | Protean | MPC-9604 | 233126-BO 236534-BO 236532-BO 236533-BO | 2003 | NEW |

| Equipment/ Instrument | Manufacturer | Model Number | Serial Number | Year(s) Put into Service | Condition When Received |
|--|--------------|--------------|--|-----------------------------|-------------------------------|
| GFPC – “Orange” | Protean | MPC-9604 | 08217155 08217156 08217154 08217153 10181186 10181187 | 2008-2010 | NEW |
| GFPC – “Purple” | Protean | MPC-9604 | 10181185 10181184 10029177 10029178 10029179 10029180 | 2010 | NEW |
| GFPC “Green” | Tennelec | LB5100 | 31360 | 2000 | NEW |
| LSC – “3180” Pink Teal Aquau Brown | Packard | Tricarb 3180 | DG06095123 DG01117382 DG01117385 DG01117384 DG01117383 | 2009-2011 | NEW |
| LSC – “3170” | Packard | Tricarb 3170 | 429670/429774 | 2002 | NEW |

Table 20-2. Example: Schedule of Routine Maintenance

Inductively Coupled Plasma

DAILY OR AS NEEDED - CHECK

- Gas supply
- Waste and rinse solution levels
- Droplet size (nebulizer)
- Replace orange/green tubing

WEEKLY

- Check water level in cool flow
- Nebulizer rinse
- Replace waste line
- Clean injector tip
- Check /Clean plasma torch assembly
- Replace sample tubing
- Clean spray chamber

MONTHLY

- Check /Clean air filter of power unit
- Clean fast autosampler valve and rotor

ANNUALLY

- Check vacuum system oil
- Check /Replace coolant water filter

Inductively Coupled Plasma/Mass Spectrometer

DAILY OR AS NEEDED

- Check Waste and rinse water container levels
- Check/ Replace sample, internal and waste lines
- Clean cones (7500, 7700)
- Clean cone

WEEKLY

- Check /Clean interface cones
- Check Roughing pump oil level and color
- Replace Waste Tubing

MONTHLY

- Check /Change pump oil (6100)
- Check /Clean auto lens (6100)
- Clean torch & injector tip (6100)
- Clean auto lense (6100)
- Clean torch (7500, 7700)
- Move data set files (7500, 7700)

Cold Vapor Automatic Analysis

DAILY OR AS NEEDED

- Check /Pump and drain tubing
- Check Gas pressure
- Instrument parameter check

WEEKLY

- Check /Change sample, reductant and draining tubings

MONTHLY

- Change/rinse tubing
- Check/change waste tubing

QUARTERLY

- Check /Change drying tube

TOX

DAILY OR AS NEEDED

- Cell Performance Test
- Electrodes
- Cell Fluid, Dehydrating Fluid and Electrolyte
- Adsorption module (cleaned at end of use)

Autoanalyzer Traacs- 1

DAILY

- Washout procedure (at end)

AS NEEDED

- Check /Change tubing
- Lubricate Probe shaft
- Lubricate oil rollers

TOC

DAILY OR AS NEEDED

- Air Supply and Gas Flow Rate (150mm)
- Humidifier
- A/LS Rinse Tank

MONTHLY

- Check /Inspect SO₃ scrubber – change if crystals at inlet are not white.
- Check /Inspect halogen scrubber – change if black color approaches outlet end.

ANNUALLY

- Check /Change CO₂ absorber

Ion Chromatography

DAILY OR AS NEEDED

- Plumbing for leaks
- Gases and Pump Pressure
- Conductivity meter
- Fill eluent
- Column replacement

UV Spec

DAILY OR AS NEEDED

- Rinse out Sample Cuvettes (after each use)

BOD

DAILY

- Calibration

As Needed

- Change membrane

Discrete Analyzer

DAILY

- Auto zero
- Perform rinse at completion of analysis
- Check DI water bottle/refill

Alpha Spectrometer

DAILY

- Pulsars

MONTHLY

- Backgrounds
- Clean detectors
- Continuing calibration verifications

ANNUALLY

- Calibrations

Gamma Spectrometer

DAILY

- Continuing calibration blank/continuing calibration verification

MONTHLY

- Clean/Long Backgrounds

ANNUALLY

- calibration checks

Gas Flow Proportional Counting

DAILY OR AS NEEDED

- Gas level
- Calibration verifications

MONTHLY

- Clean/Long Backgrounds

ANNUALLY

- Calibrations

Liquid Scintillation Counter

WEEKLY OR AS NEEDED

- Clean Fan

YEARLY

- Serviced by vendor

Semi-volatile Gas Chromatography / Mass Spectrometer

DAILY OR AS NEEDED

- Gas supply, column flow and inlet pressure
- Fill solvent rinse vials
- Check /Injection Port Cleaning
- Check /Change Septum, injection port liner, and seals
- Check /Trim Column
- Check/replace injection syringe

ANNUALLY

- Check /Replace pump oil

AS NEEDED

- Replace column
- Clean ion source
- Replace multiplier
- Replace electronic circuit board
- Replace detector
- Replace transfer lines

Volatile Gas Chromatography / Mass Spectrometer

DAILY OR AS NEEDED

- Gas supply, column flow and inlet pressure

QUARTERLY

- Check Trim Column
- Check/Change Trap

SEMI-ANNUALLY

- Check/Replace Column
- Check/Clean Source
- Check/Injection port maintenance

ANNUALLY

- Check/ Replace pump oil

High Pressure Liquid Chromatograph (HPLC)

DAILY OR AS NEEDED

- Ensure column flow and pressure are correct
- Ensure HPLC solvents are sufficient to run
- Ensure proper DAD signals are on
- Visibly check for leaks

MONTHLY

- Check/Change Purge Valve Frit

SEMIANNUALLY

- Check/Change Guard Cartridge and Frit Cap

BIANNUALLY

- Check/Replace Column
- Check/Replace UV Source
- Check/Replace Visible Source
- Check/Replace pump seals

Semi-Volatile Gas Chromatograph (Dual ECD)

DAILY OR AS NEEDED

- Ensure column flow and inlet pressure are correct
- Ensure temperature for oven, inlet(s), and detector(s) are correct
- Ensure solvent rinse vials are full
- Ensure injection syringe is secure in tower and plunger is engaged

MONTHLY

- Check/Replace injection port septum
- Visibly inspect injection port liner; replace if contaminated
- Check /Remove injection syringe and ensure plunger is free moving
- Check system for leaks (injection port, detector(s) and any column connectors)

SEMIANNUALLY

- Perform Radioactive leak test

Semi-Volatile Gas Chromatograph (FID)

DAILY OR AS NEEDED

- Check gas supply, column flow, and inlet pressure
- Fill solvent rinse vials

MONTHLY

- Check/Replace septum, injection port liner and seals
- Check/ Trim Guard Column

SEMIANNUALLY

- Check/ Replace Column

Volatile Gas Chromatograph

DAILY OR AS NEEDED

- Check gas supply, column flow and inlet pressure
- Change trap
- Trim column

SEMIANNUALLY

- Check/Replace Column
- Check/Injection port maintenance

ANNUALLY

- Check /Clean PID/FID

Liquid Chromatograph Mass Spectrometer Mass Spectrometer (LCMSMS)

DAILY OR AS NEEDED

- Check level of solution in reservoirs
- Check gas supply, column flow and system pressure
- Sonicate inlet check valves
- Clean ionization probes/corona pin
- Ballast Rough Pump

SEMIANNUALLY

- Check/Replace Column
- Check/Clean source
- Check/Injector maintenance

ANNUALLY

- Check/Replace pump oil

Table 20-3 Example: Periodic Calibration

| Instrument | Type of Calibration/ Number of Standards | Frequency | Acceptance Limits | Corrective Action |
|----------------------------------|--|--|--|---|
| Analytical Balance | Accuracy determined using working weights that are annually checked against weights traceable to the International System of Units (SI) through a NMI. Minimum of 2 standards bracketing the weight of interest. Inspected and checked by ISO17025 accredited vendor annually. | Each day of use | $\pm 0.1\%$ (QSM requires $\pm 0.1\%$ or ± 0.5 mg, whichever is greater) | Clean, check level, insure lack of drafts, and that unit is warmed up, recheck. If fails, call service. |
| Top Loading Balance | Accuracy determined using ISO17025-accredited NIST weights. Minimum of 2 standards bracketing the weight of interest. Inspected and checked by ISO17025 accredited vendor annually | Each day of use | $\pm 2.0\%$ (QSM requires $\pm 2\%$ or ± 0.02 g, whichever is greater) | Clean. Replace. |
| ISO17025-accredited NIST Weights | Verification of standard mass using weights traceable to the International System of Units (SI) through a NMI | 5 years | Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory. | Replace. |
| NIST-Traceable Thermometer | Accuracy determined by ISO17025-accredited measurement laboratory. | 5 years | As per certificate. | Replace. |
| Thermometer | Against NIST-traceable thermometer | Yearly at appropriate temperature range for intended use | $\pm 1.0\text{ }^{\circ}\text{C}$ | Replace |
| Digital thermometer | Against NIST-traceable thermometer | Quarterly | $\pm 1.0\text{ }^{\circ}\text{C}$ | Replace |

| Instrument | Type of Calibration/ Number of Standards | Frequency | Acceptance Limits | Corrective Action |
|---|--|---|---|--|
| Refrigerator | Temperature checked using NIST-traceable thermometer. | Daily. If out of range, check again after several hours | 0 – 6 °C | Adjust. Repair. While waiting for repair, seal door, attach “Out of Service” sign, move items to functional unit. Notify supervisor. |
| Freezer | Temperature checked using NIST-traceable thermometer | Daily. If out of range, check again after several hours | <-10 °C | Adjust. Repair. While waiting for repair, seal door, attach “Out of Service” sign, move items to functional unit. Notify supervisor. |
| Oven | Temperature checked using NIST-traceable thermometer. | When in use. | 103 ± 2 °C (moisture determination) 180 ± 2°C (TDS) (DoD: ±5% of set temp) | Adjust. Replace. |
| Incubator | Temperature checked using NIST-traceable thermometer. | When in use. For microbiology, twice daily when in use. | BOD: 20 ± 1.0 °C | Adjust. Replace. |
| Water Bath | Temperature checked using NIST-traceable thermometer. | When in use. | ± 5 °C | Adjust. Replace. |
| Volumetric Dispensing Devices - pipettes | On delivery by weight. Using DI water, dispense into tared vessel. Record weight with device ID number. Before first use: 10 replicate measurements with %RSD ≤ 1%. | Day of use 3 reps | ± 2% bias Precision RSD ≤ 1% | Adjust. Replace. |
| Non-volumetric labware (applicable only when measuring initial sample vol. or final extract/digestate volume) | Gravimetric – 10 reps before use | By lot before first use or upon evidence of deterioration | Bias: Mean within ± 3% of nominal volume Precision RSD ≤ 3% of stated value (based on 10 replicate measures) | replace |
| Volumetric glassware | The laboratory uses only Class A volumetric glassware. Calibration not required | N/A | Check for deterioration | Replace |

| Instrument | Type of Calibration/ Number of Standards | Frequency | Acceptance Limits | Corrective Action |
|---------------------------------|--|---|----------------------------------|---|
| Glass Microliter Syringes | None | Accuracy must be initially demonstrated if syringe was not received with a certificate attesting to established accuracy. | $\pm 1\%$ | Not applicable. |
| Conductivity Meter | Cell impedance calibrated with three KCl standards. | Each use. | $r \geq 0.99$ | Recalibrate. |
| Deionized Water | Check in-line conductivity meter on system with conductivity meter in Inorganic Department. | Daily | $<10 \mu\text{mhos}/\text{cm}^2$ | Record on log. Report discrepancies to QA Department |

Table 20-4 Radiochemistry Calibration, Verification & Background Criteria

| Instrument | Calibration Procedure | Frequency of Calibration | Acceptance Criteria |
|--------------------|-----------------------|--|--|
| Gamma Spectroscopy | Initial Calibration | Energy, FWHM and energy calibrations shall be established for the germanium spectroscopy systems annually , or when the calibration quality control check indicates an unacceptable change in the energy calibration parameters. | The curve should have eight calibration points used to determine the energy relationship of the calibration. The calibration source must have radionuclides that "blanket" the intended range of calibration. The energy difference should be less than 0.05% for all points or with 2 keV for calibration points. Computed efficiency test for all points should have a percent difference less than 8%. The FWHM must be less than 3.0 keV at 1332 keV. FWHM difference should be less than 8% for all points. |
| Gamma Spectroscopy | Initial Background | Background subtraction spectrum shall be established for the germanium spectroscopy systems monthly , or when the background quality control check indicates an unacceptable change in the daily background parameters, or as needed per client requirements. | Background count time is 12 hours. |
| Gamma Spectroscopy | Continuing | Daily Checks The energy, resolution and efficiency calibrations for a detector shall be checked with its respective source each day that the germanium spectroscopy system is used. The detector background shall be checked each day that the germanium spectroscopy system is used. | Calibration (efficiency, resolution, energy alignment, and background) quality control parameters will be found not acceptable if the result is outside the established limits (2σ to 3σ range) and marked as "action". The Daily QC check may only be recounted once without corrective action. |
| Alpha Spectroscopy | Initial Calibration | Energy calibrations shall be established for the alpha spectroscopy systems yearly , or when the calibration quality control check indicates an unacceptable change in the energy calibration parameters. Efficiency calibrations shall be established for the alpha spectroscopy systems yearly , or when the calibration quality control check indicates an unacceptable change in the efficiency calibration parameters. | Energy Calibrations shall be performed using at least three isotopes within the energy range of 3-6 meV. Final peak energy positions of all observed isotopes shall be within ± 40 keV of expected energy. Efficiency should fall between 20 and 32%. |

| Instrument | Calibration Procedure | Frequency of Calibration | Acceptance Criteria |
|-------------------------------|-----------------------|--|---|
| Alpha Spectroscopy | Initial Background | Background subtraction spectrum shall be established for the alpha spectroscopy systems monthly , or when the background quality control check indicates an unacceptable change in the daily background parameters. | Background count time is 960 minutes. |
| Alpha Spectroscopy | Continuing | Daily Checks Routine pulser quality control verifications are to be performed each day of use. The pulser energy, peak centroid, peak resolution, peak area quality control for a detector shall be checked each day that the alpha spectroscopy system is used. | Routine calibration, background and pulser quality control parameters using the "Boundary" out-of-range test will be found unacceptable if the value is outside reasonable parameter tolerance. The routine quality control check should be rerun to determine the statistical significance of the errant parameter. |
| Gas Flow Proportional Counter | Initial Calibration | Mass attenuation alpha/beta curves should be performed on an annual basis, or when the calibration quality control check indicates an unacceptable change in the efficiency calibration parameters. | The efficiency calibration shall consist of at least seven single or dual sets of mass attenuated calibration standards. The standards shall have enough activity to generate at least 10,000 counts in 90 minutes of count time for the most highly attenuated source. The count rate shall not exceed 5,000 counts per second. The coefficient of determination (r^2) shall be greater than or equal to 0.9. |
| Gas Flow Proportional Counter | Initial Background | Background established for the GFPC monthly , or when the background quality control check indicates an unacceptable change in the daily background parameters. | Backgrounds are counted for 1,000 minutes Alpha < 0.2 counts per minute Beta < 2.0 counts per minute |
| Gas Flow Proportional Counter | Continuing | Daily Checks Efficiency check and background check | |

SECTION 21. MEASUREMENT TRACEABILITY

21.1 Overview

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. (Refer to Section 20.3). With the exception of Class A Glassware and glass microliter syringes, quarterly accuracy checks are performed for all mechanical volumetric devices that are used to deliver volume critical measurements. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware and glass microliter syringes should be routinely inspected for chips, acid etching or deformity (e.g., bent needle). If the Class A glassware or syringe is suspect, the accuracy of the glassware will be assessed prior to use.

21.2 NIST-Traceable Weights and Thermometers

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), APLAC (Asia-Pacific Laboratory Accreditation Cooperation), or EA (European Cooperation for Accreditation) or another accreditation organization that is a signatory to a MRA (Mutual recognition Arrangement) of one or more of the following cooperation's – ILAC (International Laboratory Accreditation Cooperation) or APLAC (Asia-Pacific Laboratory Accreditation Cooperation).. A certificate and scope of accreditation is kept on file at the laboratory.

The calibration report or certificate submitted to TestAmerica St. Louis contains, in a well designed format, a traceability statement, the conditions under which the calibrations were made in the context of any potential influence, a compliance statement with an identified metrological specification and the pertinent clauses, a clearly identified record of the quantities and functional test results before and after re-calibration, and no recommendation on the calibration interval. All calibration reports are filed in the QA Office.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All liquid thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

21.3 Reference Standards / Materials

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by A2LA, NVLAP, and NIST with an accompanying Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a unique Reagents Log Identification Number generated by LIMS and an expiration date. All documentation received with the reference standard is retained as a QC record and references the Standards Log Standard Identification Number. Reference standards that are used in the radiochemical laboratory shall be obtained from NIST, or suppliers who participate in supplying NIST standards or NIST traceable radionuclides. When traceable standards are not available, written approval for use must be obtained from DOE clients.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. Radiochemical standards must be verified prior to initial use. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Corporate Environmental Health & Safety Manual and the analytical method SOPs "Standards and Reagents" section for additional details. Radiochemical standards and reference material are stored separately from samples and are protected in a controlled cabinet or refrigerator. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory. The laboratory must have documented contingency procedures for re-verifying expired standards.

21.4 Documentation and Labeling of Standards, Reagents, and Reference Materials

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company-wide purchase. [Refer to TestAmerica's Corporate SOP (CA-Q-S-001), Solvent and Acid Lot Testing and Approval.] Purchased stock mixtures and reagents are labeled to indicate the date they are opened.

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained in a

directory on the laboratory network drive. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to method specific SOPs and ST-QA-0002, "Standard and Reagent Preparation".

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material.

21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS, and are assigned a unique identification number. The following information is typically recorded in the electronic database:

- Standard ID
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds; these records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

21.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- Standard ID (assigned by the LIMS)

- Special Health/Safety warnings if applicable

Records must also be maintained of the date of receipt for commercially purchased items or date of preparation for laboratory prepared items. Special Health/Safety warnings must also be available to the analyst. This information is maintained in the MSDS documents available on the TestAmerica intranet site).

21.4.3 In addition, the following information may be helpful:

- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Recommended Storage Conditions
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include an expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and raw data.

All reagents and standards must be stored in accordance to the following priority:

1. with the manufacturer's recommendations;
2. with requirements in the specific analytical methods as specified in the laboratory SOP.

SECTION 22. SAMPLING

22.1 Overview

The laboratory does not provide sampling services. The laboratory's responsibility in the sample collection process lies in supplying the sampler with the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory

22.2 Sampling Containers

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Any certificates of cleanliness that are provided by the supplier are maintained at the laboratory.

22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether

prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

22.3 Definition of Holding Time

The date and time of sampling documented on the COC form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in “days” (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in “hours” (e.g., 6 hours, 24 hours, etc.) is measured from date and time zero. The first day of holding time ends twenty-four hours after sampling. Holding times for analysis include any necessary reanalysis. However, there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is.

22.4 Sampling Containers, Preservation Requirements, Holding Times

The preservation and holding time criteria specified in the laboratory SOPs are derived from the source documents for the methods. If method required holding times or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. As soon as possible or “ASAP” is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time. The laboratory SOP ST-PM-0002 contains a table listing preservation, container and holding time information.

22.5 Sample Aliquots / Subsampling

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory’s responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots & sub-sampling are located in SOP ST-QA-0038, “Procedure for Compositing and Sub-sampling”.

NOTE: Unless otherwise noted by individual preparation SOPs, the following statements apply to sample aliquots of volume (liquid) for testing analysis.

- Density Requirement – If a sample is known or suspected (based upon client knowledge, project scope, or site history) to have a high density (>1.2 g/mL, e.g. a brine or waste) or a low density (<0.98 g/mL, e.g. mixed solvent), the sample density will be measured and the volume determined arithmetically (sample mass divided by the density equals the volume).
- Volume Determination – Aliquot volume is calculated by gravimetric determination assuming a sample density of 1. Samples that are not aqueous, or suspected of having a density greater than 1.2, will have aliquots taken for density analysis to correct volume for density

SECTION 23. HANDLING OF SAMPLES

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.1 Chain of Custody (COC)

The COC form is the written documented history of any sample and is initiated at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 23-1.

23.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 23-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used

- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

When the sampling personnel deliver the samples directly to TestAmerica personnel, the samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory personnel. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. When sampling personnel deliver the samples through a common carrier (Fed-Ex, UPS), the COC relinquished date/time is completed by the field personnel and samples are released to the carrier. Samples are only considered to be received by lab when personnel at the fixed laboratory facility have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The receipt from the courier is stored with the other login paperwork.

23.1.2 Legal / Evidentiary Chain-of-Custody

If samples are identified for legal/evidentiary purposes on the COC, login will complete the custody seal, retain the shipping record with the COC, and initiate an internal COC for laboratory use by analysts and a sample disposal record.

23.2 Sample Receipt

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are described in SOP ST-PM-0002, "Sample Receipt and Chain of Custody".

23.2.1 Laboratory Receipt

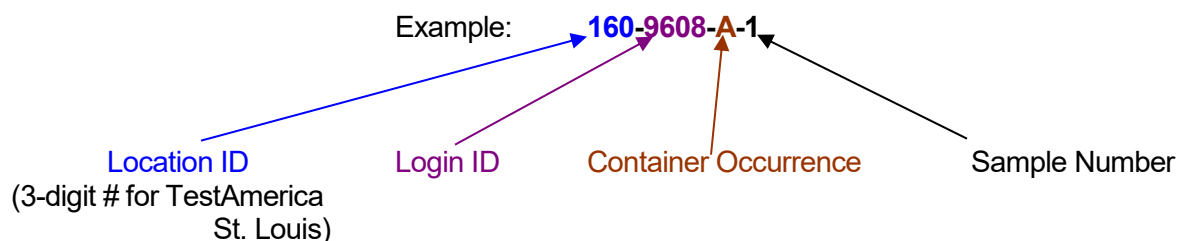
When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. Coolers received from a known or potential radiologically contaminated site are frisked prior to opening. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on a "Condition Upon Receipt" form (CUR) and brought to the immediate attention of the client. The COC,

shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

23.2.1.1 Unique Sample Identification

All samples that are processed through the laboratory receive a unique sample identification to ensure that there can be no confusion regarding the identity of such samples at anytime. This system includes identification for all samples, subsamples and subsequent extracts and/or digestates.

The laboratory assigns a unique identification (e.g., Sample ID) code to each sample container received at the laboratory. This Primary ID is made up of the following four pieces of information:



The above example indicates TestAmerica St. Louis (location 160), Login ID 9608 (unique to a particular job/client), container “A” of sample number 1.

If the primary container goes through a prep step that creates a “new” container, then the new container is considered secondary and gets another ID. For example, when a 1-liter amber bottle is sent through a Liquid/Liquid Extraction and extraction vial is created from the prep step. The vial would be a secondary container and would be labeled as follows:

160-9608-A-1-A

Secondary Container Occurrence - the Secondary ID has five components

The IDs are ‘bar-coded’ on the LIMS generated laboratory sample label attached to each container.

These steps allow the samples to be tracked through the laboratory in every step from receipt to disposal.

23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy (Figure 23-2) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;

- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);
- the Project Manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined and noted in the Case Narrative.

23.3.1 After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.

23.3.2 For samples received from a potentially radioactive site, an aliquot is removed from the container to perform a "rad screen."

23.3.3 Any deviations from these checks that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or
- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Once sample acceptance is verified, the samples are logged into the LIMS according SOP ST-PM-0002.

23.4 Sample Storage

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators, freezers or protected locations suitable for the sample matrix. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples having high levels of radiochemical contamination are labeled as such. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and are analyzed every two weeks.

Analysts and technicians retrieve the sample container allocated to their analysis from the designated refrigerator and place them on carts, analyze the sample, and return the remaining sample or empty container to the refrigerator from which it originally came. All unused portions of samples, including empty sample containers, are returned to the secure sample control area. All samples are kept in the refrigerators for two to four weeks after analysis, which meets or exceeds most sample holding times. After two to four weeks the samples are moved to a dry

room temperature sample archive area where they are stored for an additional four weeks before they are disposed of. This eight week holding period allows samples to be checked if a discrepancy or question arises. Special arrangements may be made to store samples for longer periods of time. This extended holding period allows additional analyses to be performed on the archived sample and assists clients in dealing with legal matters or regulatory issues.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

23.5 Hazardous Samples and Foreign Soils

To minimize exposure to personnel and to avoid potential accidents, hazardous and foreign soil samples are stored in an isolated area designated for hazardous waste only. The sample itself is clearly "HAZARDOUS" or "FOREIGN SOIL". Any sample that is known to be hazardous at the time of receipt or, if after completion of analysis the result exceeds the acceptable regulatory levels, the sample is labeled as such. Potentially radioactive samples are "screened" prior to release to the laboratory. The RAD category is entered into the LIMS and alerts the analyst to the radiation level associated with the sample. All hazardous samples are either returned to the client or disposed of appropriately through a hazardous waste disposal firm that lab-packs all hazardous samples and removes them from the laboratory. Foreign soil samples are sent out for incineration by a USDA-approved waste disposal facility (see SOPs ST-HS-0006, "Quarantine Soils Procedure", and the Radiation Protection SOPs for more details).

23.6 Sample Shipping

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses (see Note). The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they are not inadvertently omitting a key part of regulatory compliance testing.

23.7 Sample Disposal

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded.

Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP: ST-HS-0004, "Hazardous Waste Management Plan"). All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than two months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, and return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record should be completed.

Figure 23-1. Example: Chain of Custody (COC)

| Chain of Custody Record | | | | Temperature on Receipt _____ | | | | Drinking Water? Yes <input type="checkbox"/> No <input type="checkbox"/> | | | |
|--|--|--|--|--|--|--|--|--|--|--|--|
| Project Name and Location (State) | | | | Project Manager | | | | Date | | | |
| City | | | | State | | | | Zip Code | | | |
| Contract/Purchase Order/Quote No. | | | | Site Contact | | | | Lab Contact | | | |
| Sample I.D. No. and Description (Containers for each sample may be combined on one line) | | | | Date | | | | Time | | | |
| <div style="display: flex; justify-content: space-between;"> <div> Containers & Preservatives <input type="checkbox"/> Unpres <input type="checkbox"/> H2SO4 <input type="checkbox"/> HNO3 <input type="checkbox"/> HCl <input type="checkbox"/> NaOH <input type="checkbox"/> ZnAc <input type="checkbox"/> NaOH </div> <div> Matrix <input type="checkbox"/> Air <input type="checkbox"/> Aquatics <input type="checkbox"/> Sed <input type="checkbox"/> Soil </div> </div> | | | | <div style="display: flex; justify-content: space-between;"> <div> Sample Disposal <input type="checkbox"/> Return to Client <input type="checkbox"/> Disposal By Lab <input type="checkbox"/> Archive For _____ Months </div> <div> QC Requirements (Specify) 1. Received By _____ Date _____ 2. Received By _____ Date _____ 3. Received By _____ Date _____ </div> </div> | | | | <div style="display: flex; justify-content: space-between;"> <div> Possible Hazard Identification <input type="checkbox"/> Non-Hazard <input type="checkbox"/> Flammable <input type="checkbox"/> Skin Irritant <input type="checkbox"/> Poison B <input type="checkbox"/> Unknown </div> <div> Turn Around Time Required <input type="checkbox"/> 24 Hours <input type="checkbox"/> 48 Hours <input type="checkbox"/> 7 Days <input type="checkbox"/> 14 Days <input type="checkbox"/> 21 Days <input type="checkbox"/> Other _____ </div> </div> | | | |
| | | | | | | | | | | | |
| <div style="display: flex; justify-content: space-between;"> <div> Possible Hazard Identification <input type="checkbox"/> Non-Hazard <input type="checkbox"/> Flammable <input type="checkbox"/> Skin Irritant <input type="checkbox"/> Poison B <input type="checkbox"/> Unknown </div> <div> Turn Around Time Required <input type="checkbox"/> 24 Hours <input type="checkbox"/> 48 Hours <input type="checkbox"/> 7 Days <input type="checkbox"/> 14 Days <input type="checkbox"/> 21 Days <input type="checkbox"/> Other _____ </div> </div> | | | | <div style="display: flex; justify-content: space-between;"> <div> Sample Disposal <input type="checkbox"/> Return to Client <input type="checkbox"/> Disposal By Lab <input type="checkbox"/> Archive For _____ Months </div> <div> QC Requirements (Specify) 1. Received By _____ Date _____ 2. Received By _____ Date _____ 3. Received By _____ Date _____ </div> </div> | | | | <div style="display: flex; justify-content: space-between;"> <div> Possible Hazard Identification <input type="checkbox"/> Non-Hazard <input type="checkbox"/> Flammable <input type="checkbox"/> Skin Irritant <input type="checkbox"/> Poison B <input type="checkbox"/> Unknown </div> <div> Turn Around Time Required <input type="checkbox"/> 24 Hours <input type="checkbox"/> 48 Hours <input type="checkbox"/> 7 Days <input type="checkbox"/> 14 Days <input type="checkbox"/> 21 Days <input type="checkbox"/> Other _____ </div> </div> | | | |
| | | | | | | | | | | | |
| <div style="display: flex; justify-content: space-between;"> <div> Possible Hazard Identification <input type="checkbox"/> Non-Hazard <input type="checkbox"/> Flammable <input type="checkbox"/> Skin Irritant <input type="checkbox"/> Poison B <input type="checkbox"/> Unknown </div> <div> Turn Around Time Required <input type="checkbox"/> 24 Hours <input type="checkbox"/> 48 Hours <input type="checkbox"/> 7 Days <input type="checkbox"/> 14 Days <input type="checkbox"/> 21 Days <input type="checkbox"/> Other _____ </div> </div> | | | | <div style="display: flex; justify-content: space-between;"> <div> Sample Disposal <input type="checkbox"/> Return to Client <input type="checkbox"/> Disposal By Lab <input type="checkbox"/> Archive For _____ Months </div> <div> QC Requirements (Specify) 1. Received By _____ Date _____ 2. Received By _____ Date _____ 3. Received By _____ Date _____ </div> </div> | | | | <div style="display: flex; justify-content: space-between;"> <div> Possible Hazard Identification <input type="checkbox"/> Non-Hazard <input type="checkbox"/> Flammable <input type="checkbox"/> Skin Irritant <input type="checkbox"/> Poison B <input type="checkbox"/> Unknown </div> <div> Turn Around Time Required <input type="checkbox"/> 24 Hours <input type="checkbox"/> 48 Hours <input type="checkbox"/> 7 Days <input type="checkbox"/> 14 Days <input type="checkbox"/> 21 Days <input type="checkbox"/> Other _____ </div> </div> | | | |
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| <div style="display: flex; justify-content: space-between;"> <div> Possible Hazard Identification <input type="checkbox"/> Non-Hazard <input type="checkbox"/> Flammable <input type="checkbox"/> Skin Irritant <input type="checkbox"/> Poison B <input type="checkbox"/> Unknown </div> <div> Turn Around Time Required <input type="checkbox"/> 24 Hours <input type="checkbox"/> 48 Hours <input type="checkbox"/> 7 Days <input type="checkbox"/> 14 Days <input type="checkbox"/> 21 Days <input type="checkbox"/> Other _____ </div> </div> | | | | <div style="display: flex; justify-content: space-between;"> <div> Sample Disposal <input type="checkbox"/> Return to Client <input type="checkbox"/> Disposal By Lab <input type="checkbox"/> Archive For _____ Months </div> <div> QC Requirements (Specify) 1. Received By _____ Date _____ 2. Received By _____ Date _____ 3. Received By _____ Date _____ </div> </div> | | | | <div style="display: flex; justify-content: space-between;"> <div> Possible Hazard Identification <input type="checkbox"/> Non-Hazard <input type="checkbox"/> Flammable <input type="checkbox"/> Skin Irritant <input type="checkbox"/> Poison B <input type="checkbox"/> Unknown </div> <div> Turn Around Time Required <input type="checkbox"/> 24 Hours <input type="checkbox"/> 48 Hours <input type="checkbox"/> 7 Days <input type="checkbox"/> 14 Days <input type="checkbox"/> 21 Days <input type="checkbox"/> Other _____ </div> </div> | | | |
| | | | | | | | | | | | |
| <div style="display: flex; justify-content: space-between;"> <div> Possible Hazard Identification <input type="checkbox"/> Non-Hazard <input type="checkbox"/> Flammable <input type="checkbox"/> Skin Irritant <input type="checkbox"/> Poison B <input type="checkbox"/> Unknown </div> <div> Turn Around Time Required <input type="checkbox"/> 24 Hours <input type="checkbox"/> 48 Hours <input type="checkbox"/> 7 Days <input type="checkbox"/> 14 Days <input type="checkbox"/> 21 Days <input type="checkbox"/> Other _____ </div> </div> | | | | <div style="display: flex; justify-content: space-between;"> <div> Sample Disposal <input type="checkbox"/> Return to Client <input type="checkbox"/> Disposal By Lab <input type="checkbox"/> Archive For _____ Months </div> <div> QC Requirements (Specify) 1. Received By _____ Date _____ 2. Received By _____ Date _____ 3. Received By _____ Date _____ </div> </div> | | | | <div style="display: flex; justify-content: space-between;"> <div> Possible Hazard Identification <input type="checkbox"/> Non-Hazard <input type="checkbox"/> Flammable <input type="checkbox"/> Skin Irritant <input type="checkbox"/> Poison B <input type="checkbox"/> Unknown </div> <div> Turn Around Time Required <input type="checkbox"/> 24 Hours <input type="checkbox"/> 48 Hours <input type="checkbox"/> 7 Days <input type="checkbox"/> 14 Days <input type="checkbox"/> 21 Days <input type="checkbox"/> Other _____ </div> </div> | | | |
| | | | | | | | | | | | |
| <div style="display: flex; justify-content: space-between;"> <div> Possible Hazard Identification <input type="checkbox"/> Non-Hazard <input type="checkbox"/> Flammable <input type="checkbox"/> Skin Irritant <input type="checkbox"/> Poison B <input type="checkbox"/> Unknown </div> <div> Turn Around Time Required <input type="checkbox"/> 24 Hours <input type="checkbox"/> 48 Hours <input type="checkbox"/> 7 Days <input type="checkbox"/> 14 Days <input type="checkbox"/> 21 Days <input type="checkbox"/> Other _____ </div> </div> | | | | <div style="display: flex; justify-content: space-between;"> <div> Sample Disposal <input type="checkbox"/> Return to Client <input type="checkbox"/> Disposal By Lab <input type="checkbox"/> Archive For _____ Months </div> <div> QC Requirements (Specify) 1. Received By _____ Date _____ 2. Received By _____ Date _____ 3. Received By _____ Date _____ </div> </div> | | | | <div style="display: flex; justify-content: space-between;"> <div> Possible Hazard Identification <input type="checkbox"/> Non-Hazard <input type="checkbox"/> Flammable <input type="checkbox"/> Skin Irritant <input type="checkbox"/> Poison B <input type="checkbox"/> Unknown </div> <div> Turn Around Time Required <input type="checkbox"/> 24 Hours <input type="checkbox"/> 48 Hours <input type="checkbox"/> 7 Days <input type="checkbox"/> 14 Days <input type="checkbox"/> 21 Days <input type="checkbox"/> Other _____ </div> </div> | | | |
| | | | | | | | | | | | |

Figure 23-2. Example: Sample Acceptance Policy

TestAmerica St. Louis Sample Acceptance Policy

NELAC specifies requirements under which any NELAC accredited laboratory will accept samples. STL St. Louis will review your sample shipment against those requirements listed below, and will communicate any discrepancies to you. Your project manager will assist you in the appropriate resolution of any issues related to sample receipt. Please contact your project manager with any questions.

When completing the chain of custody form, sign your name in the "relinquished by" box.

NELAC requirements are as follows:

- Proper, full and complete documentation, which includes sample identification, the location, date and time of collection, the collector's name, the preservation type, the sample matrix type, the requested testing method, and any special remarks concerning the samples shall be provided.
- Each sample shall be labeled with unique, durable and indelible identification.
- The samples shall be collected in the appropriate sample containers.
- The samples shall arrive at the laboratory within the specified holding time for the analyses requested.
- Sufficient sample volume must be available to perform the requested analyses.
- The laboratory will notify the client upon sample receipt if the samples exhibit obvious signs of damage, contamination or inadequate preservation.

DoD QSM SAMPLE ACCEPTANCE POLICY:

NELAC specifies requirements under which any NELAC accredited laboratory will accept samples. TestAmerica St. Louis will review your sample shipment against those requirements listed below, and will communicate any discrepancies to you. Your project manager will assist you in the appropriate resolution of any issues related to sample receipt. Please contact your project manager with any questions.

When completing the chain of custody form, sign your name in the "relinquished by" box.

NELAC requirements are as follows:

- Proper, full and complete documentation, which includes sample identification, the location, date and time of collection, the collector's name, the preservation type, the sample matrix type, the requested testing method, and any special remarks concerning the samples shall be provided.
- Each sample shall be labeled with unique, durable and indelible identification.
- The samples shall be collected in the appropriate sample containers.
- The samples shall arrive at the laboratory within the specified holding time for the analyses requested.
- Sufficient sample volume must be available to perform the requested analyses.

The laboratory will notify the client upon sample receipt if the samples exhibit obvious signs of damage, contamination or inadequate preservation. Samples shall be considered "compromised" if the following conditions are observed upon sample receipt:


- Cooler and/or samples are received outside of temperature specification.
- Samples are received broken or leaking.
- Samples are received beyond holding time.
- Samples are received without appropriate preservative.
- Samples are received in inappropriate containers.
- COC does not match samples received.
- COC is not properly completed or not received.
- Breakage of any Custody Seal.
- Apparent tampering with cooler and/or samples.
- Headspace in volatiles samples.
- Seepage of extraneous water or materials into samples.
- Inadequate sample volume.
- Illegible, impermanent, or non-unique sample labeling.

When "compromised" samples are received, it must be documented on a Condition Upon Receipt Form (CUR) for the project records and the client must be contacted for instructions. If the client decides to proceed with analysis, the project report shall clearly indicate any of the above conditions and the resolution.

If the conditions listed on the Acceptance Policy are not satisfactory and when lacking direction from the client to the contrary, the sample will be rejected.

For DoD QSM project work, sample containers must be certified to meet the "less than" ½ the RL criteria for the analytes of concern. Analytes for which this certification can not be obtained will be noted in the Case Narrative. Upon DoD project approval, the laboratory will analyze method blanks prepared in the containers of concern, qualify and narrate the sample analytes which do not meet the criteria, or take other appropriate action as determined by the DoD project site.

Figure 23-3. Example: Cooler Receipt Form



THE LEADER IN ENVIRONMENTAL TESTING

Lot #(s): _____

CUR Form #: _____


CONDITION UPON RECEIPT FORM

Client: _____

Quote No: _____

COC/RFA No: _____

Initiated By: _____ Date: _____ Time: _____



Shipping Information

Shipper: FedEx UPS DHL Courier Client Other: _____

Shipping # (s):*

| | |
|----------|-----------|
| 1. _____ | 6. _____ |
| 2. _____ | 7. _____ |
| 3. _____ | 8. _____ |
| 4. _____ | 9. _____ |
| 5. _____ | 10. _____ |

Multiple Packages: Y N

Sample Temperature (s):**

| | |
|----------|-----------|
| 1. _____ | 6. _____ |
| 2. _____ | 7. _____ |
| 3. _____ | 8. _____ |
| 4. _____ | 9. _____ |
| 5. _____ | 10. _____ |

*Numbered shipping lines correspond to Numbered Sample Temp lines

**Sample must be received at 4°C ± 2°C- If not, note contents below. Temperature variance does NOT affect the following: Metals-Liquid; Rad tests- Liquid or Solids; Perchlorate

Condition (Circle "Y" for yes; "N" for no and "N/A" for not applicable):

| | | | | | |
|----|---------|--|-----|---------|---|
| 1. | Y N | Are there custody seals present on the cooler? | 8. | Y N | Are there custody seals present on bottles? |
| 2. | Y N N/A | Do custody seals on cooler appear to be tampered with? | 9. | Y N N/A | Do custody seals on bottles appear to be tampered with? |
| 3. | Y N | Were contents of cooler frisked after opening, but before unpacking? | 10. | Y N N/A | Was sample received with proper pH? (if not, make note below) |
| 4. | Y N | Sample received with Chain of Custody? | 11. | Y N N/A | Containers for C-14, H-3 & I-129/131 marked with "Do Not Preserve" label? |
| 5. | Y N N/A | Does the Chain of Custody match sample ID's on the container(s)? | 12. | Y N | Sample received in proper containers? |
| 6. | Y N | Was sample received broken? | 13. | Y N N/A | Headspace in VOA or TOX liquid samples? (If Yes, note sample ID's below) |
| 7. | Y N | Is sample volume sufficient for analysis? | 14. | Y N N/A | Was Internal COC/Workshare received? |

¹ For DOE-AL (Pantex, LANL, Sandia) sites, pH of ALL containers received must be verified, EXCEPT VOA, TOX, Oil & Grease and soils

Notes: _____

Corrective Action:

☐ Client Contact Name: _____ Informed by: _____

☐ Sample(s) processed "as is"

☐ Sample(s) on hold until: _____ If released, notify: _____

Project Management Review: _____ Date: _____

THIS FORM MUST BE COMPLETED AT THE TIME THE ITEMS ARE BEING CHECKED IN. IF ANY ITEM IS COMPLETED BY SOMEONE OTHER THAN THE INITIATOR, THEN THAT PERSON IS REQUIRED TO APPLY THEIR INITIAL AND THE DATE NEXT TO THAT ITEM.

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SECTION 24. ASSURING THE QUALITY OF TEST RESULTS

24.1 Overview

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS), tracers and carriers). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. Quality control samples are to be treated in the **exact** same manner as the associated field samples being tested. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance. PT samples must be evaluated the same as regular environmental samples. The laboratory shall employ the same quality control, sequence of analytical steps, and replicates as used when analyzing routine samples.

24.2 Controls

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

24.3 Negative Controls

Table 24-1. Example – Negative Controls

| Control Type | Details |
|--------------------|---|
| Method Blank (MB) | <p>are used to assess preparation and analysis for possible contamination during the preparation and processing steps.</p> <p>The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.</p> <p>The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples.</p> <p>The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).</p> <p>Reanalyze or qualify associated sample results when the concentration of a targeted analyte in the blank is at or above the reporting limit as established by the method or by regulation, AND is greater than $1/10$ of the amount measured in the sample.</p> |
| Calibration Blanks | are prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve. |
| Instrument Blanks | are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content. |

Table 24-1. Example – Negative Controls

| Control Type | Details |
|-------------------------------|--|
| Trip Blank ¹ | are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses (or as specified in the client's project plan). Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples. |
| Field Blanks ¹ | are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER) |
| Equipment Blanks ¹ | are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC) |
| Holding Blanks | also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory |

¹ When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

24.4 Positive Controls

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous

volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard is reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).

The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

- For methods that have 1-10 target analytes, spike all components.
- For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
- For methods with more than 20 target analytes, spike at least 16 components.
- Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
- Exception: Due to analyte incompatibility between the various PCB aroclors, aroclors 1016 and 1260 are used for spiking as they cover the range of all of the aroclors. Specific Aroclors may be used by request on a project specific basis.

24.5 Sample Matrix Controls

Table 24-2. Sample Matrix Control

| Control Type | Details |
|--------------|---------|
|--------------|---------|

Table 24-2. Sample Matrix Control

| Control Type | Details | |
|-------------------------|--------------------------------|---|
| Matrix Spikes (MS) | Use | Used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used; |
| | Typical Frequency ¹ | At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects. If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details |
| | Description | Essentially a sample fortified with a known amount of the test analyte(s). |
| Surrogate | Use | Measures method performance to sample matrix (organics only). |
| | Typical Frequency ¹ | Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery. |
| | Description | Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples. |
| Duplicates ² | Use | For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure. |
| | Typical Frequency ¹ | Duplicate samples are usually analyzed with methods that do not require matrix spike analysis. |
| | Description | Performed by analyzing two aliquots of the same field sample independently or an additional LCS. |
| Internal Standards | Use | Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements. |
| | Typical Frequency ¹ | All organic and ICP methods as required by the analytical method. |
| | Description | Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance. |
| Tracers and Carriers | Use | Chemically mimic and do not interfere with the target analytes through radiochemical separations. Isotopic tracers are typically radioactive materials while carriers are typically non-radioactive |
| | Typical Frequency ¹ | Added to each client sample, method blank, LCS and matrix QC sample, as required by the specific method. |
| | Description | Added to samples to determine the overall chemical yield of the analytical preparation steps. Each sample is spiked separately with the same material and individual sample yields are determined. The tracer/carrier is added to the sample at the very beginning of the preparation steps. For solid samples the tracer/carrier is added after grinding, but before muffling or dissolution. |

¹ See the specific analytical SOP for type and frequency of sample matrix control samples.

² LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

24.6 Acceptance Criteria (Control Limits)

As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits

with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

Once control limits have been established, they are verified, reviewed, and updated if necessary on a semi-annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

Laboratory generated % Recovery acceptance (control) limits are generally established by taking ± 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

- Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV) (unless the analytical method specifies a tighter limit).
- In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.
- The lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable and identifiable.
- The maximum acceptable recovery limit will be 150%.
- The maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 10%.
- If either the high or low end of the control limit changes by $\leq 5\%$ from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

24.6.1 The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits. The QA department can generate a Quality Control Limit summary that contains tables that summarize the precision and accuracy acceptability limits for the analyses performed at TestAmerica St. Louis. The information is stored in the LIMS and includes an effective date and is updated each time new limits are generated. Unless otherwise noted, these limits are laboratory generated. The limits are approved in the LIMS system after review by the QA department. The LIMS maintains an archive of all limits used in the laboratory. Historical limits can be found in the LIMS program. See laboratory SOP ST-QA-0014, "Evaluation of Analytical Accuracy and Precision through the Use of Control Charts".

24.6.2 A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

- The analyte results are below the reporting limit and the LCS is above the upper control limit.
- If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

Or, for NELAC and Department of Defense (DoD) work, there are an allowable number of Marginal Exceedances (ME):

| | |
|------------------|-------------------------------------|
| <11 analytes | 0 marginal exceedances are allowed. |
| 11 – 30 Analytes | 1 marginal exceedance is allowed |
| 31-50 Analytes | 2 marginal exceedances are allowed |
| 51-70 Analytes | 3 marginal exceedances are allowed |
| 71-90 Analytes | 4 marginal exceedances are allowed |
| > 90 Analytes | 5 marginal exceedances are allowed |

- Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (NELAC).
- Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken. The laboratory has a system to monitor marginal exceedances to ensure that they are random.

Though marginal exceedances may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

24.6.3 If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.

24.6.4 If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of

the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis.

24.6.5 If radiochemical tracer or carrier recovery is outside limits the sample is re-analyzed to confirm matrix interference. If recoveries confirm, or there was obvious interference, results are reported from the original run and a note is included with the case narrative. If the re-analysis meets the recovery criteria, the second run is reported (or both are reported if requested by the client). When samples are non-detect for the target analytes and the carrier/tracer recovery indicates a high bias in the analysis, the samples are not re-run unless required by the client.

24.7 Additional Procedures to Assure Quality Control

The laboratory has written and approved method SOPs to assure the accuracy of the test method; including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples (see Section 15).

A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

- Use of formulae to reduce data is discussed in the method SOPs and in Section 20.
- Selection of appropriate reagents and standards is included in Section 9 and 21.
- A discussion on selectivity of the test is included in Section 5.
- Constant and consistent test conditions are discussed in Section 18.
- The laboratories sample acceptance policy is included in Section 23.

SECTION 25. REPORTING RESULTS

25.1 Overview

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 7. A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client. Review of reported data is included in Section 19.

25.2 Test Reports

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title (e.g. Analytical Report for Samples) with a “sample results” column header.

25.2.2 Each report cover page printed on company letterhead, which includes the laboratory name, address and telephone number.

25.2.3 A unique identification of the report (e.g. job number or SDG number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

Note: Page numbers of report are represented as page # of ##. Where the first number is the page number and the second is the total number of pages.

25.2.4 A copy of the chain of custody (COC)

- Any COCs involved with Subcontracting are included.
- Any additional addenda to the report must be treated in a similar fashion so it is a recognizable part of the report and cannot accidentally get separated from the report (e.g., Sampling information).

25.2.5 The name and address of client and a project name/number, if applicable.

25.2.6 Client project manager or other contact

25.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

25.2.8 Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

25.2.9 Date reported or date of revision, if applicable.

25.2.10 Method of analysis including method code (EPA, Standard Methods, etc).

25.2.11 Practical quantitation limits or reporting limit.

25.2.12 Method detection limits (if requested)

25.2.13 Definition of Data qualifiers and reporting acronyms (e.g. ND).

25.2.14 Sample results.

25.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.

25.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 25.2.4 regarding additional addenda).

25.2.17 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

25.2.18 A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory.

25.2.19 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue; authorized signatories are qualified Project Managers appointed by the Manager of Project Managers.

25.2.20 When NELAC accreditation is required, the lab shall certify that the test results meet all requirements of TNI or provide reasons and/or justification if they do not.

25.2.21 A narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

25.2.22 When soil samples are analyzed, a specific identification as to whether soils are reported on a "wet weight" or "dry weight" basis.

25.2.23 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

25.2.24 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., preliminary data). A complete report must be sent once all of the work has been completed.

25.2.25 Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

25.2.26 A clear statement notifying the client that non-accredited tests were performed and directing the client to the laboratory's accreditation certificates of approval shall be provided when non-accredited tests are included in the report.

Note: Refer to the Corporate SOP on Electronic Reporting and Signature Policy (No. CA-I-P-002) for details on internally applying electronic signatures of approval.

25.3 Reporting Level or Report Type

The laboratory offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level I is a report with all of the elements outlined in Section 25.2 above, excluding 25.2.15 (QC data).
- Level II is a Level I report plus summary information, including results for the method blank reported to the laboratory MDL, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form and as an electronic (pdf) file. Initial reports may be provided to clients by facsimile. Procedures used to ensure client confidentiality are outlined in Section 25.6.

25.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services. TestAmerica St. Louis offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), New Agency Standard (NAS), Format A, Excel, Dbase, GISKEY, and Text Files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

25.4 Supplemental Information for Test

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report.

Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature.

Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

Note: Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This necessitates a limited scope of interpretation, and this work is performed by the QA Department. This is the only form of "interpretation" of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

25.5 Environmental Testing Obtained From Subcontractors

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the Corporate SOP on Subcontracting (SOP No. CA-L-S-002).

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of TestAmerica are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

25.6 Client Confidentiality

In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the Client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Note: This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

25.6.1 Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

This material is intended only for the use of the individual(s) or entity to whom it is addressed, and may contain information that is privileged and confidential. If you are not the intended recipient, or the employee or agent responsible for delivering this material to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify us immediately by telephone at the 1-800-765-0980 (or for e-mails: please notify us immediately by e-mail or by phone (1-800-765-0980) and delete this material from any computer).

25.7 Format of Reports

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.8 Amendments to Test Reports

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained on the Archive data server, as is the original report. The revised report is stored in the Archive data server under the job number/SDG number followed by "rev".

When the report is re-issued, a notation of "Revised" is placed on the cover/signature page of the report *and at the top of the narrative page* with a brief explanation of reason for the re-issue.

25.9 Policies on Client Requests for Amendments

25.9.1 Policy on Data Omissions or Reporting Limit Increases

Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error
- Sample identification is indeterminate (confusion between COC and sample labels).

- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely no possible impact on the interpretation of the analytical results and there is no possibility of the change being interpreted as misrepresentation by anyone inside or outside of our company.

25.9.2 Multiple Reports

TestAmerica does not issue multiple reports for the same work order where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

SECTION 26. REVISION HISTORY

26.1 CHANGES TO REVISION 0

- 26.1.1 Updated to conform to new corporate Template. Information that was specific to the company at large and less specific to the individual laboratory was removed from the template and is now found in the Corporate Quality Management Plan (CQMP).
- 26.1.2 The Quality Policy Statement was updated to include compliance with NELAC standards.
- 26.1.3 Section 10 (Services to Client) was merged with Section 7 (renamed)
- 26.1.4 Section 10 was left intentionally blank.
- 26.1.5 Section 16 (Audits) was given new text.
- 26.1.6 Section 17 (Management Reviews) revised QA report section, some tables were removed
- 26.1.7 Section 21 (Calibrations) removed information that can be found in method SOPs
- 26.1.8 Radiochemistry calculations in Appendix 6 were updated
- 26.1.9 Tables, figures and appendices were updated and re-numbered

26.2 CHANGES TO REVISION 1(06/02/09)

- 26.2.1 Added reference to ASME NQA-1-2000 to Section 3.1
- 26.2.2 Updated Ethics Agreement in Appendix 1
- 26.2.3 Updated radiochemistry calculations in Appendix 6.

26.3 CHANGES TO REVISION 2 (08/31/09)

- 26.3.1 Added reference to DoD QSM 4.1 to Section 3.1
- 26.3.2 Updated QA Manager job description in Section 4.2.3
- 26.3.3 Updated laboratory organizational chart
- 26.3.4 Added Quality Program objectives to Section 5.1; clarified staff responsibilities regarding QA documents
- 26.3.5 Added QAM review cycle to Table 16-1
- 26.3.6 Added freezer temperature criteria to Section 21.3.4
- 26.3.7 Updated Calibration information in Table 21-3
- 26.3.8 Added current Florida NELAC cert to Appendix 3
- 26.3.9 Signatures moved from Title Page to Cover per DoD Requirements

26.4 CHANGES TO REVISION 3 (08/31/10)

- 26.4.1 Section 2: list of Cross-walk references to the ISO 17025 requirements added
- 26.4.2 Section 4.2: QA Manager responsibilities updated
- 26.4.3 Section 4: Organizational Charts updated in figure 4-1
- 26.4.4 Section 5.1: Addition to quality Policy Statement regarding continuous improvement
- 26.4.5 Section 7: Figure 7-1 removed
- 26.4.6 Section 13: Table 13-3 "General Corrective Actions" added
- 26.4.7 Section 13.3.3: Root cause analysis added
- 26.4.8 Sections 3.1 & 20.4: Source methods references updated
- 26.4.9 Section 18.3: Evidence of successful training added
- 26.4.10 Section 20.15.5: text on manual integrations and Mint Miner[®] expanded
- 26.4.11 Section 21: Table 21-1 "instrument List", updated
- 26.4.12 Section 21.3.5: requirement for non-volumetric labware added
- 26.4.13 Section 21.4: calibration standards section expanded
- 26.4.14 Section 24.2.2: Unique sample ID section added

- 26.4.15** Section 24.3: Sample Acceptance Policy moved to appear in Table of Contents
- 26.4.16** Section 24.6: added note on Trip blanks
- 26.4.17** Section 26.2.18: added narrative requirement reproduction of laboratory reports
- 26.4.18** Information in Appendices 1,2,3,5 & 7 updated
- 26.4.19** Added "End of Document" statement
- 26.4.20** General grammatical edits and corrections

- 26.5** **CHANGES TO REVISION 4**
- 26.5.1** 10/08/10: Added Section 20.4.2.4 to address DOCs for tests without analyte spikes
- 26.5.2** 8/31/11: Removed the 'effective date' by section and applied it to the entire document. Continuous document pagination implemented.
- 26.5.3** 2009 TNI Standard references added to the Table of Contents only – citations removed from the section titles within the document. Updated all references from the 2003 NELAC Standards to the 2009 TNI standard
- 26.5.4** Use of the title 'Technical Manager' from the TNI Standard is defined and implemented.
- 26.5.5** Section 10 (previously left empty) removed. Other section numbers adjusted accordingly.
- 26.5.6** Section 4: Additional Quality Assurance and Technical Manager (a.k.a., Supervisors) responsibilities assigned based on the TNI Standard
- 26.5.7** Section 8: Clarification of subcontracting procedures
- 26.5.8** Table 12-1: Updated for additional corrective action procedures
- 26.5.9** Section 15: Updates reflect current internal audit process as defined in CA-Q-S-004. Table 15-1 updated.
- 26.5.10** Section 19: Verification of MDLs/RLs updated to TNI Standard
- 26.5.11** Section 25: added statement regarding the listing of non-accredited methods in the lab report
- 26.5.12** Appendix 2: updated laboratory floor plan
- 26.5.13** Appendix 4: added/removed glossary terms/acronyms
- 26.5.14** Appendix 5: Certification table updated
- 26.5.15** Appendix 6: updated and clarified calculations
- 26.5.16** Appendix 7: updated SOP list

- 26.6** **CHANGES TO REVISION 5**
- 26.6.1** Grammatical and format corrections made throughout entire document
- 26.6.2** Updated signature page
- 26.6.3** REFERENCED CORPORATE SOPs AND POLICIES updated
- 26.6.4** Section 4.3: Deputies updated
- 26.6.5** Figure 4-1 Corporate and Laboratory Organization Charts updated
- 26.6.6** Section 5.5: Criteria for Quality Indicators updated
- 26.6.7** Changed TNI to NELAC where applicable
- 26.6.8** Section 9.3.3: Specifications: updated compressed gasses paragraph
- 26.6.9** Replaced Clouseau with LIMS where applicable
- 26.6.10** Section 11.2: Responsibilities and Authorities removed COO
- 26.6.11** Section 12: Removed Clouseau screen shots
- 26.6.12** Section 14: Replaced reference to standards log program with LIMS
- 26.6.13** Section 15: updated reference to Internal Auditing SOP to CA-Q-S-003
- 26.6.14** Section 15: Added Audit Planning/Reporting section
- 26.6.15** Sections 19.15.2 & 19.15.3: updated

- 26.6.16 Section 20.2: Added "tagged-out" requirements
- 26.6.17 Table 20-1, 20-2, 20-4 updated
- 26.6.18 Section 22.5: Addition of aqueous sample aliquot density requirement and volume determination
- 26.6.19 Section 23.2.1.1: Replaced QuantIMS with TALS unique sample identification.
- 26.6.20 Section 23.3: Updated to indicate that variation from policy to be noted in case narrative
- 26.6.21 Section 24.6.1: updated to reference LIMS instead of QC Browser
- 26.6.22 Appendix 3: updated NELAC certification
- 26.6.23 Appendix 4: added new glossary terms and acronyms
- 26.6.24 Appendix 5: updated St. Louis certifications
- 26.6.25 Appendix 6: added organic calculation "On column concentrations"
- 26.6.26 Appendix 7: updated laboratory SOP listing

26.7 CHANGES TO REVISION 6

- 26.7.1 Section 3.1, updated references
- 26.7.2 Section 4.1, changed Chief Operating Officer to Chief Executive Officer
- 26.7.3 Section 4.2, updated QA Manager, Technical Manager and Technical Director Responsibilities
- 26.7.4 Section 4.3, updated responsibilities table of key personnel
- 26.7.5 Figure 4-1, updated Corporate and Lab Org Chart
- 26.7.6 Table 14-1, removed 7 year requirement and replaced it with reference to HR Manual
- 26.7.7 Section 19.13.4, revised explanation of the meaning of the lab's uncertainty statement to more closely conform to A2LA and NIST language
- 26.7.8 Table 20-4, updated to reflect practice
- 26.7.9 Section 24.1, statement added to clarify and emphasize treatment of QC samples and PT samples
- 26.7.10 Appendix 3: updated NELAC certification
- 26.7.11 Appendix 5: updated St. Louis certifications
- 26.7.12 Appendix 6: updated calculations
- 26.7.13 Appendix 7: updated SOP listing

26.8 CHANGES TO REVISION 7 (02/02/2015)

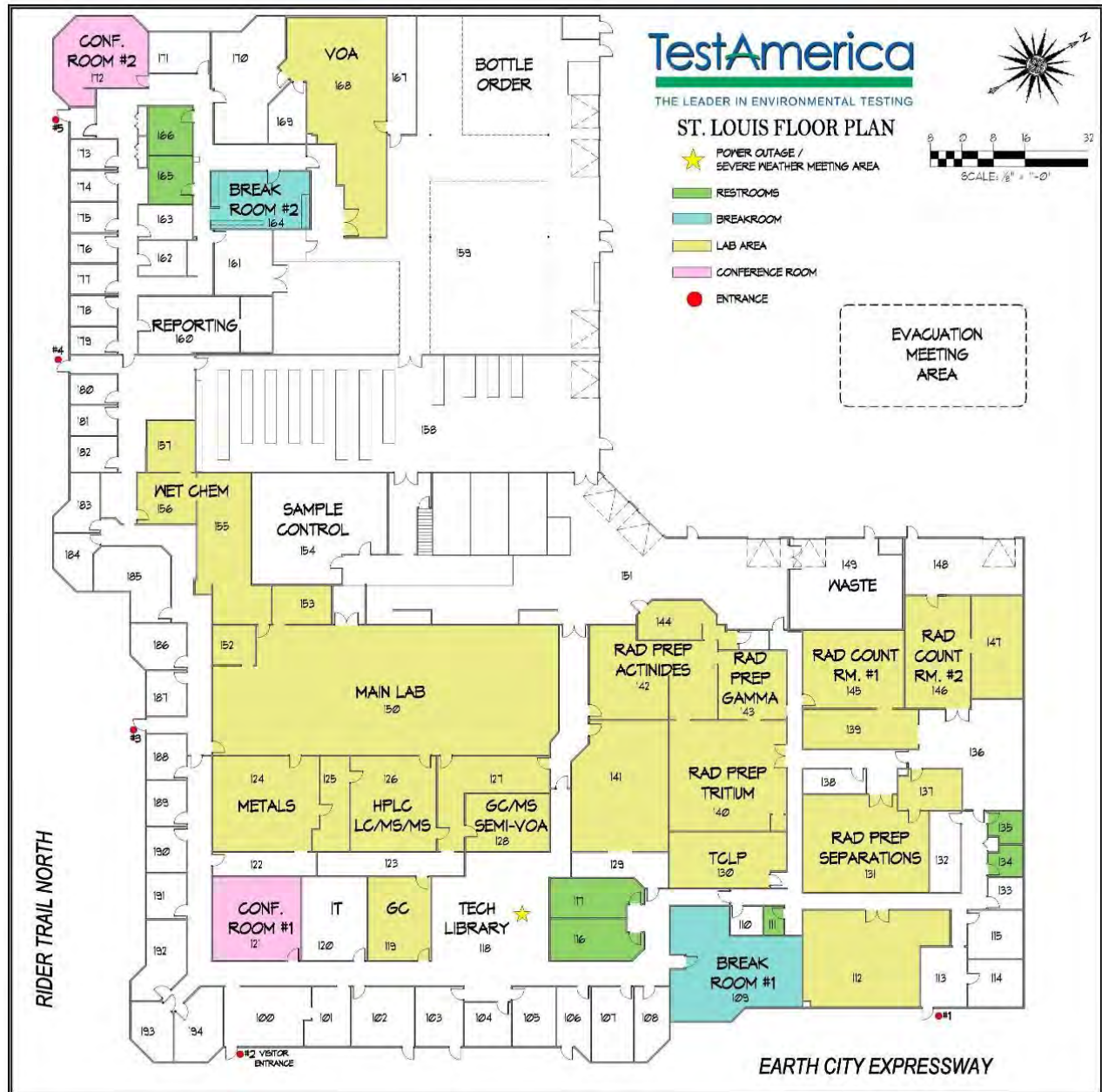
- 26.8.1 Section 4.3, updated Key Personnel Deputy table
- 26.8.2 Figure 4-1, updated organizational charts
- 26.8.3 Section 17.3, added reference to see SOP ST-QA-0044 Training
- 26.8.4 Table 20-3, updated Example: Periodic Calibration
- 26.8.5 Appendix 5, update lab certifications, accreditations, validations

26.9 CHANGES TO REVISION 8 (05/23/2016)

- 26.9.1 Updated signatures page
- 26.9.2 Removed appendices: Ethics and confidentiality agreements; NELAC/TNI certified test
- 26.9.3 Updated Corporate SOPs and Policies table as well as references throughout
- 26.9.4 Added reference in section 3 to DOE Order 414.1D
- 26.9.5 Updated corporate titles throughout
- 26.9.6 Updated deputies, section 4.3
- 26.9.7 Updated Org charts

- 26.9.8** Updated section 5.5 with name of the app used
- 26.9.9** Section 8.2.3, changed responsibilities from QAM to CSO
- 26.9.10** Section 8.3 updated
- 26.9.11** Section 9.3 & 9.3.2 updated
- 26.9.12** Section 9.5 updated
- 26.9.13** Section 11.2 & 11.3 updated
- 26.9.14** Section 12.3.4 added information about iCAT
- 26.9.15** Section 13, added list of opportunities for improvement
- 26.9.16** Table 15-1, added SOP Method Compliance
- 26.9.17** Section 15.1.3 updated
- 26.9.18** Section 16.3 updated
- 26.9.19** Section 20.3.3 added guidance of when a single point or range is required
- 26.9.20** Section 25.2.19 changed from LD to PM as the one that appoints
- 26.9.21** Section 25.3 added detail about Level I

Appendix 1. Laboratory Floor Plan



Appendix 2. Glossary/Acronyms

Glossary:

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Activity, of radionuclides: The expected number of spontaneous nuclear decays (transformations) in unit time from a specified energy state (excluding prompt decays from a lower nuclear level) for a given amount of a radionuclide. Its standard unit (SI) is the Becquerel (Bq), where one Bq equals one decay per second. Activity has often been expressed in curies (Ci), where 3.7×10^{10} Bq equals 1 Ci, exactly. (ANSI)

Aliquot: A discrete, measured, representative portion of a sample taken for analysis. (QSM)

Analysis: A combination of sample preparation and instrument determination. (QSM)

Analyst: The designated individual who performs the “hands-on” analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analyte: The specific chemicals or components for which a sample is analyzed; it may be a group of chemicals that belong to the same chemical family and are analyzed together. (QSM)

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (NELAC)

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (NELAC)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (NELAC)

Background: Ambient signal response recorded by measurement instruments that are independent of radioactivity contributed by the radionuclides being measured in the sample. (ANSI)

Batch: Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one (1) to twenty (20) environmental samples of the same quality systems matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be twenty-four (24) hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) and/or those samples not requiring preparation, which are analyzed together

as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples. (NELAC)

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (NELAC)

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (NELAC)

- 1) In calibration of support equipment the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).
- 2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve: The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

Calibration Standard (Source): A substance or reference material used to calibrate an instrument (QAMS)

Carrier: Carriers are stable counterparts of the radioactive isotope(s) to be measured. When used, carriers are added to all samples in an analytical batch so that each sample has a specific measurable QC parameter (yield). The carrier yield is used in the data calculation to correct for all sources of analytical losses. The term carrier can also be used for a non-radioactive compound added to assist in the isolation of the target analyte(s).

Certified Reference Material (CRM): A reference material

Chain of Custody (COC) Form: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. (NELAC)

Check source: a radioactive source, not necessarily traceable to a national standards body such as NIST in the USA that is used to confirm the continuing satisfactory operation of an instrument. (ASTM)

Clouseau: TestAmerica custom software developed to document, track and trend non-conformances throughout the laboratory. The software interfaces with the laboratory information management system, QuantIMS and the report narrative generating software, KATO, to provide the laboratory with a corrective action system.

Compromised Samples: Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified.

Confidential Business Information (CBI): Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. NELAC and its representatives agree to safe-guarding identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to Second Column Confirmation; Alternate wavelength; Derivatization; Mass spectral interpretation; Alternative detectors or Additional Cleanup procedures. (NELAC)

Conformance: An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Control Chart: A graphical representation of data taken from a repetitive measurement or process. Control charts may be developed for various characteristics, (e.g., mean, standard deviation, range, etc.) of the data.

“A control chart has two basic uses: (1) as a tool to judge if a process was in control, and (2) as an aid in achieving and maintaining statistical control. For applications related to radiation detection instrumentation or radiochemical processes, the mean (center line) value of a historical characteristic (e.g., mean detector response), subsequent data values and control limits placed symmetrically above and below the center line are displayed on a control chart.” (MARLAP)

Count rate: The rate at which detector pulses are being registered in a selected voltage interval. The unit is reciprocal seconds (i.e., s^{-1}). Generally the count rate is uncorrected for detector efficiency. The count rate divided by the detector efficiency for a specific particle and energy will yield the source activity.

Count time: The time interval for the counting of a sample or source by a radiation detector. Depending upon the context used, this can be either the “clock” time (the entire period required to count the sample), or “live” time (the period during which the detector is actually counting). Live time is always less than or equal to clock time. (MARLAP)

Continuing Calibration Verification: The verification of the initial calibration. Required prior to sample analysis and at periodic intervals. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and nonlinear calibration models. (QSM)

Correction: Actions necessary to correct or repair analysis specific non-conformances (e.g. the acceptance criteria for method specific QC and protocols as well as the associated corrective actions). The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis. No significant action is taken to change behavior, process or procedure.

Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402) A root cause analysis may not be necessary in all cases. (QSM)

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria).

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors, and collation into a more useable form. (NELAC)

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (NELAC)

Detection Limit (DL): The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration with 99% confidence. At the DL, the false positive rate (Type I error) is 1%. A DL may be used as the lowest concentration for reliably reporting a detection of a specific analyte in a specific matrix with a specific method with 99% confidence. (QSM)

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)

Duplicate Analyses: The analyses or measurements of the variable of interest performed identically on two sub-samples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Energy Calibration: The correlation of the multi-channel analyzer (MCA) channel number to decay photon energy, obtained from the location of peaks from known radioactive standards.

Equipment Blank: Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.

External Standard Calibration: Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

False Negative: A result that fails to identify (detect) an analyte or reporting an analyte to be present at or below a level of interest when the analyte is actually above the level of interest. (QSM)

False Positive: A result that erroneously identifies (detects) an analyte or reporting an analyte to be present above a level of interest when the analyte is actually present at or below the level of interest. (QSM)

Field Blank: Blank prepared in the field by filling a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Holding Times: The maximum time that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Initial Calibration Verification (ICV): Verifies the initial calibration with a standard obtained or prepared from a source independent of the source of the initial calibration standards to avoid potential bias of the initial calibration. (QSM)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (NELAC)

Internal Standard Calibration: Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank: A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Detection Limit (IDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is $\pm 100\%$. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Laboratory Information Management Systems (LIMS): The entirety of an electronic data system (including hardware and software) that collects, analyzes, stores, and archives electronic records and documents. (QSM)

Least Squares Regression (1st Order Curve): The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit(s) of Detection (LOD) [a.k.a., Method Detection Limit (MDL)]: A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. (NELAC)

QSM Clarification: The smallest concentration of a substance that must be present in a sample in order to be detected at the DL with 99% confidence. At the LOD, the false negative rate (Type II error) is 1%. A LOD may be used as the lowest concentration for reliably reporting a non-detect of a specific analyte in a specific matrix with a specific method at 99% confidence.

LOD Verification [a.k.a., MDL Verification]: A processed QC sample in the matrix of interest, spiked with the analyte at no more than 3X the LOD for single analyte tests and 4X the LOD for multiple analyte tests and processed through the entire analytical procedure.

Limit(s) of Quantitation (LOQ) [a.k.a., Reporting Limit]: The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. (NELAC)

QSM Clarification: The smallest concentration that produces a quantitative result with known and recorded precision and bias. For DoD/DOE projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard and within the calibration range.

(QS) Matrix: The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater effluents, and TCLP or other extracts.

Drinking Water: Any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-Aqueous Liquid: Any organic liquid with <15% settleable solids.

Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.

Air & Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (NELAC)

Matrix Spike (spiked sample or fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.

Measurement Uncertainty: An estimate of the error in a measurement often stated as a range of values that contain the true value, within a certain confidence level. The uncertainty generally includes many components which may be evaluated from experimental standard deviations based on repeated observations or by standard deviations evaluated from assumed probability distributions based on experience or other information. For DoD/DOE, a laboratory's Analytical Uncertainty (such as use of LCS control limits) can be reported as the minimum uncertainty. (QSM)

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Method Detection Limit: The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

Minimum Detectable Activity or Concentration (MDA/MDC): For radiological analyses it is the smallest amount of activity/concentration that can be detected given the conditions of a specific sample. It is reported at the 95% confidence interval, meaning that there is a 5% chance that a false signal was reported as activity/concentration and a 5% chance that the true activity/concentration went undetected.

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Non-conformance: An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Performance Audit: The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC)

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (NELAC)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (NELAC)

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (NELAC)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within specified acceptance criteria. (NELAC)

Operator Aid: A technical posting, other than formal procedures, rules, instructions (such as poster, operating manual, or notepad) that assists workers in routine tasks and are not required to be posted or displayed by any organization or procedure. All operator aids must be controlled by the facility.

Qualitative Analysis: Analysis designed to identify the components of a substance or mixture. (QSM)

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item or service is of the type of quality needed and expected by the client. (NELAC)

Quality Assurance [Project] Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control: The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against “out of control” conditions and ensuring that the results are of acceptable quality. (NELAC)

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. (NELAC)

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (NELAC)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC activities. (NELAC)

Quantitative Analysis: analysis designed to determine the amounts or proportions of the components of a substance. (QSM)

RadCapture: Software used to process and report radiochemical data.

Radioactive: exhibiting radioactivity or containing radionuclides. (MARLAP)

Radioactive decay: Process by which a spontaneous change in nuclear state takes place. This process is accompanied by the emission of energy and subatomic particles.

Radioactivity: spontaneous emission of radiation, either directly from unstable atomic nuclei or as a consequence of a nuclear reaction.

Radionuclide: a nuclide that is radioactive (capable of undergoing radioactive decay). (MARLAP)

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (NELAC)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Material: Material or substance one or more properties of which are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (NELAC)

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or a given location. (NELAC)

Reporting Limit: A customer-specified lowest concentration value that meets project requirements for quantitative data with known precision and bias for a specific analyte in a specific matrix. (QSM)

Sample Transfer Utility (STU): TestAmerica custom software developed to document and track samples through the laboratory. The software interfaces with the laboratory information management system, QuantIMS. STU employs barcode technology for rapid processing of sample transfer events including removal from storage, transfer between personnel and sample disposal.

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Second Order Polynomial Curve (Quadratic): The 2nd order curves are a mathematical calculation of a slightly curved line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2nd order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. (NELAC)

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

Spike: A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies. (NELAC)

Standard Deviation: the square root of a variance of a random variable. The variance is a measure of the variation of the observations within a measurement set. The standard deviation is often estimated using a set of measurements of the random variable. The standard deviation has the same units as the measured quantity and therefore, is particularly convenient when describing the variability of the measured quantity. (ANSI)

Standard Operating Procedure (SOP): A written document which details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks. (NELAC)

Storage Blank: A blank matrix stored with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

Systematic error: An error component that produces a fixed bias in the underlying expected value of a determination, from measurement to measurement. (ANSI)

Systems Audit (also Technical Systems Audit): A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Manager: A member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of accreditation and reporting of results

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (NELAC)

Tracer: Tracers are radioactive and/or massless. Where used, they are added to all samples in an analytical batch so that each sample has a specific measurable QC parameter (yield). Tracers are counted and the yield is used in data calculations to correct for and all sources of analytical loss.

Trip Blank: A blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Uncertainty: A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

Unethical actions: Deliberate falsification of analytical or quality control results, where failed method or contractual requirements are made to appear acceptable. (QSM)

Acronyms:

| | |
|------|--|
| %R | Percent Recovery |
| ANSI | American National Standards Institute |
| App | Application |
| ASTM | American Society for Testing and Materials |
| Bq | becquerel |
| CAR | Corrective Action Report |
| CCV | Continuing Calibration Verification |
| CF | Calibration Factor |
| CFR | Code of Federal Regulations |
| Ci | Curie |
| CLP | Contract Laboratory Program |
| CoA | Certificate of Analysis |
| COC | Chain of Custody |
| cpm | Counts per minute |
| cps | Counts per second |
| CRM | Certified reference material |
| CSU | Combined standard uncertainty |
| CWA | Clean Water Act |
| DEQ | Department of Environmental Quality |

| | |
|---------|--|
| DER | Duplicate Error Ratio |
| DOC | Demonstration of Capability |
| DoD | Department of Defense |
| DOE | Department of Energy |
| DOECAP | DOE Consolidated Audit Program |
| DOT | Department of Transportation |
| dpm | Disintegrations per minute |
| DQO | Data Quality Objectives |
| DUP | Duplicate |
| EDD | Electronic data deliverable |
| EHS | Environment, Health and Safety |
| EPA | Environmental Protection Agency |
| FWHM | Full width half maximum |
| GC | Gas Chromatography |
| GC/MS | Gas Chromatography/Mass Spectrometry |
| GFPC | Gas-flow Proportional Counter |
| HPGe | High-purity germanium |
| HPLC | High Performance Liquid Chromatography |
| ICP | Inductively Coupled Plasma Atomic Emission Spectroscopy |
| ICP-MS | ICP/Mass Spectrometry |
| ICV | Initial Calibration Verification |
| IDL | Instrument Detection Limit |
| iDOC | Initial Demonstration of Capability |
| IH | Industrial Hygiene |
| IS | Internal Standard |
| ISO | International Organization of Standardization |
| keV | Kilo electron volts |
| LAN | Local area network |
| LCL | Lower control limits |
| LCS | Laboratory Control Sample |
| LCSD | Laboratory Control Sample Duplicate |
| LIMS | Laboratory Information Management System |
| LLD | Lower Level of Detection |
| LOD | Limit of Detection |
| LLQ | Lower Level of Quantitation |
| LOQ | Limit of Quantitation (PQL) |
| LSC | Liquid scintillation counter |
| MAPEP | Mixed Analyte Performance Evaluation Program |
| MARLAP | Multi-Agency Radiological Laboratory Analytical Protocol |
| MCL | Maximum contaminant limit |
| MDA/MDC | Minimum Detectable Activity/Concentration |
| MDL | Method Detection Limit |
| MDLCK | MDL Check Standard |
| MDLV | MDL Verification Check Standard |
| ME | Marginal exceedance |
| MeV | Mega electron volts |
| MQC | Minimum quantifiable concentration |
| MQO | Measurement quality objective |
| MRL | Method Reporting Limit Check Standard |
| MS | Matrix Spike |
| MSD | Matrix Spike Duplicate |
| NCM | Non-conformance memo |
| NELAC | National Environmental Laboratory Accreditation Conference |
| NELAP | National Environmental Laboratory Accreditation Program |

| | |
|-------|---|
| NIST | National Institute of Standards and Technology |
| NVLAP | National Voluntary Laboratory Accreditation Program |
| pCi | picocurie |
| PE | Performance Evaluation |
| PT | Performance Testing |
| TNI | The NELAC Institute |
| QAM | Quality Assurance Manual |
| QA/QC | Quality Assurance / Quality Control |
| QAMS | Quality Assurance Management Systems |
| QAPP | Quality Assurance Project Plan |
| RCRA | Resource Conservation and Recovery Act |
| RDL | Required detection limit |
| RF | Response Factor |
| ROI | Region of interest |
| RPD | Relative Percent Difference |
| RPP | Radiation Protection Plan |
| RSD | Relative Standard Deviation |
| RSO | Radiation Safety Officer |
| SAP | Sample and analysis plan |
| SD | Standard Deviation |
| SDS | Safety Data Sheets |
| SMO | Sample Management Office |
| SOP | Standard Operating Procedure |
| SOW | Statement of work |
| SQC | Statistical quality control |
| SRM | Standard reference material |
| TAT | Turn-Around-Time |
| TCLP | Toxicity characteristic leaching procedure |
| TLD | Thermoluminescent dosimeter |
| TPU | Total propagated uncertainty |
| TSS | Total suspended solids |
| μohms | Resistivity unit of measure |
| WET | Whole effluent toxicity |
| WMP | Waste Management Plan |
| WP | Water pollution |
| VOA | Volatiles |
| VOC | Volatile Organic Compound |

Appendix 3: Laboratory Certifications, Accreditations, Validations

TestAmerica St. Louis maintains accreditations, certifications, and approvals with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:



TestAmerica Certifications

| Laboratory | Program | Authority | Identification | Expiration Date |
|-----------------------|---------------|-------------------|-----------------|-----------------|
| TestAmerica St. Louis | DoD ELAP | L-A-B | L2305 | 04/06/2019 |
| TestAmerica St. Louis | Federal | USDA | P330-07-00122 | 01/09/2017 |
| TestAmerica St. Louis | NELAP | Florida | EB7689 | 06/30/2016 |
| TestAmerica St. Louis | NELAP | Illinois | 003757 | 11/30/2016 |
| TestAmerica St. Louis | NELAP | Kansas | E-10236 | 07/31/2016 |
| TestAmerica St. Louis | NELAP | Louisiana | 04080 | 06/30/2016 |
| TestAmerica St. Louis | NELAP | Louisiana (DW) | LA160008 | 12/31/2016 |
| TestAmerica St. Louis | NELAP | New Jersey | MC002 | 06/30/2016 |
| TestAmerica St. Louis | NELAP | New York | 11616 | 03/31/2017 |
| TestAmerica St. Louis | NELAP | Pennsylvania | 68-00540 | 02/28/2017 |
| TestAmerica St. Louis | NELAP | Texas | T104704193-15-9 | 07/31/2016 |
| TestAmerica St. Louis | NELAP | Utah | MC000542015-7 | 07/31/2016 |
| TestAmerica St. Louis | NELAP | Virginia | 460230 | 06/14/2016 |
| TestAmerica St. Louis | NRC | NRC | 24-24817-01 | 12/31/2022 |
| TestAmerica St. Louis | State Program | Alaska | MC00054 | 06/30/2016 |
| TestAmerica St. Louis | State Program | California | 2886 | 03/31/2018 |
| TestAmerica St. Louis | State Program | Connecticut | PH-0241 | 03/31/2017 |
| TestAmerica St. Louis | State Program | Iowa | 373 | 12/01/2016 |
| TestAmerica St. Louis | State Program | Kentucky (DW) | 90125 | 12/31/2016 |
| TestAmerica St. Louis | State Program | Maryland | 310 | 09/30/2016 |
| TestAmerica St. Louis | State Program | Missouri | 780 | 06/30/2016 |
| TestAmerica St. Louis | State Program | Nevada | MC000542016-1 | 07/31/2016 |
| TestAmerica St. Louis | State Program | North Dakota | R207 | 06/30/2016 |
| TestAmerica St. Louis | State Program | Oklahoma | 9997 | 08/31/2016 |
| TestAmerica St. Louis | State Program | South Carolina | 85002001 | 06/30/2016 |
| TestAmerica St. Louis | State Program | Washington | C592 | 08/30/2016 |
| TestAmerica St. Louis | State Program | West Virginia DEP | 381 | 08/31/2016 |

* Certification Valid - Laboratory is Pending Renewal with the Program Authority

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The certificates and parameter lists (which may differ) are available, upon request, from a laboratory representative. For each organization or may be found on the corporate web site, the laboratory's public server, the final report review table, and in the following offices: QA, marketing, and project management.

Appendix 4: Calculations

Common Calculations

- Percent Recoveries (ICV, CCV, LCS, Surrogates) are calculated according to the equation:

$$\%R = 100 \left(\frac{\text{Found}}{\text{True}} \right)$$

- Tracers and Carriers

$$\text{Recovery}(\%) = \frac{\text{measured}}{\text{added} - \text{native}} \times 100$$

Where:

Measured is the amount of tracer/carrier measured

Added is the amount of tracer/carrier added (spiked) into the sample

Native is the amount of tracer/carrier analyte native to the sample

- Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = 100 \left(\frac{\text{SSR} - \text{SR}}{\text{SA}} \right)$$

Where:

SSR = Spike Sample Result

SR = Sample Result

SA = Spike Added

- The relative percent difference (RPD) of matrix spike/matrix spike duplicates is calculated according to the following equation:

$$RPD = 100 \left[\frac{|\text{MSD} - \text{MS}|}{\left(\frac{\text{MSD} + \text{MS}}{2} \right)} \right]$$

Where:

MS = determined spiked sample concentration

MSD = determined matrix spike duplicate concentration

- Due to the nature of radioactive decay (random process) and the fact that Radiochemistry results are reported down to (and below) the MDC, dual criterion are used for replicate precision. When significant activity (well above the MDC) is present for a nuclide in the sample, the best representation of replicate precision is the RPD (Relative Percent Difference), which is calculated as follows:

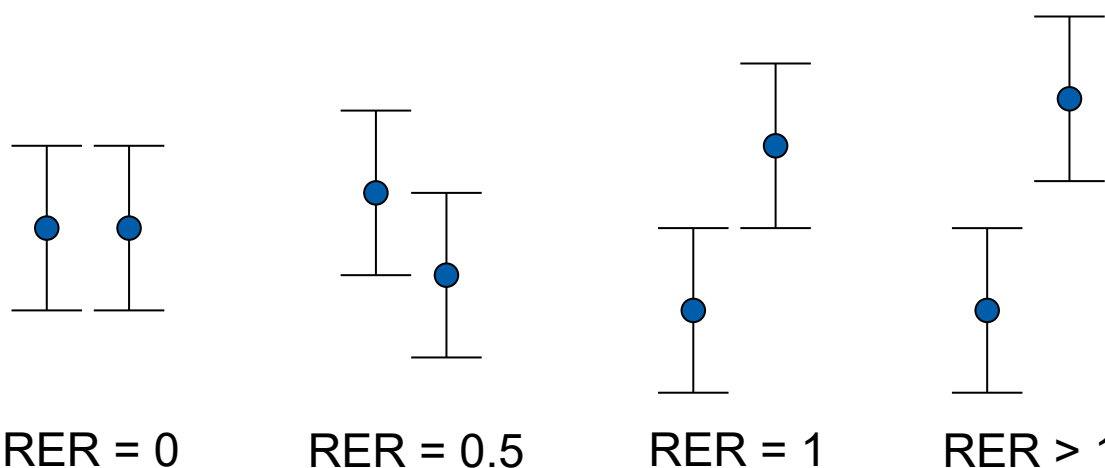
$$RPD = \frac{|\text{Sample} - \text{Duplicate}|}{\left(\frac{\text{Sample} + \text{Duplicate}}{2} \right)}$$

- Typically, the RPD is expected to be within a certain range (e.g. $\pm 40\%$), dependent upon matrix and sample type. However, as the sample activity approaches the MDC, the RPD tends to “blow up” out of proportion due to the statistical error involved. Thus, we also look at the RER (Relative Error Ratio):

$$RER = \frac{|Sample - Duplicate|}{(Sample\ 2\sigma\ Unc. + Duplicate\ 2\sigma\ Unc.)}$$

The RER is most meaningful near or below the MDC, and is expected to be ≤ 1 . As the sample activity increases, the RER tends to “blow up” out of proportion, and the RPD is more representative of replicate reproducibility.

Looking at the RER pictorially, when the 2σ error bars touch or overlap, the $RER \leq 1$.



Thus, when evaluating replicate precision for Radiochemistry results, a dual criteria is applied. Either $RPD \leq$ control limit (e.g. $\pm 40\%$) or $RER \leq 1$.

- The percent difference (%D) is calculated as follows:

$$\%Difference = \frac{|R_1 - R_2|}{R_1} \times 100$$

Where:

R_1 = First result

R_2 = Second result

- Standard Deviation (SD) is calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^N (X_i - \bar{X})^2}{N - 1}}$$

Where:

X_i = Value of X as i through N
N = Number of points
X = Average value of X_i

ADDITIONAL Calculations for Metals

- The final concentration for a digested aqueous sample is calculated as follows:

$$mg / L = \frac{C \times V1 \times D}{V2}$$

Where:

C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

- The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$mg / Kg, dry weight = \frac{C \times V \times D}{W \times S}$$

Where:

C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight in Kg of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on wet weight basis the "S" factor should be omitted from the above equation.

Additional Calculations for Organics

- The calibration factor for an external calibration standard is calculated as follows:

$$\text{Calibration Factor (CF)} = \frac{\text{Area or Height of Peak}}{\text{Mass Injected (ng)}}$$

- Relative Standard Deviation (%RSD), applicable to initial calibration, is calculated as follows:

$$\%RSD = \frac{SD}{CF_{avg}} \times 100$$

Where:

CF_{avg} = The average of the initial CFs for a compound

SD = The standard deviation (using $n-1$) of the initial calibration CFs for a compound

- Aqueous sample concentration using external standard calibration is calculated as follows:

$$\text{Concentration (mg / L)} = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times V_s)}$$

Where:

A_x = Response for the analyte in the sample

V_i = Volume of extract injected, μL

D_f = Dilution factor

V_t = Volume of total extract, μL

V_s = Volume of sample extracted or purged, mL

CF = Calibration factor, area or height/ng

- Non-aqueous sample concentration using external standard calibration is calculated as follows:

$$\text{Concentration (mg / kg)} = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times W \times D)}$$

Where:

A_x = Response for the analyte in the sample

V_i = Volume of extract injected, μL

D_f = Dilution factor

V_t = Volume of total extract, μL

CF = Calibration factor, area or height/ng

W = Weight of sample extracted or purged, g

$$D = \frac{100 - \%Moisture}{100} \quad (D = 1 \text{ if wet weight is required})$$

- On column concentration

On Column Concentration ($\mu\text{g/mL}$):

$$[OC] = \frac{A_x}{CF}$$

Where:

$[OC]$ = On Column Concentration [typically expressed in $\mu\text{g/mL}$ (ppm)]

Then substitute/derive

$$[C] = [OC] \left(\frac{V_t * D}{V_i * V_s} \right)$$

When *on column concentration* $[OC]$ is equal to the *CAL-AMT (calibration amount)* of the low level standard needed to support the *reporting limit* ($\mu\text{g/L}$) and we solve the equation for *concentration* ($\mu\text{g/L}$)

Then

$$[C] \equiv RL \equiv [OC] \left(\frac{V_t * D}{V_i * V_s} \right)$$

Where:

$RL = \text{Reporting Limit}$

Additional Calculations for GC/MS SVOA

- Concentration calculation using average response factor:

$$C_{ex} = \frac{R_x C_{is}}{R_{is} \overline{RF}}$$

- Concentration calculation using linear fit:

$$C_{ex} = A + B \frac{(R_x C_{is})}{R_{is}}$$

Where:

C_{ex} = Concentration in extract, $\mu\text{g/ml}$
 R_x = Response for analyte
 R_{is} = Response for internal standard
 C_{is} = Concentration of internal standard
 A = Intercept
 B = Slope

- Concentration calculation using quadratic fit:

$$C_{ex} = A + B \left(\frac{R_x C_{is}}{R_{is}} \right) + C \left(\frac{R_x C_{is}}{R_{is}} \right)^2$$

Where:

C = Curvature

- Aqueous sample concentration is calculated as follows:

$$\text{Concentration, ug / L} = \frac{C_{ex} V_t}{V_o}$$

Where:

V_t = Volume of total extract, μL , taking into account dilutions
 V_o = Volume of water extracted (ml)

- Sediment/soil, sludge and waste concentration is calculated as follows:

$$\text{Concentration, ug / kg} = \frac{C_{ex} V_t}{W_s D}$$

Where:

W_s = Weight of sample extracted or diluted in grams
 D = (100 - % moisture in sample)/100, for a dry weight basis
or 1 for a wet weight basis

Additional Calculations for GC/MS VOA

- Calculation (x) for water and water-miscible waste:

$$x = \frac{(A_x)(I_s)(D_f)}{(A_{is})(V_o)}$$

Where:

A_x = Area of characteristic ion for the compound being measured
 A_{is} = Area of the characteristic ion for the internal standard
 I_s = Amount of internal standard added in ng
 V_o = Volume of water purged, mL

$$D_f = \text{Dilution Factor} = \frac{\text{Total volume purged (mL)}}{\text{Volume of original sample used (mL)}}$$

- Calculation (x) for medium level soils:

$$x = \frac{(A_x)(I_s)(V_t)(1000)(D_f)}{(A_{is})(V_a)(W_s)(D)}$$

Where:

A_x , I_s , D_f , A_{is} are the same as for water
 V_t = Volume of total extract, mL (typically 25 mL)

V_a = Volume of extract added for purging, μL
 W_s = Weight of sample extracted, g

$$D = \frac{100 - \% \text{moisture}}{100}$$

- Calculation (x) for low level soils:

$$x = \frac{(A_x)(I_s)}{(A_{is})(W_s)(D)}$$

Where:

A_x , I_s , A_{is} are the same as for water
 D is the same as for medium level soils
 W_s = Weight of sample added to the purge vessel, g

The Percent Difference is calculated as follows:

$$\% \text{ Difference} = \frac{(\text{CF(v) or RF(v)}) - (\text{Avg. CF or RF})}{(\text{Avg. CF or RF})} \times 100$$

Where:

CF(v) or RF(v) = CF or RF from verification standard
Avg. CF or RF = Average CF or RF from Initial Calibration.

The Percent Drift is calculated as follows:

$$\% \text{ Drift} = \frac{\text{Result} - \text{True Value}}{\text{True Value}} \times 100$$

The Percent Recovery is calculated as follows:

$$\% \text{ Recovery} = \frac{\text{Result}}{\text{True Value}} \times 100$$

Gamma Activity Concentration

The activity concentration of a sample will be calculated using the following equation.

$$ACT_s = \frac{\text{Net_Counts}}{2.22 * E * t_s * Ab * V_A * D_C * D_S}$$

where:

| | | |
|------------|---|---|
| ACT_s | = | the activity in pCi/(units of the volume) |
| Net_Counts | = | the net area of a peak |
| 2.22 | = | the correction factor to pCi |
| E | = | the efficiency – corrected for transmission |
| t_s | = | the count time in minutes |

| | | |
|----------------|---|--|
| Ab | = | the gamma abundance factor |
| V _A | = | the sample aliquot volume |
| D _C | = | the decay correction during the analysis |
| D _S | = | the decay correction from collection date to start of analysis |

Gamma Uncertainty of Concentration (at 2σ confidence level)

The Total Propagated Uncertainty (TPU) will be calculated using the following equation.

The software calculates the 2σ TPU term by incorporating the stochastic counting uncertainty and by examining the nuclide library for the error in the nuclide half-life and abundance for their respective contributions. The software routine also includes the standard certificate file and the calibration standard uncertainties. Finally, a 1% factor is added in quadrature due to the uncertainty in the preparation of the sample. This is attributed to the maximum allowable variability of the balances.

$$TPU_S = 1.96 * ACT_S * \sqrt{\left(\frac{\Delta P}{P}\right)^2 + \left(\frac{\Delta Ab}{Ab}\right)^2 + \left(\frac{\Delta \varepsilon}{\varepsilon}\right)^2 + \left(\frac{\Delta V}{V}\right)^2 + \left(\frac{sys}{100}\right)^2 + (\Delta Decay)^2}$$

Where:

$$\Delta Decay = \left[\frac{\Delta T_{1/2}}{T_{1/2}} \right] * \left[\frac{\lambda E_r}{1 - e^{-\lambda E_r}} - \lambda (T_s + E_r) - 1 \right]$$

Where:

| | | |
|-------------------|---|--|
| TPU _S | = | the 2σ uncertainty of the activity of the sample |
| ACT _S | = | the activity in pCi/(units of volume) |
| 1.96 | = | the statistical multiplication factor for 95% confidence level |
| ΔP | = | the uncertainty in the peak area |
| ΔAb | = | the uncertainty in gamma abundance |
| Δε | = | the uncertainty in the efficiency ε |
| ΔV | = | the uncertainty in the volume |
| sys | = | the systematic error estimate (in %)* |
| ΔT _{1/2} | = | the uncertainty in the half-life |
| T _{1/2} | = | the half-life of the nuclide of interest |
| λ | = | the decay constant |
| E _r | = | the elapsed real time during count |
| T _s | = | the sample collection time |

Gamma MDC

The minimum detectable concentration will be calculated using the following equation.

$$MDC = \frac{4.65 * \sqrt{R_B * t_S} + 2.71}{2.22 * E * t_S * Ab * V_A * D_C * D_S}$$

Where:

| | | |
|----------------|---|--|
| MDC | = | Minimum Detectable Activity of the sample |
| R _B | = | Count rate of detector background (in cpm) |
| t _S | = | Count time for analysis |
| E | = | Detector efficiency |
| Ab | = | Abundance of the gamma emission |
| V _A | = | sample aliquot volume |
| D _C | = | Decay during sample analysis |
| D _S | = | Decay from collection to start of analysis |

Alpha Tracer Yield Recovery

Tracer Yield Recovery

$$Y = \frac{(C_T - C_B)}{E * A_T * t_S}$$

Where:

| | | |
|----------------|---|------------------------------|
| Y | = | Chemical Yield |
| C _T | = | Tracer Counts |
| C _B | = | Tracer ROI background counts |
| A _T | = | Tracer dpm |
| t _s | = | Count time for analysis |
| E | = | Detector efficiency |

Ra-226 Ingrowth factor:

$$I = 1 + 3(1 - e^{-\lambda t})$$

Where:

λ = ln(2)/Rn-222 Half-life (in days)

Rn-222 Half-life = 3.824 days

t = Time between BaPrecipitationTime and CountMidPoint (in days)

Note that for validation of data from TALS Level IV reports, BaPrecipitationTime = lngDecDate2 from the Ra-226 prep Batch Worksheet. CountMidPoint is the date Analyzed from the Analysis Detail Report plus one half of the count duration (Ts).

Ra-228 Ingrowth/Decay factors:

$$I = \left(\frac{1 - e^{-\lambda t_2}}{\lambda t_2} \right) (1 - e^{-\lambda t_3}) (e^{-\lambda t_1})$$

Where:

$\lambda = \ln(2)/\text{Ac-228 Half-life (in days)}$

Ac-228 Half-life = 0.2563 days

t_1 = Time between YttriumPrecipitationTime and StartOfCount

t_2 = SampleCountDuration

t_3 = Time between YttriumIngrowthStartTime and YttriumPrecipitationTime

Note that for validation of data from TALS Level IV reports, YttriumPrecipitationTime = IngDecDate1 and YttriumPrecipitationTime = IngDecDate2 from the Ra-228 prep Batch Worksheet. StartofCount is the date Analyzed from the Analysis Detail Report.

Total Strontium Ingrowth factor:

$$I = 1 + (1 - e^{-\lambda t})$$

Where:

$\lambda = \ln(2)/\text{Y-90 Half-life (in days)}$

Y-90 Half-life = 2.67 days

t = Time between StrontiumPrecipitationTime and CountMidPoint (in days)

Note that for validation of data from TALS Level IV reports, StrontiumPrecipitationTime = IngDecDate1 from the Total Strontium prep Batch Worksheet. CountMidPoint is the date Analyzed from the Analysis Detail Report plus one half of the count duration (Ts).

Sr-90 Ingrowth/Decay factors:

$$I = (1 - e^{-\lambda t_1}) (e^{-\lambda t_2})$$

Where:

$\lambda = \ln(2)/\text{Y-90 Half-life (in days)}$

Y-90 Half-life = 2.67 days

t_1 = Time between StrontiumPrecipitationTime and YttriumPrecipitationTime

t_2 = Time between YttriumPrecipitationTime and CountMidPoint

Note that for validation of data from TALS Level IV reports, StrontiumPrecipitationTime = IngDecDate1 and YttriumPrecipitationTime = IngDecDate2 from the Sr-90 prep Batch Worksheet. StartofCount is the date Analyzed from the Analysis Detail Report.

Additional Information for Radiochemistry Calculations:

Zero Count Uncertainty

Certain analyses with intrinsic low background may lead to instances where both the background and the sample count results may be zero (e.g. alpha spec, Ni-59). In such circumstances, the counting uncertainty (CU) and total propagated uncertainty (TPU) will evaluate to zero. To provide a non-zero estimate of the counting uncertainty (and thus a non-zero TPU) in such an occasion, a value of one (1) will be substituted for the sample counts in the counting uncertainty and critical level equations.

Crosstalk Calculation

Alpha into Beta Crosstalk

$$\alpha \gg \beta \text{ crosstalk} = \frac{CPM_{XT}}{CPM_{\alpha} + CPM_{XT}} = y$$

$$yCPM_{\alpha} + yCPM_{XT} = CPM_{XT}$$

$$CPM_{XT} = \frac{y}{(1-y)} CPM_{\alpha} \text{ where } CPM_{\alpha} \text{ is net alpha CPM}$$

Where:

| | | |
|-----|---|---|
| CPM | = | counts per minute (S=Sample, B=Background, XT=crosstalk, α=alpha) |
| T | = | count duration in minutes (S=Sample, B=Background) |
| E | = | Efficiency |
| V | = | aliquot volume |
| UF | = | uncertainty factor (e.g. 0.05) |
| Act | = | activity |

RadCapture Version 5.1.63

Calculation equations for all methods were updated to create consistency. All methods now use the form:

$$Activity = \frac{\left(\frac{Cs}{Ts} - \frac{Cxt}{Ts} - \frac{Cb}{Tb} \right)}{D * E * I * V * R * A} * DF * UCF$$

$$\text{UncCnt } (1\sigma) = \frac{\sqrt{\frac{C_s}{T_s^2} + \frac{C_{xt}}{T_s^2} + \frac{C_b}{T_b^2} + \text{Chi}^2}}{D * E * I * V * R * A} * DF * UCF$$

$$\text{UncTot } (1\sigma) = \sqrt{\text{UncCnt}^2 + (\text{TPUFact} * \text{Activity})^2}$$

$$\text{MDC} = \left(\frac{3.29 \sqrt{\frac{C_b}{T_b * T_s} + \frac{C_{xt}}{T_s^2} + \frac{C_b}{T_b^2} + \text{Chi}^2}}{D * E * I * V * R * A} + \frac{3}{D * E * I * V * T_s * R * A} \right) * DF * UCF$$

$$\text{DLC} = \left(\frac{1.645 \sqrt{\frac{C_b}{T_b * T_s} + \frac{C_{xt}}{T_s^2} + \frac{C_b}{T_b^2} + \text{Chi}^2}}{D * E * I * V * R * A} \right) * DF * UCF$$

Where:

| | | |
|-----|---|--|
| Cs | = | Sample Counts |
| Cb | = | Background Counts |
| Cxt | = | Crosstalk Counts (currently only gross beta) |
| Ts | = | Sample Count Duration |
| Tb | = | Background Count Duration |
| D | = | Decay |
| E | = | Efficiency |
| I | = | Ingrowth |
| V | = | Aliquot Volume |
| R | = | Recovery |
| A | = | Abundance (Branching Ratio) |
| DF | = | Dilution Factor |
| UCF | = | Units Conversion Factor |
| Chi | = | non-Poisson variance |

For the count uncertainty, if both Cs and Cb = 0, then 1 is forced into Cs.
For the DLC, if Cb = 0, then 1 is forced into Cb.

Gross Alpha/Beta is the only method which currently employs a crosstalk factor (and only for alpha into beta crosstalk). However, a crosstalk factor is included for all methods to create consistency. For all methods except Gross Alpha/Beta, Cxt is set to zero in the code.

Similarly, the non-Poisson variance (Chi) has only been employed for a specific client, and only for LSC methods. It is included for all methods to create consistency in the calculation equations. A table is set up in the database to list the Chi factor for each analyte. This factor may be updated on a periodic basis to reflect current operating conditions. This is controlled by an "active" date assigned in the table. The Chi factor is currently set to only be applied for

specific projects (client-based). When not directed to the Chi Table, the calculation uses zero (currently the default for all).

When both the crosstalk and Chi factors are zero, all equations are essentially equivalent to previous versions. The new DLC equation has a marked distinction modification in that it essentially represents a “non-paired” situation to take into account variation in count durations of the background and sample. When the sample and background count durations are the same, the DLC result of the new “non-paired” equation equals the result of the previous equation. Thus, for this verification only the DLC is calculated manually when the sample and background count durations are different. In addition, the factor in the second portion of the MDC equation has been changed to “3” (updated from “2.71” to reflect current generally accepted industry practice).

Equations for Isotopes by Mass and Activity ICP-MS (Uranium by Mass)

Activity Calculation:

$$A_c = M_c \times S$$

Where:

A_c = Activity concentration of Nuclide (e.g. pCi/g or pCi/L)

M_c = Mass concentration of nuclide (e.g. ug/g or ug/L)

S = Specific Activity of the Nuclide

The specific activity of a nuclide is a constant based upon the halflife.

Total Uranium, by Mass:

$$M_{Total} = M_{U-233} + M_{U-234} + M_{U-235} + M_{U-236} + M_{U-238}$$

Where:

M = Mass for each isotope from ICP - MS results

Total Uranium, by Activity:

$$A_{Total} = A_{U-233} + A_{U-234} + A_{U-235} + A_{U-236} + A_{U-238}$$

Where:

A = Activity for each isotope using conversion above from ICP - MS results

Percent U-235 (by mass):

$$\text{Percent U-235} = \left(\frac{M_{U-235}}{(M_{U-233} + M_{U-234} + M_{U-235} + M_{U-236} + M_{U-238})} \right) \times 100$$

Where:

M = Mass for each isotope

Specific Activity values utilized in the calculations above were obtained from NuclideNavigator Version 3.4 and are based upon the PCNUDAT data file from the National Nuclear Data Center (NNDC) at Brookhaven National Laboratory (BNL).

| <u>Nuclide</u> | <u>Specific Activity (pCi/ug)</u> |
|----------------|-----------------------------------|
| Technetium | 17120 |
| Uranium 233 | 9636 |
| Uranium 234 | 6222 |
| Uranium 235 | 2.161 |
| Uranium 236 | 64.67 |
| Uranium 238 | 0.3361 |

Uranium, by Mass:

$$M = \frac{(A \times C) \times (G / L)}{N}$$

Where :

A = Activity in pCi/L for liquid, pCi/g for soil

C = conversion factor from pCi to Bq = 0.037

G = gram formula weight

L = Lamda = 0.693 / halflife in seconds

N = Avegado's Number = 6.02252E + 23

Total Uranium, by Mass:

$$M_{Total} = M_{U-234} + M_{U-235} + M_{U-238}$$

Where :

M = Mass for each isotope from above equation

Percent U-235:

$$\text{Percent U} - 235 = \left(\frac{M_{U-235}}{(M_{U-234} + M_{U-235} + M_{U-238})} \right) \times 100$$

Where :

M = Mass for each isotope

Appendix 5 Laboratory SOP Listing

| SOP Number | SOP Title |
|------------|--|
| ST-GC-0005 | Extractable Total Petroleum Hydrocarbons |
| ST-GC-0013 | Extraction and analysis of Phenols |
| ST-GC-0014 | Aromatic Volatiles and Volatile Petroleum Hydrocarbon |
| ST-GC-0015 | PCB GC Analysis |
| ST-GC-0016 | Pesticide GC Analysis |
| ST-GC-0017 | Herbicide GC Analysis |
| ST-GC-0018 | Analysis of Water Miscible Non-Halogenated Organic |
| ST-GC-0019 | RSK-175 |
| ST-HS-0001 | Waste Minimization Plan |
| ST-HS-0002 | Facility Addendum to Corporate Safety Manual |
| ST-HS-0003 | St. Louis Facility Contingency Plan |
| ST-HS-0004 | Hazardous Waste Management Plan |
| ST-HS-0005 | Laboratory Security Systems |
| ST-HS-0006 | Quarantine Soils Procedure |
| ST-HS-0007 | Fume Hood Calibration |
| ST-IP-0001 | Reactive Cyanide & Sulfide |
| ST-IP-0002 | Acid Digestion of soil |
| ST-IP-0004 | Labware Prep for Inorganic & Trace Metal Analysis |
| ST-IP-0013 | Acid Digestion of Aqueous Samples & Extracts |
| ST-IP-0014 | Alkaline digestion of Cr+6 |
| ST-IP-0015 | Filtration Procedure for Dissolved Metals Analysis |
| ST-IP-0019 | Sulfide Distillation |
| ST-IP-0020 | Distribution Coefficients of Inorganic Species by the Batch Method |
| ST-IS-0001 | Software Change Management |
| ST-IS-0002 | Software Testing, Validation & Verification |
| ST-IS-0003 | Information Systems |
| ST-LC-0001 | HPLC Analysis of PAH/PNA |
| ST-LC-0002 | Analysis of Nitroaromatic & Nitroamine Explosives |
| ST-LC-0004 | Analysis of Perchlorates by LC/MS/MS |
| ST-LC-0005 | Analysis of Nitroaromatics by LC/MS/MS |
| ST-LC-0006 | Analysis of Herbicides by Method 8321 |
| ST-MS-0001 | GC/MS Analysis based on 8270C and 625 |
| ST-MS-0002 | Volatile Organics by GCMS |
| ST-MT-0001 | Metals by ICP/MS |
| ST-MT-0003 | Metals by ICP-AES |
| ST-MT-0005 | Mercury in Aqueous Samples by CVAA |
| ST-MT-0007 | Mercury in Solid Samples by CVAA |
| ST-MT-0008 | Total Uranium by Laser Induced Phosphorimetry (KPA) |
| ST-OP-0001 | Labware Preparation for Organic Analysis |
| ST-OP-0002 | Extraction & Cleanup of Organic Compounds from Water |
| ST-OP-0007 | Extraction of Herbicides - Water & Soil |
| ST-OP-0008 | Extraction of Nitroaromatics |
| ST-OP-0009 | TCLP/SPLP and CWET Procedures |
| ST-PM-0001 | Project Setup and Quote |

| SOP Number | SOP Title |
|------------|---|
| ST-PM-0002 | Sample Receipt & Chain of Custody |
| ST-PM-0003 | Bottle Kit Preparation |
| ST-PM-0004 | Data Review, Verification & Reporting |
| ST-QA-0002 | Standard and Reagent Preparation |
| ST-QA-0005 | Calibration & Verification Procedure for Thermometer |
| ST-QA-0014 | Evaluation of Accuracy and Precision via Control C |
| ST-QA-0016 | IDL/MDL Determination |
| ST-QA-0021 | Internal Surveillance |
| ST-QA-0023 | Document Control |
| ST-QA-0024 | Preventative Maintenance |
| ST-QA-0028 | Water System Maintenance & Monitoring |
| ST-QA-0031 | VOA Holding Blank Analysis |
| ST-QA-0035 | Preparation and Management of SOPs |
| ST-QA-0036 | Non-Conformance Memo Process |
| ST-QA-0037 | Procurement of Quality Related Items |
| ST-QA-0038 | Procedure for Compositing and Subsampling |
| ST-QA-0039 | Sample Transfer Utility |
| ST-QA-0040 | Manual Integration Procedure |
| ST-QA-0041 | Lead Auditor |
| ST-QA-0042 | 10CFR 21 Defects and Non-Compliances |
| ST-QA-0043 | DoD QSM 4.X |
| ST-QA-0044 | Training |
| ST-QAM | Quality Assurance Manual |
| ST-RC-0002 | Planchet Prep for Radiochemistry & Radiological Sc |
| ST-RC-0003 | Drying & Grinding of Soil & Solid Samples |
| ST-RC-0004 | Prep of Soil, Sludge, Filter, Biota &)/G Samples |
| ST-RC-0010 | Screening Samples for Presence of Radioactive Mate |
| ST-RC-0014 | Bulk Drying and Grinding of Soil and Solid Samples |
| ST-RC-0015 | Total Activity Screening Procedure by LSC |
| ST-RC-0020 | Determination of Gross Alpha/Beta Activity |
| ST-RC-0021 | Gross Alpha Radiation in Water - Coprecipitation |
| ST-RC-0025 | Preparation of Samples for Gamma Spectroscopy |
| ST-RC-0030 | Determination of Tritium in Water, Fluids, Soil & |
| ST-RC-0031 | Tritium Determination by Cryogenic Distillation |
| ST-RC-0036 | Chlorine-36 |
| ST-RC-0039 | Radium 226 by Alpha Spec |
| ST-RC-0040 | Total Alpha Emitting Isotopes of Radium |
| ST-RC-0041 | Radium-226 & Radium-228 by Chemical Separation |
| ST-RC-0042 | Iodine-129 in Water |
| ST-RC-0050 | Preparation of Strontium 89 & 90 |
| ST-RC-0055 | Determination of Fe55, Ni59 & Ni63 by LSC |
| ST-RC-0056 | Carbon-14 by LSC |
| ST-RC-0057 | Carbon -14/Inert Gas |
| ST-RC-0058 | Soil Prep for Sr-89, Sr-90 & Total Sr using Extraction Chromatography |
| ST-RC-0100 | Actinide Co-precipitation |
| ST-RC-0125 | Determination of TC99 using Eichrom TEVA Resin |

| SOP Number | SOP Title |
|------------|--|
| ST-RC-0210 | Determination of Po210 by Alpha Spectrometry |
| ST-RC-0211 | Determination of Pb210 by LSC |
| ST-RC-0232 | Isotopic Th/Np in Various Matrices by Eichrom TEVA |
| ST-RC-0238 | Isotopic U by Eichrom UTEVA Resin for Various Matrices |
| ST-RC-0240 | Isotopic Am/Cu/Pu/Th/U in Various Matrices Eichrom |
| ST-RC-0241 | Am/Pu/Cu/U in Various Matrices by Eichrom UTEVA & |
| ST-RC-0242 | Isotopic Th/Pu/U in Various Matrices by Eichrom Se |
| ST-RC-0245 | Determination of Pu241 by LSC |
| ST-RC-0246 | Isotopic Am/Cu/U in Various Matrices by Eichrom S |
| ST-RC-0247 | Promethium247 & Samarium151 Lanthide Resin Separation |
| ST-RC-0300 | NJ 48 Hour Gross Alpha Testing PWTA |
| ST-RC-5006 | Decontamination of Lab Glassware, Labware & Equip. |
| ST-RD-0102 | Gamma Vision Analysis |
| ST-RD-0210 | Alpha spectroscopy |
| ST-RD-0302 | Liquid Scintillation Counter Analysis |
| ST-RD-0403 | Low Background Gas Flow Proportional Counting System |
| ST-RP-0001 | Radiation Protection Program |
| ST-RP-0005 | ALARA Program |
| ST-RP-0010 | Internal Exposure Control |
| ST-RP-0020 | External Exposure Control |
| ST-RP-0030 | Radiological Contamination |
| ST-RP-0031 | Radiation Work Permits |
| ST-RP-0032 | Instrumentation and surveillance |
| ST-RP-0033 | Radiological Areas and Posting |
| ST-RP-0034 | Engineered Controls |
| ST-RP-0042 | Handling of Sealed Sources |
| ST-RP-0050 | Purchase, Receipt, Handling and ID of Radioactive |
| ST-RP-0051 | Packaging/Transportation of Radioactive Material |
| ST-RP-0100 | Radiation Protection Records |
| ST-RP-0110 | Radiation Protection Training |
| ST-RP-0120 | Emergency Response & notification |
| ST-RP-0140 | Quality Assurance in Radiological Protection |
| ST-WC-0001 | Turbidity |
| ST-WC-0002 | Cyanide Analysis by Technicon TRAACS 800 Autoanaly. |
| ST-WC-0003 | Hardness |
| ST-WC-0004 | Chemical Oxygen Demand |
| ST-WC-0005 | Percent Solids Determination |
| ST-WC-0006 | Total Organic Halides in Water (TOX) and Soil(EOX) |
| ST-WC-0011 | Analysis of pH in Water & Soil |
| ST-WC-0012 | Analysis of Sulfide in Water |
| ST-WC-0013 | Phosphorus, all Forms |
| ST-WC-0014 | Analysis of Ammonia as N in Water & Soil |
| ST-WC-0015 | Biochemical Oxygen Demand |
| ST-WC-0016 | Total Organic Carbon |
| ST-WC-0017 | Phenolics, Total Recoverable |
| ST-WC-0018 | Acidity of Water & Wastewater |

| SOP Number | SOP Title |
|------------|---|
| ST-WC-0019 | Alkalinity in Water & Soil |
| ST-WC-0020 | Prep and determination of TKN |
| ST-WC-0023 | Nitrate/Nitrite analysis by TRAACS |
| ST-WC-0025 | Conductivity in Water & Soil |
| ST-WC-0026 | Flashpoint by Pensky-Martens Closed Cup |
| ST-WC-0028 | Anions by Ion Chromatography |
| ST-WC-0029 | Residual Chlorine |
| ST-WC-0031 | Paint Filter |
| ST-WC-0033 | Hexavalent Chromium |
| ST-WC-0034 | Heat of Combustion |
| ST-WC-0036 | Determination of Solids in Water and Wastewater |
| ST-WC-0037 | Perchlorate by IC |
| ST-WC-0039 | Method 1664, N-Hexane Extractable Material |
| ST-WC-0042 | Chlorophyll-a |
| ST-WC-0044 | POTENTIOMETRIC DETERMINATION OF FLUORIDE ISE |
| ST-WC-0045 | Cation Exchange |
| ST-WC-0046 | Reactivity to Air, Water, Physical Properties |
| ST-WC-0047 | TOC in soil |
| ST-WC-0050 | Std Method for Moisture, Ash & Organic Matter |

Mr. Chris Scieszka
DTE Electric Company
June 30, 2020

Attachment C

Groundwater Statistical Evaluation Plan

**REVISED
GROUNDWATER STATISTICAL EVALUATION PLAN**

**INACTIVE BOTTOM ASH IMPOUNDMENT
DTE MONROE PLANT
MONROE, MICHIGAN**

Prepared for:

DTE Energy
One Energy Plaza
Detroit, MI 48226

April 2019, Revised April 2020

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- 1 Monitoring Well Location Map

1.0 INTRODUCTION

On December 28, 2018, the State of Michigan enacted Public Act No. 640 of 2018 to amend Part 115 of the Natural Resources and Environmental Protection Act, PA 451 of 1994, as amended (Part 115). The December 2018 amendments to Part 115 were developed to provide the State of Michigan oversight of coal combustion residual (CCR) impoundments and landfills and to better align existing state solid waste management rules and statutes with the CCR Rule. This alignment would ensure compliance with the CCR standards through a state-approved permitting program that would be deemed to be “equivalent to” or “as protective as” through an administrative application that would be reviewed and authorized by U.S. EPA.

This Groundwater Statistical Evaluation Plan was initially developed to comply with the requirements set forth in the 2015 U.S. EPA CCR Rule and its subsequent amendments. In response to the 2018 State of Michigan rule amendments noted above (Part 115), this document was reviewed by DTE Energy and a third-party consultant to align the information presented herein with the applicable Part 115 amendments. AECOM concurs with this alignment and has revised this Plan accordingly

1.1 Regulatory Framework

Regulatory guidance provided in Title 40 Code of Federal Regulations (CFR) §257.90 and Part 115 specifies that the owner or operator of a CCR unit must develop a groundwater monitoring program that includes selection and certification of the statistical procedures to be used for evaluating groundwater quality data as required by 40 CFR §257.93 and Part 115 R 299.4908 of the Michigan Solid Waste Management Rules. This certification must also include a narrative description of the statistical method that will be used for evaluating groundwater monitoring data. Groundwater quality monitoring data has been collected under the detection monitoring program for the inactive Bottom Ash Impoundment (a single CCR unit) including analysis of eight (8) independent groundwater samples from each background and downgradient well.

Title 40 CFR §257.93(f) and Part 115 R 299.4908 outline the statistical methods available to evaluate groundwater monitoring data. The statistical test(s) chosen will be conducted for each constituent in each monitoring well and will be appropriate for the constituent data and the data set distribution.

In accordance with 40 CFR §257.93(f)(6), a qualified professional engineer must certify that the selected statistical method is appropriate for evaluating the groundwater monitoring data for the CCR unit.

1.2 Site Hydrogeology

The bedrock in the site vicinity is overlain by approximately 40 to 50 feet of unconsolidated deposits of glacial origin. The deposits are comprised of two (2) distinct units: a hard glacial till immediately overlying bedrock and lacustrine (lake bed or lake shore) deposits which overlay the till unit. The till is comprised of over consolidated (highly compacted) gray silty to sandy clay with some cobbles and boulders, and ranges from approximately 20 to 50 feet in thickness. The overlying lacustrine deposits are composed of 10 to 30 feet of fine-grained sand and silt with some soft clay except where there is a thin, discontinuous coarse sand unit at the base of the lacustrine sequence. A detailed site hydrogeologic summary is presented in the Revised Monitoring Well Installation Report, Inactive Bottom Ash Impoundment, DTE Monroe Plant, Monroe, Michigan, dated April 2019, Revised April 2020.

Under parts of the Plant, the Inactive Bottom Ash Impoundment, and Process Pond areas, this sand unit ranges in thickness from 5 to 20 feet and yields groundwater. The sand unit thins progressively to the west, having a thickness of approximately 12 feet on the east side of the discharge canal and thinning to less than a few feet within 150 feet to the west of the discharge canal. Further to the west the sand unit is not evident in soil borings for monitoring wells drilled in 2016 around the Fly Ash Basin. This is consistent with the expectation that lake-deposited materials will decrease in thickness with distance away from Lake Erie. Accordingly, it appears that this sand unit is a localized lakeshore beach deposit formed by westward

aggradation with rising lake level and subsequently blanketed by finer lacustrine deposits. Groundwater in the sand unit is under semi-confined conditions with groundwater elevations ranging between approximately 572.6 and 575.6 feet above mean sea level (msl).

In this scenario, the groundwater monitoring system wells do not serve as simple upgradient or downgradient monitoring points. This is because of two main factors:

- The sand unit located at the bottom of the lacustrine deposits is limited in extent. The unit is present in the inactive Bottom Ash Impoundment area and extends a limited distance north into the main Monroe Plant area. As noted above, the sand unit extends westward but also thins out and is not present in monitoring wells located greater than 500 feet west of the CCR unit. As a consequence, there is no representative upgradient or background monitoring position available for the unit.
- There is a strong confined hydraulic pressure in the sand unit aquifer. The overlying finer grained lacustrine deposits are relatively dry but water levels in the monitoring wells installed in the sand unit rise to within 2.5 to 12.0 feet below ground surface (bgs), likely driven by hydraulic pressure from the underlying bedrock aquifer system.

2.0 GROUNDWATER MONITORING SYSTEM

The following sections provide a summary of the monitoring well network for the inactive Bottom Ash Impoundment and the constituents required for the Detection and Assessment Monitoring phases.

2.1 Groundwater Monitoring System

The monitoring well network for the inactive Bottom Ash Impoundment (a single CCR unit) consists of the following monitoring wells (shown on Figure 1):

| | | | | | |
|-------|-------|-------|-------|-------|-------|
| MW-1S | MW-2S | MW-3S | MW-7S | MW-9 | MW-10 |
| MW-11 | MW-12 | MW-13 | MW-14 | MW-15 | |

The number, spacing, and depth of monitoring wells was based on a thorough characterization of the hydrogeologic factors included in § 257.91 (b)(1)&(2) and a review of applicable portions of Part 115 R 299.4906. Details are presented in the *Revised Monitoring Well Installation Report, Coal Combustion Residuals (CCR) Rule, Inactive Bottom Ash Impoundment, DTE Monroe Plant* dated April 2019, revised April 2020.

2.2 Constituents for Detection Monitoring

R 299.4440 describes the requirement for detection monitoring. The following inorganic constituents are required to be monitored as part of the Detection Monitoring Program under Section 11511a.(3)(c) of Part 115:

| | |
|------------------------------|----------|
| Boron | Calcium |
| Chloride | Fluoride |
| Iron | Sulfate |
| pH | |
| Total Dissolved Solids (TDS) | |

These constituents are inclusive of both the Appendix III list under Subsection 257.94 of the CCR Rule and detection monitoring constituents required in Section 11511a. (3)(c) of Part 115.

2.3 Constituents for Assessment Monitoring

Assessment Monitoring (Part 115 R 299.4441) is required if a statistically significant increase (SSI) over background is identified for one or more Appendix III constituents under the Detection Monitoring program. In addition to the detection monitoring constituents listed above in Section 2.2, the following inorganic constituents are required to be monitored as part of an Assessment Monitoring Program under the CCR Rule (Subsection 257.95) and Section 11511a. (3)(c) of Part 115:

| | | |
|-----------|----------|----------------------------------|
| Antimony | Arsenic | Barium |
| Beryllium | Cadmium | Chromium |
| Cobalt | Copper | Lead |
| Lithium | Mercury | Molybdenum |
| Nickel | Selenium | Silver |
| Thallium | Vanadium | Radium 226 and 228 (combined) |
| Zinc | | |

These constituents are inclusive of the Appendix IV list under Subsection 257.95 of the CCR Rule and Section 11511a. (3)(c) of Part 115.

3.0 STATISTICAL ANALYSIS

The plan for statistical analysis of the groundwater monitoring data includes a series of initial steps, and subsequent series of evaluation steps specifically applicable to Detection Monitoring, Assessment Monitoring, or Corrective Action as described in the following sections. Statistical methods specified in Part 115 R 299.4908(1) must be used to evaluate groundwater monitoring data. The statistical tests must meet the performance standards outlined in Part 115 R 299.4908(2). The goal of the statistical evaluation is to determine whether a CCR unit has released contaminants into underlying groundwater. This determination is made by identifying a statistically significant increase (SSI), or in the case of pH either a SSI or a statistically significant decrease (SSD), over background. The specific statistical procedure selected for a given data set depends on several factors including the distribution of the data and the percentage of not detected values within the data for each constituent. Parametric or non-parametric prediction intervals are generally considered the preferred method of evaluating detection or assessment monitoring data and will be used at this site.

3.1 Interwell vs Intrawell Statistical Approach

The first step in evaluating the data is to determine whether an interwell or an intrawell statistical approach is appropriate. Interwell testing is appropriate when there is an identifiable upgradient or background location that is not impacted by the CCR unit. Intrawell testing may be appropriate where there is no clear upgradient or background condition for comparison to the waste boundary aquifer condition. As noted in Section 1.2 above, the available hydrogeologic information indicates that the extent of the uppermost aquifer (sand unit overlying glacial till) is limited, which suggests that an intrawell approach may be applicable for evaluating groundwater data. Other support for intrawell testing includes:

- The hydraulic confinement of the uppermost aquifer and its relatively shallow potentiometric surface (2.5 to 12 feet bgs) indicates that there is relatively little hydraulic head difference to drive vertical movement of water through the overlying finer grained lacustrine deposits.

- The water quality of the uppermost aquifer (sand unit) includes the presence of naturally occurring ionic constituents, but their relative concentrations are not suggestive of CCR impact.

Accordingly, an intrawell approach has been selected for statistical testing of the inactive Bottom Ash Impoundment groundwater monitoring system data.

3.2 Outlier Evaluation

Outliers are inconsistently large or small data values that may be the result of sampling, analytical, or transcription errors, laboratory or field contamination, or extreme values with a population. The monitoring data will be initially evaluated graphically using box or time series plots to determine whether outliers may be present in the data for each well and constituent. Outliers for constituents with less than or equal to 50 percent non-detect data will be evaluated using Dixon's outlier test. Definitive outliers will be removed from the background data as appropriate.

3.3 Normality Tests

Tests to determine whether the data exhibit a normal distribution will be performed using the Shapiro-Wilk test for data sets comprised of less than or equal to 50 observations or the Shapiro-Francia test of normality will be used for datasets comprised of greater than 50 observations. Distributions will be determined using the ladder-of-powers for untransformed (raw data), $\ln(x)$, $x^{1/3}$, $x^{1/2}$, x^2 , x^3 . The first distribution in the ladder-of-powers having a Shapiro-Wilk W statistic greater than the critical value will be used to calculate the background summary statistics and determine whether the data exhibit a normal or non-normal distribution. Normally distributed data will be evaluated using parametric tests. Nonparametric tests will be used when data cannot be normalized.

3.4 Evaluation of Non-Detects

Constituent concentrations that are reported below the practical quantitation limit (PQL), typically referred to as non-detects, will be evaluated differently depending on the percentage of non-detect values for a particular constituent in a given well. Data that are normally distributed and have less than 15 percent non-detects will be evaluated by substituting one-half of the detection limit to calculate the prediction limit. If more than 15 percent but less than 50 percent of the data are non-detects and the data are normally distributed, the prediction limit will be calculated using Aitchison's, Cohen's, or the Kaplan-Meijer adjustment. For data that contain 50 percent or more, a non-parametric prediction limit will be used.

3.5 Parametric or Nonparametric Prediction Limits

Intrawell parametric prediction limits will be used to statistically analyze constituents that are normally distributed and have less than 50 percent non-detects. Nonparametric prediction limits will be used to statistically analyze constituents do not fit a normal distribution, or that may be normally distributed but have 50 percent or more non-detect values. Parametric prediction limits are calculated as outlined in the U.S. Environmental Protection Agency's (EPA's) Unified Statistical Guidance (USEPA 2009). A nonparametric prediction limit is determined as the largest constituent concentration (excluding outliers) measured during the background period. For parameters comprised of 100 percent non-detect data, the most recent practical quantitation limit (PQL) will be set as the nonparametric prediction limit. It is noted that if there is a new lower PQL utilized by the laboratory in the future, the statistical limit will be maintained at the previous higher PQL until there are a minimum of eight observations reported using the new lower PQL. The statistical limit will be re-evaluated once eight (8) results at the lower PQL are available.

Semiannual sampling results will be compared to the parametric or nonparametric prediction limits to determine if results exhibit any SSIs above background. For parameters where background is comprised of 100 percent non-detect data, the double quantification rule will be applied, wherein an exceedance of the PQL by a quantified constituent concentration will be considered a SSI, and may be verified by

resampling. Two or more consecutive SSIs are required to confirm a constituent as exhibiting a SSI over background.

3.6 False Positive and Negative (Statistical Power)

To achieve the site-wide false positive rates (SWFPR) recommended in the EPA's Unified Statistical Guidance (USEPA 2009), the verification resampling program outlined in Section 4.2 is required. Without verification resampling, the SWFPR cannot be reasonably met, and much larger statistical limits would be required to achieve a SWFPR of 5 percent or less for a semi-annual sampling event. Furthermore, the false negative rate would also be greatly increased. Power curves will be calculated to verify that the SWFPR is achieved for each sampling event.

3.7 Updating Background

Due to the complex behavior of groundwater and the need for sufficiently large sample sizes, background data should not be regarded as a single fixed quantity. Background should be sampled regularly throughout the life of the facility, and periodically reviewed and revised as necessary to account for changes in background water quality that are not attributable to a CCR unit. There are no firm rules on how often to update background data. The EPA's Unified Statistical Guidance (USEPA, 2009) adopts the general principle that updating should occur when enough new measurements have been collected to allow a two-sample statistical comparison between the existing background data and a potential set of newer data. At least 4 to 8 new measurements should be gathered to enable such a test; this implies that updating would take place every 2 to 4 years with semi-annual sampling.

3.8 Verification Sampling

Verification resampling is an integral component of the statistical methods outlined above. Verification resampling provides a way to evaluate unexpected or errant sample results and can help avoid unnecessary entry into assessment monitoring. A verification resample would only be collected from the well(s) where an outlier or statistically significant concentration increase was observed, and only for the relevant analyte(s). The same sampling procedures used for Detection Monitoring would also be used for verification resampling. The facility will take reasonable efforts to complete verification resampling within 30 days of identifying the need to resample. A "1 of m" sampling protocol will be used to verify initial statistical exceedances. A "1 of 2" sampling method is defined as the collection of an initial sample and one confirmatory resample. A SSI is only flagged when a verification sample confirms the initial result.

4.0 ASSESSMENT MONITORING

According to R 299.4440(8), if the facility determines, pursuant to R 299.4908(5), that there is a SSI over background levels for one or more of the detection monitoring constituents, the facility will, within 14 days of the determination of a SSI, place a notice in the operating record that indicates which constituents show a SSI and notify EGLE. Within 45 days of detecting a SSI, the facility will prepare and submit an assessment monitoring plan as required by Rule 299.4441 and a response action plan as required by Rule 299.4442 <or> demonstrate that:

- A source other than the CCR unit caused the SSI, or
- The SSI resulted from error in sampling, analysis, statistical evaluation, or natural variation in groundwater quality.

The owner or operator must complete a written demonstration (i.e., Alternative Source Demonstration (ASD)), of the above within 30 days of confirming the SSI and submit the ASD to EGLE as required by R

299.4440(9). If a successful ASD is completed, a certification from a qualified professional engineer is required, and the CCR unit may continue with detection monitoring. If a successful ASD is made, the facility must determine if the constituents in groundwater render the unit unmonitorable in accordance with R 299.4440(9)(b).

If the facility is notified that a successful demonstration has not been made, then, within 15 days of notification, the facility shall prepare and submit an assessment monitoring plan as required by R 299.4441 and a response action plan as required by R 299.4442. The facility will initiate the assessment monitoring program within 60 days of the submittal of the assessment monitoring plan as required in R 299.4441 and within 90 days of detecting a SSI as described further in Section 5.

4.1 Assessment Monitoring Program

The facility must begin assessment monitoring for the CCR unit if a SSI is identified, and the SSI cannot be attributed to an ASD. Per R 299.4441, assessment monitoring must begin within 60 days of submitting an assessment monitoring plan. Per the CCR Rule, assessment monitoring must begin within 90 days of identification of a SSI that is not attributed to an alternative source. Wells included in the groundwater monitoring system will be sampled for assessment monitoring constituents included in Section 11519b. (2) of Part 115. Within 14 days of receiving sample results, the owner or operator will place a notice of the detected assessment monitoring parameters in the operating record and notify EGLE as required under R 299.4441(4)(a). Within 90 days of obtaining the results from the first assessment monitoring event, all of the wells will be sampled for detection monitoring and the detected assessment monitoring constituents in the initial assessment monitoring event. Background will be established for the Section 11519. (2) constituents not already included in the CCR Rule Appendix IV (i.e., copper, nickel, silver, vanadium, and zinc) throughout eight sampling events in accordance with R 299.4441(4)(c).

If assessment monitoring is triggered pursuant to R 299.4440(8), data are compared to Groundwater Protection Standards (GWPSs) developed in accordance with R 299.4441(9) or background groundwater quality. The CCR Rule [§257.95(h)] and the Part 115 rules [R 299.4441(4)(d)] require GWPSs to be established for assessment monitoring constituents that have been detected during baseline sampling. The GWPS is set at the lowest of the EPA maximum contaminant level (MCL), the EPA Regional Screening Level (RSL), the lowest applicable Michigan Part 201 residential criteria (Part 201 RC), or a value based on background data. The lowest of the MCLs or RSLs or applicable Part 201 RC will be the GWPSs unless the background concentration is greater than the MCL or RSL or applicable Part 201 RC, in which case, the statistically-determined background value becomes the GWPS. For GWPSs that are established using background, tolerance limits are anticipated to be used to calculate the GWPS. The background will be updated every two years, along with the resulting GWPS, consistent with the EPA's Unified Statistical Guidance (USEPA 2009). If additional assessment monitoring parameters become detected during the assessment monitoring, GWPSs will be developed for those parameters in the same manner as the initial parameters.

Consistent with the EPA's Unified Statistical Guidance (USEPA 2009), the preferred method for comparisons to a fixed standard will be confidence limits. An exceedance of the standard occurs when the 95 percent lower confidence level of the downgradient data exceeds the GWPS. Confidence intervals will be established in a manner appropriate to the data set being evaluated (proportion of non-detect data, distribution, etc.). If the statistical tests conclude that an exceedance of the GWPS has occurred, verification resampling may be conducted by the facility. Once the resampling data are available, the comparison to the GWPS or background will be evaluated.

If the statistical tests and verification resampling conclude that an exceedance of the GWPS has occurred, the facility will conduct an assessment of corrective measures, select a remedy for affected groundwater, and implement a remedial action plan in accordance with the requirements and schedules outlined in R 299.4443, R 299.4444, and R 299.4445.

5.0 CERTIFICATION STATEMENT

CCR Unit: DTE Electric Company, Monroe Power Plant – Inactive Bottom Ash Impoundment

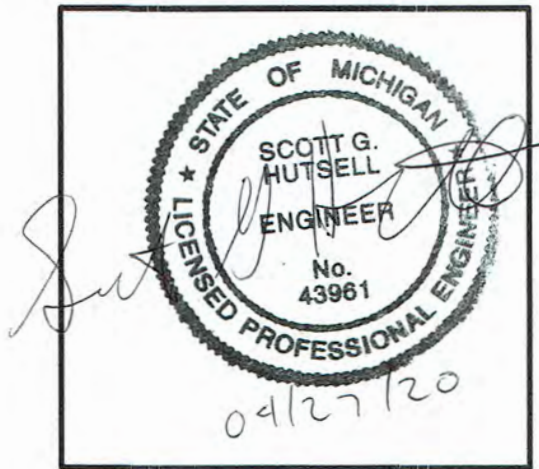
I, SCOTT HUTSELL, being a Registered Professional Engineer in good standing in the State of Michigan do hereby certify, to the best of my knowledge, information, and belief that the information contained in this certification is prepared in accordance with the accepted practice of engineering. I certify, for the above-referenced coal combustion residuals (CCR) Unit, that the statistical approach selected for the groundwater monitoring system, as described in this document, is appropriate for evaluating the groundwater monitoring data for the CCR management area.

SCOTT G. HUTSELL

Printed Name

04/27/20

Date



6.0 REFERENCES

AECOM, April 2019, revised April 2020. *Monitoring Well Installation Report, Inactive Bottom Ash Impoundment, DTE Monroe Plant, Monroe, Michigan, April 2019, revised April 2020*

USEPA. 1989. *Statistical Analysis of Groundwater Monitoring Data at RCRA Facilities, Interim Final Guidance*. Office of Solid Waste.

USEPA. 2009. *Statistical Analysis of Groundwater Monitoring Data at RCRA Facilities, Unified Guidance*. Office of Conservation and Recovery. EPA 530/R-09-007.

Figure 1
Monitoring Well Location Map

Document Path: G:\Cincinnati\DCS\GIS\ArcMap_GeoDB_Projects\ID\TE\DTE-Monroe Plant\Data-Tech\GIS\IDTE_Monroe_Fig2_WellLocationMap_WithDeepWells_2018_20190114.mxd



Aerial and Google Earth 2016 Aerial Imagery

- LEGEND:**
- CCR Program Monitoring Well
 - Investigation Monitoring Well (Potentiometry Only)

- Unit Separation Berm
- Approximate Plant Boundary
- Revised Approximate Boundary of Inactive Bottom Ash Impoundment

0 700 1,400 Feet



Monroe Power Plant

FIGURE 1
INACTIVE BOTTOM ASH IMPOUNDMENT
WELL LOCATION MAP
2018

DATE: 3/20/2019

SCALE 1 inch = 720 feet

JOB NO. 60516675