	Remediation and Redevelopment Division POLICY AND PROCEDURE	DEPARTMENT OF ENVIRONMENTAL QUALITY						
Original Effective Date: October 22, 2004 Revised Date: March 10, 2016 Reformatted Date: March 10, 2016	<table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <td colspan="2" data-bbox="470 191 1153 254">Subject: Standard Operating Procedure for Methanol Preservation in the Field</td> </tr> <tr> <td colspan="2" data-bbox="470 254 1153 363">Program Name: Part 201, Environmental Remediation Part 213, Leaking Underground Storage Tanks</td> </tr> <tr> <td data-bbox="470 363 906 443">Number: RRD-35</td> <td data-bbox="906 363 1153 443">Page 1 of 5</td> </tr> </table>	Subject: Standard Operating Procedure for Methanol Preservation in the Field		Program Name: Part 201, Environmental Remediation Part 213, Leaking Underground Storage Tanks		Number: RRD-35	Page 1 of 5	Category: <input checked="" type="checkbox"/> Internal/Administrative <input type="checkbox"/> External/Non-Interpretive <input type="checkbox"/> External/Interpretive Type: <input type="checkbox"/> Policy <input checked="" type="checkbox"/> Procedure <input type="checkbox"/> Policy and Procedure
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A Michigan Department of Environmental Quality (MDEQ) Policy and Procedure cannot establish regulatory requirements for parties outside of the MDEQ. This document provides direction to DEQ staff regarding the implementation of rules and laws administered by the MDEQ. It is merely explanatory; does not affect the rights of, or procedures and practices available to, the public; and does not have the force and effect of law.

PURPOSE

This Standard Operating Procedure (SOP) is explanatory and provides direction to MDEQ staff and their contractors pertaining to the collection and preservation of soil samples using the procedures of U.S.EPA SW-846 Method 5035 for analysis volatile organic compounds, commonly referenced as methanol preservation. This SOP is available as a technical reference that may be informative and may be used as a reference for parties outside of the MDEQ when conducting methanol preservation.

Please note that because this SOP was written for MDEQ staff, it may contain references to specific equipment for field investigations that the MDEQ currently uses. Such references do not represent endorsements for particular products or vendors.

The MDEQ has designated analytical methods capable of achieving the target detection limits in accordance with MCL 324.20101(1)(bbb). In designating the analytical method, the appropriate preparation techniques included within the method are also designated. The designated analytical methods for volatile organic compounds contained in the MDEQ published list of Target Detection Limits and Designated Analytical Methods for soil samples include preservation of the samples using U.S. EPA SW-846 Method 5035A. This document contains the technical specifications for methanol preservation in the field.

SOP FOR METHANOL PRESERVATION IN THE FIELD

Health and Safety

Safety Data Sheets (SDS) provide health and safety data and emergency procedures. The SDS should accompany staff in the field. Methanol ampoules, tubes, and vials must be provided to field staff inside protective containers to hold any spillage. Methanol is a toxic and flammable liquid. Handle with proper safety precautions. Wear safety glasses and protective gloves. Nitrile, rubber or Viton gloves are recommended. Avoid inhalation. Store and handle in a ventilated area away from sources of ignition and extreme heat. Store the methanol in a cool place, preferably in sample coolers on ice. This is especially important for methanol in tubes where pressure buildup due to extreme heat may result in rupture. Vials should be opened and closed quickly during collection. In the event of eye contact, immediately flush with large

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amounts of water for at least 15 minutes, occasionally lifting upper and lower lids. Seek medical attention immediately.

Shipping

The shipping of methanol may be regulated by the United States Department of Transportation, Title 49 of the Code of Federal Regulations.

Apparatus and Materials Needed for Sample Collection

- Absorbent Material: If the samples are to be shipped by common carrier, vermiculite or a similar material should be added to the shipping container sufficiently to completely absorb any potential spilled methanol.
- Calibration Weight: Near or equal to the target sample weight (i.e., 10 grams).
- Field Balance: Capable of holding sampling vial, syringe and the wide mouth jar used to prevent balance contamination and measurement within + 0.2 grams. Note that the procedure in Method 5035A requires a balance capable of measuring to 0.01 grams. The requirement of the method or this SOP can be followed. A reliable spring scale is also acceptable as an alternative to the field balance for weighing the sample.
- SDS: Should accompany sample collection personnel in the field.
- Methanol Preservation Sampling Kit.
 - Certified Methanol: Methanol certified for purge-and-trap gas chromatography is analytically verified prior to sampling (by lot). For this SOP, the methanol is provided in the MDEQ lab sampling kit in sealed Teflon tubes. Some laboratories may provide methanol in the sampling vial.
 - Sub-Coring Device: A syringe-type device whose material has been tested and found free of contaminants. This device is used to sub-sample the targeted amount of soil for transfer into methanol in the field.
 - VOC Vials: Vials with Teflon™ lined septa, pre-weighed, with labels.
- Protective Wear: Nitrile, rubber or Viton gloves. Splash proof safety goggles.
- Plastic Bags: Airtight seals to hold sample VOC vials and sub-coring device.
- Protocol to be used for the collection of samples.
- Wide Mouth Jar (for holding methanol tubes): Of suitable size to allow temporary storage and shipment of the methanol tubes.
- Wide Mouth Jar (for preventing balance contamination): Of suitable size to allow temporary storage of the syringe-type sampler and VOC sample vial on the field balance.

MDEQ Laboratory Specifications for Sample Collection

The following specifications apply to the methanol preservation sample collection kit provided by the MDEQ laboratory.

Target Soil Weight = 10 grams

Allowed Weight = 9 to 11 grams

Size of VOC Sampling Vials = 40 mL

Plastic bag for sample containers

Methanol Volume (provided in Teflon tubes) = 10 mL

Soil Coring Device and Cap = 10 mL

Label

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Sample Containers, Preservation, and Holding Times

- Containers: Sample containers are VOC vials with Teflon™ lined septa of suitable size to hold the soil plus methanol, supplied with pre-weighed labels.
- Preservation: Samples are preserved in the field approximately with a one-to-one ratio of soil weight to methanol volume using pre-weighed vials and a field balance. The exact sample weights and ratios are determined at the laboratory. When soil weights are less than the specified minimum or more than the specified amounts of methanol are added, the reporting limits are increased. Samples must be stored and transferred at less than or equal to 6° C.
- Holding Times: The maximum allowable holding time is 14 days from the sample collection to analysis. If the maximum allowable holding time is exceeded, interpret the results as minimum concentrations of the measured compounds.

Methanol

Only purge-and-trap grade methanol verified to be suitable for methanol preservation should be used. Field staff should maintain documentation of the methanol lot numbers for all associated samples. If consistently high levels of compounds are measured in methanol field blanks associated with a specific lot number, request the laboratory to verify the quality of the methanol lot used to preserve the samples.

Contamination

Contamination by airborne VOCs in the air is possible by diffusion through the vial septum during shipment, storage, collection, and analysis. To control such contamination:

- Use appropriate VOC sample vials.
- Avoid sources that generate VOCs such as petroleum products, especially auto exhaust fumes.
- Keep sample containers in coolers as much as possible.
- Collect samples quickly.
- Use methanol provided in sealed ampoules, tubes, or VOC vials.
- Store methanol tubes in glass jar.

Attempt to isolate the source of contamination and incorporate the appropriate procedures to avoid similar circumstances.

Field Balance Calibration Check

The field balance calibration should be checked as needed and whenever necessary because of handling in the field. Record this check in the field logbook.

MDEQ RRD Field Sampling Procedures

1. Make arrangements with the laboratory to obtain the appropriate Methanol Preservation Sampling Kits.
2. Record the tracking or lot number(s) for the methanol in the field logbook. If more than one lot is used, each lot must be associated with the samples for which it was used.

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3. Prior to any sample collection, check the calibration of the balance. Also estimate the amount of the soil to be collected using the steps in this SOP. The soil used for the estimations must be discarded.
4. Prior to sample collection, prepare a sufficient quantity of methanol field blanks, i.e., at least one per cooler and one per methanol lot as follows:
 - a. Select an area free of VOC sources.
 - b. Remove a methanol tube.
 - c. Use scissors to cut off the top and place the methanol into one of the pre-weighed sample vials.
 - d. Place the cap on the vial and tighten it. Avoid over-tightening.
 - e. Identify it as a methanol field blank.
5. Place the wide mouth glass jar used to prevent balance contamination on the balance.
6. Record the location, date, and time of sampling in the field logbook. **Do not place any labels, stickers, tape, etc., on the pre-weighed sample vials.**
7. For methanol field blanks, remove the cap from a methanol field blank which was prepared in Step 4 above, place the opened vial in the collection area for the approximate time it takes to collect a sample, then cap the methanol field blank for storage, and transport to the laboratory.
8. Place a pre-weighed VOC vial and syringe in the wide mouth jar on the balance.
9. Record the weight in the field logbook. If the balance features re-zeroing, zero the balance.
10. Remove the syringe. If a cap is provided, remove the cap and place it in the jar.
11. Insert the open end of the syringe into a fresh face of undisturbed soil. Fill it as appropriate according to the amount of soil needed.
12. If necessary, use your gloved finger (decontaminate before next sample) or other appropriate instrument and push the soil deeper into the syringe sampler.
13. If a cap was provided, immediately cap the end of the syringe.
14. Place the syringe in the jar on the balance. Read the weight, and if necessary, subtract the weight of the syringe, vial, and jar as appropriate to determine the weight of the soil.
15. If the weight of the sample is determined to be more than the maximum amount allowed, extrude enough soil to obtain the target amount within the specified tolerance and re-weigh.
16. If the weight of the sample is less than the minimum amount allowed, re-sample.
17. Record the soil weight in the field logbook. **Do not record the weight on the sample vial label.**
18. Remove the cap from the sample vial and place it in the jar on the balance with the septum upwards.
19. Insert the open end of the syringe sampler into the mouth of the vial and carefully extrude the soil, taking care to avoid spillage.
20. If the required amount of methanol is not included in the pre-weighed vial, immediately remove a methanol tube from the wide mouth glass storage jar. Holding the tube upright, use scissors to cut (plastic) off one end and pour the methanol into the sample vial, taking care to avoid spillage. Loss of several drops will not make a significant difference in the results. If a significant amount is spilled, a new sample must be collected or the sample must be appropriately flagged to indicate estimated results. If methanol does not cover soil, add another tube and note on chain of custody.

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21. Using a clean brush, paper towel, or other suitable material, thoroughly wipe excess soil particles from the threads and vial body. Particles left on the threads will prevent a good seal.
22. Place the VOC cap on the sample vial. The cap must be tight; however, over-tightening should be avoided. Complete the label. **Do not add any labels, stickers, or tape to the vial.**
23. Gently swirl the sample and methanol for about ten seconds to break up the soil. **DO NOT SHAKE.**
24. Place the sample in a plastic bag on ice in a cooler.
25. If a jar was requested for another analysis, a syringe for dry weight does not need to be submitted, skip to step 29.
26. Using the syringe sampler, take another sample from the soil.
27. Cap and label the syringe with the sample identification.
28. Place the syringe with the sample vial in the plastic bag. This sample is for dry weight determination.
29. Decontaminate the jar/balance using decontamination procedures appropriate for the type and level of contamination.
30. Unused methanol must be returned to the laboratory for disposal.

REMEDATION AND REDEVELOPMENT DIVISION CHIEF APPROVAL:



Robert Wagner, Chief

March 10, 2016
Date