

**PROTOCOL FOR COLLECTION AND HANDLING  
OF SOIL AND SEDIMENT SAMPLES FOR  
GASOLINE RANGE ORGANIC ANALYSIS**

**Maine Department of Environmental Protection  
Leaking Underground Storage Tank Program**

Standard Operating Procedure: **GRO 1**

Revision: **2**

Written by: Peter Eremeta

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Revised by: Troy Smith

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Reviewed By: Deb Stahler

**Approved by:**

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**George Seel**

**Date**

## **1.0 Summary**

The purpose of this document is to describe the Maine Department of Environmental Protection, Bureau of Remediation and Waste Management, Leaking Underground Storage Tank (MEDEP/LUST) Program procedure for collecting and handling soil/sediment and other “solid” material for gasoline range organic (GRO) analysis.

## **2.0 Purpose**

The details provided in this method are intended to reduce losses of GRO during sampling, storage and transport. These include:

- collection by coring to minimize sample disruption,
- preservation by slurring the sample with methanol (or optionally by maintaining the sample(s) at dry ice temperature) to deter biodegradation and
- isolation by cleaning the cap and vial threads to facilitate tight closure and reduce volatilization, and by using polytetrafluoroethylene(PTFE)-lined lids.

## **3.0 Scope and Applicability**

The techniques described in this SOP are intended for use for GRO in soils. They are mandatory for sampling soils for GRO analysis using any method adopted by the Maine Department of Human Services as applicable for certified analysis. The techniques described are to be used when investigating, remediating, and closing LUST sites that meet the intermediate and stringent soil cleanup standards.

Managers or supervisors are responsible for ensuring that the staff they supervise are familiar with and adhere to this SOP when performing or procuring soil sampling for GRO analysis.

## **4.0 Detailed Description of the Method**

### **4.1 Preservative**

Samplers shall use methanol (**or** dry ice) to preserve soil samples. The methanol must be appropriate for the purge and trap method of analysis. It is necessary that the methanol be subjected to quality control analysis before use. The target ratio of sample to methanol shall be one-to-one by volume. Waste liquid methanol and methanol-preserved samples not submitted for laboratory analysis must be managed as hazardous waste.

### **4.2 Containers**

Samplers shall use clean, quality-controlled, glass containers with PTFE-lined lids. The recommended container volume is 60 mL. The mouth must be wide enough to allow insertion of the coring syringe without contacting the container. It is preferable that the containers be pre-weighed, with the weight recorded on the labels. Containers not pre-weighed must be tared after analysis. The sample plus methanol volume shall not be less than one-tenth of the container volume. When using dry ice as a preservative, take care to minimize headspace.

### 4.3 Sampling devices (coring syringes)

- 4.3.1 Use disposable open-barrel (without Luer-tip end) plastic syringes for sampling. Store in clean polyethylene zipper closure bag, or other suitable clean container. Twenty milliliter (20 mL) syringes are recommended for most applications, especially for coarse soils. Ten milliliter (10 mL) syringes are acceptable. Use a clean coring syringe for each sample collected for GRO analysis. Syringes having rubber or other elastomer seals are not acceptable.
- 4.3.2 Alternatively, reuseable coring devices may be used in lieu of disposable plastic syringes (e.g. stainless steel, PTFE, brass, etc.). The chosen sampling device must be decontaminated before each use.

### 4.4 Sampling Procedure

- 4.4.1 Collect samples for GRO analysis as soon as the soil has been exposed to the atmosphere. Respond to any delay by preparing a fresh sampling surface and starting over.
- 4.4.2 Examples of sample types collected during a sampling event are those from split spoons, Geoprobos®, bucket augers, test pit walls (naturally exposed soil horizons), backhoe buckets or surface grid locations. Expose the sample site (i.e., open the split spoon, scrape the pit wall surface, remove vegetation and top soil from a surface grid location, etc.) and quickly collect the soil sample.
- 4.4.3 Samplers shall use appropriate personal protective equipment (PPE) for the specific sampling event, including eye protection and methanol-compatible gloves. To prevent cross contamination, samplers shall wear clean gloves for each sample collected.
- 4.4.4 At each sampling location, prepare a sample for GRO analysis by transferring a soil plug **less than one-half the container volume** to the sample container using a coring syringe. Collect a co-

located sample for dry weight determination of the associated GRO sample, if required by your laboratory.

- 4.4.5 Before coring the exposed soil, retract the plunger of the coring syringe leaving barrel space for the desired soil sample volume. The recommended volume is 10 mL.
- 4.4.6 Insert the coring syringe into the soil surface far enough to fill the preset barrel space. If the soil medium being sampled does not have adequate depth, or if stones prevent the collection of the total amount desired in a single insertion, repeat the motion until the coring syringe contains the desired volume.
- 4.4.7 It is recommended that methanol be added to the pre-weighed container before the soil is cored and added. Insert the coring syringe into the mouth of the pre-weighed container and expel the soil plug into the container by pushing the plunger of the syringe. After expelling the soil plug, ensure that the threads and sealing surface of the container are clean. Cap the container securely. (It is not recommended that methanol be added to the sample container after the soil plug because of the possibility of increased volatile loss.)
- 4.4.8 Completely disperse samples in methanol by shaking to produce a slurry. Transport cohesive clay soils that do not disperse well in the field to the laboratory as soon as possible, and instruct the laboratory to disperse the sample by appropriate means immediately upon receipt. Note the need for dispersal on the Chain-of-Custody form.
- 4.4.9 For the dry ice preservative option, samples should be placed in the sample container without the methanol preservative. Sufficient volume of sample should be placed in the container to minimize or eliminate any headspace when the cover is placed on the container. Care should be taken to remove particles from the threads on the container. Place sample(s) on dry ice immediately after collection. Maintain samples at dry ice temperature until dispersed in methanol by the laboratory. (Note: Dry ice requires special handling. Do not use without appropriate training and equipment.)

#### **4.5 Sample Storage**

To prevent cross contamination, separate all methanol-preserved soil samples from all other samples in a clean shipping/storage container. Clean the outside of each sample container before placing in the

shipping/storage container. Neat materials should not be placed in any container used for the transport or storage of environmental samples.

#### **4.6 Sample Holding Times**

Maximum holding time for GRO samples is 14 days. Transport samples to the laboratory expeditiously.

#### **4.7 Chain of Custody**

Maintain Chain-of-Custody procedures for each sampling event.

#### **5.0 Quality Assurance**

5.1 Document all deviations from the procedures described in this SOP, and all choices of elective alternatives, in the field notebook or approved work plan, and any subsequent report.

5.2 Compositing of soil samples for GRO analysis is not acceptable.

5.3 Handle and store field QA samples (i.e., trip and field blanks, co-located samples) in the same manner as environmental soil samples.

5.4 Collect background samples when high levels of naturally occurring organic compounds are suspected (e.g., peat and septage).

5.5 Take additional co-located samples for replicate GRO analysis when necessary. The recommended minimum sampling frequency for co-located samples is one for every ten environmental samples collected; at least one per sampling event. Meaningful co-located samples can only be obtained from undisturbed soil horizons.

5.6 Collection and analysis of the following QA samples is **mandatory**:

5.6.1 Prepare one methanol trip blank per field batch when using pre-preserved sample containers. (Not required when dry ice preservation is used.) Each sample storage and shipping container should contain a trip blank. Trip blanks are not required when adding methanol in the field.

5.6.2 Prepare at least one field blank per day per sampling event. If the methanol is added before mobilization, open the container in the field as if adding a sample. If the methanol is added in the field, add it in the same environment and in the same manner as though a sample were being preserved. Take additional field blanks at sample locations where air-borne contamination is specifically

suspected. Identify the samples that are associated with each field blank.

5.7 GRO results must be expressed on a dry weight basis, using the laboratory-determined sample dry weight, or when necessary, using the associated co-located sample dry weight. Evaporation of the analyzed sample to dryness is preferred over co-located sample use.

5.8 Request laboratory analysis and results of all QA samples.

5.9 All analytical data for QA samples and environmental samples reported by the laboratory, must appear together in any subsequent report.

### **Definitions:**

**A sampling event** is a single cycle of mobilization and sample collection, at a single physical site, carried out by a single team of personnel.

**Co-located samples** are second samples collected as near to and as close in time to first samples as feasible. They are not true replicate soil samples for volatile analytes, because it is not feasible to demonstrate homogeneity of the matrix. A co-located sample represents the best attainable approximation of such a replicate, for the given location and matrix.

**Field blanks**, for the purposes of this SOP, are samples of methanol from the same source as that used for sample preservation. They are exposed to the atmosphere at the sampling site to serve as a check on air-borne contamination. They are required because methanol is an avid solvent for gasoline range organics.

**Trip blanks** are sample containers containing methanol from the same source as that used for sample preservation. They are preferably prepared by the laboratory, alternatively by field personnel in the clean area where the methanol and sample containers are stored, before leaving for the field. Their purpose is to detect contamination of samples associated with transportation and handling and as a check for any contamination in the containers or methanol as received from the supplier.

**A field batch of samples** (as distinct from a laboratory batch of samples) is a group of samples collected during one sampling event, and stored and transported in a single shipping container, regardless of the number of samples in the group.