

**Department of Environmental Protection
Bureau of Remediation & Waste Management
LUST Program
Standard Operating Procedure Change Record**

Title: FIELD SCREENING OF SOIL SAMPLES UTILIZING PHOTOIONIZATION AND FLAME-IONIZATION DETECTORS

Identification #: RWM-DR 011

SOP Originator: Brian Beneski

Author	Revision Number	Description of Change	Date
Deb Stahler	TS 01	<p>Substitute MEDEP/Lust Program in the place of MEDEP/DR</p> <p>Section 2.0: Replace with: "MEDEP/LUST Program is responsible for investigation and remediation of soil and water contaminated with gasoline and fuel oil from leaking underground storage tanks. This procedure is approved for determination of hydrocarbon content of oil contaminated soils as outlined in Appendix Q of Chapter 691."</p> <p>Section 7.0 Procedure: Include the updated PID/FID calibration set-points guidance. For key project decisions and site closure, use all procedures listed in Appendix Q of Chapter 691 as attached.</p> <p>Section 10.0 Documentation: All sampling events must be documented in a field notebook or field note forms. Chain of custody forms must be completed, and a completed, signed copy retained in the project file.</p>	8/1/2009
	TS 02	New Set Points	8/19/04
	TS 03	New Set Points	9/13/06
	TS 04	New Set Points	11/24/08

Approved by:

Bruce Hunter, Environmental Hydrogeology Supervisor

George Seel, LUST Program Director

Appendix Q: Field Determination of Soil Hydrocarbon Content by Jar / Poly Bag Headspace Technique

1. **Introduction.** The following is a procedure acceptable to the commissioner for determination of the hydrocarbon content of soils contaminated only by oil and petroleum products. A soil sample is placed in a sealed jar or polyethylene bag and the volatile hydrocarbons are allowed to come to equilibrium with the jar headspace. The headspace hydrocarbon concentration is then measured with a calibrated photo- or flame-ionization (PID or FID) instrument, approved by the commissioner.
2. **Applicability.** This procedure is intended for estimating gasoline, #2 heating oil, diesel fuel, kerosene, and other chemically and physically similar oil contamination in mineral soils, having water contents between bone-dry and saturation. The procedure is not intended for estimating concentrations of heavy oils, lubricating oils, waste oil, and other low volatility hydrocarbon products. Soil grain size distribution and organic carbon content may effect the partitioning of hydrocarbon between soil, liquid, and vapor phases. Weathering of the hydrocarbon product also will decrease the proportion of volatile and soluble constituents, thereby decreasing instrument response. None of these limitations invalidate the method as a technique for approximation of low-level petroleum hydrocarbon concentrations.
3. **Equipment Required.**
 - A. Shovel; trowel;
 - B. Lab containers (VOA or SVOA) of type and quantity for hydrocarbon to be sampled at expected concentrations;

NOTE: Laboratory should be consulted in advance to determine their needs.

- C. Metal dial-type thermometer, -10°C to 50°C;
- D. (Jar headspace method only) Glass, wide-mouthed, metal screw-top, 16 oz. jars, with cardboard lid liner removed, and 1/4" hole drilled through center of lid;
- E. (Jar headspace method only) Roll of heavy duty aluminum foil;
- F. (Poly bag method only) 1-quart, Zip-Lock[®] type polyethylene bags;
- G. Means of measuring 250 gm soil sample, plus or minus 10 gms. (e.g., a "calibrated" container, a "Weight Watchers" spring balance);
- H. Photoionization (PID), or flame ionization (FID) instrument approved by the commissioner;

NOTE: A list of approved instruments and their calibration set points is available from the commissioner. The department also has developed a protocol whereby manufacturers of other instruments may generate calibration data for commissioner evaluation and approval. Copies are available from the Bureau of Remediation and Waste Management.

- I. Calibration equipment for instrument chosen; and
- J. Decontamination equipment including soapy water and clean distilled water in squirt bottles or pressurized canisters.

4. Analytical Procedure.

- A. Determine the location at which the sample is to be taken. If possible, identify an uncontaminated location at the same site from which soil of similar texture and moisture content can be obtained, to serve as a field "blank".
- B. Measure a 250 gm. sample of the soil into a wide-mouthed jar or polyethylene bag. In so far possible, samples should be mineral soil free of vegetation and stones larger than 1/2" in diameter. Seal the samples immediately in the jars by placing a square of foil over the mouth and screwing on the lid, and the bag by zipping the closure. Sufficient air should be left in the bag so that the instrument can withdraw an adequate headspace sample.
- C. Repeat this procedure for three (3) more samples, all gathered within a 2'x2' area.
- D. Shake the jars for 30 seconds to thoroughly mix the contents. If bags are used, they may be kneaded until the contents are uniform.
- E. Measure the samples' temperature by sacrificing one jar or bag. If necessary, adjust all sample temperatures to between 15°C and 25°C by bringing sample containers into a warm vehicle or immersing in a water bath. In warm weather, samples should be kept in a shaded, ventilated area during headspace development and analysis.
- F. Allow at least 15 minutes but not more than 1 hour for soil hydrocarbons to reach equilibrium with the headspace.
- G. If samples are to be taken for laboratory analysis, they should be collected and preserved per laboratory protocols at this time. Preferably, these samples should bracket a wide range of hydrocarbon concentrations including the highest and lowest concentration at the site.
- H. Warm up and calibrate the PID or FID instrument to be used to the calibration set point determined by the commissioner for the make of instrument in use and the product(s) present at the facility.

NOTES:

1. These calibration set points have been established by testing the instruments against weathered petroleum headspace surrogates. Therefore no conversion of the readings to their benzene equivalent is necessary.
 2. The UV source in PID instruments should be cleaned at least weekly per the manufacturer's recommended procedure. Both PID and FID instruments must be recalibrated after four hours of continuous use, as well as at the beginning of field use, since their calibration may drift with battery condition.
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- I. Shake the jars or knead the bags again for thirty (30) seconds.
 - J. Measure the samples' headspace concentration. If the jar headspace technique is used, break the foil seal through the drilled hole in the jar lid using a pencil or nail. Insert the instrument's probe about 1/2" into the jar. If using the poly-bag technique, insert the probe through the bag opening while squeezing the bag tight around the probe. Record the highest reading that remains steady for 1-2 seconds (i.e., that is not due to instrument needle inertia). Repeat this step until all jars have been measured.

NOTE: Both PID and FID instruments withdraw a headspace sample from the jar. In the jar headspace technique, air replaces this sample, diluting the headspace as it is being measured. In the poly bag technique, the bag collapses as its headspace is used by the instrument. In either case it is important to obtain an instrument reading immediately after the seal is broken -- preferably within 10 seconds. Once a jar or bag has been used, it may not be used again, even if sufficient time is allowed to re-establish headspace equilibrium.

K. Repeat all steps at each other location of interest at the site. Finally, repeat all steps for the "field blank" obtained from the uncontaminated location.

L. Average the three readings obtained from each soil sample within each 2'x2' area. Blank results must be reported but must not be used to adjust the readings obtained on other samples.

NOTE: Because calibration set points have been established by testing the instruments against weathered petroleum headspace surrogates, no conversion of the readings to their benzene equivalent is necessary.

CALIBRATION SET POINTS

November 2008 Update

DATE: November 24, 2008

TO: All Persons Performing Site Assessments Pursuant To "Regulations for Registration, Installation, Operation & Closure of Underground Oil Storage Facilities (Appendix P of CMR, Chapter 691)"

FROM: George Seel, Director Division of Technical Services, Bureau of Remediation & Waste Management

SUBJ: Calibration Set Points For Photoionization- (PID) and Flame Ionization- (FID) Detectors Used in Field Headspace Determinations at Maine Petroleum Remediation Sites

The following table gives the set points for various PIDs and FIDs when calibrated with manufacturer-recommended span gas. The listed set points were determined for each make and model of PID using the lamp normally supplied by the manufacturer for petroleum investigations, usually in the 10.2 – 10.6 eV range. The set points are not valid for lamps of other energies.

Please note that the set points previously established for several instruments have changed (new or changed values are shown in red). This was necessary due to a recent change in the test gas formulation. Set points are intended only to normalize response of various PID and FID models to complex petroleum mixtures, not to evaluate the cleanup level achieved at most sites. DEP's guidance for determining cleanup standards, *DEP Procedural Guidance For Establishing Standards For The Remediation Of Oil-Contaminated Soil And Groundwater In Maine* ("Decision Tree") requires laboratory analysis of soil or groundwater for the closure of any Stringent (ST) or Intermediate (IN) site. These changes, therefore, should seldom affect the extent of a remediation or produce outcomes inconsistent with past practices.

Only the makes and models of instrument listed below may be used in Maine site assessments pursuant to Chapter 691 closure requirements. The notification level using instruments adjusted to these set points is 100 ppm for motor vehicle fuels, aviation fuel, marine diesel fuel, and middle distillate heating products. The headspace method is not appropriate and should not be used for evaluating heavy oil or waste oil sites.

Instruments calibrated to the listed set points **may** be used to determine compliance with the cleanup standards at Baseline (BL) sites, where a petroleum discharge poses minimal risk to human and ecological health or environmental resources.

Instruments may be made to read directly by entering the appropriate set point when the calibration routine requests the span gas concentration. Alternatively, the instrument may be calibrated to the actual span gas concentration and its readings later multiplied by the set point divided by 100. Concentrations obtained by either method should not be corrected to "benzene equivalents," as suggested by some instrument manufacturers.

This list is periodically updated as set points are established for additional instruments. For the most current listing, please contact the Division of Technical Services, Bureau of Remediation & Waste Management (BRWM) at (207) 287-2651.

Photoionization Instruments (PIDs)

Make	Model	Gasoline Set Point	Fuel Oil Set Point
GasAlert	Micro 5 PID	260	385
Hnu Systems	HNu 101 Series	320	400
	HNu 102 Series	210	290
Ion Science	PhoCheck Series	140	130
MSA	Photon Gas Detector	225	225
	Passport PID II OVM	200	220
	Sirius Multigas Detector	285	385
Photovac	MicroTIP Series	225	225
	2020 ProPLUS	120	130
RAE Systems	MiniRAE 2000	130	140
	MiniRAE 3000	220	260
Thermo Environmental	OVM 580 Series	210	240
Foxboro	TVA-1000 (PID mode)	210	250

Flame Ionization Instruments (FIDs)

Make	Model	Gasoline Set Point	Fuel Oil Set Point
Photovac	MicroFID	100	90
Thermo Environmental	OVM 680	80	45
Foxboro	TVA-1000 (FID mode)	100	90

**COVERSHEET
STANDARD OPERATING PROCEDURE**

Operation Title: **FIELD SCREENING OF SOIL SAMPLES UTILIZING
PHOTOIONIZATION AND FLAME-IONIZATION DETECTORS**

Originator: **Brian Beneski
Quality Assurance Coordinator
Division of Remediation
Bureau of Remediation and Waste Management**

Standard Operating Procedure: **DR#011**
REVISION: **02**
DATE: **March 16, 2009**
Written/Revised by: **Nick Hodgkins**
Reviewed by: **Troy Smith**

Five Year Review No Changes Needed:

Print Name: _____ Signature: _____ Date: _____

Print Name: _____ Signature: _____ Date: _____

Print Name: _____ Signature: _____ Date: _____

Print Name: _____ Signature: _____ Date: _____

1.0 PURPOSE

The purpose of this document is to describe the Maine Department of Environmental Protection, Bureau of Remediation and Waste Management, Division of Remediation's (MEDEP/DR) procedure for field screening volatile organic content of soils using a closed container and a photoionization detector (PID) or a flame ionization detector (FID).

2.0 APPLICABILITY

MEDEP/DR is responsible for the investigation and remediation of uncontrolled hazardous substance sites throughout Maine. The procedure described herein will provide a screening tool for determining relative levels of volatile organic compounds (VOCs) present in soil with a field PID or FID instrument.

3.0 RESPONSIBILITES

This procedure applies to all staff in the MEDEP/DR who are involved with performing field activities in the investigation of uncontrolled hazardous substance sites. Generally, it is the field personnel of MEDEP/DR and MEDEP/Technical Services (MEDEP/TS) (the Oil and Hazardous Materials Specialist and Geologist positions) who will be responsible for performing this task. Project managers of MEDEP/DR can assist and/or perform this task with field personnel present, or after receiving specific training in this activity.

All managers and supervisors are responsible for ensuring that staff who are responsible for performing this procedure understand and adhere to it for all events.

4.0 INTRODUCTION

In conducting this procedure, a soil sample is placed in an approved container and the volatile constituents are allowed to come to equilibrium. The headspace is then measured with a calibrated PID or FID, with a result expressed in parts per million (ppm). Due to the different ionization potentials of various compounds, actual levels of contamination cannot be determined. However, this technique provides an effective means of screening soil to determine "hot spots", extent of contamination, and as a means of screening samples for submittal for laboratory analysis.

This methodology is not a substitute for actual laboratory analysis; it is a screening tool in the field for determining "hot spots" and other areas of high or low concentrations of VOCs present in soil, or for when choosing samples from a site to submit for laboratory analysis.

5.0 PLANNING

As with any sampling event, a sampling and analysis plan (SAP) and a health and safety plan (HASp) must be developed. Protocol for the development of a Sampling and Analysis Plan can be found in DSR's SOP #014 – Development of a Sampling and Analysis Plan.

6.0 EQUIPMENT

The following equipment is required for conducting the procedure:

- Soil sampling equipment (shovel, bucket auger, soil borer);
- Approved containers (one quart freezer zip lock bags are most commonly used, see section 6.1);
- A PID or FID; and
- Calibration equipment, including users manual, for particular PID or FID to be used.

6.1 SPECIAL CONSIDERATIONS REGARDING CONTAINERS

Currently, the most commonly used (and recommended) containers are one quart sized polyethylene zip lock freezer bags (various manufacturers make these types of bags). Freezer bags are recommended as they are usually constructed from thicker material, and have better quality zip locks. Also used are wide mouthed, metal screw top 16 oz jars, with a ¼ inch hole drilled through center, with foil over the top to provide the seal.

7.0 PROCEDURE

- 1) Collect the soil sample, as outlined in the site specific Sampling and Analysis Plan (SAP)(See SOP DR#014 - Development of a Sampling and Analysis Plan) with appropriate soil sampling equipment.
- 2) Place approximately 250 grams of the soil sample into a an approved container as stated in the SAP. The same type of container should be consistently used at the site for comparison purposes; do not mix or reuse headspace containers (unless the approved container is reusable and cleaned appropriately between uses). In so far as possible, samples should be mineral soil free of vegetation and stones larger than ½ inches in diameter. If soil samples are of different type (loam, sand, silt), this should be identified in the field log book. If a duplicate sample is to be submitted to the laboratory for analysis, this sample should be containerized and preserved as appropriate **immediately**. Soil that has been screened with this procedure should not be submitted for laboratory analysis, unless so documented. If using jars, the jars should be sealed now by placing a square of foil over the mouth and screwing on the lid. If using a bag, the bag should be zipped closed leaving sufficient air in the bag so that the instrument can withdraw an adequate headspace sample.
- 3) Shake the container for 30 seconds to thoroughly mix the contents. If bags are used, they may be kneaded until the contents are uniform.
- 4) Let Sample equilibrate. Allow at least fifteen minutes but not more than two hours for VOCs to reach headspace equilibrium with the headspace. An attempt should be made to allow the same amount of equilibration time for each sample.
- 5) Warm up and calibrate the PID and FID instrument to be used according to the manufacturers recommended procedure (See Section 8 - Additional Considerations With Use of PID/FID). The PID and/or FID should be ready for use prior to collection of the first sample.
- 6) Shake containers/knead bags again for thirty seconds.

7) Measure and record the samples headspace concentration with the instrument. Collect a sample of the headspace by inserting the PID/FID probe into the appropriate opening for the container you are using. Record the highest reading on the instrument after allowing the probe to “sniff” the container for 10 – 15 seconds. It is important to obtain insert the probe as quickly as possible after the seal to the container has been broken. Documentation of headspace results should be outlined in the SAP.

8.0 ADDITIONAL CONSIDERATIONS WITH USE OF A PID/FID

Use of a PID/FID can be found in SOP DR#019 – Protocol for Use of a PID/FID.

The are limitations of PIDs and FIDs. A PID or FID cannot detect all VOCs, nor do they detect all VOCs equally. Factors that influence the response of the particular compound include ionization potential of compound, particular energy rating of lamp, calibration standard used, response factor, response curve, etc. In some instances, such as when the contaminant of concern is a single known compound, it is possible to calibrate the instrument so that a relatively accurate measurement, when compared to laboratory analysis, can be obtained. Because of this, it is recommended that the operator of the particular instrument that will be conducting this procedure take the time before the sampling event to familiarize themselves with the particular instrument that will be used, if they are not already familiar with that instrument. This includes reviewing the specific user manual, and calibration and practice with the instrument prior to the sampling event.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

Data quality objectives (DQOs) should be stated in the SAP (See SOP DR#014). QA/QC samples may be collected if needed to meet your data quality objectives. The following are typical QA/QC samples or tasks conducted for PID/FID field screening. Additional sampling or tasks may be added based on the DQO requirements of the project.

9.1 RECALIBRATION DURING USE

During the course of the work day, the PID/FID should be recalibrated after all long work stoppages (such as lunch break). Additionally, the TVA’s response should be periodically tested by challenging it with calibration gas. If the TVA does not read within 15% of the calibration gas, it should be recalibrated. All recalibration and meter challenges must be documented in the field notebook.

9.2 DUPLICATE SAMPLES

Duplicate samples may be collected at a rate of 5% to assess sample location variability.

10.0 DOCUMENTATION

Field notes should be collected following the standard procedures outlined in SOP DR#013 - Documentation of Field Activities and Development of a SETR. It is important that documentation include the specific lamp energy rating, calibration standard, and special

response factors or curves that may be employed for the particular sampling event. When documenting such a sampling event, one should include enough information so that a person at a later date can easily duplicate the sampling and be able to compare the results.

As this type of screening is done in the field by the sampling team conducting the sampling, no chain of custody is required.

Specialized forms may be developed for recording field screening data. Additionally, some PID/FIDs have software which can record data. Any special method of recording and documenting results must be outlined in the SAP.