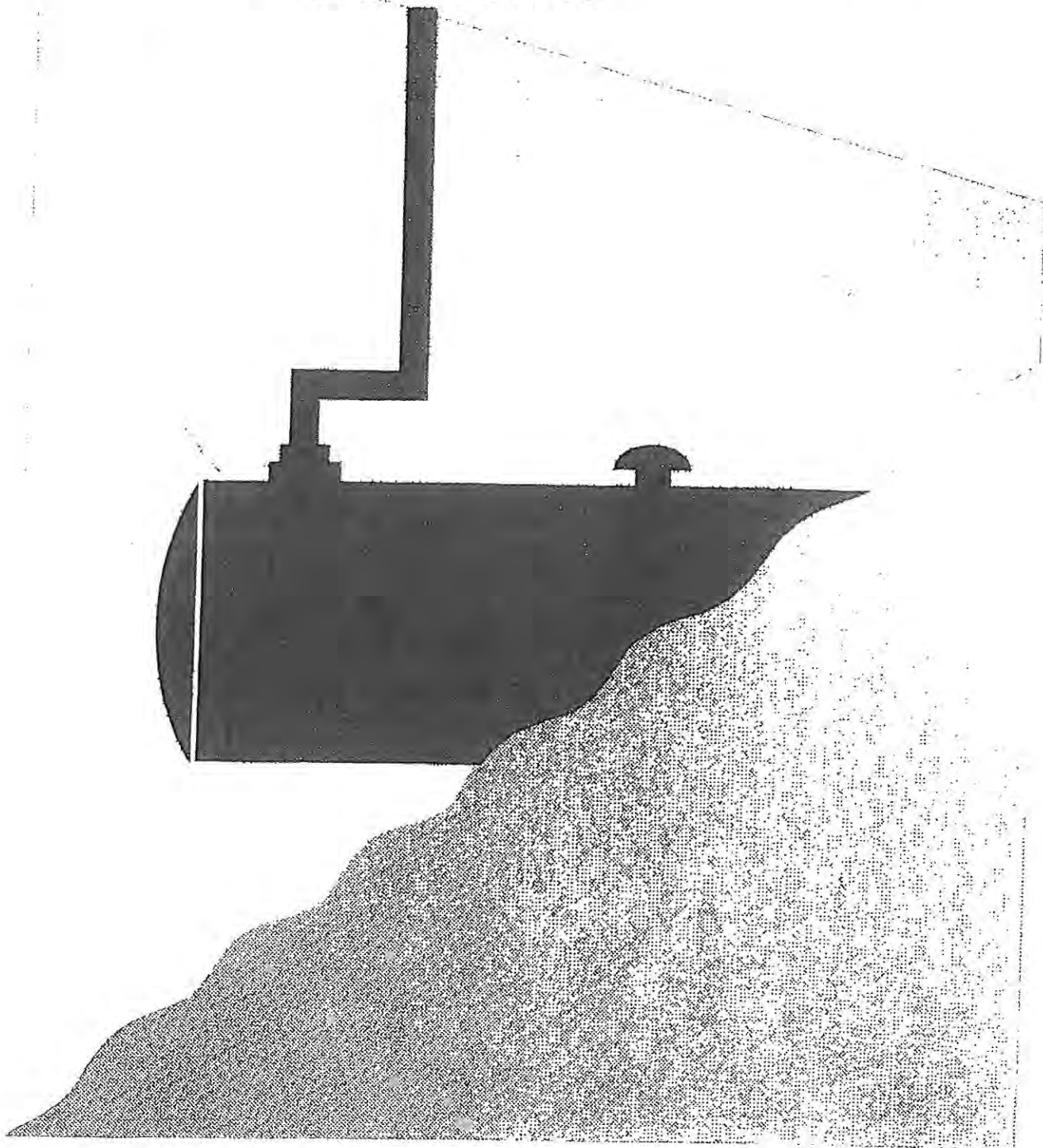




Field Measurements

Dependable Data When
You Need It



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U. S. Environmental Protection Agency
Office of Underground Storage Tanks
Washington, DC 20460

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September 1990

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Disclaimer

This document has been reviewed in accordance with U.S. Environmental Protection Agency policy and approved for publication. Mention of trade names or commercial products does not constitute EPA's endorsement or recommendation for use. Other field measurement procedures may currently exist or be developed.

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Why Are Field Measurement Techniques Desirable?

Hundreds of thousands of underground storage tank (UST) sites will need to be investigated over the next few years to determine if a release has occurred or to find out how much a release has spread. To protect ground-water resources and public health, state and local UST personnel must evaluate these sites to determine as quickly as possible whether corrective action must be taken. Many methods commonly used to evaluate the degree and extent of contamination can be time consuming (as in the use of laboratory analyses) or may not provide sufficiently accurate information for decisionmaking (as in the use of sight-and-smell field observations). Field measurement procedures provide an advantageous alternative to these more commonly used site assessment methods.

Field measurement procedures result in better protection of ground-water resources by dramatically reducing the time required to conduct a site assessment at an UST facility. Field measurement procedures immediately provide accurate, on-site information about the severity and extent of contamination. This information can be effectively used to direct additional investigations, determine where to place monitoring wells, and make wise cleanup decisions.

Currently, many states and consultants stress accuracy over speed, choosing to perform laboratory analyses of soil and water samples collected at UST sites. The results may take as long as 45 days to come back from the laboratory, and they frequently indicate the need for additional site sampling work. It is not unusual for this type of site investigation to take several months, which can unnecessarily delay the start of site cleanup.

Delaying cleanup can cause significant environmental damage and make recovery of released product more difficult with each passing day. Although the use of sight-and-smell field observations also produces immediate measurements, these measurements are considerably less accurate than field measurement techniques or laboratory analyses. In addition, sight-and-smell methods pose direct health risks to the people making the field observations.

INTRODUCTION

In addition to obtaining faster results, field measurements have other important advantages over laboratory analyses. In many cases they may be more accurate than laboratory analyses, which can suffer from loss of contaminants due to biodegradation and volatilization during sample holding. For example, EPA studies show that losses in the range of 10 to 35 percent can occur during a 2- to 5-day holding time for benzene, toluene, ethylbenzene, and xylenes (BTEX) in water samples. Also, the lower cost of field measurements can reduce the cost of investigation and allow more samples to be taken at the site. Taking more samples gives a clearer picture of the extent of a release, which results in better cleanup decisions.

What Information Does This Guide Contain?

Currently, investigators are using a variety of field measurement techniques and procedures to assess contamination at UST sites. This guide presents information on field measurement procedures currently used for UST site investigations and identifies applications and limitations of those procedures. This information is organized as follows:

- A comparative overview of the most common field measurement procedures (Section II);
- Descriptions of two general procedures and three specific procedures (Section III);
- Descriptions of the field instruments used in most of these procedures (Section IV);
- A list of manufacturers and distributors of field sampling and analysis equipment (Section V); and
- A glossary of terms used in this document (Section VI).

Figure 1 on the next page is a flow chart illustrating how this document is organized. As you read the descriptions of the general and specific procedures, you will see part of this flow chart reproduced in the left hand margins to help guide you during reading of the document.

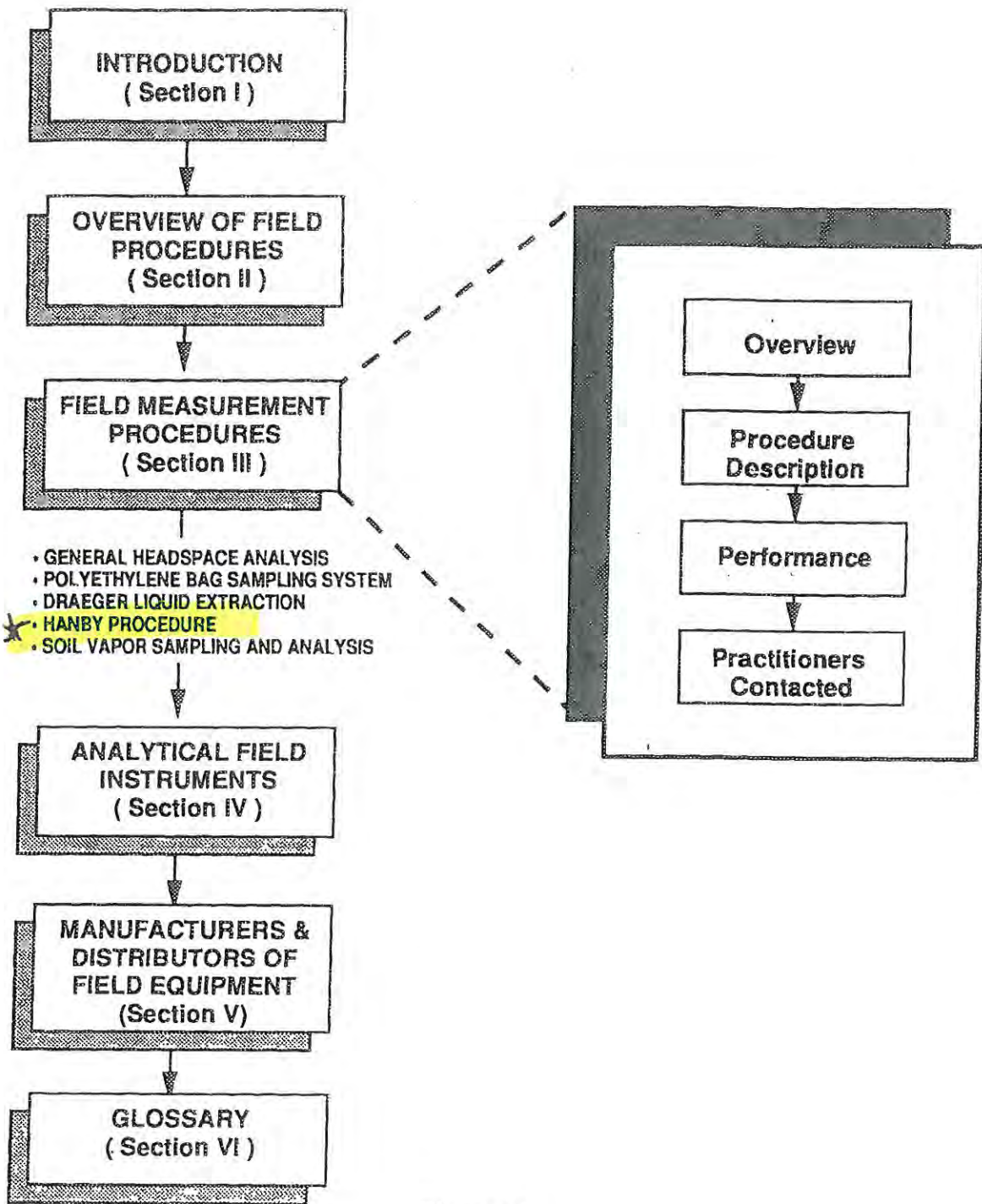


Figure 1

Organization of Document

The analytical field procedures described in this document represent the general types of procedures currently being used or developed for on-site evaluation of petroleum contamination at suspected UST release sites. Descriptions of the procedures are based on information obtained from individuals experienced in conducting them and who have helped develop them. There are a wide variety of field procedures currently being used. Some procedures are very simple to conduct and are used as a quick indicator of contamination, while other procedures are used to quantify the magnitude of contamination. The advantages and disadvantages of each field procedure are highlighted in this section. The information presented below will allow the reader to compare the application, operation, and performance of each procedure relative to one another. For detailed descriptions of each field measurement procedure, see Section III.

A qualitative evaluation of the performance of each procedure for different field applications is presented in Table 1. This qualitative evaluation is based on discussions with practitioners and, where available, on performance data. It should be noted that documented performance data has been identified for only a few of the procedures (e.g., dynamic headspace analysis using a polyethylene bag). Known detection limits and performance factors for each procedure are presented in Tables 2 and 3. Procedures with little or no performance data have been evaluated subjectively. A more quantitative or objective evaluation of these procedures can be made when additional data become available. Table 4 summarizes performance factors for each analytical device.

General Headspace Analysis of Soil and Water

This procedure involves collecting a soil or water sample, placing it in an air-tight container, and then analyzing the headspace vapor using a portable analytical instrument. Some investigators agitate the sample prior to analysis to facilitate volatilization of organic compounds into the headspace immediately above the sample. This is referred to as a "dynamic" headspace analysis. Other investigators conduct a "static" headspace analysis; instead of agitating the sample, the sample is kept still (or static) for a period of time to allow volatile compounds to collect in the container headspace.

Table 1
Applications of Field Measurement Procedures ⁽¹⁾

PROCEDURE	Media						
	Confirm Presence of Contamination	Quantity Contamination Level (See Table 3)	Trace Source of Highest Contamination	Determine Placement Of Monitoring Wells	Determine Limits of Soil Excavation	Measure GW Remed. Progress	
General ⁽²⁾ Headspace Analysis	Soil, Water	○	○	○	○	○	○
Polyethylene Bag Sampling System	Soil, Water	●	○	○	●	●	●
Draeger Liquid Extraction	Water	●	○	○	○	○	○
Hanby	Soil, Water	●	○	○	●	●	●
Soil Vapor	Soil Vapor	○	○	○	○	○	○

Suitability for Application

- High
- Medium
- Low
- ☒ Not Applicable

⁽¹⁾ The information presented above reflects the applications reported by investigators interviewed for this document.
⁽²⁾ The VOA vial procedure developed by G. Robbins for water analysis performs very well in these applications.
⁽³⁾ Dynamic and Static Headspace Analysis

Table 2
Field Procedure Performance Factors ⁽¹⁾

PROCEDURE	Media	Skill Level For Procedure	Lab and Field Correlation Data Available?	Interference From High Clay Content	Interference From High Soil Moisture	Interference From High Organic Content In Soil
General ⁽²⁾ Headspace Analysis	Soil, Water	<input type="radio"/>	No	<input checked="" type="radio"/>	<input checked="" type="radio"/>	<input checked="" type="radio"/>
Polyethylene Bag Sampling System	Soil, Water	<input checked="" type="radio"/>	Yes	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>
Draeger Liquid Extraction	Water	<input type="radio"/>	No	<input checked="" type="radio"/>	<input checked="" type="radio"/>	<input checked="" type="radio"/>
Hanby	Soil, Water	<input checked="" type="radio"/>	Yes ⁽⁴⁾	<input type="radio"/>	<input checked="" type="radio"/>	<input checked="" type="radio"/>
Soil Vapor	Soil Vapor	<input checked="" type="radio"/>	Yes	<input checked="" type="radio"/>	<input checked="" type="radio"/>	<input checked="" type="radio"/>



(1) The information presented above reflects the applications reported by investigators interviewed for this document
 (2) Dynamic and Static Headspace Analysis
 (3) High degree of interference with the Static Headspace Analysis
 (4) Limited lab and field correlation data available

Skill Level/Degree of Interference
 High
 Medium
 Low
 Not Applicable

Table 3
Analytical Methods and Device Performance

PROCEDURE	Measuring Device	Lower Detection Limits (LDL) For Gasoline		Estimated Time for Sample Collection & Analysis (min.)
		Soil & Water (ppm)	Soil Vapor (ppm)	
General ⁽¹⁾ Headspace Analysis	FID/PID/Colorimetric Detector Tube	10's-100's		10-20
	GC	ppb		20
Polyethylene Bag Sampling System	FID/PID/Colorimetric Detector Tube	1		10-20
	GC	ppb		20
Draeger Liquid Extraction	Colorimetric Detector Tube	Water: 0.5		15
Hanby		Soil: 1 Water: 0.1		Soil: 45-60 Water: 10-15
	FID/PID/Colorimetric Detector Tube		10's-100's	10-30
Soil Vapor	GC		ppb	15-35

4-6 minutes for either,



⁽¹⁾ Dynamic and Static Headspace Analysis

Not Applicable

Table 4
Summary of Analytical Device Performance ⁽¹⁾

ANALYTICAL METHOD/DEVICE	Operational Factors			
	Skill Level For Analysis	Calibration Frequency	Ease of Maintenance ⁽²⁾	Operational Factors
Portable FID	Medium	1-3 times every day	Easy	<ul style="list-style-type: none"> • Detects methane • Low oxygen levels cause flame out • Ambient air must be >40° F • Requires battery recharge every 8 hours • Hydrogen gas is required • Restricted flow rate may produce unreliable readings
Portable PID	Medium	1-3 times every day	Very Easy	<ul style="list-style-type: none"> • Photoionization lamp requires periodic cleaning/recharging • High relative humidity (>90%) "quenches" signal • Interference from dust particles, nearby AC or DC lines, high voltage radio wave transmitters • Less accurate when detecting concentrations >150 ppm
Portable GC	High	Every 5 to 10 samples	Difficult	<ul style="list-style-type: none"> • Operates under limited temperature range
Handy	Medium	None	Minimal Maintenance	<ul style="list-style-type: none"> • High clay content increases sample preparation time • Residual water on glassware consumes catalyst • Sunlight degrades reaction
Colorimetric Detector Tubes	Low	None	No Maintenance	<ul style="list-style-type: none"> • Limited shelf life • High humidity can reduce sensitivity

⁽¹⁾ See Section IV A for more details.

⁽²⁾ On-site routine maintenance by the operator



OVERVIEW

The static headspace procedure yields results that are likely to be less sensitive than results obtained from dynamic headspace analysis. Agitating a sample will facilitate volatilization of light hydrocarbons into the headspace of the container.

The dynamic and static headspace procedures are very simple and can be conducted quickly with minimal equipment and operator experience. Results obtained are qualitative and can be used as an indicator of contamination. Limited performance data are available for both of these general headspace procedures. Data that are available indicate significant interferences from soil matrix effects.

Dynamic Headspace Analysis of Soils and Water Using the Polyethylene Bag Sampling System

Field analyses using the Polyethylene Bag Sampling System (PBSS) involve collecting a soil or water sample, placing it in a reclosable freezer bag, agitating the sample to release vapors in the bag, and then measuring the concentration of these vapors using an analytical field instrument. This field procedure is relatively straightforward and easy to perform once the practitioner has been properly trained and is familiar with the operational concepts and equipment. The procedure includes generating a calibration curve with field standards, which is used to determine sample concentrations and as a quality control check of analytical results.

The quality of data (precision and accuracy) obtained with this procedure is very good for both water and soil analyses (see Tables 2 and 3). Performance data obtained using this procedure (shown in Table 3) indicate that volatile petroleum products can be measured in soil and water at relatively low concentrations.

Compared to most other field procedures, the PBSS procedure provides more reproducible results for soil analyses. This is accomplished by adding distilled water to the soil sample, which breaks up the soil and facilitates partitioning of volatile organics from the soil sample into the bag headspace.

Draeger Liquid Extraction and Analysis of Water

This extraction procedure for field analysis of water samples involves passing a fixed volume of air through the sample. Volatile contaminants are "extracted" from the sample and quantified using colorimetric detector tubes. This procedure is easy to use, gives rapid measurement, and requires only the equipment provided with the manufactured kit.

OVERVIEW

The volatile compounds that can be measured using this procedure depend on the type of detection tube selected. The typical range of relative standard deviations is ± 20 -30 percent and the procedure can measure some contaminants (e.g., benzene) at concentrations less than 1 ppm.

Hanby Procedure for Soil and Water Analysis

The Hanby procedure involves extraction of aromatic compounds from soil or water samples and colorimetrically indicates the concentration and type of contaminants present. The color of the reacted extract solution indicates the type of compound (gasoline, diesel, solvent), and the intensity of the color indicates the concentration of the compound. Training and "hands-on" experience are necessary to accurately perform these analyses and to interpret the results.

This procedure provides quantitative results with high levels of precision and accuracy (see Tables 2 and 3). However, some practitioners have indicated that soil analysis (especially samples containing fine clays and silts) takes longer to conduct and the results are not as accurate as water analysis.

Soil Vapor Sampling and Analysis

This procedure generally involves measuring the volatile hydrocarbon concentrations in a soil vapor sample collected in situ by pumping (i.e., active transport) the sample to a field instrument for analysis. There are several procedures for collecting soil vapor samples: 1) drilling or augering a borehole and inserting the analytical instrument probe directly into the borehole; 2) driving a hollow steel probe into the subsurface and collecting a sample with a gas-tight syringe; 3) driving a hollow steel probe into the subsurface and collecting a sample in a collapsible bag; or 4) driving a hollow steel probe into the subsurface and sampling directly from the probe using a portable analytical field instrument. Sample analysis can be conducted using a variety of portable field instruments such as gas chromatographs and photoionization detectors. The skill level required to perform the procedure depends greatly on the analytical instrument selected. For example, an operator using a gas chromatograph (GC) with a driven probe will require more training than one using a photoionization field instrument (PID) inserted in a borehole.

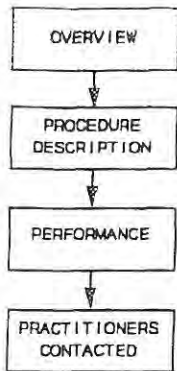
Soil vapor procedures are used for a variety of applications. They are used primarily to provide an indirect estimate of the vertical and lateral extent of contamination, to identify areas of potentially high contamination, and to determine locations for

OVERVIEW

installation of ground-water monitor wells. However, this procedure cannot be used to determine the concentration of contaminants in soil or ground water. The results provide qualitative information on contaminant concentrations in soil vapor, and should be interpreted by evaluating the level of contamination relative to other soil vapor sample points.

Hanby Procedure for Soil and Water Analysis

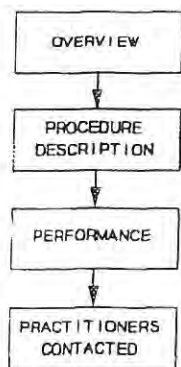
Overview



The Hanby Field Test Kit is a procedure for analysis of petroleum aromatic hydrocarbons over a wide range of concentrations in water and soil. The Hanby procedure provides a colorimetric indication of the presence of aromatic compounds (BTEX, gasoline, diesel, etc.). The soil procedure requires two extraction steps; whereas the water procedure involves one extraction step. Figures 8 and 9 show the process of sample preparation and analysis for soil and water, respectively.

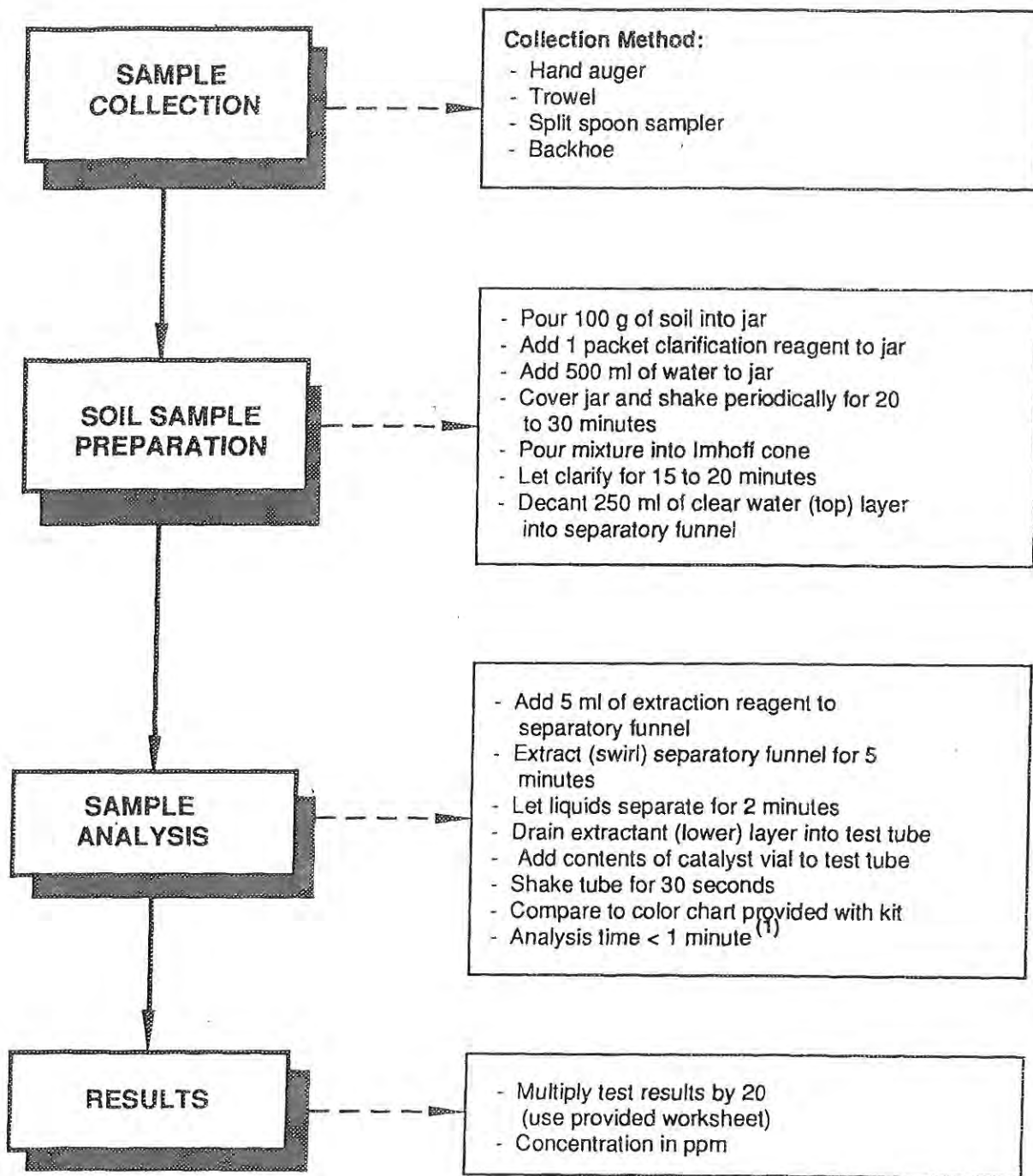
This field procedure is being used by investigators to assess the presence of contamination during tank removals and to quantify levels of contamination to determine the extent of contaminant plumes. As noted in Table 1, this procedure can be used in other applications, including selecting locations for installation of ground-water monitoring wells, determining limits of soil excavation, and measuring progress of ground-water remediation.

Procedure Description



SAMPLE COLLECTION - Soil and Water

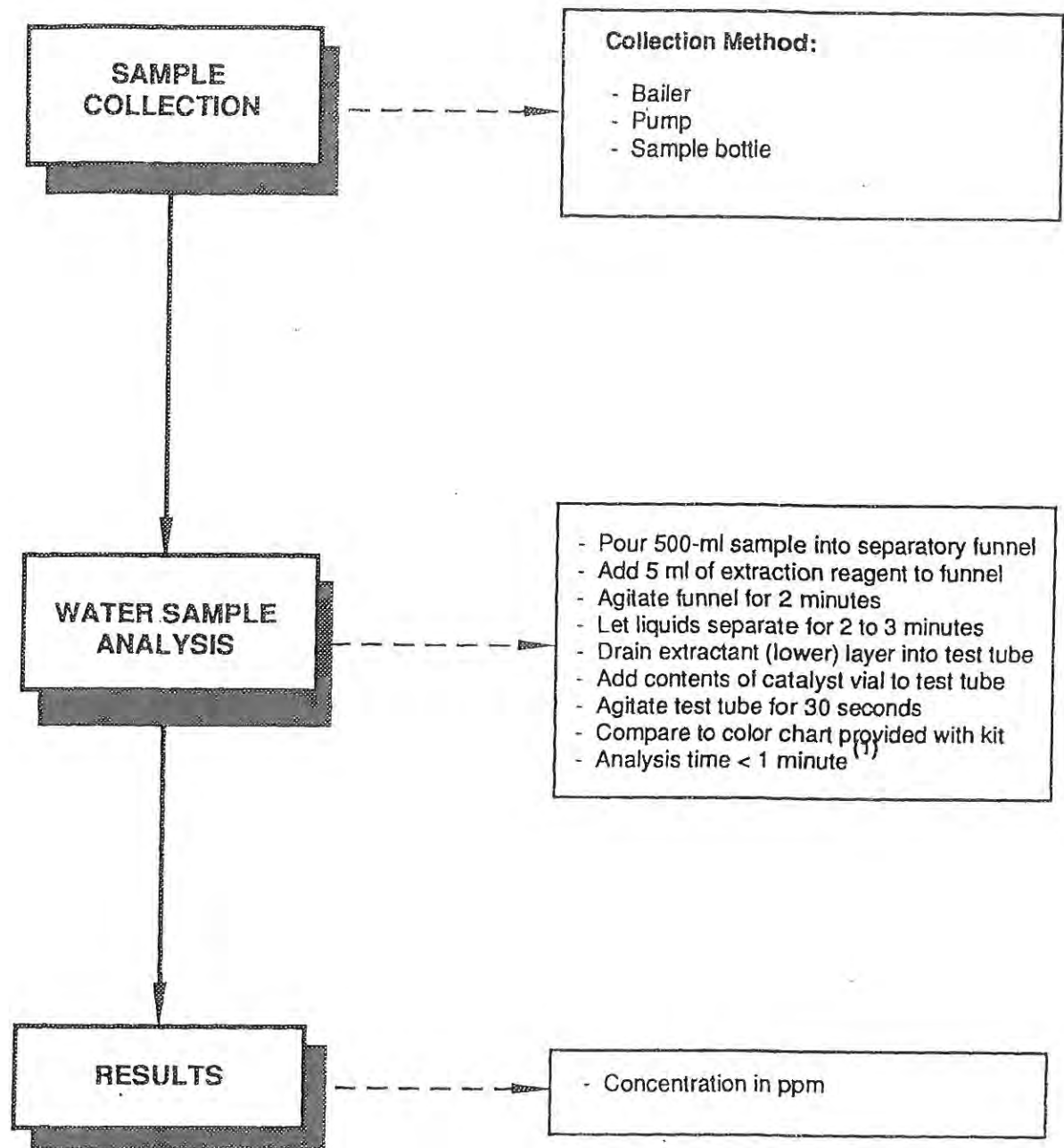
1. Samples may be collected by a number of conventional methods. Soil samples can be obtained using a trowel, backhoe, auger, or split spoon sampler. Water samples may be obtained using pumps, bailers, or sample bottles.
2. Minimize mixing, aerating, heating, or otherwise disturbing the sample to prevent loss of any volatile organic compounds.
3. Decontaminate sampling and testing equipment between each sample analysis to prevent cross-contamination. This can be accomplished using clean water to thoroughly rinse all apparatus.



(1) Keep out of direct sunlight: colorimetric reaction will be degraded by sunlight in as little as 2 minutes.

Figure 8

Hanby Procedure: Colorimetric Analysis of Soil

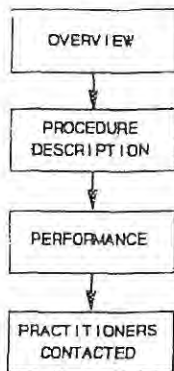


(1) Keep out of direct sunlight: colorimetric reaction will be degraded by sunlight in as little as 2 minutes.

Figure 9

Hanby Procedure: Colorimetric Analysis of Water

HANBY METHOD



SAMPLE PREPARATION AND ANALYSIS

The Hanby Field Test Kit provides the reagents and equipment necessary for the extraction and colorimetric analysis of aromatics from contaminated soil and water. This kit includes: a 500-ml separatory funnel, a tripod ring stand, a 10-ml graduated cylinder, thirty 5-ml ampules, thirty color development reagent vials, six 15-ml test tubes with rack, safety glasses, vinyl gloves, and a color chart depicting test results at various concentrations of eleven aromatic containing substances. A soil accessory kit contains an Imhoff cone and 30 packets of clarifying reagent. The following items are needed to perform soil tests but are not provided in the Test Kit: quart jars with air-tight, screw-on lids (e.g., glass canning jars) and a balance or scale accurate to 0.1 g.

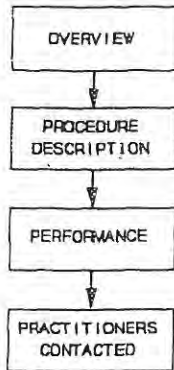
Extraction of Soil

1. A soil sample of 100 grams and one packet of clarification reagent are placed into a 1-quart jar. Next, 500 ml of distilled water is added to the soil-reagent mixture.
2. The jar is sealed with the lid; the soil-water mixture is shaken in the jar intermittently for 20 to 30 minutes.
3. The soil-water mixture is poured into an Imhoff cone and allowed to settle for 15 to 20 minutes. Fine clays may take as long as 30 to 60 minutes to settle.
4. After settling, 250 ml of the clear water layer is carefully decanted into the separatory funnel. One 5-ml ampule of extraction reagent is added to the separatory funnel. After the stopcock and glass stopper are closed, the separatory funnel is gently swirled for 5 minutes while the glass stopper is held in place. The funnel is periodically inverted and vented by opening the stopcock.

Extraction of Water

1. A water sample of 500 ml is placed into a separatory funnel. One 5-ml ampule of extractant reagent is added to the separatory funnel, and the water-reagent mixture is agitated for 2 minutes.

HANBY METHOD



Analysis of Soil and Water


1. The liquid in the separatory funnel will form two layers. After the contents of the separatory funnel are allowed to settle for 2 to 3 minutes, the lower liquid layer is drained into a test tube, without allowing water from the upper layer into the test tube (water consumes the catalyst making the colorimetric reaction impossible). A cotton swab or paper towel can be used to remove any visible moisture from the stem of the funnel. Empty the test tube into the 10-ml graduated cylinder to measure the amount of extraction reagent recovered. Empty the graduated cylinder into another clean, dry test tube.

2. One vial of catalyst is added to the test tube. The test tube is covered and shaken for 30 seconds. Care should be taken to keep the test tube out of direct sunlight once the catalyst has been added. Sunlight reverses the colorimetric reaction in as little as 2 minutes.

3. The color of the test tube contents is compared to the color chart provided with the test kit.

Note: To obtain aromatic concentration in ppm in soil, multiply test results by 20. Use the worksheet provided by the manufacturer for soil samples to arrive at the final concentration (mg/kg) in the sample. For heavily contaminated soils (>400 ppm), water leaching (soil extraction steps 1-4) may be replaced by adding four 5-ml ampules of extraction reagent directly to 10 grams of soil and extracting for 2 minutes. After transferring 4.2 ml of clear liquid to a test tube, follow step 2 (analysis of soil and water) above. Multiply test results by 200 to obtain aromatic concentration in ppm.

METHOD RESULTS

 The Hanby procedure is a colorimetric test that provides qualitative and quantitative identification of the presence of petroleum compounds in soil or water. Table 5 provides a summary of the optimum range of colors and concentrations that can easily be determined for each compound, and a reference for identifying the type of contaminant present.

HANBY METHOD

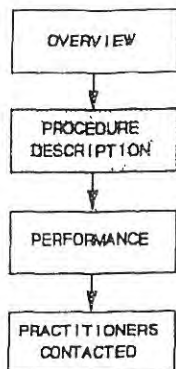


Table 5
Optimum Colors and Concentration Ranges for
Qualitatively Identifying Types of Compounds

<u>Compound(s)</u>	<u>Color/Concentration Range</u>
Benzene	Yellow (1 ppm) to Orange (10 ppm)
Toluene, Ethylbenzene, Xylenes	Orange-Yellow (1 ppm) to Burnt Orange (10 ppm)
Gasoline	Orange (5 ppm) to Rust Red-Brown (20 ppm)
Diesel	Beige (5 ppm) to Green (20 ppm)
Naphthalene (& PNAs)	Light Violet (0.1 ppm) to Blue Violet (2.5 ppm)

A mixture of different compounds will result in a color somewhere between the colors shown on the chart. For example, gasoline, which yields a rust test color, includes a mixture of benzene (yellow) and substituted benzenes (toluene, ethylbenzene, xylenes) (orange).

This procedure provides a quantitative measurement from the shade or intensity of the color, which corresponds to the concentration of the detected aromatic compound. Generally, an optimum contaminant concentration range of 1 to 5 ppm is necessary to clearly distinguish the different colors on the Hanby color chart. Distinctions can be made with increasing difficulty down to 0.05 ppm. Although precise color identification is not possible, discoloration of the test tube contents with respect to a blank sample will be apparent to anyone with normal vision. For instance, at 0.05 ppm it may not be possible to determine that the light hue is benzene, TEX, or gasoline; however, it is clearly not naphthalene.

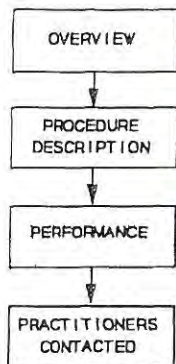
HANBY METHOD

Naphthalene or polynuclear aromatics produce a stronger color intensity at this concentration level, and generally are detected at lower concentration levels because of their larger molecular size.

The time that it takes to perform each analysis is approximately 45 to 60 minutes for soil samples and 15 minutes for water samples. When several analyses are conducted together, the time required to perform each analysis decreases.

(4 to 6 minutes for either test)

Performance



The Hanby procedure is used to determine the presence of most aromatic compounds. It can permit quantitative assessment of the level of aromatic contamination, and depending on concentration, can permit qualitative identification of a particular compound or class of compounds. Practitioners consulted have used the procedure to provide a quick, inexpensive means of detecting aromatic contamination.

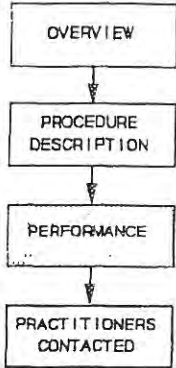
ENVIRONMENTAL INFLUENCES

Top soil and humics containing natural aromatics can interfere with this method when the heavily contaminated soil test method (>400 ppm) is used on soils containing less than 400 ppm aromatics. Some practitioners have indicated that soils containing fine clays and silts take an inordinate amount of time to settle in step 3 of the soil extraction. The clarification reagent was recently reformulated to address this problem.

REPRODUCIBILITY

The developer of this test kit, John Hanby, claims that the Hanby procedure is comparable in reproducibility to purge-and-trap GC methods. This approximately corresponds to a variation in accuracy of 10 percent, which reportedly has been confirmed with split samples analyzed both by purge-and-trap methods and by the Hanby procedure.

HANBY METHOD



One source of error in reproducibility of the procedure is the draining of the lower layer from the separatory funnel. The procedure specifies draining the lower layer until it reaches the portion of the stem just above the stopcock, leaving 0.8 ml in the separatory funnel (this specification is to minimize the likelihood that water will be drained as well.) Because the method is calibrated to draining exactly 4.2 ml, the amount drained in the first analysis step must be measured and corrected (if not 4.2 ml) by using the worksheet provided with the kit. If this correction is not made, the contamination may be overstated by as much as 16 percent.

DETECTION LIMITS

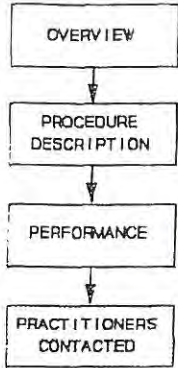
The developer of the test kit reports that the following minimum detection limits can be achieved when comparing the least visible color changes to a blank sample. This comparison does not allow identification of the compound. Although the compound or class of compounds may not be identified, the test provides visible evidence of aromatic contamination as shown in Table 6 below.

Table 6
Lower Detection Limits for Petroleum Hydrocarbon
Contaminants in Soil and Water

<u>Contaminants</u>	<u>Concentration (ppm)</u>	
	<u>Water</u>	<u>Soil</u>
Gasoline and Diesel	0.1	1
BTEX	0.05	0.5

HANBY METHOD

Practitioners Contacted



The following individuals were contacted to obtain information on the Hanby Field Test Kit.

Jeff Billings	Billings & Associates, Inc.
Dale Birdwell	GMS
Jim Dixon	Seneca Environmental Services
John Hanby	Hanby Analytical Labs
Rick Kiel	Vector Engineering
Frank Peduto	New York State Department of Environmental Conservation