7.0 Use of Field Instruments to Monitor Vapors

7.1 Introduction

Typically, the vapors monitored by FDGTI personnel can be divided into two general categories: organic vapors from petroleum products, and non-petroleum vapors such as vapors from chlorinated compounds. Vapors are monitored for a variety of reasons, including:

- monitoring vapor levels in soil from borings/excavations as a means of establishing whether the material is contaminated and the approximate level of contamination;
- monitoring vapor levels in the unsaturated (vadose) zone of in-situ soils as an indicator of whether the material is contaminated and the approximate level of contamination;
- monitoring the vapor levels in soil from excavations to establish whether the material must be considered hazardous and, therefore, disposed of accordingly;
- monitoring vapor concentrations in the work space when working in the known or potential presence of hazardous substances to determine the appropriate level of protection;
- monitoring vapor levels in and around buildings and residences where the presence of contaminants is known or suspected; and
- measuring the air quality of the discharge from air strippers and soil vent systems as a permit requirement.

For practical purposes, vapor monitoring can be classified into three general types:

1. monitoring vapors in soil to assess environmental quality;
2. monitoring the effluent air stream of treatment systems for regulatory compliance and determination of remedial progress; and
3. monitoring ambient air quality to assess potential risk (contaminant exposure or explosive hazard) to humans.

7.2 Purpose

The purpose of this document is to present standard procedures for monitoring air quality for the presence of known or suspected vapors using a variety of field instruments and associated methodologies.

There are numerous methods for monitoring vapors, each method tailored to specific conditions such as the type contaminant being monitored and the medium in which the vapors are contained (i.e., air or soil). A number of selected methods that are typically used during normal FDGTI fieldwork are presented in this SOP. Other methods which are not typically used by FDGTI but may at some time become important in fulfilling special monitoring requirements at specific sites, such as particulate monitoring of ambient air, are presented in the references listed below. Likewise, the types and numbers of monitoring devices that exist on the market today are too numerous to address individually in this SOP. Individual regions and offices tend to favor specific instruments, technologies, and methodologies, all of which are valid and some of which are mandated by state and local regulatory agencies.

This SOP will therefore address standard procedures for the use of general (i.e., not brand-specific) types of vapor monitoring devices (i.e., photoionization detectors, flame ionization detectors, LEL/O_2 sensors, detector tubes, and activated charcoal tubes).

7.3 References

The following ASTM Standards were consulted in the preparation of this SOP:

D 1357-94	Practice for Planning the Sampling of the Ambient Atmosphere
D 3249-90	Practice for General Ambient Air Analyzer Procedures
D 3614-90	Guide for Laboratories Engaged in Sampling and Analysis of Atmospheres and Emissions
D 3686-89	Practice for Sampling Atmospheres to Collect Organic Compound Vapors (Activated Charcoal Tube Adsorption Method)
D 4096-91	Test Method for Determination of Total Suspended Particulate Matter in the Atmosphere (High-Volume Sampler Method)
D 4490-90	Practice for Measuring the Concentration of Toxic Gases or Water Vapors Using Detector Tubes
D 4597-92	Practice for Sampling Workplace Atmospheres to Collect Organic Gases or Vapors with Activated Charcoal Diffusion Samplers
D 4598-87	Practice for Sampling Workplace Atmospheres to Collect Gases or Vapors with Liquid Sorbent Diffusional Samplers
D 4599-90	Practice for Measuring the Concentration of Toxic Gases or Vapors Using Length-of-Stain Dosiometers
D 4844-88(93)	Guide for Air Monitoring at Waste Management Facilities for Worker Protection
D 4861-94	Practice for Sampling and Analysis of Pesticides and Polychlorinated Biphenyls in Air
D 5314-92	Standard Guide for Soil Gas Monitoring in the Vadose Zone
D 5337-92	Practice for Flow Rate for Calibration of Personal Sampling Pumps
D 5466-93	Test Method for Determination of Volatile Organic Chemicals in Atmospheres (Canister Sampling Methodology)
E 884-82(93)	Practice for Sampling Airborne Microorganisms at Municipal Solid-Waste Processing Facilities
E 1370-90	Guide for Air Sampling Strategies for Worker and Workplace Protection
E 1553-93	Practice for Collection of Airborne Particulate Lead During Abatement and Construction Activities

Note: an ASTM serial designation in bold type denotes a major reference used in the preparation of this SOP. Non-bold serial designations denote references useful in obtaining additional information pertinent to the subject matter.

7.4 Monitoring Vapors for Environmental Assessment

7.4.1 General

For the purpose of environmental assessment of soils conducted by FDGTI, two general methods monitor vapors can be identified:

1. headspace screening of collected soil samples, and
2. measurement of in-situ soil vapors (i.e., soil gas survey).

The methodology for conducting a soil gas survey to monitor in-situ vapors is fully discussed in SOP No. 8 and is therefore not presented here.

7.4.2 Soil Sample Vapor Monitoring (Jar Headspace Screening)

FDGTI field personnel commonly conduct soil sampling for subsequent headspace sampling (commonly referred to as headspace screening). Similar methodology is also used at times`to screen water samples to indicate the presence of dissolved phase contaminants. This practice is not however recommended for water except as a preliminary indicator, and as such it is not considered reliable. Even when performed on soils, headspace screening is qualitative at best, and should be used as a preliminary indicator of contamination for the selection of samples that will be laboratory analyzed.

The following are recommended procedures for conducting headspace screening of volatile contaminated soil samples using a portable photoionization detector (PID) or flame ionization detector (FID) (Section 7.7, below, discusses the operation of these instruments in detail). This procedure is used to monitor vapor concentrations in collected soil samples (i.e., samples collected from boreholes during drilling or from open excavations while digging):

Half-fill two clean glass jars with the sample to be analyzed. Quickly cover each open top with one or two sheets of clean aluminum foil and subsequently apply screw caps to tightly seal the jars. Never use plastic wrap to cover the samples as it may emit measurable vapors. 16 oz. (approx. 500 ml) soil or "mason" type jars are preferred; jars less than 8 oz. (approx. 250 ml) total capacity may not be used.

Allow headspace development for at least 10 minutes. Vigorously shake jars for 15 seconds both at the beginning and end of the headspace development period. Where ambient temperatures are below 32 F (0 C), headspace development should be within a heated vehicle or building. Soil samples should be at 50 F(10 C) or warmer. Note temperature and background VOC levels in field notes during screening.

Subsequent to headspace development, remove screw lid and expose foil seal. quickly puncture foil seal with instrument sampling probe, and insert probe into the jar to about one-half of the headspace depth. Exercise care to avoid uptake of water droplets or soil particles.

> As an alternative, syringe withdrawal of a headspace sample with subsequent injection to instrument probe or septum-fitted inlet is an acceptable contingent upon verification of methodology accuracy using a test gas standard.

Following probe insertion through foil seal and/or sample injection to probe, record the highest meter response as the jar headspace concentration. Using the foil seal/probe insertion method, maximum response should occur at about 2 to 5 seconds. Erratic meter response may occur at high organic vapor concentrations or conditions of elevated

headspace moisture, in which case headspace data should be discounted.

The headspace screening data from both jar samples should be recorded and compared; generally, replicate values should be consistent to plus or minus 20%.

When monitoring for petroleum hydrocarbon vapors, PID and FID field instruments shall be operated and calibrated to yield "total organic vapor" in ppm (v/v) as benzene. PID instruments must be operated with a 10.2* eV (=/-) lamp source. Operation, maintenance, and calibration shall be performed in accordance with the manufacturer's specifications. For jar headspace analysis, instrument calibration shall be checked/adjusted no less than once every 10 analyses, or daily, whichever is more frequent (see Section 7.7, below, for instrument calibration procedures).

* Reference the operator's manual to verify if 10.2 eV is the correct energy level for optimum detection of the chemicals of concern. Many chlorinated solvents require higher (11.7) eV lamp.

Instrumentation with digital (LED/LCD) displays may not be able to discern maximum headspace response unless equipped with a "maximum hold" feature or strip-chart recorder.

Record all vapor measurements, other than those taken during drilling, onto a Vapor Monitor Information form or other relevant form used to record project data, making certain to fully describe the sample location and sampling conditions so that the vapor monitoring procedure can be duplicated, if necessary. An example of a completed Jar Headspace Analysis field form is attached to this SOP. Measurements taken during drilling should be noted on the Well/Boring Log. (See SOP No.10)

Deviations, departures and/or additions to the above procedures will be considered on a case-by case basis by the regulatory representative, on-site coordinator or project manager. In such cases, compelling technical justification must be presented and documented by the methodology proponent.

7.5 Monitoring Emissions from Treatment Systems

Vapor treatment systems (air strippers, carbon canisters, thermal oxidizers, catalytic oxidizers and internal combustion engines) are routinely monitored while in operation in order to determine the vapor concentrations in both the inlet and the outlet (i.e., the influent and effluent) air stream of the system. There are a number of reasons for this vapor monitoring, including:
regulatory compliance regarding acceptable levels of toxic vapors in the exhaust stream,
determination of treatment system efficiency by comparing inlet and outlet vapor concentrations,
determination of the decline in inlet concentrations over time to determine the rate of subsurface remediation, and
determination of concentrations of errant compounds (e.g., methane) which may interfere with the readings of the compounds of concern or compounds (e.g. lead) that can foul system components.

Air streams associated with vapor treatment systems can be monitored a number of ways, including:
using and FID, PID or detector tubes inserted into the vapor stream to obtain direct analysis of the vapor stream (quickest and least accurate method),

collecting short-duration air bag (grab) samples for subsequent laboratory analysis either through a positive-pressure port in the system, or using a mechanical sampling pump, and

collecting time-integrated (long duration, 1 hr. to several days) air samples into stainless steel canisters for subsequent laboratory analysis; this method is commonly used when demonstrating compliance of the system with regulatory emissions requirements (most accurate method).

7.5.1 Direct Analysis Using Field Instruments

The protocol for using field instruments to monitor treatment system vapors is identical to that for using these instruments for other types of monitoring (see Section 7.7, below). However, the following considerations apply when monitoring treatment systems:

Heated exhaust vapors and high positive air pressures in treatment system air streams can be both a hindrance and a safety hazard to vapor monitoring. High air pressure can extinguish the flame of an FID and damage intake fans and other internal parts. Elevated air temperatures can also damage equipment. Therefore, it is essential that the system be equipped with designated sampling ports where the air flow can be regulated. Where it is not prudent to affix an instrument directly to the vapor stream, air samples can be collected into tedlar bags and subsequently fed into the analyzing instrument.

It is important to note the limitations of the instrument being used to take readings. For example, naturally occurring methane in the subsurface will pass directly through activated carbon and into the atmosphere. A PID will not detect this methane while an FID will, but will not differentiate it from other gases. In a situation where methane is not a regulated emission, carbon canisters can appear to have reached saturation, breakthrough and non-compliance if the FID detects methane in the effluent air stream. It is therefore important to have initial (and periodic) compound-specific laboratory analyses performed on treatment system air samples to verify the validity of field readings.

7.5.2 Air Bag Sampling

When collecting air samples into sample bags (i.e., tedlar bags) for subsequent analysis using a field analyzer, the air sample should be fed into the analyzer as quickly as possible to prevent degradation of the sample. When collecting an air bag sample for laboratory analysis, standard documentation (i.e., chain-of-custody procedures) handling and transport procedures apply.

7.5.3 Canister Sampling

Stainless steel canisters are leak-free pressure vessels of desired volume with a valve and passivated interior surfaces (e.g., a SUMMA® canister). Collection of atmospheric samples in canisters provides for:
convenient integration of air samples over a specific time period;
remote sampling and central laboratory analysis;
ease of storing and shipping samples;
unattended sample collection;
analysis of samples from multiple sites with one analytical system;
dilution or additional sample concentration to keep the sample size introduced into the analytical instrument within the calibration range;
collection of sufficient sample volume to allow assessment of measurement precision or analysis, or both, of samples by several analytical systems; and
can be performed in remote access areas using a vacuum regulator flow controller if electricity is not available.

Subatmospheric samples (i.e., samples below atmospheric pressure) can be collected into the canisters by evacuating the canister to 64 Pa (5.0 mm Hg) or less prior to sample collection. When open to the atmosphere containing the VOCs to be sampled, the differential pressure causes the sample to flow into the canister. This technique may be used to collect grab samples (10 to 30 sec.) or time-integrated samples (12 to 24 hrs.) taken through a flow-restrictive inlet.

Pressurized sampling is used when longer-term integrated samples or higher volume samples are required. The sample is collected into the canister using a pump and flow control arrangement to achieve a typical 103-206 kPa (15-30 psig) final canister pressure. A flow control device is chosen to maintain a constant flow into the canister over the desired sample period. The flow rate can be calculated by:

$$F = (P \times V) / (t \times 60)$$

where:
F = flow rate, mL/min,
P = final canister pressure, atmospheres absolute. P is approximately equal to [(kPa gauge)/101.2] +1,
V = volume of the canister , mL, and
t = sample period, hrs.

7.6 Monitoring Ambient Air Quality to Assess Potential Risk

7.6.1 General

Ambient air quality is measured at a site for two reasons:

1. to determine if the air is safe for workers to breathe (i.e., contains sufficient oxygen and no contaminants exceeding acceptable concentrations), and
2. to determine if an explosive hazard may exist.

Ambient air quality can be measured a number of ways:
using hand-held field instruments (FID, PID, LEL/O_2 sensor, or detector tubes);
by affixing portable air sample collection devices to workers' clothing (charcoal tubes, liquid sorbent diffusional samplers, dosiometers) and
by setting up fixed sample collectors around the area to be monitored.

The use of field instruments to monitor ambient air and the use of charcoal tubes to collect air samples for laboratory analysis are discussed here. Other methods are discussed in detail in the referenced ASTM standards (see Section 7.3).

7.6.2 Ambient Air Monitoring Using Field Instruments

Four types of instruments are generally used by FDGTI field personnel to monitor ambient air quality at a site:

1. flame ionization detector (FID),
2. photoionization detector (PID),
3. combustible gas indicator (CGI)and oxygen sensor, also referred to as lower explosive limit/oxygen (LEL/O_2) meter, and
4. detector tubes.

Ambient air measurements are taken in the following areas to assure healthy and safe air quality in those areas:
in the workers' breathing zone during drilling of boreholes and the digging of excavations;
in places (usually but not always enclosed) where vapor accumulation and/or oxygen depletion could occur from a known or suspected source of contamination (e.g., chemical storage areas, buildings housing treatment systems, buildings beneath which contamination has occurred, etc.);
in pits, trenches, basements, excavations and other subsurface areas that humans are likely to access where subsurface contaminants can potentially accumulate and/or oxygen depletion may occur;
around joints, valves and open ends of above-ground piping and storage vessels containing hazardous substances; and
around the exhaust stacks of treatment systems or other operating machinery capable of emitting hazardous vapors.

It should be noted that some of the areas noted above could be classified as confined spaces. This document does not cover confined space entry procedures, but does cover proper ambient air quality monitoring procedures that are applicable to monitoring confined spaces. Before entering any enclosed space, it must first be carefully scrutinized to determine if it qualifies as a confined space. **Confined space entry by FDGTI workers or subcontractors is expressly prohibited unless stringent confined space entry procedures are followed by workers who have been certified for confined space entry.**

The following general procedures are used to monitor ambient air quality:

Choose the appropriate instrument for the contaminant and the space being monitored. A PID or FID can be used to monitor ambient air quality in open areas such as the work space around a drill rig or soil piles. Detector tubes should be used if specific known or suspected chemicals or chemical suites are of concern (e.g., specific organic compounds, mercury, benzene, etc.) PIDs, FIDs and detector tubes cannot be used as the primary indicators of air quality in an enclosed or confined space prior to access because they are incapable of monitoring oxygen level which is of primary importance in an enclosed area. Also, if oxygen is depleted or displaced by hazardous vapors, an FID, which relies on oxygen as a catalyst, is likely to yield an erroneous reading or no reading at all. An LEL/O_2 sensor must be used for this type of monitoring. LEL/O_2 meters should also be used when inerting a storage tank before removal to make sure that the inerting medium has removed sufficient oxygen and combustibles from within the tank.

Start up and calibrate the field monitoring instrument before use; make sure batteries are fully charged (Section 7.7, below, discusses operation of specific instruments).

When monitoring open work areas, take readings periodically; more frequent readings should be taken where elevated contaminant concentrations are encountered (odor can indicate this). Record all readings on an appropriate field form. If vapor concentrations reach or exceed action levels (refer to the site safety plan) take the appropriate action immediately.

When monitoring an enclosed or confined space prior to entering it, use an LEL/O_2 meter and begin monitoring at the entrance and slowly work the probe into the enclosed air space, noting any changes in combustible gas and oxygen concentrations as the probe is moved. Specifically monitor the lowest areas (such as along the floor in a vault) and “dead air” pockets as they can accumulate heavier-than-air vapors and can be oxygen-deficient.

When measuring vapors inside of a building, measure around the perimeter of the room, approximately 1 foot above the floor. Pay particular attention to corners, construction seams and joints, and “dead air” spaces.

When monitoring fixtures such as valves, containers such as drums and tanks, or exhaust ports or stacks on machinery for vapor emissions, take readings in every direction possible, taking readings as close to the source as possible then moving outward into the work space.

7.6.3 Ambient Air Monitoring Using Charcoal Tubes

This practice covers a method for the sampling of atmospheres for determining the presence of certain organic vapors by means of adsorption on activated charcoal using a charcoal tube and a small sampling pump worn by a worker. Annex A1, attached, gives a partial list of chemicals for which this method is applicable, along with their OSHA Permissible Exposure Limits.

It is important to note that water mist and vapor can interfere with the collection of organic compound vapors. Humidity greater than 60% can significantly reduce the adsorptive capacity of activated charcoal. Presence of condensed water droplets in the sample tube will indicate a suspect sample.

A sampling tube consists of a length of glass tubing containing two sections of activated charcoal which are held in place by nonadsorbant material and sealed at each end. The tubes are commercially available and range in size from 100/50 mg to 800/400 mg, which means the tubes are divided into two sections with the front section containing 100 to 800 mg of activated charcoal and the back section containing 50 to 400 mg of activated charcoal. The 100/50 mg tube is the most frequently used. The back section of charcoal adsorbs vapors that penetrate the front section and serves as a warning that breakthrough may have occurred.

Any pump whose flow rate can be accurately determined and be set at the desired sampling rate is suitable. This practice is intended for use with small sampling pumps. Pumps having stable low flow rates (10 to 200 mL/min) are preferable for long period sampling (up to 8 hrs.) or when the concentration of organic vapors is expected to be high. Reduced sample volumes will prevent exceeding the adsorptive capacity of the charcoal tubes. (Suggested flow rates and sampling times are given in Annex A1 for anticipated concentration ranges). At a minimum, pumps should be calibrated before and after sampling. Pumps should be calibrated with the charcoal tube in the proper sampling position.

The following procedures must be followed when monitoring ambient air quality with activated charcoal tubes:

Calibrate the sampling system, including pump, flow regulator, tubing to be used, and a representative charcoal tube (or an equivalent induced resistance) with a primary or calibrated secondary flow-rate standard to +/- 5%. A primary standard practice is given for the calibration of low flow-rate pumps in Annex A2, attached.

Break open both ends of the charcoal tube to be used for sampling, ensuring that each opening is at least one half the inside diameter of the tube.

Insert the charcoal tube into the sampling line, placing the back-up section nearest the pump. At no time should there be any tubing ahead of the sampling tube.

For a breathing zone sample, fasten the sampling pump to the worker, and attach the sampling tube as close to the worker's breathing zone as possible. Position the tube in a vertical position to avoid channeling of air through the adsorber sections.

> **Warning** - assure that the presence of the sampling equipment is not a safety hazard to the worker.

Turn on the pump and adjust the flow rate to the recommended sampling rate.

Record the flow rate and starting time or, depending on the make of pump used, the register reading.

Sampling volumes - the minimum sampling volume will be covered by the detection limit of the analytical method, and the maximum sampling volume will be determined by either the adsorptive capacity of the charcoal or limitations of the pump battery. To calculate the required sampling volume, determine first the sampling range over which it is important to report and exact number, for example from 0.2 to 2 times the permissible exposure concentration, and then calculate the sample volumes as follows:

Minimum sample volume, m^3

$$= \frac{\text{minimum detection limit, mg}}{0.2 \times \text{permissible exposure limit, mg/m}^3}$$

Maximum sample volume, m^3

$$= \frac{\text{tube capacity for vapors, mg}}{2 \times \text{permissible exposure limit, mg/m}^3}$$

Select a sampling rate that, in the sampling time desired, will result in a sample volume between the minimum and maximum calculated above. Generally, a long sampling rate at a low flow rate is preferable. A sample flow rate of less than 10 L/min, however, should not be used. Approximate sample volumes and sample times are given in Annex A1.

When spot checks are being made of an environment, a sample volume of 10 L is adequate for determining vapor concentrations in accordance with exposure guidelines.

At the end of the exposure period recheck the flow rate, turn off the pump, and record the following information: time, register reading, and if pertinent, temperature, barometric pressure, and relative humidity.

Seal the charcoal tube with the plastic caps provided. Label the tube with the appropriate information to identify it.

At least one charcoal sampling tube should be presented for analysis as a field blank with every 10 to 15 samples, or for each specific inspection or field study. The purpose of the field blank is to assure that if the sampling tubes adsorb vapor that is extraneous to the sampling atmosphere, the presence of the contaminant will be detected. To prepare a field blank, break the sealed ends off the tube and cap it with the plastic caps. Do not draw air through the tube, but in all other ways, treat it as an air sample.

Results from the field blanks shall not be used to correct sample results. If a field blank shows contamination, the samples taken during the test must be assumed to be contaminated.

Calculation of sample volume:

For sample pumps with flow-rate meters:

f times t
SQRT
FUNC { P Sample volume, mL =
SUB 1

where:
f = flow rate sampled, mL/min,
t = sample time, min,
P_1 = pressure during calibration of sampling pump (mm Hg or kPa),
P_2 = pressure of air sampled (mm Hg or kPa),
T_1 = temperature during calibration of sampling pump (K), and
T_2 = temperature of air sampled (K).

For sample pumps with counters:
{ (R SUB 2 - R SUB 1) times V } OVER I times P
SUB 1 OVER 760 times 298 OVER { T SUB 1 + 273
} Sample volume, mL =

where:
R_2 = final counter reading,
R_1 = beginning counter reading,
V = volume,(1) mL-count (1),
P_1 = barometric pressure,
T_1 = temperature, ^{o}C, and
T_2 = temperature of air sampled (K).

Samples must be capped securely and identified clearly.

Samples collected in charcoal tubes must not be kept in warm places or exposed to direct sunlight.

Samples of highly vaporous or low-boiling materials, such as vinyl chloride, must be stored and transported in dry ice.

Although there is no published data on the effects of conditions in cargo holds on capped samples, the preferred method is to carry the samples on board.

Ship samples as soon as possible, keeping them stored under refrigeration until they are analyzed, and analyzed if possible within 5 working days.

Migration or equilibration of the sampled material within the sampling tube during prolonged or adverse storage or handling could be interpreted as break-through. This can be prevented by separating the front and back sections immediately after sampling, by having each section in a separate tube and capping them separately.

In some situations, calibration standards may be made up at the facility where the study is being made and submitted as quality control checks.

Never ship or store bulk solvent samples with the collected air samples.

Ambient air quality can also be monitored using activated charcoal diffusional samplers which are similar to the activated charcoal sampling tube discussed above except that the vapors are adsorbed onto the charcoal sampler by diffusion of air as it naturally flows across an area (i.e., no pump is used to collect the sample). The sampling procedure is similar to the procedure described above, except that a pump is not attached to the sample tube, and the sampling rate of the diffusional sampler is determined by either reference to the manufacturer's literature or by subjecting the samplers to known exposure in the laboratory.

7.7 Instruments that Measure Vapor Concentrations in the Field

7.7.1 Instrument Calibration

One of the most important steps in analyzer operation is proper calibration of the instrument. Various calibration techniques may be used depending on the sample's physical or chemical property requiring measurement. Frequency of calibration depends largely on the application, degree of accuracy, and reliability expected.

The following general procedures apply to calibration of field instruments typically used for field vapor monitoring (i.e., PIDs and FIDs):

Perform calibration using a standard reference sample and utilize the analyzer adjustments recommended by the manufacturer.

Consult the instrument supplier to determine the calibration procedure necessary for the particular analysis involved as preliminary instrument adjustments using zero and upscale standards may be necessary.

Charts and calibration curves are essential and should be routinely verified.

The standard used for calibration must be as representative as possible of the compound(s) to be analyzed (e.g., isobutylene to simulate benzene), although it cannot always contain representations of all potential interfering substances.

Use a calibration standard of known concentration.

Check all operating parameters of the system in accordance with the instrument specifications and data for specific analysis. Allow sufficient time for the analyzer to reach equilibrium as indicated by a stable output.

Introduce the standard reference sample into the analyzer using the recommended instrument operational procedure.

After sufficient standard has been allowed to flow through the analyzer, adjust the readout to conform with the benchmark value (this applies to the use of both zero gas and calibration gas). This establishes a single calibration point. Many newer instruments calibrate their readouts automatically during this procedure.

Continue introducing standard sample and record analysis after a stable response is achieved.

Discard any standard when any change in composition is detected.

Specific instructions for calibrating each type of instrument are presented in the operational procedures presented below.

7.7.2 Flame Ionization Detectors

Flame ionization detectors (FIDs) generate electrical current when gases containing carbon atoms are oxidized to carbon dioxide in a hydrogen flame and potential is applied across the flame. The magnitude of the electric current generated is termed the detector response. FIDs are responsive to hydrocarbon contaminants in a vapor stream and are commonly employed for this purpose. FIDs are durable for field use, and have a wide linear range and nearly uniform response to organic gas species. FIDs are generally unresponsive to inorganic gases and water vapor. Although versatile, these detectors are not selective for halogenated compounds (i.e., chlorinated organics). Also, They require supplies of fuel gas which require careful safety practices in handling and flame ignition.

Older models of FIDs have analog (needle gauge) readouts while newer models are generally digital. The older models also contain more manual controls and adjustments while the newer computerized instruments perform certain standard functions (such as flame ignition and shut-down) automatically. The following are general procedures for operating a typical (e.g., Foxboro Brand) FID. Procedures will vary from one brand to another and one model to another, so these procedures may not all apply to a specific instrument. A field instrument should never be used without first reading through the owner’s manual because improper operation of an FID is potentially dangerous and is likely to yield erroneous data.

Review owner’s manual before use.

CAUTION: The FID uses hydrogen gas which is extremely flammable and potentially explosive. Great care should be taken to avoid all external sources of ignition when re- filling the gas cylinder and when operating the instrument. Follow start-up and shut-down procedures exactly to avoid leakage of hydrogen gas. And follow all safety regulations for transporting flammable pressurized gases when transporting or shipping an FID.

Always start with a fully charged battery and a half to full supply of hydrogen in the gas cylinder.

☐

Turn the instrument on and follow the designated start-up procedure for the specific unit: it will involve a sequence that includes opening the gas cylinder valve (some instruments will inform the user when to open the gas valve).

Once the gas is turned on it must be ignited, either using a manual ignition switch or automatically by the instrument itself. Wait the appropriate time before igniting the flame (some instruments will prompt the user when ready for ignition).

Observe flame (ignition) indicator; if the indicator does not show that the flame has ignited, repeat the flame ignition procedure according to instrument instructions.

Once the flame has been ignited, allow the unit to stabilize for at least 5 minutes.

Follow manufacturer's instructions to calibrate the instrument to a zero reading. This will involve connection of the instrument probe to a container of "zero gas" (i.e., ultra-clean air) which allows the instrument to be "zeroed". If zero air gas is unavailable, the instrument can be zeroed to clean ambient air (i.e., move away from work areas, storage facilities, motor vehicles and any other potential source of vapors before calibrating).

Follow manufacturer's instructions to calibrate the instrument using a standard calibration gas. This will involve connection of the instrument probe to a container of calibration gas (standard FDGTI calibration gas is a known concentration of isobutylene in air). Generally, the "response" of the calibration gas against the actual contaminant (e.g., isobutylene to benzene) must be entered into the calibration sequence - the owners manual should provide a procedure and conversion table to facilitate this. This will adjust the instrument's response so that it will read the concentration of the actual contaminant correctly.

When calibration is completed, the instrument is ready to use. If the instrument has a manual range setting (e.g., readout x10, x100, x1,000) set it for the anticipated contaminant concentrations.

Place the end of the sampling probe near the material being tested Air will be drawn into the probe by suction.

Read vapor concentrations on the appropriate scale.

DO NOT allow pressure in the gas cylinder to drop below a prescribed amount (about 50 p.s.i.) because loss of pressure will cause contamination of the cylinder.

When finished using the instrument, check the amount of charge left in the battery and the pressure in the gas cylinder. Follow the manufacturer's instructions for shutting the instrument off. It is extremely important to be certain the flame has been properly extinguished and the gas flow turned completely off.

If gas in the cylinder is not sufficient for another day's use, refill the cylinder with hydrogen gas following all safety precautions involving hydrogen.

If the battery is not sufficiently charged for another day's use, recharge it. Note in the owner's manual if there is a maximum allowable recommended charging time and don't exceed it (some battery chargers automatically shut off and some don't). Also, certain types of batteries last longer if fully discharged before recharging while others can be damaged by fully discharging them. Find out what kind of battery your instrument has and treat it accordingly.

Most instruments have one or more filters (air inlet and/or outlet filters)that should be removed, inspected and cleaned between daily uses.

7.7.3 Photoionization Detectors

Photoionization detectors (PIDs) employ ultraviolet radiation to ionize contaminant molecules. Positive ions and free electrons are formed which migrate to the detector electrode(s), resulting in an electric current that is proportional to contaminant concentration at the detector. PIDs are extremely sensitive to aromatic hydrocarbons due to the great efficiency of ionization of pi bonds under ultraviolet radiation. Efficiency of ionization of sigma bonds is lower, resulting in a higher PID detection limit for aliphatic hydrocarbons. The selectivity of the method can be adjusted by selecting lamps of different energies, causing a change in response of contaminants with fixed ionization potentials to changing lamp energies. Tables exist of ionization potentials of compounds within classes common to vapor-phase contaminants. Methane has an ionization potential higher than the energies of commercially available lamps, limiting the PID to detection of compounds other than methane. PIDs are further limited by their tendency to conceal the presence of low-sensitivity compounds when high-sensitivity compounds (aromatics) are present. PID response can be impacted by condensation of water vapor in the lamp.

As with FID, older models of PIDs have analog (needle gauge) readouts and manual controls while newer models are generally computerized and digital. The following are general procedures for operating a typical (e.g., HNU Brand) PID. Procedures will vary from one brand to another and one model to another, so these procedures may not all apply to a specific instrument. A field instrument should never be used without first reading through the owner's manual because improper operation of an PID can yield erroneous readings that could place site workers in jeopardy if using the instrument to monitor ambient air quality.

Review owner's manual before use.

Check to see that the battery is properly charged before use.

Follow manufacturer's instructions to calibrate the instrument to a zero reading. This will involve connection of the instrument probe to a container of "zero gas" (i.e., ultra-clean air) which allows the instrument to be "zeroed". If zero air gas is unavailable, the instrument can be zeroed to clean ambient air (i.e., move away from work areas, storage facilities, motor vehicles and any other potential source of vapors before calibrating).

Follow manufacturer's instructions to calibrate the instrument using a standard calibration gas. This will involve connection of the instrument probe to a container of calibration gas (standard FDGTI calibration gas is a known concentration of isobutylene in air). Generally, the "response" of the calibration gas against the actual contaminant (e.g., isobutylene to benzene) must be entered into the calibration sequence - the owners manual should provide a procedure and conversion table to facilitate this. Also PIDs generally have a “span” setting that is used to calibrate the instrument to a known gas. Follow manufacturer instructions regarding the proper span setting as this is critical for obtaining accurate readings.

When calibration is completed, the instrument is ready to use. If the instrument has a manual range setting (e.g., readout x10, x100, x1,000) set it for the anticipated contaminant concentrations.

Place the end of the sampling probe near the material being tested. The internal fan will draw an air sample through the probe tip. Avoid placing the probe too close to the sample to reduce the intake of excessive soil and dirt which may damage the probe or cause the fan to lock.

PID readings are relative to the ambient air temperature; the same sample will yield a higher concentration reading at a higher ambient air temperature than at a lower air temperature. For consistency, monitor all soil samples at room temperature (not less than 70 degrees F (21^{o} C)).

Record the ambient air temperature at which the samples were measured.

Be particularly careful not to allow the probe to suck in moisture. Even a small amount of moisture can condense on the lamp and affect its performance (or cause it to stop working). If this happens, shut off the instrument and follow the owner’s manual procedures for disassembling, inspecting and cleaning the lamp. **Be extremely careful not to scratch or mar the surface of the lamp when cleaning or drying it Use only a very soft cloth or tissue.** In-line moisture traps are available for use in wet conditions.

Some PIDs are sensitive to air currents; widely fluctuating readings will usually indicate air movement past the end of the probe.

When finished using the instrument, check the amount of charge left in the battery. Follow the manufacturer’s instructions for shutting the instrument off.

If the battery is not sufficiently charged for another day’s use, recharge it. Follow manufacturer’s instructions regarding recommended battery charge/discharge procedures.

Inspect the lamp between daily uses to make sure its surface is clean, dry and undamaged. If cleaning is necessary, follow the owner’s manual procedures for disassembling, inspecting and cleaning the lamp. **Be extremely careful not to scratch or mar the surface of the lamp when cleaning or drying it Use only a very soft cloth or tissue.** If the lamp surface is scratched, inform the person in charge of instrument maintenance.

Most instruments have one or more filters (air inlet and/or outlet filters)that should be removed, inspected and cleaned between daily uses.
PIDs are outfitted with 10.2 eV lamps for monitoring petroleum hydrocarbon vapors. This lamp will not detect methane nor will it detect the volatilized halogenated (chlorinated) compounds. To monitor chlorinated compounds, an 11.7 eV lamp must be used (this will

easily interchange with the 10.2 eV lamp). The 11.7 eV lamp cannot be used to monitor petroleum hydrocarbons.

When switching lamps, the span setting must be adjusted to match the compound you are testing for. Follow manufacturer's instructions to do this.

On an HNU PID, Span settings for typical compounds are:

1,2 Dichloroethane	5.5
Benzene	5.0
Methylene Chloride	4.4
Carbon Tetrachloride	4.0
MEK	1.8
Chloroform	1.5

7.7.4 Combustible Gas Indicators and Oxygen Sensors

Instruments capable of analyzing air for the presence of combustible gas and oxygen are generally referred to as lower explosive limit/oxygen meters (or LEL/O_2 meters), because combustible gas readings are generally given as the percentage of the lower explosive limit (%LEL) of the vapors present in the air being tested. The meter will also read the concentration of oxygen in the air as a percentage.

LEL/O_2 meters are generally used to monitor air quality in enclosed or confined spaces prior to entry and to monitor the vapors in storage tanks during tank removal operations.

Vapors associated with gasoline are the most common substances monitored by FDGTI personnel. Gasoline's flammable range is 1.4 to 7.6% by volume in air. Therefore, 1.4% is the LEL for gasoline (below 1.4%, not enough fuel vapors are available to burn. Therefore 100% LEL on an LEL/O_2 meter corresponds to 1.4% gasoline vapor by volume. A reading of 10% LEL or greater requires that all work be stopped and personnel leave the area immediately.

Normal atmosphere is 21% oxygen by volume. The safe breathing range is 19.5 to 21%. A reading above or below this range requires that all work be stopped and personnel leave the area immediately.

The following are general procedures for operating a typical LEL/O_2 sensor. A field instrument should never be used without first reading through the owner's manual because improper operation of an LEL/O_2 meter can lead to personnel working in dangerous conditions.

Review owner's manual before use.

Always start with a fully charged battery, and battery charge should be checked periodically throughout the day.

Start the instrument up and check calibration according to manufacturer's instructions. The instrument must be properly calibrated for the substance(s) being monitored. If the sensor will not calibrate it cannot be used and must be sent to a qualified service center for repair.

Most instruments have an alarm that is either pre-set to sound above or below a certain reading, or can be set by the user. Be aware of how the alarm is set, and if it can be manually adjusted, set it to sound at a reading of 10% LEL.

To take a reading, insert the probe into the space where air quality is being monitored and hold it there until the reading stabilizes. Note also the maximum LEL value that registered on the instrument. Take appropriate action as necessary.

For large spaces such as basements, the probe should be moved through the air space and any variations noted as it is moved. Take particular note of air quality close to the floor, in corners and near seams. When monitoring fixtures such as valves take readings in every direction possible, and as close to the fixture as possible.

For periodic readings, the instrument should be cleared after each reading, particularly after high levels of contaminant vapors have passed through the instrument. Move away from all vapor hazard areas, move fresh air through the instrument, and check that the instrument is properly reading ambient air quality.

After taking readings of particularly high vapor concentrations, the instrument may have to be recalibrated. Erratic readings or failure of the LEL to decline back to ambient air readings indicate the need for recalibration It will sometimes take several minutes for residual vapors in the instrument to "burn off"). If the instrument can't be recalibrated properly, the sensor may have been poisoned and the instrument is no longer functional.

Avoid the introduction of dirt, water or other liquids into the probe as these substances can foul the sensors. Also, tetra-ethyl lead and silicon can poison the sensors by permanently coating them, so prolonged use of the instrument in atmospheres containing these substances should be avoided.

When finished using the instrument, check the amount of charge left in the battery. Follow the manufacturer's instructions for shutting the instrument off.

If the battery is not sufficiently charged for another day's use, recharge it. Follow manufacturer's instructions regarding recommended battery charge/discharge procedures.

If the instrument has an inlet or outlet filter, it should be removed, inspected and cleaned between daily uses.

7.7.5 Detector Tubes

A detector tube consists of a glass tube containing an inert granular material that has been impregnated with a chemical system which reacts with the gas or vapor of interest. As a result of this reaction, the impregnated chemical changes color. The granular material is held in place within the glass tube by porous plugs of a suitable inert material. The ends of the glass tube are flame-sealed to protect the contents during storage.

Detector tubes may be used for either short-term (grab) sampling (1 to 10 minutes), or long term (dosiometer) sampling (1 to 8 hours) of atmospheres containing toxic gases or vapors. This document will address the use of detector tubes for short-term sampling only.

A given volume of air is pulled through the tube by a mechanical, hand-operated, aspirating pump. Two types of pumps are commercially available: piston-operated and bellows-operated. The pumps generally have a capacity of 100 mL for a full pump stroke. By varying the number of pump strokes, the sample volume is controlled. If the substance for which the detector tube was designed is present, the indicator chemical in the tube will change color. The concentration of that substance may be estimated by either (a) the length-of-stain compared to a calibration chart, or (b) the intensity of the color change as compared to a set of standards.

To monitor vapors using detector tubes, carefully follow the instruction sheet of the manufacturer for the proper use of each detector tube. In general, the instruction sheet will include the following information:

- storage conditions,
- shelf life,
- chemical reaction and color change,
- test procedure,
- significant interferences,
- temperature and humidity correction factors, if necessary,
- correction for atmospheric pressure, and
- measurement range.

Sampling pumps should be maintained and calibrated periodically. Each manufacturer provides a maintenance and calibration procedure for his particular pump.

The Safety Equipment Institute has a certification program for certain detector tubes used in short-term monitoring, where the tubes are required to meet a minimum level of accuracy. In general, the accuracy of any detector tube depends on the construction and chemistry of the tube along with the actual composition of the test atmosphere and the conditions under which the tube is read. There is also some variation in accuracy between manufacturers tubes designed to detect the same compound(s). Therefore the user should verify the accuracy with the tube manufacturer or run his own tests to determine accuracy. The accuracy of a detector tube towards a specific compound depends on the cross-sensitivity of the tube to other gases or vapors present in the test atmosphere, the characteristics of which are generally unknown. Accuracy is therefore expected to be limited when sampling unknown atmospheres.

Table 1, attached, presents a partial list of chemicals which can be monitored using detector tubes.

7.7.6 Portable Gas Chromatograph

The portable gas chromatograph allows qualified personnel to quickly and accurately characterize contaminants in water, soil and air, on site and in real time.

The basis of chromatography is the separation of various components of a sample due to their differences in solubility or in adsorption on a stationary bed of material. The sample is introduced into a carrier gas as a vapor which flows through the chromatographic system. Upon separation by the stationary phase, the sample travels through the column at different speeds, entering the detector where individual components are identified, then recorded.

Because of the gas chromatograph's speed, it can be used as an emergency response tool. Other uses include: leak detection and evaluation; mapping of a contamination plume; screening for contaminants; pollutant identification; continuous monitoring of target chemicals; and, waste site profiling.

Compounds which can be detected to 1 ppb or less include:

- ethylene oxide
- vinyl chloride or other chloroethylenes
- benzene, toluene, xylene, ethane and higher alkanes to octane
- isoprene
- ethylene
- methylene chloride and other chloromethanes
- light chlorobenzenes
- hydrogen sulfide
- light mercaptans
- organic sulfides to DMDs
- acetone and MEK
- arsine
- phosphine
- acetaldehyde
- aldehydes up to hexanal

Compounds detectable to 50 ppb or less:

- glycol ethers
- fluorochloromethanes (freons)
- methyl isocynate
- chloroethanes
- cyclohexanone
- ethyl acrylate
- light alcohols

Consult with the engineering staff or staff chemist when preparing any site investigation, water treatment system, pump test or other procedure where the portable gas chromatograph may be applicable.

EXAMPLE OF COMPLETED FIELD FORM

JAR HEADSPACE ANALYSIS

(Readings in Parts Per Million (PPM))

PROJECT: Petro Spill 1,000
ANALYST: D. Sniffer
DATE: 5-5-88 TEMP.: 60 F.
INSTRUMENT: HNU PI-101 LAMP : 10.2 eV. NO.: 4
CALIBRATION DATE: 5-4-88

Background Concentration: 5 ppm

SOIL SAMPLE ID	JAR VOLUME	DEVELOPMENT PERIOD (min.)	SCALE	REPLICATE PEAK READINGS A	B	DIFFERENCE (as % of scale)
SS-1	16 oz.	10	20	12	15	15
SS-2	16 oz.	10	200	45	35	5
SS-3	16 oz.	10	2000	265	310	2
SS-4	16 oz.	10	20	ND	4	20

NOTE: ND = not detected.

XXXXXXXXXXXXXXXXXXXXXXXXXXXXXX
ATTACH ANNEXES A1 AND A2 FROM ASTM D 3686
XXXXXXXXXXXXXXXXXXXXXXXXXXXXXX

XXXXXXXXXXXXXXXXXXXXXXXXXXXXXX
ATTACH TABLE 1 FROM ASTM D 4490
XXXXXXXXXXXXXXXXXXXXXXXXXXXXXX