

FIELD AND LABORATORY PROCEDURES
FOR
SOIL VAPOR SAMPLING

Playa Vista
Los Angeles, California

Prepared by:
Exploration Technologies, Inc.
3698 Westchase Drive
Houston, Texas

January 5, 2000

TABLE OF CONTENTS

- 1.0 INTRODUCTION
- 2.0 FIELD PROCEDURES
 - 2.1 Preparation of Soil Vapor Sampling Bottles
 - 2.2 Collection of Soil Vapor Samples
 - 2.3 Quality Control Samples
 - 2.4 Field Recording of Samples
 - 2.5 Field Labeling/Recording of Samples
 - 2.6 Shipment of Samples
 - 2.7 Chain of Custody Procedures/Documentation
 - 2.8 Water Source
 - 2.9 Disposition of Soil Vapor Collection Holes
- 3.0 CHAIN OF CUSTODY AND DOCUMENTATION
 - 3.1 Field Logbook
 - 3.2 Sample Documentation
 - 3.2.1 Sample Labels and/or Tags
 - 3.2.2 Chain of Custody Records
 - 3.3 Corrections to Documentation
 - 3.4 Investigation Derived Waters
- 4.0 LABORATORY PROCEDURES FOR ANALYSIS OF SOIL VAPOR SAMPLES
 - 4.1 Summary of Methodology
 - 4.2 Suite of Analysis and Reporting/Detection Limits
 - 4.3 Interferences
 - 4.4 Data Collection and Archival
 - 4.5 Calibration and Results
 - 4.6 Quality Control
 - 4.7 Sample Analysis and Holding Times

1.0 INTRODUCTION

The field procedures and protocols implemented for the Exploration Technologies, Inc. (ETI) proposed soil vapor sampling methodology is described in this plan. This Field Sampling Plan (FSP) describes the methodologies to be used during collection and analysis of soil vapor samples and the requirements for documentation and reporting.

In preparing this soil gas work plan, the following documents were consulted and implemented in developing the proposed field and analytical procedures:

- ASTM D5314-92 *Standard for Soil Gas Monitoring in the Vadose Zone*
- Los Angeles RWQCB *Interim Guidance for Active Soil Gas Investigation*

2.0 FIELD PROCEDURES

The field procedures to be used during collection of soil vapor samples are as follows:

2.1 Preparation of Soil Vapor Sampling Bottles

All soil vapor samples are collected in 22, 50 or 125-cubic centimeter (cc) glass serum bottles, depending upon available soil gas volumes. All bottles are pre-washed and soaked by filling with a detergent solution for 24 hours. These sample bottles are rinsed by filling with water and soaking for an additional 24 hours. After rinsing, the bottles are heated to 150^o C for 24 hours, purged with pre-purified nitrogen (defined as 99.998% pure nitrogen with maximum levels of oxygen, total hydrocarbons and water not to exceed 5 parts per million volume (ppmv), 1 ppmv and 3 ppmv, respectively), capped and sealed with a butyl rubber septum and a crimped aluminum cap with a removable center protector.

2.2 Collection of Soil Vapor Samples

Soil vapor samples are collected in accordance with the following procedures and methodology:

- 2.2.1. Before initiating field activities, a utility locator will survey and clear each proposed boring or sampling location for any subsurface utilities or interferences. If an underground utility is identified within the proposed sampling location, the boring will be repositioned or relocated nearby and resurveyed for underground utilities.
- 2.2.2. After each sampling location is cleared of utilities, the sample hole is made with a manually operated ½- inch outside diameter steel plunger bar to the specified sampling depth of 4, 7 or 12 feet below ground surface. This is generally located within the vadose zone above the capillary fringe, although water samples can also be collected through ETI's soil gas probe.

- 2.2.3. For each sampling location, two of the pre-prepared septum top glass 125-cc sample bottles are evacuated onsite with a hand pump to a vacuum of approximately 20 inches of mercury for use in collecting soil vapor and ambient air samples.
- 2.2.4. After each boring has been punched to the specified sampling depth, the ½inch outside diameter plunger bar is removed from the hole.
- 2.2.5. Before inserting the stainless-steel sampling probe into the pre-drilled borehole, one of the evacuated sample bottles is attached to a three-way stop cock valve mounted on the top of the probe with a new 20-gauge needle attached to a 60 cc hypodermic syringe. The three-way valve is opened to allow a sample of ambient air to fill the evacuated bottle through the sampling probe and to collect a background air sample for quality control between sampling locations. An additional 60 cc of ambient air is injected into the blank sample bottle using the new syringe, after which the sample bottle is removed from the valve and the puncture hole is sealed with a silicone rubber adhesive sealant.
- 2.2.6. After the blank sample is collected, the sampling probe is inserted into the sample hole and purged by withdrawing at least 15 cc of ambient air using the syringe mounted on the three-way valve attached to the top of the probe. The stainless steel sampling probe has an outside diameter of ½inch and an inside diameter of 1/8 inch and a perforated tip for collecting the soil vapor sample at the bottom of the pre-drilled hole. This volume of purge is adequate to remove ambient air from the probe, while providing minimal disturbance to the soil gas near the probe tip. A 4-foot-long sampling probe with a 1/8-inch inside diameter has an internal volume of 9.65 cc.
- 2.2.7. Collection Of Light Methane through Butane Volatiles. The four foot long pounder bar is then removed from the hole and a four foot long sampling probe is immediately inserted into the four foot deep sampling hole. A 22 cc septum top sampling bottle is evacuated on-site with a hand pump to a vacuum of approximately 20 inches of mercury.

Before the soil gas sample is collected in the sample bottle, the probe is purged by withdrawing 15 cc of soil gas from the subsurface through the probe using the 60 cc syringe mounted on the three-way valve located on the top of the probe. Following this purging of the probe with the subsurface soil gas, the 22 cc evacuated sampling bottle is placed on the probe needle with the three-way valve closed to the bottle. A 60 cc syringe is then used to extract 50 cc's of soil gas through the probe. The three-way valve is rotated to connect the bottle to the syringe and this 50 cc of soil gas is injected through the three-way valve into the 22 cc bottle to overpressure the sampling bottle to approximately 2 atmospheres. The sample bottle is removed and sealed with a silicone rubber adhesive cement (as above for the blank).

- 2.2.7.1 Collection and Analysis of Hydrogen Sulfide. If real-time analysis for hydrogen sulfide (H₂S) is desired, then an H₂S sensitive instrument (typically a Jerome 631-X) is attached directly to the probe. For this analysis the stainless steel probes should be coated with teflon to reduce adsorption of H₂S in the probe. The Jerome instrument is set in its most sensitive mode. In this mode the instrument is programmed to extract 25 cc of soil gas from the probe using its internal sampling pump. The sample inlet for the H₂S instrument is attached with a very short tygon tube (no more than 2 inches in length) to the top of the probe and 25 cc of soil gas is withdrawn and analyzed on site for H₂S. The H₂S reading is recorded on the field sheet and in the field notebook.
- 2.2.7.2 Collection and Analysis of BTEX (Benzene, Toluene, Ethylbenzene and Xylenes). If analysis for BTEX (or chlorinated solvents) is desired, then a second 22 cc septum top bottle is evacuated on site and attached to the top of the probe. The three-way valve is set to isolate the sampling bottle. The 60 cc syringe is again used to extract an additional 50 cc of soil gas through the probe. The three-way valve is rotated to connect the second bottle to the syringe and the second 50 cc of soil gas is injected into the 22 cc bottle. The second bottle is removed and sealed with a silicone rubber adhesive cement (as above for the blank and the first sampling bottle). The syringe is discarded following the collection of the last sample at each location.
- 2.2.7.3 Collection of Soil Gas Samples for Analysis of Carbon and Hydrogen Isotopes. For analysis of carbon and hydrogen isotopes, a third 125 cc bottle can be used to collect an additional sample. This third bottle is evacuated on site and mounted on the probe, where the syringe and three-way valve can be used to collect this isotope sample. Using the syringe, this third bottle is filled to atmospheric pressure and then over-pressured by 60 cc to prevent leakage. The positive pressure on the bottle ensures that no exchange of sample gases with atmospheric air occurs during shipping from the field to the laboratory.
- 2.2.8. All sampling equipment is decontaminated between sample collection. The ½ inch- diameter sampling probe is washed both outside and inside by injecting a detergent solution through the probe, followed by a distilled water rinse before for collecting a soil vapor sample from each location. After rinsing, the inside of the probe is flushed with compressed air at approximately 25 pounds per square inch (psi) pressure using bottled breathing air.

The ETI sampling protocol is designed to collect only a small volume of equilibrium soil vapor sample from the subsurface sediments at the selected

sampling depth under various conditions. If impermeable and/or water saturated soils are encountered at the selected sampling depth, the field personnel will observe a significant vacuum in the syringe mounted on the three-way valve such that the syringe plunger cannot be withdrawn. It will be necessary to relieve the high vacuum before a soil gas sample can be collected. In cases where high vacuum is encountered, one of the following options can be implemented depending on actual conditions in the field:

2.2.8.1 The probe can be pulled up a few inches to clear the free water and/or wet clays that are sealing the bottom of the probe tip.

2.2.8.2 A new hole can be redrilled one to two feet from the initial sampling location. In most cases, this impermeable subsurface condition is not uniformly present across the site.

Under extreme impermeable conditions, the volume of the sample to be collected can be reduced from 125 cc to 50 cc or even 22 cc.

All sampling equipment is decontaminated between sampling locations. The manually operated sampling probes and any other field equipment is decontaminated between sampling locations using a high-pressure steam cleaner. Waste or rinse water generated during steam cleaning and decontamination is contained for proper disposal offsite. The soil vapor probe is also steam-cleaned, washed with soap, rinsed and blown dry with compressed air, using bottled breathing air as described above.

2.3 Quality Control Samples

Quality control samples will include ambient air samples collected through the probe at each location and one trip blank for each day of field activity. All trip blanks and 20 percent of ambient air samples collected will be analyzed using the same analytical procedures for the suite of analytes proposed for the soil vapor samples.

2.4 Field Recording of Samples

All soil vapor collection bottles will be labeled at each sample site with an appropriate map or grid reference number. A base map will be posted daily with all completed sites, and a list of samples collected will be retained by the sampler as part of the field notes. A copy of the field form to be used during soil vapor sampling is attached.

2.5 Field Labeling/Recording of Samples

A bound record book will be used by field personnel to document and record field observations and data collected during soil vapor sample collection. The record will include the times, locations, and the person collecting the samples. Each soil vapor sample container will be labeled in the field with the following information: site number, sample collection depth, date and time of sample collection, person collecting the sample. Records of field observations/measurements will be maintained for record keeping.

2.6 Shipment of Samples

Samples will be shipped/delivered to ETI's, or to any other designated analytical laboratory for analyses of constituents of concern following the recommended procedures of the U.S. Environmental Protection Agency (EPA) and American Society for Testing and Materials (ASTM). Samples are shipped/delivered to the designated analytical laboratory within 24 hours of collection and within the specified holding times for each analysis following appropriate chain of custody procedures as described below.

2.7 Chain of Custody Procedures/Documentation

A chain of custody form will accompany all samples collected and submitted to ETI's, or to any other designated laboratory for analysis, and are maintained as part of record keeping and documentation of the soil vapor sampling activities. All samples are maintained under chain of custody control during transportation and until transfer and receipt by the laboratory. Immediately upon receipt by the laboratory, the samples are logged in with the appropriate sample designation, matrix, time and date of sampling, analyses required, client, and the sample designation. A copy of the chain of custody form is attached.

2.8 Water Source

An onsite potable water source will be identified by site personnel for use during field activities. Deionized water used for decontamination is normally purchased from a retail store.

2.9 Disposition of Soil Vapor Collection Holes

After the soil vapor samples are collected, each soil gas sample hole is backfilled with bentonite and/or neat cement as required by the local culture and finished to grade to match existing surface materials. All wastes generated during equipment cleaning are managed in accordance with the appropriate environmental procedures.

3.0 CHAIN OF CUSTODY AND DOCUMENTATION

The following section describes the project documentation requirements and procedures to be followed during field activities and sampling.

3.1 Field Logbook

A bound logbook dedicated to the project that has consecutively numbered pages is maintained. All fieldwork performed is recorded in this logbook. At a minimum, the following information is included in the logbook:

- Date and time of arrival and departure
- Weather conditions
- Personnel on site
- Level of personal protection
- Deviations from work plan standards
- Purpose of site visit

- Timed entries of the site activities performed
- All sample identification numbers and description of sample (including related QC samples)
- Field instruments used and calibration information
- Description of the number of shipping coolers and shipping method
- Name of receiving laboratory or laboratories
- Signature of the person maintaining the logbook

In cases where separate field sheets or forms are used to record data, the specific sheets are referenced by title in the logbook. All entries in the logbook will be made with waterproof markers. The logbook is maintained for record keeping for the duration of the project.

Other information, which is recorded, includes:

- Field screening instrument readings, if any
- Brand name and amount of each material used
- Any problems encountered and their resolutions
- Date and time of start and completion of soil gas samples, and notation as to depths
- Boundaries between individual lithologies

3.2 Sample Documentation

The following sections describe the sample documentation procedures that will be used during soil vapor sampling. Complete sample documentation is required from the time of sample collection to the preparation of analytical reports to ensure the integrity of sample data generated.

3.2.1 Sample Labels and/or Tags

Each sample collected will have a label affixed immediately following sample collection. If more than one container is collected for each location, then each container from that sample location will have identical information on the sample labels plus information regarding the time that each sample is collected. Each sample label will contain the following information:

- Project code, site name, or project number
- Sample identification number
- Sampler's name
- Preservative information
- Requested analysis
- Date and time of collection
- Type of sample, either soil gas or water

3.2.2 Chain of Custody Records

Chain of custody (COC) documents is used to maintain a record of sample collection, transfer of samples between personnel, sample shipping, and receipt by the laboratory. Sample information is entered on the COC documents at the time of sample collection. If there is any transfer of samples prior to shipment, the COC will reflect the change of possession. Samples are considered to be under custody if one or more of the following criteria are met:

- The sample is in the sampler's possession
- The sample is in the sampler's view after being in possession
- The sample was in the sampler's possession and was then locked up to prevent tampering
- The sample is in a designated secure area

All samples, including quality assurance/quality control samples, will be entered on a COC form. The COC form will include name, address, phone number, and project contact; project code, site name, and project number; full sample identification numbers; sampler's name; sample matrix; sample type; number of sample containers for each identification number; requested analyses; and any other pertinent information required by the laboratory. The COC form will be signed, dated, and timed by the relinquishing and receiving party each time sample possession is transferred. Transfer of sample custody will be kept to a minimum to simplify the COC record.

3.3 Corrections to Documentation

Any corrections made to field documentation, either in the field or during review, will be made by a single strike-through, the correct information will be recorded adjacent to the corrected information, and the person making the correction will initial and date next to the correction. The person who made the initial entry will make the corrections.

3.4 Management of Investigation-Derived Wastes

Waste soil and water generated during field activities and soil vapor sampling will be stored on site. These investigation-derived wastes (IDW) will be stored in proper containers pending characterization and proper disposal to a permitted facility.

4.0 LABORATORY PROCEDURES FOR ANALYSIS OF SOIL VAPOR SAMPLES

4.1 Summary of Methodology

Analysis of the permanent gases and light hydrocarbons in a gaseous sample is accomplished using gas chromatographs following a modified procedure outlined in EPA Method 8000 in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW-846 (Third Edition). If a sample loop is used to introduce the sample onto the columns, it is attached to a multi-port valve and is flushed with the carrier gas following rotation of the valve. Direct injection by gas tight syringe is acceptable. The permanent gases are analyzed using a thermal conductivity detector (TCD). The light hydrocarbons are analyzed using a flame ionization detector (FID). C5+ compounds are analyzed using a flame ionization detector (FID). The data is transferred to a computer where it is converted to digital format, stored, and processed using a chromatography data system.

This method is recommended for use by (or under the supervision of) analysts experienced in sample preparation, the operation of gas chromatographs and in the interpretation of chromatograms.

4.2 Suite of Analysis and Reporting/Detection Limits

Concentrations of analytes in the gas sample will be reported in percent by volume (for permanent gases) and parts per million by volume (PPMV) in accordance with the following detection limits:

Light Hydrocarbons	Reporting limits, FID	Reporting limits, TCD
Methane *	0.04 PPMV	0.10% *
Ethane	0.01	PPMV
Ethene	0.01	PPMV
Propane	0.01	PPMV
Propene	0.01	PPMV
I-Butane	0.01	PPMV
N-Butane	0.01	PPMV

Permanent Gas	Reporting limits, TCD
Hydrogen	0.5 PPMV
Carbon dioxide	0.03%
Oxygen	1%
Nitrogen	5%

*** NOTE:** *Samples and standards that contain high levels of methane must be reported using both TCD and FID methods. The results must agree to within 15% RPD.*

C5 Plus Analyses

The C5 plus analysis, which includes BTEX, contains many compounds, depending upon the fuel types present on the site. Because of the wide variations possible from site to site, it is generally convenient to group and report the C5 plus data according to the relative boiling points of the following compounds:

C5-Benzene

The sum of all hydrocarbons with a boiling point greater than pentane and less than benzene are reported as ppmv benzene equivalents.

Benzene-Toluene

The sum of all hydrocarbons with a boiling point equal to or greater than benzene and less than toluene are reported as benzene equivalents.

Toluene-Xylene

The sum of all hydrocarbons with a boiling point equal to or greater than toluene and less than xylene are reported as benzene equivalents.

Xylene Plus

The sum of all hydrocarbons with a boiling point greater than p-xylene are reported as benzene equivalents.

The reporting limit of each group of components in the C5+ analysis is 1.0 PPMV.

4.3 Interferences

The most likely source of "interference" is ambient air. Due to the relatively high concentrations of oxygen and nitrogen in air, a very small amount of air as a contaminant will seriously skew the results. The analyst must take care to ensure that air is flushed from the gas tight syringe before sample preparation and that no air has entered the syringe or needle prior to injection of the sample into the gas chromatograph.

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. An unrestricted flow of pure carrier gas from a 10 psig source should be allowed to flow through each sample loop for 30 seconds prior to each analyses.

Syringes should be cleaned with laboratory soap and water (Alconox or equivalent) between sample extraction and analysis to insure absence of carryover from previous samples.

As required, the analyst should demonstrate the absence of carryover contamination by analysis of the contents of the sample loop when purged with carrier gas. This demonstration should be performed when carryover contamination is suspected (after high samples). In the event that 'ghost peaks' (peaks similar to previous sample) appear when a pure carrier gas sample is analyzed, measures should be taken to eliminate the carryover contamination.

4.4 Data Collection and Archival

The output of the chromatograph is directed to a computer where the signal is converted to digital format, stored, and processed using a chromatography data system.

Tabulated data is to be made available in electronic format as specified by the client. Data will be preserved and archived for a period of time as specified by the client.

4.5 Calibration and Results

The standard calibration gas should be introduced in the same manner, as is the sample (sample loop or direct injection). Measured peak areas are converted to concentrations using certified commercial gas standards traceable to NIST standards (Matheson Gas Products and Scott Specialty Gases). Dilutes may be made to achieve multi point calibration curves.

Initial calibration is accomplished by analyzing multiple standards of appropriate calibration ranges. The results should agree to within 10% RPD. These results will be used to establish a multi-point calibration curve.

A Continuing Calibration Verification (CCV) standard will be run for every 20 samples (or more frequently if contractually required). If the instrument response for any CCV standard varies by more than 20%, the analyst will not analyze samples until the reason is determined and the problem is corrected.

4.6 Quality Control

The quality control procedures to be implemented for analysis of soil gas samples for the analytes listed in Section 2.0 shall be as follows:

- 4.6.1 If the requirements set forth above are not met, the analytical program will be terminated until the cause is determined and a solution is effected.
- 4.6.2 The analyst should demonstrate the absence of ambient air in the sample preparation system by filling a sample syringe with inert gas and injecting the inert gas onto the columns in the same manner as a sample. The results of this 'syringe blank' should show all analyte levels below the minimum detection limits.
- 4.6.3 Before and during sample analysis, instrument blanks (sample loop filled with flush inert gas) should be analyzed to assure the absence of interference as described in Section 3.0 above.
- 4.6.4 An experienced analyst should examine all chromatograms.
- 4.6.5 Calibration records are generated in electronic and hard copy formats and stored. All such records will be maintained in the laboratory during the course of the project and thereafter as determined by the client.

4.7 Sample Analysis and Holding Times

Each soil gas sample will be analyzed for C1-C4 and C5 plus compounds within ten working days of collection. Unless otherwise specified, all samples will be held for 30 days after the report has been submitted. Samples are then segregated, when appropriate, by type and disposed of in the proper manner. If the laboratory cannot dispose of sample remnants in an environmentally safe manner, they may be returned to the client for disposal.

Turnaround times are dependent on the tests that are required and the holding times for the various tests. In the case of holding times, they will always dictate the turnaround time of the sample. When rapid turnaround is required, it should be specified by the client and arranged in advance.

