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## Environment Programs Directorate Engineering & Technology

### Standard Operating Procedure

for **SAMPLING SUBSURFACE VAPOR**

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## 1.0 PURPOSE AND SCOPE

The purpose of this procedure is to describe the process of sampling sub-surface air for vapor phase contaminants at the Los Alamos National Laboratory (Laboratory).

## 2.0 BACKGROUND AND PRECAUTIONS

### 2.1 Background

Sampling systems and sampling equipment vary between sampling projects; therefore, all portions of this procedure are not required for every port that is sampled. All sections of the procedure applicable to a single type of sampling system or equipment are required and are followed in the order presented. The individual work plan will specify the sampling systems and the samples to be collected using this procedure.

This procedure is to be used in conjunction with an approved Environmental, Safety, and Health (ES&H) Plan or an Integrated Work Document (IWD).

The sample train, sample containers, and sampling systems are the primary components of the sub-atmospheric air-sampling system. The Laboratory uses three types of sampling systems:

- Down-hole packer(s) – removable devices that isolate the sample depth interval within the borehole or casing.
- FLUTE system - a borehole-specific sampling device that is configured and left in place to sample specific intervals for pore gas. FLUTE installation may be performed by the FLUTE manufacturing subcontractor, using their own installation procedures. The FLUTE system uses nylon or PVDF tubing to transport soil gas from the sampling port to the surface.
- Stainless steel tubing system with sampling ports permanently installed at specific depths. Sampling ports are surrounded by a sand filter pack. Bentonite seals are placed above and below the sand pack to prevent soil air flow between ports.

The sample train may be fitted with or adapted to various field screening instruments such as an air-flow gauge, vacuum gage, a photo-ionization detector (PID), a Brüel and Kjaer (B&K) Multi-gas Analyzer, a LANDTEC gas extraction meter, MultiRAE IR Multigas Monitor, or gas chromatograph/mass spectrometer (GC/MS). Field screening of oxygen and carbon dioxide is required to assure representative samples are collected. Other field screening instruments may be used for other sampling objectives. The sample train will be fitted to a SUMMA canister for VOC sample collection and to a silica gel cartridge for tritium sample collection.

### 2.2 Precautions

- If both volatile organic compounds (VOCs) and tritium are sampled at a single sampling port, the order in which the tritium cartridge sample and the VOC SUMMA canister sample are collected must be consistent for each sampling period. See discussion in Attachment 1.
- Properly documented field procedures must be followed to ensure wells and boreholes do not become damaged or contaminated during sampling activities;
- Because of the harsh conditions in the field, the plastic tubing of the sampling lines and vapor ports may degrade over time, depth tags on the tubing or ports may become unreadable, or plugs may be lost. Any unusual conditions of the sampling lines (tubing) or vapor ports must be documented in the field logbook; Repairs to these components should be undertaken according to the manufacturer's specifications as soon as they are discovered.
- Field screening and monitoring equipment shall have factory calibration certification as prescribed in the equipment's manufacturer's manual. These certifications are required for Appendix B Field Methods of Consent Order reports.

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- Naturally occurring radon may be present on some of the sampling lines or vapor ports. Radon can collect on the sampling lines or vapor ports because the plastic has a slightly negative static charge. The human body has a slight positive charge, and the hands may attract minute amounts of radon when they come in contact with the vapor ports or plastic tubing. Clapping the hands to eliminate any built-up static charge may alleviate this problem. Nitrile gloves should be worn for protection, and they may also alleviate the problem of static charge.
- Preparation of field screening equipment, packer systems, silica gel, and silica gel cartridges require preparation and maintenance activities described in this procedure prior to sampling; be aware of these timelines prior to sampling.
- If a vehicle or generator is used to supply power to purge pumps, ensure that exhaust is directed away (downwind) from the borehole sampling operation to prevent contamination of samples.
- Special care should be taken during the installation of the silica gel cartridges into the sample train to minimize exposure to ambient air. When installed into the sampling train, the cartridge must be oriented so that air flow passes through the silica gel and then the filter, so that silica gel is not pulled out of the sample cartridge and into the pump.
- Silica gel water vapor collection efficiency is significantly reduced during hot weather. For sampling efficiencies during the warm spring/summer months, it is recommended that the silica gel cartridge connected to the sample train be placed in a cooler with ice during sampling to reduce the time required to collect 5 grams of water.
- Water vapor may condense and freeze in sampling lines during cold weather, restricting flow. Sample lines may be heated using a heat gun to re-vaporize condensed water and restore air flow.
- When collecting a SUMMA canister field blank from a gas cylinder of pure nitrogen, close the SUMMA canister valve and the nitrogen cylinder valve when the SUMMA pressure valve equals zero, to avoid over pressurizing the SUMMA canister.
- Packer systems can become contaminated when used in multiple boreholes. Precautions to prevent cross contamination should be identified prior to the sampling campaign. Different strategies can be taken: packers can be decontaminated between use, packers can be assigned for use at only one borehole, etc.
- Clear handwriting of data in field collection logs and logbooks is essential to assure data quality.
- Waste generated from sampling activities must be handled in accordance with SOP-5238, Characterization and Management of Environmental Program Wastes.

### **3.0 EQUIPMENT AND TOOLS**

#### **3.1 All Sampling Systems**

- Gas flow meter.
- Differential pressure gauge or manometer for measuring static subsurface pressure.
- Hand tools – adjustable wrench, screwdrivers, pliers, utility knife.
- Nitrile gloves for skin protection and minimizing contamination, in accordance with the approved ES&H plan or Integrated Work Document (IWD).
- Teflon tubing that delivers pore gas from the vapor sampling port to the sample or field screening instruments/containers
- Vacuum pump(s) capable of 300 ml/min with flow rate meters. Maximum flow rates are 2 – 15 liters/min.

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- Calibration gases for field screening instruments.
- Pure (99.99%) nitrogen gas for field blank samples
- SUMMA Canisters - The SUMMA canister captures and contains the air sample for transport to the analytical laboratory for analysis of VOCs.
- Field screening instruments such as a LANDTEC gas extraction meter or MultiRAE IR Multigas Monitor for monitoring oxygen and carbon dioxide.

### 3.2 Sampling tritium

- Sampling manifold if sampling from multiple ports simultaneously
- Silica gel cartridge for tritium analysis. Cartridges must hold 135 grams of silica gel. Cartridges must have end plugs, to isolate the silica gel before and after sampling.
- Balance capable of reading 1000 +/- 0.01 g
- 500 g and 1000 g weights, National Institute of Standards and Technology (NIST) traceable
- Cloth gloves for handling balance weights
- Silica gel, 6–12 mesh, 135 grams per sample
- Laboratory oven capable of maintaining 110 °C for 48 hours
- Coolers with ice during hot weather. The silica gel cartridge, connected to the sample train tubing, can be placed in the cooler during sampling
- 2 crucibles for each lot of silica gel to be tested for bound water percentage. Crucibles shall be able to hold 50 - 100 grams of silica gel.
- Laboratory grade oven capable of maintaining 1000 °C for 2 hours
- Tongs, gloves, eye protection for removing silica gel from oven
- High-temp marker or etched markings to mark crucibles prior to denaturing silica gel
- 5 grams distilled water for each field blank sample
- "T" connection for sampling train for collecting duplicate tritium sample
- Heat gun for thawing frozen sample lines during cold weather

### 3.3 Sampling VOCs with B&K

- CGA 590 Bolt Compressed Gas Regulator;
- two Tedlar™ bags (include extras in case a bag is torn);
- a large adjustable wrench;
- a length of ¼-inch Teflon™ tubing; and
- calibration gasses: TCA - 100 ppm, TCE – 60 ppm

### 3.4 Equipment and tools for the Packer sampling system

- Dedicated packers
- Pulling rig or sampling tripod for lowering and raising packer systems into the borehole.

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- Teflon tubing with an outer diameter of approximately one quarter inch. This tubing is attached at the surface to stainless steel tubing and fittings that must be air tight. Fittings along the stainless steel tubing that will accommodate attachments for the sample containers.
- Pure nitrogen gas (99.99%) for decontaminating re-usable sample tubing
- Calibrated depth measuring device to ensure that packer is deployed at the correct depth.
- Down-hole camera system, calipers, or equivalent, for inspecting borehole for irregularities. The borehole is inspected each time prior to the installation of the packer system.

## 4.0 STEP-BY-STEP PROCESS DESCRIPTION

### 4.1 Develop Field Data Collection Forms

- Field Team Leader
- Each sampling campaign includes different sampling equipment and different numbers of boreholes and ports to be sampled. Example forms for field data collection are provided in Attachments 2-5. Each sampling campaign should develop field data collection forms, containing the data specified in the Attachments, appropriate to the specific sampling campaign. Sufficient space should be allowed in each data collection form to allow for legible handwriting.
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- Record the time to purge the sample train on Attachment 4. It is calculated as follows:  
 For boreholes constructed using sand pack filters, the purge time prior to screening for CO<sub>2</sub> is 10 minutes.  
 For boreholes to be sampled through FLUTE systems or open boreholes, the sample train should be purged for either 10 minutes, or the time required to purge the entire volume of the sample lines/port volumes, whichever is greater.  
 For FLUTE systems or open boreholes, calculate the time required for purging 3 one volume of sample line/borehole volume for each port to be sampled. The time to purge-one volume is calculated as (the total volume of sample lines and port volumes)/pump flow rate.  
 The volume of sample line is calculated as  $L * \pi * D^2/4$ , where L is the sample line length and D is the inner diameter of the sample line.  
 If the borehole is open and sampling intervals are designated with inflatable packers, the volume of the borehole between the packers is added to the volume of the sample line, to obtain the entire volume that requires purging prior to screening. The volume of space between the packers is calculated as  $P * \pi * DB^2/4$ , where P is the distance between the packers and DB is the diameter of the borehole.  
 If the borehole has a FLUTE system, the volume of the cylinder surrounding the port is included to obtain the entire volume that requires purging prior to screening. This volume is calculated as  $L * [\pi * DB^2/4] - L * [\pi * (DB - t)^2/4]$ , where L is the interval length of the port, DB is the borehole diameter, and t is the thickness of the FLUTE sample port membrane.

### 4.2 Periodic Evaluation of Silica Gel Bound Water Percentage for Tritium Sampling

- Field Team Member
- NOTE:** This service may be supplied by an organization separate from the field sampling team.  
 Silica gel shall be tested periodically to measure bound water percentage, as this value can change over time or with different manufacturers. Silica gel shall be tested when procured from a new manufacturer. If silica gel is purchased more than once per year, silica gel shall be tested once every 6 months. If silica gel is purchased less than once per year, silica gel shall be tested when new silica gel is purchased.

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Field Team Member (cont.)	2.	Silica gel is purchased in containers of varying sizes. Newly procured silica gel containers are called "lots" in this procedure. Select a minimum of 2% of newly procured silica gel lots to be used in testing prior to its use in field sampling.
	3.	Dry the silica gel in a drying oven at 110°C for at least two days. Dry the silica gel in a tray or in the original metal can with the lid removed. Place the dried silica gel into a container that can be closed immediately after removal from the oven to minimize the exposure of silica gel to ambient air. Allow the silica gel to cool before proceeding to the next steps
	4.	Weigh each crucible with lid while empty (CE). Mark each crucible with a number using a high-temp marker or etched markings. Record the weight of each crucible with lid on a data collection form similar to Attachment 2.
	5.	From the dried silica gel selected for testing, partially fill 2 crucibles.
	6.	Weigh each setup of crucible, lid, and silica gel (CSGI) and record the data on a data collection form similar to Attachment 2.
	7.	Place the crucibles in furnace and heat at 1000 °C for two hours.
	8.	Remove crucibles after oven has cooled. Use tongs, wear gloves and eye protection and stand back when opening furnace. When crucibles are cool enough to handle, weigh and record final crucible + silica gel weight (CSGF) on a data collection form similar to Attachment 2.
	9.	Calculate the percent moisture of silica gel from each crucible: $\frac{(CSGI - CSGF)}{CSGI - CE} \times 100$ Record this value on a data collection form similar to Attachment 2 for each crucible of silica gel tested (e.g. 5.54%).
	10.	Calculate the percent moisture value of the silica gel lot as the average of the individual crucibles tested. Record this value on a data collection form similar to Attachment 2 (e.g. 6.35%). Record this value in sampling logbooks. This value must also be retained with the lot of silica gel.

### 4.3 Preparation of Tritium Cartridges, prior to Field Deployment

Field Team Member	1.	Ensure that all materials and equipment that will come into contact with the silica gel are clean and dry before proceeding.
	2.	Dry the silica gel to be used for field sampling in a drying oven at 110°C for at least two days. Dry the silica gel in a tray or in the original metal can with the lid removed. Place the dried silica gel in a container and close the container after removal from the oven to minimize the exposure of silica gel to ambient air. Allow the silica gel to cool before proceeding to the next steps.
	3.	Zero the balance. Put on a cloth glove to handle check weights. Use the 500 g and 1000 g weights to check the balance function. Ensure the balance reads zero with no weights but with any supports to be used. Record the results in the log book.

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| Field Team Member (cont.) | 4. Record the percent moisture value for the lot of silica gel in the logbook. (see Section 4.2)  |
|                           | 5. Check the cartridge to assure that a filter is in place in one of the end caps. Mark the direction of sample air flow on the cartridge. Sample air moves towards the filter.   |
|                           | 6. Weigh each empty cartridge with end plugs to the nearest 0.01 g. Record the weight of each cartridge in the log book.  |
|                           | 7. Add approximately 135 grams of silica gel to each canister and close both openings. Weigh each cartridge containing silica gel to the nearest 0.01 g. Record the weight of the cartridge with silica gel in the log book.  |
|                           | 8. Calculate the amount of dry silica gel added to the cartridge. This value is the weight of the cartridge containing silica gel minus the weight of the empty cartridge. Record the weight of the dry silica gel (which will be approximately 135 grams) in the log book. |

#### 4.4 Preparation of Packer System prior to Field Deployment

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| Field Team Member | 1. If re-usable sample lines are used with the packer systems, decontaminate sample line prior to use. Purge line with nitrogen for a minimum of three sample line volumes. |
|                   | 2. If re-usable packers are used, decontaminate packers prior to use.   |
|                   | 3. Check the inflatable packers and air line fittings for leaks before the packer system is sent down-hole.   |

#### 4.5 Planning for QA/QC Samples

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| Field Team Members | 1. QA/QC samples are collected at a frequency greater than or equal to 10% per sampling event. Two QA/QC samples are collected for VOCs using SUMMA canisters: a field duplicate (FD) sample, and a field blank of pure nitrogen (99.99%). Two QA/QC samples are collected for tritium: a field duplicate (FD) sample and a field blank sample of distilled water. |
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#### 4.6 Pre-Sampling Activities

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| Field Team Members | 1. Ensure the documentation of all field logbook entries is in accordance with SOP-5181, Notebook and Logbook Documentation for Environmental Directorate Technical and Field Activities.   |
|                    | 2. Document at a minimum the following pre-sampling activities into the field logbook: <ul style="list-style-type: none"> <li>• Date, time, location</li> <li>• sample train inspection;</li> <li>• current calibration of all instruments;</li> <li>• port conditions;</li> <li>• tubing problems/solutions;</li> <li>• field screening instrumentation operational check</li> </ul> |

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Field Team Members (cont.)

3. Inspect all tubing, fittings, and valves on the sample train for degradation.
4. Inspect Swagelok fittings for degradation.
5. Tighten, as necessary, all fittings and valves that make up the assembly.
6. Ensure the power supply is functional
7. Determine the time required to purge 3 volumes for each port to be sampled, if not previously calculated in section 4.1 Step 2. The time required to purge three volumes from each port is calculated as the  $3 * (\text{volume of sample tubing} + \text{volume of sample interval}) / (\text{pump flow rate})$ . This value is recorded for each port on Attachment 2.

#### **4.7 Daily Field Operational Check of Screening Instruments (Landtec Gas Extraction Meter or MultiRAE IR Multigas Monitor, etc)**

Field Team Member

1. Ensure that the monitor is charged before taken out in the field. Ensure that the external power supply is functional.
2. Follow manufacturer's procedure for performing daily operational checks of field screening instrumentation. Field screening is required for oxygen and carbon dioxide. Field screening may be required for other VOCs as specified in the site specific work plan. For Landtec or MultiRAE IR Multigas Monitor meter, check O<sub>2</sub> and CO<sub>2</sub>. Oxygen values should be +/- 1% of 20.9%. Carbon dioxide should read 0%. Record results on a form similar to Attachment 3. If readings are not within manufacturer's specifications, follow repair instructions or contact the manufacturer.

#### **4.8 Daily Field Operational Check of B&K Multi-gas Analyzer**

Field Team Member

1. Before operating the B&K unit, read the Operational Manual.
2. Follow manufacturer's procedure for performing daily operational checks of field screening instrumentation. For B&K analysis, collect VOCs as required by the site-specific work plan (typically TCA and TCE, possibly others). Record results on a data collection form similar to Attachment 3. If readings are not within manufacturer's specifications, follow repair instructions or contact the manufacturer.
3. Adjust setup conditions of the B&K unit to ambient pressure and temperature.
4. Select first calibration gas. Write gas name and concentration on Tedlar™ bag.
5. Ensure the regulator valve is closed.
6. Connect the regulator to the calibration gas bottle, and connect the Tedlar™ bag to the regulator.
7. Open the valve on the Tedlar™ bag and the valve on the bottle.



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- Field Team Member (cont.)
8. Slowly open the regulatory valve and fill the Tedlar™ bag. Do not over-inflate.
  9. Close the regulator valve, the Tedlar™ bag valve, and the calibration gas bottle valve.
  10. Remove the Tedlar™ bag from the regulator.
  11. Open then close the regulator valve to release any gas in the regulator.
  12. Remove the regulator from the gas bottle.
  13. Turn on the B&K unit.
  14. Connect the Tedlar™ bag to the B&K inlet line.
  15. Open the Tedlar™ bag valve.
  16. Allow the B&K unit to take several samples of gas from the Tedlar™ bag. Document B&K readings on a data collection form similar to Attachment 3.
  17. Close the Tedlar™ bag valve and remove it from the inlet line. Dispose of the Tedlar™ bag.
  18. Turn off the B&K unit.
  19. If the B&K operational check does not quantify the results of the analysis within 20% of the known concentration of the laboratory-certified gas, perform one or more of the following actions:
    - change the setup parameter (refer to B&K Operational Manual for instructions);
    - inspect the Tedlar™ bags [NOTE: The bags may degrade and fail (leak) over time]; or
    - call the manufacturer
- Repeat Steps 4-19 for other calibration gases

#### **4.9 Preparation and Installation of Packer Systems**

- Field Team Members
1. If re-usable sample lines are used with the packer systems, decontaminate sample line prior to each use. Purge line with nitrogen for a minimum of 3 sample line volumes.
  2. Ensure that the equipment to be used is contaminant-free by scanning it with field screening instruments before lowering it into the borehole to prevent cross-contamination from other boreholes.
  3. Check the inflatable packers and air line fittings for leaks before they are sent down-hole.
  4. Ensure packer(s) are the appropriate diameter for the borehole.

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- Field Team Members (cont.)
5. Before the packer system is lowered into an open borehole, ensure the borehole is free of any irregularities such as sloughing or the state wherein the borehole is curved due to incorrect auger tracking. A down hole camera system may be used for this inspection.
  6. Using the appropriate equipment (pull rig or sampling tripod), deploy the packer(s) to the desired depth. Use a calibrated depth measurement device to position the packer at the correct depth.
  7. Inflate the packer to the desired inflation pressure according to the manufacturer's specification.
  8. If the pressure drops following inflation, it may indicate a packer-inflation leak. Remove the packer from the borehole and either fix or replace the leaking component prior to sampling.

#### **4.10 Sampling Activities at a borehole: Purging and screening (Landtec Gas Extraction Meter or MultiRAE IR Multigas Monitor, etc)**

- Field Team Members
1. Confirm the borehole number and location. Document on sample collection log and field logbook.
  2. Open borehole cover. Document any finding that may impede sampling of the borehole.
  3. Using the differential pressure gauge, measure and document the static pressure of the formation at the sample interval on a form similar to Attachment 4.
  4. Connect the purge pump to the top of the vapor port system using a length of Teflon™ tubing. Purge the sample train for the time calculated in section 4.1 with the purge pump to remove all stagnant air within the tubing and valves. Document purge flow rate and start/stop times on a data collection form similar to Attachment 4.
  5. If purge rate is less than 1/3 of the nominal purge pump flow rate, the port may be blocked. If you suspect that the port is blocked, do not continue sampling at this port. Document in log book and sample collection log. Contact Project Leader for further instructions at the time of this discovery.
  6. Following purging (step 4), connect the screening instrument to the vapor port and monitor CO2/O2 values. Record screening values of methane, CO2, and O2 readings in 1 minute intervals on a data collection form similar to Attachment 4. When 2 consecutive readings of CO2 have changed by less than 10%, the values have stabilized and a representative sample can be collected.

#### **4.11 Sampling Activities at a Borehole: Screening with B&K Multi-Gas Analyzer**

- Field Team Members
1. B&K unit screening is only performed following Landtec or MultiRAE IR Multigas Monitor screening.
  2. Ensure that the operational check for the B&K unit has been performed before use (see section 4.9).

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| Field Team Members (cont.) | 3. Turn the B&K unit ON and ensure that it is in "Continuous Monitoring" mode. Steps 4-7 are required to clear out any sample air remaining in the B&K unit, prior to sampling.  |
|                            | 4. Program the sample tubing length to 300 feet.   |
|                            | 5. Press the "Standby" button on the B&K unit control display to start the sampling cycle.   |
|                            | 6. Allow the B&K unit to take three ambient air measurements, and record the last set of values on a data collection form similar to Attachment 5, on the line marked "ambient". If values of VOCs are non-zero, repeat this step. |
|                            | 7. Press the "Standby" button on the B&K unit control display to stop the sampling cycle.  |
|                            | 8. Connect the sample tubing to the vapor port and to the B&K unit to begin sampling.  |
|                            | 9. Press the "Standby" button on the B&K control panel to start sampling.  |
|                            | 10. Take readings at one minute intervals until the CO <sub>2</sub> reading varies by less than 10%. Record the CO <sub>2</sub> and TCA readings on a data collection form similar to Attachment 5.                                |
|                            | 11. When the CO <sub>2</sub> reading has stabilized, record the full measurement of the B&K analysis (TCA, TCE, H <sub>2</sub> O, pressure differential), the date, and time on a data collection form similar to Attachment 5.    |
|                            | 12. Press the "Standby" button to stop the sampling.   |
|                            | 13. Disconnect the sample train inlet line from vapor port.  |

#### 4.12 SUMMA Canister Sampling

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| Field Team Members | 1. SUMMA Canister sampling is only performed following purging and screening with the field screening instrument and, if applicable, screening with the B&K Multi-gas Analyzer.            |
|                    | 2. Connect pressure valve with the vacuum gauge to the vapor port.   |
|                    | 3. Ensure all the valves are closed.   |
|                    | 4. Attach the SUMMA canister to the pressure valve.  |
|                    | 5. Open the valve on the SUMMA canister and check the vacuum gauge for proper vacuum. Document the starting pressure on the sample collection log, and the date/time of sample collection. |
|                    | 6. Open the pressure valve.<br><b>NOTE:</b> The SUMMA canister will draw an air sample because of the vacuum in the canister   |

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| Field Team Members (cont.) | 7. Close the valve on the canister when the gauge indicates the pressures in the canister and atmospheric pressure have equilibrated (i.e. reading on pressure gage = 0 psig). Document the final pressure on the sample collection log. |
|                            | 8. Complete the identification tag of the canister.  |
|                            | 9. Document SUMMA canister sampling in the field logbook, in the sample collection log, and on the chain-of-custody forms.   |
|                            | 10. Disconnect pressure valve from vapor port.   |
|                            | 11. Store the SUMMA canister in the shipping container, and ship to the Laboratory SMO in accordance with EP-ERSS-SOP-5057, Handling, Packaging, and Transporting Field Samples.   |

#### **4.13 Collect SUMMA Canister QA/QC Samples**

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| Field Team Members | 1. To collect the field duplicate sample, connect a second SUMMA canister to the sample train immediately following the collection of the original sample. Complete steps in 4.12                                      |
|                    | 2. To collect the field blank sample, connect the regulator to the nitrogen tank, with the tank and regulator valves closed. Connect the sample train to the nitrogen tank regulator.                                  |
|                    | 3. Open the nitrogen tank valve. Then purge the sample train with nitrogen by slowly opening the regulator valve for a few seconds. Close the regulator valve.   |
|                    | 4. Attach SUMMA canister to the pressure valve and to the sample train. Close all valves on sample train.  |
|                    | 5. Open valve on SUMMA canister. Document the starting pressure on the sample collection log, and the date/time of sample collection.  |
|                    | 6. Open the valve on the sample train leading to the nitrogen tank.  |
|                    | 7. Open the regulator valve on the nitrogen tank slightly allowing nitrogen to flow through sample train into the SUMMA canister until SUMMA pressure is zero. Record the final pressure on the sample collection log. |
|                    | 8. When SUMMA canister pressure valve equals zero, close the SUMMA valve and the regulator valve. Do not over pressurize the SUMMA canister.   |
|                    | 9. Slowly open the regulator valve, to release any gas remaining in the regulator  |
|                    | 10. Store the SUMMA canister in the shipping container, and ship to the Laboratory SMO in accordance with EP-ERSS-SOP-5057, Handling, Packaging, and Transporting Field Samples.                                       |

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#### 4.14 Tritium Sampling

- Field Team Members
1. Tritium samples are only collected after the system has been purged with a field screening instrument and, if applicable, the B&K unit.
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2. Connect the tritium cartridge to the purge fittings of the sample manifold. Orient the cartridge so that the filtered end cap is on the pump side of the sample train.
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3. Activate pump(s) to pull subsurface pore gas through the tritium cartridge. Periodically disconnect the tritium cartridge from the sample train, install both end plugs, and weigh it. The sample is complete when the weight of the cartridge has increased by at least 5 g above the original cartridge weight determined in Section 4.3. If the weight has not increased by at least 5 g, re-install the cartridge in the sample train and continue pulling pore gas through the sample cartridge until the weight of the cartridge has increased by at least 5 g.  
  
Record the final weight of the cartridge and sample to the nearest 0.01 g.  
**NOTE** – Tritium sampling may take several hours.
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4. Quickly remove cartridge and seal the ends.
- 
5. Document the tritium cartridge sample in the field logbook and in the sample collection log or the chain-of-custody forms. Record the (a) initial weight of dry silica gel used in the sample, (b) the weight of the cartridge plus dry silica gel before sampling, and (c) the weight of the silica gel cartridge and silica gel after sampling on the sample collection log. Record the silica gel bound water percentage for this lot of silica gel (from the field log book or Attachment 2) on the sample collection log or chain-of custody form.
- 
6. Submit samples to the Laboratory’s Sample Management Office (SMO) in accordance with EP-ERSS-SOP-5057, Handling, Packaging, and Transporting Field Samples.

#### 4.15 Collect Tritium QA/QC Samples

- Field Team Members
1. To collect field duplicate samples, connect a second tritium cartridge to the sample train using a “T” connection into the two columns, so that air is pulled through both samples simultaneously. Follow steps 2-6 in 4.14 for sample collection, data recording, and submission.
- 
2. To collect a field blank sample, add 5 grams of distilled water to a tritium cartridge. Add end plugs to the cartridge. Weigh the cartridge with silica gel and distilled water. Follow steps 5-6 in 4.14 for sample documentation and submission.

#### 4.16 Complete Sampling through the Packer System

- Field Team Members
1. Deflate the packer(s) before pulling them out of the borehole. Pull the packers.
- 
2. Close borehole.

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- 
3. Dispose of any tubing that is visibly damaged or contaminated.
- 
4. Document date/time of completion of packer system removal in field log book and note any abnormal condition of the packer system.

#### **4.17 Post-sampling Activities**

- Field Team Members
1. Ensure that the packer system is contaminant-free by scanning it with field screening instruments following sampling to prevent cross-contamination from other boreholes. Alternately, decontaminate sampling equipment following EP-ERSS-SOP 5061 Field Decontamination of Equipment. Document results in sample logbook.

#### **4.18 Records**

- Field Team Members
1. Submit the following records generated by this procedure to the Records Processing Facility:
    - Completed chain-of-custody/request for analysis forms;
    - Closed out field logbooks;
    - Attachments from this procedure; and
    - Completed sample collection logs.

## **5.0 PROCESS FLOW CHART**

None

## **6.0 ATTACHMENTS**

Attachment 1: Sampling Considerations

Attachment 2: Silica Gel Bound Water Percentage Worksheet

Attachment 3: Daily QA/QC Operational Checks for Landtec or MultiRAE IR Multigas Monitor and B&K Instruments

Attachment 4: Purge/Screening Using Landtec or MultiRAE IR Multigas Monitor

Attachment 5: Purge/Screening Using the B&K

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## 7.0 REVISION HISTORY

Revision No. <i>[Enter current revision number, beginning with Rev.0]</i>	Effective Date <i>[DCC inserts effective date for revision]</i>	Description of Changes <i>[List specific changes made since the previous revision]</i>	Type of Change <i>[Technical (T) or Editorial (E)]</i>
0.0	02/09/07	New document number, reformatted and renumbered. Supersedes SOP-06.31	E
IPC 1	TBD	Adds one item to the equipment and tools list. Adds more detail to the weight measurement steps in Section 4.7.	T
IPC-2	July 2009	Standardized drying silica gel prior to use. Recording initial mass of silica gel, prior to adding to sampling cartridge. Calculation of corrected tritium pore gas concentrations. Superseding EP-ERSS-SOP-5074, R 0 IPC-1.	
1	6/22/2010	Add routine testing of silica gel bound water. Standardize purging requirements for VOC sampling. Standardize use of CO <sub>2</sub> and O <sub>2</sub> screening steps for VOC sampling. Superseding EP-ERSS-SOP-5074, R 0 IPC-2	T
IPC-1	7/23/2010	Change sample line purge to 10 minutes prior to CO <sub>2</sub> screening in Section 4.10, Step 4.	T
2	9/17/2010	Formalized changes documented in IPC-1 into the procedure as Revision 2.	T/E

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## Attachment 1: Sampling Considerations

### General

There is not one standard method for collecting subsurface vapor contaminants, due to the lack of equilibrium in subsurface contaminant vapor plumes and due to the variability of vadose zone geology from site to site. The lack of equilibrium in subsurface contaminant vapor plumes is a function of the source of vapor phase contaminants, methods of disposal, rate of degradation in the soil, and the time since disposal.

The ASTM standard recommends that sampling in a consistent manner, from time period to time period, is of greatest importance to obtain a credible data set over time. This procedure calls for the consistent use of sampling equipment and methods from time period to time period, but does not require that each site use the exact same methods due to the variability in site geology and the variability in contaminant vapor plumes from site to site.

Each site (primarily Material Disposal Areas at LANL) is governed by an individual sampling plan approved by the NMED. And so, there is variability from site-to-site in sampling requirements (frequencies, depths, VOCs, tritium, etc). The NMED approved sampling plan will specify the locations, port depths, and contaminants to be monitored. This procedure covers all aspects of subsurface vapor monitoring, to implement the NMED approved sampling plan.

### Sample Pump Flow Rates

Consistent use of pumps for sample line/borehole purging and sampling tritium is required. A minimum sample pump flow rate is 300 ml/min. Typical sample pump flow rates are 3-15 liters/minute due to the variability of port depths to be sampled and the porosity of the vadose zone soils at Los Alamos.

### Sampling VOCs and Tritium

The order of sample collection for VOC SUMMA canisters and tritium cartridges must be the same at each site, from one sampling period to the next. In all cases, the sample lines/borehole volumes are purged and then screening sampling is done prior to the collection of a VOC SUMMA canister sample or a tritium sample.

At locations where there are higher concentrations of VOCs in subsurface vapor plumes (e.g. MDA L), it is recommended that VOC SUMMA canisters samples are collected before tritium samples, following the purge of the sample line/borehole volumes and documentation/verification of stabilized subsurface air measurements. This order of sampling (VOCs first then tritium) reduces the chance of impacting the equilibrium concentration of VOCs collected by the SUMMA canister that might happen following a long sampling period required (12+ hours) for collecting the tritium sample.

At locations with lower concentrations of VOCs in subsurface vapor plumes (e.g. MDA T), the tritium sample can be collected before or after the VOC SUMMA canister sample. Whichever sample is collected first (VOC or tritium), this same order of sampling must be followed each time.

Staff in ADEP-Environmental Technology can provide guidance on selecting the order of sampling VOCs and tritium for a new site, not previously sampled.



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### Screening for Identifying Sample Representativeness

Following purging of the sample lines/borehole, screening is required to demonstrate a representative sample of the vapor contaminant plume is being collected, as opposed to atmospheric air. Typically, solvent contaminant plumes include carbon dioxide produced as a byproduct of bacteria breaking down solvents. The goal of screening is to identify when carbon dioxide concentrations representative of the contaminant plume are identified rather than carbon dioxide naturally occurring in atmospheric air.

The carbon dioxide screening measurement becomes more complicated at sites where boreholes are in direct connection with the atmosphere (basalt, near the edge of a mesa, etc.) or where there is a low level of organic material. In these cases, the subsurface vapor sampling may pull in atmospheric air from adjacent canyons, or there may be very little CO<sub>2</sub> produced by degradation of organics. Screening sampling instruments must be able to differentiate between subsurface air and ambient air. For sampling at these more challenging sites, a more CO<sub>2</sub> sensitive screening instrument is required, such as a MultiRAE IR Multigas Monitor, rather than a LANDTEC gas extraction meter, for example.

### Reference Procedures for Sampling Subsurface Air

ASTM, March 2009. "Standard Guide for Soil Gas Monitoring in the Vadose Zone," ASTM D 5314-92.

US EPA, 1996. "Soil Gas Sampling," SOP 2042, <http://www.epa.gov/region5/sites/multisite-mgp/multi-site-qapp-appendices-opt.pdf>.

California Department of Toxic Substances Control, 2003. Advisory – Active Soil Gas Investigations.

US EPA Region 4, 2007. "Soil Gas Sampling Operating Procedure," SESDPROC-307-R1.

Sandia National Laboratory, 2007. ASSOP 07-02, "Soil-Vapor Sampling at the Mixed Waste Landfill."

Sandia National Laboratory, 2007. ASSOP 01-04, "Active Soil-Gas Sampling Using Method TO-14 at the Corrective Action Management Unit (CAMU)".

Missouri Department of Natural Resources, 2005. "Soil Gas Sampling Protocol," Missouri risk-based corrective action for Petroleum Storage Tanks.

**Attachment 2: Silica Gel Bound Water Percentage Worksheet**

Date of Silica Gel Bound Water Percentage Tests: \_\_\_\_\_ Sampler \_\_\_\_\_

Dates of use of average silica gel bound water percentage (typically 6 months): \_\_\_\_\_ Reviewed by: \_\_\_\_\_

Sampling locations of use of average silica gel bound water percentage: \_\_\_\_\_ Review date: \_\_\_\_\_

Crucible number	Silica gel can number	Position of gel in can (relative to top, e.g. 1/3 down)	Weight of empty crucible plus lid, grams (CE)	Initial weight of silica gel plus crucible plus lid, grams (CSGI)	Final weight of silica gel plus crucible plus lid (after denaturing), grams (CSGF)	Calculated silica gel bound water percentage: $\frac{(CSGI - CSGF)}{CSGI - CE} \times 100$	Comments
Average of all crucible silica gel bound water percentages for the batch of silica gel cans							

**Attachment 3: Daily QA/QC Operational Checks for Landtec or MultiRAE IR Multigas Monitor and B&K Instruments**

Sampling Date \_\_\_\_\_ B&K Instrument Name/Number \_\_\_\_\_ Sampler \_\_\_\_\_

Cal Gas Lot # \_\_\_\_\_ Reviewed by: \_\_\_\_\_ Date \_\_\_\_\_

Standard	Time	TCA (ppm)	TCE (ppm)	Calibration Verified? (>80%)
Standard 1				
Standard 2				
Standard 3				

**Example**

Sampling Date \_\_\_\_\_ Landtec Instrument Name/Number \_\_\_\_\_ Sampler \_\_\_\_\_

Instrument is checked against ambient air \_\_\_\_\_ Reviewed by: \_\_\_\_\_ Date \_\_\_\_\_

Standard	Time	Reading 1	Reading 2	Reading 3	Expected Value	Calibration Verified?
Methane					0%	
CO2					0%	
Oxygen					20.9% +/- 1%	

**Attachment 4: Purge/Screening Using Landtec or MultiRAE IR Multigas Monitor**

Borehole number \_\_\_\_\_ Landtec No \_\_\_\_\_ Sampler \_\_\_\_\_

Sampling Date \_\_\_\_\_ Static Pressure of Port \_\_\_\_\_ Reviewed by: \_\_\_\_\_ Date \_\_\_\_\_

Purge Flow Rate: \_\_\_\_\_ Time Required to purge 3 sample line volumes at nominal flow rate \_\_\_\_\_

Purge Start time: \_\_\_\_\_ Purge Complete Time: \_\_\_\_\_

Depth (ft)	Date	Screening Time	CH4 %	CO2 %	O2 %	

*Example*

