

Collection of Bedrock Vapor Samples Using a Packer

1.0 Scope and Application

This standard operating procedure (SOP) offers an approach for the collection of field readings of subsurface vapor from unlined bedrock core holes. The objective of this sampling approach is to collect a vapor sample from the unsaturated zone that is representative of concentrations in equilibrium with subsurface bedrock, pore water, and groundwater. This procedure outlines the collection of bedrock vapor parameters and an analytical laboratory sample from a bedrock corehole above the water table.

This SOP should be used when its application is consistent with the project's data quality objectives (DQOs) and in conjunction with the SOP for Packer Testing. Only persons trained in the collection of vapor samples should attempt this procedure.

Site-specific Considerations

Prior to attempting bedrock vapor sampling, there should be an understanding of subsurface conditions at the site:

- **Depth to Groundwater**—Bedrock vapor samples are to be collected in the vadose zone (and above the capillary fringe). Generally, bedrock vapor samples should not be collected less than 5 feet below ground surface without guidance from vapor intrusion (VI) senior technical staff. At least 2 feet of open corehole must be available for sampling, to use this procedure.
- **Bedrock Permeability**—It might not be feasible to collect vapor from intervals with low permeability such as unfractured bedrock or clays, and these intervals should be avoided. Care should be taken during purging and sampling so that the vacuum in the sampling system never exceeds 7 inches of mercury ("Hg) (100 inches of water) in order to obtain representative concentrations of subsurface conditions. If the vacuum does exceed 7"Hg, then this interval has very low permeability and is likely to have a significantly reduced role in subsurface VOC migration and accumulation; therefore, another interval should be selected.

Other Considerations

- Vapor sampling should not be performed until 5 days after a significant rain event (defined as greater than 0.5 inch of rainfall). This timeframe is to allow re-equilibration of volatile organic compounds (VOCs) following the infiltration of rainwater and the passage of dramatic swings in barometric pressure common to storm events.
- Methane (CH₄) and carbon dioxide (CO₂) can cause positive bias with a helium leak detector, as detailed below in the helium leak-check procedure of this SOP. If methane or CO₂ are expected or encountered at the site, then it may be necessary to explore different strategies to establish borehole/packer seal integrity. CH₄ and CO₂ can be expected near landfill or petroleum hydrocarbon spill sites.
- Equipment used for the collection of bedrock vapor field parameters, typically VOCs, oxygen (O₂), CO₂, and CH₄, should be selected based on the expected range of concentrations as well as the monitoring goals. Equipment may be either too sensitive or not sensitive enough for concentrations encountered in the field. Discuss expected contaminants of concern (COCs) and expected range of concentrations with the equipment provider prior to field mobilization.

2.0 Equipment and Materials

- Appropriate diameter packer(s) (typically a dual packer assembly, for collecting depth-discrete vapor samples in the vadose zone) outfitted with Teflon tubing, ¼-inch outside diameter (OD) sample tubing and Swagelok ¼-inch nut and ferrule sets (part #SS-400-NFSET) for connecting the packer tubing to the sampling manifold; foot pump to inflate packer and a gauge to monitor packer inflation pressure
- GEM-2000 Landfill Gas Meter (Optional if field measurements of CO₂, O₂, or CH₄ are necessary [aerobic biodegradation parameters typically measured for petroleum hydrocarbon sites])
- MultiRae Five gas meter (Optional if onsite atmospheric gas analysis is required)
- The helium leak-check equipment (or equivalent), including the enclosure, helium cylinder (high purity helium), and helium detector (Dielectric MGD is preferred); the helium detector can be rented from an equipment rental company; the enclosure must encompass the borehole, sampling system, and SUMMA Canister
- Appropriate monitors for expected gases and concentrations—photoionization detector (PID) or flame ionization detector (FID) for health and safety to verify that breathing-zone VOC concentrations remain below levels specified by the Health and Safety Plan (HASP)
- Gas sampling bag (for example, a Tedlar bag) to collect the purged vapor so the volume of purged vapor can be measured and field screening with a PID and/or GEM-2000 meter can be performed on the purged gas
- Vacuum pump for purging with rotometer to control flow to 1 liter per minute (L/min) (should be a Cole Parmer # R-79200-00 grey diaphragm pump, or equivalent); appropriate electric supply for the pump must be provided (either generator or power inverter with adapter for car battery)
- Sampling manifold consisting of Swagelok gas-tight fittings with three valves and one pressure gauge to attach the probe to the air pump and the sample canister as shown on Figure 1 (provided in Section 3.2.1); this manifold must be clean, free of oils, and flushed free of VOCs prior to use
- Flow controller or critical orifice, certified-clean, and set at desired sampling rate; these are typically cleaned, set, and provided by the laboratory
- Wrenches and screwdriver (clean and free of contaminants), various sizes as needed for connecting fittings and making adjustment to the flow controller; a 9/16-inch wrench fits the ¼-inch Swagelok fittings, which most canisters and flow controllers have
- Canister, stainless-steel, SUMMA polished, certified-clean, and evacuated (Canisters are typically provided by the laboratory.)
- Negative-pressure (vacuum) gauge, oil-free and clean, to check canister vacuum. The vacuum gauges are typically provided by the laboratory. The laboratory may either provide one vacuum gauge to be used with all of the canisters or a vacuum gauge for each canister to be left on during sample collection. Sometimes the canisters are fitted with built-in vacuum gauges that are not removable.
- A CH2M HILL-owned and -maintained PG5 Digital Pressure Gauge from Automation Products Group (APG) with National Institute of Standards and Technology (NIST)-traceable calibration certificate, or equivalent; the digital vacuum gauge will be used to check the initial and final negative pressures of the canisters
- Shipping container, suitable for protection of canister during shipping; typically, strong cardboard boxes are used for canister shipment; the canisters should be shipped back to the laboratory in the same shipping container in which they were received
- Personal protective equipment (PPE) per the HASP

3.0 Procedures and Guidelines

3.1 Packer Installation and System Set-up

A packer is used to isolate the desired sample interval from ambient air for purging and sampling. Packers will be inflated to the specified pressure based on depth and the manufacturer specifications for that depth with the specific model or serial number associated with the packer assembly;

1. Obtain the target sample intervals from work plan and/or the VI or Subsurface Remediation senior technical consultant.
2. Acquire all the necessary hardware and sampling equipment shown in Figure 1 (included in Section 3.2.1). Be sure to use ¼-inch diameter Teflon sample tubing. **Do not connect the SUMMA canister at this time.**
3. Refer to Figures 2, 3, and 4 (provided at the end of this SOP) for packer system installation diagrams. Verify that the Teflon tubing sampling inlet will be located at least 6 inches below the packer (or top packer if a dual packer assembly is to be used); and that the downhole leak test injection inlet is no more than 5 feet above the packer. If collecting depth-discrete vapor samples, initial packer placement should be at the shallowest desired interval, sequentially moving down to deeper intervals for subsequent samples. Measure the depth of the packer placement with a cloth tape measure. Do not allow the sample tubing to become submerged below the static water level. Inflate the packer using the foot pump. Do not allow the packer to become deflated; pull gently on the packer tubing to check the seal. The pressure inside the packer must remain between 5 and 10 pounds per square inch (psi) to provide a proper seal; this could vary depending on the packer used.
4. Adjust the purge system evacuation pump sampling rate to achieve the desired flow rate of 1 L/min. Flow rate measurements should be performed at the outlet of the vacuum pump prior to purging, either by using a suitable flow meter or by determining the amount of time required to fill a 1-L gas-sampling bag.
5. Sampling canisters are evacuated prior to shipment by the laboratory. The vacuum will need to be verified in the field with the APG Digital Pressure Gauge **and** the laboratory-supplied vacuum gauge and properly recorded prior to use.
6. Attach the air pump to the sampling manifold and the Tedlar bag to the air pump exhaust. See Figure 2. **Do not attach the SUMMA canister at this time.**

3.2 System Leak Checking and Purging

Vapor sample integrity is verified by using a real-time leak checking procedure before taking each sample. This must be done after packer installation and prior to sampling, as well as before each subsequent vapor sample from each packer interval, VMP, or monitoring well.

3.2.1 Physical Leak Check

The physical leak check documents that the sampling apparatus is pneumatically tight, and that the sampling will not introduce dilution air. Perform a leak check of the sample manifold system as follows:

1. Make sure the gas probe valve (Valve 1) is closed and the sample valve (Valve 2) is open.
2. Open the purge valve (Valve 3) and start the purge pump. Verify that the flow is set to 1 L/min.
3. Close the sample valve (Valve 2) and achieve a vacuum gauge reading of 7"Hg (approximately 100 inches of water column) on the gauge located on the sampling manifold or to a vacuum that will be encountered during sampling, whichever is greater.
4. A leak-free system will be evident by closing off the purge valve (Valve 3), turning off the vacuum pump, and observing no loss of vacuum within the sampling manifold system for a period of 30 seconds (that is, the needle on the pressure gauge within the sampling manifold remains at 7"Hg for 30 seconds). Repair

any leaks prior to sample collection by tightening the fittings on the manifold. Re-test to make the sure the manifold passes the physical leak check before proceeding.

5. Record the leak check date and time on the field sampling log.



FIGURE 1
Sampling Manifold

3.2.2 System Purge and Helium Leak Check

A purge of the packer interval and sampling manifold system is required before taking each sample. The helium leak-check procedure is also performed during this step. This helium leak check will verify the integrity of the seal of the packer and bedrock interface. This is accomplished through the following steps:

1. Affix Teflon tubing to the regulator attached to the helium cylinder. Make sure the tubing is long enough to connect to the downhole leak injection tubing.
2. The downhole leak injection tubing is to be installed as the packer assembly is placed. The opening of the tubing should be within the first 5 feet above the top packer.
3. Place the helium enclosure over the bedrock corehole to achieve a buildup of helium in the corehole and the enclosure. Make sure the enclosure is large enough to accommodate this. The enclosure should not be tightly sealed and there should be an exhaust for the helium so that pressure does not build up in the enclosure.
4. Start the flow of helium and let the helium fill the injection tubing, and from there allow it to fill the corehole and enclosure for a couple of minutes.
5. Turn the helium leak detector on while in outdoor air and check that the detector is not reading any helium before proceeding. Verify that the helium concentration inside the leak-check enclosure is greater than 5 percent by placing the probe of the helium detector into the hole where the sample tubing comes out or under the enclosure wall. It is not necessary to verify that the helium concentration is 100 percent, as this may damage the detector. When the helium leak detector is purged and reading zero, connect to the sample tubing (the tubing extending into the packed off interval).
6. Calculate one purge volume. Be sure to take into account the packered interval borehole volume and the inside diameter and length of the Teflon sample tubing. Purging is carried out by pulling in situ gases

through the system at a rate of 1 L/min for a time period sufficient to achieve a purge volume that equals at least one dead volume (internal volume) and no more than two dead volumes of the packer interval, sample line, and sampling manifold system. If during the purge (or sampling) the vacuum exceeds 7"Hg, then reduce the pump flow rate. The system vacuum must stay below this level at all times. Purge monitoring will be as follows:

- In-line PID readings of the purge vapor will be collected at a minimum of 60-second intervals.
 - Purge a minimum of one casing volume.
 - After one casing volume, cease purging after either (1) the peak vapor concentration is measured (signaled by two consecutive readings lower than 0.3 parts per million less than the maximum value), or (2) a two-casing purge volume is reached.
7. Open the sample valve (Valve 2) and the purge valve (Valve 3) and start the purge pump. Verify that the flow rate is still 1 L/min.
 8. To start the packer purge, open the gas probe valve (Valve 1) and close the sample valve (Valve 2) at the same time, and start timing. Turn off the helium at this time.
 9. Carefully watch the tubing as the pump is turned on. If water is observed in the sample tubing, **immediately shut off the pump**. Bedrock vapor collection will not be feasible if the probe is in contact with water.
 10. During the entire purge time, measure the concentration of helium in the purge gas using the helium detector (make sure that the helium detector is exposed to ambient air and "zeroes out" before measuring the purged bedrock vapor). The helium concentration in the purged bedrock vapor must be less than 1 percent of what is in the corehole, as measured either inside the helium enclosure during purging or from vapor sampled at depth, from the leak test injection tubing. To pass the leak test (5,000 parts per million by volume [ppmv] if the helium concentration was 50 percent). Either: 1) calculate what 1 percent of the helium concentration was in the enclosure from the measured concentration in Step 5; or 2) use a limit of 0.5 percent (5,000 ppmv), which allows for a 10-times safety margin. If the probe fails the leak check, then corrective action is required. This includes first checking the fittings and connections, then increasing the packer pressure, and possibly deflating and reinflating the packer before trying another purge and leak check. It may also be necessary to remove the packer and re-install it at a different interval. **Note: Helium leak detectors could be sensitive to high concentrations of methane or other atmospheric gases. If these are expected to be present in the bedrock vapor, then caution should be used with this technique, because false positive readings could be encountered during leak testing. Use a GEM-2000 landfill gas meter to evaluate whether methane is present in bedrock vapor (see No. 14).**
 11. During the last 1 minute of the purge, connect a Tedlar bag to the purge pump exhaust and collect a sample for field screening measurements.
 12. If the vacuum gauge reads greater than 7"Hg during the purge, then close the purge valve (Valve 3) and monitor the vacuum in the manifold and probe. If there is no significant change after a minute, then there is an insignificant amount of bedrock vapor and the vacuum is too great to take a vapor sample. Several things can cause this. Consult with the VI senior technical consultant and take corrective action.
 13. At the end of the calculated purge time and after the system is verified to be leak free, close the purge valve (Valve 3), close the valve to the gas sampling bag, and turn off the pump. Do not open the purge valve again. Doing so will result in loss of the purge integrity and will require re-purging.
 14. The purged bedrock vapor in the Tedlar bag can be screened with a GEM-2000 landfill gas meter to obtain field measurements of CO₂, O₂, and CH₄ and/or a MiniRae PID can be used to measure concentrations of total VOCs in the field.

15. Record the purge volume, the field measurement readings, and the leak check information on the Soil Gas Sampling Log.
16. Once the packer purge is complete, immediately move onto the sampling phase. Little to no delay should occur between purging and sampling.

3.3 Sample Collection

“Clean” sampling protocols must be followed when handling and collecting samples. This requires care in the shipping, storage, and use of sampling equipment. Cleanliness of personnel who come in contact with the sampling equipment is also important: no smoking, no eating, no drinking, no perfumes, no deodorants, no dry-cleaned clothing, etc. Canisters should not be transported in vehicles with gas-powered equipment or gasoline cans. Sharpie markers should not be used for labeling or note-taking during sampling.

The air sampling canisters are certified clean and evacuated by the laboratory to about 30”Hg vacuum. Initial canister vacuums that are less than certified by the laboratory are a potential indication of leakage that could affect the accuracy of analytical results. Care should be used at all times to prevent inadvertent loss of canister vacuum. Never open the canister’s valve unless the intent is to collect a sample or check the canister vacuum with an attached gauge.

Do not sample using a canister without sufficient initial vacuum, as shown in Table 1; be advised that sampling data might be flagged or rejected from canisters with low initial vacuum (less than 28”Hg):

1. Verify that the canister has sufficient (28 to 30”Hg) initial vacuum for sampling. Measure the initial canister vacuum using an external digital vacuum gauge as follows:
 - a. Remove the protective cap from the valve on the canister. **Make sure the canister knob is closed!**
 - b. Measure the initial canister pressure using a digital vacuum gauge with 0.25 percent accuracy at the -30 to 0”Hg range and NIST-traceable calibration for vacuum measurements. Open the canister knob and record the reading. Close the canister knob and remove the digital vacuum gauge.
 - c. If using assigned analog vacuum gauges (one for each canister), attach the vacuum gauge to the canister, then attach the flow controller.

Table 1 identifies the field team’s response based on the initial vacuum reading for a canister. In addition, Table 1 also identifies the potential bias to results at different initial canister vacuums.

TABLE 1

Vacuum Reading Responses

NASA SSFL Collection of Bedrock Vapor Samples Using a Packer

Initial Vacuum Reading	Potential Error in Analytical Results Due to Leakage	Field Team Response
>30 to 28” Hg	Up to -10% error	Use canister for sampling – no limitations on use.
>26 to 28” Hg	Up to -21% error	Use canister for sampling if necessary; replace canister with a spare if spares are available.
>24 to 26” Hg	Up to -30% error	Sampling with canister is not advisable. Contact project manager and obtain direction before sampling with this canister. Be advised that qualifiers may be applied to analytical results sampled with canisters with vacuums less than 26” Hg.
<24” Hg	>-30% error	Do not use this canister for sampling. Analytical results will be rejected.

2. With canister knob still closed, attach the canister to the flow controller and then connect the flow controller to the sample valve (Valve 2) on the sampling manifold. Open the sample valve (Valve 2).

3. Before collecting the sample, confirm that the sampling system valves are set as follows: 1) the purge valve (Valve 3) is confirmed to be closed; 2) the gas probe valve (Valve 1) is open; and 3) the sample valve (Valve 2) is open.
4. Slowly open (counter-clockwise) the canister's knob approximately one full turn.
5. After sampling for the appropriate amount of time (determined from project instructions), close the sample valve (Valve 2) and the canister's knob. If the canister has a built-in or assigned vacuum gauge, allow the canister to fill until the vacuum reaches 2 to 10" Hg for 6-liter canisters and 2 to 5" Hg for 1-L canisters and record the final pressure. With the canister knob securely closed, remove the canister from the sampling manifold.
6. Measure the final canister pressure using the same digital vacuum gauge used to measure initial canister pressure. Open the canister knob and record the reading. Close the canister knob again and remove the digital vacuum gauge.
7. If using a single laboratory provided external vacuum gauge, re-attach it, open the canister knob, and record the final vacuum. Close the valve, remove the gauge, and replace and tighten the cap on the canister. Ideal final vacuum in the canister is between 2 and 10" Hg. More than 10" Hg of vacuum can greatly increase reporting limits; however, a small amount of vacuum should be left in the canister so the laboratory can confirm that the canister was not opened during shipment.
8. Consult with the project manager and the project specific senior technical consultant before submitting the sample to the laboratory if a final vacuum greater than 10" Hg, or less than 2" Hg are encountered.

Use Table 2 for guidance for actions to take, depending on the final vacuum measurements.

TABLE 2

Field Team Response based on Final Vacuum Readings

NASA SSFL Collection of Bedrock Vapor Samples Using a Packer

Final Vacuum Reading	Field Team Response
< 2" Hg	Contact Project Manager and project VI senior technical consultant before submitting sample. Notify analytical laboratory to report the laboratory-measured pressure and to get direction from the Project Manager before analyzing sample.
> 2" Hg and <10" Hg	Submit sample for analysis - no limitations on data use
>10" Hg	Contact Project Manager and project VI senior technical consultant before submitting sample. Verify final vacuum with the analytical laboratory before analysis.

9. Canisters with no vacuum left (that is, 0" Hg) should not be analyzed. Contact the Project Manager and project senior technical consultant before submitting a sample with a final vacuum of 0" Hg to establish the appropriate course of action. One option is to verify the final vacuum with the analytical laboratory. If there is vacuum remaining in the canister according to the laboratory vacuum gauge, the Project Manager and/or project senior technical consultant may direct the analytical laboratory to analyze the sample.
10. Record the sampling date, times, canister identification (ID), flow controller ID, vacuum gauge ID(s), and any other observations pertinent to the sampling event on the Soil Gas Sampling Log. Also record the weather conditions (temperature, barometric pressure, precipitation, etc.) during sampling.
11. Fill out appropriate documentation (sampling forms, sample labels, chain of custody, sample tags, etc.).
12. Disassemble the sampling system. Decontaminate the packers prior to reassembly at subsequent wells to be sampled, following the Equipment Decontamination SOP. If another interval is to be packed off

and sampled, decontaminate packers between depth-specific intervals if PID readings of purged vapor exceed 10 ppmv.

3.4 Sample Handling and Shipping

After the appropriate documentation (chain of custody and sample tags) has been completed, return canisters and equipment to the laboratory;

1. The canisters should be shipped back to the laboratory in the same shipping container in which they were received. The samples do not need to be cooled during shipment. **DO NOT** put ice in the shipping container.
2. When packing the canisters for shipment, verify that the valve is sealed (just past finger tight) and valve caps are snug (1/4 turn past finger tight), and use sufficient clean packing to prevent the valves from rubbing against any hard surfaces. Never pack the cans with other objects or materials that could cause them to be punctured or damaged.
3. Do not place sticky labels or tape on any surface of the canister!
4. Place a custody seal over the openings on the shipping container.
5. Make sure to insure the package for the value of the sample containers and flow controllers.
6. Ship canisters for overnight delivery. **NOTE:** If sampling on a Friday, verify that the laboratory accepts samples on Saturdays.

3.5 Quality Control

1. Canisters supplied by the laboratory must follow the performance criteria and quality assurance prescribed in U.S. Environmental Protection Agency (EPA) Method TO-14/15 for canister cleaning, certification of cleanliness, and leak checking. SOPs are required.
2. Flow controllers supplied by the laboratory must follow the performance criteria and QA prescribed in EPA Method TO-14/15 for flow controller cleaning and adjustment. SOPs are required.

4.0 Key Checks and Items

- Wear proper PPE per the HASP.
- Operation of the packer system can be hazardous if carried out by inexperienced staff; therefore, proper training and supervision must be carried out for all personnel involved.