EMISSION ISOLATION FLUX CHAMBER SAMPLING

CONTENTS

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1.0 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to describe the procedures for using a stainless steel double-shell SUMMA passivated isolation flux chamber (hereafter called a flux chamber) to measure the emission rate (upward contaminant flux) of vapor-phase volatile organic compounds (VOCs) associated with impacted subsurface sources (i.e., contaminated soils and/or groundwater). The procedure allows for the calculation of emissions produced from surface or subsurface contamination. This direct emission measurement sampling technique uses a surface enclosure (flux chamber) to isolate a known surface area for emission flux (rate per area) measurement. The flux chamber is designed to isolate those emissions that emanate from the subsurface, and the measured flux emission rate should be reflective of the natural flux emission rate with respect to subsurface fate and transport processes occurring at a site. Sampling of emissions may be performed utilizing any one of the following configurations: with Tedlar® bags, evacuated SUMMA passivated stainless steel canisters, or adsorbent tube cartridges. An example of an emission flux chamber sampling train is illustrated in Figure 1 and Figure 2 in Appendix A.

A Quality Assurance Project Plan (QAPP) in Uniform Federal Policy (UFP) format describing the project objectives must be prepared prior to deploying for a sampling event. The sampler needs to ensure that the methods used are adequate to satisfy the data quality objectives (DQOs) listed in the QAPP for a particular site.

The procedures in this SOP may be modified, dependent on site conditions, equipment limitations or other procedural limitations. In all instances, the procedures employed must be documented on a Field Change Form and attached to the QAPP. These changes must be documented in the final deliverable.

2.0 METHOD SUMMARY

Emissions enter the open bottom of the flux chamber from the exposed surface. Ultra-high purity (zero air) sweep air is added to the flux chamber through the sweep air inlet at a metered rate. Within the flux chamber, the sweep air is mixed with emitted subsurface vapors and gases based on the physical design of the flux chamber. The sweep air creates a slight wind velocity above the emitting surface, preventing a buildup of the emission concentration in the boundary layer directly above the emitting surface. The exit port (sample outlet) is used for measurement of the concentration of the air within the flux chamber or for sampling and subsequent analysis. A port in the top center of the flux chamber fitted with a pressure relief valve prevents pressure buildup within the flux chamber that might occur during sampling. A temperature port also allows excess pressure to escape to the atmosphere.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Once the samples are collected from the flux chamber by one of the selected configurations (Tedlar bags, SUMMA canisters, or adsorbent tube cartridges) and proper sample documentation protocols have been followed, the samples are transported to a laboratory for analysis. The handling of samples should follow the respective QAPP and SOP for the selected collection media.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Sweep air flow rate is the single most important operating factor in flux chamber sampling. If the sweep air flow rate is not maintained at constant rate throughout the sampling event, the accuracy and precision of the results can be affected significantly. In addition, sweep air flow rate must be sufficiently high to minimize re-deposition of compounds from the air to the ground. This objective is typically accomplished by using sweep air flow rate that ensures a complete air exchange every four minutes within the flux chamber.

A positive or negative pressure in the flux chamber enclosure can affect the quality of the emission results. Over-pressurization of the flux chamber may be caused by an obstruction within the pressure relief valve, and under-pressurization of the flux chamber may be caused by a low sweep air pressure or too high of a sampling rate. In addition, flux rates may be affected due to changes in atmospheric pressure and ambient temperature. Barometric pressure changes may induce a change in the subsurface flux rate, and a drastic ambient temperature change may induce a change in the flux of surface contamination.

Sample bias associated with residual contamination of the flux chamber sampling train components may be introduced if the sampling train components are not properly cleaned before use. The potential for sample bias associated with carryover of a contaminant is compound-specific, but the potential increases if high concentrations of VOCs are detected at a sampling point.

5.0 EQUIPMENT/APPARATUS

The flux chamber sampling train design described in this SOP consists of the following components:

5.1 Sweep Air Delivery System

- Zero Air Cylinder, to provide a positive pressure of clean ultra-pure sweep air to the flux chamber.
- Air Pressure (two-stage) Regulator, to provide precise pressure regulation regardless of cylinder pressure changes or air flow fluctuations
- Flowmeter, for precise measuring of sweep air flow rates calibrated to measure 1 to 10 liters per minute (L/min).

5.2 Double-Shell Flux Chamber

- Sweep Air Inlet Line, to allow the transfer of zero air into the flux chamber. The sweep air inlet line is positioned below the flux chamber dome with sweep air exiting from four equally spaced holes pointing toward the center of the chamber that are positioned parallel to the emitting surface to allow for adequate mixing.
- Temperature Readout, to measure the temperature inside the flux chamber. The temperature port vents to the atmosphere.
- Pressure Relief Valve, to maintain atmospheric pressure within the flux chamber.
- Vacuum Pressure Gauge, to maintain vacuum conditions in the insulated outer shell of the chamber of the flux chamber and to minimize the greenhouse effect that can cause an elevation of the internal air/gas temperature. This is critical when measuring the flux from exposed vents.
- Sample Exit Line, to facilitate the flow of the sweep air and maintain a well-mixed and homogeneous sample. The sample exit line is perforated with two columns of holes facing perpendicularly and is extended outside of the flux chamber for sampling (Flux Sample Outlet).

- Sampling Lines and Three-Way Valve, to connect the sampling train. The sampling lines are made of Teflon tubing (1/4" outer diameter) fitted with the necessary Swagelok stainless steel fittings for interconnections. Up to six sections of tubing may be required: one 3-foot section to connect the zero sweep air to the flowmeter, one 2-foot section to connect the flowmeter to the flux chamber sweep air inlet, one 3-foot section to connect the flux chamber sample outlet to the three-way valve, and three 2-foot sections to connect the three-way valve to each sampling configuration. The three-way valve (Whitey Co., SS-42XS4 or equivalent) is required for selection of configuration sampling (valve positions 1, 2, and 3). One flow controller (VALCO Instruments Co., or equivalent) calibrated to 2 L/min is required for accurate SUMMA canister sampling.

5.3 Configuration Sampling System

- Tedlar Bag Sampling - vacuum box fitted with a tubing pass through, personal sampling pump with flow rate calibrated to 2 L/min and Tedlar bags free of visible contamination. Consult U.S. EPA Environmental Response Team (ERT) SOP, *Tedlar Bag Sampling.*

- SUMMA Canister Sampling - leak-free 6-liter stainless steel SUMMA canister fitted with a flow controller calibrated to 2 L/min to connect to the three-way valve or directly to the flux chamber sample outlet. Consult ERT SOP, *SUMMA Canister Sampling* for details.

- Sorbent Tube Sampling - personal sampling pump with flow rate calibrated to 2 L/min. Fresh supply of sorbent tube cartridges. Consult the site-specific QAPP and/or compound-specific sampling SOPs for details.

6.0 REAGENTS

Typically, reagents are not used. After the collection of each sample, post-sampling cleaning usually includes a zero air purge of the system (a minimum of three volumes are recommended) to physically remove adhered particles. If the flux chamber needs additional decontamination, a soap solution wash followed by a hexane rinse will be employed. After completion of the decontamination procedure, the system should be allowed to air dry before reuse or shipping.

7.0 PROCEDURES

7.1 Sampling Train Preparation

Upon deciding on the location to be sampled, the following procedures must be performed:

1. Arrange the flux chamber sampling train on the ground in the order of its components.

2. Set the zero air cylinder on a steady surface and connect the zero air (sweep air) to the flowmeter utilizing approximately 3-feet of Teflon tubing.

3. Place a clean flux chamber on the emitting surface and advance into the ground surface to a depth of 1-2 inches (2.5-5 centimeters). Verify that the gaps around the base of the flux chamber have been sealed.

4. Connect the flowmeter to the flux chamber sweep air inlet utilizing approximately 2-feet

of Teflon tubing.

5. Connect the flux chamber sample outlet to the three-way valve utilizing approximately 3-feet of Teflon tubing.

7.2 Field Operation

Upon completion of the sampling train assembly setup, the following procedures must be performed:

1. Begin sweep air flow. Set the sweep air flow rate at the flowmeter to 3-5 L/min (0.003-0.005 cubic meters per minute).

2. Allow three to four residence times (15-20 minutes) for steady-state concentrations to be reached inside the flux chamber before initiation of sampling.

3. Record date, time, meteorological conditions, and temperature on a field data sheet.

4. Start to collect samples.

7.2.1 Tedlar Bag Sampling

1. Connect the calibrated sampling pump (2 L/min) to the outlet of the vacuum box.

2. Place a Tedlar bag inside the vacuum box. Pass the Teflon tubing from the flux chamber exhaust port or from the three-way valve (position 1) through the inlet of the vacuum box directly to the Tedlar bag. Close the vacuum box.

3. Turn on the sampling pump. Allow the Tedlar bag to fill. Verify that the seal on the vacuum box is intact by listening for air leaks and checking for indentation of the box as it becomes evacuated.

4. Turn off the sampling pump. Remove the Tedlar bag from the vacuum box, lock, and place in a clean cooler or opaque trash bag to prevent photodegradation.

5. Record the appropriate sample information on the field data sheet.

7.2.2 SUMMA Canister Sampling

1. Connect the inlet of the SUMMA canister directly to the flux chamber exhaust port or to the three-way valve (position 2) utilizing approximately 2-feet of Teflon tubing.

2. Open SUMMA canister valve. Allow canister to fill.

3. Close SUMMA canister valve. Record canister final pressure in inches of mercury.

4. Record the appropriate sample information on the field data sheet.

7.2.3 Sorbent Tube Cartridge Sampling

See site specific QAPP and/or compound-specific sampling SOPs.

1. Connect the calibrated sampling pump (2 L/min) to the outlet of the tube manifold.

2. Crack the tube ends and place in manifold with arrow pointing in the direction of air flow and verify that the tube is securely held in place.

3. Connect the inlet of the tube manifold directly to the flux chamber exhaust port or to the three-way valve (position 3) utilizing approximately 2-feet of Teflon tubing.

4. Turn on the sampling pump to collect desired sample volume.

5. Turn off the sampling pump. Remove tube from manifold and cap ends with plastic caps.

6. Record the appropriate sample information on the field data sheet.

7.3 Post-Operation

Decontamination of the flux chamber sampling train is described in Section 6.0. Upon completion of each sample, the Teflon tubing must be replaced.

8.0 CALCULATIONS

During constant atmospheric pressure and temperature conditions, the emission flux rate of VOCs from the ground can be determined by the following equation:

$$E_i = \frac{C_i \text{ x } Q}{A}$$

Where:

E_i = emission flux of component i in micrograms per square meter per minute (µg/m^2-min);
C_i = concentration of component i at the flux chamber outlet in micrograms per cubic meter (µg/m^3);
Q = sweep air flow rate into flux chamber in cubic meters per minute (m^3/min); and
A = surface area enclosed by the flux chamber in square meters (m^2).

All parameters in the equation are measured directly. The surface area for the flux chambers utilized with this SOP is 0.073 m^2. Sweep air flow rate will be set at 3 L/min (0.003 m^3/min).

The calculation of actual surface contamination must be corrected to account for atmospheric pressure and temperature at the time of sampling. The use of an onsite meteorological station is recommended.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

Specific quality assurance/quality control (QA/QC) activities that apply to the implementation of these procedures will be listed in the QAPP prepared for the applicable sampling event. The following general QA procedures will also apply:

1. All sample collection data, including sample number, sample location, start and end times, start and end flow rates, pump number, SUMMA number, media used and analysis/method must be documented on site logbooks or Field Sampling Worksheets.

2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer or instrument-specific SOPs, unless otherwise specified in the QAPP. Equipment check-out and calibration is necessary prior to sampling and must be done according to the instruction manuals supplied by the manufacturer.

3. Records must be maintained, documenting the training of the operators that use instrumentation and equipment for the collection of environmental information.

4. The collection of field blanks is recommended to evaluate the potential for contamination from media. The field blank should be handled in the same manner as the samples, except that no air is drawn through it. Field blanks are collected with the frequency of one per sampling event or a frequency of 5 percent (%), whichever is greater.

   Additional blanks may be required. C onsult the individual sampling SOP and site-specific QAPP for additional information.

10.0 DATA VALIDATION

Data verification (completeness checks) must be conducted to ensure that all data inputs are present for ensuring the availability of sufficient information. This may include but is not limited to location information, start and end times, sampling method and total volume sampled. These data are essential to providing an accurate and complete final deliverable. The ERT contractor's Task Leader is responsible for completing the UFP-QAPP verification checklist for each project.

Results of the QA/QC samples will be evaluated for contamination during the data validation process. This information will be utilized to qualify the environmental sample results accordingly with the data quality objectives of the project.

11.0 HEALTH AND SAFETY

Based on Occupational Safety and Health Administration (OSHA) requirements, a site-specific health and safety plan (HASP) must be prepared for response operations under the Hazardous Waste Operations and Emergency Response (HAZWOPER) standard, 29 CFR 1910.120. Field personnel working for EPA's ERT should consult the Emergency Responder Health and Safety Manual currently located at https://response.epa.gov/_HealthSafetyManual/manual-index.htm for the development of the HASP, required personal protective equipment (PPE) and respiratory protection.

12.0 REFERENCES

Eklund, B. 1992. Practical Guidance for Flux Chamber Measurements of Fugitive Volatile Organic Emission Rates. Presented at the 85th Annual Meeting and Exhibition of the Air and Waste Management Association (Paper 92-66.07), Kansas City, Missouri, June 21-26, 1992.

Eklund B, Petrinec C, Howlett L. U.S. EPA 1991. Database of Emission Rate Projects. EPA/4501-91/003, U.S. EPA, Research Triangle Park, NC.

Hartman, Blayne. 2003. How to Collect Reliable Soil-Gas Data for Upward Risk Assessments, Part 2: Surface Flux-Chamber Method. LUSTLine Bulletin 44, August 2003. Retrieved from http://www.handpmg.com/documents/LustLine-44-Flux_Chambers_Part_2.pdf

Kienbusch, M. U.S. EPA 1986. Measurements of Gaseous Emission Rates from Land Surfaces Using an Emission Isolation Flux Chamber-User's Guide. EPA/600/8-86/008, U.S. EPA, Washington, DC.

13.0 APPENDICES

A - Figures

APPENDIX A
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SOP: ERT-PROC-2117-21
January 2021

FIGURE 1. Picture of Flux Chamber Sampling Train

FIGURE 2. Diagram of Emission Flux Chamber Sampling Train (Setup with a three-way valve)