COLLECTION OF TRANSPIRATION GASES DURING PHYTOREMEDIATION STUDIES

CONTENTS

These sections affected by Revision 0.0

SUPERCEDES: SOP #2032; Revision 0.0; 01/29/99; U.S. EPA Contract 68-C4-0022

1.0 SCOPE AND APPLICATION

The objective of this Standard Operating Procedure (SOP) is to illustrate the setup and operation of the apparatus used to collect plant transpiration gases during phytoremediation studies. Transpiration gas samples are collected *in-situ* and submitted to the laboratory for analysis. In addition, the condensate that occurs during the collection of the gases may be separately obtained and analyzed.

Phytoremediation is the use of living plants to reduce the level(s) of the contaminants of concern (COCs) [i.e., volatile organics (VOCs)] in soil or water. This reduction may be due to direct plant uptake or by providing a hydraulic barrier to the movement of contaminants. Transpiration gases and the condensate associated with a phytoremediation activity may contain the COCs.

A section of a shrub or tree (i.e., several branches) or group of herbaceous stems (e.g. *Phragmites*) is enclosed in a 100-liter (L) Tedlar bag and the transpiration gases are collected, after a recorded time interval, in a suitable vessel (such as a SUMMA canister) appropriate for the COC. A subsample of the resultant gas sample is later removed for analysis.

2.0 METHOD SUMMARY

An open 100-L Tedlar bag is pulled over a suitable, preselected section of vegetation. In all experiments, the 100-L Tedlar bag is isolated from the outside ambient air with a clay seal. Instruments to measure carbon dioxide (CO_2), relative humidity (Rh), and temperature are sometimes placed in close proximity to the area of vegetation being analyzed.

Satisfactory results may be obtained using a static Tedlar bag. Additional devices may be used in conjunction with the bag in an attempt to maximize the transpiration rate by controlling the temperature and Rh within the bag. However, there is no clear evidence at this time to show that the addition of such devices will enhance results. If not controlled, the temperature and Rh could quickly rise within the sealed bag. Probes may be used within the bag to monitor the temperature and humidity.

A micro-fan may be placed in a 100-L Tedlar bag near the meter probes to circulate the air within the bag. A line for recirculating the air inside the Tedlar bag may also be installed such that the negative pressure, or vacuum, line is at the lowest point in the bag, and the positive pressure line is in close proximity to the enclosed branches or stems. The Teflon lines may then connected to a “cold trap” (e.g. ice bath) and recirculating pump. The purpose of the air recirculating line, if utilized, is to circulate the air inside the Tedlar bag through a cold-trap that condenses water vapor. Removing water vapor helps to maintain lower internal Rh and temperature levels inside the bag that may otherwise inhibit the transpiration rate. The micro-fan is utilized to help uniformly distribute the air inside the Tedlar bag.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

Vapor-phase samples from the 100-L Tedlar bag are collected in 1- or 5-L Tedlar bags, SUMMA canisters, or appropriate sorbent tubes. Refer to the appropriate Scientific, Engineering, Response, and Analytical Services (SERAS) SOP regarding the type of sample collection media to be used.

The following is a list of appropriate SERAS SOPs used for the sampling, preservation, storage, and handling of vapor phase samples collected from 100-L Tedlar bags during phytoremediation studies:

SOP#	SOP Title
1704	*Summa Canister Sampling*
2001	*General Field Sampling Guidelines*
2003	*Sample Storage, Preservation and Handling*
2102	*Tedlar Bag Sampling*
2103	*Charcoal Tube Sampling in Ambient Air*
2104	*Tenax/CMS Tube Sampling*

The liquid condensate collected in the sample jar of the cold trap system and water droplets from the walls of the 100-L Tedlar bag can be combined prior to analysis or analyzed separately. The following is a list of SERAS SOPs used for the sampling, preservation, storage, and handling of liquid phase samples collected from 100-L Tedlar bags during phytoremediation studies:

SOP#	SOP Title
2001	*General Field Sampling Guidelines*
2003	*Sample Storage, Preservation and Handling*

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Elevated temperatures and Rh, as well as low levels of CO_2, can affect the transpiration rate of the study plants. Transpiration rates are extremely variable. Variation is due to several factors including but not limited to the plant species, health, stage of development, growing conditions, sunlight, soil moisture, season, temperature, and time of day. When employed, devices such as the recirculating system are designed to remove water vapor from the inside of the sealed 100-L Tedlar bag to keep Rh from reaching saturation. However, due to the tremendous variability inherent in this type of study, good results have been obtained in static systems without these supplemental instruments and devices.

5.0 EQUIPMENT/APPARATUS

The system for the collection of transpiration gases during phytoremediation studies is shown in Figure 1, Appendix A. The cold-trap is shown in more detail in Figure 2, Appendix A.

The 1-, 5-, and 100-L Tedlar bags used must be chemically inert and have a sampling value that can be easily opened and closed (SKC, part numbers: 231-01, 231-05, 231-50, or equivalent). Sampling bags must allow the transmission of visible light without selective absorption of photosynthetic important light wavelengths. Tedlar bags transmit light above 0.4µM wavelength (Figure 3, Appendix A,) which is within the visible light spectrum of 0.45 to 0.75 microns (µM).

Vapor-phase samples can be collected through the Tedlar bag-sampling valve without compromising the clay seal of the 100-L Tedlar bag.

Liquid samples can be collected at the end of the sampling period from the condensate jar of the cold-trap if present or from the 100-L Tedlar bag itself. Generally, condensate develops on the inside walls of 100-L Tedlar bag, and then collects in the lowest part of the bag. The 100-L Tedlar bag clay seal is opened and the liquid is allowed to collect in one of the corners. The bag is cut open and the condensate is collected into an appropriate sample jar.

The recirculation lines are chemically inert 1/4" outer diameter (OD) Teflon tubes (SKC part number 231-9-23, or equivalent). The flexible tubing used in the head of the peristaltic pump must be chemically inert such as silicone (Masterflex C-flex tubing, part number 06 424-15, or equivalent). The peristaltic pump has an interchangeable pump head that can be easily disassembled to change the section of flexible tubing for each phytoremediation study (GeoTech pump model GeoPump-2, Masterflex pump head part number 7015-00, or equivalent).

The temperature, Rh, or CO2 monitoring instruments inside the Tedlar bag allow the measurement of these parameters using the smallest sample volume possible so as to minimize interference with VOC concentrations. Data logging of these parameters over time may also desirable. Meters with internal data logging capabilities can be used to measure temperature, Rh and CO_2 concentrations inside Tedlar bags (Cole-Parmer Rh/temperature meter Model Tri-sense, Solomat Model MPM-4100 temperature/Rh meter, CEA instruments Model RI-411A portable infrared gas analyzer or HP model Micro-GC portable gas chromatograph).

6.0 REAGENTS

Ultra-zero ambient air (Scott Gas, or equivalent) with a certified CO_2 concentration $_{in}$ the range of 315-385 parts per million by volume (ppmv).

7.0 PROCEDURES

The optimal condition for generating and collecting transpiration gases is still in development. The system outlined in this SOP has not been tested widely or frequently enough to determine if all the components in the system are necessary or if methods or components need amendment. It is suggested that solar radiation, local meteorological conditions, and tree sap flow rates be monitored during the collection of the transpiration gases. The frequency of sampling and the sample collection duration depends upon the age or size of the tree leaves, ambient temperatures, and on-site monitoring and analytical capabilities. For best results, samples should be collected during the optimal solar radiation period of the day, typically from 10:00 to 14:00 hours. Sampling duration is typically up to four (4) hours maximum, or until the leaves inside the 100-L Tedlar bag begin to show signs of stress or wilting. Samples may be taken periodically (e.g. ½ hour intervals) or once at the end of the sampling period. Samples for confirmatory analysis are usually taken at the end of the sampling duration using sorbent tubes or SUMMA canisters. All sampling times and observations must be documented in appropriate field logbooks. The use of static bags without ultra-zero air or additional equipment will often produce satisfactory results.

1. Select branches or groups of stems, preferably in bright light, which appear to be representative of the phytoremediation area. There should also be a section of the stem clear of leaves and branches to facilitate sealing of the 100-L Tedlar bag.

2. Cable tie temperature, Rh, and CO_2 meters, micro-fan, and Teflon lines approximately midway on

the branches or stems to be enclosed, if applicable. Place positive pressure Teflon tube at the midway position on the stem or branch and extend the negative pressure or vacuum end of the Teflon tube beyond the end of the stem or branch. This end of the tube is placed at the lowest corner of the 100-L Tedlar bag in Step #4.

3. Cut the 100-L Tedlar bag at the bottom, approximately 1 to 2 inches from the end and opposite to the valve end. Pull the 100-L Tedlar bag over the branches or stems. It may be necessary to gently bend some of the outer branches to fit inside the bag.

4. Position the negative pressure or vacuum Teflon tube so that it is in the farthest corner of the 100-L Tedlar bag. This corner should also be the lowest in elevation and will be used to collect any water that accumulates in the bag.

5. Select a clear area on the stem or branch and position the open ends of the 100-L Tedlar bag. Clamp one of the wooden splints, with the gasket seals to the inside, on one end of the bag and secure with binder clamps. The first set of splints should seal about one half the bag.

6. Clamp the remaining section of the bag with a second set of wooden splints, as close to the first set as possible. Make sure the electronic wires from the meters and micro-fan, and the Teflon tubes are between the splints and are adequately secured, if applicable.

7. Place a lump of modeling clay in the gap between the splints. Work the clay among the tubes and wires to completely seal the 100-L Tedlar bag against the stem of the branch. It may be necessary to moisten the clay to make it pliable in order to form a proper seal around the stem and bag.

8. Immediately fill the 100-L Tedlar bag with ultra-zero air. Connect the battery to the micro-fan to start the air circulating, if applicable. Connect the Teflon tubes to the recirculation system (peristaltic pump, cooling coils, cold trap) and start the pump. The 100-L Tedlar bag may have to be supported by tying the corners to adjacent branches to make sure it is level, facing the sun, and secure.

9. Record start time, initial temperature, Rh, CO_2, and any other pertinent parameters (cloud cover, rain, winds).

10. Collect initial sample through the sampling valve of the 100-L Tedlar bag.

11. Collect samples at the designed frequency and monitor temperature, Rh, and CO_2 throughout the duration of the phytoremediation study and sample collection activities.

12. If CO_2 levels drop, add approximately 35 milliliters (mL) of pure CO_2 to increase the level inside the 100-L Tedlar bag to approximately 350 ppmv.

8.0 CALCULATIONS

The amount of pure (99 %) CO_2 necessary to maintain the CO_2 inside the 100-L Tedlar bag in the range of 315 to 380 ppmv has been calculated to be approximately 35 mL, as follows:

$$\frac{35\ mL}{100\ L} \times 99\% \approx 350\ ppmv$$

9.0 QUALITY ASSURANCE/QUALITY CONTROL

There are no specific quality assurance (QA) activities which apply to the implementation of these procedures. However, the following general QA procedures apply:

1. All data must be documented on field data sheets or within field logbooks.

2. All instruments must be operated in accordance with operating instructions supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration activities must occur prior to sampling/operation and must be documented in field logbooks.

10.0 DATA VALIDATION

This section is not applicable to this SOP.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow United States Environmental Protection Agency (U.S. EPA), Occupational Safety and Health Administration (OSHA), and corporate health and safety procedures.

12.0 REFERENCES

This section is not applicable to this SOP.

13.0 APPENDICES

A - Figures

APPENDIX A
Figures
SOP #2032
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FIGURE 1. Transpiration Gas Collection Apparatus

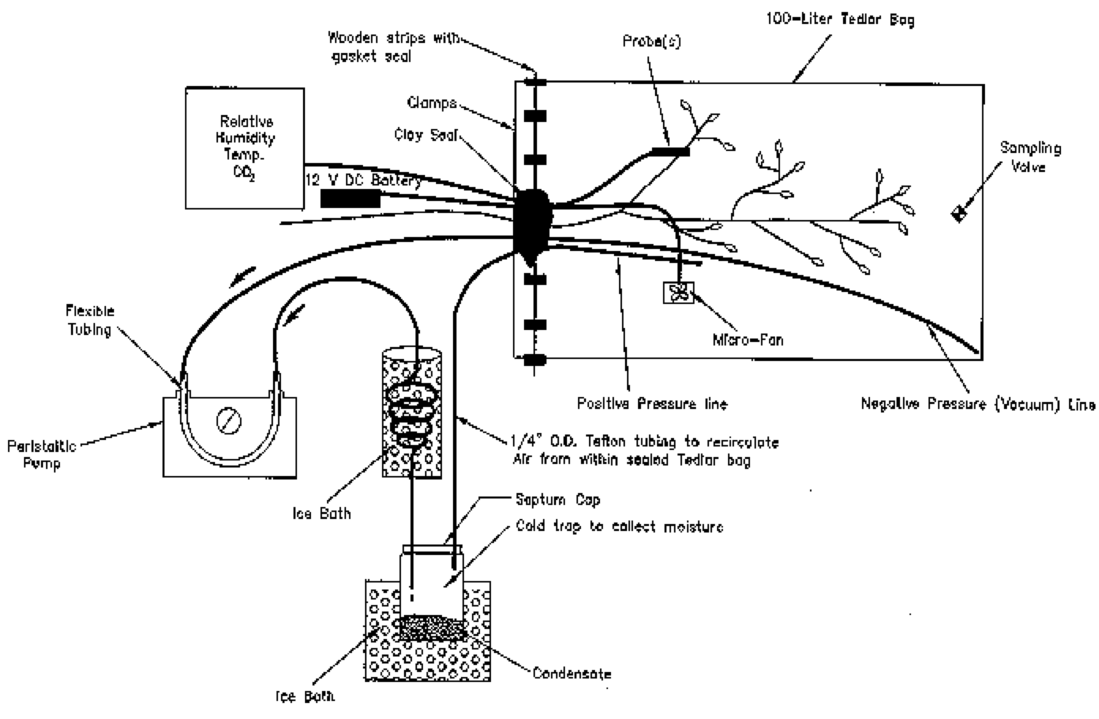

FIGURE 1

Transpiration Gas Collection Apparatus

FIGURE 2. Cold-Trap System