PROCESSING AIR SAMPLES WITH THE PORTABLE SAMPLE CONCENTRATOR

CONTENTS

SUPERCEDES: SOP #1730; Revision 1; 06/30/90; U.S. EPA Contract EP-W-09-031.

PROCESSING AIR SAMPLES WITH THE PORTABLE SAMPLE CONCENTRATOR

1.0 SCOPE AND APPLICATION

The purpose of this procedure is to define the means of processing air samples with a portable sample concentrator. The sample concentrator is a field portable sorption tube concentration device used to concentrate dilute air samples prior to chromatographic analysis. It can be used with any gas chromatograph that allows the directed injection of air samples. The concentrator has been successfully used on air samples with vapor pressures ranging from 1-2600 millimeters of mercury (mmHg), and concentration factors up to 250 have been achieved.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent on site conditions, equipment limitations or limitations imposed by the procedure or other procedure limitations. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. EPA endorsement or recommendation for use.

2.0 METHOD SUMMARY

A large volume of sample with a dilute concentration of organic contaminants is passed through a tube filled with 80-100-mesh Tenax and Spherocarb (the "trap"). The compounds adsorb onto these materials and when the trap is heated, the compounds are desorbed with helium and collected in a smaller volume. Thus, a constant number of organic molecules are taken from a large volume and put into a small volume, effectively increasing the concentration.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Samples are loaded into the concentrator using an internal sample pump. A manual syringe injection of sample onto the adsorption traps is an option if the use of a sample pump is not desired. These samples can be collected in a large container such as a Tedlar or other polymer film bag. All bags should be leak-tested before use. Although satisfactory cleaning of the bags has been demonstrated, cross-contamination problems can be avoided by disposing them after each use. In addition, because solvents found in the glue on labels and in marking pens will permeate the bags, samples should be identified by affixing a label to the edge of the bag, outside of the seam. If Tedlar air bags are used, all samples should be analyzed within 48 hours of collection.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

The sample concentrator is not weatherproof and should be protected from water at all times; nor is it intrinsically safe, and so must be kept out of explosive atmospheres. Only particulate-free gas samples should be injected into the traps. The effects of corrosive gases on concentration efficiency have not been investigated and introduction of such gases may reduce the service life of the unit. **Liquids should not be injected directly into the concentrator**. Further, operation of the concentrator in environments with ambient temperatures greater than 90°F may adversely affect trapping efficiency for highly volatile compounds.

5.0 EQUIPMENT/APPARATUS

The Concentrator was developed and manufactured at Louisiana State University's Institute for Environmental Studies. Its operation requires the use of the following equipment:

- Beckton-Dickerson 50-mL glass syringe (with Yale metal Luer-Lok tip and individually fitted barrel and plunger - VWR Scientific, Cat. #BD2315 or equivalent).

- Beckton-Dickerson 2-mL glass syringe (with Yale metal Luer-Lok tip and individually fitted barrel and plunger - VWR Scientific, Cat. #BD2310 or equivalent).

- Each syringe is fitted with a Mininert Syringe Valve (Precision Sampling, Inc., Baton Rouge, LA, Cat. # 654051 or equivalent).

- 1.5 L Tedlar bag (with on/off valve - Teklab, Inc., Baton Rouge, LA, Cat. # 20212-03 or equivalent).

A brief manual for the unit appears in Appendix A.

6.0 REAGENTS

- Helium, 99.995% minimum purity.

- Certified compress gas standard(s) cylinder (Scott Gas, Matheson, or other reliable source). Concentrations should be within ±2% of listed values.

7.0 PROCEDURE

7.1 Concentrator Set-Up

1. Attach the helium line to the 1/8 inch Swagelok fitting (helium) and adjust the delivery pressure to 10-15 psig (Figure 1).

2. Place the following switches in the "off " position (either the "down" or the "left" positions): main switch (power), fan power switch (Fan On), and pump power switch (Pump). Place the Trap Heat switch in the center "off " position, and plug the AC power cord first into a power outlet, and then into the concentrator unit. The Trap Heat switch is a triple pole switch which selects the trap to be heated. It also controls a relay which automatically selects the proper thermocouple for temperature monitoring and heat control.

3. Turn on the Power switch. If any indicator lights come on, immediately pull the power cord from the front panel, re-check all of the switches, and repeat the procedure. The digital panel on the heater control unit should come on when the Power switch is in the "on" position. If it does not, check the fuse, the AC power source, and the cord.

4. Turn the Fan On switch on, check that the fan turns, and that the amber fan indicator light comes on. If the light comes on but the fan does not turn, switch the fan off, and ensure that the fan blades turn freely. If the fan turns freely, turn off the main power switch, unplug the power cord from the front panel, and remove the concentration unit from the housing. Eight screws secure the unit to the housing: one in each corner of the front panel, and four on the bottom of the housing. Check the leads going to the fan, then reinstall the concentration unit in the housing using all eight screws (two screws on the bottom

counteract the torque generated in turning the handle on the switching valve; the other two secure the pump).

5. Place an empty 2-mL syringe in the sample port. Open the Trap valve by pulling the handle to a vertical position. Open the Syringe valve (push the green button) and, with the switching valve in the Run 1/Load 2 position, check to see that there is gas flowing through trap.

   The proper flow rate is 1-mL in approximately 15 seconds and is controlled by the "Trap Adj" needle valve (turn counterclockwise to open).

6. When the flow through Trap 1 is established, place the switching valve in the Run 2/Load 1 position and check the flow through Trap 2 in the same manner.

7. Connect a 1/8" OD Teflon line from the sample to pump port.

8. After closing the Trap valve, the concentration unit is ready.

7.2 Concentrating a Sample

1. Because elevated trap temperatures may decrease trapping efficiency, be sure the temperature of the trap to be used is no more than 30°C. Trap temperature is monitored by switching the Run/Temp switch to the "Temp" position, and the Trap Heat switch to the position corresponding to the trap of interest.

2. Begin the concentration procedure by estimating the amount of the sample needed to be put into the trap to give the analytical system a detectable peak, using a desorb volume of 1 ml.

3. Connect a 1/16" OD Teflon line between the Vent 1 port and the bottom port of the rotameter. Connect a 1/16" Teflon line between the Sample from Pump port to the Load 1/Run 2 port. Place the Switching valve in the "Load 1/Run 2" position and place the pump vent valve in the "Sample To Vent" position. The pressure drop created by the 1/16" OD plumbing of the concentrator will prevent the pump from starting while in-line-starting the pump with the pump vent in the wrong position may damage the pump motor. Connect the sample to be analyzed to the Sample to Pump port via the 1/8" Teflon line.

4. Switch both the pump and the Pump Vent valve to the "Sample To Trap" position. Adjust the sample flow (with the Sample Adjust valve) to the desired rate and begin timing the trap loading period. When the desired period has elapsed, switch the Pump Vent valve to the "Sample To Vent" position and switch off the pump.

5. Close the Trap valve and place the Switching valve in the "Run 1/Load 2" position. Then switch the Trap Heat switch to the "1" position. Place the temp/heat switch (located by the temperature display panel) to the "heat" position. At the pre-set temperature (20°C), both the power indicator for the heater controller and the heat indicator light should begin to cycle on and off.

6. Wait approximately one minute after the temperature reaches 230°C before desorbing the sample. Place a clean, empty, 2-mL syringe on the sample port with the Syringe valve open. Begin desorbing the trap with helium by opening the Trap Toggle valve. Close the Syringe valve when 1 ml of sample

enters the syringe, and allow the helium flow to purge the trap while it is heated. Place a section of tubing on the sample port to vent any remaining vapors away from the working area and into a carbon scrubber or fume hood. This cleans the trap in preparation for the next sample. Since the plumbing is not heated, the time required to clean the instrument depends upon the concentration and volatility of the compounds in the preceding sample, and upon the ambient temperature. Total mass loads of 300 ng/ml of compounds with vapor pressures of 75 mm Hg or greater require approximately eight minutes for cleaning when the ambient air temperature is 75°F. The same mass loading of compounds with vapor pressures ranging from 10 to 75 mm Hg will require 12 minutes for cleaning at the same ambient air temperature. The time required for cleaning should be determined on a case-by-case basis and is accomplished by running successive blanks while the trap is being cleaned. When the trap is clean, turn the Trap Heat switch to the center "off" position. Place the Temp/Heat switch to the "temp" position to monitor cooling off the trap.

7. The sample is ready for analysis after the 2-mL syringe is removed from the Sample Out port of the concentrator. Samples are concentrated with Trap 2 by switching the 1/6" OD pump vent tube to the Vent 2 port, switching the 1/16" OD sample loading tube to the Load 2 port, and placing the switching valve in the "Run 1/Load 2" position. To desorb the sample, place the switching valve in the "Run 2/Load 1" position.

8.0 CALCULATIONS

The concentration factor and the actual concentration in the sample are calculated as follows:

$$\text{Concentration Factor} = \frac{\text{volume of sample loaded}}{\text{volume of desorbed sample)}}$$

$$\text{Actual Concentration} = \frac{\text{measured concentration}}{\text{concentration factor}}$$

For example, if a 500-mL sample is loaded and desorbed into 1 mL, the concentration factor will be 500. If the analysis of the concentrated sample yields a concentration of 500 ppb, the original concentration of the sample was 1 ppb.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

Trap blanks should be run at the beginning of each day, using a gas which is free of organic contaminants (the helium carrier gas can be used). Each trap's ability to concentrate a sample should also be checked once daily. Certified calibration gas standards should be concentrated using three or more concentration levels, preferably bracketing the ranges expected in the field samples. Expected concentration should be calculated and compared to a previous calibration curve of the certified standard. The analysis of the concentrated sample should be within ±30% of the analysis of the certified standard. If the analysis does not fall within this range, the concentration should be leak-tested and the above-mentioned procedure repeated. If the problem continues, replace the defective trap.

At this time, the service life of these traps is unknown. Acceptable recoveries have been achieved even with traps which have processed an estimated 400 samples.

10.0 DATA VALIDATION

The sample concentrated on the field portable concentrator system will undergo subsequent GC analysis. GC analysis by its nature relies on the Retention Time Index (RTI) to identify compounds in the sample matrix. If the RTI's of sample peaks are within 5 to 10% of the standard gas mixture, they are considered identical. It is quite possible that two or more compounds could elute at similar RTIs. Alternative analytical methods, such as GC/MS, would be required to confirm the identity of these co-eluting peaks. Adsorption/desorption efficiencies on the trap, preferably in similar background matrices as the sample, should be determined prior to entering the field. This will define the optimum loading capacity of the trap. From these studies, "hot" samples, as determined by GC or other analytical means, could be evaluated to determine whether they needed to be re-run at a lower loading. If any questions remain as to the trapping efficiency and/or recovery from the system, a series of known additions can be performed. In that case, portions of the sample are dosed with several known levels of standards and the recovery through the system can be evaluated. Lower recoveries will be recorded and noted in a results table from the field analysis run.

11.0 HEALTH AND SAFETY

The sample concentrator should be used in a well ventilated room. A vent line should be placed in the Sample port after the sample has been taken, and while it is still in the run mode. This will allow any contaminants that may be purging out of the trap to be vented to the outside or into a carbon scrubber for clean-up. Disposable protective gloves are required only if liquid reagents are to be used, or if the samples are highly contaminated.

When working with potentially hazardous materials, refer to U.S. EPA, OSHA or corporate health and safety practices.

12.0 REFERENCES

Overton, E.B., T.H. McKinney, C.F. Steele, E.S. Collard, and R.W. Sherman, "Rapid Field Analysis of Volatile Organic Compounds in Environmental Samples", Proceedings of the Third Annual Symposium on Solid Waste Testing and Quality Assurance, USEPA, Washington, DC, July, 1978.

Overton, E.B., R.W. Sherman, E.S. Collard, P. Klinkhachorn, and H.P. Dharmasena, "Current Instrumentation for Field Deployable Analysis of Organic Compounds", Proceedings of the Joint Army-Navy-NASA-Air Force Safety and Environmental Protection Subcommittee Meeting, Monterey, CA, May, 1988.

Overton, E.B., R.W. Sherman, H.P. Dharmasena and C.F. Steele, "Remote Sensing Using Gas Chromatography Via a Macintosh Interface", Proceedings of the Sixth Annual Hazardous Materials Management Conference International, Atlantic City, N.J., June, 1988.

Overton, E.B., R.W. Sherman, C.F. Steele, and H.P. Dharmasena, Correlation "Chromatography with a Portable Microchip Gas Chromatograph", Proceedings of the First International Symposium for Field Screening Methods for Hazardous Waste Site Investigations, Las Vegas, NV, Cleveland, OH.

Overton, E.B., C. Steele, R. Sherman, E. Collard, S. and T. McKinney, "Field Useable Compound Specific Analytical Device" Proceedings of Safety and Environmental Protection Subcommittee of the Joint Army-Navy-NASA-Air

Force Interagency Propulsion Committee, May, 1987, Cleveland, OH.

Sherman, R., T. McKinney, M. Solecki, R. Gaines, and B. Shipley, "Field Use of a Microchip Gas Chromatograph", Proceedings of the First International Symposium for Field Screening Methods for Hazardous Waste Site Investigations, Las Vegas, NV, October, 1988.

Sherman, R.W., M.K. Solecki, E.S. Collard, T.H. McKinney, L.H. Grande, and E.B. Overton, "Development of a Field Portable Concentrator/Purge and Trap Device for Analysis of VOC in Ambient Air and Water Samples", Proceedings of the First International Symposium for Field Screening Methods for Hazardous Waste Site Investigations, Las Vegas, NV, October, 1988.

APPENDIX A
Sample Concentrator Manual
SOP #1714
December 1994

PROCESSING AIR SAMPLES WITH THE PORTABLE SAMPLE CONCENTRATOR

PROCEDURE

Concentrator Set-Up:

1. Attach the helium source to the 1/8 inch Swagelok fitting (helium) and adjust the regulator pressure to 10-15 psig.

2. Place the Power Fan and Pump switches in the "off" (left) position.

3. Place the Temp/Heat switch in the "Temp" position.

4. Plug the A.C. power cord into an electrical outlet and then into the concentrator unit.

5. Turn on the Power switch. The digital panel of the temperature controller should come on. If any other indicator lights come on, turn the unit off and recheck the switch settings.

6. Turn on the Fan switch and verify that the indicator light comes on and that the fan blades are turning.

7. Place an empty 2-mL syringe, with a Miniert™ syringe valve attached, in the Sample port and open the Trap valve by lifting the handle to a vertical position. Open the syringe valve (push the green button) and, with the trap selection valve in the "Load 2/Run 1" position, check to see that gas is flowing through trap #1). The proper flow rate is approximately 1 ml per 15 seconds and is controlled by the Trap Adj needle valve (counter-clockwise to increase flow). Switch the trap selection valve to the "Load 1/Run 2" position and similarly verify the flow through trap #2.

8. Connect a 1/8 OD Teflon line to the Sample to Pump port. Close the Trap valve and the unit is ready to concentrate samples.

To Concentrate a Sample:

1. Verify that the temperature of the trap to be used is no more than 30°C by placing the Temp/Heat selector in the "Temp" position and the Heat selector to the proper position for the trap being loaded (either 1 or 2). This will give the temperature of the selected trap and neither trap will be heated.

2. Connect a 1/16" OD Teflon line between the Sample from Pump fitting and the Load 1 or Load 2 fitting, as appropriate. Also connect a 1/16" OD line between the appropriate Vent fitting and the bottom port of the rotameter.

3. Place the trap selection valve in the "Load 1/Run 2" position if trap #1 is to be loaded or in the "Load 2/Run 1" position if trap #2 is to be loaded.

4. Place the pump vent valve in the "Sample to Vent" position and turn on the pump switch.

   CAUTION: Starting the pump with the pump vent valve in the wrong position may damage the pump.

5. Connect the sample to the Sample to Pump port using the previously attached 1/8" Teflon tubing.

6. Switch the pump vent valve to the "Sample to Trap" position and adjust the flow rate as desired by using the Sample adjust needle valve adjacent to the Sample to Pump port.

7. Allow the pump to run until the desired volume of sample has passed through the trap. Switch the pump vent to "Sample to Vent" and turn off the pump.

To desorb (or Run) a sample:

8. Verify that the Trap valve is off and switch the trap selection valve to the alternate position (e.g. from Load 1/Run 2 to Load 2/Run 1 if trap #1 was being loaded).

9. Place the Heat selector switch to the desired trap (the trap being "Run").

10. Place a clean 2-mL syringe, with a syringe valve attached, on the Sample port adjacent to the trap selection valve. Open the syringe valve and switch the Temp/Heat switch to "Heat". The temperature will rise to 230-240°C in approximately 45 seconds.

11. After the temperature reaches the set point (230°C), wait one minute and then open the Trap valve. Gas will begin to flow into the syringe. When 1 ml of gas has flowed into the syringe, close the syringe valve and remove the syringe. The sample is now ready for analysis.

12. Leave the Trap valve open and continue heating the trap for 2 minutes* to prepare it for the next sample. After this time, continue the purge gas flow and discontinue trap heating by switching the Temp/Heat switch to "Temp".

* Because the plumbing is not heated, the time required to clean the instrument depends on the concentration and volatility of the compounds in the preceding sample as well as the ambient temperature. Total mass loads of 300 ng/mL of compounds with vapor pressures of 75 mm Hg or greater require approximately eight minutes for cleaning the plumbing lines when ambient temperatures are 24°C (75°C). Given the same ambient temperature and mass loading, compounds with vapor pressure in the 10 to 75 mm range will require about 12 minutes. The actual time required should be determined on a case by monitoring the purge gas effluent at the Sample port.

13. The alternate trap can be loaded while the previously loaded trap is being purged. Note, however, that the temperature of one trap cannot be monitored while the other trap is being heated.

Calculations

The concentration factor and the actual concentration in the sample are calculated as follows:

$$\text{Concentration Factor, } F = \frac{V_S}{V_d}$$

$$\text{Actual concentration} = \frac{C_m}{F}$$

where:

V_s = volume of sample passed through the trap
V_d = volume of desorb gas in the syringe
C_m = measured value of the concentrated sample

the Purge and Trap Portable Concentrator
SOP #1714
December 1994

PROCESSING AIR SAMPLES WITH THE PORTABLE SAMPLE CONCENTRATOR

VOLATILE ORGANICS FROM SOILS AND WATER USING THE
PURGE AND TRAP PORTABLE CONCENTRATOR

1.0 INTRODUCTION

The purpose of the "purge and trap" is to remove volatile organic compounds from water or soil so that they may be analyzed easily by using the Micromonitor. This is accomplished by bubbling helium or clean compressed air through the water, stripping the organics from the samples, and passing the volatile organics onto the Tenax traps. The traps are then desorbed in the normal manner to yield a sample ready for analysis.

2.0 SAMPLE PREPARATION

To use the purge and trap for water samples it is necessary to have the water in a small vial with a septum cap. The level of the water should be no higher than one inch from the top of the cap. If you are analyzing soil, mix some soil into clean water. Try to mix it so that you have as much soil as possible (the more sample the better) without making the slurry too thick.

For waters, 5-mL of sample in a 10-mL septum vial is satisfactory. For soils, 2 grams of soil plus 3-mL of clean water placed in a 10-mL septum vial is used. The actual volumes of waters and weights of soils may vary according to application, but all values should be recorded for later concentration calculations.

3.0 BUBBLER SET-UP

1. Using two pieces of clean Teflon tubing with male Luer fittings on both ends, hook one piece of tubing to Bubbler port and hook the other piece of tubing to the appropriate Load port.

2. Attach a short thick (18 or 20 gauge) syringe needle to the Load tubing, and attach a long thin (26 gauge) syringe needle to the Bubbler tubing. If possible, the Bubbler syringe needle should be long enough to reach the bottom of the vial.

3. Poke the Bubbler needle through the septum, pushing it down to the bottom of the vial or as far as it will go.

4. Poke the Load needle through the septum, but keep the point of the needle as close to the septum as possible.

To start bubbling through the sample, be sure that you are hooked up according to the instructions given in "Bubbler Set-Up", and open the "Bubbler" valve. The flow rate of the purge gas can be controlled with the needle valve. The flow rate can be measured by connecting on-line at the appropriate "vent" trap port to the bottom from the concentrator's rotameter. Set the flow at 50-100 cc/min and bubble 3-5 minutes, collecting 300-500 cc onto the trap.

4.0 PREPARING FOR THE NEXT RUN

As when you are concentrating air samples, "clean" the Tenax traps by desorbing helium while the trap is heated

after every run. Be sure that the Tenax trap is "cooled" to at least 30-35°C before you start to load the next sample. If you are reusing Teflon tubing, clean the tubing by blowing helium through the tube using the Bubbler port. Be aware that by reusing syringe needles or Teflon tubing you run the risk of contaminating later samples. If you want to check for this, run a "blank" by bubbling through clean water, and analyze with the Micromonitor.

5.0 RECOVERY CALCULATIONS

If you have an estimate of the concentration of the organics in the desorbed sample analyzed by the Micromonitor, the concentration in water may be determined using the following calculations:

For the desorbed sample in the 2-mL syringe:

(conc in ppm) X MW/24.5 = conc in mg/m^3 = conc in ng/mL, where MW is the molecular weight of the contaminant.

If you used 5-mL of contaminated water:

(conc in ng/ml) X ml air/5-mL water = conc in water in ng/mL, or ppb in water on a weight basis.

This calculation assumes 100% efficiency in the purging, trapping, and desorbing, which is unlikely. However, this will give you an estimate of the water concentration. If you analyzed soil by putting it into water it is necessary to adjust your calculations to account for the amount of soil that was mixed into the water.