SUMMA CANISTER CLEANING PROCEDURES

CONTENTS

SUPERCEDES: SOP #1703; Revision 2.1; 05/24/91; U.S. EPA Contract EP-W-09-031.

SUMMA CANISTER CLEANING PROCEDURES

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) is intended for use when cleaning Summa polished stainless steel canisters. Summa canisters provide a medium to sample gas phase Volatile Organic Compounds (VOCs) on-site at concentrations of one part per billion by volume (ppbv) and greater. This procedure is to assure that canisters have been sufficiently cleaned prior to sampling, to the extent that no VOC contamination is present at concentrations greater than 0.2 ppbv.

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

After use, canisters are logged in and physically inspected. These canisters are vented to the outside air under an operating exhaust hood. Canisters are connected to a manifold which is attached to a vacuum pump via a cryogenic trap. The canisters and lines are evacuated and then the canisters are heated to an elevated temperature for a prescribed time period. During the heating period, the canisters are filled with humidified nitrogen and pressurized. The process is repeated. The filling and pressurizing functions are followed by evacuation and heating and are performed a total of three times.

Canisters are confirmed free of VOC contamination by pressurizing the canisters with ultra high purity nitrogen and analyzing on the GC/MS. If no VOC contamination is present at concentrations greater than 0.2 ppbv, the canister is determined clean. Clean canisters are leak-tested by pressurizing with nitrogen for 24 hours. Canisters that have been determined clean and without leaks are evacuated. These canisters are logged in as cleaned and certified and are stored in the evacuated state with brass cap fittings until needed for sampling.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

3.1 Canister Receipt

1. The overall condition of each sample canister is observed. Any canister having physical defects requires corrective action.

2. Each canister should be observed for an attached sample identification number.

3. Each canister is recorded in the dedicated laboratory logbook by its Summa canister number.

3.2 Canister Storage

1. Canisters are stored in an evacuated state of less than 0.05 mm Hg and with a brass cap in place. The canisters remain in this state until needed.

2. An identification tag is attached to the neck of each canister for field notes and to complete the chain of custody record.

3. Each canister is recorded in the dedicated laboratory logbook stating the canister status and storage location. Also noted on the identification tag are the date cleaned and date certified clean, as well as the initials of the operator.

4.0 INTERFERENCE AND POTENTIAL PROBLEMS

Contamination may occur in the sample canisters if they are not properly cleaned before use. All other equipment used in this process must be sufficiently clean. All gases and solvents used must be certified 99.99% pure to avoid contamination. Canisters must be stored with the valve closed and the brass caps in place to avoid vacuum loss.

5.0 EQUIPMENT/APPARATUS

5.1 Canister

Sample canister - Leak free stainless steel pressure vessels at desired volume (e.g., 6L), with valve and Summa passivated interior surfaces (Scientific Instrumentation Specialists, Inc., P.O. Box 8941, Moscow, ID, 83843 or Anderson Samplers, Inc., 4215-C Wendell Dr., Atlanta, GA, 30336), or equivalent.

5.2 Canister Cleaning System (Figure 1, Appendix A)

Vacuum pump - capable of evacuating sample canister(s) to an absolute pressure of <0.05 mm Hg.

Manifold - stainless steel manifold with connections for simultaneously cleaning several canisters.

Shutoff valve(s) - two on/off toggle valves (Valves A, and B).

Stainless steel vacuum gauge (pressure gauge) - capable of measuring vacuum in the manifold to an absolute pressure of <0.05 mm Hg or less.

Cryogenic trap - stainless steel U shape open tubular trap cooled with liquid nitrogen to prevent contamination from back diffusion of oil from vacuum pump.

Stainless steel two stage pressure regulator 0-690 kPa (0-100 psig) to regulate nitrogen pressure.

Teflon tee with a septum port - an injection port capable of introducing distilled, deionized water to provide moisture to the nitrogen supply line.

Isothermal oven - a system for heating canisters (Fisher Scientific, Pittsburgh, PA, Model 349) or equivalent.

6.0 REAGENTS

Gas cylinders of nitrogen, ultra high purity grade.

Cryogen - liquid nitrogen (bp -195°C).

Distilled, deionized water, ultra high purity.

7.0 PROCEDURE

7.1 System Set-Up

1. All connections in the vacuum system except the canisters and manifold are sealed. All connections, lines, and valves are checked for leaks by pressurizing the line to 30 psig and using a soap solution. The septum is checked for leaks by visual inspection.

2. The liquid nitrogen is added to the cryogenic trap and allowed to equilibrate.

3. Check the pump to assure proper working order by achieving a vacuum of 0.05 mm Hg in the line that normally attaches to the manifold but is now capped. Valve A is open and Valve B is closed. After the vacuum test is completed, turn the pump off and remove the cap to break the vacuum.

4. Check the oven to assure proper working order by heating the oven to 100°C and measuring the internal temperature with a thermometer.

5. Check reagents to assure proper purity.

6. Set the back pressure on the nitrogen to 30 psig.

7.2 Cleaning

1. All canisters are vented to the outside air under an operating exhaust hood.

2. Connect the canisters (with the valves closed on the canisters) to the manifold by the Swagelok fittings. Connect the manifold to the vacuum system by the Swagelok fitting.

3. Open Valve A, assure Valve B is closed, and start vacuum pump.

4. Once a vacuum (0.05 mm Hg) is obtained in the line and the manifold, Valve A is closed. The system is then examined for leaks by comparing the initial vacuum reading and a second vacuum reading three minutes later. If the vacuum deteriorates more than 5 mm Hg, a leak exists and corrective action, such as tightening all fittings, is necessary.

5. If no leaks are observed, Valve A is opened and the Canister 1 valve is opened. Evacuate Canister 1 to 0.05 mm Hg, then close Canister 1 valve. By evacuating one canister at a time, cross contamination between canisters is minimized.

6. Evacuate all other canisters in the same manner as described in the above step.

7. After all four canisters are evacuated, open all canister valves. Turn on the oven and heat to 100°C.

8. Continue evacuating canisters for one hour at 100°C. Document the time.

9. After one hour, Valve A is closed and Valve B is opened, with the regulator metering the flow of nitrogen.

10. Inject 100 µL of distilled, deionized water via a syringe through the humidity injector port in the nitrogen line.

11. Allow the canisters to pressurize to 30 psig for 15 minutes.

12. Close Valve B.

13. Close canister valves.

14. Repeat steps 5 through 13, twice.

15. Close valves on canisters.

16. Close Valve A.

17. Turn off vacuum pump.

18. Disconnect manifold from cleaning system.

19. Disconnect canisters from the manifold and place a brass cap on each canister.

20. Choose the one canister of this set of four that was analyzed as being the most highly contaminated previous to cleaning. Fill this canister with ultra high purity nitrogen to a pressure of 30 psig.

21. Analyze the above canister for VOC contamination by GC/MS. If this canister is sufficiently clean to the extent that no VOC contamination is present at concentrations greater than 0.2 ppbv, then all canisters in that set of four are considered clean. Document the results.

22. Evacuate the above canister again to 0.05 mm Hg, cap it with a brass fitting, and store it with the other three of the lot. Document the location.

23. If the above canister is not sufficiently clean (i.e. VOC contamination is present at concentrations greater than 0.2 ppbv), then all canisters in that lot must be cleaned again until the canisters meet the prescribed criteria. Document the results.

7.3 Leak-Testing

1. Once the canister lot is determined as being clean, the canisters are pressurized to 30 psig with nitrogen.

2. The initial pressure is measured via the pressure gauge, the canister valve is closed, and the brass cap is replaced. Document the time and pressure.

3. After 24 hours, the final pressure is checked. Document the time and pressure.

4. If leak tight, the pressure should not vary more than ±13.8 kPa (±2 psig) over the 24-hour period. If this criterion is met, the canister is capped with a brass fitting and stored. If a leak is present, corrective action such as tightening all fittings, is required. Document the results.

8.0 CALCULATIONS

There are no calculations for this SOP.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

The following specific quality assurance/quality control procedures are applicable for Summa canister cleaning:

1. All connections, lines, and valves are checked to assure no leaks are present.

2. The septum is checked, to assure no leaks are present, by removing the septum and visually examining it.

3. The pump is checked to assure proper working order by achieving a vacuum of 0.05 mm Hg prior to cleaning.

4. The oven is checked to assure proper working order by comparing the oven setting at 100°C to the internal temperature with a thermometer.
5. The reagents are checked to assure sufficient purity.

6. All canisters are to be evacuated to 0.05 mm Hg during each cycle of the cleaning process and the results are to be documented.

7. All canisters are to be evacuated at 100°C for one hour during each cycle of the cleaning process. Results are to be documented.

8. All canisters are to be evacuated, heated, and pressurized three times during the cleaning process. Document each cycle.

9. The selected canister from the cleaning lot to be tested must be analyzed by GC/MS as shown to be sufficiently cleaned to the extent that no VOC contamination is present at concentrations greater than 0.2 ppbv for the canister lot to be considered cleaned. If the VOC contamination is greater than 0.2 ppbv, the canister lot must be cleaned again. In either case, the results will be documented.

10. All canisters will be leak-tested for 24 hours and the results will be documented.

11. All canisters will be stored evacuated and capped with a brass fitting. The pressure and location will be documented.

10.0 DATA VALIDATION

This section is not applicable to this SOP.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA or corporate health and safety practices. More specifically, liquid nitrogen is used to cool the cryogenic trap. Its boiling point is -196°C. Insulated gloves, lab coat, face shield, and safety glasses must be worn when using this material. Liquid nitrogen must be transported only in properly constructed containers.

Ultra high purity nitrogen is used to clean the canisters and must be labeled properly. All cylinders must be securely fastened to a stationary object. The cylinder valve should only be opened by hand. The proper regulator must be used and set correctly.

The oven is set to a temperature of 100°C. Insulated gloves should be worn when handling items heated to this temperature.

Prior to cleaning, canisters are to be vented to the atmosphere under an operating exhaust hood. The hood must be in proper working order.

Canisters are pressurized during the cleaning operation. No canister is to be pressurized above 30 psig. The maximum pressure limit for the Summa canisters is 40 psig.

12.0 REFERENCES

ASTM Standards D1356-73A - Standard Definitions of Terms and Relating to Atmospheric Sampling and Analysis.

Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air EPA/600/4-87/006, September 1986, Method TO-14 - Determination of Volatile Organic Compounds (VOCs) in Ambient Air Using Summa Canister Sampling and Gas Chromatographic Analysis.

APPENDIX A
Figures
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FIGURE 1. Canister Cleaning System