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**USEPA REGION 9 LABORATORY
RICHMOND, CALIFORNIA**

STANDARD OPERATING PROCEDURE #312

**CLEANING AND CERTIFICATION OF SPECIALLY PREPARED
CANISTERS FOR AIR SAMPLING**

Signature & Title

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1.0 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) applies to the cleaning, leak testing and certification of specially prepared canisters that are used for collection of air samples that are subsequently analyzed for volatile organic compounds. Canisters included are SUMMA® polished interior canisters and SilcoSteel™ silica coated interior canisters. The procedures are based on EPA methodologies, specifically Compendium Method TO15, titled "The Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)".

2.0 METHOD SUMMARY

The canister cleaning procedure involves repeatedly filling a canister with humidified nitrogen followed by evacuation of the canister to a vacuum < 50 mtorr (0.05 mm Hg) while heating the canister to 105°C.

Canisters are leak tested by pressurizing them to approximately 30 psig with humidified zero air. The initial pressure is then measured and recorded. After 24 hours the pressure is again measured and recorded to determine if a leak is present.

Canisters are certified clean by pressurizing them to approximately 30 psig with humidified zero air. A aliquot of the air in the canister is then analyzed according to SOP #310 to verify the absence of target analytes.

The canister cleaning system and the air toxics analytical system are located in Room 203 which is the Region 9 Air Analyses Laboratory. The laboratory is maintained at positive pressure to prevent intrusion of volatile contaminants from the surrounding laboratories.

3.0 DEFINITIONS

- 3.1 SUMMA® Canister spherical stainless steel container that has undergone a SUMMA® treatment that results in a highly polished interior surface resistant to adsorption of volatile organic compounds
- 3.2 SilcoSteel™ Canister spherical stainless steel container with fused silica interior

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- 3.3 lining that resists adsorption of volatile organic compounds
GC/MS Gas Chromatograph/Mass Spectrometer

4.0 HEALTH AND SAFETY

- 4.1 Due to the unknown and potentially hazardous characteristics of samples all sample handling must be performed in or vented into a laboratory fume hood.
- 4.2 Specially-prepared canisters should never be pressurized beyond 40 psig which is the maximum allowable pressure for specially-prepared canisters.
- 4.3 When using liquid nitrogen; insulated gloves, a lab coat, and safety glasses must be worn. Liquid nitrogen must be transported in a properly constructed container.

5.0 SAMPLE CANISTER HANDLING AND STORAGE

- 5.1 After an air sample has been analyzed and after storage of the sample for the time period outlined in SOP #310, indicate that the canister is ready for cleaning by applying a red sticker.
- 5.2 Canister cleaning, leak testing and certification are logged into the canister cleaning logbook (See Attachment A). A separate page is maintained in the logbook for each canister. Each canister has a unique number.

6.0 INTERFERENCES

- 6.1 Impurities in the dilution gas, organic compounds out-gassing from the cleaning system, and solvent vapors in the laboratory may result in contamination problems. To minimize this possibility, the cleaning apparatus should be assembled of clean, high quality components. Nitrogen and zero air should be of high purity and should be passed through a VOC trap or zero air generator. The nitrogen and zero air should be humidified with organic free water that has been prepared according to SOP #205, "Preparation of Organic-Free Method Blank Water".

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- 6.2 Canisters should be capped tightly with the valve closed during storage and shipping to minimize contamination of the canister.
- 6.3 Solvents and other compounds which are target analytes must never be introduced into the laboratory where volatiles analysis is performed. Methylene chloride and other common laboratory chemicals are target analytes under this SOP and must be excluded from Room 203.

7.0 APPARATUS AND MATERIALS

7.1 Canister Cleaning System

Examples of canister cleaning systems can be found in References 11.1, 11.2 and 11.5 of this SOP. The canister cleaning system at the Region 9 Lab is assembled from the following components.

- 7.1.1 Vacuum Pump. Capable of evacuating canister to an absolute pressure of <0.05 mm Hg
- 7.7.2 Manifold. Stainless steel manifold with four connections for simultaneously cleaning four canisters (Scientific Instrumentation Specialists; Moscow, Idaho)
- 7.7.3 Shut-off Valve: On-off toggle valve.
- 7.7.4 Convectron Vacuum Gauge capable of measuring vacuum in the manifold to an absolute pressure of 50 mtorr or less. (Granville-Phillips Series 275).
- 7.7.5 Cryogenic Trap: stainless steel U-shaped open tubular trap cooled with liquid nitrogen to prevent contamination from back diffusion of oil from vacuum pump and to provide clean zero air to sample canisters.
- 7.7.6 Stainless Steel Gas Regulator to control delivery pressure of zero air.
- 7.7.7 Water Sparger constructed of Swagelok fittings to humidify zero nitrogen entering the canisters.
- 7.7.8 Isothermal Oven capable of heating canisters to 105°C (Thermolyne Series 9000).

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7.7.9 Stainless Steel Flow Control Valves: two for controlling flow of zero gas.

7.7.10 Stainless steel pressure gauge for monitoring zero gas pressure.

7.7.11 Vacuum Isolation Valve a manual bellows valve that isolates the canister from the vacuum system

7.1.12 Stainless steel tubing and fittings

7.1.13 Gas Purifier connected in-line between nitrogen and cleaning system inlet to remove moisture and organic impurities from gas streams (Supelco Supelcarb HC Hydrocarbon Trap and a S/P Moisture Trap packed with 13X Molecular Sieve).

7.2 Reagents

7.2.1 Nitrogen, high purity

7.2.2 Air, ultra high purity

7.2.3 Organic-free water (Region SOP #205)

7.3 Analytical System

The analytical system and associated reagents and standards are detailed in Region 9 SOP #310, "Analysis of Volatile Organic Compounds (VOCs) in Air Collected in Specially Prepared Canisters".

8.0 QUALITY CONTROL

When analyzing air samples to certify canisters as clean, the QA/QC detailed in Region 9 SOP #310, "Analysis of Volatile Organic Compounds (VOCs) in Air Collected in Specially Prepared Canisters" must be followed.

9.0 PROCEDURES

9.1 Canister Cleaning

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- 9.1.1 Canisters tagged with a red sticker are ready to be cleaned. Open canister valve in a hood to bring contents to ambient pressure then close canister valve.
- 9.1.2 Close valve between pump and cleaning oven and turn on the pump and the oven. Set oven temperature to 105 degrees C.
- 9.1.3 Check o-rings on oven manifold to determine that they are intact. With canister valves closed, connect canisters to oven manifold making sure to tighten the connection enough so no leak will occur (finger tight should be sufficient).
- 9.1.4 Fully open canister valves and close oven door.
- 9.1.5 Open the vacuum valve to begin pump down. Continue pump down until temperature reaches the set point and monitor the vacuum. Pump down to <50 mtorr on the digital vacuum gauge. When temperature and pressure reach set points, note the time and hold the heat and vacuum for 15 minutes.
- 9.1.6 Turn oven off and open oven door. Make sure the humidifier in the gas line contains organic free water. After 10 minutes, close the vacuum valve and open the gas line valve slowly. Fill cans to 30 psi pressure with humidified nitrogen then close the gas inlet valve. Keep a close eye on the pressure to ensure that it doesn't exceed the canister limit of 40 psi.
- 9.1.7 Close the gas line valve. Turn on oven and open the vacuum valve to begin second pump-down. Continue pump down until temperature reaches the set point and monitor the vacuum. Pump down to <50 mtorr on the digital vacuum gauge. When temperature and pressure reach set points, note the time and hold the heat and vacuum for one hour.
- 9.1.8 Turn off oven and open oven door. Allow canisters to cool to near ambient temperature. Close valves, close canisters and disconnect canisters from manifold.
- 9.1.9 Add 200 - 250 l of organic-free water to each canister and pressurize canisters to approximately 30 psi with ultra high purity air.

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9.1.10 Perform leak test as outlined in Section 9.2 of this SOP.

9.2 Leak Testing

9.2.1 After cleaning the canisters, allow canister to reach room temperature. Check the canister pressure and record it in the canister cleaning logbook. Record the time and tag canisters with a yellow sticker to indicate leak test.

9.2.2 Allow canisters to stand overnight (16-24 hours) and remeasure the pressure. No decrease in pressure should be apparent (± 2 psig is the method limit). If the canister passes the leak test, record canister pressure and tag canister with a blue sticker (indicating canister is ready for GC/MS analysis). If the canister fails the leak test, determine the cause of the leak and repair it.

9.3 Canister Certification

9.3.1 Following Region 9 SOP #310, analyze an aliquot of gas from each canister that has been determined to be leak tight. If any target compounds are detected at 0.5 ppbv or greater, reclean and reanalyze the canister. In addition, if more than 5 non-target compound peaks are present and are $\geq 10\%$ of the nearest internal standard peak or if any single non-target compound peak is $\geq 50\%$ of the nearest internal standard peak, then the canister must be recleaned and reanalyzed.

9.3.2 If the cleanliness check is satisfactory, tag the canister with a green tag, record the date that the canister was cleaned and the date the canister was certified on the canister label, and record the certification date in the canister cleaning log.

9.3.3 Store canisters in this pressurized condition until they are ready to use. Store the canister in the cabinet in Room 203. Prior to use, evacuate the canister to <50 mtorr. Record canister vacuum in inches of Hg on the canister label prior to sending canisters out to the field.

9.3.4 The canisters are considered clean for a period of 6 months after being

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certified clean. If upon recertification, the cleanliness check is not satisfactory the canister must be re-cleaned, leak tested and certified.

10.0 DOCUMENTATION

- 10.1 Data from the Region 9 Laboratory is presented to the client in one of two general reporting formats: a complete, validatable data package or a summary report. For the former the laboratory prepares summary forms of calibration, quality control, and sample results and provides this information along with the raw data, a case narrative, and an analytical report spreadsheet to the client. The requirements for a complete data package for the determination of volatile organic compounds in air are outlined in Region 9 SOP #310, "Analysis of Volatile Organic Compounds (VOCs) in Air Collected in Specially Prepared Canisters". As indicated in that SOP, the data package must include documentation on the cleaning and certification of canisters used to collect air samples or to prepare dilutions of air samples. Canister certification data that should be included in data packages include relevant pages from the canister cleaning log, the raw data quantitation report, the reconstructed ion chromatogram (RIC) of the data file, the raw spectra and enhanced spectra of the target compounds detected in the sample, as well as the enhanced spectra of the corresponding target compound in the calibration file, in order of elution, and enhanced spectra of non-target compounds detected in the canister with a library search listing the three best fits of a forward library search of the non-target compounds.

11.0 REFERENCES

- 11.1 USEPA; Compendium Method TO-15; Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS); January, 1997.
- 11.2 USEPA; Compendium Method TO-14A; Determination of Volatile Organic Compounds (VOCs) in Ambient Air Using Specially Prepared Canisters With Subsequent Analysis by Gas Chromatography. January 1997.
- 11.3 USEPA; Statement of Work (SOW) for the Analysis of Air Toxics from Superfund Sites. Draft report, June 1990.

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- 11.4 Region 9 SOP 315, Cleaning and Certification of Specially Prepared Canisters for Air Sampling.
- 11.5 USEPA: Compendium of ERT Air Sampling Procedures. May 1992.

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APPENDIX A

DEVIATIONS FROM EPA METHOD TO-15

1. Method TO-15 defines cleanliness as less than 0.2 ppbv of targeted VOCs. This SOP defines cleanliness as less than 0.5 ppbv of targeted VOCs.
2. Method TO-15 does not address the presence of non-target compounds in its definition of canister cleanliness, whereas this SOP does.
3. Method TO-15 does not define a time period that canisters can be considered clean after certification. This SOP considered canisters to be clean for a period of 6 months after certification.

