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PROCEDURES FOR AUTOMATED SUMMA CANISTER CLEANING

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1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) is intended for use when cleaning polished stainless steel SUMMA type or glass-lined Silco type canisters. These canisters provide a medium for sampling gas phase volatile organic compounds (VOCs) on-site at parts per trillion by volume (pptv) concentrations and greater. This procedure has been developed to ensure that canisters have been sufficiently cleaned prior to sampling by the Environmental Response Team/Scientific, Engineering, Response and Analytical Services (ERT/SERAS) Laboratory.

2.0 METHOD SUMMARY

After use, canisters are logged into a logbook and physically inspected for irregularities. The canisters are connected to an automated canister cleaning system designed to simultaneously clean multiple canisters by cycling between an evacuation cycle and a refilling cycle using clean humidified nitrogen. The canisters are also heated by means of heating bands or blankets to further facilitate the removal of any VOCs or surface bound contaminants. Based on the extent of contamination, this process may be repeated for an unlimited number of cycles.

Canisters are pressurized with ultra high purity humidified air and confirmed free of VOC contamination by analysis using a gas chromatograph/mass spectrometer (GC/MS). The canisters are considered clean when target compounds and non-target compounds are not detected above 0.2 parts per billion by volume (ppbv) and 2 ppbv, respectively, for the full scan TO-15 analysis. Optionally, canisters may be cleaned down to a site-specific reporting limit (RL) of 70 pptv and are considered clean when target compounds of interest are not detected above this RL. Canisters are then logged in as "cleaned and certified" and stored in the evacuated state with brass or stainless steel cap fittings until needed for sampling.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

The overall condition of each sample canister is observed. Any canister having physical defects requires corrective action. Canisters are stored in an evacuated state of less than (<) 0.05 millimeter of mercury (mm Hg) or approximately 30 inches (") of Hg with a brass cap placed over the valve. The canisters remain in this state until needed or for three months. After the three month shelf period has expired, these canisters are then re-checked for leaks. If the vacuum in a canister is < -27" Hg, the canisters are returned to the storage shelf. If the vacuum is greater than (>) 27" of Hg, the canister valves are checked and repaired and the canister is re-cleaned. All canisters should be re-cleaned and re-certified after six months of storage. Each canister is recorded in a dedicated laboratory logbook (Figure 1) by the canister number (i.e., barcode number) with its current status.



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An identification tag is attached to the neck of each canister for field notes and/or to complete the chain of custody record. The identification tag contains information that documents the date cleaned, date certified, certification number (lab-generated sequential number), initials of the person who cleaned the canister and the name of the certifying analyst.

4.0 INTERFERENCE AND POTENTIAL PROBLEMS

Sample contamination may occur if the canisters are not properly cleaned before use. Care must be taken to ensure the cleanliness of the equipments used for the cleaning process. All gases and solvents used must be 99.99 percent (%) pure to avoid contamination. Canisters must be stored with the valve closed and capped.

5.0 EQUIPMENT/APPARATUS

- Sample canister, leak-free stainless steel pressure vessels at desired volume (e.g., 6-liter [L]), 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.
- Canister cleaning system, Entech Model 3100 Canister Cleaning System or equivalent (Figures 2 and 3)
- Stainless steel vacuum gauge (pressure gauge), capable of measuring vacuum in the SUMMA canister to -29" Hg vacuum
- Stainless steel two-stage pressure regulator, 0-690 kilopascals (kPa) or 0-100 pound force per square inch gauge (psig), to regulate nitrogen pressure
- Valve, 3-way for switching from nitrogen to zero air
- Heat source, heating bands (Entech), canister blankets (Meriter) or equivalent
- Identification tags, green for canisters used for samples and white for canisters used for standards

6.0 REAGENTS

- Gas cylinders of air, ultra high purity grade
- Cryogen Liquid nitrogen (boiling point -195 degrees Centigrade [°C])
- Distilled or de-ionized water



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7.0 PROCEDURE

7.1 Canister Receipt

As canisters are returned from use, they are logged or scanned as received. A chargeable work assignment number (WA#) should be legibly written on the identification tag and the canister placed on the designated shelving area for cleaning.

7.2 Canister Cleaning

Connect the canisters (with their valves closed) to the manifold by their Swagelok fittings. Proceed according to manufacturer's instructions for canister cleaning.

A step-by-step procedure for the Entech Model 3100 Canister Cleaning System is as follows:

- 1. Power "ON" the Model 3100 Canister Cleaner. Start the rough pump and toggle the "Start" switch ON.
- 2. Boot computer, if not already started.
- 3. Double click on the "3100 Cleaner" icon (Figure 4). Click on "Open". Choose the "canclean.m30" method. Enter the number of cycles to run, ex. ≥4.
- 4. Click on "RUN". This will open to the "Run Control Screen" (Figure 5).
- 5. Attach canisters. Plug any unused positions in the manifold.
- 6. Slip heaters or blankets over canisters. Leave canister valves closed.
- 7. In the "Run Control Screen", click "ON" button next to "Rough Pump" and wait until pressure reads 2.0 pound per square inch absolute (psia) or less.
- 8. Click "ON" button next to "H.V. Pump". Vacuum should drop below 100 millitorr (mtorr) in about 1 to 5 minutes. This ensures that the system and the valves are leak free. If not, a leak exists in the canister connection or "thru" the canister valve. Tighten each canister fitting and valve one-by-one. If necessary, remove canisters one-by-one to see which has the greatest effect on the vacuum.
- 9. Once leak tight, click on the "ALL OFF" button.
- 10. Open all the canister valves. Vent excess pressure through the charcoal filter to the outside by means of the Swagelok valve at the front of the manifold. Do not start the cleaning system with canisters above 15 psia. Close valve once system SERAShes between 17-14.7 psia.
- 11. Plug in alternating current (AC) cords for the heaters. At the computer, click on "GO" to start the process.



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- 12. Automated cleaning will occur over the next 1 to5 hours depending on the parameters chosen and the number of canisters being cleaned.
- 13. A final vacuum of 50 mtorr, equivalent to 0.05 mm Hg, is targeted. **NOTE:** If a final vacuum below 100 mtorr cannot be attained, a leak may be present in one of the canisters at the valve weld. To find the leaking canister, try closing each canister valve one at a time and note any change in the vacuum.
- 14. Once a vacuum of 50 mtorr or less has been achieved, maintain the vacuum until all canister valves are closed.
- 15. Turn off power to the heaters and/or blankets.
- 16. Wait approximately 15 minutes or longer for canisters to cool.
- 17. One or all canisters can be filled with humidified air for certification by opening the valve and clicking the "ON" button next to "Diluent". Fill the canister(s) with zero humidified air to a pressure of 12 psia or greater to be able to draw the appropriate volume off for analysis.
- 18. Close all valves and remove canisters from the manifold. Place a cap on each valve.
- 19. After a minimum of 12 hours of "aging", the canisters are documented on a chain of custody (Figure 6) and submitted for VOC analysis.. The canisters are considered clean when target compounds and non-target compounds are not detected above 0.2 ppbv and 2 ppbv, respectively, for the full scan TO-15 analysis. Optionally, canisters may be cleaned down to 70 pptv and are considered clean when target compounds of interest are not detected above a site-specified reporting limit (RL). Document the results.
- 20. Once certified clean, the canisters are re-evacuated to -29" Hg. Cap the canisters and stored on shelving designated for certified clean canisters.

7.3 Canister Release for Use

The vacuum must be checked on all canisters prior to release using the stainless steel gauge to verify that at least 27"Hg vacuum has been maintained. Document the actual vacuum reading and date of measurement in the logbook.



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7.4 Leak-Testing for Pressurized Sampling

When canisters will be pressurized during sampling (i.e. Anderson Sampling), the following leak test must be performed prior to use:

- Once the canisters have gone through the cleaning process, the canisters are pressurized to > 12 psia with zero humidified air and sent to the ERT/SERAS Laboratory for certification.
- 2. The initial pressure is measured via the pressure gauge, the canister valve is closed, and the cap is placed back on the canister. Document the time and pressure.
- 3. After 24 hours, the final pressure is checked. Document the time and pressure.
- 4. If the canister is leak tight, the pressure should not vary more than ±13.8 kPa (±2 psia) over the 24-hour period. If this criterion is met, the canister is capped 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 canister cleaning:

- 1. All connections, lines, and valves are checked to ensure that no leaks are present. Follow manufacturer's instructions for leak testing.
- 2. All canisters are to be evacuated, heated and pressurized at least four times during the cleaning process. The Entech Model 3100 provides for an unlimited number of cleaning cycles.
- 3. All canisters are to be evacuated to a final vacuum of -29" Hg or less. All canisters will be stored in an evacuated state and capped.
- 4. All canisters must be leak-tested, either by positive or negative pressure testing, prior to release for use. All results are to be documented on the identification tag/log book.
- 5. Any unused canisters will be re-submitted for certification after six months.



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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. Environmental Protection Agency (EPA), Occupational Safety and Health Administration (OSHA) and corporate health and safety practices. More specifically, liquid nitrogen is used to clean the canisters, its boiling point is -196°C. Insulated gloves, lab coat, face shield and safety glasses must be worn when using this material. Liquid nitrogen and compressed air must be transported only in properly constructed containers.

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 heater bands are set to a temperature of 100°C by the manufacturer. 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 or vent. The hood must be in proper working order.

Canisters are pressurized during the cleaning operation. Pressurize the canisters to 10 psig less than the maximum pressure limit of the canister. The maximum pressure limit for the canisters is to be observed.

12.0 REFERENCES

American Society for Testing and Materials (ASTM). 2000. Standard D 1356-00a - Standard Terminology Relating to Sampling and Analysis of Atmospheres.

U.S. EPA. 1999. Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, 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), EPA/625/R-96/010b.





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FIGURE 1. Example of Laboratory Logbook

SUMMA#	Date Cleaned & Initials	COC#	PSI	QA	Date Certified	Site Name	WA#
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- 110						***************************************	
<u> </u>						****	
<u> </u>	+						
							
<u> </u>							
							
A second							

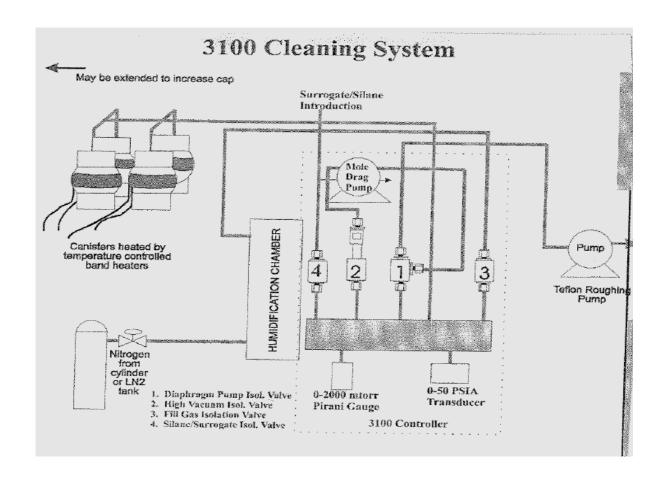
	1						
			-				
			-		 		
				1			

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FIGURE 2. Entech Model 3100 Canister Cleaning System

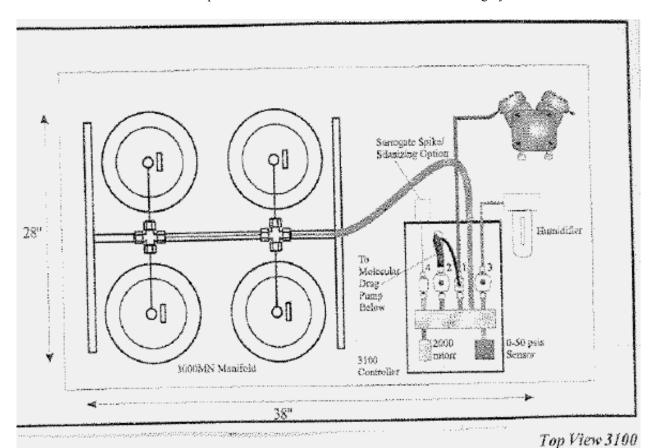






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FIGURE 3. Top View of the Entech Model 3100 Canister Cleaning System

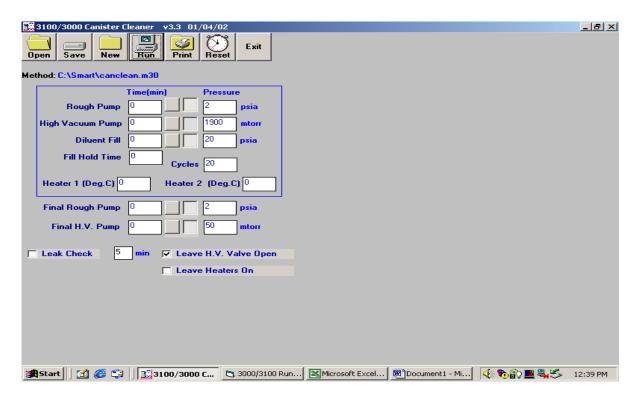






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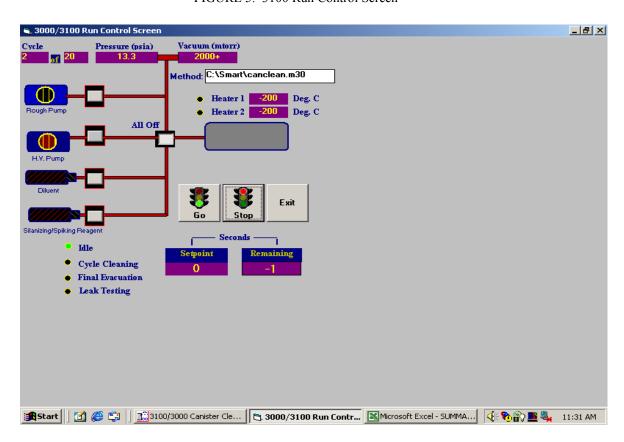
FIGURE 4. 3100 Canister Cleaner Screen





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FIGURE 5. 3100 Run Control Screen





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FIGURE 6. Chain of Custody for SUMMA Canister Certification

