

Title: Automated Cleaning of Sample Canisters		Copy No: ##
SOP No.: 7.01/1.5/S	Effective Date: September 11, 2013	Location: ###

QSM Approval: _____

Automated Cleaning of Sample Canisters

1. Scope

This Standard Operating Procedure provides instructions for proper operation of the Air Toxics Unit's automated canister cleaning system, and explains the purpose of the various steps in the procedure.

2. Procedure

2.1 Check the Methane Heat pyrometer gauge on the clean air generator. The gauge should indicate a temperature of $290^{\circ}\text{C} \pm 10^{\circ}\text{C}$. A red light above the gauge should be cycling on and off. Seek assistance if this temperature reading is not observed.

2.2 Setup

2.2.1 Prior to cleaning any canisters, check their ID tags to ensure that all required analyses have been performed. These are identified by the instrument on which they need to be run and will be highlighted in specific colours when done: MSD or Non-Polar (Green), FID (Pink) and Polar (Orange). There should also be an indication that the results have been checked – a check mark or a line through the tag. If there is any doubt that the canister is ready for cleaning, consult the appropriate analyst.

2.2.2 Check the canister valves to ensure that they are in the proper position. If there is a guard surrounding the valve assembly, the knob should be centered on the opening. If it is turned towards the side, loosen the 9/16" fitting directly below the valve using a 9/16" wrench with a 7/8" wrench to hold the valve in place, reposition the valve and tighten the fitting.

2.2.3 Attach canisters to manifold lines in oven by hand (finger-tight) to check for damaged threads on canister valve fittings. Reverse or replace valve on any canister with seriously-damaged threads. Tighten fittings with a 9/16" wrench, while stabilizing the valve by grasping firmly with the other hand. If the valve twists easily, tighten fittings connecting valve to canister. Ensure that all canister valves are closed. Record the canister ID numbers in the log book, include date, whether the cleaning is during the "day" or "night," and initial. Generally 18 canisters will be cleaned at a time; however, up to 36 x 3 L canisters may be cleaned with the addition of extra lines. If there are not enough canisters of one type to make up



Title: Automated Cleaning of Sample Canisters		Copy No: ##
SOP No.: 7.01/1.5/S	Effective Date: September 11, 2013	Location: ###

18, 6 L and 3 L canisters may be cleaned at the same time so long as the overall volume does not change by more than 20%.

2.2.4 Inspect water level in the glass cold trap. If level is too high, in excess of 4 cm, request assistance from lab staff familiar with procedure for removing trap. **CAUTION: This trap is normally under vacuum. Attempting to remove trap while under vacuum can result in breakage and serious injury.**

2.2.5 On Mondays, drain the oil from the filter atop the high vacuum pump by removing the plug from the front of the filter and allowing the oil to drain into the provided can. Ensure that the oil level on the pump is within 25-75% of the maximum level. Add oil as required.

2.2.6 Raise dewar over cold trap, clamp securely in place, and fill slowly to brim with liquid nitrogen. Cover the opening with the Styrofoam cap to minimize evaporation.

2.2.7 Manually turn on the high vacuum pump by means of the toggle switch on the pump.

CAUTION: This vacuum pump must ALWAYS be on whenever the cold trap is immersed in liquid nitrogen. Failure to observe this precaution could, under certain circumstances, result in an eventual explosion.

2.2.8 If the red lights on the Chron Trol module for "1" and "4" are not illuminated, press "1" on the numeric keypad, then press the "ON" key and repeat for "4". (This circuit provides power to the Magnelatch solenoid valve in the clean air supply line. A red light for circuit 4 will be displayed on the programming module.)

2.2.9 Set the relay timer for the high vacuum pump to 45 minutes for 6 L canisters, 30 minutes for 3 L canisters. Set the relay timer for the diaphragm pump to minimum setting. Press "3" on the numeric keypad, then press the "ON" key. (A red light for circuit 3 will be displayed on the programming module. The diaphragm pump will turn on briefly, then the solenoid valve to the high vacuum pump will open. The canister manifold will be evacuated in a matter of a few seconds for leak-testing purposes.)

2.2.10 When the manifold is fully evacuated (pressure gauge reading at, or very near, -30 in. Hg) press "3", then press the "OFF" key. (The red light for circuit 3 will go off. The canister manifold is now isolated from the vacuum pump.) **OBSERVE THE PRESSURE GAUGE FOR POSSIBLE LEAKS IN THE SYSTEM. IF NO LEAK IS IMMEDIATELY APPARENT, WAIT AT LEAST 5 MINUTES. AN ACCEPTABLE LEAK CHECK IS ACHIEVED IF THE RATE OF RISE OF PRESSURE DOES NOT EXCEED 2 IN. HG PER 5**

Title: Automated Cleaning of Sample Canisters		Copy No: ##
SOP No.: 7.01/1.5/S	Effective Date: September 11, 2013	Location: ###

MINUTES. IF THE RATE EXCEEDS THIS, REPEAT THE LEAK CHECK PROCEDURE TO VERIFY EXCESSIVE LEAKAGE. IF VERIFIED, TRACE AND ELIMINATE THE SOURCE OF THE LEAK, AND REPEAT THE LEAK CHECK UNTIL AN ACCEPTABLE TEST IS ACHIEVED.

- 2.2.11 After an acceptable leak check is achieved, open all canister valves.
- 2.2.12 Close the oven door and turn on the switch that controls the oven temperature. The display above the door will read 70°C).
- 2.2.13 Set the relay timer for the diaphragm pump to approximately 10 minutes.
- 2.2.14 Adjust the flow control valve for the supply air, if necessary, to adjust the rate at which the canisters will fill with clean, humidified air. (Each fill cycle lasts for approximately 9 minutes. At optimum flow rate, canisters will fill to a pressure of 10 to 20 psig in this time. This can be achieved by setting the flow control valve to the “4:30” position for 6 L canisters and to the “5:30 position for 3 L canisters.)

2.3 Programming for Automated Operation

- 2.3.1 On the Chron Trol module, press “1”. (This represents program 1, not circuit 1). No red light will be displayed. Program the system for automated operation as follows:
 - Press the “CIRCUIT” key
 - Press “3”
 - Press “ON”
 - **Enter the desired START time for commencement of cleaning cycles, using the numeric keypad. Enter the time as a four digit number (e.g. 0830 for 8:30 a.m.). The default time setting is “a.m.”. After entering the start time, if required, press the “AM/PM” key to specify a “p.m.” start time. A red dot in the lower right corner indicates a “p.m” setting.**
 - Press the “VARY” key, then enter the number **5500** using the numeric keypad (representing a time of 55 minutes and 00 seconds until the start of the following cycle).
 - Press “OFF”.
 - **Enter the desired END time for the first cleaning cycle. THE “OFF” TIME MUST BE SET 54 MINUTES LATER THAN THE “ON” TIME.** Ensure that the entered time reads “a.m.” or “p.m” (red dot), as appropriate.
 - Press the “VARY” key, then enter the number **5500** once more (this time representing automatic shifting of the END time for subsequent cycles.)

Title: Automated Cleaning of Sample Canisters		Copy No: ##
SOP No.: 7.01/1.5/S	Effective Date: September 11, 2013	Location: ###

- Press the “ENTER” key.
- Press “1”
- Press the “CYCLE” key.
- Press “0” (representing a cycle of less than one hour.)
- Press “ENTER”.
- Note that all of the above times are for cleaning 6 L canisters. They are also to be used when 3 L canisters are cleaned overnight or if 32-36 x 3 L are being cleaned. If 18 x 3 L canisters are to be cleaned during the day, the total time for each cycle is 40 minutes. Enter 4000 instead of 5500 with an end time of 39 minutes later than the “ON” time.
- Programming is now complete. **VERIFY THAT THE PROGRAM HAS BEEN ENTERED CORRECTLY BY PRESSING ALL PROGRAMMING KEYS IN THE SEQUENCE SPECIFIED ABOVE. RE-ENTER VALUES ONLY FOR ANY DISPLAYED TIME SETTINGS WHICH ARE INCORRECT. IF A TIME WAS INCORRECTLY ENTERED AS “A.M” (OR “P.M.), THE TIME MUST BE RE-ENTERED IN FULL WITH THE APPROPRIATE SETTING (I.E. SIMPLY PRESSING THE “AM/PM” KEY WILL NOT CORRECT THE ERROR).** Red lights for circuits 1 and 4 should be lit. The system will perform repeated cleaning cycles beginning at the specified start time. A ten minute evacuation stage using the diaphragm pump will be followed by a 35 minute (20 minutes for 3 L canisters) evacuation with the high vacuum pump. This is followed by a 9 minute canister filling stage. The total of these three stages is 54 minutes (or 39 minutes for the 3 L canisters). There is a one minute interval when all solenoid valves are closed, then the cycle is repeated. The one minute delay time is required because the end time of one cycle must be different from the start time of the next cycle.

2.4 Shut Down

2.4.1 While the cleaning cycle is in an evacuation stage using the high vacuum pump with the pressure gauge reading -30 in. Hg, **AND WITH AT LEAST 4 CLEANING CYCLES COMPLETED**, shut down the system as follows:

- Press “3”.

Title: Automated Cleaning of Sample Canisters		Copy No: ##
SOP No.: 7.01/1.5/S	Effective Date: September 11, 2013	Location: ###

- Press “OFF” (The solenoid valve to the vacuum pump will close).
- Press “1”
- Press “CIRCUIT”
- Press “0”
- Press “ENTER”
- Set relay timer for high vacuum pump to maximum and relay timer for diaphragm pump to minimum.
- Press “3”.
- Press “ON”.
- Turn off the oven heater and open the door. If this is not done, canisters will be too hot to handle without gloves when the evacuation is complete.
- Allow canisters to evacuate for 45 minutes.
- Close all canister valves.
- Press “3”.
- Press “OFF”.
- Manually turn off the high vacuum pump by means of the toggle switch at the back of the pump. **CAREFULLY LOWER THE DEWAR OF LIQUID NITROGEN FROM THE COLD TRAP.**
- On the Chron Trol module, press “4”.
- Press “OFF”.
- Disconnect the canisters from the manifold and install and tighten caps on all canisters. Attach labels with the applicable cleaned month and date highlighted to the tags on the canisters. Remove cleaned, evacuated canisters to storage shelves.

3. Power Failure

In the event of a power failure, the Chron Trol module will lock. Press “103” and “Enter” to unlock the unit. Note that the numbers will not appear on the display.

4. Revisions

- | | |
|-----------|--|
| Sep 2009: | Section 1: changed Section to Unit
Section 2.4: added procedure for further 45 minutes evacuation at end of cleaning cycles |
| Sep 2011 | Section 2.2.1: added requirement to ensure analyses completed
Section 2.2.2: added checking for proper valve position |

Title: Automated Cleaning of Sample Canisters		Copy No: ##
SOP No.: 7.01/1.5/S	Effective Date: September 11, 2013	Location: ###

Section 2.2.3: added reference to “day” or “night” cleaning
 Section 2.2.4: made water level more specific
 Section 2.2.5: added instruction for draining oil filter and filling pump
 Section 2.2.6: added reference to covering dewar
 Section 2.2.7: made high vacuum pump switch location generic
 Section 2.2.8 noted need to ensure that light “1” is lit
 Section 2.2.12: changed location of oven heater switch and added reference to temperature display
 Section 2.3.1: indicated timings for 3 L canisters for day and night cycles
 Section 2.4.1: moved instruction to open oven door and turn off heater up the list
 Section 3.0: added instruction for unlocking control module in event of power failure
 Deleted revisions prior to 2007

Aug 2013

Section 2.2.3 included provision for adding extra lines and mixing canister volumes
 Replaced references to using circuit “2” with circuit “3”
 Deleted revisions prior to 2009

Lead Reviewer: Gale Bryant
Title: Chemist, Air Toxics Unit

Approved By: Daniel Wang
Title: Senior Project Scientist, Air Toxics

THIS DOCUMENT MUST NOT BE PHOTOCOPIED

Any hard copies of this document missing any pages, or without a copy number and the initials of the Quality Systems Manager is uncontrolled. Any electronic copy of this document anywhere but on SharePoint is uncontrolled.

DO NOT USE THIS DOCUMENT FOR OPERATIONAL PURPOSES IF IT IS AN UNCONTROLLED COPY