California Environmental Protection Agency

Air Resources Board

STANDARD OPERATING PROCEDURE FOR CLEANING SUMMA POLISHED CANISTERS

SOP MLD020

Northern Laboratory Branch
Monitoring and Laboratory Division

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DISCLAIMER: Mention of any trade name or commercial product in this Standard Operating Procedure does not constitute endorsement or recommendation by the Air Resources Board (ARB). Specific brand names and instrument descriptions listed in the Standard Operating Procedure are for equipment used by ARB's laboratory. This method should be operated under supervision of trained personel.

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SOP MLD020

PROCEDURE FOR CLEANING SUMMA™ POLISHED CANISTERS

1 SCOPE

This document describes the method for cleaning and certifying SUMMA™ passivated canisters used for ambient sampling of volatile organic compounds (VOCs) for the Air Resources Board's Toxic Air Contaminant Program.

2 SUMMARY OF METHOD

Canisters must be cleaned and certified prior to sampling. Using the laboratory canister cleaning apparatus, up to twelve canisters are connected to the device and subjected to a series of evacuations and pressurization cycles while exposed to above ambient temperatures in an oven. Typically, the canisters are evacuated to less than 0.10 torr (0.10 mm Hg) and pressurized to approximately 10 psig during the cycle while being heated to at least 55°C. Ultra high pure humidified nitrogen or air is used during the pressurization cycle. Canisters are checked for cleanliness then released for sampling.

3 INTERFERENCES AND LIMITATIONS

Canisters containing high VOC concentrations may be difficult to clean and may require more than one cleaning session to meet specified cleanliness criteria. Canister pressure, as indicated by the laboratory, must not differ by more than 2psi when the canister is installed for sampling. A change in pressure indicates a leak and the canister may not be used for sampling. Microleaks, that are not easily detected, may introduce contamination. Therefore, canisters that have not been used for sampling within 60 days from cleaning must be recertified.

4 APPARATUS

4.1. Safety glasses.

4.2. Cryogenic gloves.

4.3. 9/16 inch compound wrench.

4.4. Laboratory canister cleaning apparatus consisting of a vacuum pump, capable of reaching 0.05 torr connected to a vacuum manifold mounted in

a temperature controlled oven or enclosure. If an oil filled pump is used there must be a trap to prevent oil mist back streaming. Additional valves and gas controls are included to regulate the cleaning cycle. A humidification device is connected in line with the compressed gas used for canister pressurization.

5 MATERIALS

5.1. Liquid nitrogen.

5.2. Compressed grade 5 ultra-pure nitrogen or zero air.

5.3. Organic free water.

5.4. Vacuum pump oil.

6 PRECAUTIONS AND SAFETY

6.1. Use safety glasses and cryogenic gloves when handling liquid nitrogen.

6.2. Do not pressurize the canisters to more than 30 psi.

6.3. Do not over tighten the valves on the canisters. Hand tighten only.

6.4. If a liquid nitrogen dewar is used, keep it at least ½ filled whenever the vacuum pump is running to prevent back streaming.

6.5. Check the vacuum pump oil level prior to operation. Add or change oil when the level gets near the low indicator on the sight glass or if the oil becomes discolored.

6.6. All canisters must be vented in the safety fume hood before cleaning.

6.7. The nitrogen or zero air cylinder must not be used if the cylinder pressure drops below 500 psi. Replace it with a full cylinder. If a compressed gas system is used, its purity shall be monitored each quarter by filling a canister for analysis as a contamination check.

6.8. The compressed gas cylinder output pressure to the canisters should not exceed 40 psi. Check regulator pressures prior to operation.

6.9. Check the humidification device for proper water levels.

the canister for further leak testing. (Do not remove it at this time.) Close canister sampling valves and allow oven and canisters to cool.

7.1.11 At least one canister from each cleaning batch must be analyzed to verify cleanliness for each analytical method. Canisters that have been previously used for this contamination check are marked on the bottom of the can. This allows for rotation of the canisters and avoids using the same canister for this check. The canister chosen for the contamination check should be selected in this order: 1) one that was shown to contain a high concentration sample, 2) one that contained calibration or control standards, or 3) a randomly selected canister that has not previously been used as a contamination check. Record which canisters will be used for contamination checks in the logbook. These will require pressurization as directed in the work instructions.

7.1.12 Analyze all canisters in a batch twice per year to verify that all cans in the batch have been cleaned consistently.

7.2 Pressurize evacuated canisters as directed in the work instructions.

8 QUALITY CONTROL/CANISTER CERTIFICATION

8.1. Each batch of cleaned canisters must be held until analyses of contamination check canisters are processed. The canisters that are marked as contamination checks are analyzed for all target compounds. There may be one canister per batch for multiple analytical methods or one canister for each analytical method. After appropriate analyses are complete the associated canisters are released for sampling if they meet cleanliness criteria. If the analytical results are above the limit of detection (LOD) for any target compound all canisters in the batch must be re-cleaned.

8.2. Canisters that have been used for contamination checks are marked with a color code to indicate the analytical method performed. Canisters should not be used repeatedly as contamination checks on the same method. The goal is to rotate canisters used for contamination checks so that all canisters get used for this purpose.

8.3. Each canister is held at -30 Hg vacuum and pressure is visually checked before it is shipped out to field sites for sampling. If vacuum is not at -30 Hg the canister is not used for sampling. The canister is leak tested and repaired, if possible.

6.10. Canisters containing samples with high concentrations of target compounds may require additional purging and cleaning.

7 PROCEDURES

Refer to the manual supplied by the manufacturer for the operation of the laboratory canister cleaning system.

7.1. CANISTER CLEANING PROCEDURE

7.1.1. Turn on the vacuum pump and adjust the compressed gas pressures to manufacturer recommendations. Verify there is adequate water in the humidification chamber. Turn on the canister cleaning apparatus and open the gas supply lines.

7.1.2. Slowly fill the dewar with liquid nitrogen to approximately two thirds of its volume.

7.1.3. Make certain all canisters to be attached to the cleaning apparatus are vented by fully opening their valves.

7.1.4. Any canisters that contained calibration or control standards or high sample concentrations must be purged at least five times with nitrogen after venting and prior to cleaning.

7.1.5. Record the canister numbers and cleaning date into the cleaning logbook.

7.1.6. Connect the canisters to the vacuum manifold in the oven with the canister sampling valves closed. Make certain any unused manifold positions are plugged.

7.1.7. Perform a leak check procedure on the attached canisters as directed in the work instructions.

7.1.8. Open all canister valves.

7.1.9. Close and latch the oven door. Verify the correct oven temperature is set.

7.1.10. Start the cleaning process. When the cleaning process is complete open the oven door. Confirm that all canister pressure gauges are at -30" Hg. If a canister gauge reads anything other than -30" Hg mark

SOP MLD 020 Revision History

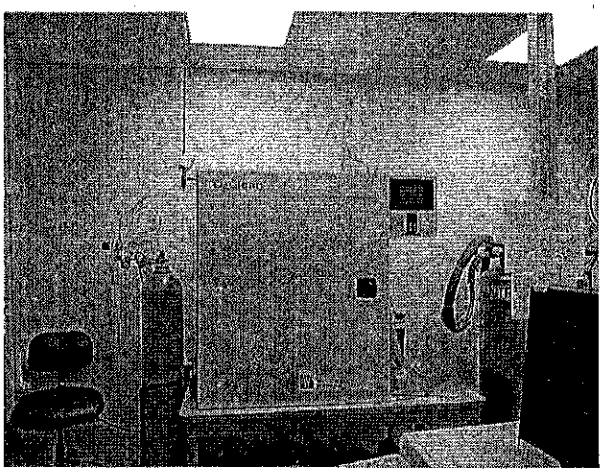
Revision Number	Revision Date	Effective Date	Revisions Made
1.0	unknown		
2.1	unknown	June 1, 1989	
3.0	April 4, 1994	May 1, 1991	Automation by using solenoid valves in place of manually operated valves.
3.1	unknown		
3.2	Oct 29, 1999	March 1, 1997	
3.3		Jan 2, 2002	
4.0		April 1, 2013	Created general instructions and QC requirements for canister cleaning. Referenced work instructions for specifics on operating TO-Clean can cleaning system.

8.4. Canisters are labeled with the cleaning date and must be used for sampling within 60 days. If not used within 60 days the canisters must be pressure tested with a calibrated gauge to verify stable pressure. If the pressure has changed by more than 2 psi the canister must be leak tested and repaired, if possible.

9 DATA HANDLING

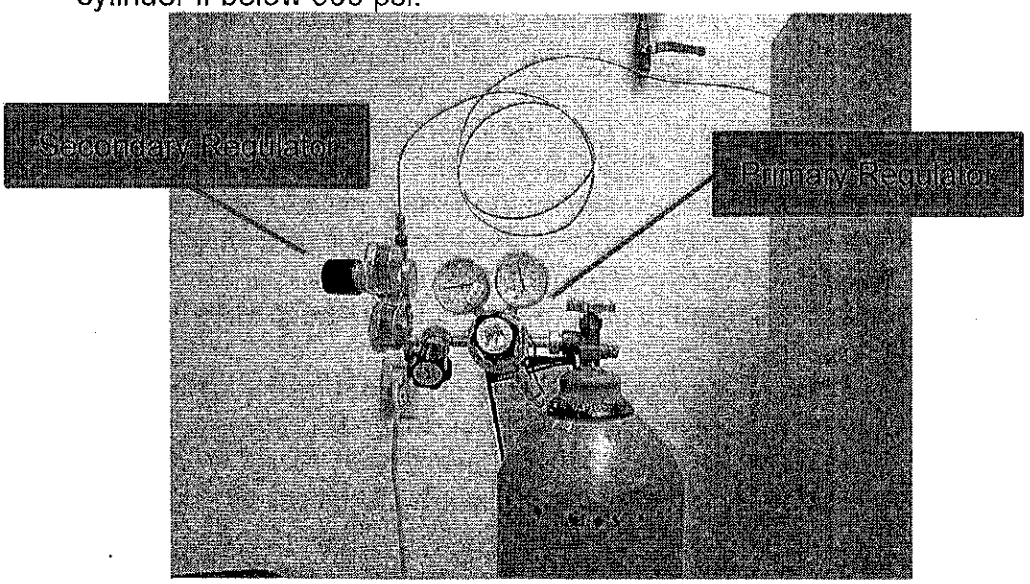
Contamination check results are recorded in the Laboratory Information Management System (LIMS) and "pass" or "fail" is noted in the logbook for each batch. LIMS allows for tracking canister cleaning and use.

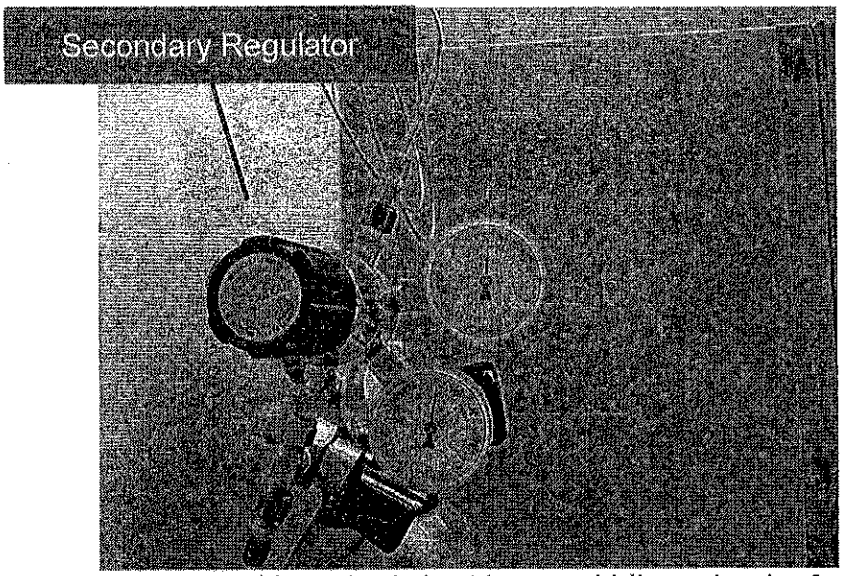
WI-MLD020-Rev.1
Work Instructions for TO-Clean Canister Cleaning System



Start up

1. Check supply of compressed gas (typically nitrogen or zero air), liquid nitrogen and water (humidifier should be at least 1/4 full with organic free water – do not fill past maximum fill line).
2. Check oil level of vacuum pump. Add oil to at least the ½ level in sight glass. Turn on the vacuum pump (power strip switch at left rear of table).
3. Ensure the compressed gas cylinder valve is open. Primary regulator pressure should be between 55 and 60 psig, secondary pressure regulator set near 40 psig. Primary regulator controls pressure to actuator valve, secondary regulator controls pressure to fill canisters. Record cylinder pressure in logbook (right side gauge on primary regulator). Do not use cylinder if below 500 psi.





4. Turn on the TO-Clean (switch at lower middle on back of electronics enclosure).
5. Open the compressed gas toggle switch in line with the humidifier.
6. Fill the dewar 2/3 full of liquid nitrogen. (Wear personal protective equipment: safety glasses and cryogenic gloves.)
7. Gather the canisters to be cleaned. Choose those needed for contamination checks for each method and mark on the bottom with a color indicating analytical method.
8. Exhaust all canisters under a safety fume hood prior to placing in the oven.
9. If canister contained calibration or control standards or had high concentrations of any target compound it must be filled and purged 5 times with nitrogen under the safety fume hood prior to placing in the oven.
10. Place canisters in oven and attach the flexible lines to each making certain the fitting is correctly attached and tighten using 9/16 inch wrench. DO NOT OVERTIGHTEN. As each canister is attached, be sure the canister valve is closed. It is convenient to place the canisters to be used for contamination checks in the front row of the upper shelf of the oven for later pressurization.
11. Leak test the canisters by running the Diagnostics Leak Test (to 30 psig for at least 1 minute – default entry). From the initial screen, press *<DIAGNOSTICS> <LEAK TEST> <START>*
12. If the leak test does not pass – recheck all connections to the canisters and rerun the leak test.
13. If the leak test passes wait for audible click then press <DONE>. Load the 5CYCLE program. From the Leak Test FINISHED screen, press *<CANCEL> <CLEAN> <LOAD METHOD> <up/down arrow> to <5CYCLE> <LOAD> <CLEAN>* (open each canister valve as the program starts to evacuate the canisters).

14. Close and latch the oven door and check that the oven temperature controller is set for about 60 degrees C. Use the up/down arrow keys on the controller to adjust the setting.
15. While cans are being cleaned list canister numbers in logbook noting those to be used as contamination checks and for which method (*= MLD070, **= MLD066 and ***= MLD058).
16. Assign one barcoded LIMS (Laboratory Information Management System) contamination check identification number to all canisters cleaned in the batch. Record this number in the logbook to be used for tracking. Log the canisters into LIMS using a barcode reader, if available.
17. The screen will read FINISHED when cleaning is complete (about 3 hours).
18. Press the *<DONE>* button, open the oven door and close all the canister valves. Let oven cool down to where the canisters may be comfortably handled (about 30 min). Go to Valve control screen and turn the vacuum valve on and then off and *<CANCEL>*.

To fill contamination check canisters

19. Use the Diagnostics-Leak Test (see 11) to fill the selected canisters for the contamination check. Set Leak Test to 10 psi for 2 minutes, start the leak test and quickly open the valves on the canisters to be used for contamination checks. When the pressure reaches 10 psig and the Leak Test timer starts, close the canister valves and press *<CANCEL>*.
20. Remove the canisters from oven. Fill out identification tag(s) for contamination check canister(s). Add a colored tag to each contamination check and to one additional canister in the cleaned batch for easy visual identification.
21. Distribute contamination check canisters to the appropriate analyst and place the other remaining canisters together on a shelf in the "waiting for contamination check" area. When analyses are complete and verified to meet cleanliness criteria, all canisters in the batch may be released for sampling.

To evacuate canisters

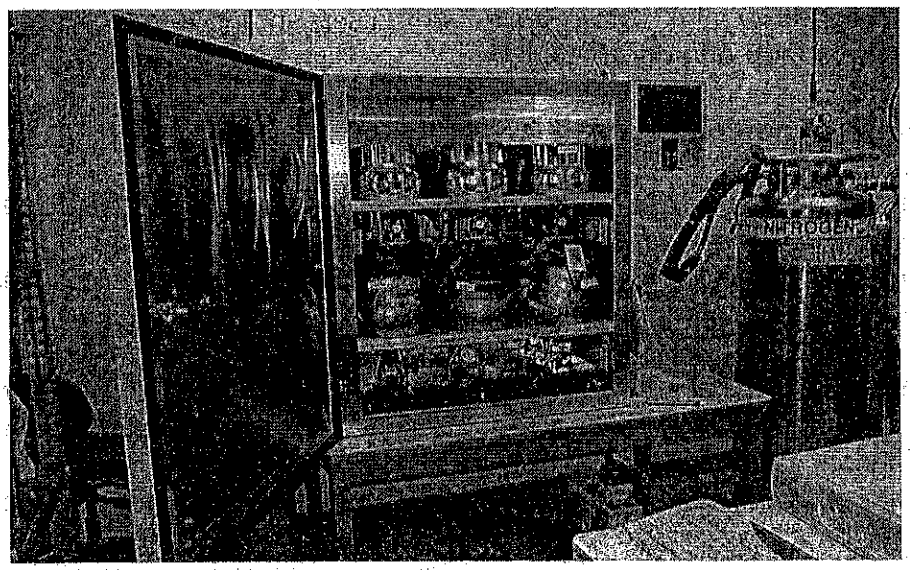
22. Place canisters in oven and attach the flexible lines to each making certain the fitting is correctly attached and tight. DO NOT OVERTIGHTEN. As each canister is attached, be sure the canister valve is closed.
23. Leak test the canisters by running the Diagnostics Leak Test (to 30 psig for at least 1 minute – default entry). From the initial screen, press *<DIAGNOSTICS> <LEAK TEST> <START>*
24. If the leak test does not pass – recheck all connections to the canisters and rerun the leak test.
25. If the leak test passes load the EVAC program and open each canister valve as the program starts to evacuate. From the Leak Test FINISHED screen,

press *<CANCEL> <CANCEL> <CLEAN> <LOAD METHOD> <up/down arrow> to <EVAC> <LOAD> <CLEAN>.*

26. The oven doors can stay open while evacuating as no heat is required. When the program is finished, close the valves on the canisters and remove them from the oven.

Shut down

27. Cap all the lines inside the oven – finger tight is sufficient.
28. Close the compressed gas toggle switch in line with the humidifier.
29. Set TO-Clean power switch to the off position.
30. Turn off power to the pump (at power strip).



Methods for TO-Clean

5CYCLE Program

See TO-CLEAN manual for instructions on programming.
Program 5 cycles of vacuum soak at 0.5 Torr for 3 minutes followed by a pressure soak at 10 psig for 3 minutes.
Final vacuum is set for 0.1 Torr.

EVAC Program

See TO-CLEAN manual for instructions on programming.
Program to vacuum to 0.1 Torr.

A02 MLD020

State of California
California Environmental Protection Agency
Air Resources Board
MLD/QMS-066 (NEW 4/14)

QUALITY MANAGEMENT DOCUMENT ADDENDUM

Section 1. ARB Document
☐ Quality Management Plan (QMP)
☐ Quality Assurance Project Plan (QAPP)
☒ Standard Operating Procedure (SOP)

Section 2. Information		
Submitter Name:	Judy Hodgkins	
Submitter Signature/Date:		

Section 3. Document Title *(specify exact title, revision #, and date of ARB Document(s) that your District proposes to modify)*	Date
SOP MLD020 Revision Number 4.0	April 1, 2013

Section 4. Revision(s)
(specify exact section(s), page number(s) and language in existing ARB document that will be modified and then specify modification (including any spreadsheets or forms).

Section 7.1.11, page 4, states "At least one canister from each cleaning batch must be analyzed to verify cleanliness for each analytical method. Canisters that have been previously used for this contamination check are marked on the bottom of the can. This allows for rotation of the canisters and avoids using the same canister for this check. The canister chosen for the contamination check should be selected in this order: 1) one that was shown to contain a high concentration sample, 2) one that contained calibration or control standards, or 3) a randomly selected canister that has not previously been used as a contamination check. Record which canisters will be used for contamination checks in the logbook. These will require pressurization as directed in the work instructions."

ARB modification to section 7.1.11 - Delete and change to: "All canisters in each cleaning batch will be tested for all target compounds for contamination. Canisters will require pressurization as directed in the work instructions."

Section 7.1.12, page 4, states "Analyze all canisters in a batch twice per year to verify that all cans in the batch have been cleaned consistently.

ARB modification to Section 7.1.12 – Delete 7.1.12

Section 8.1, page 4, states "Each batch of cleaned canisters must be held until analyses of contamination check canisters are processed. The canisters that are marked as contamination checks are analyzed for all target compounds. There may be one canister per batch for multiple analytical methods or one canister for each analytical method. After appropriate analyses are complete the associated canisters are released for sampling if they meet cleanliness criteria. If the analytical results are above the limit of detection (LOD) for any target compound all canisters in the batch must be re-cleaned."

ARB modification to Section 8.1 Delete and change to: "After each canister is checked for contamination by each current analytical method it may be released for sampling if it met the cleanliness criteria. If the analytical results are above the limit of detection (LOD) according to the applicable analytical method SOP for any target compound the canister must be re-cleaned and retested prior to release."

Section 8.2, page 4, states "Canisters that have been used for contamination checks are marked with a color code to indicate the analytical method performed. Canisters should not be used repeatedly as contamination checks on the same method. The goal is to rotate canisters used for contamination checks so that all canisters get used for this purpose."

ARB modification to 8.2 – Delete 8.2

Section 5. Justification for Deviation(s) *(provide explanation of why modification(s) to existing ARB document is necessary)*
OLS studies have shown that there are variations in the canisters and a single canister is not always representative of the cleanliness of all canisters in the batch. Therefore all canisters must be analyzed to confirm cleanliness criteria have been met.

Section 6. Attachment(s) ☐ *(specify attachment titles and number of pages, include modified spreadsheets or forms)*	**# of Pages**
WI-MLD020 Rev. 2 Work instructions modified to match changes in this addendum.	**5**

Section 7. ARB Approval		
Name/Phone Number:	Patrick Rainey	916 327-4756
Title:	Manager, Quality Management Section	
Signature/Date:		5/14/15
Addendum Number	A02 MLD020	

Completed form must be scanned/emailed or mailed to:

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