





CALIFORNIA

AIR RESOURCES BOARD

STANDARD OPERATING PROCEDURE FOR CLEANING STAINLESS STEEL AIR CANISTERS

MLD020
Revision 4.1

Northern Laboratory Branch
Monitoring and Laboratory Division

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STANDARD OPERATING PROCEDURE FOR CLEANING STAINLESS STEEL AIR CANISTERS

1. INTRODUCTION AND SCOPE

The California Air Resources Board (CARB) established the statewide comprehensive air toxics program in the early 1980s. The Toxic Air Contaminant Identification and Control Act (AB 1807, Tanner 1983) created California's program to reduce exposure to air toxics. In 1985, CARB established a twenty-station toxic monitoring network within major urban areas to provide data to determine the average annual concentrations of toxic air contaminants as input to the identification process and to assess the effectiveness of controls. Laboratory analyses of ambient air samples are conducted using validated methods. Ambient samples are collected in electropolished and silica-lined stainless steel canisters and are analyzed in the laboratory by gas chromatography/mass spectrometry (GC/MS) for volatile organic compounds (VOCs), aromatic and halogenated compounds, oxygenated and nitrile hydrocarbons, and greenhouse gases for the toxics network and special studies.

2. SUMMARY OF METHOD

Air canisters must be cleaned and certified prior to sampling. Using the laboratory canister cleaning apparatus such as the Wasson-ECE TO-Clean, up to twelve canisters are connected to the device and subjected to a series of evacuation and pressurization cycles while exposed to elevated ambient temperatures in an oven. Typically, the canisters are evacuated to less than 0.100 torr and pressurized to approximately 10 pounds per square inch gauge (psig) during the cycle while being heated to at least 60 degrees Celsius (°C). Ultra-High Purity (UHP) humidified nitrogen or air is used during the pressurization cycle. Canisters are checked for cleanliness by the GC/MS, verified to meet quality control (QC) criteria, and then released for sampling. MLD020 Standard Operating Procedure (SOP) Revision 4.1 should be used in conjunction with the current Work Instructions (WI) version.

3. ACRONYMS

Acronym or Term	Definition
°C	Degrees Celsius
CARB	California Air Resources Board
GC/MS	Gas Chromatography/Mass Spectrometry
inHg	Inches of Mercury
LIMS	Laboratory Information Management System
LOQ	Limit of Quantitation
MDL	Method Detection Limit
mTorr	Millitorr
NATTS	National Air Toxics Trends Station
NLB	Northern Laboratory Branch
PPBV	Parts Per Billion By Volume
PSIG	Pounds Per Square Inch Gauge
QC	Quality Control
SOP	Standard Operating Procedure
TAD	Technical Assistance Document
UHP	Ultra-High Purity
VOCs	Volatile Organic Compounds
WI	Work Instructions

4. 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 vacuum, as indicated by the laboratory, must not differ by more than 2 inches of Mercury (inHg) when the canister is installed for sampling. A change in vacuum indicates a leak and the canister may not be used for sampling. Microleaks, that are not easily detected, may introduce contamination.

5. PERSONNEL QUALIFICATIONS AND TRAINING

- 5.1. Prior to performing this procedure, new personnel must be trained by staff with expert knowledge of this procedure. Personnel must be trained to understand the program's requirements per any applicable State and federal regulations and guidance, and this SOP. Personnel will also be trained on how to safely and properly operate the equipment needed to perform the procedure, the quality assurance components, and

Laboratory Information Management System (LIMS) functionality pertaining to the program.

- 5.2. Personnel should provide an initial demonstration of capability prior to performing this procedure on real-world samples to obtain data for record.
- 5.3. Training will be documented and maintained by the laboratory supervisor.

6. SAFETY REQUIREMENTS

- 6.1. All personnel must follow the general health and safety requirements found in NLB's Chemical Hygiene Plan.
- 6.2. Use safety glasses and cryogenic gloves when handling liquid nitrogen.
- 6.3. Do not pressurize canisters greater than 30 pounds per square inch gauge (psig).
- 6.4. Refer to safe handling practices regarding compressed gases when moving and installing cylinders.
- 6.5. Check regulator pressures prior to operation. The compressed gas cylinder output pressure to the canisters should not exceed 40 psig.
- 6.6. Avoid touching surfaces inside oven until cool.

7. HAZARDOUS WASTE

No hazardous waste is generated by this method.

8. EQUIPMENT AND SUPPLIES

- 8.1. 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 (Figure 1). If an oil filled pump is used there must be a trap installed to prevent oil mist from backstreaming. Additional valves and gas controls are included to regulate the cleaning cycle. A humidification chamber is connected in line with the compressed gas used for canister pressurization.

- 8.2. Compressed UHP nitrogen or air.
- 8.3. Liquid nitrogen.
- 8.4. Deionized water.
- 8.5. Vacuum pump oil.
- 8.6. Electropolished/silica-lined stainless steel canisters.
- 8.7. 7/16", 1/2", and 9/16" compound wrench.
- 8.8. Crescent wrench.
- 8.9. 225mL Erlenmeyer flask.
- 8.10. 225mL graduated cylinder.
- 8.11. Fume hood.



Figure 1. TO-Clean Canister Cleaning System

9. PROCEDURES

- 9.1. Verify there is sufficient oil in the vacuum pump, adequate water in the humidification chamber, and greater than 500 psig in the nitrogen cylinder.
 - 9.1.1. The nitrogen or zero air cylinder must not be used if the cylinder pressure drops below 500 psig. 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.
- 9.2. Turn on the vacuum pump and adjust the compressed gas pressures to manufacturer recommendations.
 - 9.2.1 Check the vacuum pump oil level prior to operation. Add oil when the level drops below 50% or as indicated on the sight glass. Change the oil if it becomes discolored.
- 9.3. Open the toggle valve that is connected in between the humidification chamber and air/nitrogen supply line.
- 9.4. Turn on the canister cleaning apparatus.
- 9.5. Carefully fill the dewar with liquid nitrogen to approximately 75% of its volume.
 - 9.5.1. If a liquid nitrogen dewar is used, keep it filled to at least 25% whenever the vacuum pump is running to prevent backstreaming.
- 9.6. Record the date, TO-Clean identification number, nitrogen pressure, canister numbers, and identify which canister(s) will be used for contamination checks into the canister-cleaning logbook. Assign a LIMS contamination check identification number.
 - 9.6.1. All canisters must be vented in the safety fume hood before cleaning. Any canister that contains calibration, control, or high sample concentrations must be evacuated and pressurized at least five times in the safety fume hood using the custom manifold and vacuum pump. Canisters containing samples with high concentrations of target compounds may require an additional set of five evacuations and pressurizations.
- 9.7. Connect the vented canisters to the vacuum manifold in the oven, being sure the canister valves are closed. Make certain any unused manifold ports are plugged. Do not overtighten canister valves. Hand tighten only.

- 9.8. Perform a leak check as directed in the work instructions.
- 9.9. Close the oven door and allow the temperature to equilibrate to 60°C.
- 9.10. Open the oven door, start the programmed cleaning process, open all of the canister valves, and close the oven door.
- 9.11. When the programmed cleaning process is complete, open the oven door and allow the canisters to cool before handling.
- 9.12. Depending on how many canisters are cleaned, one or two of them will need to be filled with nitrogen or air and analyzed to verify cleanliness.
- 9.13. Close all of the canister valves that will not be used for contamination checks.
- 9.14. Fill the selected canister(s) for cleanliness verification with nitrogen to approximately 15 psig.
- 9.15. Close the canister(s) valve(s).
- 9.16. Shutdown the canister cleaning system as directed in the work instructions.
- 9.17. Place canisters in designated areas in the laboratory for analysis or holding. Identify the canisters designated to be used as contamination checks.
- 9.18. Canisters must sit at least 24 hours before being analyzed to equilibrate to room temperature.
- 9.19. Designated contamination check canisters are analyzed for all VOC and greenhouse gas methods and the results are compared to QC criteria.
- 9.20. If canisters meet QC criteria, evacuate the canisters in the TO-Clean as directed in the work instructions.
- 9.21. If canisters do not meet QC criteria, the entire batch will need to be re-cleaned.

10. TROUBLESHOOTING AND MAINTENANCE

- 10.1 See sections 6.0 to 8.0 in MLD020 WI for details.

11. QUALITY CONTROL

A summary of quality control for cleaning canisters is shown in Table 1.

- 11.1. The verification for canister cleanliness is to analyze canisters for all target compounds and to verify no concentration is $> 3x$ method detection limit (MDL) as per National Air Toxics Trends Station (NATTS) Technical Assistance Document (TAD) Revision 3 (FINAL October 2016). See pages 11 and 12 of MLD020 Work Instructions.
- 11.2. Each canister is held at 30 inHg and the vacuum is visually checked before it is shipped out to the field for sampling. If the gauge reads below 30 inHg, the canisters vacuum is checked with a calibrated gauge. If the canisters calibrated gauge reads 29.5 inHg or higher, the canister can still be used. Any canister with a calibrated gauge reading below 29.5 inHg is not used and is checked for leaks.
- 11.3. Canisters are labeled with an expiration date and must be used for sampling within 60 days of cleaning. If not used within 60 days, the canisters must be tested using a calibrated gauge to verify the vacuum. If the vacuum has changed by more than 0.5 inHg, the canister must be leak tested; otherwise, a new 30-day expiration date is assigned to the canister.
- 11.4. New canisters are received at the lab under vacuum. The vacuum shall be tested to determine if a leak is present by recording any changes over a two-week period. If the canister vacuum changes by more than 1 inHg during the two-week period, notify the manufacturer for replacement. If the canisters pass the two-week period, they can be placed in the TO-Clean for cleaning. Each canister receives its own LIMS number and is analyzed for all VOC and greenhouse gas methods.
- 11.5. If cleaning 10 canisters, one (1) canister is selected for contamination testing by analyzing for all methods with which the canister may be used. If the selected canister fails the contamination check, the entire batch needs to be re-cleaned. If cleaning 12 canisters, two (2) canisters are selected for contamination testing by analyzing for all methods with which the canisters may be used. If one (1) canister fails the contamination check, the entire batch needs to be re-cleaned. If both canisters fail the contamination check, this may be by chance or more likely contamination to the TO-Clean system. Investigate the cause and re-clean the entire batch.

Table 1. QC Reference

QC TYPE	FREQUENCY	CRITERIA	CORRECTIVE ACTION
Vacuum Verification	All canisters prior to shipping or use in making standards	29.5 inHg and higher	If gauge reads below 29.5 inHg, vacuum is checked with calibrated gauge. Canisters with vacuum below 29.5 inHg are not used.
Expiration Date	All canisters prior to shipping or use in making standards	Must be used 60 days after cleaning	If not used within 60 days, verify vacuum using calibrated gauge. If vacuum has changed by 0.5 inHg, the canister must be leak tested. If vacuum has not changed by 0.5 inHg, a new 30-day expiration date is assigned.
New Canisters	Every new canister	Two-week vacuum test	Canister vacuum is tested over a two-week period. If canister vacuum changes by more than 1 inHg, notify the manufacturer for replacement.
Contamination Checks	One (1) canister per batch of 10 or two (2) canisters per batch of 12	< 3x MDL	If one (1) canister out of 10/12 fails the contamination check, the entire batch of canisters needs to be re-cleaned. If two (2) canisters out of 12 fail the contamination check, investigate and re-clean entire batch.

12. CANISTER DATA MANAGEMENT

- 12.1. Barcodes associated with canisters are scanned into LIMS to indicate that the canister was used as a contamination check.
- 12.2. Data management consists of canisters and contamination check information. Program and maintenance notebooks and/or logbooks are to be kept with the cleaning apparatus at all times. Contamination check results are recorded in the canister logbook as pass or fail. The concentrations of the target compounds in canisters that fail contamination checks are recorded and the results updated in the sample logbook.

13. REFERENCES

- 13.1. California Air Resources Board MLD Northern Laboratory Branch: Final Chemical Hygiene Plan for Northern Laboratory Branch, 1927 13th Street, 1900 14th Street, March 2016.
- 13.2. California Air Resources Board MLD Northern Laboratory Branch: Laboratory Quality Control Manual, Revision 4.0, September 17, 2018.
<https://ww2.arb.ca.gov/sites/default/files/2018-10/nlbqcm.pdf>
- 13.3. California Air Toxics Program – Background, December 13, 2017.
<https://www.arb.ca.gov/toxics/background.htm>
- 13.4. Restek, Wasson-ECE Instrumentation TO-Clean Manual, Version 4.5.
https://www.restek.com/pdfs/restek_to_clean_manual.pdf
- 13.5. Standard Operating Procedure, Review Checklist, ARB QMB QMS, MLD/QMS-065, Revision 03/13.
<http://www.arb.ca.gov/aaqm/qa/pqao/sop/sopchecklist.pdf>
- 13.6. Technical Assistance Document for the National Air Toxics Trends Stations Program, October 2016.
https://www3.epa.gov/ttnamti1/files/ambient/airtox/NATTS%20TAD%20Revision%203_FINAL%20October%202016.pdf
- 13.7. Toxics Air Contaminants Monitoring, Laboratory Analysis, June 9, 2016.
<https://www.arb.ca.gov/aaqm/toxics.htm>

14. ATTACHMENT

- 14.1 MLD020 Work Instructions Revision No. 4.1 (2018).

15. REVISION HISTORY

REVISION NUMBER	EFFECTIVE DATE	UPDATED REVISION
1.0	May 1, 1989	
2.1	June 1, 1989	1. Added figures
3.0	May 1, 1991	1. Automation by using solenoid valves in place of manually operated valves. 2. Updated figures.
3.2	October 29, 1999	1. Added conditioner controller operating instructions. 2. Updated figures and schematics.
3.3	January 2, 2002	
4.0	April 1, 2013	1. Created general instructions and QC requirements for canister cleaning. 2. Referenced work instructions for specifics on operating TO-Clean can cleaning system.
	May 14, 2015 (A02 MLD020)	3. Added addendums relating to contamination checks and required that all canisters in each cleaning batch be tested for target compounds for contamination.
4.1	November 26, 2018	1. Updated per MLD076 Revision 0.0 SOP for Preparation of NLB's SOPs. 2. Section 7.1.11 is now Section 11.5. Approved addendum removed and updated to NATTS TAD (October 2016) Section 4.2.4.2.4 changes that allowed no less than one out of every ten canisters be verified for canister cleanliness. 3. Section 7.1.12 has been removed per approved addendum. 4. Section 8.1 is now Section 11.1. 5. Section 8.2 has been deleted per approved addendum. 6. Added QC Table to section 11. 7. Added section 11.4 for new canisters. 8. Added references section.

WI-MLD020-Rev. 3.0 Work Instructions for TO-Clean Canister Cleaning System



Figure 1. TO-Clean Canister Cleaning System

1.0 START-UP

- 1.1. Collect up to twelve (12) canisters.
 - 1.1.1. Ensure all canisters are vented in the fume hood before cleaning.
 - 1.1.2. Ensure that calibration, control, and high concentration canisters have been purged/evacuated five (5) times in the fume hood.
- 1.2. In the logbook, record the date, TO-Clean identification number, nitrogen pressure, canister number, and identify which canister(s) will be used for contamination checks.
- 1.3. Assign a Laboratory Information Management System (LIMS) contamination check identification number.
 - 1.3.1. If cleaning ten (10) canisters, one (1) canister is selected for contamination testing by analyzing for all methods with which the

canisters may be used. If cleaning twelve (12), two (2) canisters will be chosen for analyses.

- 1.3.2. The first LIMS number will represent the first ten (10) canisters and the second LIMS number will represent canisters eleven (11) and twelve (12).
- 1.4. Place a LIMS number sticker next to each selected canister number in the logbook.
- 1.5. Fill out a corresponding LIMS tag and place one into each of the selected canisters' holder.
- 1.6. Place like-colored tags in each of the canisters' holder to identify those cleaned together in a single batch.

2.0 CLEANING

- 2.1. Ensure the nitrogen gas cylinder is open. The primary regulator pressure should be maintained between 55 and 60 psig. The secondary pressure regulator should be set near 40 psig (Figure 2). The primary regulator controls the pressure to the actuator valve and the secondary regulator controls the pressure, which fills the canisters. Note: Check the nitrogen gas cylinder pressure. Do not use the nitrogen gas cylinder if gauge reading is below 500 psig.

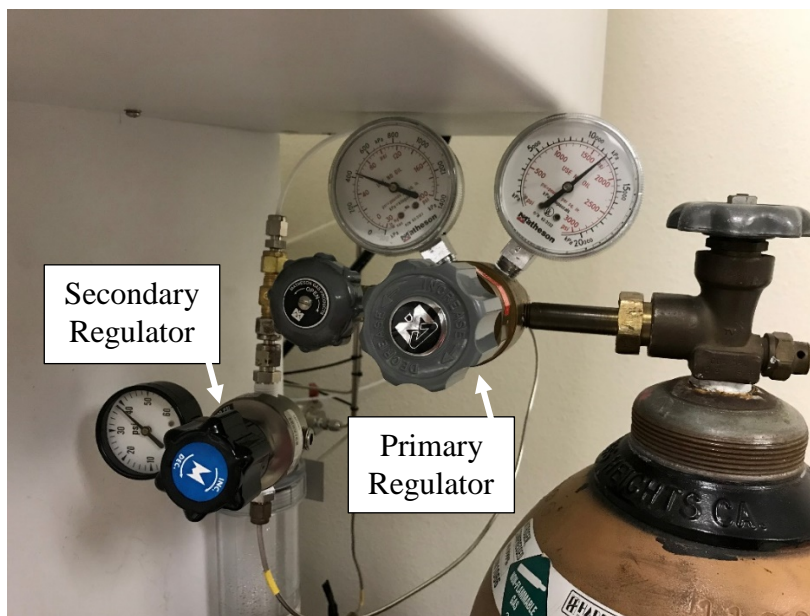


Figure 2. Nitrogen Tank and regulators

- 2.2. Check that the vacuum pump oil-level is between the MAX and MIN level marks on the bezel of the sight-glass (Figure 3).

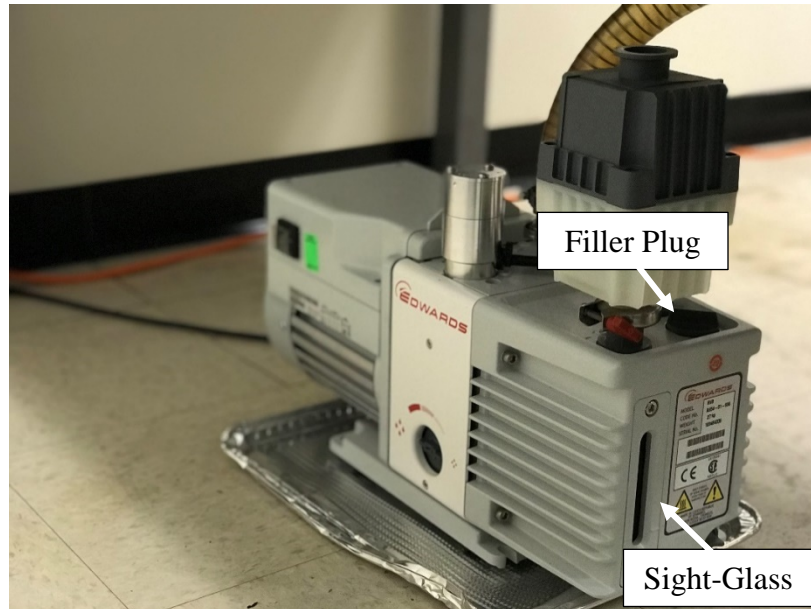


Figure 3. Vacuum Pump

- 2.2.1. If the oil-level is near to or below the MIN level mark, remove one of the filler-plugs and pour more oil into the reservoir until the oil reaches the MAX level mark.
- 2.3. Turn on TO-Clean by flipping the switch on the back of the unit.
- 2.4. Turn on the vacuum pump.
 - 2.4.1. TO-1: Power strip is located to the left of the door.
 - 2.4.2. TO-2: Use switch located on the vacuum pump.
- 2.5. Leave oven door open.
- 2.6. Wearing safety gloves and glasses, fill the dewar to 75% with liquid nitrogen (Figure 4).



Figure 4. Liquid Nitrogen Dewar

- 2.7. Place canisters in the oven starting with the rear bottom rack working your way up from the back to the front on each rack.
 - 2.7.1. When seating canisters on racks, ensure canister valves are facing forward for easier access.
 - 2.7.2. Place the canister(s) used for contamination testing on the top front rack.
 - 2.7.3. Attach the flexible lines to each canister and tighten by hand. Tighten with wrench until there is resistance. **DO NOT OVERTIGHTEN.**
- 2.8. Run Diagnostics Leak Test for one (1) minute at 30 psig.
 - 2.8.1. From the initial screen, press <DIAGNOSTICS> <LEAK TEST> <START>.
 - 2.8.2. If leak test fails, recheck all connections to the canisters and rerun the leak test.
 - 2.8.3. If leak test fails again, the U-trap may need to be emptied. See Cleaning U-trap in Section 6.1 of WI for details.
- 2.9. Close the oven door and let the temperature ramp up and equilibrate to 60°C. This may take up to 30 minutes.

2.10. Run 6CYCLE Program to clean canisters.

2.10.1. From the leak test FINISHED screen, press <CANCEL> <CANCEL> <CLEAN> <LOAD METHOD> <up/down arrow> to <6CYCLE> <LOAD> <CLEAN>.

2.10.2. Open each canister valve as the program starts the evacuation process.

2.10.3. Close the oven door.

2.11. The cleaning process will take between 3-4 hours and the screen will read FINISHED when complete.

2.12. Press <DONE> and open the oven door. Allow the canisters to cool down to room temperature before handling.

3.0 FILLING CANISTERS

3.1. Each representative canister needs to be filled with nitrogen to check for cleanliness and to check for system contamination.

3.2. Close the valves on the canisters that will not be used for contamination checks.

3.3. Use the Diagnostics Leak Test in Cleaning Step 2.8. to fill each canister with nitrogen.

3.3.1. Set Leak Test to 18 psig for two (1) minute. Once started, let nitrogen flow for about fifteen (15) seconds and press <CANCEL>. Restart the leak test in ten (10) seconds. Sometimes when the filling process is first initiated, the flow of nitrogen will bypass the canisters, not filling them up. This technique ensures the canisters are filled.

3.4. When the actuator stops clicking and the timer starts, close all of the canister valves and press <CANCEL> on the screen.

3.5. Remove all canisters from the TO-Clean.

3.6. Place representative contamination check canisters on shelf for analysis. Place others on shelf to hold until cleared for shipping.

4.0 EVACUATING CANISTERS

- 4.1. Once the selected canisters have passed all applicable contamination check analyses, they will need to be evacuated prior to being sent out to the field.
- 4.2. Depending on circumstances, canisters may be evacuated after the system has been turned on or right after a cleaning 6CYCLE Program has terminated.
- 4.3. Evacuating canisters while the system is off.
 - 4.3.1. Follow Cleaning steps 2.1. through 2.5.
 - 4.3.2. Wearing safety gloves and glasses, fill the dewar to 25% with liquid nitrogen.
 - 4.3.3. Cap any lines with plugs that will not be used. DO NOT OVERTIGHTEN.
 - 4.3.4. Place canisters inside the oven with valves facing forward.
 - 4.3.5. Attach the flexible lines to each canister and tighten by hand. Tighten with wrench until there is resistance. DO NOT OVERTIGHTEN.
 - 4.3.6. Run Diagnostics Leak Test for one (1) minute at 30 psig.
 - 4.3.6.1. From the initial screen, press <DIAGNOSTICS> <LEAKTEST> <START>.
 - 4.3.6.2. If leak test fails, recheck all connections to the canisters and rerun the leak test.
 - 4.3.6.3. If leak test fails again, the U-trap may need to be emptied. See Cleaning U-trap in section 6.1 of WI for details.
 - 4.3.7. If leak test passes, load the EVAC program and open each canister valve as the program starts the evacuation process.
 - 4.3.7.1. Once the screen shows FINISHED, press <CANCEL> <CANCEL> <CLEAN> <LOAD METHOD> <up/down arrow> to <EVAC> <LOAD> <CLEAN>.

- 4.4. Evacuating canisters after 6CYCLE Program termination.
 - 4.4.1. If the dewar has less than 25% liquid nitrogen, fill to at least 25%.
 - 4.4.2. Keep the oven door open during evacuation.
 - 4.4.3. Cap any lines with plugs that will not be used. DO NOT OVERTIGHTEN.
 - 4.4.4. Place canisters inside the oven with valves facing forward.
 - 4.4.5. Attach the flexible lines to each canister and tighten by hand. Tighten with wrench until there is resistance. DO NOT OVERTIGHTEN.
 - 4.4.6. Run Diagnostics Leak Test for one (1) minute at 30 psig.
 - 4.4.6.1. From the initial screen, press <DIAGNOSTICS> <LEAKTEST> <START>.
 - 4.4.6.2. If leak test fails, recheck all connections to the canisters and rerun the leak test.
 - 4.4.6.3. If leak test fails again, the U-trap may need to be emptied. See Cleaning U-trap in section 6.1 of WI for details.
 - 4.4.7. If leak test passes, load the EVAC program and open each canister valve as the program starts the evacuation process.
 - 4.4.7.1. Once the screen shows FINISHED, press <CANCEL> <CANCEL> <CLEAN> <LOAD METHOD> <up/down arrow> to <EVAC> <LOAD> <CLEAN>.

5.0 SHUT DOWN

- 5.1. Cap all of the lines inside the oven to finger tight.
- 5.2. Close the nitrogen toggle valve attached to the humidification chamber.
- 5.3. Turn off TO-Clean.
- 5.4. Turn off vacuum pump.

6.0 PREVENTATIVE MAINTENANCE

The manufacturer performs preventative maintenance on a yearly basis under contract with CARB.

Lab staff perform the following preventative maintenance:

6.1 CLEANING U-TRAP



Figure 5. U-trap Inside Liquid Nitrogen Dewar

At times, the U-trap (Figure 5) fills with water attributable to high humidity in the samples. The water in the U-trap will cause the pump to evacuate inefficiently. Using safety gloves and glasses, untighten the hose connections to both sides of the U-trap. Remove the U-trap from the dewar and run hot water through it at the sink. Chunks of ice may be seen flowing out.

Dry the U-trap using the in-house air nozzle inside the fume hood. Prop the U-trap against the air nozzle inside the fume hood and allow air to flow for 10-15 minutes to dry out. Once dried, carefully reinsert the U-trap back into the dewar and reconnect the hoses. Rerun the leak test program.

6.2 SYSTEM CONTAMINATION

System contamination can occur from exposure to laboratory air or from impure water in the humidifier. If contamination is suspected, it is recommended to purge the system with humidified air or nitrogen before cleaning canisters. This can be done by setting up a cleaning method with multiple cycles while running with the manifold capped. This procedure should be done anytime the water in the humidifier or nitrogen cylinder is changed, or when the sample lines have been left uncapped.

6.3 REPLACING DEIONIZED WATER

When nitrogen cylinders need to be replaced, the deionized water inside the humidification chamber should also be exchanged. This is done by releasing the pressure inside the chamber. First, ensure that the toggle valve (Figure 6) is closed. Untighten the fitting that connects the humidifier to the TO-Clean. Pressure inside will release once the connection is removed. The cap on the humidifier can now be unscrewed.

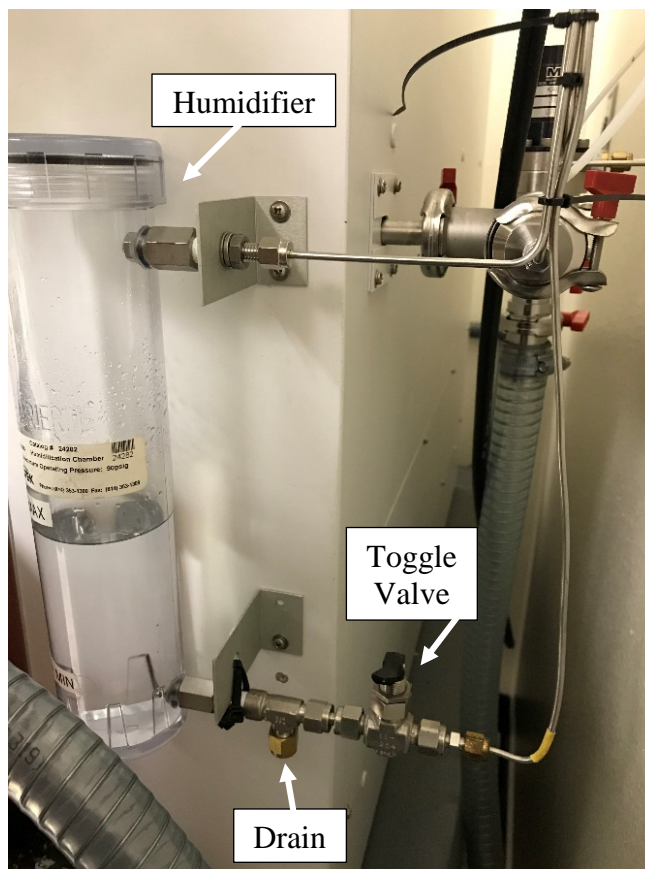


Figure 6. Humidifier, Drain, and Toggle Valve

Using a wrench, remove the drain cap from the humidifier line and collect the water using a graduated flask. Once drained, add 250 mL of deionized water to collect the water that is trapped at the bottom of the humidifier. Replace the drain cap and fill the humidifier to the fill-line. Replace humidifier cap to hand-tight only. Replace the TO-Clean line. Open the toggle valve and check for leaks by inspecting connections.

6.4 DISCONNECTING THE NITROGEN SOURCE

To ensure uninterrupted operation of the TO-Clean, the following procedures must be followed when reducing nitrogen supply pressure or changing nitrogen cylinders.

- 6.4.1. After the last cleaning cycle is finished, reduce oven temperature set point to 25°C. Open the door for faster cooling.
- 6.4.2. Once the oven is sufficiently cooled, close all the canister valves.
- 6.4.3. Close the valve on the nitrogen cylinder.
- 6.4.4. On the Start Menu, select Diagnostics.
- 6.4.5. On the Diagnostics menu, select Valve Control.
- 6.4.6. On the Valve Control menu, click the Vent Valve "On". This will bring the manifold and lines between V3 and V1 to atmospheric pressure.
- 6.4.7. Click the Pressure Valve "On". You should hear nitrogen flowing out of the purge vent.
- 6.4.8. Once the regulator pressure reads "0", close the regulator.
- 6.4.9. On the Valve Control screen, click the Vent Valve and Pressure Valve "Off".
- 6.4.10. Replace the nitrogen cylinder.
- 6.4.11. Open the nitrogen cylinder valve and set the regulator to desired pressure, 35 psig maximum.
- 6.4.12. The system is now ready to run cleaning cycles.

7.0 NEW AIR CANISTERS

Newly purchased canisters are received at the lab under vacuum. The vacuum shall be tested to determine if a leak is present by recording any changes over a two-week period. If the canister vacuum changes by more than 1 inHg, notify the manufacturer for replacement. If the canisters pass the two-week period by maintaining vacuum, they can be placed in the TO-Clean for cleaning. Each canister receives its own LIMS number and is analyzed for all methods the lab performs using canisters.

8.0 TECHNICAL ASSISTANCE DOCUMENT FOR THE NATIONAL AIR TOXICS TRENDS (NATTS) PROGRAM OCTOBER 2016 SECTION 4.2.4.2.4

Verification of Canister Cleanliness. Following completion of canister cleaning activities, minimally one canister per batch cleaned must be pressurized to approximately the pressure of field collected samples with humidified purge gas, held minimally overnight, and analyzed to ensure all target compounds are < 3x MDL or < 0.2 ppbv, whichever is lower. Cleanliness criteria must be lowered for agencies which dilute field samples such that the cleanliness criteria are met for undiluted samples. For instance, if a laboratory dilutes all samples by two-fold by addition of gas to the collected sample canister, the cleanliness criteria are not doubled, but are cut in half. A detected concentration of benzene at 0.15 ppbv (assuming 3x MDL is higher) at the instrument would not pass criteria, as the concentration adjusted for dilution is 0.30 ppbv which exceeds the 0.2 ppbv criterion.

Analysis of more than one canister from each batch is highly recommended and should be no less than one out of every ten canisters. A best practice is to survey every canister in a cleaning batch. Following analysis, canisters are re-evacuated to ≤ 50 mTorr. If only a subset of the canisters in the batch is able to be analyzed, the selected canisters should be those which indicated the highest total VOC concentration or the highest single target compound concentration in the previous sample. Other conventions for selecting the batch blank canister include random selection or evaluating high molecular weight compounds or oxygenated compounds which are more difficult to completely remove from canisters.

A composite batch blank sample may be prepared by closing the valve of a chosen canister (which is still under vacuum). The manifold is then pressurized with clean purge gas such that the other connected canisters are pressurized. The chosen batch blank canister is then opened to fill the canister with the composite gas from all of the canisters connected to the manifold.

Actions must be taken to further investigate failure of batch blanks to meet the cleanliness criteria. If each cleaned canister from the batch is surveyed, only those canisters which fail the criteria must be re-cleaned. If one canister representing the batch fails, either the entire batch can be re-cleaned (recommended) or two canisters from the batch can be selected and analyzed to confirm the batch does not pass criteria. If both of these canisters pass, only the failing canister must be re-cleaned, otherwise, the batch must be re-cleaned. Continued failure of batch blanks may indicate that the manifold or other parts of the system has become contaminated.