

Roy Cooper
Governor
Elizabeth Biser
Secretary
Michael A. Abraczinskas
Director



DAQ-03-005.2 Standard Operating Procedure (SOP)
Entech 3100A Canister Cleaner
OPERATOR RESPONSIBILITIES

Revision 1



North Carolina Department of Environmental Quality | Division of Air Quality
217 West Jones Street | 1641 Mail Service Center | Raleigh, North Carolina 27699-1641
919.707.8400

1.0 Approval Sign Off-Sheet

I certify that I have read and approve of the contents of the Entech 3100A Canister Cleaner Standard Operating Procedure with an effective date of August 23rd, 2021.

Director, Air Quality Division

Michael Abraczinskas

Signature: [DocuSigned by: Michael Abraczinskas] Date: 8/25/2021

Ambient Monitoring Section Chief

Patrick Butler

Signature: [DocuSigned by: Patrick Butler] Date: 8/25/2021

Laboratory Analysis Branch Supervisor

Jim Bowyer, Environmental Program Supervisor II

Signature: [DocuSigned by: Jim Bowyer] Date: 8/25/2021

Projects and Procedures Branch Supervisor

Joette Steger, Environmental Program Supervisor II

Signature: [DocuSigned by: Joette Steger] Date: 8/20/2021

Primary SOP Author

Brian D. Velleco, Chemist I

Signature: [DocuSigned by: Brian D. Velleco] Date: 8/20/2021

EPA Region 4

Designated Approving Official

Signature: _____ Date: _____

Disclaimer: This document, and any revision hereto, is intended solely as a reference guide to assist individuals in the operation of the instrument, related to the North Carolina Division of Air Quality's Ambient Monitoring Program.

SOP Acronym Glossary

ADQ - Audit of data quality

ASTM – American Society for Testing and Materials

AQS - Air Quality System (EPA's Air database)

°C – degrees Celsius

CCV – Continuing Calibration Verification

CFR – Code of Federal Regulations

CGA – Compressed Gasses Association

Chief – Ambient Monitoring Section chief

COC – Chain of custody

DAQ - North Carolina Division of Air Quality

DAS – Data acquisition system

DEQ – North Carolina Department of Environmental Quality

DI – Deionized

Director – Division of Air Quality Director

ECB – Electronics and Calibration Branch

e-log – electronic logbook

EPA – United States Environmental Protection Agency

FEM – Federal equivalent method

FRM – Federal reference method

GC-MS – Gas Chromatography – Mass Spectrometry

HPLC – High Performance Liquid Chromatography

HV – high vacuum

in - inches

L – liters

LAB – Laboratory Analysis Branch – DAQ Lab located at 4403 Reedy Creek Rd in Raleigh, NC

LED – Light emitting diode

MDL – Method detection limit

mtorr – millitorr

NATTS – National Air Toxics Trend Station

NC – North Carolina

PAMS – Photochemical assessment monitoring station

PM – Particulate matter

PPB – Projects and Procedures Branch

ppb_v - parts per billion volume

PPE – Personal Protective Equipment

psi – pounds per square inch

psi_A - pounds per square inch – absolute scale

PT – performance trial (audit sample)

QA – Quality assurance

QA/QC - Quality assurance/quality control

QAPP - Quality assurance project plan

QC – Quality control

RCO – Raleigh central office

RPM – Revolutions per minute

SOP - Standard operating procedure

TSA - Technical systems audit

UAT – Urban Air Toxics

UHP – Ultrahigh purity

USB – Universal Serial Bus

VOC – Volatile organic compound

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2.0 SCOPE AND PURPOSE

The scope and purpose of this SOP is to describe the steps required to successfully operate the Entech 3100A Canister Cleaner employed to clean 6 L Silco-treated Summa canisters for ambient air collection, instrument calibration standards or QC sample preparation at the DAQ Laboratory Analysis Branch (LAB).



Figure 1: Entech 3100A Canister Cleaning System

The Entech 3100A canister cleaner houses twelve 6 L canisters, canisters depicted below in Figures 2 and 3, mounted onto a silonite manifold with ¼-in compression fittings. The system heats the canisters in a closed oven at 75 degrees Celsius (°C) and cycles canisters under vacuum and then fills the canisters using ultra-high purity nitrogen as diluent gas. The canister cleaner utilizes a roughing pump and molecular drag pump operating at 27000 revolutions per minute (RPM) to attain a 10 millitorr (mtorr) vacuum on the canisters. Clean ultra-high purity nitrogen is humidified by the Entech 3100A Canister Cleaning System and is then introduced into the canisters via a pre-programmed, above ambient pressure, set point. This cycle of evacuation and pressurization is repeated cyclically to volatilize and displace VOCs inside the canisters and replace them with clean humidified nitrogen gas. The resultant canisters are batch analyzed by **SOP DAQ-03-003.2 Markes-Agilent Gas Chromatography-Mass Spectrometry VOC Analysis** for cleanliness. Canisters are stored after certification by SOP DAQ-03-003.2 for a period of no longer than 30 days.



Figure 2: Entech Silonite Silco-Treated 6 L Canister



Figure 3: Restek Silco-Treated SilcoCan 6 L Canister

3.0 EQUIPMENT CHECKS AND MATERIALS

This section describes the equipment and materials that are required to complete the steps described in this document. Additional subsection(s) or SOPs will also describe the equipment and materials as needed.

3.1 Equipment and Material List

- Entech 3100A Canister Cleaning System
- VacuumBrand rough pump or equivalent
- Computer and monitor with Windows operating system installed with:
 - Entech 3100A Canister Cleaning System software
 - Universal Serial Bus (USB) mouse and keyboard
- 12 x 6 L Entech Silonite Silco-treated Canisters or equivalent
- Ultrahigh purity (UHP) Nitrogen (Arc3 Gases Grade 5.0 or equivalent – See Appendix A for gas purity information)
- Compressed Gasses Association (CGA) pounds per square inch (psi) 580 Regulator
- Cylinder Transportation Cart
- Cylinder mount with straps (to secure to bench)
- Deionized (DI) water (HPLC Grade, Submicron filtered or ASTM Type 1 DI Water or equivalent)
- DI water filled squeeze bottle
- DAQ-03-019.2 Canister Batch Sheet (see Appendix B for a blank **DAQ-03-019.2** form)
- Canister Cleaning System Binder
- Canister Tags (see Appendix C for a blank canister tag)
- 12 x ¼-in compression fitting male nut caps, brass, or stainless-steel
- 12 x ¼-in compression fitting female nut caps, brass, or stainless-steel
- ¼-in replacement ferrules (vespel-graphite or stainless steel)
- Entech TrueSeal replacement Silonite Silco-treated canister valves or equivalent
- Hand Tools:
 - 9/16-in wrench
 - 7/8-in wrench
 - 12-in adjustable wrench or larger
 - ¼-in Die
 - Dremel tool and accessories
- PPE:
 - Heat resistant gloves
 - Heat resistant sleeve
 - Nonslip steel toed shoes or boots
 - Safety glasses: prescription or impact resistant are acceptable

3.2 Support Equipment Checks

3.2.1 UHP Nitrogen Cylinder Check

- 1 Open the UHP nitrogen cylinder and read the regulator pressure. Set the outlet pressure to 50 psi.

- 2 If the UHP nitrogen cylinder gauge reads below 250 psi, change the cylinder by closing the cylinder and loosening the regulator with an adjustable wrench.
- 3 Place the regulator on the bench and replace the cylinder. Be sure to use a cylinder cart to transport cylinders safely.
- 4 Reinstall the regulator by tightening the nut and open the cylinder. The pressure will read about 2400 psi. Open the regulator.
- 5 Record this check by completing the **UHP Nitrogen supply OPEN** section of the canister batch sheet.
- 6 Record the lot number of the current cylinder on the canister batch sheet.
- 7 Close the cylinder and regulator when Entech 3100A Canister Cleaner operations are completed.

3.2.2 System Pump Check

- 1 Switch on the rough pump underneath the bench of the canister cleaning system. It will begin to vibrate and pump air through it. This can be heard audibly.
- 2 Switch on the Entech 3100A high vacuum (HV) pump by toggling the pump switch on the front panel of the 3100A.
- 3 Visually verify that the HV pump has reached maximum speed of 27000 RPM as indicated by the illumination of the green light emitting diode (LED) on the system front panel.
- 4 Record this check by completing the **both pumps ON** section of the canister batch sheet.

3.2.3 Humidification Chamber Check

- 1 Verify water is visible in the humidification chamber located in the back of the 3100A but not above the **Water Level Line** located behind the chamber.
- 2 If the water level is not visible, fill the reservoir by loosening the 7/8-in nut and removing the chamber. Add deionized water to the chamber and reinstall the reservoir. Retighten the 7/8-in nut to secure the chamber to the system.
- 3 Record this check by completing the **water in humidification chamber** section of the canister batch sheet. **Record the lot number of the DI water added** to the reservoir.

3.2.4 System Pressure Check

- 1 Verify the displayed ambient pressure on the Entech 3100A canister cleaning software is 14.7 pounds per square inch actual (psia). The pressure is displayed on the top of the 3000/3100 Run Control Screen as depicted below under the **Pressure (psia)** header:

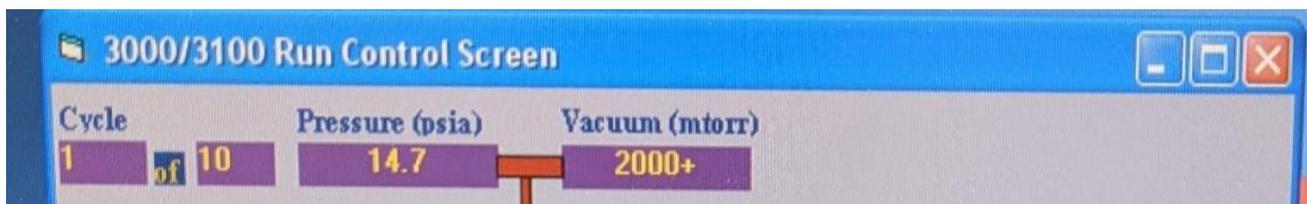


Figure 4: Entech 3100A Canister Cleaning System Pressure Read-back

- 2 If the display pressure does not match within ± 0.5 psia contact the LAB electronics technician for assistance. Do not proceed with canister cleaning.
- 3 Record this check by completing the **ambient pressure is at 14.7 ± 0.5 psia** section of the canister batch sheet.

4.0 SITE CHECKS

This section is reserved. Site checks do not apply to analysis instruments housed at the Reedy Creek Laboratory.

5.0 DETAILED PROCEDURES

5.1 System Start Up and Preparation

- 1 Select a canister cleaning system: either 1 or 2 and denote it on the canister batch sheet.
- 2 Boot, if required, and login to the selected system computer.
- 3 Double click the 3100A icon on the desktop to connect to the cleaning system.
- 4 The link active LED on the 3100A front panel will illuminate once connected.
- 5 Visually check and verify that the humidification chamber has a visible level of DI water. If the water level is not visible, fill the reservoir by loosening the 7/8-in nut and removing the chamber. Add DI water to the chamber to the marked **Water Level Line** and reinstall the reservoir. Retighten the 7/8-in nut to secure the chamber to the system. Record this step by completing the **water in humidification chamber** section of the canister batch sheet. Record the lot number of the DI water added to the reservoir.
- 6 Open the Entech 3100A Canister Cleaner oven doors and remove the caps from the manifold.
- 7 Select 12, 6-L canisters to be cleaned and record canister numbers on the canister batch sheet. Complete the other applicable sections of the canister batch sheet: **operator, date, and batch number**. The **batch number** will be T-#### for canister cleaning system 1 and H-#### for canister cleaning system 2. The batch number will begin at one and be incremented by one for each subsequent batch. (E.g., Batch T-0001 will occur on canister cleaning system 1, then T-0002, T-0003...). Refer to the canister cleaning system binder for the last batch run on each system.
- 8 Connect the canisters to the canister cleaner manifold using a 9/16-in wrench. Take care not to over tighten the connections and make sure all canister valves are closed. A best practice is one-quarter turn past finger tight to create a good compression fitting seal.
- 9 Turn on the canister cleaner oven and heat switch located on the left panel of the oven. The oven is pre-set for 75°C. It will take several minutes to reach the desired temperature. Record that the oven has hit temperature by completing the **oven temperature at set point (75°C)** section of the canister batch sheet. The current temperature of the oven is displayed in green on the LED screen in the top left corner of the oven chassis. A set-point of 75°C will be displayed in orange as shown below in Figure 5:



Figure 5: Entech 3100A Canister Cleaning System Oven Control Panel

Note: The Silonite treated canisters can begin to breakdown if heated beyond 80°C. The silonite layer within the canister is a thin film deposited by chemical vapor deposition and exposure to higher temperatures can cause the layer to wear and create active sites within a given canister. **Do not change the set point of the oven.**

- 10 Open the regulator to the UHP nitrogen supply cylinder and set the outlet pressure to 50 psi. Record this step by completing the **UHP Nitrogen supply OPEN** section of the canister batch sheet. Record the lot number of the current cylinder on the canister batch sheet.

- 11 Turn on the rough pump switch manually and then turn on the HV pump switch on the front of the 3100A system.
- 12 Visually verify that the HV pump has reached maximum speed of 27000 RPM as indicated by the illumination of the green LED on the system front panel.

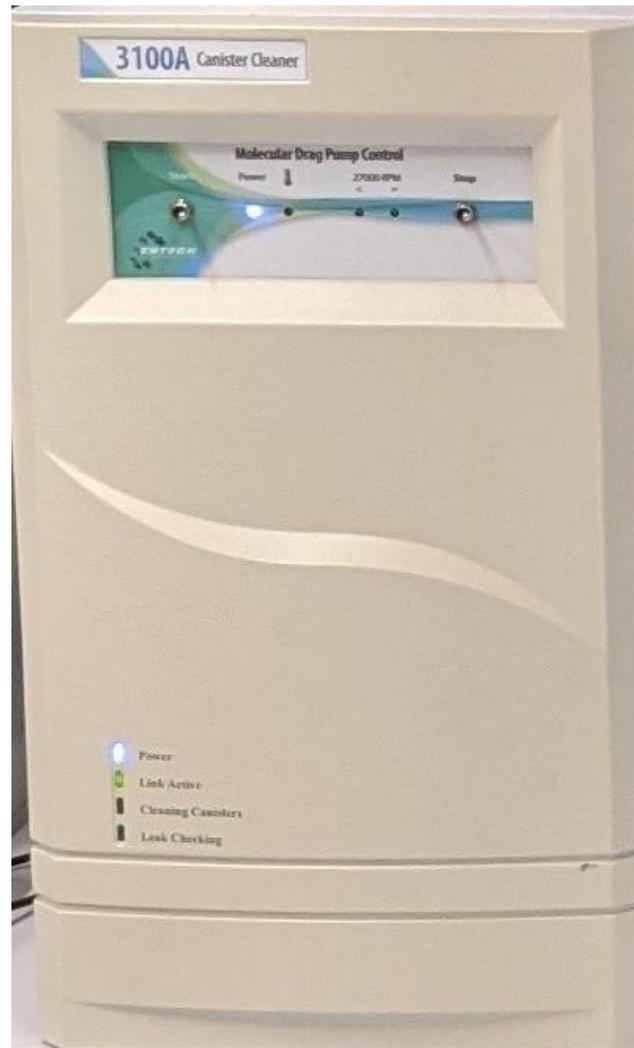


Figure 6: Entech 3100A Canister Cleaning System Front Panel

- 13 Record the pumps are functional by completing the **both pumps ON** section of the canister batch sheet.
- 14 The operator can now proceed with a system leak check.

5.2 System Leak Check

- 1 In the 3100A software, load the canister method with the desired cycle count. **No less than five cleaning cycles should be used when operating the canister cleaning system.** Select a method by clicking open on the top tab and a method menu will appear as shown in Figure 7:

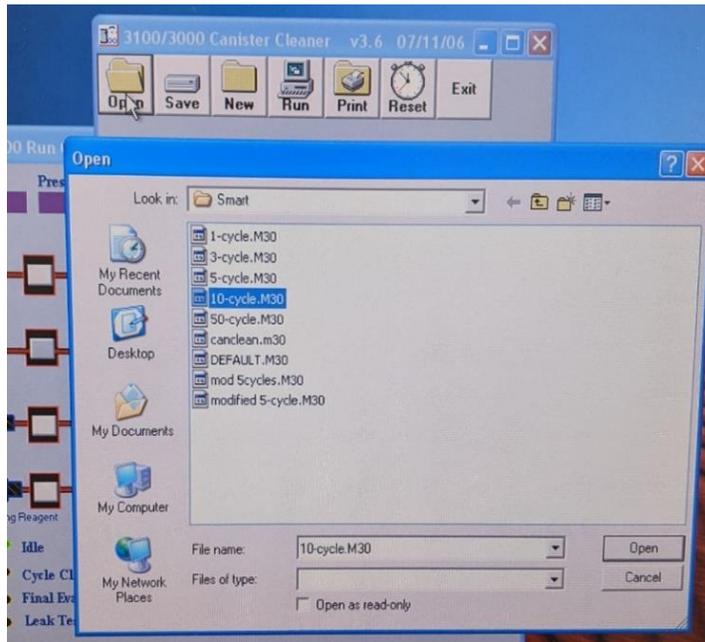


Figure 7: Entech 3100A Canister Cleaning System Method Selection

- 2 Verify the displayed ambient pressure on the Entech 3100A canister cleaning software is 14.7 psi_A. If the display pressure does not match within ± 0.5 psi_A contact the LAB electronics technician for assistance. Do not proceed with canister cleaning. Record this step by completing the **ambient pressure is at 14.7 \pm 0.5 psi_A** section of the canister batch sheet. Record the current ambient pressure displayed by the system.
- 3 Using the mouse cursor, click the rough pump button in the run screen. The read-back **pressure (psia)** in the window will begin to drop. The run screen is shown in Figure 8:

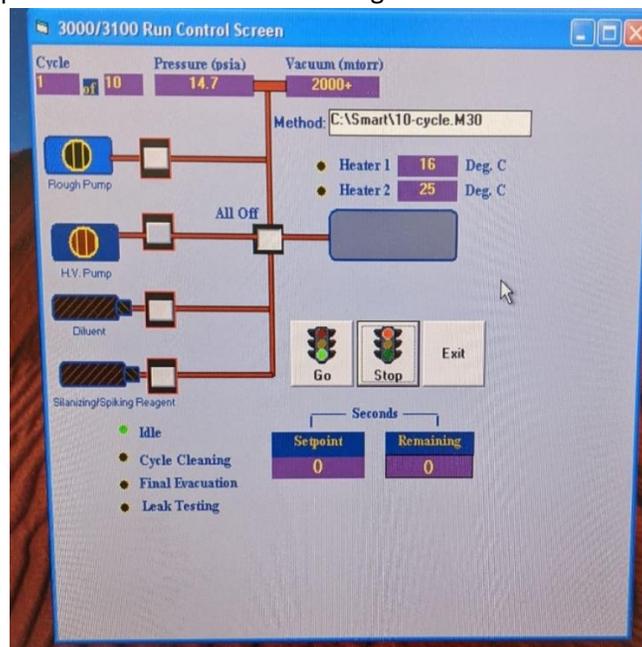


Figure 8: Entech 3100A Canister Cleaning System Run Screen

- 4 After a vacuum of less than 0.2 psi has been achieved, click the HV button in the run screen. The **Vacuum (mtorr)** read-back will begin to drop.
- 5 The HV has been pre-set to 10 mtorr and the system should achieve it after several minutes of evacuation.
- 6 Once the system has achieved a HV of 10 mtorr, monitor the read-back for 1 minute. If no fluctuations occur the system is deemed leak-free. This is considered a system batch leak check. If any of the above steps fail, see section 9.1 for corrective action.
- 7 After the leak check has been deemed successful, click **all off** in the run screen.
- 8 Record the passing leak check in the **system leak check passes** section of the canister batch sheet.

5.3 Canister Cleaning Operation

- 1 After a successful system leak check, open the oven doors and open all canister valves. Wear heat resistant gloves and sleeves to protect from burns. Close oven doors when finished and allow the system to return to 75°C. Record that all canister valves are open by completing the **all canister valves OPEN** section of the canister batch sheet.
- 2 Click the **Go** button in the run screen of the 3100A software to execute the cleaning method. The **cleaning canisters** LED on the system front panel will illuminate.
- 3 The cleaning processes takes about 6 hours to complete for a 10-cycle cleaning batch; the system will return to the idle status after all cycles have been run. The canisters will achieve a vacuum of 10 mtorr on the system run screen.
- 4 Once canister cleaning is completed open oven doors while wearing heat-resistant gloves and sleeves and close one of the front canister valves. Record this canister on the canister batch sheet as the **batch certification canister #** and close the oven doors.
- 5 Click the **diluent** button in the run screen, the pressure read-back should begin to rise.
- 6 Fill with diluent UHP nitrogen until the pressure reads between 25-35 psi on the run screen, then click the **all off** button.
- 7 Open the oven doors and while wearing heat-resistant gloves and sleeves to open the canister that was closed so that the diluent gas in the other 11 canisters will flow into the certification canister. This canister will serve as the batch certification canister.
- 8 Equilibrium has been observed on the pressure read-back when the pressure stops dropping. When equilibrium has been achieved close the valve of the batch certification canister. Close the oven doors.
- 9 Evacuate the remaining 11 canisters by engaging the rough pump in the run screen.
- 10 Once a vacuum of 0.2 psi or lower has been achieved, engage the HV pump in the run screen.
- 11 Once the HV reading has achieved 10 mtorr for a minute or longer, open the oven doors and close all the canisters using heat-resistant gloves. **Record the final canister pressure in mtorr on the canister batch sheet.**
- 12 Power off the oven heater and allow the system to cool to ambient conditions.
- 13 Remove the batch certification canister from the cleaning system. **Affix a canister tag** and deliver it to the LAB chemist for verification of cleanliness. Fill out the **can number** and **batch number field** of the **tag**. In the comment field write **certification canister**.
- 14 Once cleanliness has been verified via **SOP DAQ-03-003.2 Markes-Agilent GC-MS VOC Analysis**, remove all canisters from the cleaning system.
- 15 Cap the oven manifold ports with ¼-in nuts to finger tight.
- 16 **Complete and affix a canister tag** to each canister with the correct canister number and cleaning batch number.
- 17 Cap each canister with a ¼-in female compression nut.

- 18 Store clean canisters on canister racks until needed.
- 19 Place the completed canister batch sheet and associated gas chromatography-mass spectrometry (GC-MS) data for the batch canister check in the canister cleaning system binder.

6.0 ENTECH 3100A CANISTER CLEANER DATA REVIEW

This section of the SOP describes steps required for the operator to perform a self/Level 1 review on the canister cleaning system data. Additionally, this section describes steps required for LAB personnel not directly involved in the canister cleaning process to perform a peer/Level 2 data review. The review of the canister cleaning data, while separate in scope, is also considered in this SOP. GC-MS operation is not covered in this SOP and is reserved for qualified operators only. Please see **SOP DAQ-03-003.2 Markes-Agilent GC-MS VOC Analysis** for details.

6.1 Self/Level 1 Canister Batch Sheet Data Review (Canister Cleaner Operator)

Review canister batch sheet. Are all sections complete? Have all canisters been correctly identified on the form? Is all writing legible? Make any corrections necessary by putting a line through the error, initialing and dating. Write the correction next to the errant entry. The operator will initial and date the canister batch sheet indicating level 1 review has occurred. Submit for peer, level 2 review. It is the responsibility of the primary, level 1, operator to find a suitable reviewer in a timely manner.

6.2 Peer/Level 2 Canister Batch Sheet Data Review

Review the canister batch sheet for completeness and accuracy. Make any corrections necessary by putting a line through the error, initialing and dating. Write the correction next to the errant entry. The peer reviewer will initial and date the canister batch sheet indicating level 2 review has occurred.

6.3 Self/Level 1 Canister Certification Data Review (LAB Chemist)

- 1 Check the continuing calibration verification (CCV) recovery before and after certification canister injection and flag compounds that fail. The CCV must be within $\pm 30\%$ recovery to be acceptable for each compound. A list of the compounds identified via TO-15 GC-MS analysis is shown in Table 1:

Table 1: TO-15 Standard Cylinder Peak Table (Bold = NATTS Core I and Target PT compounds)	AQS Compounds ID
1,1,1-Trichloroethane	43814
1,1,2,2-Tetrachloroethane	43818
1,1,2-Trichloroethane	43820
1,1-Dichloroethane	43813
1,1-Dichloroethene	43826
1,2,3-Trimethylbenzene	45225
1,2,4-Trichlorobenzene	45810
1,2,4-Trimethylbenzene	42508
1,2-Dibromoethane	43843
1,2-Dichlorobenzene	45805
1,2-Dichloroethane	43815

Table 1: TO-15 Standard Cylinder Peak Table (Bold = NATTS Core I and Target PT compounds)	AQS Compounds ID
1,2-Dichloropropane	43829
1,3,5-Trimethyl Benzene	45207
1,3-Butadiene	43218
1,3-Dichlorobenzene	45806
1,4-Dichlorobenzene	45807
1,4-Dioxane	46201
1-Bromopropane	43853
1-Butanol	43305
2-Pentanone	43562
3-Hexanone	43557
3-Pentanone	43553
Acetaldehyde	43503
Acetone	43551
Acetonitrile	43702
Acrolein	43505
Benzene	45201
Benzyl chloride	45809
Bromodichloromethane	43828
Bromoform	43806
Bromomethane	43819
Carbon disulfide	42153
Carbon Tetrachloride	43804
Chlorobenzene	45801
Chloroethane	43812
Chloroform	43803
Chloromethane	43801
Cis-1,3-dichloropropene	43831
Cyclohexane	43248
Cyclopentane	43242
Ethanol	43302
Ethylbenzene	45203
Freon 11	43811
Freon 113	43821
Freon 114	43208
Freon 12	43823
Freon 22	43359
Hexanal	43517
Hexane	43231
Iodomethane	43808
Isobutene	43270

Table 1: TO-15 Standard Cylinder Peak Table (Bold = NATTS Core I and Target PT compounds)	AQS Compounds ID
Isoprene	43243
Isopropyl Alcohol	43312
m/p-Xylenes	45109
Methacrolein	43515
Methyl Butyl Ketone	43559
Methyl Ethyl Ketone	43552
Methyl Isobutyl Ketone	43560
Methyl tert-Butyl Ether	43372
Methyl Vinyl Ketone	43558
Methylene Chloride	43802
n-Butanal	43510
n-Pentane	43220
o-Xylene	45204
Propanal	43504
Propylene	43205
Styrene	45220
Tetrachloroethylene	43817
Toluene	45202
Trans-1,2-dichloroethene	43838
Trans-1,3-dichloropropene	43830
Trichloroethylene	43824
Vinyl Acetate	43447
Vinyl Chloride	43860

The compounds in **bold** are NATTS **Core I** compounds and target PT compounds for the TO-15 analysis.

- 2 Flag certification canister compounds detected at or above 0.2 parts per billion by volume (ppb_v) as failing.
- 3 Compare failing CCV compounds against failing certification canister compounds. Disqualify compounds with CCV failures and/or certification can failures.
- 4 Failing certification blank compounds that pass CCV recovery (before and after) must be flagged **CC**. Failing compounds must be flagged such that they can be easily identified during data review processes.
- 5 See section 9.3 for certification batch failure corrective action.
- 6 Once the data is confirmed valid, the operator will print out the CCV analyses and blank certification canister analysis and include the data with the canister batch sheet in the canister cleaning system binder. The analyst will initial and date the data to indicate that level 1 review has occurred. It is the responsibility of the primary, level 1, LAB chemist to find a suitable reviewer in a timely manner.

6.4 Peer/Level 2 Canister Certification Data Review

- 1 Verify that the data submitted by the LAB chemist passes CCV recovery before and after certification canister analysis. The CCV must be within $\pm 30\%$ recovery to be acceptable for each compound.

- 2 Confirm that all analytes are below 0.2 ppb_v and failing certification blank compounds that pass CCV recovery (before and after) must be flagged **CC**. Failing compounds must be flagged such that they can be easily identified during data review processes.
- 3 Once the data is confirmed valid, the peer reviewer will initial and date the data to indicate that level 2 review has occurred in the canister cleaning binder.

Note: This review can occur by the Entech 3100A operator or another LAB Chemist, independent from the initial reviewer (the chemist who did the analysis cannot peer review their own work).

7.0 FILE MANAGEMENT

This section of the SOP describes the different files generated during the canister cleaning process and the individual required to manage the file, either the operator, LAB chemist, or peer reviewer. Files include the canister batch sheet and GC-MS data associated with the batch check canister.

7.1 Canister Batch Sheet (Entech 3100A Operator)

The operator will store the completed canister batch sheet in the canister cleaning system binder. It is the responsibility of the Entech 3100A Canister Cleaner operator to maintain this record.

7.2 GC-MS Data Files (LAB Chemist)

The LAB Chemist will store the batch certification data with the canister batch sheet in the canister cleaning system binder. This includes system QC checks associated with the canister certification process. The data can be stapled to the canister batch sheet.

7.3 Peer Reviewed Canister Batch Sheet and GC-MS Data Files (Peer/Level 2 Reviewer)

The Entech 3100A Canister Cleaner operator will store the completed canister cleaner form in the canister cleaning system binder. The LAB Chemist will store the batch certification data with the canister batch sheet inside the canister cleaning system binder. The reviewer does not need to maintain this entry.

8.0 FILE QUALITY ASSURANCE AND DATA HANDLING

8.1 Records Retention

Printed records will be held at the DAQ LAB for the period specified in the DAQ document retention guidelines within the appropriate canister cleaning system binder.

8.2 Monthly Data Archival and Digitization (Data Backup)

Printed records from each month will be scanned and archived on the P: network drive in the LAB folder under the folder Canister Cleaning. The file path is: **P:\LAB (Laboratory Analysis Branch)\Canister Cleaning**

9.0 TROUBLESHOOTING AND CORRECTIVE ACTIONS

9.1 System Leak Check Failure Corrective Action

- 1 If the 3100A Canister Cleaning System fails to leak check to 10 mtorr, then click **all off** in the 3100A software control window to stop the system pumps.
- 2 Confirm that all canisters are tightened appropriately and that ferrules are in good condition. Replace any ferrules that are cracked, damaged, or warped. A Dremel tool can be used to cut the ferrule away if it is metallic.
- 3 Attempt to leak check the system again as described in section 5.2.
- 4 If the above process fails, then a leak is likely present between the connection to the 3100A manifold and the canister valve. The canisters can be removed one-by-one and the system leak check performed.
- 5 Once the leaking canister has been identified, inspect the threads around the canister orifice. If damaged, run a ¼-in dye over the thread to repair the canister and reinstall it in the canister cleaning system.
- 6 Leak check the system again. If it passes, proceed with cleaning; if it does not pass, replace the bad canister and set it aside. See section 9.2 for further action. Try again with a different canister until the system leak checks.
- 7 Contact the LAB Electronics Technician if these steps fail to produce a passing leak check.

9.2 Canister Valve Replacement

- 1 In the event a canister valve is damaged and will no longer pass leak check, replace the valve by loosening the nut on the bottom of the valve assembly with a 9/16-in wrench.
- 2 Put a new replacement valve on the exposed canister stem and tighten the nut.
- 3 Run the canister through the canister cleaning system after exposure to ambient air.

9.3 Failed Batch Certification Corrective Action

- 1 Re-analyze another canister from the same cleaning batch to verify the failing condition. If all compounds pass then, it is okay to proceed with batch certification.
- 2 If re-analysis confirms failing compound(s) above 0.2ppb_v, re-clean batch and retest when finished. If canisters are deemed dirty, a larger number of cleaning cycles can be employed (up to 50) to adequately clean the canisters at operator discretion.

10.0 REVISION HISTORY

1. Rev 0 – **BDV 04/09/2021** Original Publication
2. Rev 1 – **BDV 08/23/2021** Change History:
 - a. Canister Batch Sheet assigned document control number **DAQ-03-019.2** and formatting updated. A copy of the blank form was updated in appendix B.
 - b. Ambient pressure gauge reading acceptance criteria widened from ± 0.1 psi to ± 0.5 psi per the maximum tolerance allowed in the approved QAPP **DAQ-01-007**. All references in the document updated to reflect this change.
 - c. Items in quotations were changed to **bold** print for emphasis.
 - d. Sec 8.2 quarterly data archival and digitization updated to monthly data backup per QAPP **DAQ-01-007**.

11.0 REFERENCES

1. Entech Instruments (2021). Entech 3100A Canister Cleaner: Manual. Rev C. Simi Valley, CA. Entech Instruments

12.0 APPENDICES

1. Appendix A – Arc3 Purity Plus Specialty Gases Grade Specification Sheet
2. Appendix B – Blank Canister Batch Cleaning Sheet
3. Appendix C – LAB Canister Tag

Appendix A – Arc3 Purity Plus Specialty Gases Grade Specification Sheet



PURE GAS GRADE SPECIFICATIONS

CONTAMINATE LEVELS (UNLESS OTHERWISE NOTED AS %)

PRODUCT	GRADE	PURITY	PART #	VALVE	O2	THC*	H2O	CO	CO2	N2
ACETYLENE										
ATOMIC ABSORPTION	2.6	99.6%	ACE-26-300	510	< 4000 PPM (combined)	-	-	-	-	-
⇄ < 20 PPM PH3										
AIR										
ULTRA ZERO	0.1	-	AIR-UZ-300	590	19.5% - 23.5%	<0.1 ppm	<3 ppm	<1 ppm	<1 ppm	-
ZERO	1.0	-	AIR-ZE-300	590	19.5% - 23.5%	<1 ppm	-	-	-	-
EXTRA DRY	-	-	AIR-ED-300	590	19.5% - 23.5%	-	<8 ppm	-	-	-
ARGON										
RESEARCH	6.0	99.9999%	CDI-50-60	580	<0.2 ppm	<0.1 ppm	<0.2 ppm	<0.1 ppm	<0.1 ppm	<0.4 ppm
CHROMATOGRAPH	5.5	99.9995%	ARG-55-XX	580	<1 ppm	<0.1 ppm	<1 ppm	-	-	<3 ppm
NITROGEN FREE	5.0	99.9990%	ARG-NF-XX	580	<2 ppm	<0.5 ppm	<2 ppm	-	-	<4 ppm
UHP	5.0	99.999%	ARG-50-300	580	<1 ppm	<0.5 ppm	<1 ppm	-	-	-
PREPURIFIED	4.8	99.998%	ARG-48-300	580	<5 ppm	<2 ppm	<5 ppm	-	-	-
ZERO	4.8	99.998%	ARG-ZE-300	580	-	<0.5 ppm	-	-	-	-
CARBON DIOXIDE										
RESEARCH	5.0	99.999%	CDI-50-50	320	<1 ppm	<0.5 ppm	<2 ppm	<0.1 ppm	99.999%	<1 ppm
Scientific	4.8	99.998%	CDI-48-50	320	<2 ppm	<2 ppm	<3 ppm	<1 ppm	99.998%	-
LASER	4.5	99.995%	CDI-45-50	320	<5 ppm	<1 ppm	<5 ppm	-	99.995%	-
COLEMAN/ INSTRUMENT	4.0	99.99%	CDI-40-50	320	<20 ppm	-	<10 ppm	-	99.99%	<50 ppm
ANAEROBIC	3.0	99.9%	CDI-30-50	320	<20 ppm	-	-	-	99.9%	-
BONE DRY	2.8	99.8%	CDI-28-50	320	-	-	<20 ppm	-	99.8%	-
HELIUM										
GRADE 7	7.0	99.99999%	HEL-70-300	580	<50 ppb	<20 ppb	<50 ppb	<20 ppb	<20 ppb	<50 ppb
RESEARCH	6.0	99.9999%	HEL-60-300	580	<0.2 ppm	<0.1 ppm	<0.2 ppm	<0.1 ppm	<0.1 ppm	<0.4 ppm
CHROMATOGRAPH	5.5	99.9995%	HEL-55-XX	580	<1 ppm	<0.5 ppm	<1 ppm	-	-	<3 ppm
UHP	5.0	99.999%	HEL-50-300	580	<1 ppm	<0.5 ppm	<1 ppm	-	-	-
PREPURIFIED	4.7	99.997%	HEL-47-300	580	<5 ppm	-	<5 ppm	-	-	-
ZERO	4.8	99.998%	HEL-48-300	580	-	<0.5 ppm	-	-	-	-
HYDROGEN										
RESEARCH	6.0	99.9996%	HYD-60-300	350	<0.2 ppm	<0.1 ppm	<0.5 ppm	<0.1 ppm	<0.3 ppm	-
Grade 5.5	5.5	99.9995%	HYD-55-300	350	<0.5 ppm	<0.2 ppm	<2 ppm	<0.2 ppm	<0.1 ppm	<2 ppm
UHP^	5.0	99.999%	HYD-50-300	350	<1 ppm	<0.5 ppm	<1 ppm	-	-	<5 ppm
ZERO	4.5	99.995%	HYD-45-300	350	-	<0.5 ppm	-	-	-	-
PREPURIFIED	4.0	99.99%	HYD-40-300	350	<20 ppm	-	<10 ppm	-	-	-
NITROGEN										
RESEARCH	6.0	99.9999%	NIT-60-300	580	<0.2 ppm	<0.1 ppm	<0.2 ppm	<0.1 ppm	<0.1 ppm	-
CHROMATOGRAPH	5.5	99.9995%	NIT-55-XX	580	<1 ppm	<0.1 ppm	<1 ppm	<1 ppm	<1 ppm	-
UHP	5.0	99.999%	NIT-50-300	580	<1 ppm	<0.5 ppm	<1 ppm	-	-	-
ZERO	4.8	99.998%	NIT-ZE-300	580	-	<0.5	-	-	-	-
PREPURIFIED	4.8	99.998%	NIT-48-300	580	<5 ppm	-	<5 ppm	-	-	-
Oxygen Free	4.8	99.998%	NIT-OF-300	580	<0.5 ppm	-	-	-	-	-
NITROUS OXIDE										
ATOMIC ABSORPTION	2.6	99.6%	N2O-26-50	326	*	-	<30 ppm	-	-	*
* N2O-AA: AIR<2000 ppm										
OXYGEN										
RESEARCH***	5.0	99.999%	OXY-50-300	540	99.999%	<0.5 ppm	<1 ppm	<1 ppm	<5 ppm	-
UHP**	4.3	99.993%	OXY-43-300	540	99.993%	<0.5 ppm	<3 ppm	-	-	<10 ppm
ZERO	2.8	99.8%	OXY-28-300	540	99.8%	<0.5 ppm	-	-	-	-
EXTRA DRY	2.6	99.6%	OXY-26-300	540	99.6%	-	<10 ppm	-	-	-
					O2-UHP <40 ppm Ar	*O2-RESEARCH <5 ppm Ar				
ppm = Parts Per Million					ppb = Parts Per Billion			THC = Total Hydrocarbons		

Appendix B – Blank Canister Batch Cleaning Sheet



NORTH CAROLINA
Environmental Quality

Division of Air Quality

Laboratory Analysis Branch (LAB)

Canister Cleaning System 1 | 2 (circle one)

Canister Cleaning Record

Batch #: _____

Number of cycles: _____

Operator: _____

Date: _____

Batch Certification Canister #: _____

Canister Numbers of Canisters Cleaned

Procedure Checklist

- Both pumps ON
- Water in humidification chamber | DI Water (Optima grade) Lot Number: _____
- UHP Nitrogen supply OPEN | Cylinder Lot Number: _____
- Ambient Pressure is within 14.7 ± 0.5 psi | Ambient Pressure (psi): _____
- All canister valves OPEN
- System leak check passes
- Oven Temperature at set point (75°C)
- Final canister pressure: _____ mtorr

Level 1 (self) Data Review: _____

Level 2 (peer) Data Review: _____

DAQ-03-019.2 Revision 1
For use with SOP DAQ-03-005.2

Appendix C – LAB Canister Tag

NC DAQ LABORATORY ANALYSIS BRANCH

4403 Reedy Creek Road, Raleigh, NC 27607

SAMPLE CANISTER LABEL -- Use Black Ballpoint Ink Pen

Can Batch Number _____ Sample Number _____

Can Number _____ Sampling Site _____

Start Date _____ End Date _____

Start Pressure _____ End Pressure _____

COC Seal _____ Lab Number _____

Comments _____