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NORTH CAROLINA
Environmental Quality

DAQ-03-005.001 Standard Operating Procedure (SOP)
Entech 3100A Canister Cleaner
ECB RESPONSIBILITIES

Revision 0

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 April 22nd, 2021.

Director, Air Quality Division

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Signature: DocuSigned by: Michael Abraczinskas D5013FED0218423... Date: 4/28/2021

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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

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 Water

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 – Ultra high purity

USB – Universal Serial Bus

VOC – Volatile organic compound

Table of Contents

2.0 SCOPE AND PURPOSE

The scope and purpose of this SOP is to describe the steps required to successfully install and leak check 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 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°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 RPM to attain a 10-mtorr vacuum on the canisters. Clean ultra-high purity nitrogen is humidified by the Entech 3100A Canister Cleaning System and then introduced into the canisters to 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.

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
  - USB mouse and keyboard
- 6-L Silco-treated summa canisters or equivalent
- UHP Nitrogen (Arc3 Gases Grade 5.0 or equivalent – See Appendix A for gas purity information)
- UHP Helium (Arc3 Gases Grade 5.0 or equivalent)
- CGA580 Regulators
- Cylinder Transportation Cart
- Cylinder mount with straps (to secure to bench or wall)
- 1/8-in stainless-steel chromatographic grade tubing and compression fittings
- ¼-in Teflon or polypropylene tubing and compression fittings
- ¼-in stainless-steel chromatographic grade tubing and compression fittings
- 3/8-in stainless-steel chromatographic grade tubing and compression fittings
- Deionized water (HPLC Grade, Submicron filtered or equivalent)
- Deionized water filled squeeze bottle
- Entech 3100A Canister Cleaner Logbook
- 12 x ¼-in compression fitting male nut caps, brass, or stainless-steel
- ¼-in replacement ferrules (vespel-graphite or stainless steel)
- Agilent G3388B Electronic Leak Detector or equivalent
- Hand Tools:
  - 7/16-in wrench
  - 9/16-in wrench
  - 7/8-in wrench
  - 12-in adjustable wrench or larger
- PPE:
  - Heat resistant, textured grip gloves
  - 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 100 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 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 audibly heard.
2 Switch on the Entech 3100A 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 LED on the system front panel.

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.

3.2.4 System Leak Check

1 Cap and secure all system ports. The vacuum pressure is displayed on the top of the 3000/3100 Run Control Screen as depicted below under the "Vacuum (mtorr)" header as depicted in figure 4.

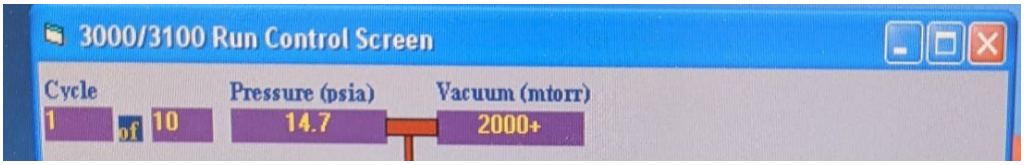

Figure 4: Entech 3100A Canister Cleaning System Pressure Read-back

2 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.
3 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.
4 The HV has been pre-set to 10 mtorr and the system should achieve it after several minutes of evacuation.
5 Once the system has achieved a HV of 10 mtorr, monitor the read-back for 1 minute. If no fluctuations occur the system is leak-free and the vacuum system is functioning as designed. If any of the above steps fail, see section 9.1 for corrective action.

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 Installation and Set up

1 Record the date, operator, and serial number of the Entech 3100A Canister Cleaner in the Entech 3100A Canister Cleaner Logbook.
2 Team lift the canister cleaning oven onto the laboratory bench and set it towards the end of the countertop.
3 Place the Entech 3100A canister cleaning system box next to the canister cleaning oven. The system box front panel is pictured below:

Figure 5: Entech 3100A Canister Cleaning System Box Front Panel

4 Place the computer, monitor and peripherals next to the Entech 3100A system box.
5 Connect all power and peripheral cables for the canister cleaning oven, system computer and Entech 3100A system box. The configuration should appear similar to what is shown in figure 1.
6 Connect the canister cleaning oven to the Entech 3100A system box with the supplied tubing and fittings.
7 Fill the humidification chamber reservoir by loosening the 7/8-in nut located on the rear of the Entech 3100A system box and removing the chamber. Add deionized 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 the lot number of the DI water added to the reservoir in the Entech 3100A Canister Cleaner Logbook.
8 Fill a DI water squeeze bottle and label the bottle with the lot number of the DI water inside. Place on the bench to be used with **DAQ-03-005.2 Entech Canister Cleaner Operation**.
9 Bench or wall mount a cylinder holder with straps.
10 Using a cylinder cart to transport, install a UHP nitrogen cylinder with a CGA580 regulator.
11 Connect the Entech 3100A canister cleaner to the UHP nitrogen cylinder regulator via the ¼-in fitting at the top of the humidification chamber.

12 Attach 1/8-in stainless steel tubing from the humidification chamber port to the "fill gas in" port on the back of the 3100A.
13 Connect the diaphragm pump to the ¼-in bulkhead labeled "rough pump" on the back the Entech 3100A system box using ¼-in Teflon or polypropylene tubing.
14 Open the UHP cylinder and set the regulator outlet pressure to 50 psi.
15 Boot and login to the system computer.
16 Double click the 3100A icon on the desktop to connect to the cleaning system.
17 The link active LED on the 3100A front panel will illuminate once connected.
18 Proceed to 5.2 System Leak Check.

5.2 System Leak Check

The 3100A canister cleaner must be leak checked after each installation to ensure leak-free operation. Leak checking without canisters is the manufacturer recommended method to check for leaks as detailed below:

1 Open the Entech 3100A Canister Cleaner oven doors and tighten ¼-in male compression nuts into the oven manifold. A best practice is one-quarter turn past finger tight to create a good compression fitting seal.
2 Turn on the rough pump switch manually and then turn on the HV pump switch on the front of the 3100A system.
3 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.
4 In the 3100A software load a canister method to access the Run/Control Screen. Select a method by clicking open on the top tab and a method menu will appear as shown in Figure 6:

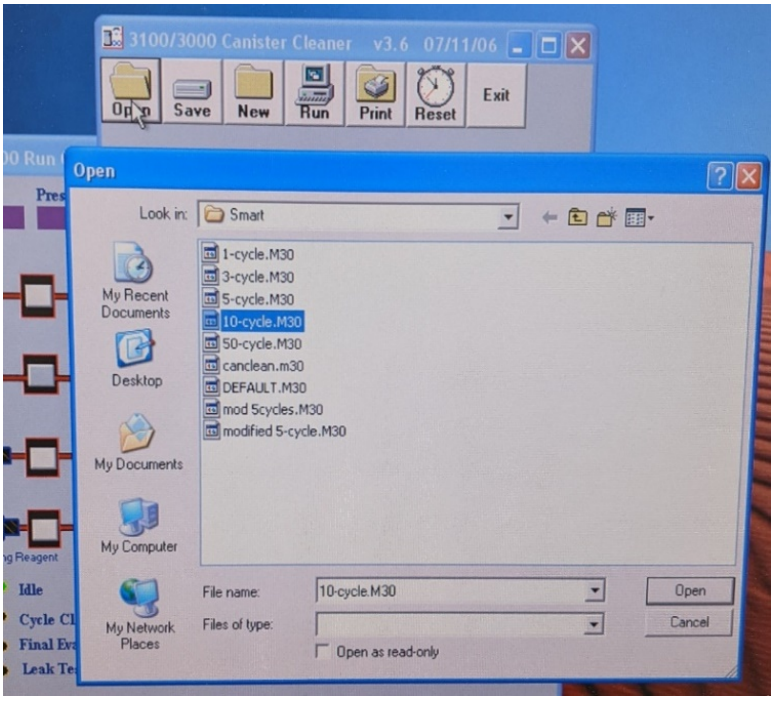

Figure 6: Entech 3100A Canister Cleaning System Method Selection

5 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 7:

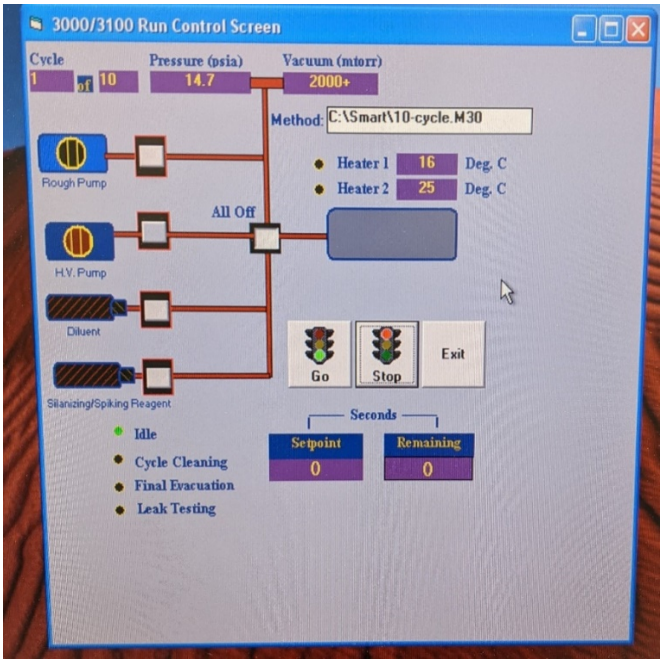

Figure 7: Entech 3100A Canister Cleaning System Run Screen

6 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.

7 The HV pump has been pre-set to draw the vacuum down to 10 mtorr and the system should achieve that vacuum after several minutes of evacuation.

8 Once the system has achieved a HV pump vacuum of 10 mtorr, monitor the read-back for 1 minute. If no fluctuations occur the system is leak-free. If any of the above steps fail, see section 9.1 for corrective action.

9 Click the "all off" button in the run screen to stop the vacuum system.

10 Record the passing installation leak check in the Entech 3100A Canister Cleaner Logbook.

6.0 ENTECH 3100A CANISTER CLEANER DATA REVIEW

This section of the SOP describes steps required for the ECB 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.

6.1 Self/Level 1 Canister Cleaner Logbook Entry Review (ECB Operator)

Review the Entech 3100A Canister Cleaner Logbook for completeness and accuracy. Initial and date the logbook entry to indicate a level 1 review has occurred and submit the logbook for secondary, level 2 peer review. A template logbook entry is available in appendix B. It is the responsibility of the primary, level 1, operator to find a suitable reviewer in a timely manner.

6.2 Peer/Level 2 Precision Diluter Data Review

Review the 3100A Canister Cleaner Logbook entry 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. Initial and date the logbook to indicate a level 2 peer review has occurred.

7.0 FILE MANAGEMENT

This section of the SOP describes the different files generated during the canister cleaner installation and the individual required to manage the file, either the ECB operator or level 2 reviewer, or both. Files include the Entech 3100A Canister Logbook Entry Template entered into the Entech 3100A Canister Cleaner Logbook.

7.1 Entech 3100A Precision Diluter Logbook Entry (ECB Operator)

The operator will store the completed Entech 3100A Canister Cleaner Logbook entry within the Entech 3100A Canister Cleaner Logbook. The template can be cut and pasted into the logbook. Initial over the interface of the logbook and the printed sheet of paper. Place a piece of tape over the signed interface of the logbook entry. Secure the entire entry to the logbook.

7.2 Entech 3100A Precision Diluter Logbook Entry (Peer/Level 2 Reviewer)

The operator will store the completed Entech 3100A Canister Cleaner Logbook Entry Template within the Entech 3100A Canister Cleaner Logbook. 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 Entech 3100A Canister Cleaner Logbook.

8.2 Quarterly Data Archival and Digitization

Printed records from each quarter will be scanned and archived on the P: network drive in the LAB folder.

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 ¼-in male nuts are tightened appropriately and that ferrules are in good condition. Replace any ferrules that are cracked, damaged, or warped.
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 ¼-in male nuts. Remove a ¼-in nut from the front of the oven manifold.

5 Fill an evacuated canister to about 30 psi with UHP helium and connect to the open port on the manifold.
6 Power on the Agilent G3388B electronic leak detector and allow it to warm up as shown below:

Figure 8: Agilent G3388B Leak Detector and Probe

The warmup procedure takes 90 seconds. Do not skip this step. The leak detector will beep once the warmup is complete. Gas can be heard audibly flowing through the meter while it is powered on.

7 Open the helium canister.
8 Using the electronic leak detector, snoop around all the oven manifold fittings to look for leaks. Wait about 10-15 seconds per point of interest to allow the gas to reach the leak detector. The detector will read R_LO and bars will fill the screen when a leak is found. The detector will also beep. Address any leaks found with the appropriate tool or parts. For parts and support contact support@entechinst.com.

10.0 REVISION HISTORY

1. Rev 0 – **BDV** 04/22/2021 Original Publication

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 – Entech 3100A Logbook Entry Template

Appendix A – Arc3 Purity Plus Specialty Gases Grade Specification Sheet

PurityPlus Specialty Gases

PURE GAS GRADE SPECIFICATIONS

PRODUCT	GRADE	PURITY	PART #	VALVE	CONTAMINATE LEVELS (UNLESS OTHERWISE NOTED AS %) 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	<0.1ppm	<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%	N20-26-50	326	*	-	<30 ppm	-	-	*
* N20-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 – Entech 3100A Logbook Entry Template

North Carolina Department of Environmental Quality, Division of Air Quality Laboratory

Entech 3100A Canister Cleaner Logbook Entry Template

Installation Date:	
Operator ID:	
Entech 4700 S/N:	
DI Water Lot Number:	

System Leak Check Pass? (Circle one) Pass Criteria: System reaches 10mtorr for 1 minute sustained	Y N

Level 1 Review:	

Level 2 Review:	