

ROY COOPER
Governor
DIONNE DELLA-GATTI
Secretary
MICHAEL A. ABRACZINSKAS
Director



DAQ-03-006.1 Standard Operating Procedure (SOP)
Entech 4700 Precision Diluter
ECB RESPONSIBILITIES

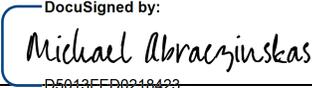
Revision 0

1.0 Approval Sign Off-Sheet

I certify that I have read and approve of the contents of the Entech 4700 Precision Diluter Standard Operating Procedure with an effective date of April 1st, 2021.

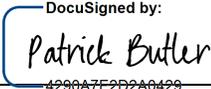
Director, Air Quality Division

Mike Abraczinskas

Signature:  Date: 4/7/2021

Ambient Monitoring Section Chief

Patrick Butler

Signature:  Date: 4/7/2021

Laboratory Analysis Branch Supervisor

Jim Bowyer, Environmental Program Supervisor

Signature:  Date: 4/7/2021

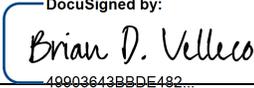
Projects and Procedures Branch Supervisor

Joette Steger, Environmental Program Supervisor

Signature:  Date: 4/6/2021

Primary SOP Author

Brian D. Velleco, Chemist I

Signature:  Date: 4/6/2021

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

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

COA – Certificate of Analysis

COC – Chain of custody

COM – Communication Port

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

in - inches

L – liters

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

LED – Light emitting diode

mbar – millibar

MDL – Method detection limit

NC – North Carolina

NIST – National institute of standards and technology

PAMS – Photochemical assessment monitoring station

PC – Personal computer

PDF – portable document format

PM – Particulate matter

PPB – Projects and Procedures Branch

ppb - parts per billion

PPE – Personal Protective Equipment

ppm – parts per million

psi – pounds per square inch

psi_A - pounds per square inch – absolute scale

PSIA - pounds per square inch – absolute scale

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

USB – Universal Serial Bus

μL - microliter

VOC – Volatile organic compound

Table of Contents

1.0	Approval Sign Off-Sheet.....	2
	SOP Acronym Glossary	3
2.0	SCOPE AND PURPOSE	7
3.0	EQUIPMENT CHECKS AND MATERIALS	10
3.1	Equipment and Material List.....	10
3.2	Chemical and Material Checks.....	11
3.3	Support Equipment Checks.....	11
3.3.1	Ultra Zero Air Cylinder Check	11
3.3.2	Analytical Gas Standard Cylinder Check.....	11
4.0	SITE CHECKS	12
5.0	DETAILED PROCEDURES.....	12
5.1	System Installation and Configuration.....	12
5.2	System Leak Check.....	16
5.3	Initial and Yearly Pressure Sensor Calibration	17
5.4	Entech 4700 Software Upgrades	19
6.0	DATA REVIEW.....	19
6.1	Self/Level 1 Precision Diluter Data Review	19
6.2	Peer/Level 2 Precision Diluter Data Review	20
7.0	FILE MANAGEMENT	20
7.1	4700 Precision Diluter Logbook Entry (Operator)	20
7.2	4700 Precision Diluter Logbook Entry (Peer/Level 2 Reviewer)	20
8.0	FILE QUALITY ASSURANCE AND DATA HANDLING.....	20
8.1	Records Retention	20
8.2	Quarterly Data Archival and Digitization	20
9.0	TROUBLESHOOTING AND CORRECTIVE ACTIONS.....	20
9.1	System Communication Errors	20
9.2	System Reads Negative Pressures Under Full Vacuum	21
9.3	Ambient Pressure does not Match NIST-Traceable Barometer	21
9.4	Failed Canister Leak Check.....	21
9.5	System Will Not Power On.....	21
10.0	REVISION HISTORY	21
11.0	REFERENCES.....	22
12.0	APPENDICES	22

Appendix A – Arc3 Purity Plus Specialty Gases Grade Specification Sheet.....23

Appendix B – Example Analytical Gas Standard Certificate of Analysis..... 24

Appendix C – NIST-Traceable Barometer and Calibration Certificate..... 26

Appendix D – Entech 4700 Precision Diluter Logbook Pressure Calibration Template 28

2.0 SCOPE AND PURPOSE

The scope and purpose of this SOP is to describe the steps required to successfully install and maintain the Entech 4700 Precision Diluter employed to create analytical gas-phase standards, QC samples and humidified blanks in 6-L treated Summa canisters for LAB instrumentation in support of UAT and PAMS monitoring. The Entech 4700 Precision Diluter includes proprietary high resolution pressure monitoring circuitry that measures and records every step of an automated dilution process, allowing for sub-ppm level dilutions from ppm level standards with high accuracy and precision.



Figure 1: Entech 4700 Precision Diluter



Figure 2: Entech Silonite Silco-Treated Summa 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 4700 Precision Diluter
- Computer and monitor with Windows 10 operating system installed with:
 - Entech 4700 Precision Diluter System software (v2.1.2.13 or later)
 - Adobe PDF Reader
 - Microsoft Office 365 Suite
 - USB mouse and keyboard
- Certified clean 6-L Silco-treated summa canisters or equivalent
 - Certification and cleaning process detailed in **SOP DAQ-03-005.2 Entech 3100A Canister Cleaner**
 - Example Canisters shown in Figures 2 and 3.
- Entech 4700 Precision Diluter Standard Preparation Binder
- Entech 4700 Precision Diluter Standard Preparation Logbook
- Ultra Zero Air (Arc3 Gases Grade 0.1 or equivalent – See Appendix A for gas purity information)
- Ultra High Purity Helium (Arc3 Gases Grade 5 or equivalent)
- Analytical Gas Standards (NIST-traceable, custom VOC blend)
- CGA590 Regulator
- CGA580 Regulator
- CGA350 Regulators (for custom VOC blend standards, one per standard)
- Cylinder Transportation Cart
- Cylinder Wall Mount Brackets (one per cylinder used)
- 2 x 1/4in compression fitting male nut caps, stainless steel
- 1/4-in compression fitting female nut cap, brass or stainless steel
- Hand Tools:
 - 9/16-in wrench
 - 1/2-in wrench
 - 7/16-in wrench
 - 12-in adjustable wrench or larger (for gas cylinder changes)
 - 1/4-in Die
 - Phillips-Head Screwdriver
 - Cordless Drill
 - Dremel Tool, Accessories and Workstation
 - Scissors
 - Adjustable Tubing Cutter
- PPE:
 - Gloves:
 - Nitrile
 - Textured grip, heat resistant work gloves

- Nonslip steel toed shoes or boots
- Safety glasses: prescription or impact resistant are acceptable
- 1/8-in Silonite tubing (to connect canisters and standards) and compression fittings
- 1/8-in Stainless Steel Chromatographic Grade tubing and compression fittings
- NIST-Traceable Barometer Fisher Scientific Traceable® or equivalent
- Agilent G3388B Electronic Leak Detector or equivalent
- Snoop Leak Detection Fluid
- Clear Tape
- Label Tape
- 2A replacement fuses

3.2 Chemical and Material Checks

All standards used in the Precision Diluter must be valid, non-expired, NIST-traceable VOC blends. Standards can be recertified by the vendor in lieu of buying a new standard as long as sufficient pressure remains. COAs and standard reference materials must be maintained by the operator with diligence as shown in appendix B. Standards are certified for a period determined by the vendor based on a specific VOC blend stability. Copies of the current cylinder inventory certificates of analyses must be stored in the back of the Entech 4700 Precision Diluter Standard Preparation Binder.

3.3 Support Equipment Checks

3.3.1 Ultra Zero Air Cylinder Check

- 1 Open the ultra-zero air cylinder and read the regulator pressure. The outlet pressure should be set to 50 psi.
- 2 If the cylinder pressure is below 100 psi or the cylinder is expired, change the cylinder by closing the cylinder and loosening the regulator with an adjustable wrench. **Note:** CGA590 cylinders are reversed threaded.
- 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. Gas purity information can be viewed in appendix A.
- 5 Record the lot and expiration date of the ultra-zero air cylinder in the 4700 Software configuration file as detailed in steps 6 and 7 of section 5.1.
- 6 Close the ultra-zero cylinder and regulator after Entech 4700 Precision Diluter operation is complete.

3.3.2 Analytical Gas Standard Cylinder Check

- 1 Open the standard cylinder of interest and read the regulator pressure. The outlet pressure should be set to 40 psi.
- 2 If the cylinder regulator gauge reads below 100 psi, then change to a new standard cylinder by closing the current cylinder and loosening the regulator with an adjustable wrench. If the standard has sufficient pressure remaining (<500 psi) and is approaching the expiration date, contact the manufacturer for recertification of the analytical standard. **Note:** CGA350 cylinders are reversed threaded.
- 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. Example gas standard certificates of analysis can be viewed in appendix B.
- 5 Record the name, lot and expiration date of the standard cylinder in the 4700 Software configuration file as detailed in steps 6 and 7 of section 5.1.
- 6 Close the standard cylinder and regulator after Entech 4700 Precision Diluter operation is complete.

3.3.3 4700 Precision Diluter Pressure Sensor Check (Ambient Conditions)

- 1 Remove the ¼-in nut from the outlet port on the front of the diluter and allow the pressure to equilibrate to ambient conditions for 30 seconds.
- 2 Compare the NIST-traceable local barometric pressure to “Current PSIA” reading in the top left corner of the 4700 Precision Diluter Software.
- 3 The Fisher Traceable® NIST-Traceable Barometer reads units of mbar and must be converted to psi_A and can be done with the following equation:

$$(1) \text{psi}_A = (\text{Ambient Pressure}_{\text{mbar}}) \left(\frac{14.50377 \text{psi}_A}{1 \text{bar}} \right) \left(\frac{1 \text{bar}}{1000 \text{mbar}} \right)$$

Report the result with the correct number of significant digits.

- 4 If the reading is within ± 0.1 psi_A of ambient pressure, the pressure calibration has been verified. Record this check in the Entech 4700 Precision Diluter logbook.
- 5 If the reading is out of specification, see section 9.3 for corrective action.

3.3.4 4700 Precision Diluter Pressure Sensor Check (Vacuum Conditions)

- 1 Remove the ¼-in nut from the outlet port on the front of the Precision Diluter and connect a fully evacuated canister run through **DAQ SOP-03-005.2**.
- 2 Open the canister and allow the pressure to equilibrate for 30 seconds.
- 3 Read the current pressure from “Current PSIA” in the top left corner of the 4700 Precision Diluter Software.
- 4 If the reading is within 0.03 psi_A of zero, the pressure calibration has been verified. Record this check in the Entech 4700 Precision Diluter logbook. **Note:** Negative values are never acceptable in an absolute measurement scale.
- 5 If the reading is out of specification, see section 9.2 for corrective action.
- 6 Close the canister and disconnect. Set it aside as a dirty, used canister.

4.0 SITE CHECKS

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

5.0 DETAILED PROCEDURES

5.1 System Installation and Configuration

- 1 Set the Entech 4700 Precision Diluter on an instrument bench, place it to the right hand side of the bench. Connect all power cords to the Precision Diluter and their respective outlets.
- 2 Setup the Entech 4700 Precision Diluter instrument computer to the left of the dilution system. Connect all peripheral equipment, network and power cords to their respective junctions.

- 3 Connect the Entech 4700 Precision Diluter to the system computer via the supplied USB Type-B cable.
- 4 Install wall brackets to the right of the dilution system to secure cylinders to the wall. Install one bracket per cylinder utilized on the system.
- 5 Carefully move the current analytical cylinder from inventory and ultra-zero air cylinder into place against the wall mounted brackets and secure them with straps. Install the correct regulators on the cylinders. **Note:** CGA350 and CGA590 cylinders are reversed threaded.
- 6 Using 1/8-in chromatographic grade stainless steel tubing, connect the ultra-zero air cylinder to the rear of the 4700 Precision Diluter. This is the diluent gas and is connected to channel 1. The rear panel of the Entech 4700 is depicted below from the Entech 4700 Precision Diluter Manual V2.0:

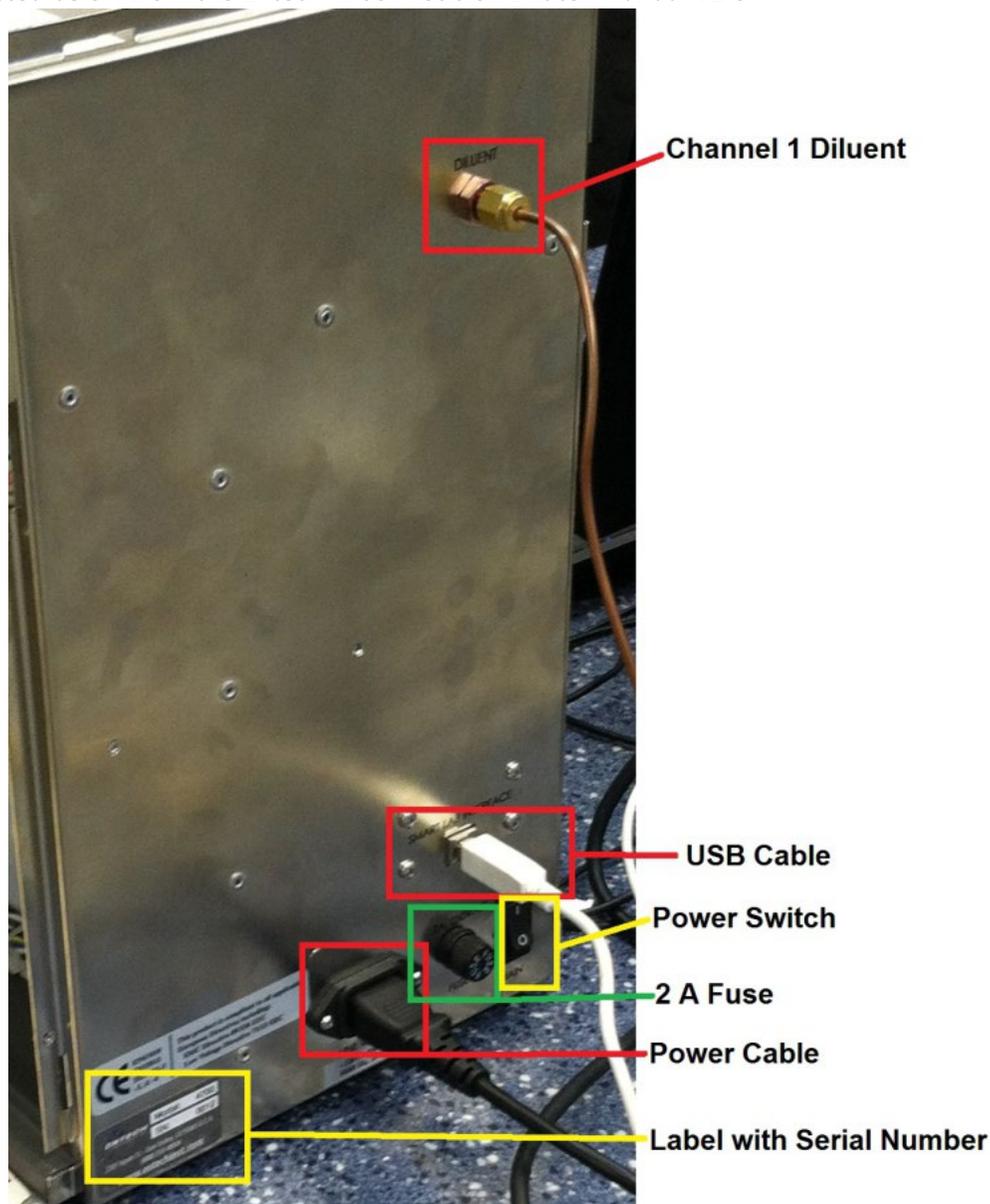


Figure 4: Rear of Entech 4700 Precision Diluter

- 7 Open the cylinder and regulator. Set the outlet pressure to 50 psi.

- 8 Using the supplied silonite treated 1/8-in tubing and fittings, connect each standard regulator to the side ports of the Entech 4700 Precision Diluter. Standards are connected to color coded ports, 2-5, on the right hand side with supplied quick connectors as depicted below from the Entech 4700 Precision Diluter Manual V2.0:

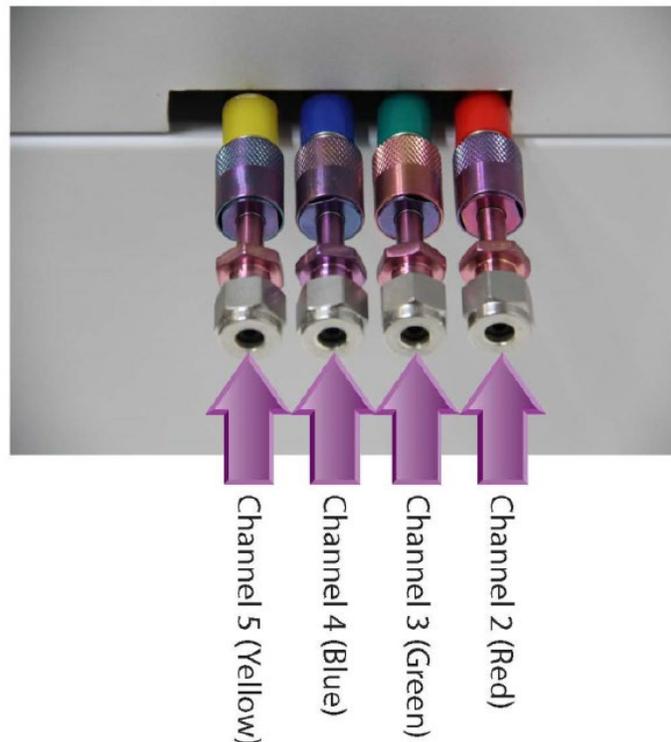


Figure 5: Entech 4700 Precision Diluter Gas Channels 2-5

- 9 To use the quick-connect, grab the female end and push it in towards the diluter. The male end will slide out. Simply reinsert to reestablish a connection.
- 10 Open the standard cylinder and regulator. Set the outlet pressure to 40 psi. Snoop for leaks. Slow leaks in standards over long periods of time can cause expensive (~\$10,000) custom VOC blends to be depleted. Close cylinders and regulators after pressure leak testing. **Note:** Only silonite inertly treated tubing is to be used to connect standards and canisters to the Entech 4700 Precision Diluter.
- 11 Connect the provided 1/8-in silonite line with 1/4-in adapter fittings to the outlet port and channel 6 re-dilute port on the front of the Entech 4700 Precision Diluter. Swage all fittings into place.
- 12 Power on all equipment, the physical set up of the unit should look similar to Figure 1 and 4.
- 13 Log into the Precision Diluter system computer.
- 14 Using the supplied Entech 4700 installation media, install version 2.1.2.13 of the Entech 4700 Precision Diluter software. Windows install shield will guide the operator through the installation and allow customization of the install directory. It is suggested that all windows updates be run until up-to-date status has been achieved prior to the installation of the Entech 4700 Precision Diluter software. **Note:** Administrative rights are required for this step. Also, Windows will only create a shortcut for the operator account profile. Each subsequent user will have to go to the install directory and create a shortcut to the "Entech4700RF.exe." The shortcut should be renamed to "Entech 4700 2.1.2.13" and placed on the user desktop. It is the responsibility of the ECB

operator to ensure that the LAB personnel involved in Precision Diluter operations have access to the software.

- 15 Run the newly installed Entech 4700 2.1.2.13 desktop shortcut and connect to the instrument. The link active LED on the 4700 front panel will illuminate once connected. See section 9.1 for communications troubleshooting.
- 16 In the Entech 4700 Precision Diluter Software, click to the “Settings” gear and wrench icon in the top menu bar. Click the “override” tab to display the current COM port. Record this port for reference as shown below:

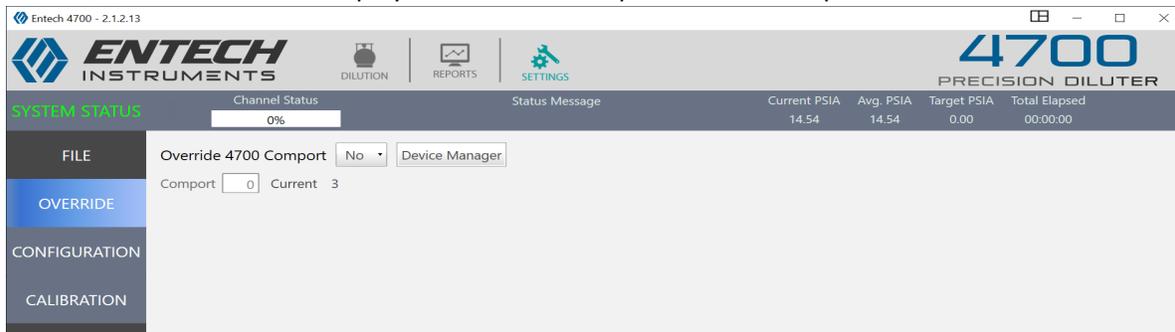


Figure 6: Entech 4700 Precision Diluter COM Port Setting

- 17 Click the “configuration” tab to set up the cylinder inventory for the laboratory.
- 18 Enter the ultra-zero air cylinder lot number into the diluent “Cylinder ID” column. Enter the date hooked up to the instrument as the “Date Rcvd.” and enter the expiration date in the “Expires” column.
- 19 Enter the standard name into the “Title” column of the desired channel. Enter the nominal concentration value of the standard into the “Init. Conc.” field. Select the appropriate units for this concentration from the drop down menu in the “Conc. Level” column. Enter the cylinder number into the “Cylinder ID,” the cylinder certification date into the “Date Rcvd.” Column and the expiration date into the “Expires” column. Repeat this step for each cylinder equipped to the instrument. Any notes about the standard can be put into the “Notes” column. All information from this step can be gleaned from the accompanying certificate of analysis as depicted in Appendix B. An example of the LAB cylinder inventory is shown below in Figure 7:

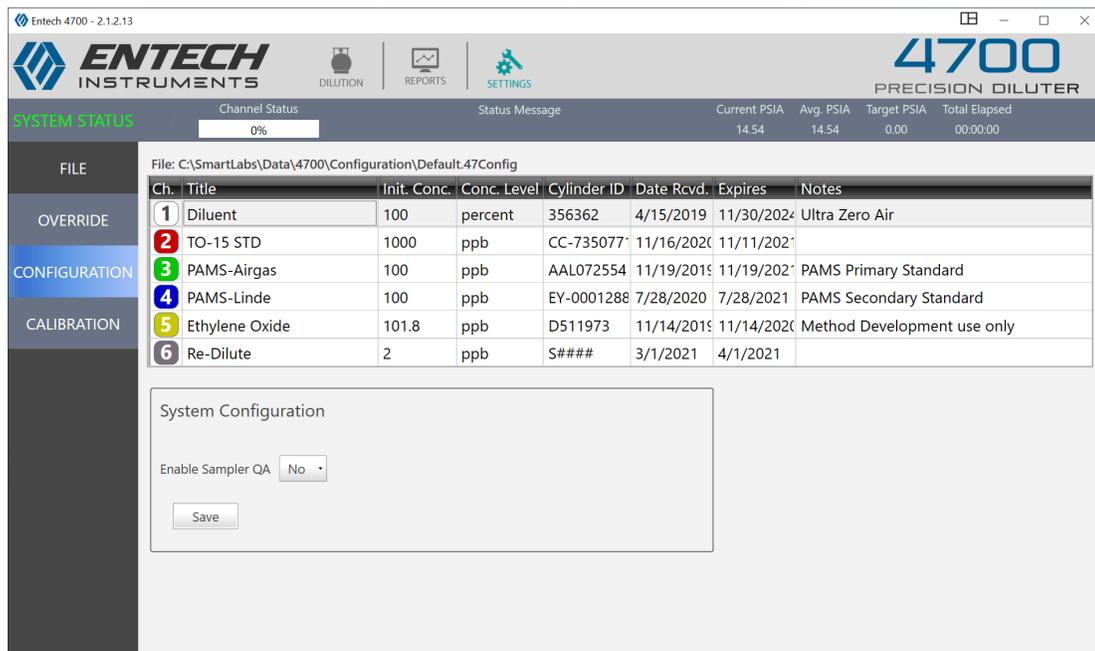


Figure 7: Entech 4700 Precision Diluter Cylinder Inventory Configuration

- 20 Click on the file tab of the left hand menu and select "save configuration." Overwrite the existing configuration file.
- 21 Place the Fisherbrand Traceable® NIST-traceable barometer on top of the Entech 4700 Precision Diluter. Place a copy of the calibration certificate into the rear of the Entech 4700 Precision Diluter Binder. An example barometer and calibration certificate is shown in Appendix C.
- 22 The system is now installed and ready for a system leak check as described in section 5.2.

5.2 System Leak Check

- 1 Fill an empty canister with 20-30 psi of helium and connect it to the Entech 4700 Precision Diluter outlet port.
- 2 Remove the cover of the Entech 4700 Precision Diluter to expose the flow path as depicted below:

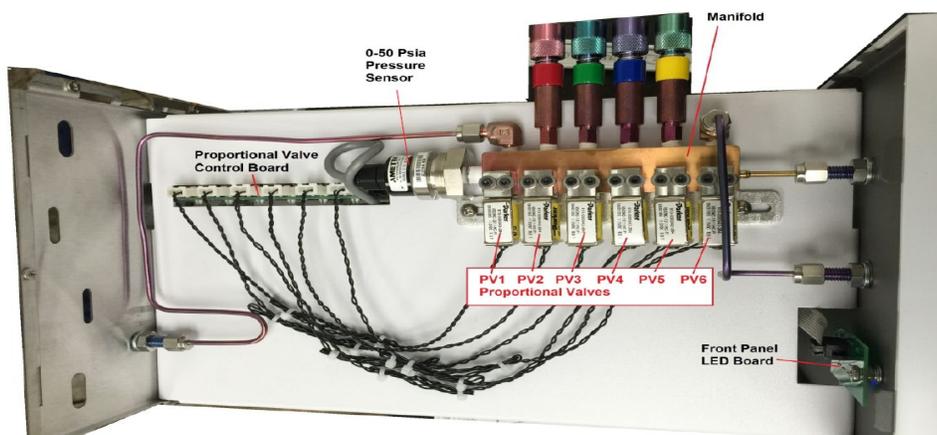


Figure 8: Entech 4700 Precision Diluter Top Panel Interior

- 3 Power on the Agilent G3388B electronic leak detector and allow it to warm up as shown below:



Figure 9: 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.

- 4 Open the helium canister and observe the "Current PSIA" value increase and stabilize to its filled value for 30 seconds.
- 5 Using the electronic leak detector, snoop around all the fittings, manifold and pressure sensor to look for leaks. Wait about 10-15seconds 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. For part support contact support@entechinst.com.

5.3 Initial and Yearly Pressure Sensor Calibration

- 1 In the top of the software window, select the "settings" wrench and gear icon to begin calibrating the 4700 Precision Diluter pressure sensor. The following window will appear after selecting "Calibration" from the left hand menu:

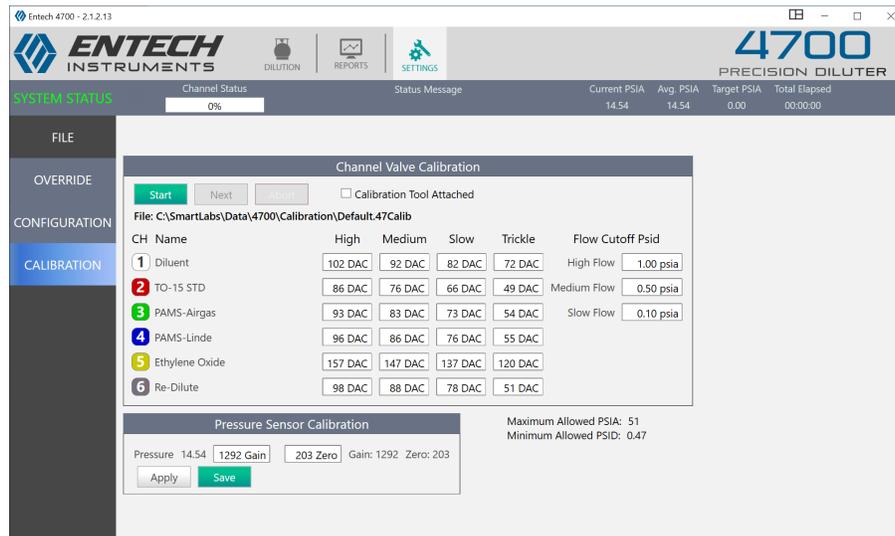


Figure 10: Entech 4700 Precision Diluter Cylinder Inventory Configuration

- 2 Remove the ¼-in nut from the outlet port and allow the pressure to equilibrate for 30 seconds
- 3 In the Entech 4700 Precision Diluter Logbook record the current pressure sensor calibration information: the current pressure reading, the gain value, and the zero-point value.
- 4 Using the NIST-Traceable barometer, record the conditions of the laboratory in the Entech 4700 Precision Diluter Logbook. Record the serial number and calibration expiration date of the barometer in the logbook.
- 5 Connect a fully evacuated canister run through **DAQ SOP-03-005.2**. Record the canister number and open the canister. Allow the pressure to equilibrate for 30 seconds. Note the stability of the “Current PSIA” reading. If the canister leaks above 0.5 psi_A over the 30 seconds, the system needs to be checked for leaks. **Note:** The canister must be at high vacuum for this step. Do not attempt a system calibration with a canister that is not near true zero.
- 6 Record the “Current PSIA” value of the canister as the pre-calibration “Current PSIA” reading for the vacuum canister.
- 7 If the canister is not within 0.03 psi_A of zero or if the absolute pressure is negative, simply enter a new value in the “zero” box of the pressure sensor calibration field and hit apply to send it to the Entech 4700 Precision Diluter. Adjust the zero calibration point until zero is attained. Record the “Current PSIA” of the canister as the Post-Calibration Adjustment “Current PSIA” reading of the vacuum canister. Record the new zero point for the final pressure sensor calibration. Otherwise, leave the calibration unchanged and record the initial zero point as the final zero point.
- 8 Close the canister and disconnect it from the Precision Diluter. Set it aside as a used canister.
- 9 Allow the instrument to equilibrate to ambient pressure for 30 seconds and record the “Current PSIA” reading as the pre-calibration “Current PSIA” reading for the barometric/ambient pressure.
- 10 Compare the result against the NIST-traceable barometer. If it is not within ±0.1 psi_A, adjust the gain of the pressure sensor calibration. Hit apply to send the new value to the precision diluter. Repeat until the reading is within specification. Record the “Current PSIA” of the ambient pressure as the Post-Calibration Adjustment “Current PSIA” reading of the ambient/barometric pressure. Record the new gain point for the final pressure sensor calibration. Otherwise, leave the calibration unchanged and record the initial gain point as the final gain point.

- 11 Click the green save button in the pressure sensor calibration box to save the calibration. The pressure sensor calibration is valid for one-year unless the operator can no longer verify the ambient pressure reading during normal operation as described in **SOP DAQ-03-006.2**.
- 12 Place a sticker on the top cover of the Entech 4700 Precision Diluter with the calibration date and the calibration expiration date (or calibration due date).

5.4 Entech 4700 Software Upgrades

- 1 Periodically the manufacturer will distribute software revisions. They can be obtained via two means: 1) register for an account at www.entechinstant.com and securely download or 2) contact support@entechinstant.com and provide the serial number of the unit to be securely provided the file via Microsoft SharePoint.
- 2 Close the Entech 4700 software and install the new software revision. Follow the Windows install wizard prompts. **Note:** This will require administrative rights.
- 3 The software will appear in the same installation directory as the previous revision as shown below:

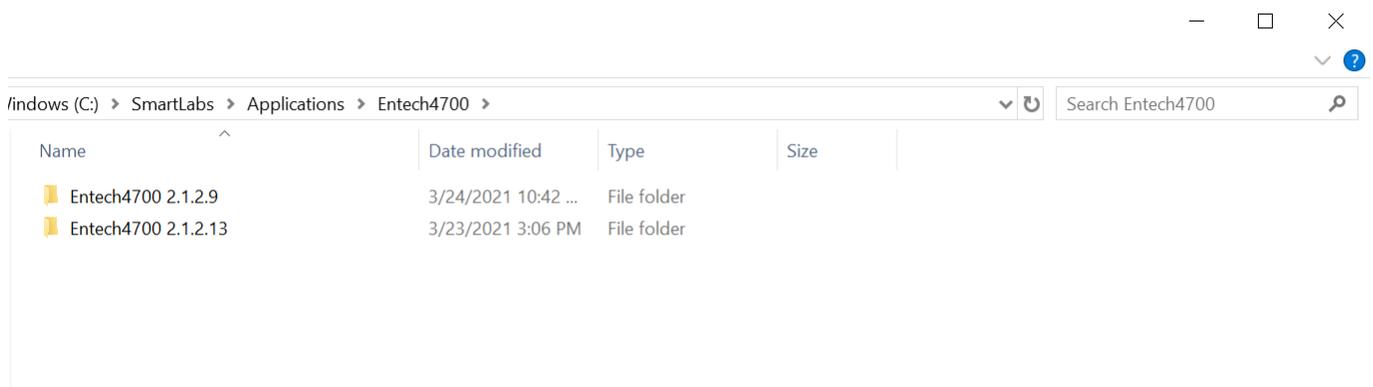


Figure 11: Entech 4700 Precision Diluter Software Directory

- 4 Open the newly installed revision folder and create a shortcut to the “Entech4700RF.exe” file on the desktop if one did not appear. Rename the shortcut to “Entech 4700 Version Number” (e.g. Entech 4700 2.1.2.13 for the version updated in Figure 11). Other users will have to make their own desktop shortcut and the ECB operator should work with LAB staff involved with Entech 4700 Precision Diluter operation to ensure the appropriate software revision is running on their individual user account.
- 5 Record the update in the Entech 4700 Precision Diluter Logbook.

6.0 DATA REVIEW

This section of the SOP describes steps required for the operator to perform a self/Level 1 review on the precision diluter system data. Additionally, this section describes steps required for LAB personnel not directly involved in the precision diluter operation process to perform a peer/Level 2 data review.

6.1 Self/Level 1 Precision Diluter Data Review

Review the Entech 4700 Precision Diluter Logbook entry for the pressure sensor calibration 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 calibration form is available in appendix D. 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 4700 Precision Diluter 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 standards preparation report and 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 precision dilution process and the individual required to manage the file, either the operator or level 2 reviewer, or both. Files include the Entech 4700 Precision Diluter Pressure Calibration template entered into the Entech 4700 Precision Diluter Logbook.

7.1 4700 Precision Diluter Logbook Entry (Operator)

The operator will store the completed Entech 4700 Precision Diluter Pressure Calibration Template within the Entech 4700 Precision Diluter 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 4700 Precision Diluter Logbook Entry (Peer/Level 2 Reviewer)

The operator will store the completed Entech 4700 Precision Diluter Pressure Calibration Template within the Entech 4700 Precision Diluter 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 4700 Precision Diluter logbook.

8.2 Quarterly Data Archival and Digitization

Printed and reviewed 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 Communication Errors

If the Entech 4700 does not connect to the PC, power off both the PC and the Entech 4700. Wait a couple minutes and power on both devices. Log in and attempt to reestablish a connection. If that fails, check the device manager for COM port settings. The software can be reinstalled using the steps in section 5.1. Lastly, replace the

USB cable connecting the diluter to the computer. It is a standard USB Type-B cable. If none of these steps rectify the communications, contact Entech support at <https://www.entechnst.com/global-support-home/>. **Note:** Administrative rights are required to control components in device manager or reinstall software.

9.2 System Reads Negative Pressures Under Full Vacuum

The Entech 4700 Precision Diluter uses an absolute psi pressure scale and should not read negative values. Perform a pressure sensor calibration if a canister under vacuum yields a negative pressure reading when opened on the dilution system. These steps are detailed in section 5.3.

9.3 Ambient Pressure does not Match NIST-Traceable Barometer

If the Entech 4700 Precision Diluter no longer accurately measures the ambient pressure of the laboratory, perform a pressure sensor calibration. These steps are detailed in section 5.3.

9.4 Failed Canister Leak Check

Begin by tightening the fittings on the output line. Make sure they are swaged into place, but not overtightened. Inspect the ferrules and line for damage and deformation. Replace the ferrule or line if it is damaged. A Dremel tool will be required to remove the metal ferrule. Silonite tubing is very expensive per foot and it is preferable to replace ferrules and cut tubing to fix leaks instead of replacing an entire run of silonite tubing. It is also worth inspecting the threads of the canister attached to the system. If they are in rough condition, run a ¼-in die over the threads to repair them. Attempt a leak check again, if it passes it is okay to proceed to normal operation in SOP DAQ-03-006.2. Leak checking as described in section 5.2 can be repeated if none of the above steps yield a passing leak check. For further support contact support@entechnst.com.

9.5 System Will Not Power On

If the 4700 Precision Diluter fails to power on, the most likely culprit is the 2A fuse located on the rear of the unit. Unplug the rear power cable. The fuse holder is located next to the power cable receptacle. Push in and turn to slide the fuse out. Replace the 2A fuse. Reinstall the fuse into the unit and reconnect the power. Power on the Entech 4700 Precision Diluter to test the replacement fuse. If the unit continues to consume fuses after replacement or continues to not power on, contact support@entechnst.com for further support.

10.0 REVISION HISTORY

1. Rev 0 – BDV 04/01/2021 Original Publication

11.0 REFERENCES

1. Entech Instruments (2018) 4700 Precision Diluter: Manual Version 2.0. Simi Valley, CA. Entech Instruments & Tim Raub.
2. CRC Press (2014-2015) CRC Handbook of Chemistry and Physics: 95th Edition. Bacon Raton, FL. W.M Hanyes, Ph.D, David R. Lide, Ph.D and Thomas J. Bruno, Ph.D. (Pg. 1-39).

12.0 APPENDICES

1. Appendix A – Arc3 Purity Plus Specialty Gases Grade Specification Sheet
2. Appendix B – Example Analytical Gas Standard Certificate of Analysis
3. Appendix C – NIST-Traceable Barometer and Calibration Certificate
4. Appendix D – Entech 4700 Precision Diluter Logbook Pressure Calibration Template

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 – Example Analytical Gas Standard Certificate of Analysis



Airgas Specialty Gases
Airgas USA, LLC
6141 Easton Road
Bldg 1
Plumsteadville, PA 18949
Airgas.com

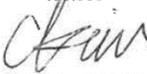
CERTIFICATE OF ANALYSIS**Grade of Product: CERTIFIED HYDROCARBON**

Part Number:	X57N199C15AC003	Reference Number:	160-401659192-1
Cylinder Number:	AAL072554	Cylinder Volume:	144.3 CF
Laboratory:	124 - Plumsteadville - PA	Cylinder Pressure:	2050 PSIG
Analysis Date:	Nov 19, 2019	Valve Outlet:	350SS
Lot Number:	160-401659192-1	Expiration Date:	Nov 19, 2021

Traceability Statement: Hydrocarbon Process standards are NIST traceable either directly by weight or by comparison to Airgas laboratory standards that are directly NIST traceable by weight.

CERTIFIED CONCENTRATIONS

Component	Requested Concentration	Reported Mole %	Accuracy
1 BUTENE	100.000 PPB	108.000 PPB	+/- 10%
1 HEXENE	100.000 PPB	98.000 PPB	+/- 10%
1 PENTENE	100.000 PPB	99.000 PPB	+/- 10%
1,2,3 TRIMETHYL BENZENE	100.000 PPB	105.000 PPB	+/- 10%
1,2,4 TRIMETHYLBENZENE	100.000 PPB	104.000 PPB	+/- 10%
1,3 DIETHYLBENZENE	100.000 PPB	97.000 PPB	+/- 10%
1,3,5 TRIMETHYL BENZENE	100.000 PPB	100.000 PPB	+/- 10%
1,4 DIETHYL BENZENE	100.000 PPB	99.000 PPB	+/- 10%
2 ETHYLTOLUENE	100.000 PPB	104.000 PPB	+/- 10%
2 METHYL PENTANE	100.000 PPB	106.000 PPB	+/- 10%
2 METHYLHEPTANE	100.000 PPB	99.000 PPB	+/- 10%
2 METHYLHEXANE	100.000 PPB	102.000 PPB	+/- 10%
2,2 DIMETHYLBUTANE	100.000 PPB	101.000 PPB	+/- 10%
2,3 DIMETHYL BUTANE	100.000 PPB	104.000 PPB	+/- 10%
2,3 DIMETHYL PENTANE	100.000 PPB	100.000 PPB	+/- 10%
2,3,4 TRIMETHYL PENTANE	100.000 PPB	99.000 PPB	+/- 10%
2,4 DIMETHYL PENTANE	100.000 PPB	102.000 PPB	+/- 10%
3 ETHYLTOLUENE	100.000 PPB	99.000 PPB	+/- 10%
3 METHYL PENTANE	100.000 PPB	105.000 PPB	+/- 10%
3 METHYLHEPTANE	100.000 PPB	102.000 PPB	+/- 10%
3 METHYLHEXANE	100.000 PPB	102.000 PPB	+/- 10%
4 ETHYL TOLUENE	100.000 PPB	95.000 PPB	+/- 10%
ACETYLENE	100.000 PPB	101.000 PPB	+/- 10%
BENZENE	100.000 PPB	100.000 PPB	+/- 10%
CIS 2 BUTENE	100.000 PPB	108.000 PPB	+/- 10%
CIS 2 PENTENE	100.000 PPB	98.000 PPB	+/- 10%
CUMENE	100.000 PPB	96.000 PPB	+/- 10%
CYCLOHEXANE	100.000 PPB	103.000 PPB	+/- 10%
CYCLOPENTANE	100.000 PPB	95.000 PPB	+/- 10%
DODECANE	100.000 PPB	103.000 PPB	+/- 10%
ETHANE	100.000 PPB	107.000 PPB	+/- 10%
ETHYL BENZENE	100.000 PPB	101.000 PPB	+/- 10%
ETHYLENE	100.000 PPB	111.000 PPB	+/- 10%
HEXANE	100.000 PPB	105.000 PPB	+/- 10%
ISOBUTANE	100.000 PPB	108.000 PPB	+/- 10%
ISOOCTANE	100.000 PPB	100.000 PPB	+/- 10%
ISOPENTANE	100.000 PPB	96.000 PPB	+/- 10%
ISOPRENE	100.000 PPB	95.000 PPB	+/- 10%
M,P XYLENE	100.000 PPB	102.000 PPB	+/- 10%
METHYL CYCLOHEXANE	100.000 PPB	102.000 PPB	+/- 10%
METHYL CYCLOPENTANE	100.000 PPB	106.000 PPB	+/- 10%
N BUTANE	100.000 PPB	107.000 PPB	+/- 10%
N DECANE	100.000 PPB	104.000 PPB	+/- 10%


Approved for Release

Page 1 of 160-401659192-1



Airgas Specialty Gases
Airgas USA, LLC
6141 Easton Road
Bldg 1
Plumsteadville, PA 18949
Airgas.com

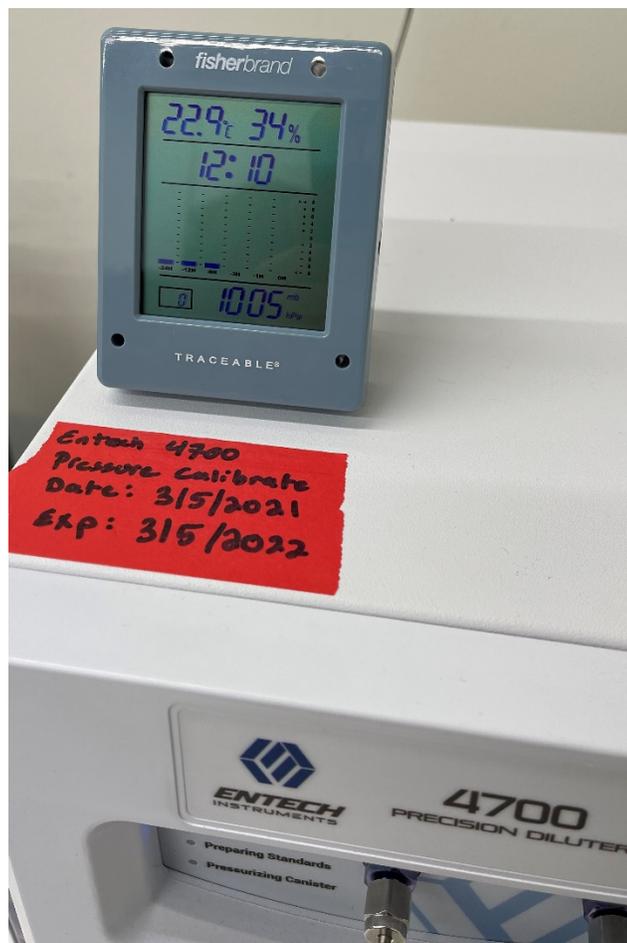
N HEPTANE	100.000 PPB	96.000 PPB	+/- 10%
N NONANE	100.000 PPB	102.000 PPB	+/- 10%
N OCTANE	100.000 PPB	101.000 PPB	+/- 10%
N PENTANE	100.000 PPB	101.000 PPB	+/- 10%
N PROPYL BENZENE	100.000 PPB	101.000 PPB	+/- 10%
O XYLENE	100.000 PPB	100.000 PPB	+/- 10%
PROPANE	100.000 PPB	109.000 PPB	+/- 10%
PROPYLENE	100.000 PPB	107.000 PPB	+/- 10%
STYRENE	100.000 PPB	99.000 PPB	+/- 10%
TOLUENE	100.000 PPB	106.000 PPB	+/- 10%
TRANS 2 BUTENE	100.000 PPB	106.000 PPB	+/- 10%
TRANS 2 PENTENE	100.000 PPB	101.000 PPB	+/- 10%
UNDECANE	100.000 PPB	103.000 PPB	+/- 10%
NITROGEN	100.0 %	99.999429 %	+/- 2%

Permanent Notes:STD TO MIX - PAMS

Approved for Release

Page 2 of 160-401659192-1

Appendix C – NIST-Traceable Barometer and Calibration Certificate



Fisherbrand Traceable® NIST-Traceable Barometer



Calibration complies with ISO/IEC
17025, ANSI/NCSL Z540-1, and 9001



Cert. No.: 6530-11810931

Traceable® Certificate of Calibration for Digital Barometer

Manufactured for and distributed by : Fisher Scientific "300 Industry Drive,,Pittsburgh,PA,15275-1001"

Instrument Identification:

Model: 14-650-118,15557603

S/N: 200810884

Manufacturer: Control Company

Standards/Equipment:

Description	Serial Number	Due Date	NIST Traceable Reference
Digital Barometer	D4540001	19 Nov 2021	1000461277
Digital Thermometer	130070752	10 Mar 2021	4000-11170557
Chilled Mirror Hygrometer	44654/2H3737	25 Nov 2021	17811
Climate Chamber	W619.0019		

Certificate Information:

Technician: 57

Procedure: CAL-31

Cal Date: 16 Dec 2020

Cal Due Date: 16 Dec 2022

Test Conditions: 44.5%RH 22.06°C 1021mBar

Calibration Data: (New Instrument)

Unit(s)	Nominal	As Found	In Tol	Nominal	As Left	In Tol	Min	Max	±U	TUR
%RH	N.A.	N.A.		50.48	51	Y	47	53	0.74	>4:1
°C	N.A.	N.A.		25.21	25.1	Y	24.81	25.61	0.05	>4:1
mb/hPa	N.A.	N.A.		805.60	806	Y	802	810	0.62	>4:1
mb/hPa	N.A.	N.A.		910.40	911	Y	906	914	0.62	>4:1
mb/hPa	N.A.	N.A.		1011.77	1012	Y	1008	1016	0.62	>4:1

This certificate indicates Traceability to standards provided by (NIST) National Institute of Standards and Technology and/or a National Standards Laboratory.

A Test Uncertainty Ratio of at least 4:1 is maintained unless otherwise stated and is calculated using the expanded measurement uncertainty. Uncertainty evaluation includes the instrument under test and is calculated in accordance with the ISO *Guide to the Expression of Uncertainty in Measurement : (GUM). The uncertainty represents an expanded uncertainty using a coverage factor k=2 to approximate a 95% confidence level. In tolerance conditions are based on test results falling within specified limits with no reduction by the uncertainty of the measurement. The results contained herein relate only to the item calibrated. This certificate shall not be reproduced except in full, without written approval of Control Company.

Nominal=Standard's Reading; As Left=Instrument's Reading; In Tol=In Tolerance; Min/Max=Acceptance Range; ± U=Expanded Measurement Uncertainty; TUR=Test Uncertainty Ratio; Accuracy= $\pm(\text{Max}-\text{Min})/2$; Min=As Left Nominal(Rounded) - Tolerance; Max= As Left Nominal(Rounded) + Tolerance;

Nicol Rodriguez

Nicol Rodriguez, Quality Manager

Marisa Elms

Marisa Elms, Technical Manager

Note :

Maintaining Accuracy:

In our opinion once calibrated your Digital Barometer should maintain its accuracy. There is no exact way to determine how long calibration will be maintained. Digital Barometer change little, if any at all, but can be affected by aging, temperature, shock, and contamination.

Recalibration:

For factory calibration and re-certification traceable to National Institute of Standards and Technology contact Control Company.

Issue Date : 16 Dec 2020

CONTROL COMPANY 12554 Galveston RD Suite B230 Webster TX USA 77598
Phone 281 482-1714 Fax 281 482-9448 sales@control3.com www.traceable.com

Control Company is an ISO/IEC 17025:2017 Calibration Laboratory Accredited by (A2LA) American Association for Laboratory Accreditation, Certificate No. 1750.01.
Control Company is ISO 9001:2015 Quality Certified by DNV GL, Certificate No. CERT-01805-2006-AQ-HOU-ANAB.
International Laboratory Accreditation Cooperation - Multilateral Recognition Arrangement (ILAC-MRA).

Appendix D – Entech 4700 Precision Diluter Logbook Pressure Calibration Template

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

Entech 4700 Precision Diluter Logbook Pressure Calibration Template			
Date:			
Operator ID:			
Entech 4700 S/N:	0316		
Initial Pressure Sensor Calibration			
Gain	Zero		
	Pre-Calibration "Current PSIA" Reading	Post-Calibration Adjustment "Current PSIA" Reading	
Vacuum Canister (psi _A)			
Barometric/Ambient Pressure (psi _A)			
‡ Barometric Pressure is converted from mbar to psi _A via the following formula: $psi_A = (Ambient\ Pressure_{mbar}) (14.50377 psi_A / 1bar) (1bar / 1000mbar)$			
NIST-Traceable Barometer S/N:		200810884	
Barometer Expiration Date:		12/16/2021	
Canister Number:			
Laboratory Conditions			
Temperature (°C)			
Relative Humidity (%RH)			
Barometric Pressure (mbar)			
Barometric Pressure (psi _A) [‡]			
Final Pressure Sensor Calibration			
Gain		Zero	
Calibration Expiration Date:			
Level 1 Review:			
Level 2 Review:			