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



DAQ-03-006.2 Standard Operating Procedure (SOP) Entech 4700 Precision Diluter OPERATOR 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: Decusigned by: <u>Michael Abracyinskas</u>	Date:
Ambient Monitoring Section Chief	
Patrick Butler	
Signature:	Date:
Laboratory Analysis Branch Supervisor	
Jim Bowyer, Environmental Program Supervisor	
Signature:	4/7/2021 Date:
Projects and Procedures Branch Supervisor	
Joette Steger, Environmental Program Supervisor	
Signature:	Date:
Primary SOP Author	
Brian D. Velleco, Chemist I	
Signature:	Date:
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.

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SOP Acronym Glossary

- ADQ Audit of data quality
- AQS Air Quality System (EPA's Air database)
- °C degrees Celsius
- 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
- HPLC High Performance Liquid Chromatography
- 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
- mL milliliter
- NC North Carolina
- NIST National institute of standards and technology
- PAMS Photochemical assessment monitoring station
- PC Personal computer

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- 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
- μ L microliter
- 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 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

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Figure 2: Entech Silonite Silco-Treated Summa 6L Canister

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Figure 3: Restek Silco-Treated SilcoCan 6L 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
 - o 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 Purity Information)
- Ultra High Purity Helium (Arc3 Gasses 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
- Deionized water (HPLC Grade, Submicron filtered or equivalent)
- Clean 30-mL Beaker
- 100-µL Hamilton gas-tight glass syringe or equivalent
- 2 x ¼-in compression fitting male nut caps, stainless steel
- ¼-in compression fitting female nut cap, brass or stainless steel
- Hand Tools:
 - o 9/16-in wrench
 - o ½-in wrench
 - o 7/16-in wrench
 - o 12-in adjustable wrench or larger (for gas cylinder changes)
 - o ¼-in Die
 - Phillips-head Screwdriver
 - o Dremel Tool, Accessories and Workstation
 - Scissors
- PPE:
 - Gloves:
 - Nitrile
 - Textured grip, heat resistant work gloves
 - Nonslip steel toed shoes or boots

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- Safety glasses: prescription or impact resistant are acceptable
- 1/8-in Silonite tubing (to connect canisters and standards) and fittings
- NIST-Traceable Barometer Fisher Scientific Traceable[®] or equivalent
- Agilent G3388B Electronic Leak Detector or equivalent
- 2A replacement fuse

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 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.
- 5 Record the lot and expiration date of the ultra-zero air cylinder in the Entech 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 regulator pressure gauge for the cylinder 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.
- 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

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 the conversion can be done with the following equation:

(1)
$$psi_A = (Ambient \ Pressure_{mbar}) \left(\frac{14.50377 psi_A}{1bar}\right) \left(\frac{1bar}{1000 mbar}\right)$$

Report the result with the correct number of significant digits.

- 4 If the reading is within ± 0.1 psi_A of ambient pressure, it is okay to proceed with a dilution. Record this check in the Entech 4700 Precision Diluter logbook.
- 5 If the reading is out of specifications, see section 9.4 for corrective action.

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 Start Up and Preparation

- 1 Boot, if required, and login to the precision diluter system computer.
- 2 Press the power button on the Entech 4700 Precision Diluter, located in the bottom left corner. The power LED will illuminate on the front panel.
- 3 Double click the Entech 4700 icon on the desktop to connect to the precision diluter.
- 4 The link active LED on the 4700 front panel will illuminate once connected. See section 9.1 for communications troubleshooting. The following software screen will appear on the computer:

🔇 Entech 4700 - 2.1.2.9													⊞ ·	- 0	×
	DILUTI		SETTINGS						47	00	Pre	cisio	on Di	luter	
SYSTEM STATUS	Ch	annel Status 0%		Status Mes	sage			Current f 14.50	PSIA A' D	vg. PSIA 14.50	Target 0.0	PSIA I O	Total Ela 00:00:	psed 00	
FILE	Error: 401. Dil	ution Method - Meth	nod Channel configu	ration misma	tch - use th	e update l	button or lo	oad a ma	itching o	hannel	configur	ation			
	File: Default.4	1700	Canister ID:	No Canister ID	C	anister Vo	olume:	6 Liter			1	Target F	inal PSI	A: 35.00) psia
STANDARDS PREP	Use Ch. Tit	tle	Initial Conc.	Initial Level	Target Conc.	Target Level	Dilution Factor	Initial Psia	Target Psia	Final Psia	Target Psid	Final Psid	Error %	Elapsed Time	
FLUSH	✓ 1 Di	luent	100	0.00 percent	100.00	percent	1.00	0.00	0.00	0.00	0.00	0.00	0.00%	00:00:00	5
	🗹 🔁 Ch	nannel2	1,000	0.00 ppb	1,000.00	ppb	1.00	0.00	0.00	0.00	0.00	0.00	0.00%	00:00:00	J
PRESSURIZE	✓ 3 Cł	nannel3	1,000	0.00 ppb	1,000.00	ppb	1.00	0.00	0.00	0.00	0.00	0.00	0.00%	00:00:00	J
	✓ 4 CF	nannel4	1,000	0.00 ppb	1,000.00	ppb	1.00	0.00	0.00	0.00	0.00	0.00	0.00%	00:00:00	S
SPIKE	🗹 🚺 Cł	nannel5	1,000	0.00 ppb	1,000.00	ppb	1.00	0.00	0.00	0.00	0.00	0.00	0.00%	00:00:00	3
	✓ 6 Re	e-Dilute	1,000	0.00 ppb	1,000.00	ppb	1.00	0.00	0.00	0.00	0.00	0.00	0.00%	00:00:00	S
MANUAL CTRL	🗹 🚺 Di	luent	100	0.00 percent	100.00	percent	1.00	0.00	0.00	0.00	0.00	0.00	0.00%	00:00:00	J
	Optional Rep	ort Parameters													
	Report ID:	No Report ID	Prepared By	Not Specifie	d										
	Comment:	No Comment													
													_		
		Method channel	ls no longer match o	urrent config	uration sett	ings. Upd	late to mate	ch? U	pdate	St	art	Abo	ort		

Figure 4: Entech 4700 Precision Diluter Software Initial Boot

5 The "Error 401. Dilution Method" will trigger at each launch of the software due to multiple user profiles. Click the update button to load the configuration file. The screen will now populate with the cylinder inventory equipped to the precision diluter:

Entech 4700 - 2.1.2.9														⊞ –		×	Page 13 OI
	DI		SETTINGS							47	00	Pre	cisio	on Dil	uter		
YSTEM STATUS		Channel Status		S	tatus Mess	age		(Current P 14.50	ISIA AV	g. PSIA I 4.50	Target 0.0	PSIA 0	Total Elap 00:00:0	sed 10		
FILE	File: Defa	ult 4700	Caniste		anister ID	6	anister Vol	ume:	6 Liter			т	arget F	inal PSIA	. 25.00	ncia	
TANDARDS PREP	Use Ch.	Title	Carriste	Initial	Initial	Target	Target	Dilution	Initial	Target	Final	Target	Final	Error	lapsed	psia	
FLUSH	I 1	Diluent		100.00	percent	100.00	percent	1.00	0.00	0.00	0.00	0.00	0.00	0.00%	00:00:00		
	2	TO-15 STD		1,000.00	ppb	1,000.00	ppb	1.00	0.00	0.00	0.00	0.00	0.00	0.00%	00:00:00		
PRESSURIZE		PAMS-Airgas		100.00	ppb	100.00	ppb	1.00	0.00	0.00	0.00	0.00	0.00	0.00%	00:00:00		
CDIVE		PAMS-Linde		100.00	ppb	100.00	ppb	1.00	0.00	0.00	0.00	0.00	0.00	0.00%	00:00:00		
JUNE		Po Diluto		100.00	ppp	101.80	ppb	1.00	0.00	0.00	0.00	0.00	0.00	0.00%	00:00:00		
		Ne-Dilute		100.00	ppo	100.00	ppb	1.00	0.00	0.00	0.00	0.00	0.00	0.00%	00.00.00		

Figure 5: Entech 4700 Precision Diluter Software with Configuration File Loaded

6 Confirm that the cylinder information is correct, in the event it needs to be updated, navigate to the "settings" gear and wrench icon on the top menu of the software. Next, select the "configuration" tab as shown below. The current configuration and cylinder inventory will appear:

🔇 Entech 4700 - 2.1.2.9									⊞ –		×
	DILUTION REPORTS	SETTINGS				4	700	Precisi	on Dilu	ter	
SYSTEM STATUS	Channel Status 0%		Status Mes	sage		Current PSIA 14.75	Avg. PSIA 14.75	Target PSIA 0.00	Total Elapse 00:00:00	9	
FILE	File: C:\SmartLabs\Data\4700\Cor	nfiguration\D	efault.47Confi	g							
	Ch. Title	Init. Conc.	Conc. Level	Cylinder ID	Date Rcvd.	Expires	Notes	_	_		
OVERRIDE	1 Diluent	100	percent	356362	4/15/2019	11/30/2024	Ultra Zero /	Air			
	2 TO-15 STD	1000	ppb	CC-7350771	11/16/2020	11/11/2021					
CONFIGURATION	3 PAMS-Airgas	100	ppb	AAL072554	11/19/2019	11/19/2021	PAMS Prim	ary Standar	d		
	4 PAMS-Linde	100	ppb	EY-0001288	7/28/2020	7/28/2021	PAMS Seco	ndary Stand	dard		
CALIBRATION	5 Ethylene Oxide	101.8	ppb	D511973	11/14/2019	11/14/2020	Method De	evelopment	use only		
	6 Re-Dilute	100	ppb		3/1/2021	3/1/2021	Rediluted O	Canister ID:			
	System Configuration										
	File: Default.4700.SYSCFG										
	Enable Sampler QA No 🔸										
	Save										

Figure 6: Entech 4700 Precision Diluter Software with Configuration File Loaded

- 7 Enter in the cylinder lot ID, receive date, expiration date for each channel, 2-5, connected to the Precision Diluter. Click on the file tab of the left hand menu and select "save configuration." Overwrite the existing configuration file.
- Open the regulator to the ultra-zero air supply cylinder and confirm the outlet pressure is set to 50 psi. 8
- 9 Open the regulator on the analytical gas standard cylinder of interest; confirm the outlet pressure is set to 40 psi.
- 10 Remove the ¼-in cap nut from the outlet stream on the front of the diluter. In the left hand menu of the Entech 4700 Precision Diluter software click the flush tab. The following screen will appear:

WINDERSON WINDERSON WINDERSON WINDERSON Mage 124 Mage 12	Entech 4700 - 2.1.2.9							8 - 0
FILE Function FUSH Channel Status Status Message Current PSIA Avg. PSIA Target PSIA Total Elapsed Obs Obs Obs Obs Obs Obs FILE Error: 410. Flush Method - At least one channel must be selected before running a flush Elect Channels Flush Configuration Oldent Diluent Diluent Flush 10 sec. PRESSURIZE PAMS-Airgas Diluent Flush Between 10 sec. SPIKE S Ethylene Oxide Status Status Status					4	700	Precisi	ion Diluter
FILE Error: 410. Flush Method - At least one channel must be selected before running a flush STANDARDS PREP • • • • •	SYSTEM STATUS	Channel Statu: 0%	5	Status Message	Current PSIA 14.49	Avg. PSIA 14.50	Target PSIA	Total Elapsed 00:00:00
Select Channels Flush Configuration TANDARDS PREP 1 Diluent I Diluent Diluent Flush I Diluent Diluent I Diluent Diluent <t< td=""><td>FILE</td><td>Error: 410. Flush Method</td><td>- At least one channel must be</td><td>selected before running a flu</td><td>sh</td><td></td><td></td><td></td></t<>	FILE	Error: 410. Flush Method	- At least one channel must be	selected before running a flu	sh			
FLUSH 1 Diluent Diluent Flush 10 sec FLUSH 2 TO-15 STD Standards Flush 10 sec PRESSURIZE 9 PAMS-Linde 10 sec SPIKE 6 Re-Dilute MANUAL CTRL Skip Abort		Select Channels	Flush Configu	iration				
FLUSH 2 TO-15 STD Standards Flush 10 sec PRESSURIZE 3 PAMS-Airgas Diluent Flush Between 10 sec SPIKE 6 Ethylene Oxide 6 Re-Dilute	STANDARDS PREP	1 Diluent	Diluent Flush	10 sec				
PRESSURIZE SPIKE SPIKE Bethylene Oxide Re-Dilute MANUAL CTRL Start Skip Apart	FLUSH	2 TO-15 STD	Standards Flush	10 sec				
PRESSURIZE PRESSURIZE		3 PAMS-Airgas	Diluent Flush Between	10 sec				
SPIKE S Ethylene Oxide 6 Re-Dilute MANUAL CTRL Start Skip Abort	PRESSURIZE	4 PAMS-Linde						
MANUAL CTRL Start Skip Abort	SPIKE	5 Ethylene Oxide						
MANUAL CTRL Start Skip Abort	of the	6 Re-Dilute						
	MANUAL CTRL		Start Skip	Abort				



11 Select the diluent channel (number 1) and the analytical standard(s) of interest. If more than one standard will be used in a given diluter session, then select all that apply. Apply a "Diluent Flush" of 30 seconds, a "Standard Flush" of 30 seconds, and a "Diluent Flush Between" of 30 seconds. A PAMS system flush is demonstrated below:

🔇 Entech 4700 - 2.1.2.9							□ –		×
		PORTS SETTINGS		4	700	Precisi	on Dilu	ter	
SYSTEM STATUS	Channel Status 0%		Status Message	Current PSIA 14.50	Avg. PSIA 14.50	Target PSIA 0.00	Total Elapsed 00:00:00		
FILE									
	Select Channels	Flush Configu	uration						
STANDARDS PREP	1 Diluent	Diluent Flush	30 sec						
FLUCT	2 TO-15 STD	Standards Flush	30 sec						
FLUSH	3 PAMS-Airgas	Diluent Flush Between	30 sec						
PRESSURIZE	4 PAMS-Linde								
SPIKE	5 Ethylene Oxide								
	6 Re-Dilute								
MANUAL CTRL		Start Skip	Abort						

Figure 8: Entech 4700 Precision Diluter Software Flush PAMS Example

- 12 Press start to begin the flush and wait for it to complete. The channel status bar will show the progression of the flush as the system purges each line.
- 13 Prepare an aliquot of deionized water in a clean 30-mL beaker. Dispense about 5 mL of water.
- 14 Using a clean 100-μL gas-tight syringe, flush the syringe with the deionized water three times. Be sure to examine the barrel of the syringe for any air bubbles and repeat flushing the syringe until all air is displaced with water.
- 15 The operator can now proceed to the system leak check in Section 5.3.

5.2 Creating Dilution Methods

- 1 Select "Standards Prep" from the left hand menu in the 4700 Diluter software. Click file and select "New Method."
- 2 Channel 1, the diluent, must be selected both times. The system operates by filling a canister with diluent gas to a specified pressure, then a specified pressure interval of standard gas, and finally tops off the dilution canister to the final target pressure with diluent gas. Uncheck the "use" box column for standards in the inventory that are not of interest for the targeted dilution.
- 3 Enter a target final pressure in psi_A in the top right corner of the window. The maximum pressure for a dilution is 50 psi_A.
- 4 In the "Target Conc." column, enter the concentration of the dilution desired or enter the dilution factor of the standard in the dilution factor column (each column will populate the other with the correct value based on the value entered). A maximum dilution factor of 100 can be achieved with the Entech 4700 Precision Dilution System. Further dilutions require a serial dilution of an already prepared standard and are detailed in section 5.4.3. An example dilution, creating a 2 ppb standard from an 100 ppb PAMS-Airgas standard, is shown below:

🔇 Entech 4700 - 2.1.2.9				🖽 – 🗆 🛛
			4700	Precision Diluter
	Channel Status 0%	Status Message	Current PSIA Avg. PSIA 14.76 14.76	Target PSIA Total Elapsed 0.00 00:00:00
FILE				
	File: Default.4700 Canist	er ID: No Canister ID Canister Volu	me: 6 Liter •	Target Final PSIA: 50.00 psia
STANDARDS PREP	Use Ch. Title	Initial Initial Target Target D Conc. Level Conc. Level F	Dilution Initial Target Final T actor Psia Psia Psia P	arget Final Error Elapsed sid Psid % Time
FLUSH	Diluent	100.00 percent 100.00 percent	1.00 0.00 0.00 0.00	0.00 0.00 0.00% 00:00:00
	2 TO-15 STD	1,000.00 ppb 1,000.00 ppb	1.00 0.00 0.00 0.00	0.00 0.00 0.00% 00:00:00
PRESSURIZE	PAMS-Airgas	100.00 ppb 2.00 ppb	50.00 0.00 0.00 0.00	0.00 0.00 0.00% 00:00:00
	A PAMS-Linde	100.00 ppb 100.00 ppb	1.00 0.00 0.00 0.00	0.00 0.00 0.00% 00:00:00
SPIKE	5 Ethylene Oxide	101.80 ppb 101.80 ppb	1.00 0.00 0.00 0.00	0.00 0.00 0.00% 00:00:00
	C 6 Re-Dilute	100.00 ppb 100.00 ppb	1.00 0.00 0.00 0.00	0.00 0.00 0.00% 00:00:00
MANUAL CTRL	Diluent	100.00 percent 100.00 percent	1.00 0.00 0.00 0.00	0.00 0.00 0.00% 00:00:00
	Optional Report Parameters			
	Report ID: No Report ID Prepa	red By Not Specified		
	Comment: No Comment			
			Star	t

Figure 9: Entech 4700 Precision Diluter Software 2 ppb PAMS Preparation Example

5 Click file and select "Save Method." Use a name that details the concentration of the standard created and the source from which it came. E.g. The standard in Figure 9 could be saved as 2ppb_PAMS-Airgas.

5.3 System Leak Check

- 1 In the Entech 4700 software navigate back to the "Standards Prep" tab in the left-hand menu.
- 2 Select a clean and evacuated standard canister for standard preparation. In the Precision Diluter Logbook document the date, operator initials, standard canister number (Note: standard canisters and ambient sample canister numbers follow separate naming conventions. Standard canisters are denoted with an S preceding the canister serial number. Ambient canisters are not to be used for standard preparation.), the cleaning batch number of the canister (located on the canister tag see appendix C for an example tag), diluent

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cylinder lot, analytical gas standard cylinder number and DI water lot number. A template logbook entry sheet for the Entech 4700 Precision Diluter is shown in appendix D.

- 3 Dispense a 75-µL aliquot of deionized water into the orifice of the standard canister.
- 4 Connect the standard canister to the outlet port with a 9/16-in wrench. Take care not to over tighten the canister connection, a quarter-turn past finger tight is a best practice for compression fittings.
- 5 Open the valve and observe the pressure read back of the system in the top left of the screen under "Current PSIA." It will drop from an ambient reading to near zero on the absolute scale.
- 6 Observe the pressure read back for 30 seconds and look for stability in the measurement. Reject canisters that the observed pressure increases by more than 0.5 psi_A over 30 seconds. See section 9.2 for corrective action.
- 7 The canister is ready to proceed through the desired preparation process; See section 5.4.1 for Analytical Standard Preparation, section 5.4.2 for Humid Blank Preparation or 5.4.3 Serial Dilution of a Prepared Standard.

5.4 Precision Diluter Operation: Standard Preparation

Select the desired subsection based on the operator objective:

5.4.1 Preparation of an Analytical Gas Standard

1 Click on the "File" tab of the left hand menu and select "Load Method." Select the desired canister preparation method. Record the method loaded and executed for a dilution in the Entech Precision Diluter logbook. An example dilution method is shown below in Figure 10:

W Entech 4700 - 2.1.2.9				Ш – I ×
		5	4700	Precision Diluter
SYSTEM STATUS	Channel Status 0%	Status Message	Current PSIA Avg. PSIA 14.49 14.49	Target PSIA Total Elapsed 0.00 00:00:00
FILE				
	File: PAMS\2ppbvPAMS_Airgas.4700 Canis	ter ID: No Canister ID Caniste	r Volume: 6 Liter •	Target Final PSIA: 50.00 psia
STANDARDS PREP	Use Ch. Title	Initial Initial Target Targe Conc. Level Conc. Leve	et Dilution Initial Target Final I Factor Psia Psia Psia	Target Final Error Elapsed Psid Psid % Time
FLUSH	☑ 1 Diluent	100.00 percent 100.00 perce	ent 1.00 0.00 0.00 0.00	0.00 0.00 0.00% 00:00:00
	2 TO-15 STD	1,000.00 ppb 1,000.00 ppb	1.00 0.00 0.00 0.00	0.00 0.00 0.00% 00:00:00
PRESSURIZE	✓ 3 PAMS-Airgas	100.00 ppb 2.00 ppb	50.00 0.00 0.00 0.00	0.00 0.00 0.00% 00:00:00
	A PAMS-Linde	100.00 ppb 100.00 ppb	1.00 0.00 0.00 0.00	0.00 0.00 0.00% 00:00:00
SPIKE	5 Ethylene Oxide	101.80 ppb 101.80 ppb	1.00 0.00 0.00 0.00	0.00 0.00 0.00% 00:00:00
	6 Re-Dilute	100.00 ppb 100.00 ppb	1.00 0.00 0.00 0.00	0.00 0.00 0.00% 00:00:00
MANUAL CTRL	☑ 1 Diluent	100.00 percent 100.00 perce	ent 1.00 0.00 0.00 0.00	0.00 0.00 0.00% 00:00:00
	Optional Report Parameters			
	Report ID: No Report ID Prep	ared By Not Specified		
	Commont: No Comment			
	comment. No comment			
			Sta	Abort

Figure 10: Entech 4700 Precision Diluter with Preparation Method Loaded

2 Fill in the "Canister ID" field with the canister number (format: S####), delete/empty the Report ID field, put the operator initials in the prepared by field, and put the 75-μL aliquot of DI water into the comment field with the lot number. An example for canister S2403 is shown in Figure 11:

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Œ Entech 4700 - 2.1.2.9 4700 Precision Diluter 🕼 ENTECH A File: PAMS\2ppbyPAMS_Airgas.4700_Canister ID: 52403 Canister Volume: 6 Liter Target Final PSIA: 50.00 psia Target Dilution Initial Target Final Target Final Level Factor Psia Psia Psia Psid Psid Use Ch. Title Level Time Conc. ✓ 1 Diluent 100.00 percent 0.00 100.00 percent 0.00 0.00% 00:00:00 1.00 0.00 0.00 0.00 2 TO-15 STD 1,000.00 ppb 1,000.00 ppb 1.00 0.00 0.00 0.00 0.00 0.00 0.00% 00:00:00 ✓ 3 PAMS-Airgas 100.00 ppb 2.00 ppb 50.00 0.00 0.00 0.00 0.00 0.00 0.00% 00:00:00 A PAMS-Linde 100.00 ppb 100.00 ppb 1.00 0.00 0.00 0.00 0.00 0.00 0.00% 00:00:00 5 Ethylene Oxide 101.80 ppb 101.80 ppb 1.00 0.00 0.00 0.00 0.00 0.00 0.00% 00:00:00 6 Re-Dilute 100.00 ppb 100.00 ppb 1.00 0.00 0.00 0.00 0.00 0.00 0.00% 00:00:00 ✓ 1 Diluent 100.00 percent 100.00 percent 1.00 0.00 0.00 0.00 0.00 0.00% 00:00:00 Optional Report Parameters Report ID: Prepared By BDV Comment: 75uL water (Lot 181732) Start

Figure 11: Entech 4700 Precision Diluter Preparation Example for Canister S2403

- 3 Press start to begin the standard preparation. The system will initially fill the canister with diluent gas, then standard gas, and finish with diluent gas. A report will generate when complete. Standard preparation times vary depending on concentration and final pressure and can take 20-40 minutes to complete depending on the parameters selected.
- 4 Close the canister valve and disconnect the canister with the 9/16-in wrench.
- 5 Click the report tab on the top row of the Entech 4700 Precision Diluter software. The screen will populate with a dilution report for the newly created standard. Click the floppy disk icon and save the report as a PDF. Name the report in the following format: YYMMDD Standard Name Concentration Canister Number. For the canister previously prepared on October 1st, 2020 at 2 ppbv of PAMS-Airgas the report would be saved as 201001 PAMS-Airgas 2ppb CanS2403. Save the report in the report folder. An example report is shown below in Figure 12:

4700 Preci	ision Dilut	ter Standaro	ds Preparatio	n Report		Date:	10/1/2020 2:09:22 PM
Dilution ID:	207		-	-		Report ID:	
Canister Size:	6000cc	Canister ID:	S2391	Target Final Psia:	50	Prepared By:	BDV
Comment:	75uL Water	Lot (181732)					

Channel Methods

Ch.	Title	Init. Conc.	Target Conc.	Dilution Factor	Init. Psia	Target Psid	Final Psid	Target Psia	Final Psia	Duration	Final Conc.
1	Diluent	100 percent	100 percent	1	0.13	11.84	11.84	11.98	11.98	00:06:48	N/A
3	PAMS - Airgas	100 ppb	2 ppb	50	11.98	1.00	1.00	12.98	12.98	00:01:48	2.01 ppb
1	Diluent	100 percent	100 percent	1	12.98	37.02	37.02	50.00	50.00	00:23:16	N/A

Total Duration: 00:32:00

Channel Configuration and Standards Inventory

Ch.	Source	Init. Conc.	Cylinder ID	Expiration Date	Notes
1	Diluent	100 percent	356362	11/30/2024	Zero Air
3	PAMS - Airgas	100 ppb	AAL072554	11/19/2021	

Figure 12: Entech 4700 Precision Diluter Standards Preparation Report

- 6 Print a copy of the 4700 Precision Diluter Standards Preparation Report and place it in the Entech 4700 Precision Diluter Binder. Initial and date the copy after reviewing it for accuracy. Cross out any errant entry with a line and write the correct value next to it. Initial and date.
- 7 Label the canister tag with the standard name. E.g. "PAMS-Airgas 2ppb" and the preparation date.
- 8 Return to the flush menu and flush the system with diluent only for 30 seconds to purge the system.
- 9 Close all analytical gas standards and ultra-zero air cylinders and corresponding regulators.
- 10 Cap the canister standard with a ¼-in female compression nut. A best practice is to let the prepared standard sit over-night prior to use to allow the water to interact with the canister and introduced compounds to ascertain equilibrium.
- 11 Cap the Entech 4700 Precision Diluter with ¼-in male compression nuts to finger tight.
- 12 Cap the newly created standard with a ¼-in female compression nut.

5.4.2 Preparation of a Humidified Blank

1 Click on the "Pressurize" tab of the left hand menu. "Error 420. Pressure Method – Select a container type to continue" will appear on the screen:

🏈 Entech 4700 - 2.1.2.9			<
	DILUTION REPORTS SETTINGS	4700 Precision Diluter	
SYSTEM STATUS	Channel Status Status Message	Current PSIA Avg. PSIA Target PSIA Total Elapsed 14.49 14.49 0.00 00:00:00	
FILE	Error: 420. Pressurize Method - Select a container type to continue		
STANDARDS PREP	1. Choose Type of Container O Glass O Stainless Steel		
FLUSH	2. Choose How to Pressurize Factor Absolute		
PRESSURIZE			
SPIKE	Target Psia: 0.00 Start Psia: 14.49 End Psia: 0.00 Psid: 0.00		
MANUAL CTRL	Start Abort		

Figure 13: Entech 4700 Precision Diluter Pressurize Tab

- 2 Select the stainless steel container type by clicking the radio button.
- 3 Select the absolute pressurize function in step 2 "chose how to pressurize." Enter 50 in the psia box that appears as shown below in Figure 14:

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Figure 14: Entech 4700 Precision Diluter Pressurization Settings

- 4 Click start and wait until the system is finished pressurizing the canister with ultra-zero air. The channel status bar will increment as the system pressurizes the canister. The system does not generate a report via this function.
- 5 Label the canister tag as humid blank. Record the date.
- 6 Close the ultra-zero air cylinder and regulator.
- 7 Cap the Entech 4700 Precision Diluter with ¼-in male compression nuts to finger tight.
- 8 Cap the newly created humid blank with a ¼-in female compression nut.

5.4.3 Preparation of a Standard by Serially Diluting a Previously Made Standard

To obtain parts-per-billion concentrations and below, standards need to be serially diluted from their source. The Entech 4700 can do this using the re-dilute, channel 6, functionality. To re-dilute a canister:

1 Select a previously created standard and connect it to the re-dilute channel on the front of the Entech 4700 with a 9/16-in wrench.

Note: A best practice is to let previously created standards equilibrate for 12-24 hours prior to the re-dilution of their contents.

- 2 Enter the settings tab and select the "Configuration" from the left hand menu.
- 3 Enter the canister number into the Cylinder ID field.
- 4 Enter the nominal concentration of the standard into the "Init. Conc." Field.
- 5 Enter the preparation date into the "Date Rcvd." Field.
- 6 Enter the expiration date in the "Expires" column.
- 7 Save the configuration file by clicking file and selecting "save configuration." Overwrite the existing configuration file. An example of the modified Re-Dilute channel configuration is shown below:

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🔇 Entech 4700 - 2.1.2.9									⊞ –	\Box ×
	DILUTION REPORTS SI					4	700	Precisi	on Dilut	er
SYSTEM STATUS	Channel Status 0%		Status Mes	sage		Current PSIA 14.76	Avg. PSIA 14.76	Target PSIA 0.00	Total Elapsed 00:00:00	
FILE	File: C:\SmartLabs\Data\4700\Confi	guration\De	efault.47Confi	g						
	Ch. Title	Init. Conc.	Conc. Level	Cylinder ID	Date Rcvd.	Expires	Notes		_	
OVERRIDE	1 Diluent	100	percent	356362	4/15/2019	11/30/2024	Ultra Zero	Air		
	2 TO-15 STD	1000	ppb	CC-7350771	11/16/2020	11/11/2021				
CONFIGURATION	3 PAMS-Airgas	100	ppb	AAL072554	11/19/2019	11/19/2021	PAMS Prim	ary Standard	ł	
	4 PAMS-Linde	100	ppb	EY-0001288	7/28/2020	7/28/2021	PAMS Seco	ndary Stand	lard	
CALIBRATION	5 Ethylene Oxide	101.8	ppb	D511973	11/14/2019	11/14/2020	Method De	evelopment	use only	
	6 Re-Dilute	2	ppb	S####	3/1/2021	4/1/2021				
	· ·									
	System Configuration									
	File: Default.4700.SYSCFG									
	Enable Sampler QA No 🔸									
	Save									

Figure 15: Entech 4700 Precision Diluter Re-Dilute Channel Configuration

8 Click the dilution button to navigate back to standards prep. Create a new method, as detailed in section 5.2, using the re-dilute channel as the analytical gas standard to target a desired dilution factor or concentration. An example is shown below on creating a 0.04-ppb standard from a 2-ppb standard:

🚷 Entech 4700 - 2.1.2.9														⊞ -	- 🗆	×
		D		TTINGS						47	00	Pre	cisio	on Di	luter	
SYSTEM STATUS		ľ	Channel Status <mark>0%</mark>		Status Mess	age			Current F 14.76	PSIA A [.] 5	vg. PSIA 14.76	Target 0.0	: PSIA 1 0	Total Ela 00:00:	osed 00	
FILE																
	File:	Defa	ult.4700	Canister ID: S##	##	Ca	nister Vo	lume:	6 Liter	•		1	Farget F	inal PSI	A: 40.00	psia
STANDARDS PREP	Use	Ch.	Title	Initial Conc.	Initial Level	Target Conc.	Target Level	Dilution Factor	Initial Psia	Target Psia	Final Psia	Target Psid	Final Psid	Error %	Elapsed Time	
FLUSH		(1)	Diluent	100.00	percent	100.00	percent	1.00	0.00	0.00	0.00	0.00	0.00	0.00%	00:00:00	
		2	TO-15 STD	1,000.00	ppb	1,000.00	ppb	1.00	0.00	0.00	0.00	0.00	0.00	0.00%	00:00:00	
PRESSURIZE		3	PAMS-Airgas	100.00	ppb	2.00	ppb	50.00	0.00	0.00	0.00	0.00	0.00	0.00%	00:00:00	
		4	PAMS-Linde	100.00	ppb	100.00	ppb	1.00	0.00	0.00	0.00	0.00	0.00	0.00%	00:00:00	
SPIKE		5	Ethylene Oxide	101.80	ppb	101.80	ppb	1.00	0.00	0.00	0.00	0.00	0.00	0.00%	00:00:00	
		6	Re-Dilute	2.00	ppb	0.04	ppb	50.00	0.00	0.00	0.00	0.00	0.00	0.00%	00:00:00	
MANUAL CTRL	\checkmark	1	Diluent	100.00	percent	100.00	percent	1.00	0.00	0.00	0.00	0.00	0.00	0.00%	00:00:00	
	Opti	onal	Report Parameters													
	Repo	ort ID	:	Prepared By Ar	alyst ID											
	Com	men	t: 75uL Water (Lot ######)													
												_				
											St	art	Abo	1		

Figure 16: Entech 4700 Precision Diluter Re-Dilute Channel Method

9 Enter the canister number into the Canister ID, the operator initials in the prepared by field and the volume and lot number of water used in the comment field.

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- 10 Press start to begin the dilution and wait for the process to complete.
- 11 Close all canisters and disconnect with a 9/16-in wrench.
- 12 Click the report tab on the top row of the Entech 4700 Precision Diluter software. The screen will populate with a dilution report for the newly created standard. Click the floppy disk icon and save the report as a PDF. Name the report in the following format: YYMMDD Standard Name Concentration Canister Number. For a canister, S1000, previously prepared on January 21st, 2021 at 2 ppbv PAMS-Airgas the report would be saved as "210121 PAMS-Airgas 2ppb CanS1000." Save the report in the report folder.
- 13 Print a copy of the 4700 Precision Diluter Standards Preparation Report and place it in the Entech 4700 Precision Diluter Binder. Initial and date the copy after reviewing it for accuracy. Cross out any errant entry with a line and writing the correct value next to it, then initial and date.
- 14 Label the canister tag with the standard name and preparation date. E.g. "PAMS-Airgas 2ppb" and the preparation date.
- 15 Return to the flush menu and flush the system with diluent only for 30 seconds to purge the system.
- 16 Cap the canister standard with a ¼-in female compression nut. A best practice is to let the prepared standard sit over-night prior to use to allow the water to interact with the canister and compounds to ascertain equilibrium.
- 17 Cap the Entech 4700 Precision Diluter with ¼-in male compression nuts to finger tight.
- 18 Cap the newly created standard with a ¼-in female compression nut.

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 Standards Preparation Report and the Entech 4700 Precision Diluter Logbook entry for completeness and accuracy. Confirm that all gas lots, DI water lot, added water volume and expiration dates are correct. Confirm that the canister ID and other information on the canister tag matches the dilution records. Initial and date both the logbook entry and standards preparation report to indicate a level 1 review has occurred and submit the logbook and binder for secondary, level 2 peer 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 Precision Diluter Data Review

Review the 4700 Precision Diluter Standards Preparation Report and 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 entry 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 4700

DAQ-03-006.2 Revision 0 04/01/2021 Page 22 of 33 Precision Diluter Standards Preparation Report and COAs of gas phase analytical standards or calibration certificates for support equipment.

7.1 4700 Precision Diluter Standards Preparation Report (Operator)

The operator will store the completed 4700 Precision Diluter Standards Preparation Report in the 4700 Precision Diluter binder. The binder is located next to the diluter instrument. The raw 4700 Precision Diluter Standards Preparation Report is also stored on the precision diluter computer as a PDF.

7.2 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 clear tape over the signed interface of the logbook entry.

7.3 4700 Precision Diluter Analytical Gas Standards Certificates of Analysis and Support Equipment Calibration Certificates (Operator)

The operator will store the completed COAs for all analytical gas standards employed by the diluter in the rear of the Entech 4700 Precision Diluter binder. The binder is located next to the diluter instrument. Support equipment calibration certificates shall be stored here as well: this includes the calibration certificate for the NIST-Traceable barometer and gas-tight syringe.

7.4 4700 Precision Diluter Standards Preparation Report (Peer/Level 2 Reviewer)

The peer reviewer will store the reviewed 4700 Precision Diluter Standards Preparation Report in the 4700 Precision Diluter binder. The binder is located next to the diluter instrument. The reviewer does not need to maintain this entry.

7.5 4700 Precision Diluter Logbook Entry (Peer/Level 2 Reviewer)

The operator will store the completed Entech 4700 Precision Diluter Logbook Entry 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 4700 Precision Diluter binder.

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.

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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. If none of these steps rectify the communications, contact Entech support at <u>support@entechinst.com</u>. **Note:** Administrative rights are required to control components in device manager.

9.2 System Leak Check Failure Corrective Action

- 1 If the 4100 Precision Diluter fails to leak check, then 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 a 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. Attempt a leak check again, if it passes it is okay to proceed to standard preparation.
- 2 Inspect the threads of the canister. If they are not even, 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 standard preparation.
- 3 Connect a canister pressurized with helium at 20-30 psi to the Entech 4700 using a 9/16-in wrench.
- 4 Remove the top cover to expose the proportional valves and pressure sensor as shown below in Figure 17:



Figure 17: Entech 4700 Precision Diluter Top Panel Interior

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



Figure 18: 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.

- 6 Open the helium canister attached to the diluter and place the G3388B probe against all the fittings, valves and the pressure sensor. Wait about 10-15 seconds per point of interest and 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.
- 9 Repair or replace the leaking section and leak test the system again. For part support contact support@entechinst.com

9.3 System Reads Negative Pressures Under Full Vacuum

The Entech 4700 Precision Diluter uses an absolute psi pressure scale and should not read negative values. Preform 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 DAQ SOP-03-006.1 ECB Responsibilities.

9.4 Ambient Pressure does not Match NIST-Traceable Barometer

If the Entech 4700 Precision Diluter no longer accurately measures the ambient pressure of the laboratory, preform a pressure sensor calibration. These steps are detailed in DAQ SOP-03-006.1 ECB Responsibilities.

9.5 Standard Preparation is Not Filling Completely and is Stuck on the First Diluent Step

Verify the diluent gas is turned on and set to 50 psi. Verify the canister's beginning evacuated pressure is under 1 psi_A. If the aforementioned items were set correctly, power down the Entech 4700 Precision Diluter and its system computer. Reboot the devices and open the software. Verify the 4700 link is active and begin the standard preparation process again. If a reboot does not solve the issue, contact support@entechinst.com for further assistance.

9.6 Standard Preparation is Stopping on a Standard Fill Step or Final Diluent Step

Verify the standard gas is turned on and set to 40 psi. The pressure of the standard gas delivered to the canister must exceed the pressure inside the canister. Conduct a leak test on the Entech 4700 Precision Diluter

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Top Panel Interior. The system is likely leaking. For further details see section 9.2 or contact support@entechinst.com.

9.7 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@entechinst.com for further support. The rear panel of the Entech 4700 is depicted below from the Entech 4700 Precision Diluter Manual V2.0:



Figure 19: Rear of Entech 4700 Precision Diluter

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 Example Canister Tag
- 4. Appendix D Entech 4700 Precision Diluter Logbook Entry Template
- 5. Appendix E NIST-Traceable Barometer and Calibration Certificate

Appendix A – Arc3 Purity Plus Specialty Gases Grade Specification Sheet



PURE GAS GRADE SPECIFICATIONS

	DE				CONTAI	MINATE LEVE	LS (UNLESS (OTHERWISE	NOTED AS	%)
PRODUCT	GRA	PURITY	PART #	VALVE	02	THC*	H2O	co	CO2	N2
ACETYLENE										
ATOMIC										
ABSORPTION∻	2.6	99.6%	ACE-26-300	510	< 4000 PPM (co	ombined)	-	-	-	-
						\$ < 2	20 PPM PH3			
				500		10.4	10			
	0.1	-	AIR-02-300	590	19.5% - 23.5%	<0.1 ppm	<3 ppm	<1 ppm	<1 ppm	-
	1.0	-	AIR-ZE-300	590	19.5% - 23.5%	<1 ppm	-	-	-	-
		-	AIN-LD-300	1 290	19.370 - 23.370	-	io ppin	-	-	-
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	50	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%	- ppm
LASER	4.5	99.995%	CDI-45-50	320	mag 5>	<1 mag 1>	mag 2>	-	99.995%	
COLEMAN/	4.0	00.000/		200		- P.P.M.	<10		00.000/	-50
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%	
				500						
GRADE /	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 npm	-	-	-	-
	1.0	00.00070	TIEE TO COO	000		ioto ppin				
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 mag	<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	mag 1>	-	-	<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			
NITROCEN										
RECEARCH	60	00.00000/	NIT 60 200	500	<0.2 mm	<0.1 mm	<0.2 mm	<0.1 mm	<0.1 mm	
	5.5	00 0005%	NIT-55-XY	580	<1 ppm	<0.1 ppm	<0.2 ppm	<1 npm	<1 npm	
	5.0	00 000%	NIT-50-300	580	<1 ppm	<0.1 ppm	<1 ppm	< ppm	< i ppm	-
ZERO	4.8	99.99976	NIT-7E-300	580	<1 ppm	<0.5 ppm	<1 ppm	-	-	
	4.8	99,998%	NIT-48-300	580	<5 ppm		<5 nnm	-	-	-
Oxygen Free	4.8	99,998%	NIT-OF-300	580	<0.5 ppm	-		-	-	-
					are pp				I	
NITROUS OXIDE										
ATOMIC										
ABSORPTION	2.6	99.6%	N20-26-50	326	*	-	<30 ppm	-	-	*
					* N20-AA: AIR<	2000 ppm				
OXYCEN										
UXYGEN	5.6	00.0006/	0)0/ 50 000	5.40	00.0000/	-0.5				.5
RESEARCH***	5.0	99.999%	OXY-50-300	540	99.999%	<0.5 ppm	<1 ppm	<1	opm	<5 ppm
	4.3	99.993%	OXY-43-300	540	99.993%	<0.5 ppm	<3 ppm	-	-	<10 ppm
	2.8	99.8%	OXT-28-300	540	99.8%	v.5 ppm	<10 ppm	-	-	-
	2.0	99.070	0/1-20-300	340	*02-LIHD -40		***O2 PI		<5 nnm	Δr
	Darte	Der Million			DZ-UFIF X40	Phili Al	02-Ri		vdroorth	
ppm =	- marts	rer willion			ppp = marts per Bi	non		nu = rotaí H	yurocarbon	5

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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

Orauc	OI I I OULOUI OLIMANIA			
Part Number: Cylinder Number: Laboratory: Analysis Date: Lot Number:	X57NI99C15AC003 AAL072554 124 - Plumsteadville - PA Nov 19, 2019 160-401659192-1	Reference Number: Cylinder Volume: Cylinder Pressure: Valve Outlet: Expiration Date:	160-401659192-1 144.3 CF 2050 PSIG 350SS 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 C	CONCENTRATIONS	
	Requested	Reported	
Component	Concentration	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%
123 TRIMETHYL BENZENE	100.000 PPB	105.000 PPB	+/- 10%
124 TRIMETHYLBENZENE	100.000 PPB	104.000 PPB	+/- 10%
1 3 DIETHYLBENZENE	100.000 PPB	97.000 PPB	+/- 10%
135 TRIMETHYL BENZENE	100.000 PPB	100.000 PPB	+/- 10%
14 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 METHYL HEPTANE	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%
2 METHVI HEDTANE	100 000 PPB	102.000 PPB	+/- 10%
3 METHYLHEYANE	100.000 PPB	102.000 PPB	+/- 10%
A ETHYL TOLLIENE	100.000 PPB	95.000 PPB	+/- 10%
ACETVIENE	100 000 PPB	101.000 PPB	+/- 10%
RENZENE	100.000 PPB	100.000 PPB	+/- 10%
CIS 2 BUTENE	100.000 PPB	108.000 PPB	+/- 10%
CIG 2 DENTENE	100.000 PPB	98.000 PPB	+/- 10%
CIMENE	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%
	1 PRIAN		
	(AWV'		

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			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%
NNONANE	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

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Appendix C – Example Canister Tag

NC DAQ LABORATORY	ANALYSIS BRANCH
4403 Reedy Creek R	oad, 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	×

Appendix D – Entech 4700 Precision Diluter Logbook Entry Template

	Entech 4700 Pr	ecision Dilute	r Logbook Entry Template				
Date:			Canister Number:				
Operator ID:			Canister Cleaning Batch Number:				
Entech 4700 S/N:	0316		Standard Name:				
Syringe:	Hamilton Gas-Tight 10	ΟμL	Diluent Gas Cylinder Lot:				
DI Water Volume (μL):			Analytical Gas Cylinder # or Rediluted Canister #:				
DI Water Lot Number:			Entech 4700 Method ID:				
NIST-Traceable Barometer S/N:	ST-Traceable Barometer 200810884		Barometer Expiration Date:		12/16/2021		
	System Pressure Che	ck	· · · · · · · · · · · · · · · · · · ·				
"Current PSIA" Reading	PAS ±(S CRITERIA 0.1psi _A of	Leak Check Pass? (PASS CRITERIA: No more than 0.5psi _A over 30seconds)	Y	N	(circle one)	
Barometric Pressure (mbar)	Ba F	rometric Pressure	4700 Pressure Calibration Expiration Date:		3/5/	/2022	
Barometric Pressure (psi _A) [‡]							
‡ Barometric Pressure is converted t	from mbar to psi _A via the following	formula:	Level 1 Review:				
psia = (Ambient Pressurembar)(1	4.50377psi₄/1bar)(1bar/100(Jmbar)	Level 2 Review:				

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

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Appendix E – NIST-Traceable Barometer and Calibration Certificate



Fisherbrand Traceable® NIST-Traceable Barometer

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	Calibration		+					Cert. N	0.: 0530-1	1810931	
_	Tracea	ble® Certi	ficate o	f Calibra	tion for	Digital	Baromet	er			
lanufacture	d for and dist	ributed by : Fish	er Scientific	: "300 Industry	y Drive,,Pitts	burgh,PA,15	275-1001"				
Instrume	nt Identifi	cation:									
Model: 1	4-650-118,	15557603		5	S/N: 20081	0884	Ma	nufacturer: (Control Comp	any	
Standard	s/Equipm	ent:									
	Descri	ption		Serial Num	ber	Due	Date	NIST	Traceable Refer	rence	
	Digital Ba	rometer		D4540001			v 2021	1000461277			
	Digital The	mometer		130070752			10 Mar 2021		4000-11170557		
	Chilled Mirror	Hygrometer		44654/2H3737			25 Nov 2021		17811		
	Climate C	hamber		W619.0019							
Certificat Techniciar	e Informa	tion:	Procedure	: CAL-31	Ca	I Date: 16 I	Dec 2020	Cal D	ue Date: 16 De	ec 2022	
Test Cond	itions: 44.	5%RH 22.06	5°C 1021	mBar							
Calibratio	on Data: (N	lew Instrum	ent)								
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	
	N A	NA		1011 77	1012	Y	1008	1016	0.62	>4:1	

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=±(Max-Min)/2; Min=As Left Nominal(Rounded) – Tolerance; Max= As Left Nominal(Rounded) + Tolerance;

Rial Rodriguy

Nicol Rodriguez, Quality Manager

Jarisa (Flms, Technical Mana

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).

Traceable® is a registered trademark of Control Company

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