Electronic Supplementary Material (ESI) for Digital Discovery.

Accelerated screening of carbon dioxide capture by liquid sorbents

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Tetramethylammonium 2,3,4,5,6-pentafluorophenolate

The NMR and FTIR characterization are shown in Figures S1-S5. Yield: 2.259 g (97 %). ^{1}H NMR (600 MHz d-DMSO) δ 3.14 (TMA) ppm. $^{13}C\{^{1}H\}$ NMR (151 MHz, d-DSMO) δ 147.8 (tt, *J*=14.1, 4.0 Hz), 140.9 (dtdd, *J*=231.9, 9.2, 4.6, 1.9 Hz), 138.6 (ddtd, *J*=235.9, 19.2, 10.6, 2.3 Hz), 123.7 (dtt, *J*=222.4, 14.8, 5.4 Hz), 54.4-54.3 (m, TMA) ppm. $^{13}C\{^{1}H\}$ NMR (151 MHz, D_2O) δ 142.0–141.7 (m), 140.8 (dm, *J*=231.6 Hz), 138.2 (dm, *J*=240.7 Hz), 129.8 (dm, *J*=232.4 Hz), 55.3–55.2 (m, TMA) ppm. $^{19}F\{^{1}H\}$ (565 MHz, d-DMSO) δ -172.0–-172.1 (m, 2F), -172.1–-172.3 (m, 2F), -196.3–-196.5 (m, 1F) ppm. FTIR (ATR)/cm^{-1} v = 3032.41 (w), 1690.86 (w), 1644.75 (w), 1599.17 (w), 1504.13 (s), 1487.98 (s), 1467.75 (s), 1248.42 (m), 1230.19 (m), 1167 (w), 1084.29 (w), 1046.34 (w), 1001.62 (s), 967.89 (s), 723.23 (w), 604.91 (w), 577.75 (w).

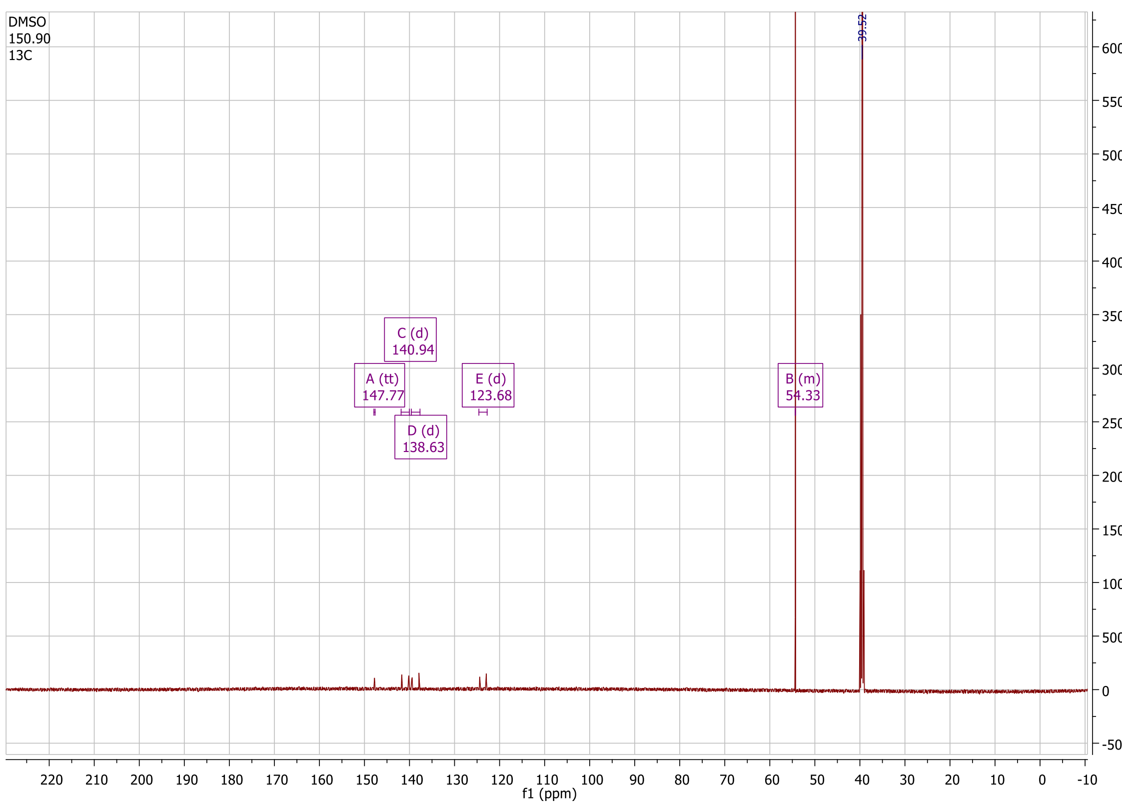


Figure S1: $^{13}C\{^{1}H\}$ NMR of TMA pentafluorophenoxide in d-DMSO.

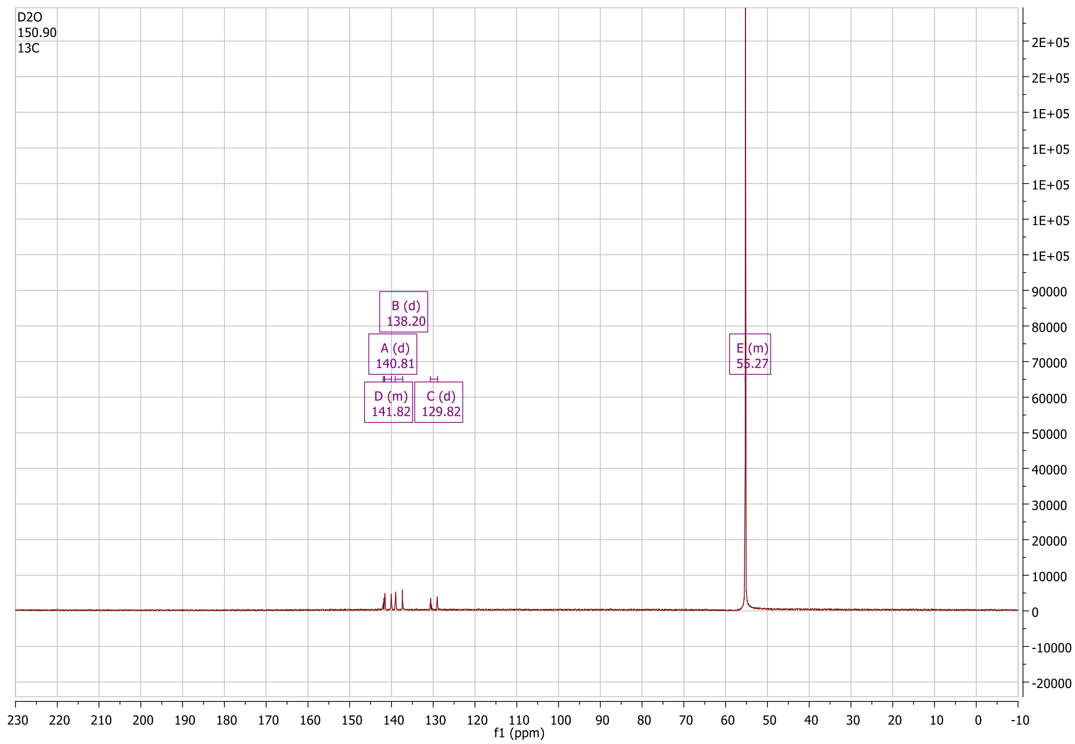


Figure S2: $^{13}C\{^{1}H\}$ NMR of TMA pentafluorophenoxide in D_2O.

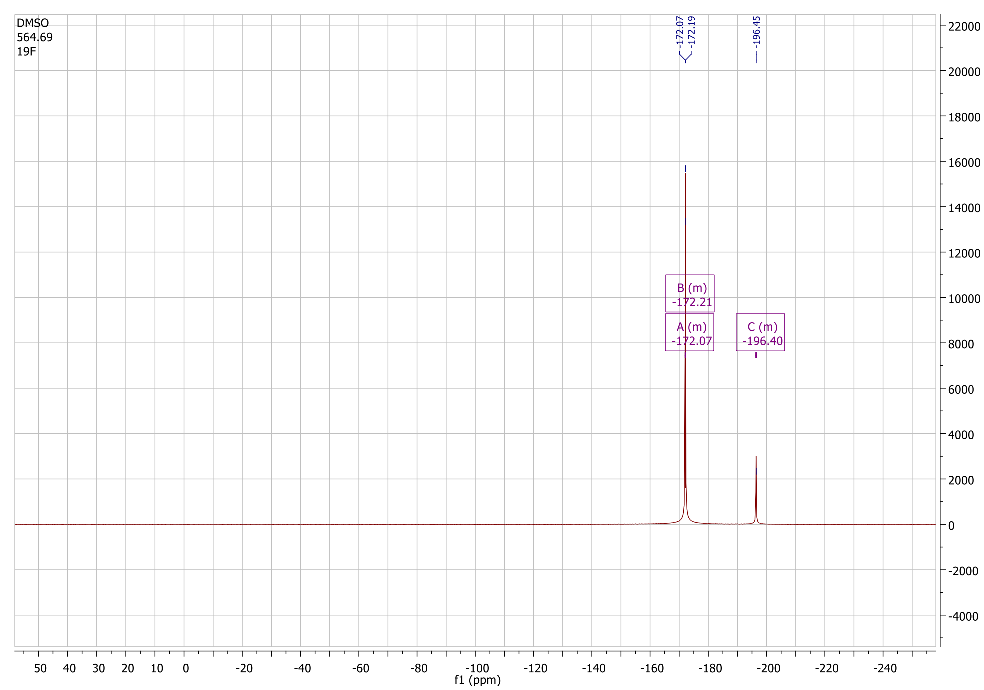


Figure S3: $^{19}F\{^{1}H\}$ NMR of TMA pentafluorophenoxide in d-DMSO.

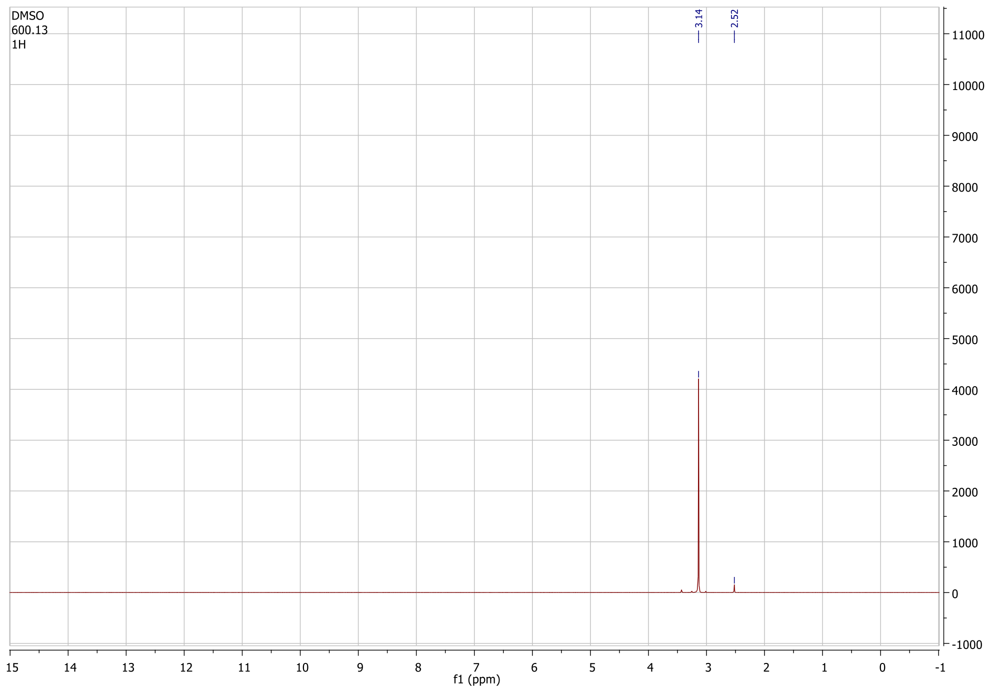


Figure S4: ^{1}H NMR of TMA pentafluorophenoxide in d-DMSO.

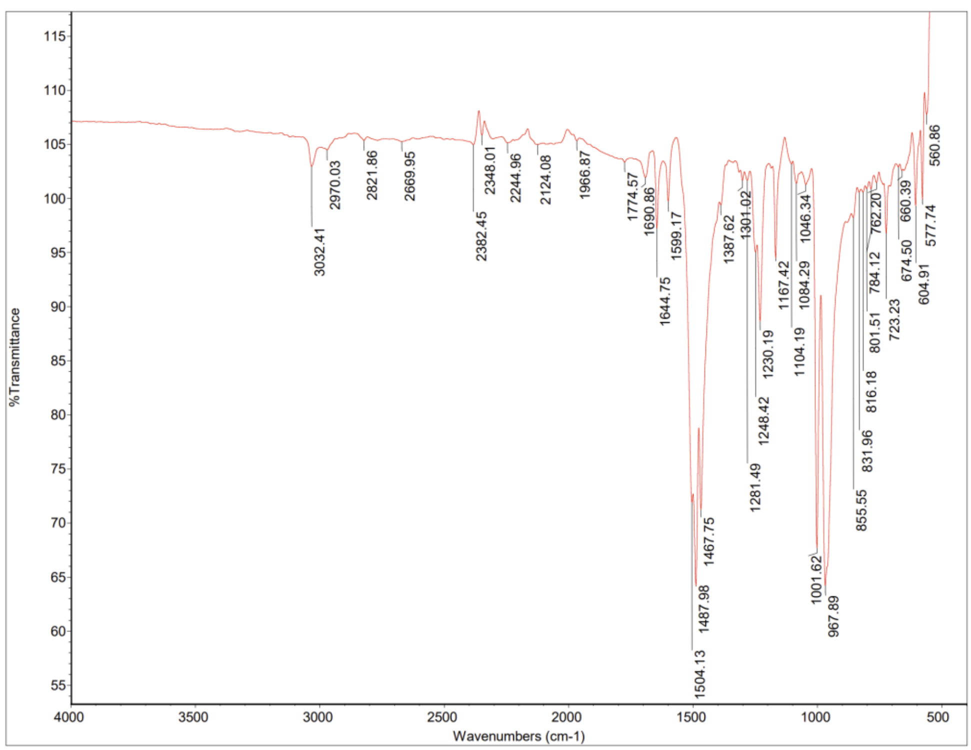
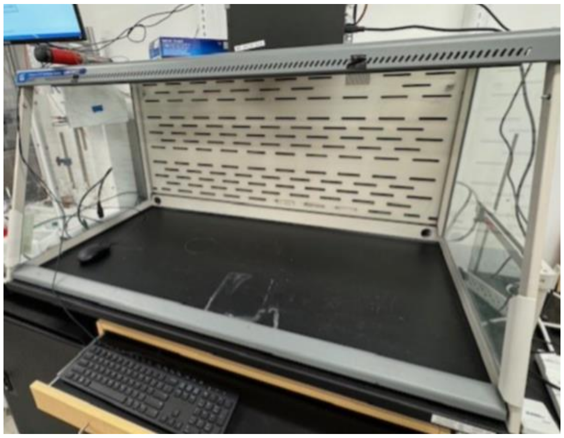


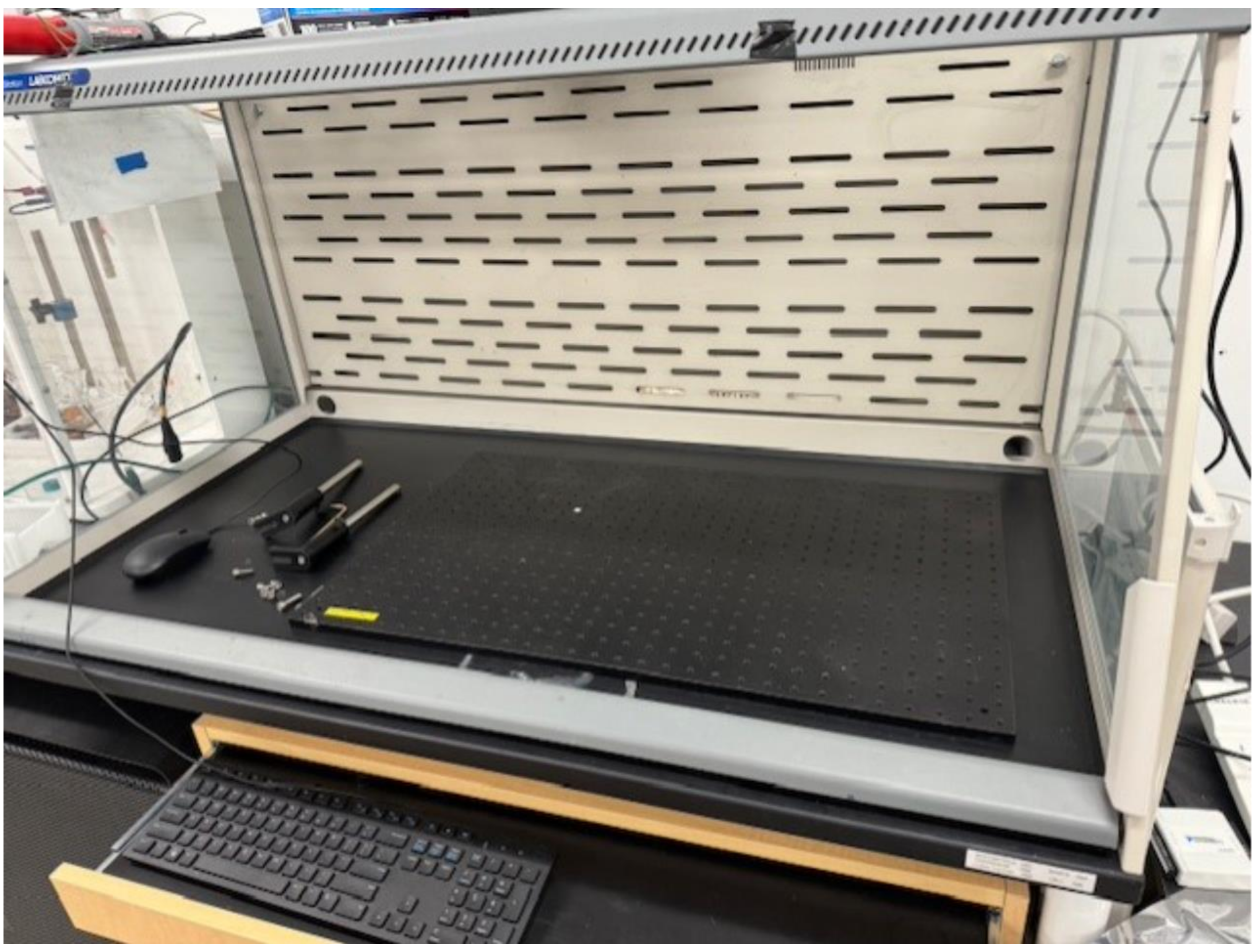
Figure S5: FTIR of TMA pentafluorophenoxide.

CCSI assembly instructions

1. Clear Hood/Work Area

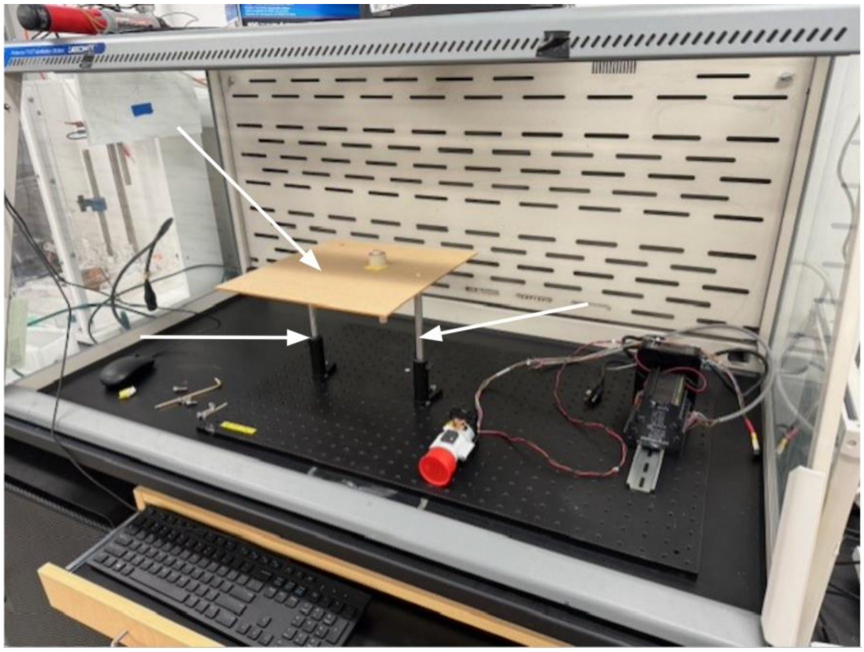


2. Place Breadboard

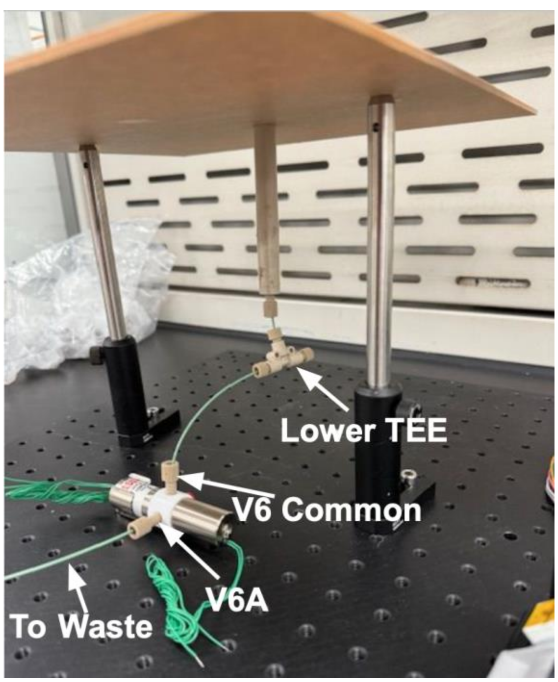


3. Wire Electrical Distribution Rack Ground and Relays, ensuring to observe proper polarity for both Control and Load signals.

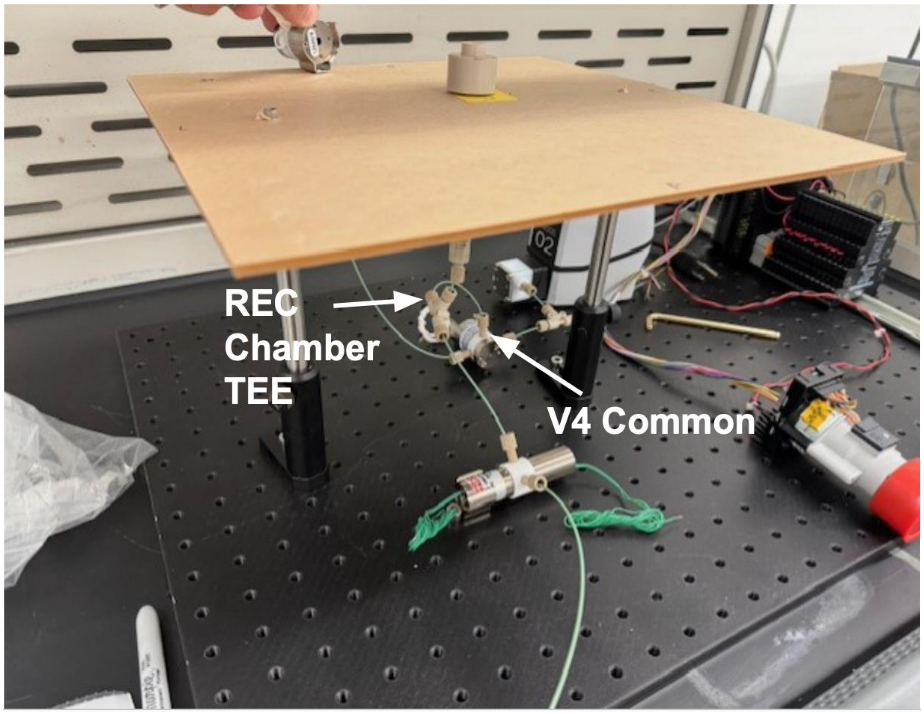
4. Mount Main Chamber Panel (12”x12” panel) onto elevated standoffs



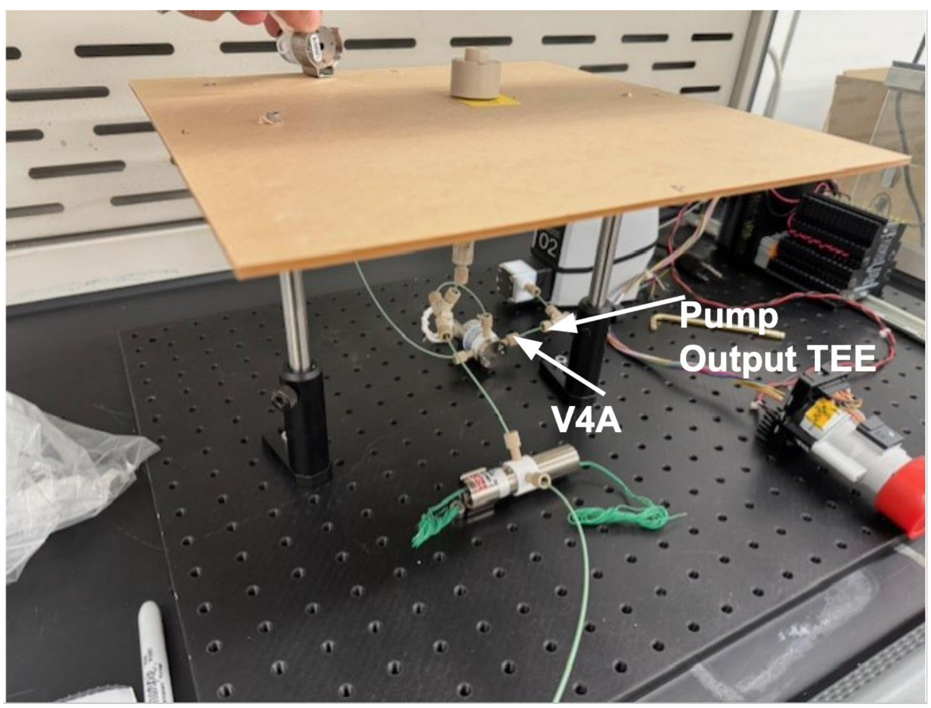
5. Insert REC Chamber into Panel and Install lower TEE as shown (REC Chamber → Tee Common port). Place Flow Selection Valve 6 on breadboard as shown. Connect the REC Chamber TEE into the Valve 6 Common as shown. Install PEEK tubing into Valve 6 A port as shown. Ensure the PEEK tubing is long enough to reach the designated Waste/Used repository.



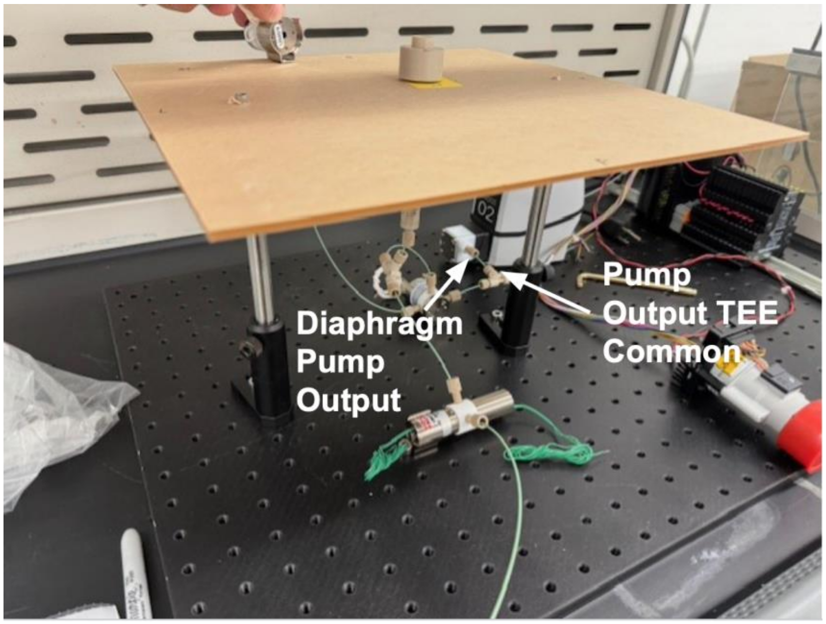
6. Place switching valve V4 on the breadboard in approximate shown location. Connect V4 Common port to the REC Chamber Tee.



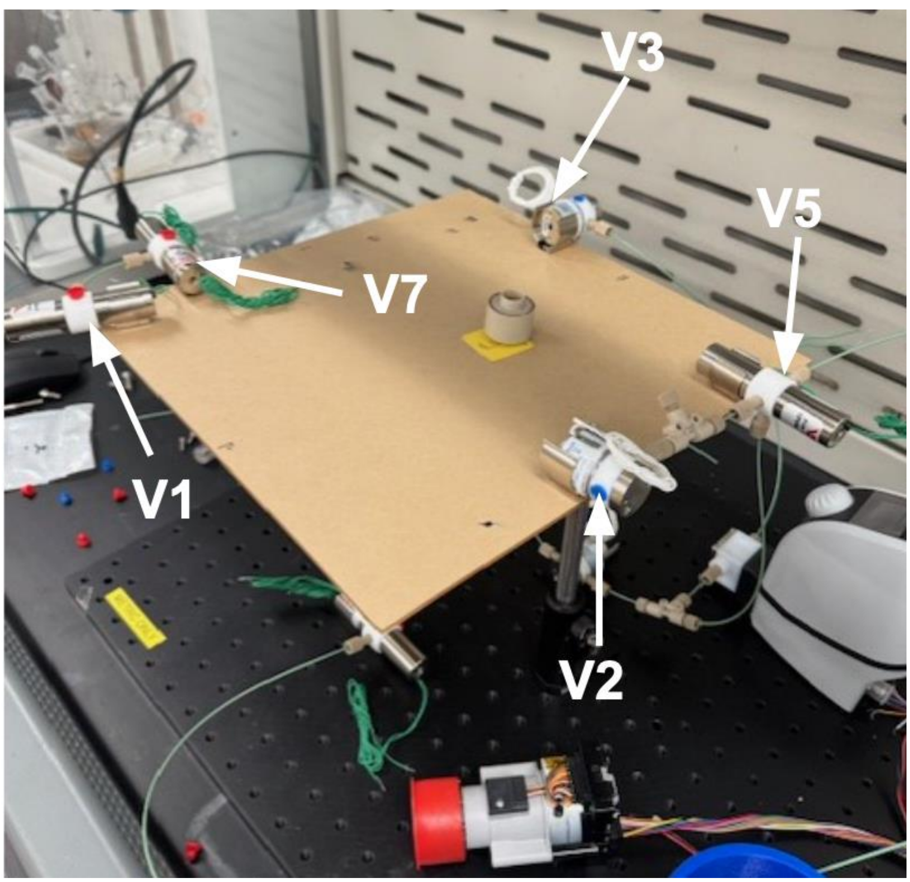
7. Connect port V4A to the Pump Output TEE, as shown.



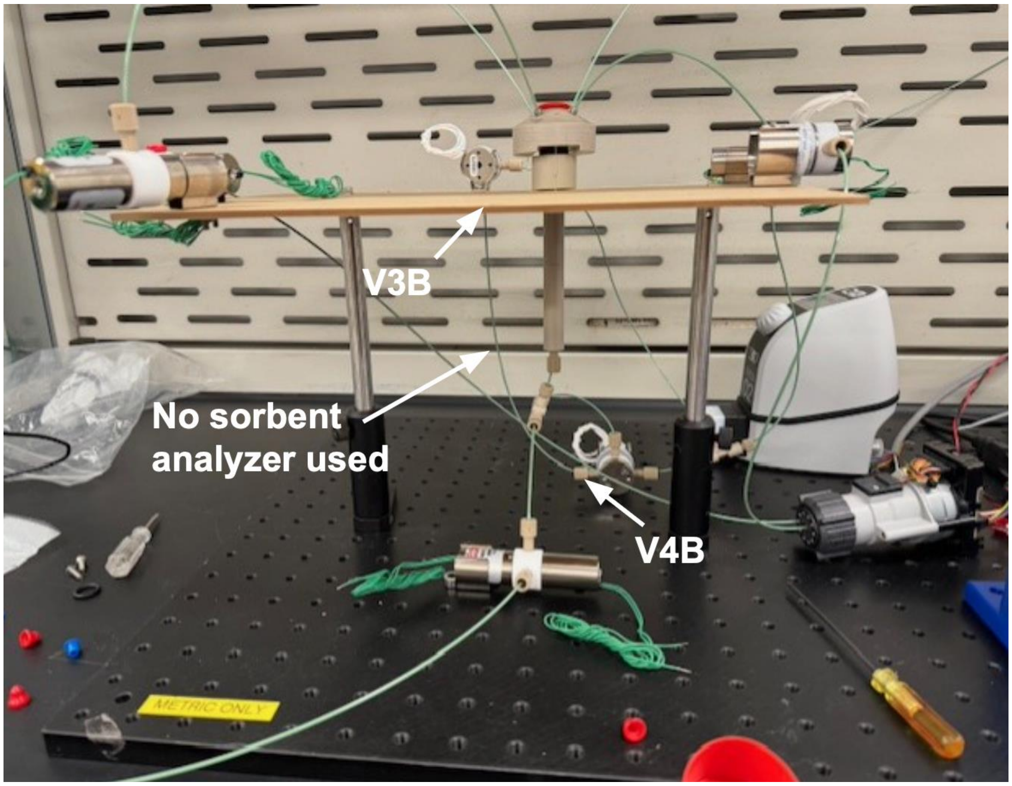
8. Connect the Pump Output Tee Common port to the output of the Diaphragm Pump, as shown. Place pump on breadboard in approximate location shown so that none of the fluidic connectors are strained/pulled from their housing.

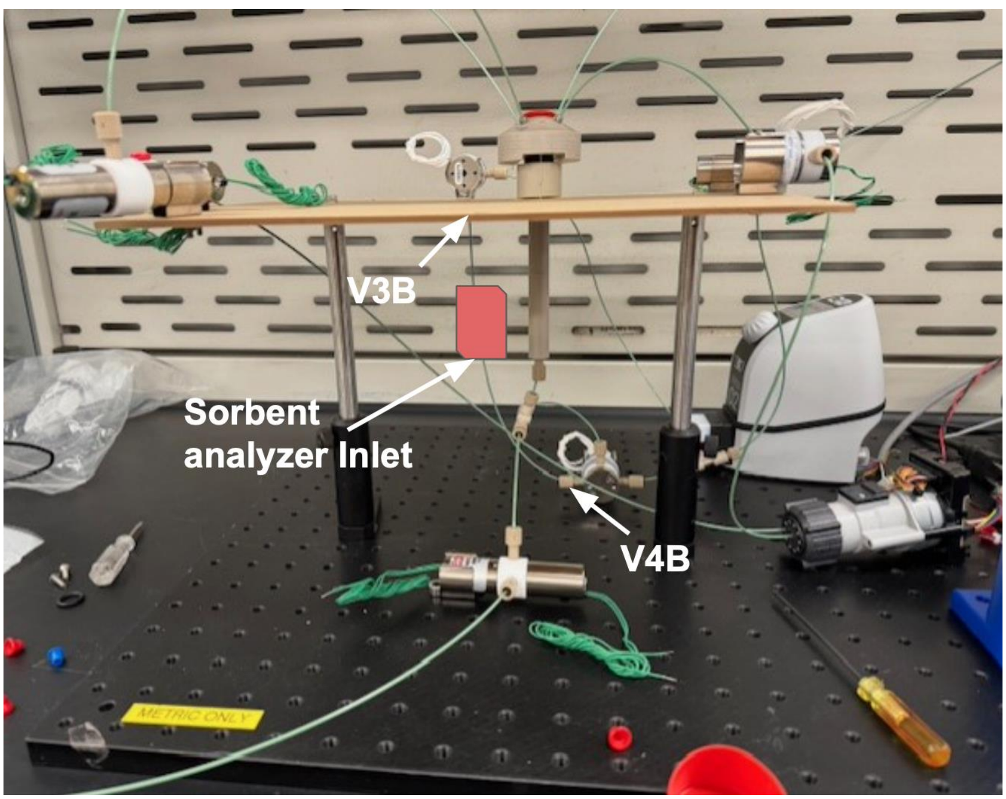


9. Mount Valves V1, V2, V3, V5, and V7 on the Main Panel in the approximate locations shown.

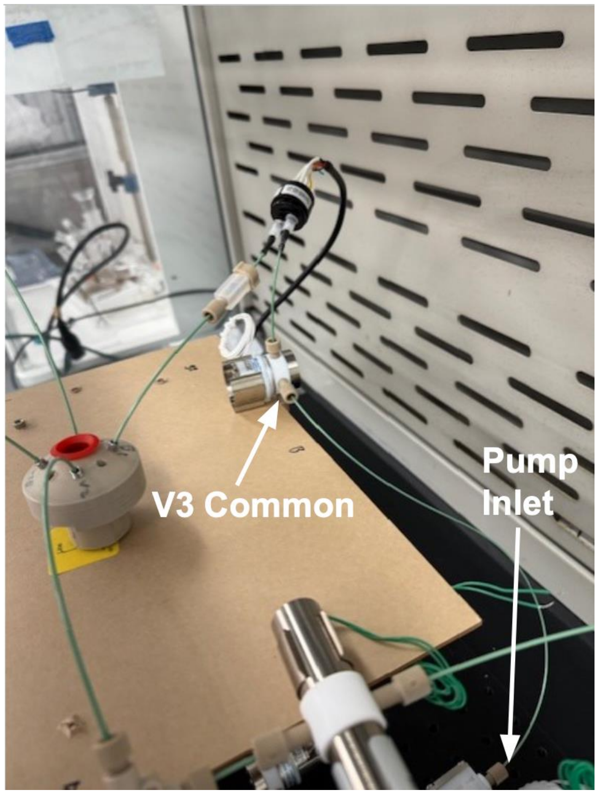


10. If no sorbent analyzer used, Connect port V4B to port V3B as shown. If using sorbent analyzer, Connect port V4B to Sorbent Analyzer Inlet, and Connect port V3B to Sorbent Analyzer Outlet.

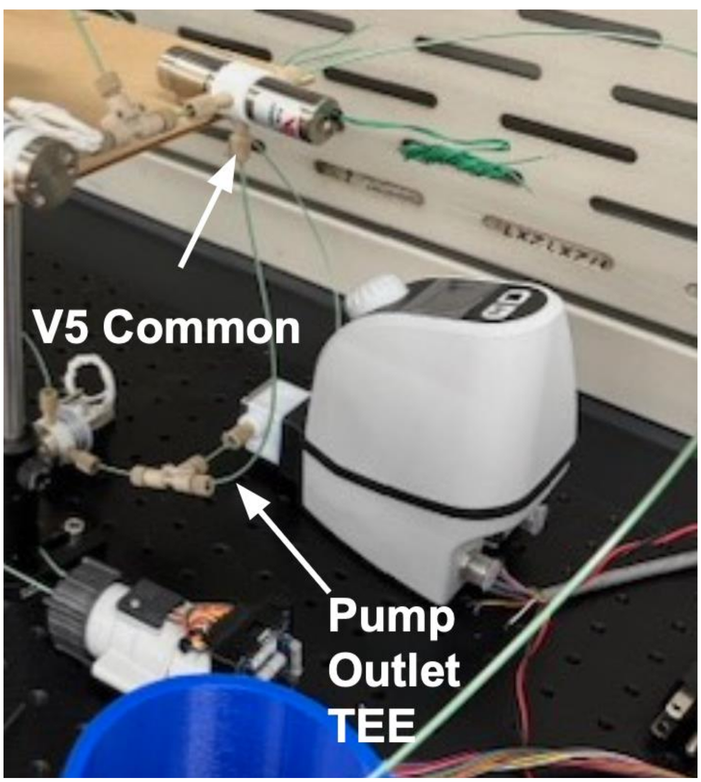




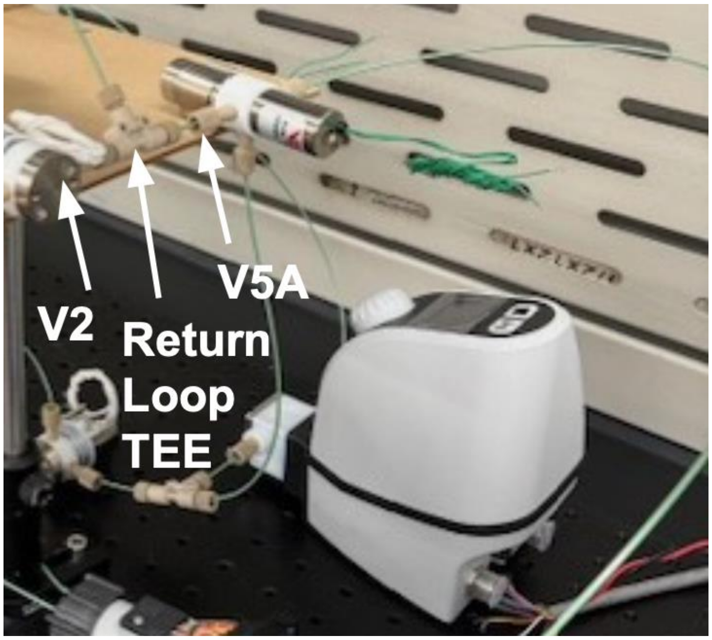
11. Connect port V3 Common to Diagraphm Pump Inlet



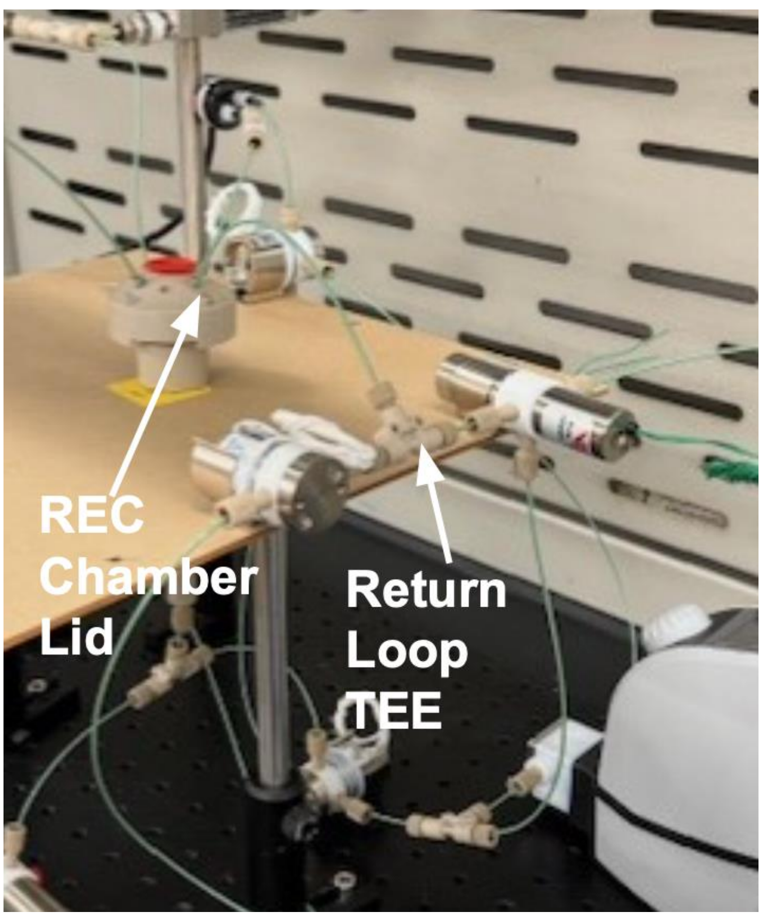
12. Connect Port V5 Common to the remaining Pump Outlet Tee Port



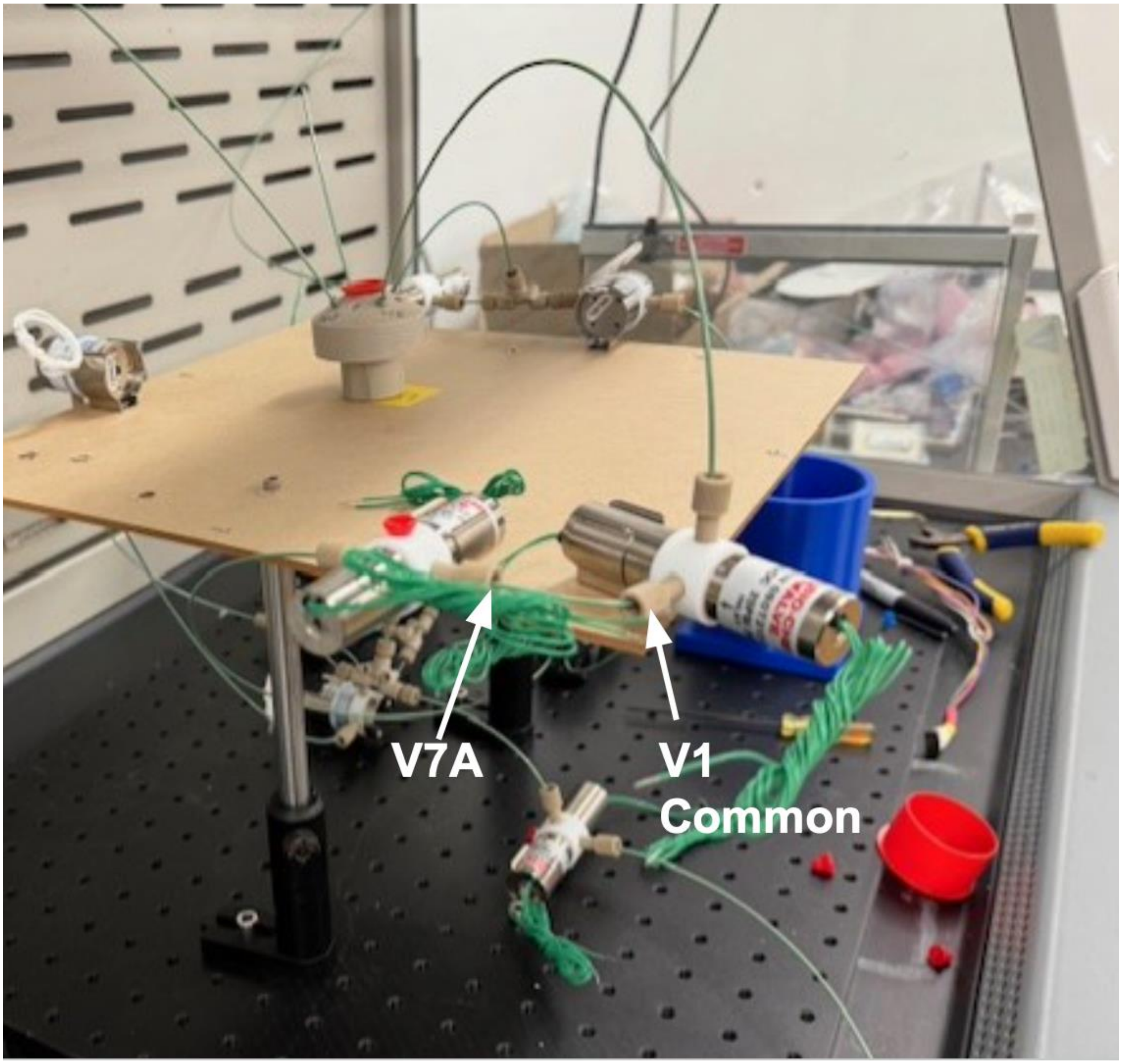
13. Connect port V5A to one end of the Return Loop TEE. Connect the other Return Loop TEE port to valve port V2 Outlet, as shown, leaving the Return Loop Tee Common open.



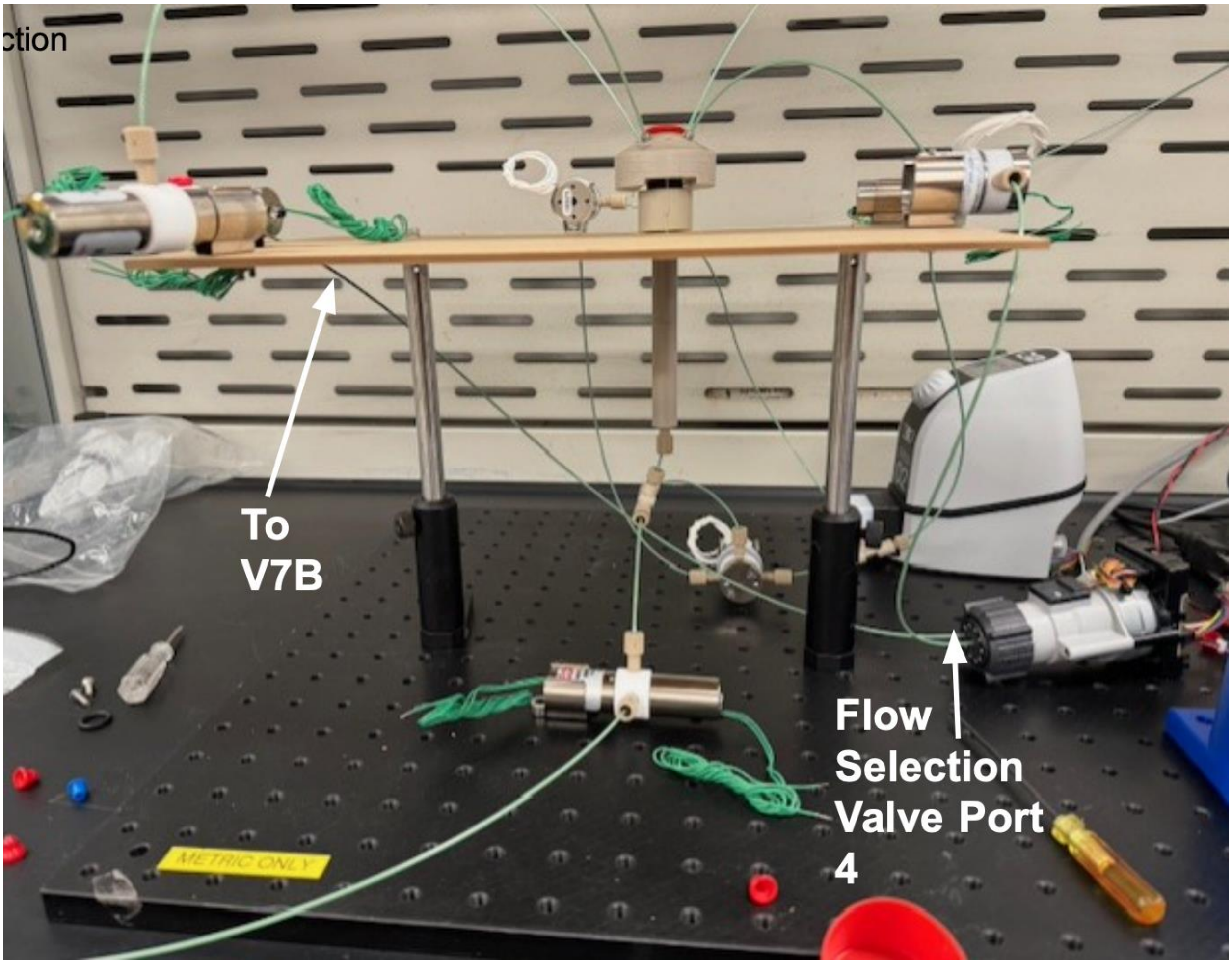
14. Connect Return Loop Tee Common port, and port V1B, to the REC Chamber Lid



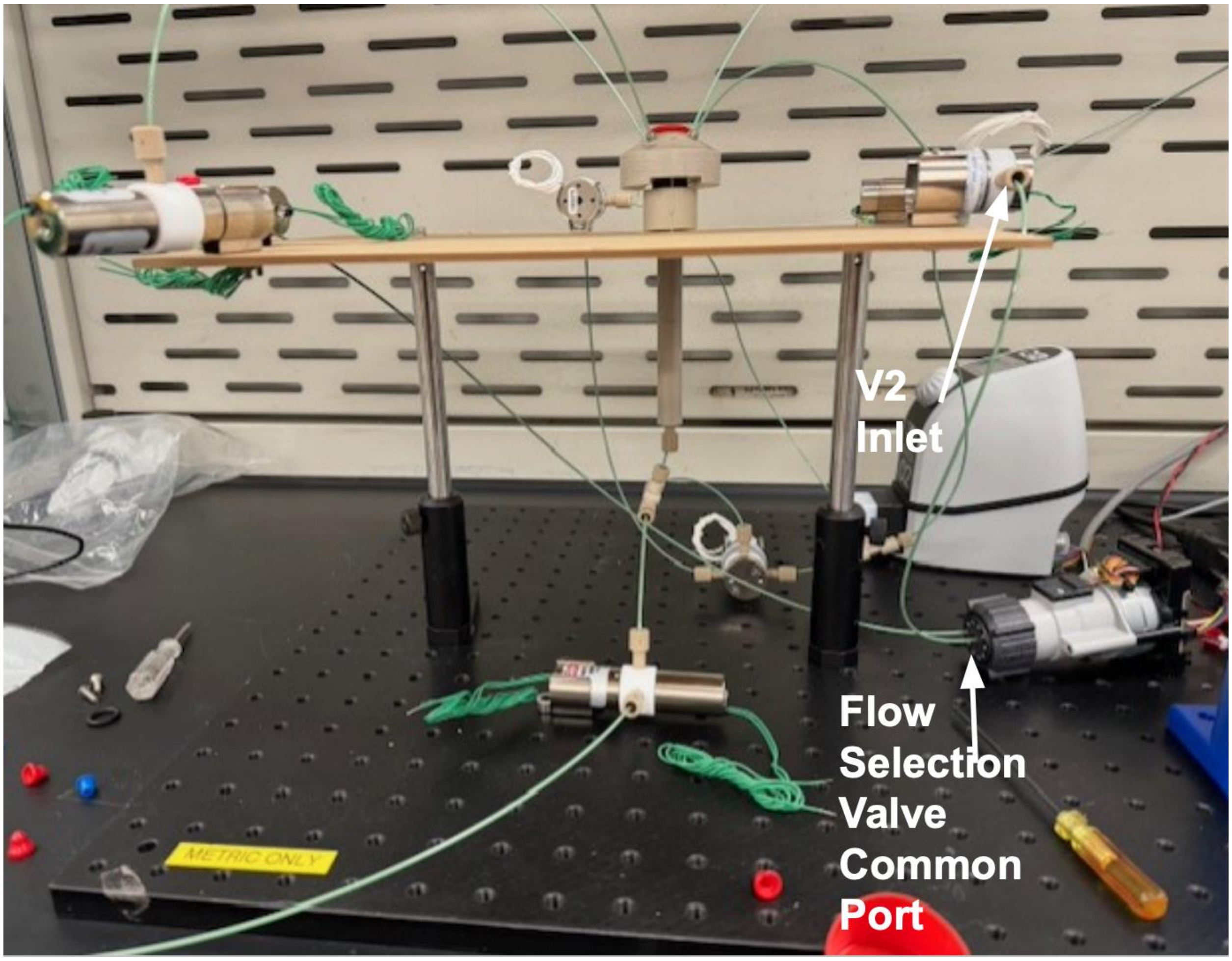
15. Connect V1 Common port to port V7A



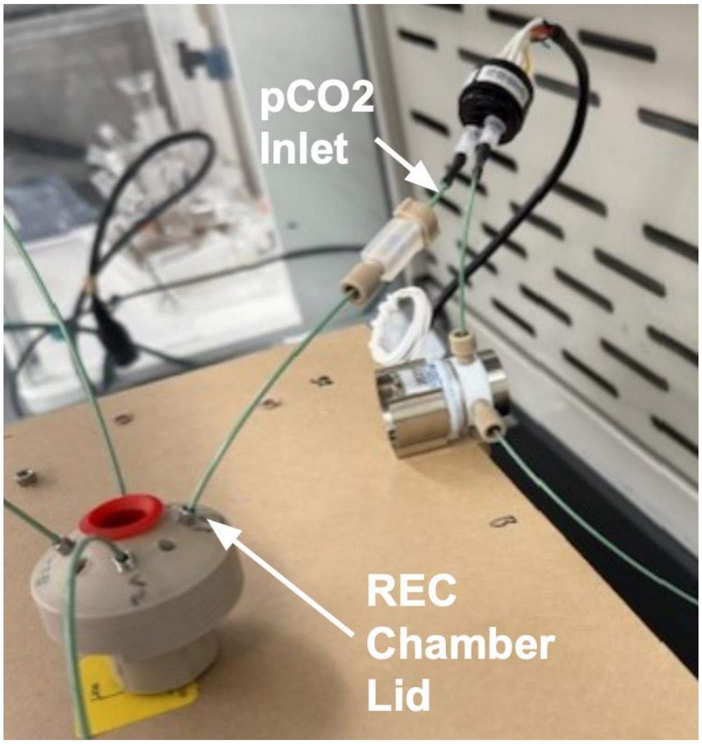
16. Connect port V7B to Flow Selection Valve Channel 4



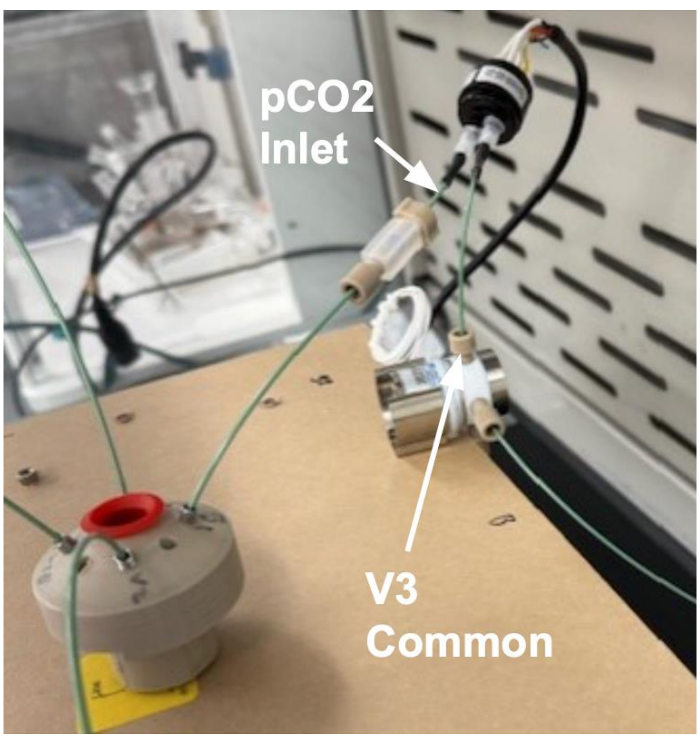
17. Connect port V2 Inlet to Flow Selection Valve Common Port (center port)



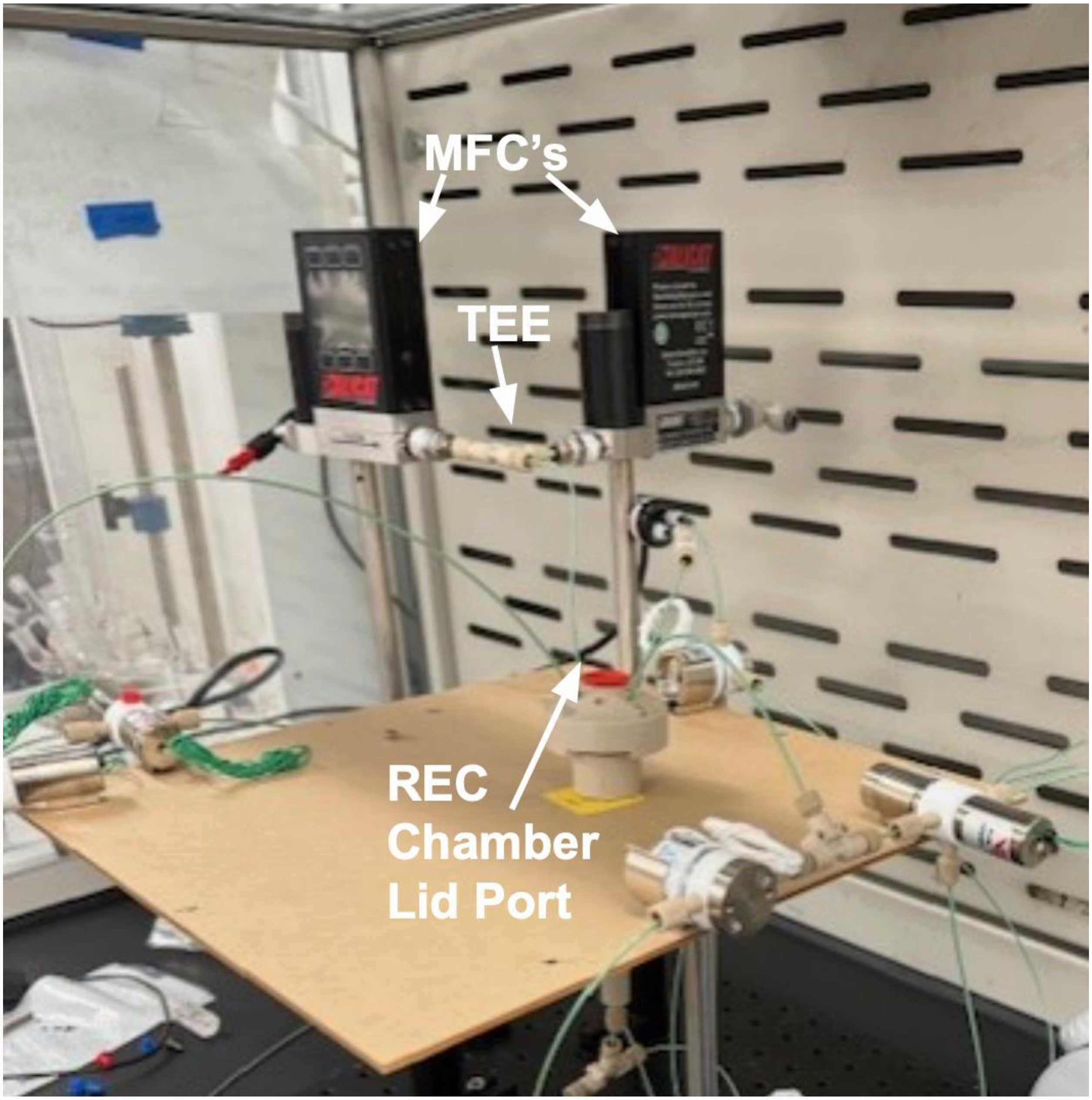
18. Connect pCO2 inlet to REC Chamber Lid



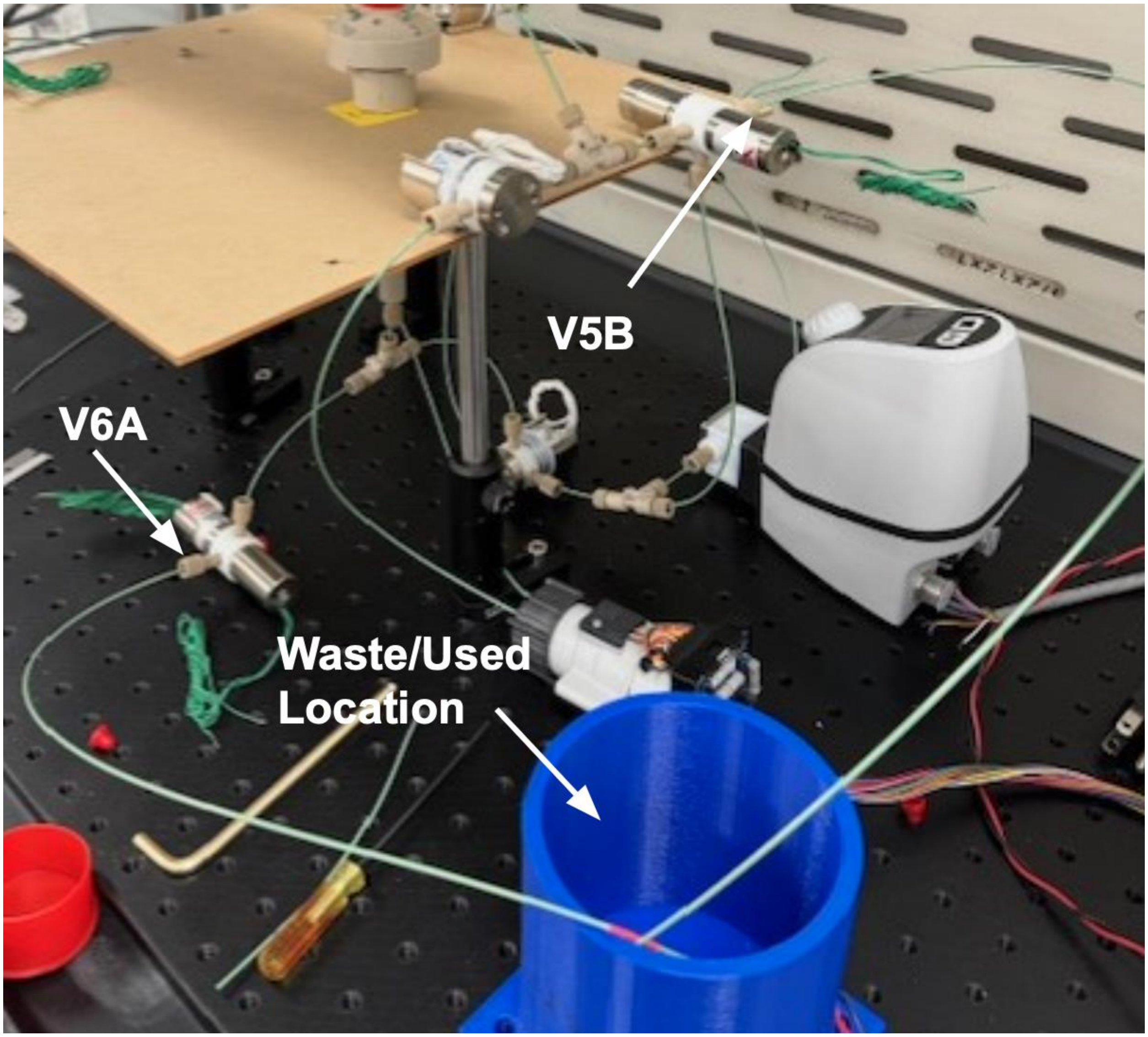
19. Connect pCO2 outlet to port V3 Common



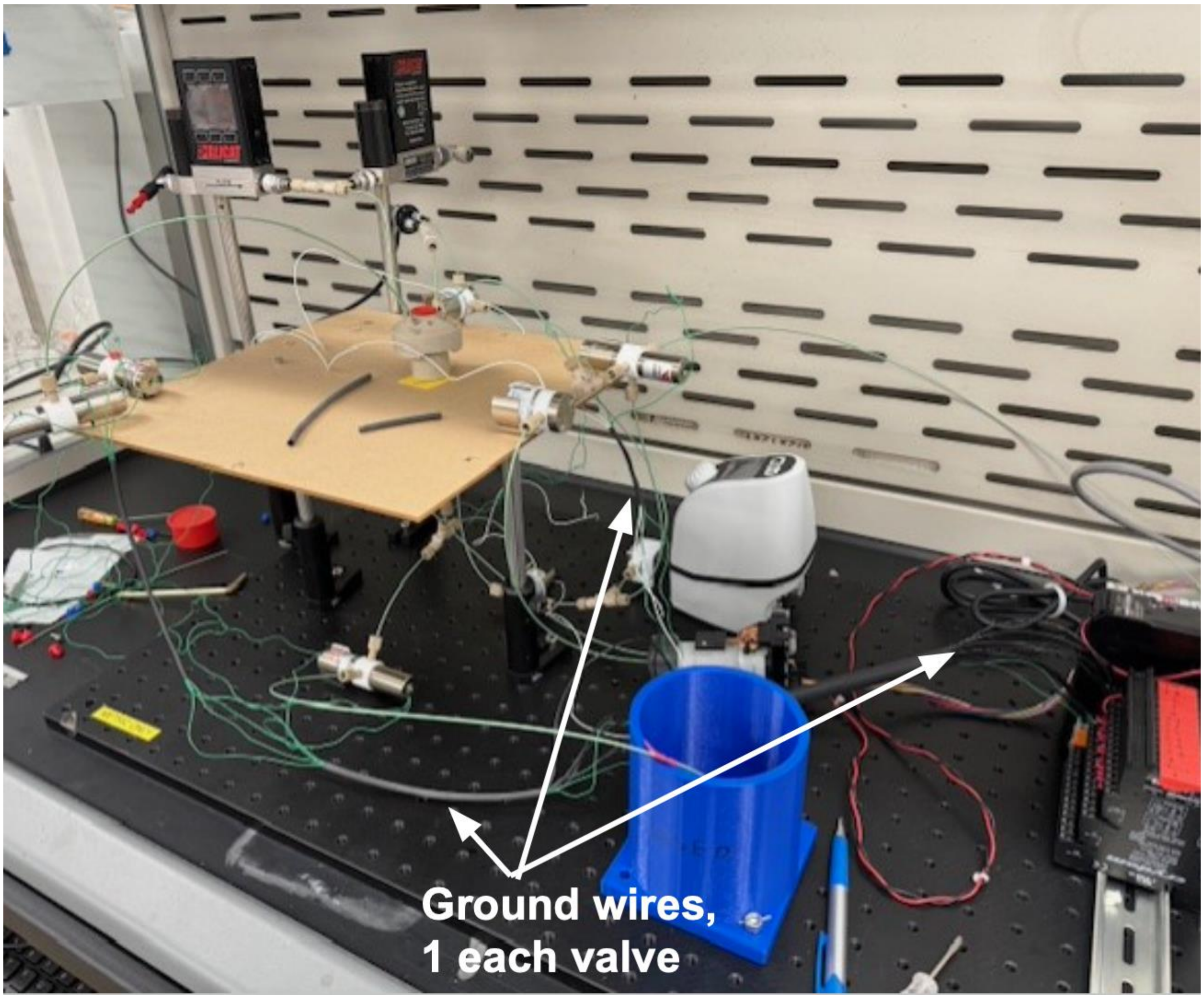
20. Configure MFC's such that they outlet into two ports of a TEE. Connect the remaining port of the Gas Metering TEE to the REC Chamber Lid



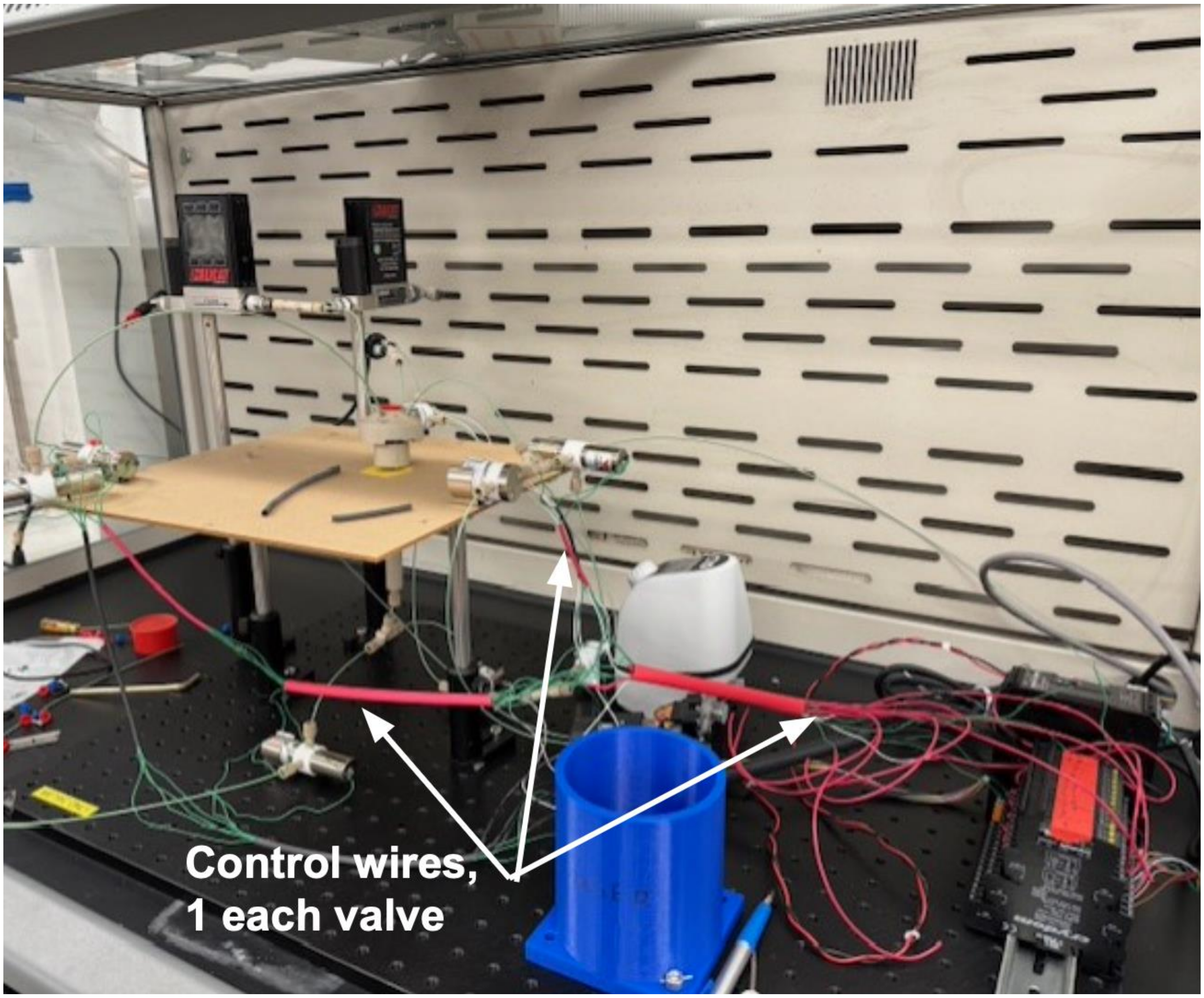
21. Route the tubes from V6A and V5B to the designated Waste/Used repository location to ensure they reach.



22. Wire all the valves to GROUND. Each valve has a pair of wires for electronic control. One wire from each pair is used to connect each valve to a common Ground, polarity is not consequential. As shown, the Omron 12V source Ground is wired to a Wago distribution panel, which allows all Valve Wires to connect to Ground simultaneously. Some wires may need to be extended if they do not reach the power distribution rack.



23. Wire all the Valves to their respective control relays. Each valve will have 1 wire available after connecting the other to ground (previous step). Ensure this free wire is connected to the proper control relay, observing relay load polarity to ensure proper function.



24. Plumb gas lines into the hood. Connect background gas to Valve port V7 Common. Connect Syringe Pumps to respective Multi Channel Valve Port

25. Test all valves and connections for leaks and proper functioning.

End of assembly instructions.

Additional guidance for assembly and diagnostics

Replication of the CCSI instrumentation requires prerequisite knowledge of, and familiarity with, the constituent components. This body of knowledge includes, but is not limited to, their safe and proper usage, and any general operational considerations that can be found in the user manual of each component. Below we provide some techniques, tips, and tricks that can aid in the assembly and validation of the CCSI.

Syringes w/ Luer Lock Tips

It is very helpful to be able to manually pressurize or evacuate the system without having to use high pressure gas cylinders or knock-out drum-coupled vacuum sources. The easiest way to do this is by using a syringe made of instrument compatible materials. For the demonstrated aqueous configuration, a 60 mL plastic syringe with rubber plunger was acceptable.

Syringes come with many types of tips for connecting to external tubing, but the authors have found those with Luer Lock (or similar twist lock systems) tips allow for the largest range of tube sizes and materials to be used. For the present work, the authors utilized barbed tip luer lock connectors to interface with Masterflex peristaltic pump tubing.

Flex Tubing (Including systems using solely hard tubing)

As alluded to, flexible plastic or rubber tubing can be very useful in quickly connecting to, and disconnecting from, other types of tubing. In low pressure situations flex tubing can slip directly onto the outer diameter of hard plastic, or metal, tubing. Increased pressures may require using a barb/luer lock connector, or flaring the OD of the hard tubing. High Pressure applications usually are not suitable for flex tubing, and hard tubing with compression fittings is recommended. Chemical compatibility with sorbent media must also be considered.

Selection Valve Solenoid Direction

Selection valves are comprised of any number of solenoid actuated diaphragm isolation valves connected in parallel to a common port. In the case of 3-way selection valves, the often-used symmetric port geometry can make the determination of which valve controls which port unclear. Determining which port is controlled by which solenoid is paramount for proper control wiring and ensuring safe operation of the instrument.

In the case of Normally Closed solenoid valves, applied pressure to the common port will not result in any flow. Thus, to test ambiguous port configurations, one only need apply pressure to the common port (e.g. via manually operated syringe pump compression) and sequentially open each unknown solenoid using an appropriate measurement method to detect flow. Common practice involves placing tubes in water and watching for which tube makes bubbles, indicating the port is controlled by the actuated solenoid valve.

CO_2 Meter Connection to Headspace Loop

The CO_2 sensor reported here (SprintIR-6S) is delivered with a black housing that has two tapered ports for external connections to tubing. Most of the time, flexible tubing like that used in peristaltic pumps is sufficient for making airtight seals on the ports. In other cases, material compatibility may prevent the use of flexible tubing, and so one must find a suitable way to connect hard tubing to the ports. The authors have found that the Luer Lock Socket style tube connectors snugly fits onto the ports. One must then find an acceptable method for mating the desired recirculation tubing to this Luer Lock Port.

For example, polyether ether ketone (PEEK) is a hard tubing with broad chemical resistance. It is also readily available and commonly used in the physical sciences, making it an attractive choice for building systems where chemical compatibility and contamination is paramount. PEEK tubing is also a choice material for chromatography systems, and there exists a wide range of available fluidic connectors for making seals between PEEK tubing and various vessels. A common port designation for these seals is the ¼”-28 Flat Bottom specification, allowing for tubing to be hermetically sealed to any vessel with this threading with the use of a specially designed nut-and-ferrule assembly. Thus, all one needs to do is use a Luer Lock Socket with ¼”-28 threading, and then utilize a Union Joint (common from HPLC suppliers) to connect to external recirculation tubing.

Liquid and gas handling procedures

The following procedure cleans the cell using liquid fille-rinse cycles with gas purge. This is the “Clean” procedure, which is also performed as needed during “Initialize”

Notes: V8 used for water syringe refill. V9 selects Background Gas and is set prior to starting												
Step: / *State Description*	**Valve States**											
Liquid Fill:	**V1A**	**V1B**	**V2**	**V3**	**V4**	**V5A**	**V5B**	**V6A**	**V6B**	**V7A**	**V7B**	**P1**
Valve Prep	ON	ON	ON	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
H2O Inject	H2O Syringe Inject (Dead Volume + Target Volume)											
Clear Liquid Valve	ON	ON	ON	A	A	OFF	OFF	OFF	OFF	OFF	ON	OFF
wait	wait= LiquidFillwait											
H2O Withdraw	H2O Syringe Withdraw (Retraction Volume)											
H2O Inject	H2O Syringe Inject (Backlash Volume)											
Headspace REC												
Gas Off	ON	ON	ON	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Isolate Liquid Valve	ON	ON	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Seal Cell	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Measure CO2	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	ON
wait	wait= WaitCO2MeasurementTime											
Pump OFF	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Liquid Drain												
Valve Prep	OFF	ON	OFF	A	A	OFF	OFF	ON	OFF	ON	OFF	OFF
wait	wait= LiquidSamplePurge											
Gas OFF	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Cleaning (Dilution)												
Valve Prep	ON	ON	ON	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF

H2O Syringe Inject	H2O Syringe Inject (Dead Volume + Clean Volume)											
Clear Liquid Valve	ON	ON	ON	A	A	OFF	OFF	OFF	OFF	OFF	ON	OFF
wait	wait= LiquidFillWait											
H2O Syringe Withdraw	H2O Syringe Withdraw (Retraction Volume)											
H2O Syringe Inject	H2O Syringe Inject (Backlash Volume)											
Gas OFF	ON	ON	ON	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Liquid Valve Close	ON	ON	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Cell Seal	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Measure CO2	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	ON
wait	wait= WaitCO2MeasurementTime											
Pump OFF	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Liquid Drain	OFF	ON	OFF	A	A	OFF	OFF	ON	OFF	ON	OFF	OFF
wait	wait= LiquidCleanPurge											
Gas OFF	OFF	OFF	OFF	A	A	OFF	OFF	ON	OFF	OFF	OFF	OFF
Seal Cell	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Syringe REFILL Prep	V8=B											
H2O Syringe Withdraw	H2O Syringe Withdraw (Target Volume + Clean Volume +Backlash Volume)											
H2O Syringe Inject	H2O Syringe Inject (Backlash Volume)											
Syringe Refill Finish	V8=A											
Headspace Fill	**V1A**	**V1B**	**V2**	**V3**	**V4**	**V5A**	**V5B**	**V6A**	**V6B**	**V7A**	**V7B**	**P1**
CO2 Line Dilute	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	ON
wait	wait= WaitCO2DilutionTime											
Pump OFF	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
HSmain purge	OFF	ON	OFF	A	A	OFF	OFF	ON	OFF	ON	OFF	OFF
wait	wait= HeadspacePurge											
Gas OFF	OFF	OFF	OFF	A	A	OFF	OFF	ON	OFF	OFF	OFF	OFF

Seal Cell	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF

The following procedure initializes the headspace using the primary process gas (9.9% CO_2 in the present work)

Notes: V8 used for water syringe refill. V9 selects Background Gas and is set prior to starting.												
<u>Step:</u> / *State Description*	Valve States											
<u>Main Headspace Purge</u>	**V1A**	**V1B**	**V2**	**V3**	**V4**	**V5A**	**V5B**	**V6A**	**V6B**	**V7A**	**V7B**	**P1**
HSMain purge via V1-6	OFF	ON	OFF	A	A	OFF	OFF	ON	OFF	OFF	OFF	OFF
wait	wait= HSpurge1_duration											
HSMain purge via V2-6	OFF	OFF	ON	A	A	OFF	OFF	ON	OFF	OFF	ON	OFF
wait	wait= ManPurge1_duration											
Line purge via V2-5	OFF	OFF	ON	A	A	OFF	OFF	ON	OFF	OFF	OFF	OFF
wait	wait											
Line purge via V2-5	OFF	ON	OFF	A	A	ON	ON	OFF	OFF	ON	OFF	OFF
wait	wait= AlphaPurge1_duration											
<u>AUX PROBE PURGE</u>												
Aux Probe purge via V5	ON	ON	OFF	B	B	OFF	ON	OFF	OFF	ON	OFF	OFF
wait												
Pump On	ON	ON	OFF	B	B	OFF	ON	OFF	OFF	ON	OFF	ON
wait	wait= ProbePurge1_Duration											
<u>pCO2 SENSOR PURGE</u>												
pCO2 Sensor purge via V5	ON	ON	OFF	B	B	OFF	ON	OFF	OFF	ON	OFF	ON
wait	wait= SensorPurge1_duration											
Pump Off and Equilibrate	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
<u>Dilution purge</u>												

Line dilution V4-Tee	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	ON
wait	wait= DelaDilute1_duration											
Pump Off	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Main Headspace Purge												
*HSMain purge via V1-6**	OFF	ON	OFF	A	A	OFF	OFF	ON	OFF	ON	OFF	OFF
wait	wait= HSpurge2_duration											
HSMain Pressure equilibration	ON	ON	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Cell Seal	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Headspace Evaluation												
HS Composition Verification	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	ON
wait	wait= WaitCO2Measurement											
Pump Off	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Evaluate	if pCO2 =/= Background, GOTO HSMain purge via V1-6* else Continue to Pre Clean Procedure REC Chamber, AUX Probe Cell, and all Lines are at 1ATM of the background gas. All Valves OFF.											
	V1A	**V1B**	**V2**	**V3**	**V4**	**V5A**	**V5B**	**V6A**	**V6B**	**V7A**	**V7B**	**P1**
Pre Clean Procedure												
Liquid Fill												
Valve Prep	ON	ON	ON	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
MCVset	MCV=H2O											
H2O Inject	H2O Syringe Inject											
wait	wait= pressureEqualization											
MCVset	MCV= GAS											
Clear MCV	ON	ON	ON	A	A	OFF	OFF	OFF	OFF	OFF	ON	OFF

wait	wait= LiquidFillwait											
Headspace REC												
Gas Off	ON	ON	ON	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
wait	wait= pressureEqualize											
MCVset	MCV= CLOSED											
Isolate Liquid Valve	ON	ON	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Seal Cell	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Measure CO2	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	ON
wait	wait= WaitCO2MeasurementTime											
Pump OFF	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Liquid Drain												
Valve Prep	OFF	ON	OFF	A	A	OFF	OFF	ON	OFF	ON	OFF	OFF
wait	wait= LiquidSamplePurge											
Gas OFF	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Cleaning (Dilution)												
Valve Prep	ON	ON	ON	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
MCVset	MCV=H2O											
H2O Inject	H2O Syringe Inject											
wait	Wait= liquidPressureEqualization											
MCVset	MCV= GAS											
Clear MCV	ON	ON	ON	A	A	OFF	OFF	OFF	OFF	OFF	ON	OFF
wait	wait= LiquidFillwait											
Gas OFF	ON	ON	ON	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
wait	wait= pressureEqualization											
MCVset	MCV=CLOSED											
Liquid Valve Close	ON	ON	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF

Cell Seal	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Measure CO2	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	ON
wait	wait= WaitCO2MeasurementTime											
Pump OFF	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Liquid Drain	OFF	ON	OFF	A	A	OFF	OFF	ON	OFF	ON	OFF	OFF
wait	wait= LiquidCleanPurge											
Gas OFF	OFF	OFF	OFF	A	A	OFF	OFF	ON	OFF	OFF	OFF	OFF
Seal Cell	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Syringe REFILL Prep	V8=B											
H2O Syringe Withdraw	H20 Syringe Withdraw (Refill Volume)											
H2O Syringe Inject	H20 Syringe Inject (Backlash Volume)											
Syringe Refill Finish	V8=A											

The following procedure is for the “Liquid fill” and “CO_2 sorption” steps for the KOH experiments in which the sorbent media is diluted with water at the desired ratio.

Notes: V8 used for water syringe refill. V9 selects Background Gas and is set prior to starting												
Step: **/** *State Description*	Valve States											
Headspace Fill* (LOOP=2)	**V1A**	**V1B**	**V2**	**V3**	**V4**	**V5A**	**V5B**	**V6A**	**V6B**	**V7A**	**V7B**	**P1**
CO2 Line Dilute	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	ON
wait	wait= WaitCO2DilutionTime											
Pump OFF	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
HSmain purge	OFF	ON	OFF	A	A	OFF	OFF	ON	OFF	ON	OFF	OFF
wait	wait= HeadspacePurge											
Gas OFF	OFF	OFF	OFF	A	A	OFF	OFF	ON	OFF	OFF	OFF	OFF
Seal Cell	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF

Liquid Fill												
Valve Prep	ON	ON	ON	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
MCVset	MCV=KOH											
KOH Inject	KOH Syringe Inject											
wait	wait= pressureEqualization											
MCVset	MCV= H2O											
H2O Inject	H2O Syringe Inject											
wait	wait= pressureEqualization											
MCVset	MCV= GAS											
Liquid Valve Clear	ON	ON	ON	A	A	OFF	OFF	OFF	OFF	OFF	ON	OFF
wait	wait= LiquidFillWait											
Gas OFF	ON	ON	ON	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
wait	wait= pressureEqualization											
MCVset	MCV=CLOSED											
Valve Pressure Equilibrate	ON	ON	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Seal Cell	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
CO2 Sorption												
Measure pCO2	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	ON
wait	wait= WaitCO2MeasurementTime											
Pump OFF	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Liquid Drain												
Valve Prep	OFF	ON	OFF	A	A	OFF	OFF	ON	OFF	ON	OFF	OFF
wait	wait = LiquidSamplePurge											
Gas OFF	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Clean (LOOP=4)												

Valve Prep	ON	ON	ON	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
MCVset	MCV=H2O											
H2O Inject	H2O Syringe Inject											
wait	Wait= liquidPressureEqualization											
MCVset	MCV= GAS											
Clear MCV	ON	ON	ON	A	A	OFF	OFF	OFF	OFF	OFF	ON	OFF
wait	wait= LiquidFillwait											
Gas OFF	ON	ON	ON	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
wait	wait= pressureEqualization											
MCVset	MCV=CLOSED											
Liquid Valve Close	ON	ON	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Cell Seal	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Measure CO2	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	ON
wait	wait= WaitCO2MeasurementTime											
Pump OFF	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Liquid Drain	OFF	ON	OFF	A	A	OFF	OFF	ON	OFF	ON	OFF	OFF
wait	wait= LiquidCleanPurge											
Gas OFF	OFF	OFF	OFF	A	A	OFF	OFF	ON	OFF	OFF	OFF	OFF
Seal Cell	OFF	OFF	OFF	A	A	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Syringe REFILL Prep	V8=B											
H2O Syringe Withdraw	H20 Syringe Withdraw											
H2O Syringe Inject	H2O Syringe Inject (Backlash Volume)											
Syringe Refill Finish	V8=A											
Continue?	If Sample Remaining = Yes GOTO ***Headspace Fill**** Else FINISH											

CCSI automation in helao-async

HELAO Environment Setup (tested on Windows 7 x64)

1. Unzip “helao_code/helao-async” and “helao_code/helao-core” from the zip archive to a local directory. The two repositories must be colocated for the setup script to function properly.
2. Install miniconda3 from https://docs.conda.io/projects/miniconda/en/latest/index.html
3. Open a miniconda command prompt and change directory to the unzipped “helao-async” path.
4. Run “setup_env.bat from” the command prompt to install the python environment packages listed in “helao_pinned_win-64.yml”
5. Run “conda activate helao” to activate the newly installed environment.
6. The unzipped code folders are not valid git repositories, but HELAO integrates version tracking using git commit hashes. For this demonstration, initialize local git repositories by running “git init” from the “helao-async” and “helao-core” directories.

Edit the instrument configuration file

1. Open “helao-async/helao/configs/ccsi1.yml” for editing
2. CCSI-specific experiments (“helao-async/helao/experiments/CCSI_exp.py”) and sequences (“helao-async/helao/sequences/CCSI_seq.py”) are loaded from lines 4 and 10 respectively. Update these paths as needed for new/modified code.
3. Modify line 15 “root: C:\INST_hlo” to the desired save location for data and instrument logs.
4. Under the “servers” group, modify all “host” key values to the local machine’s domain name, external address, or internal address. Local address 127.0.0.1 may be used to restrict access to the host machine.
5. Under the “servers/IO” subgroup, modify line 32 “galil_ip_str” to the Galil DMC-41x3 I/O controller’s IP address.
6. Under the “servers/NI” subgroup, modify the “cDAQ1Mod1/port0/line*” values to match valve-terminal wiring on the NI DAQ board.
7. Under the “servers/CO2SENSOR” subgroup, modify line 102 “port: COM9” to the correct COM port for the SprintIR-6S 20% CO2 Sensor.
8. Under the “servers/SYRINGE*” subgroups, modify lines 111 and 122 “port” key values to the correct COM ports for the KDS Scientific Legato 100 syringe pumps (SYRINGE0 for the adsorbent solution, SYRINGE1 for cleaning solution).
9. Under the “servers/MFC” subgroup, modify line 135 “port: COM7” to the correct COM port for the Alicat Scientific MC-05SCCM-D mass flow controller.
10. Comment out or remove the “servers/DB” group to disable automatic data upload to AWS.

Launch HELAO using the ccsi1 configuration file

1. Open a miniconda command prompt and run “conda activate helao” to activate the conda environment.
2. Change directory to the unzipped “helao-async” path.
3. Start the HELAO launcher script by running “helao.bat ccsi1” which will launch processes for device API servers (action servers), the scheduler API server (orchestrator), visualizers, and web UI (operator).

Using the web UI operator to setup, queue, and start a CCSI initialization sequence

1. The “CCSI_initialization” sequence prepares the instrument for subsequent measurements by purging the cell headspace with CO_2 to a predefined level.
2. Open a web browser and browse to “http://host_address:5002” where “host_address” is the value specified under the “servers/ORCH” subgroup’s host key in the edited ccsi1.yml configuration file.
3. Under the “Sequence Selection” tab, choose “CCSI_initialization” from the dropdown list. Modify sequence parameters as desired.
4. Click “Append seq to exp plan” to add the sequence to the planning buffer.
5. Scroll down to the “Orchestrator” section to review experiments listed in the sequence planning buffer.
6. Click “Add exp plan” to send the planning buffer to the HELAO orchestrator’s sequence queue.
7. Click “Start Orch” to begin processing the orchestrator queue.

Bill of materials, total estimated cost, 12,941 to 15,759 USD

Category	Item	Detail	Quantity	Price (2023 estimate, USD)	Notes/Links
Echem Cell Fabrication	Materials for Electrodes (Pt, glassy carbon, Membranes etc...)		1	450.00	
	Recirculation Cap		1	500.00	
	Recirulation Chamber		1	500.00	
Mounting / Construction	80/20 Railings (To mount Sorbent Analyzer and Frame)		1	200.00	4x 1.5x3x12, 2x 1.5x1.5x12, 1x 1.5x3x24
	80/20 Brackets and Fasteners		1	50.00	4 Each Single and Double Brackets w/ fasteners
	12x12 mounting surface		1	8.46	https://www.mcmaster.com/8560K191/
	Mounting Hardware Machining		1	100.00	
Gas Management/ Control	Alicat MFC		2	2,500.00	Custom quoted MFC depends on target gas composition and desired pressure ranges.
	Fluid valves (3 way Selection Valves, BioChem)	080T312-62-5	4	1,740.72	Various Suppliers. Arcmed quoted the present system components
	Fluid Valves (3-Way Switching Valves, NResearch	HP225T031	3	284.67	HP225T031
	Flat bottom fittings with Ferrules	Cost from cole parmer for fittings with ferrule	25	137.50	https://www.coleparmer.com/i/idex-flangeless-fitting-standard-knurl-natural-peek-1-16-od-tubing-1-4-28-flat-bottom-10-pk/0202077?PubID=UX&persist=true&ip=no&gclid=CjwKCAiAjPyfBhBMEiwAB2CCIk7-9zfrcQEinCNRwiky4puqplawSaUVCuciKgWSjsp9MkhAnAPnSRoCabcQAvD_BwE
	Alicat Power Cables		2	110.00	
	Recirculation pump	Peristaltic pump OR Diaphragm pump	1	1,018.00 OR 3,836.00	https://us.vwr.com/store/item/NA5192314/masterflex-c-l-analog-variable-speed-pump-systems-with-single-channel-pump-head-avantor OR https://knf.com/en/us/solutions/laboratory-equipment/details/simdos-02-fem-102-rc-p

Liquid Management/ Control	Tubing/tubing for organic solvent	25ft/pack	2	177.00	Green PEEK tubing: MMC# 51085K48
	PEEK Tees and chemically compatible fluidic connectors	Tees, fittings	4	185.60	https://www.coleparmer.com/i/idex-low-pressure-tee-assembly-natural-peek-0-040-bore-1-16-od-tubing-1-4-28-flat-bottom-1-ea/0201432#eb-item-specification
	Switching Valve	MLP778-606	1	1,000.00	https://www.biotechfluidics.com/product/titanextm-10-position11-port-with-pcb-2/#tab-description
	Chemically Compatible O-rings		2	10.00	https://www.mcmaster.com/9452K111/
Computer/ Electronics	Computer		1	750.00	
	5V Power Supply	Omron S8VK-G03005	1	76.61	https://www.digikey.com/en/products/detail/omron-automation-and-safety/S8VK-G03005/4079540
	12V Power Supply	Omron S8VK-G06012	1	89.06	https://www.digikey.com/en/products/detail/omron-automation-and-safety/S8VK-G06012/3929120
	24V Power Supply	Omron S8VK-C12024	1	83.31	https://www.digikey.com/en/products/detail/omron-automation-and-safety/S8VK-C12024/4987817
	Wago Distribution Panels	2946-2001-1401-ND	10	21.30	https://www.digikey.com/en/products/detail/wago-corporation/2001-1401/13549604
	Various Electrical Wiring components		1	50.00	
	Relays	Crydom DRA	15	858.60	https://www.digikey.com/en/products/detail/sensata-crydom/DRA-CN024D05/2263904?s=N4IgTCBcDaICICUCCBhAcgBjAFjhgrCALoC%2BQA
	Powerstrip		2	40.00	
	NI DAQ	cDAQ-9174	1	1,501.00	https://www.ni.com/en-us/support/model.cdaq-9174.html
Headspace measurement	CO2 sensor 1 with sensor development kit	0-60% CO2	1	499.00	https://www.co2meter.com/collections/20-percent-co2-sensor/products/sprintir-r-20-co2-sensor?variant=32046774026358