Trace Contaminant Control Test Bed for Evaluation of TCC Prototypes with Vacuum Regenerable and Non-Regenerable Sorbents

Nicholas F. Materer,[1]
Department of Chemistry, Oklahoma State University, Stillwater, OK 74078

Evgueni B. Kadossov,[2] John R. Tidwell,[3] Michael L. Teicheira,[4] Shoaib Shaikh,[5]
XploSafe, Stillwater, OK 74074

and

Cinda Chullen[6]
NASA Johnson Space Center, Houston, TX 77546

A new Space Suit Exploration Portable Life Support System (xPLSS) is being designed, built, integrated, and tested into the Extra-Vehicular Mobility Unit (xEMU). The Trace Contaminant Control (TCC) system is a component in the oxygen ventilation loop of xPLSS that removes contaminants generated by the crewmembers' metabolic processes. The primary trace contaminants that must be removed include ammonia (NH_3), carbon monoxide (CO), formaldehyde (CH_2O), and methyl mercaptan (CH_3SH). The current state-of-the-art TCC sorbent is non-regenerable activated carbon. As a non-regenerable sorbent, its use negatively impacts the logistics for future missions. Thus, an ideal solution can be a vacuum-regenerable sorbent integrated with the xPLSS CO_2/H_2O removal system. To test new designs and sorbents, XploSafe has constructed a recirculating test bed that mimics the environment within the xPLSS by providing concentrations of the trace contaminant analytes generated at the operating temperature, humidity, pressure, and flow rates of the xPLSS. This system can perform regeneration or desorption by exposing the sorbent to a pressure swing from 4.3 psia to <1 torr over approximately 2 minutes. Using this test system, XploSafe evaluated various TCC prototypes constructed using rapid prototyping techniques and identified a process that allows vacuum regenerative TCC designs to be quickly examined for flow, pressure drop, and performance. By using toluene as a segregate for volatile organics, this test system was evaluated using both vacuum regenerable and non-regenerable sorbents to demonstrate the operational effectiveness of these sorbents in maintaining the concentrations of the trace contaminant analytes below the 7-day Spacecraft Maximum Allowable Concentrations.

Nomenclature

ABS	= acrylonitrile butadiene styrene
ACFM	= actual cubic feet per minute

[1] Professor of Chemistry, Department of Chemistry, 316 Physical Sciences I, Stillwater, OK 74078
[2] Research Scientist III, XploSafe, Stillwater, OK 74074
[3] Research Scientist I, XploSafe, Stillwater, OK 74074
[4] Operations Manager, XploSafe, Stillwater, OK 74074
[5] Product Development Specialist and Program Manager, XploSafe, Stillwater, OK 74074
[6] EVA SBIR Topic & PLSS SBIR/STTR Subtopic Manager, Spacesuit Knowledge Capture Manager EC5/Space Suit and Crew Survival Systems Branch Crew and Thermal Systems Division, Lyndon B. Johnson Space Center, NASA Parkway, Houston, TX 77058

Ammonasorb II = phosphoric acid (H_3PO_4)-treated activated carbon
CH_2O = formaldehyde
CH_3SH = methanethiol (also known as methyl mercaptan)
cm = centimeter
CO = carbon monoxide
FDM = fused deposition modeling
EVA = extravehicular activity
GC/MS = gas chromatography/mass spectrometry
ISS = International Space Station
kg = kilogram
L = liter
lpm = liters per minute
L/s = liters per second
mg = milligram
mL = milliliter
mm = millimeter
mTorr = millitorr
NASA = National Aeronautics and Space Administration
NH_3 = ammonia
OD = outer diameter
OSU-6 = XploSafe's mesoporous silica sorbent
PLA = Polylactic Acid
ppm = parts per million
RCA = Rapid Cycle Amine
RH = relative humidity
s = second
SMAC = Spacecraft Maximum Allowable Concentrations
SLA = Stereolithography 3D Printing
TCC = Trace Contaminant Control
TD = thermal desorption
VOCs = volatile organic compounds
xEMU = Exploration Extra-vehicular Mobility Unit
xPLSS = Exploration Portable Life Support System
μm = micrometer

I. Introduction

With NASA's sights set on lunar, Mars, and deep-space exploration, the Extra-Vehicular Mobility Unit demands innovative upgrades. The xEMU's heart, the Exploration Portable Life Support System, houses vital functions like oxygen regulation, CO_2 removal, and air filtration.[1] Within this system, the Trace Contaminant Control plays a crucial role, scrubbing the ventilation loop of trace contaminants generated by both suit materials and the astronauts themselves.[2] Left unchecked, these contaminants, including ammonia and VOCs, pose a significant health risk. As the next-generation xEMU takes shape, so too must the evolution of its sorbents. To rigorously evaluate the current non-regenerable solution as well as emerging regenerable materials under realistic conditions, this manuscript presents the ongoing development and refinement of a dedicated TCC test rig. This apparatus mimics the xPLSS environment, enabling the precise evaluation of not only next-generation sorbents for potential integration into the TCC system but also new regenerable TCC designs. Ultimately, this research paves the way for safer, more efficient space exploration by ensuring the astronauts well-being through robust contaminant control.

II. Background

The test rig must be able to simulate the physical conditions found in the TCC loop with conditions listed in the literature.[2] Additional specifications include functioning within the xEMU atmosphere that consists of oxygen concentrations up to 26.5% with the balance composed of nitrogen, metabolic products (CO_2 and H_2O), and trace gases. The test rig must be able to precisely inject these gases into the TCC to mimic the source rates from xEMU and its operator and reliably measure their concentrations. NASA has prioritized 18 volatile organic compounds for

removal by the TCC. The contaminant list developed for the TCC consists of 18 VOCs, for which the 7-day Spacecraft Maximum Allowable Concentrations are tabulated in Table 1.[3] Of these contaminants, ammonia (NH_3), carbon monoxide (CO), formaldehyde (CH_2O), and furan (C_4H_4O) are expected to exceed their corresponding 7-day SMAC limits.[4] Hostetler and co-authors list the emission rate of each contaminant that is generated within the xPLSS internally and biologically[2]. As per NASA's specifications, the TCC must be able to remove these compounds to ensure that they do not exceed the 7-day SMAC limits during an EVA.[4] Using these limits ensures that potential exposures will never exceed the 1-hour or 24-hour SMAC limits, even after multiple EVA missions. To meet this requirement, the currently used TCC system is within an accessible area of the xPLSS to allow for the replacement of the sorbent filter after 150 EVA hours. The more sustainable solution would be a vacuum-regenerable sorbent that could be integrated with the xPLSS CO_2/H_2O removal system, eliminating the cost and logistics of supplying replaceable sorbent filters to, for example, the International Space Station. Regeneration can be performed by exposing the sorbent to the vacuum of space, which is identical to the method performed by the Rapid Cycle Amine system currently integrated into the xPLSS.[5,6,7] Thus, a test system was designed to mimic not only the conditions faced by the current TCC system but also those potentially found in the swing bed area of the xPLSS, to allow new sorbent systems to be evaluated for vacuum regeneration. Combined with 3D printing, such a system also allows rapid evaluation of new TCC designs.

Table 1. Reported 7-day SMAC limits for 18 compounds specified by NASA for removal by the TCC.[2] The Source Rate is the projected generation with time of each compound within the xPLSS system.

Compound	SourceRate (mg/day)	SMAC Limit (ppm)
Acetaldehyde	0.663	2
Acetic acid	0.227	3.01
Acetone	0.193	22
Acrolein	0.006	0.015
Ammonia	80	3
1-Butanol	0.50	25
Carbon Monoxide	18	55
Ethanol	4.51	1000
Formaldehyde	0.42	0.1
Furan	0.3	0.025
Hexamethylcyclotrisiloxane	0.00396	10
Hydrogen	42.0	4100
Methane	329.	5300
Methanol	1.02	70
Methyl-Ethyl Ketone	0.907	10
Methyl mercaptan	N/A	0.2
Toluene	1.35	4
Trimethyl Silanol	0.2	1

III. Assembly and Testing

The development of the test bed was motivated by the need to both test TCC prototypes and quantify the sorbent performance within the TCC. The design of the test bed and initial testing procedure were presented elsewhere; therefore, only significant modifications of the setup will be discussed in this paper.[8] A block diagram of the TCC test bed design is shown in Figure 1, and a photograph of the test rig is shown in Figure 2 (Top). Briefly, there are four core subsystems in the testing apparatus. The main subsystem is the sub-atmospheric recirculation loop, including a tank, blower, and the TCC and bypass loops. A commercially available stainless steel compressor tank with a volume of approximately 20 L is used to simulate the xPLSS volume. A safety release valve, relative humidity sensor, and pressure gauge are connected to the tank with KF-25 fittings. The recirculating loop includes KF-25 fittings, rigid straight sections, flexible bellows, and right-angle couplers and tees that ensure that all connections are vacuum-tight while facilitating their removal and/or replacement for potential modifications. Gas is recirculated through the unit using a powerful Micronel blower (U100HL-024KA-4). The recirculation loop has two pathways controlled by three pneumatic valves. The bypass allows the assessment of any analyte loss by the recirculation subsystem that may

impact the subsequent assessments of the TCC performance. It also allows any unwanted contamination to be identified and eliminated before testing, facilitates the replacement of the TCC unit without breaking the recirculation loop, and supports the regeneration subsystem (described in detail in Section C). On startup, bypassing the TCC unit also allows the target analytes to be loaded at the desired concentrations before the introduction of the TCC sorbent into the flow. There are two piezoelectric pressure gauges that are connected directly across the TCC unit to monitor the pressure drop. Finally, there are three Alcatel 2012A dual-stage mechanical pumps to provide vacuum for initial evacuations, pressure regulation, and regeneration. The pressure regulation vacuum pump has a gas load equal to that of the flow through the injection valve (typically less than 300 mL/min). However, the regeneration pump has a significant load as it removes contaminants absorbs water from the TCC sorbent, and requires more frequent oil changes. This pump will eventually be replaced by a dry mechanical pump. No back streaming of oil was observed in the background mass spectrum.

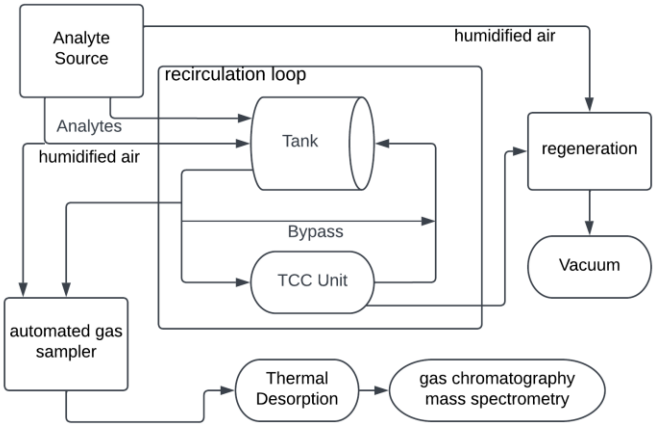

Figure 1. A block diagram of the TCC test bed designed to mimic the environment within the xPLSS. The system will be able to quantify the sorbent performance and test vacuum regeneration.

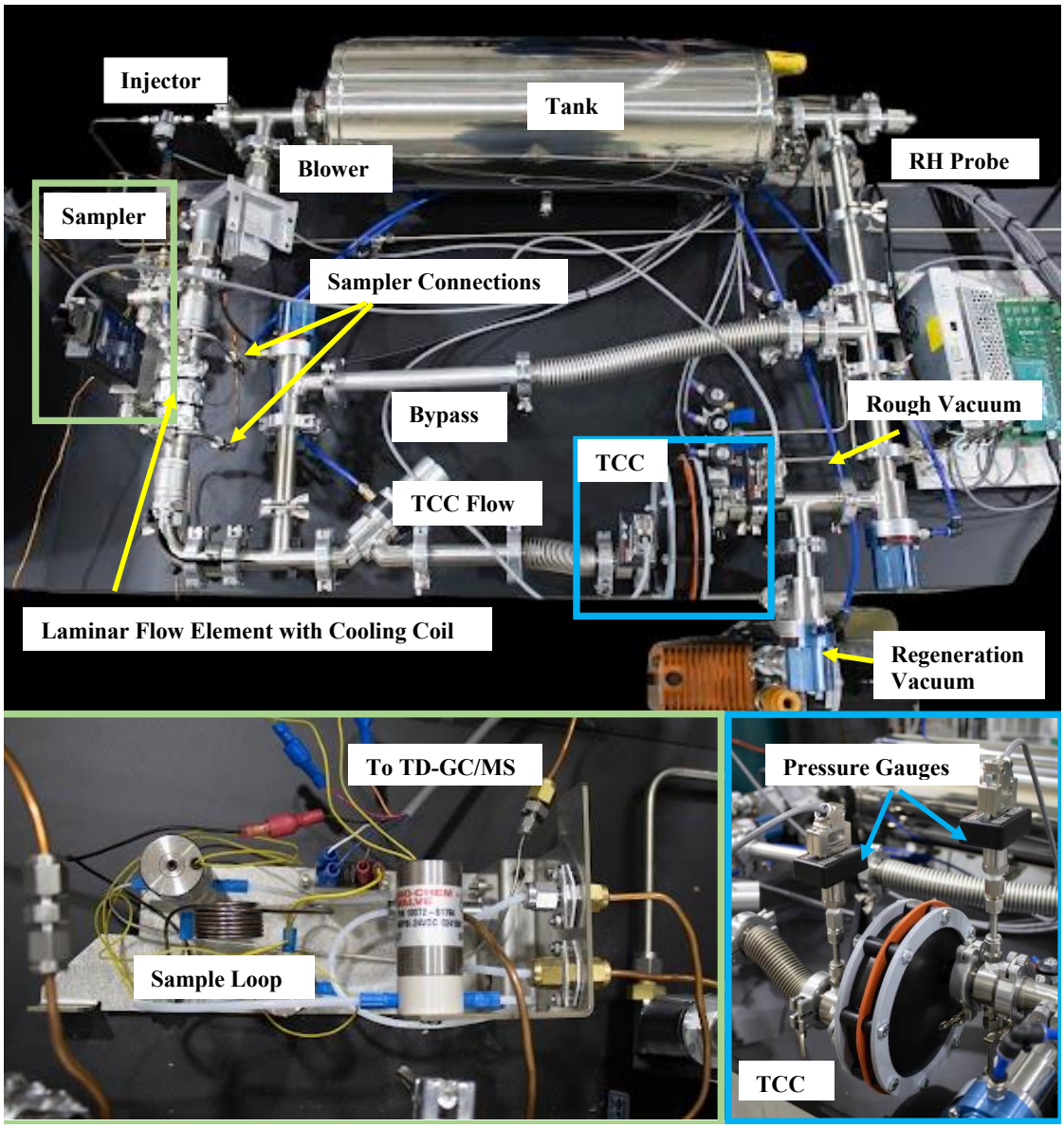

Figure 2. (Top) XploSafe's TCC test bed with a direct TCC flow loop and a bypass loop; (Bottom Left, Green) Automated gas sampler. (Bottom Right, Blue) TCC testing apparatus with a prototype TCC sorbent holder unit.

The second subsystem provides a source of humidified air to maintain a desired relative humidity level and pressure in the recirculation loop. It also includes a gas mixture generation system that can produce single or multi-component analyte vapor streams at known concentrations. Gas enters the system through a mass flow controller to control the injection rate. The third subsystem is the automated gas sampler (Figure 2, bottom left) that collects 10 mL samples from the recirculation loop to determine the gas phase concentrations of the analytes by TD-GC/MS. The TD instrument is a Markes TD-100 thermal desorption unit. The GC unit is an Agilent 6890N Network gas chromatograph connected to a turbo-pumped Agilent 5973N mass selective detector. The final component is a sub-atmospheric gas regeneration subsystem that facilitates the continuous pressure swing loop that is representative of the xPLSS. The apparatus is controlled by an Arduino microcontroller with a custom board that controls the blower speed and valves and reads the temperature, relative humidity, and pressure sensors. The microcontroller also supports the sampling subsystem and its interface to the TD and GC/MS units along with the regeneration subsystem. This microcontroller is connected to a computer with a graphical interface for higher-level monitoring, logging, and control.

A. Temperature and Pressure Control

Gas is recirculated through the unit using a Micronel blower (U100HL-024KA-4). An in-line high-flow low-pressure drop flowmeter (HFM-200 LFE) was added after the pump to directly measure the flow during operation. During testing, an external anemometer was used to measure the gas velocity that was subsequently converted into a volume flow using the cross-sectional area. The two values agreed within approximately 5% for flows between 3−8 ACFM, demonstrating the accurate performance of the in-line flow meter. The flowmeter proved to be important as changes in the flow path, induced by switching from the TCC to the bypass pathway, increase the flow due to the elimination of the TCC hardware from the pathway. The blower speed must be adjusted by the microcontroller to maintain a constant flow when switching between these two paths. In addition, different TCC geometries require different blower settings.

An unforeseen side effect of the blower running at 6 CFM was the inadvertent heating of the circulating gas and the temperature rising 5 ℃ within a half-hour of running. The laminar flow element from the in-line flow meter was utilized to remove thermal energy after the circulating gas exited from the blower. The laminar flow element consists of many small channels or tubes bunched together within the element. This element was cooled using a short coil of copper tubing wrapped around its exterior. An external chiller was used to flow a temperature-regulated water/ethylene glycol mixture through the coil. This setup was sufficient to cool the gas to 25 ℃. No condensation on the outlet of the element after completing a long experimental run was observed.

In the current design, a mass flow controller permits a known flow of the analyte gas to enter the loop to simulate a given source rate. A pressure release system maintains the internal gas pressure using two regulators. A sub-atmospheric back pressure gas regulator is connected to a vacuum pump and another standard regulator is connected to a contamination-free air source. At the junction of the two regulators, the pressure can be actively maintained in the range of 1−800 Torr. When starting with a dry system, the relative humidity will increase slowly until it equals the water concentration in the source gas. Figure 3 shows the temperature stability and relative humidity equilibrium achieved for a water vapor source. It can be modeled by assuming the input flow is equal to the outlet flow (Eq. (1)).

$$Tank\ Concentration\ =\ Injected\ Concentration \times \left(1 - e^{\frac{Inlet\ flow}{Tank\ Volume}}\right) \qquad (1)$$

Increasing the relative humidity in the model (equation) results in a bypass volume of 21 L, which is consistent with the experimental setup. Similarly fitting the flow through the TCC gives a larger volume, again consistent with the additional volume of the TCC prototype. The extended run shown in Figure 3 indicates that the system can maintain temperature and humidity stability for hours. Additional studies have shown that it is stable for time scales measured in weeks.

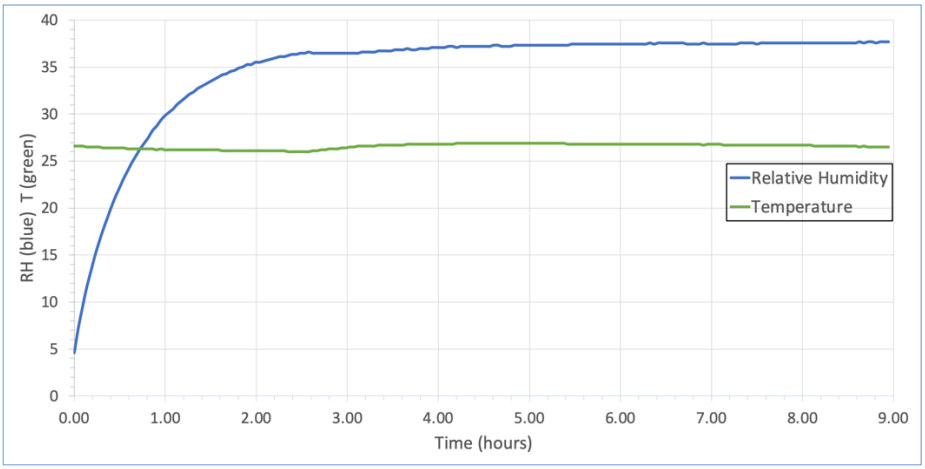

Figure 3. Temperature stability and relative humidity equilibrium achieved for a water vapor source. The system can maintain temperature and humidity stability for days to weeks.

B. TCC Prototype Unit Design and Evaluation

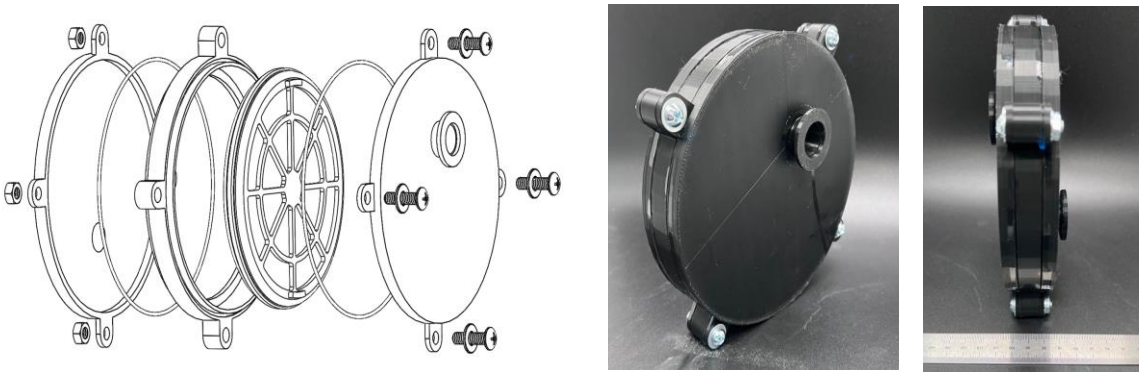

Figure 4. (Left) CAD model of the initial design components. (Middle) Assembled 3D printed prototype of the third prototype design. (Right) A side view of the third prototype design (thickness: 2 inches).

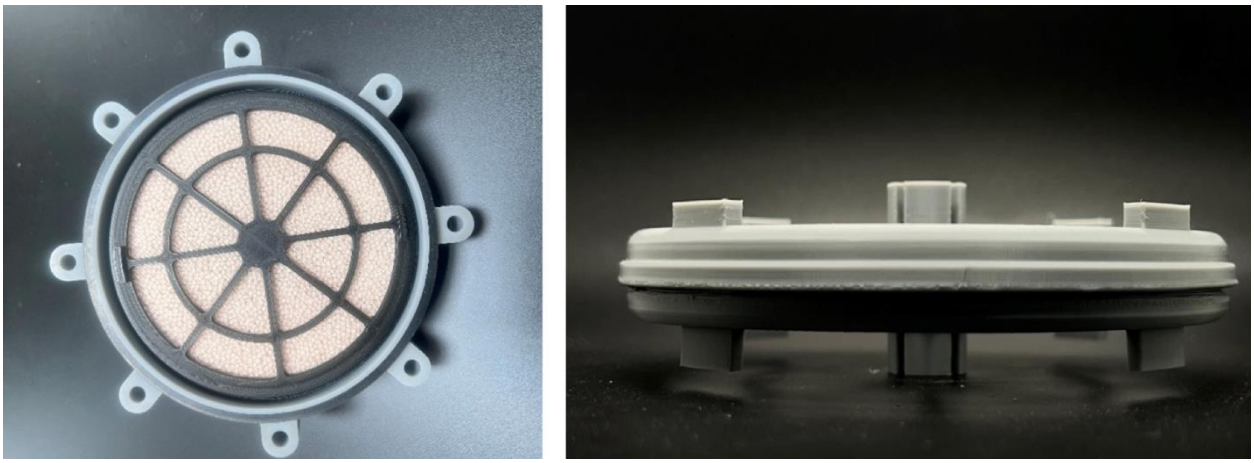

Figure 5. Inner sorbent holder of the TCC prototype: (Left) top view inside of an outer shell and (Right) side view without an outer shell.

XploSafe developed a series of prototypes for the TCC to be tested on the test bed. Rapid testing combined with 3D printing of prototypes allows one to test different configurations for the sorbent and study the flow within the TCC without costly machining. As discussed below, designs were considered with symmetric and offset arrangement gas ports. For regeneration systems, prototypes were tested to optimize the material used and the mechanical strength, with future options for modeling the evacuation process. During the model evaluation, different structures within the TCC housing were explored to provide a more uniform flow. When considering possible TCC prototypes, it was decided that the internal sorbent holder should match the current TCC design (Figure 5). The prototype shown in Figure 4 was 3D printed using polylactic acid (PLA) filaments and incorporates a set of rubber O-ring gaskets to help seal the covers along with four screws to clamp the three pieces together.

Flow simulations conducted for this prototype with ¼" and ¾" inputs (Figure 6) showed that most of the pressure drop occurs at the input to the TCC unit. The simulations also examined the effect of offsetting the inlet and outlet ports with respect to a symmetric arrangement. In the symmetric arrangement, it was found that most of the flow was through the center of the filter with the edges hardly having any flow at all. This arrangement would severely reduce the total capacity of the TCC. These calculations also provide insights into the current complex design of the TCC with the offset flanges. This observation is consistent with the current TCC 360 design that flows the gas stream at a 90° angle.

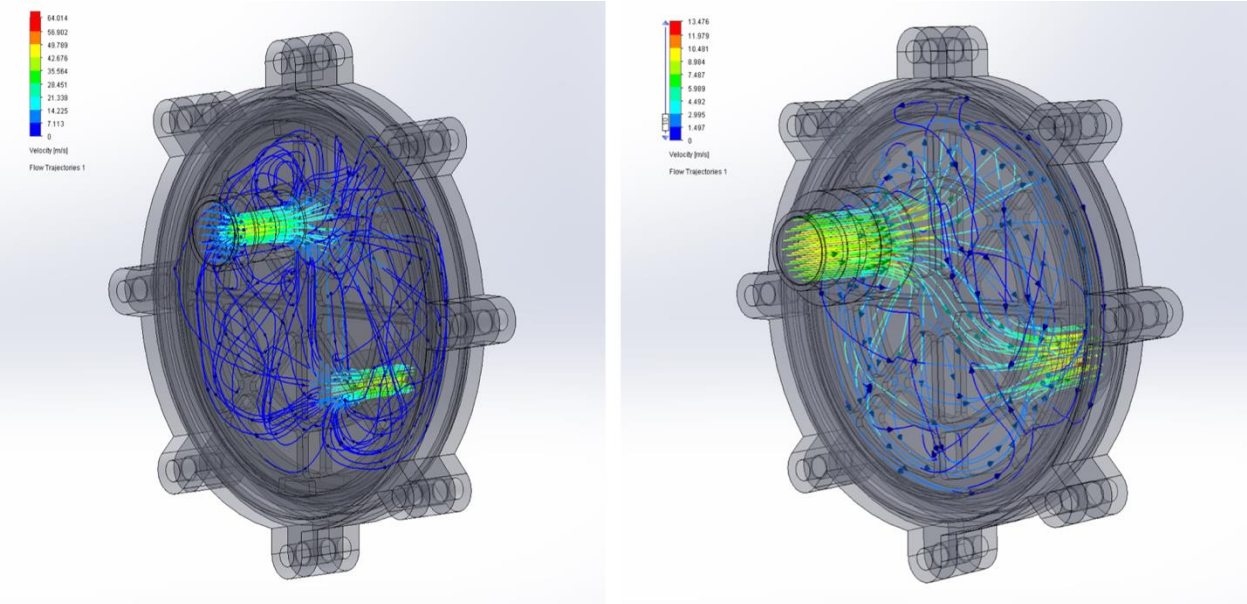

Figure 6. Flow simulations of ¼" inlets and outlets (left) versus ¾" inlets and outlets (right) on the filter. Note: dark blue airflow is closer to 0 m/s whereas green/yellow airflow is closer to inlet air velocity.

In the next prototype version, all parts were 3D printed using thermal deposition from a filament source. It was found that these prototypes leaked through voids in the layers of plastic material even at a 430 Torr pressure. The filament choice and the printing density could not resolve this issue. Stereolithography 3D printing (SLA) has been shown to be able to create vacuum and pressure-tight polymer vessels.[9] As a result, the subsequent prototypes were manufactured via 3D SLA printing. The first SLA printed prototype (see discussion and Figure 9 in the next section) had significant mechanical issues but was found to be leak-free. In further refinement, internal structural elements were added to the filter holder to help support the TCC sorbent holder body, prevent sudden cracking, and reduce bending/deformation during vacuum cycle testing. In addition, the O-rings were replaced with a silicone gasket, and the adjacent surfaces were optimized to account for the flexing of the material and imperfections. Flanged collars were also added to alleviate pressure on the bolts, helping to prevent sudden cracking of prototypes and reduce bending/deformation during vacuum cycle testing.

Figure 7 shows the final design with 3D-printed KF-25 connections. During testing, it was noted that the regeneration pressure desired (~100 mTorr) could not be achieved with several of the plastic materials used in 3D printing, predominantly due to the nature of fused deposition modeling-based printing. The voids introduced by filament printing contributed to outgassing as they trapped foreign contaminants within the print. This outgassing and leaking could be reduced by modifying the printer settings to reduce the imperfections in the printed components. Combining ABS with FDM and ABS-like resin with SLA allowed the prototype to reach the ultimate sustainable vacuum at ~100 mTorr. To increase the overall strength during the final prototypes, centering rings were changed from PLA to an ABS filament with 100% printing infill (Figures 8a and b). These prototypes passed all testing criteria in the test rig. Figure 8b shows a nylon PA-12 prototype that was externally manufactured. In this version, the main housing and interior filter cartridge were 3D printed from nylon PA-12 with two additional finishing steps. Both prototypes depicted in Figure 8 were capable of holding multiple cycles overnight with less outgassing than the prototype displayed in Figure 8a.

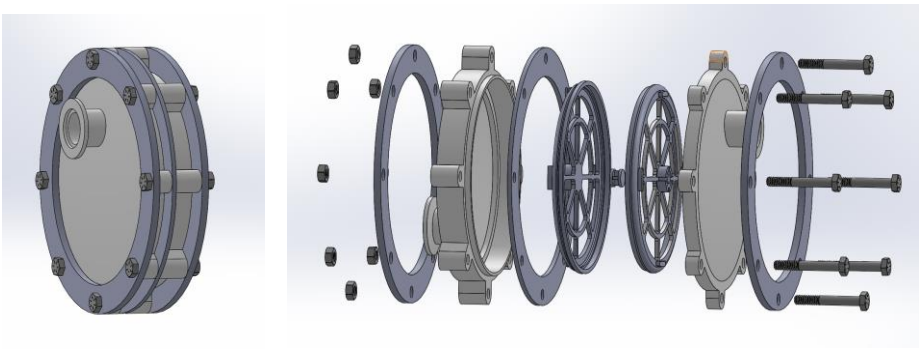

Figure 7. Final sorbent filter design with 3D printed KF-25 connections.

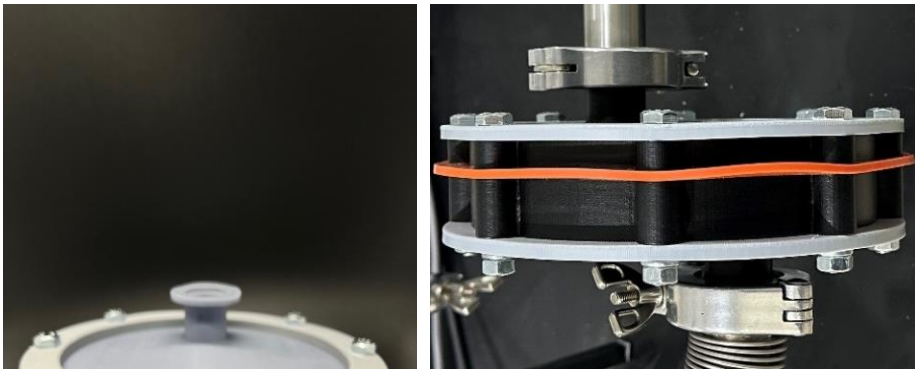

Figure 8. Photographs of the final prototypes with (left) ABS-like resin housing and ABS centering rings, and (right) nylon (PA-12) housing and ABS centering rings.

C. Vacuum Regeneration Testing

When the test rig is in the bypass mode, the TCC prototype is isolated between the two KF-25 valves shown in Figure 2. When isolated, additional solenoid valves can be used for vacuum regeneration and pressurization. A regulated 1-Torr vacuum is provided using a setup similar to that employed for regulating the pressure in the recirculation loop. A sub-atmospheric back pressure gas regulator is connected to a vacuum pump combined with another standard regulator, which is attached to a contamination-free air source. This setup maintains a stable pressure between the regulators of 1.00 ± 0.01 Torr. The pressure was consistent with the prior NASA vacuum testing for regeneration of the RCA system.[7]

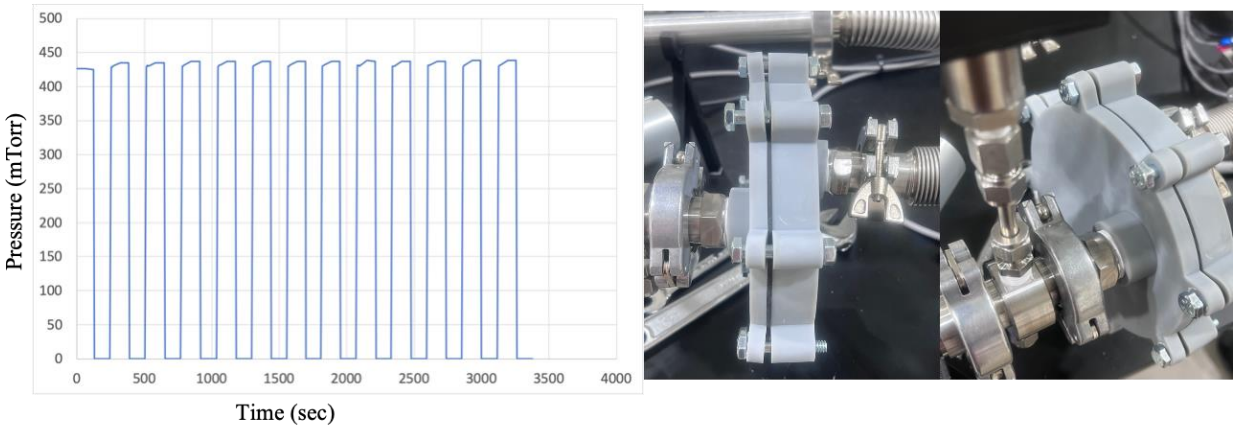

Figure 9. (Left) Measured pressure swings during cycling and (Right) physical deformation on an early prototype while under vacuum (SLA print).

Extensive testing of the TCC units required evacuations and re-pressurization. As discussed below, leakage, mechanical stability, and outgassing of the 3D printed materials represented a significant challenge. In addition, once loaded with sorbent, the conductance of the evacuation line becomes an issue. Hence, an additional two-part pumping mechanism has been designed. It consists of an initial connection to the pump through a small line (labeled "Rough Vacuum" in Figure 2), which can take the pressure down to a few Torr within seconds. Once the pressure is reduced, a larger 1-inch line is opened to pump the system down to 100 mTorr or lower. The two-stage pumping systems also allow verification of the initial pump down, preventing damage to the vacuum system in case of fractures in the TCC unit. Thus, once the TCC unit is isolated, the pressure difference between the inlet and outlet can be reduced to 100 Torr within a few seconds. After a pre-set time (i.e. 2 minutes), the vacuum is shut off, and the re-pressurization valve opens, to quickly repressurize the unit to the loop pressure, using the recirculation loop-regulated pressure source described above. Afterward, the TCC loop is reconnected to the recirculation loop until the next cycle starts. This is illustrated graphically by the recorded pressure swings in Figure 9 (left panel). The mechanical deformation on an early prototype can easily be seen in the right panel of Figure 9.

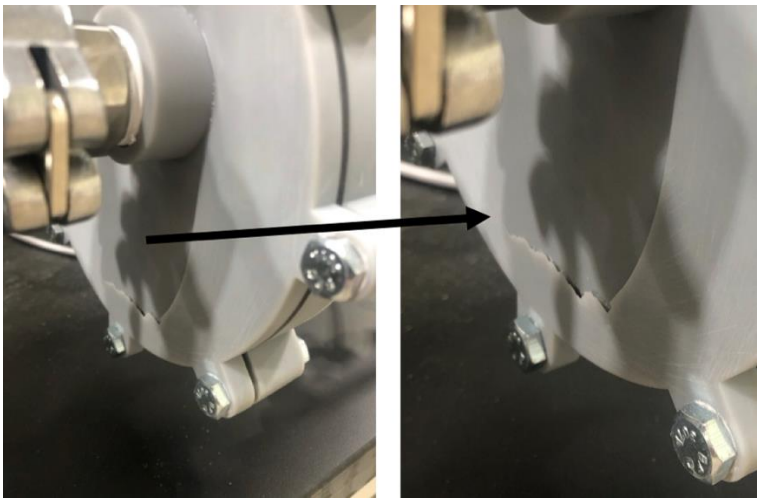

Figure 10. Fracture of the TCC prototype causing rapid and audible pressurization.

Safety concerns are paramount for long-term testing where operators can become complacent. For the current test rig, using the initial pump down at the start of the vacuum to monitor for leaks or fractures in the TCC unit and to stop the test without damaging the system or the operator is an important safety feature. The current design utilizes a plexiglass chemical blast shield to safeguard the operator in case of an implosion. Figure 10 shows a more dramatic prototype failure (other failures have occurred at the gasket seal and the KF connectors). As discussed above, changes in the design and materials were required to achieve reliable vacuum cycling for the 3D-printed parts. The final TCC prototypes have been tested successfully for over 100 cycles; the mechanical deformation of the TCC body was almost eliminated using internal structures. The final SLA printed prototype with an ABS-like resin reached the ultimate sustainable vacuum at ~100 mTorr and could withstand 100s of cycles.

D. TCC Testing Using the Analyte Source Subsystem

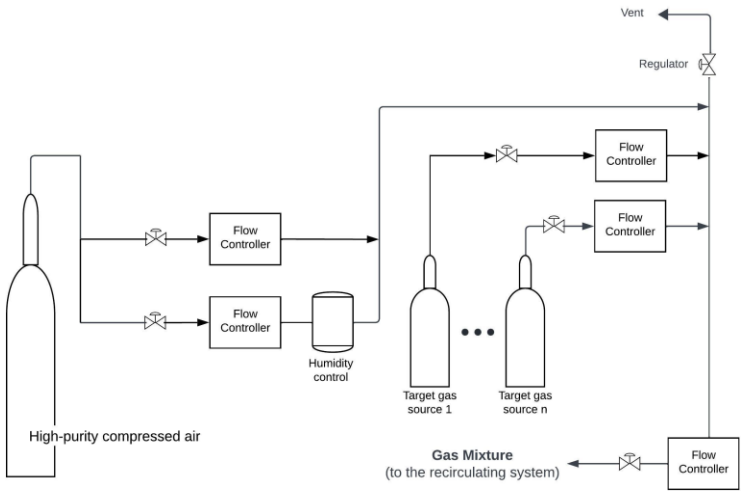

Figure 11. Schematic of the Analyte Source Subsystem showing the generation of the regulated humidified air at the correct pressure (200−760 Torr) and the gas mixture source.

The Analyte Source Subsystem (Figure 11) was designed to create gas mixtures of select NASA analytes and to supply the mixture to the main flow controller (at the TCC flow rate) on the TCC test rig. Each gas mixture component was introduced to the main flow controller from a pre-calibrated gas cylinder through a separate mass flow controller with a flow rate range of 0–20 mL/min. A humidified carrier gas was used to adjust the relative humidity. In the humidity control section, a water-saturated gas source was generated in a commercial gas washing bottle with a coarse frit to disburse the gas (Chem Glass CG-1114-14). The RH stream is then diluted to obtain the desired relative humidity in the final gas mixture. The relative humidity was validated by a sensor (OMEGA RH-USB environmental test probe) attached to the output. In addition, the relative humidity and temperature in the recirculation loop are continually monitored. In general, the total flow rate required to generate the mixtures is greater than the flow into the TCC loop. Thus, a back regulator was used to ensure that the pressure at the inlet of the main flow controller remained constant. The flow rate for each analyte into the mixer was computed based on the NASA source rate (Table 1), tank volume, gas cylinder concentration, previously measured sorbent capacity, and tank flow rate. Currently, the TCC must limit the concentrations of the trace contaminants inside the TCC loop under the 7-day SMAC limits established by NASA for at least 150 hours or 25 EVAs.

The source rates for selected contaminants are reported in Table 1. In the test rig, the source rates were simulated by using a mass flow controller to inject a known concentration of each gas at a known flow rate into the TCC loop. An injection rate of less than 500 mL/min was selected to not significantly impact the recirculation flow of 6 ACFM (170 lpm). This injection rate results in a slow rise in the concentration of the analyte with time when the filter unit is not installed. The simulated source rate is equal to the injection rate times the gas concentration in ng/mL (Eq. (2)).

$$Source\ rate\ \left(\frac{\mathrm{mg}}{\mathrm{day}}\right) = Injection\ rate\ \left(\frac{mL}{min}\right) \times Gas\ concentration\ \left(\frac{ng}{mL}\right) \times \frac{mg}{10^6 ng} \times \frac{24\ h}{day} \times \frac{60\ min}{h} \qquad (2)$$

Once the injection rate is set, the correct concentrations in ng/mL of each supplied gases can be selected by using flow controllers to mix the outputs of various calibrated gas cylinders with a balance of humidified air to control the relative humidity. The concentration in ppm at 25 °C, which enters the calculation *via* the molecular volume, can be calculated as shown in Eq. (3).

$$Gas\ concentration\ in\ \frac{ng}{mL} = Gas\ concentration\ (ppm) \times \frac{Molecular\ weight\ \left(\frac{g}{mol}\right)}{Molar\ volume\ \left(\frac{L}{mol}\right)} \qquad (3)$$

The total flow of the calibrated gases and balance air can exceed the injection flow. The concentration of each gas in the pictures can be computed by using the total flow into the mixing tube, which includes the balance air and the flow of all other gases, and the cylinder ppm (Eq. (4)).

$$Gas\ concentration\ (ppm) = Gas\ \text{cylinder (ppm)} \times \frac{Gas\ flow}{Total\ flow} \quad (4)$$

The relative humidity in the flow through balanced air post mixing in the stream is equal to the setpoint (i.e. 40%). A pressure regulator is installed before the injection mass flow controller to prevent over-pressurization (see Figure 11). Using these equations, Table 2 summarizes the flow rates for various gases required to match NASA source rates listed in Table 1. This calculation assumes a tank volume of 20 L, a temperature of 25 ℃, a relative humidity of 40%, a TCC injection flow rate of 200 mL/min, and pre-calibrated gas cylinders at the given concentrations. The gases are mixed with a humidified air stream at a flow of 250 mL/min to set the relative humidity of the mixture. Note that the total flow into the mixing chamber is greater than the gas flow rate, requiring a backpressure regulator to controllably release the excess gas.

The sorbent capacities[10] can be used to estimate the time required to reach the 80% capacity (Eq. (5)), the point where the initial breakthrough is experimentally observed. The total mass of the contaminant is equal to the injection rate times time. Capacity times 80% was utilized as the breakthrough in small-scale studies with the same bed depth of the filter occurs at this point.[10] The amounts of Ammonasorb II sorbent powder (115 g, 40/60 mesh) and OSU-6 beads (35 g) were selected to match the quaintly contained in the inner sorbent holder (see Figure 5) of the prototype TCC unit.

$$Time\ (h) = \frac{(0.80) \times sorbent\ mass\ (g) \times sorbent\ \ capacity\ \left(\frac{mg}{g}\right)}{Injection\ rate\ \left(\frac{ng}{min}\right)} \times \left(\frac{h}{60\ min}\right) \times 10^6 \quad (5)$$

Table 2. Calculated flow rates of select TCC analytes required to match the NASA source rates listed in Table 1. Calculated time is that required to reach 80% of the contaminate capacities for the amounts of Ammonasorb II sorbent and OSU-6 beads within the inner sorbent holder (see Figure 5). See text for details.

Contaminant	Cylinder Concentration (ppm)	Cylinder Flow Rate (mL/min)	Time to Reach 80% Sorbent Capacity (h)	
			OSU-6	Ammonasorb II
Acetone	50	1.4	6963	5262
Acetaldehyde	100	3.2	41	93
Ammonia	10000	10.0	12	442
1-Butanol	50	2.9	12096	39743
Ethanol	500	4.2	1073	5679
Formaldehyde	50	5.9	7	14
Furan	50	1.9	605	1987
Methanol	500	1.4	659	5628
Methyl mercaptan	50	7.3	0.32	2527
Toluene	200	10.0	743	17196

The extremely long times required for the NASA source rates pose a significant barrier to experimentation for many contaminants. Although ammonia is an important TCC contaminant, volatile organic contaminants are an internal focus. Initial experiments with a gas mixture containing methanol, ethanol, and toluene as well as a mixture with acrolein, methyl ethyl ketone, furan, and acetaldehyde were performed to test the performance of the analysis system. The difficulty of dosing gas mixtures along with the long experimental timescale shifted our effort to a single source that was readily procured at different concentrations over several orders of magnitude. Thus, to verify that the test apparatus is performing as expected, the experiments described here were performed using toluene as the target gas. Toluene is a stable gas that has low toxicity and is easily detected via TD-GC/MS.

Given the long time required to reach a breakthrough, this study primally used a 120-ppm toluene gas source injected at a flow rate of 300 mL/min into the TCC loop at ambient pressure. This injection rate corresponds to a source rate of 190 mg/day. Figure 12 (left panel) shows the sorbent holder filled with 35 g of beads made from XploSafe's mesoporous silica sorbent (OSU-6). For OSU-6 powder (particle sizes of 0.25 to 0.425 mm), a smaller sorbent holder shown in Figure 12 (right panel) was developed to be able to test the apparatus in a reasonable time. This new holder has the same bed depth but holds significantly less material. For this system, the calculated time to reach capacity was 21 hours. In this experiment, the system was allowed to equilibrate with respect to temperature at

a relative humidity of 40%. After which, toluene was injected into the TCC loop. The toluene vapor concentrations were measured via TD–GC/MS as described in a previous work.[10]

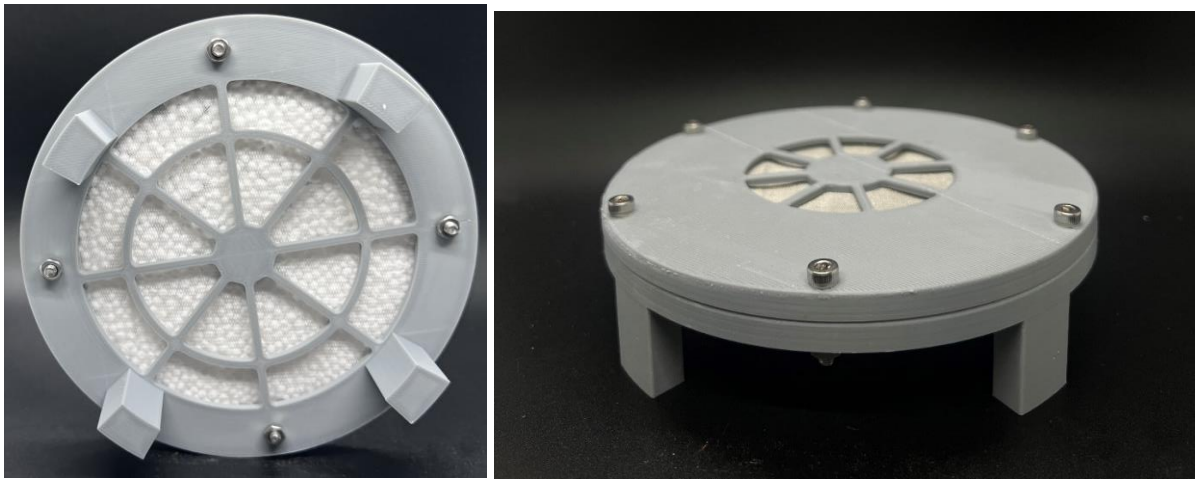

Figure 12. (Left) An ABS sorbent cartridge filled with OSU-6 beads. (Right) A reduced size ABS sorbent cartridge filled with OSU-6 power.

The concentration of toluene in the TCC loop system measured without and with the OSU-6 sorbent bed is shown in Figure 13. The fit was based on a simple model (Eq. (1)) that considers the input rate and system volume. The time required to reach 120 ppm without a sorbent was approximately 5 hours, consistent with the volume of the TCC loop. With the OSU-6 sorbent, the time to reach 120 ppm increased to 21 hours and matched the predicted value. The breakthrough is due to the toluene gas flowing over the beads and not through them as a filter, due to the large particle size (2.5 to 4 mm) and small bed depth (9 mm). To optimize the filter performance, the resonance time must be increased either by increasing the bed depth or decreasing the particle size (both options will impact the pressure drop across the TCC). The current beads result in a pressure drop that is well under the allowed pressure drop specified by NASA (0.3 in-H_2O of pressure drop at 170 lpm and 29.7 kPa).[2] In the larger sorbent cartridge (Figure 12, left), the 40/60 sieved OSU-6 also meets the pressure drop requirements. Reference 3 has a discussion of the bed diameter, depth, and particle sizing in the pressure drop. Using these results, bead sizes can be easily reduced to 1 mm or less.

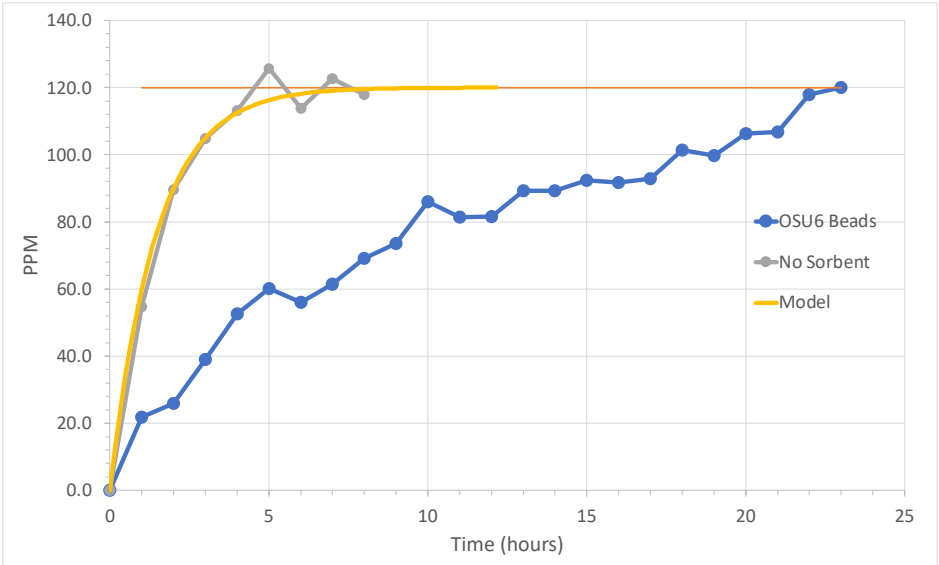

Figure 13. Toluene concentration in the TCC loop measured by TD-GC/MS without a sorbent, with a model fit based on the source rate of 190 mg/day and system volume. The blue line is the toluene concentration in the TCC loop with OSU-6 beads.

The smaller sorbent cartridge can contain between ~1−2 g of 40/60 sieved OSU-6 due to its low density and smaller overall bed volume. At a 20-ppm injection rate (source rate of 13.6 mg/day), the initial breakthrough of toluene (~2 ppm) for 1.9 g of OSU-6 is achieved in 142 hours. A similar experiment with 24.2 g of 40/60 sieved Ammonasorb II, which is significantly denser than OSU-6 and was packed tighter, had an initial breakthrough time (~2 ppm) of 135 hours at an injection rate of 190 mg/day. These values are consistent with the breakthrough times calculated using the previously measured breakthroughs in a flow experiment with only 50 mg of sorbent in a 4 mm diameter column.[10]

With regeneration, the contaminant is also lost after each cycle due to the evacuation. Assuming the TCC unit is repressurized from an external contaminate-free source, the tank concentration without the sorbent can be described by Eq. (6).

$$Tank\ Concentration\ =\ Injected\ Concentration \times \left(\frac{Inlet\ flow - Regen\ Loss}{Inlet\ flow}\right) \times \left(1 - e^{\frac{Inlet\ flow - Regen\ Loss}{Tank\ Volume}}\right) \quad (6)$$

where the regeneration loss is the volume lost in each regeneration cycle. Thus, unlike the non-regeneration case, the maximum concentration is less than the concentration of the inlet gas. Using a 4-min cycle (2 min of the TCC flow and 2 min of vacuum) and an injection rate of 190 mg/day (120-ppm gas mixture injected at 300 mL/min), the final tank concertation was measured without a sorbent and modeled using Eq. (6). The regeneration stability was tested over 200 cycles. After which, 2.4 g of OSU-6 was added, and the process was repeated. After several hundred cycles, the steady-state concentration was reduced by 74 % due to the capture and regeneration of the sorbent. Figure 14 shows the concentration with and without the sorbent with time. The results are still being evaluated and changes, including reducing the free volume in the TCC and optimizing the evacuation flow are being considered. Changes in the design to minimize mechanical stress on the sorbent during repeated vacuum cycles are also a concern that now can be investigated.

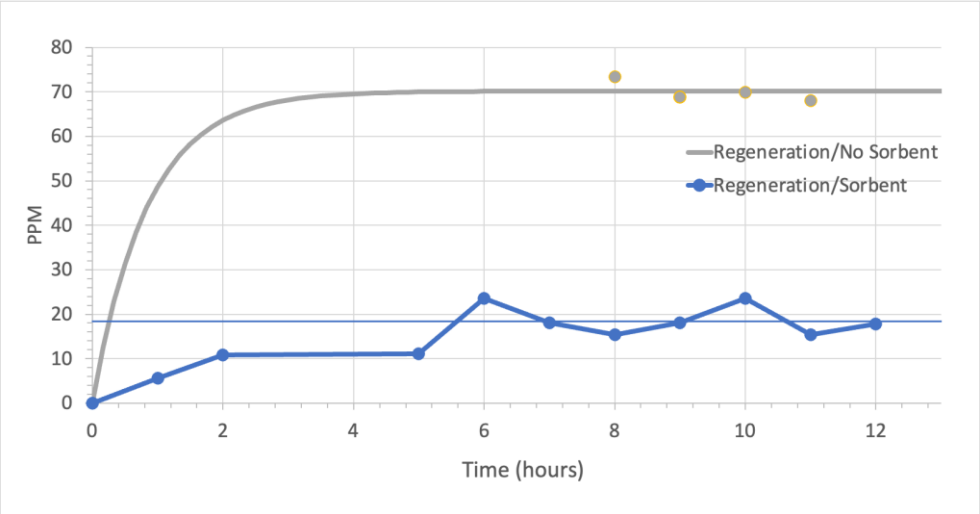

Figure 14. Toluene concentration in the TCC loop measured by TD-GC/MS with and without sorbent. The "No-Sorbent" line is computed from Eq. (6), with the dots representing the measured values. The difference between these two curves with time is the steady-state removal of toluene by the sorbent.

Conclusion

A test bed for the evaluation of different TCC designs and sorbents within the TCC module of the xPLSS was designed, assembled, and evaluated. This system operates at sub-atmospheric pressures and can evaluate the performance of a prototype TCC using known source rates of individual or mixtures of trace analytes at the specified operating conditions. In this update, the temperature, relative humidity stability, and vacuum regeneration capacities of the test bed are described. The mechanical stability and outgassing performance of various 3D-printed TCC prototype filters, holder designs, and materials during cycling between sub-atmospheric and vacuum regeneration conditions were assessed. A TCC filter unit fabricated from an ABS-like resin can be 3D-printed using SLA and is

stable under continual cycling, which exposes the prototype to both vacuum and mechanical stress. The use of an appropriate material along with the carefully placed internal supports and compression rings allows rapid experimental testing of low-cost, lightweight, 3D-printed parts with different internal designs for the optimal removal of contaminants and sorbent regeneration. The challenges of matching the source rate with the time required for reaching a breakthrough were discussed, and two different sorbent holders were designed to allow breakthrough times to occur within 24 hours for our initial testing. Initial experimental results revealed that the previously measured breakthroughs using 50 mg of sorbent in a 4-mm diameter column were accurate and valid when scaled. Finally, the system was successfully tested using a toluene source and OSU-6 sorbent composed of either beads or powder.

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