

Regenerable Trace Contaminant Control for Advanced Portable Life Support System

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The Trace Contaminant Control (TCC) System is a component in the Exploration Portable Life Support System (xPLSS) which removes contaminants present in the ventilation loop. These trace contaminants, introduced into the ventilation loop via crew metabolic processes, off-gassing of spacesuit materials, and by-products of the suit processes, such as the CO₂/H₂O removal system (e.g., Rapid Cycle Amine beds), would accumulate without the TCC and pose a threat to the crewmember. Trace contaminants are traditionally removed using non-regenerable activated carbon. While effective, the downside of the current state-of-the-art is a high associated life cycle operating cost resulting from a low regeneration capability, a large canister size, and significant power consumption during regeneration. This provides a logistics impact for future missions.

Precision Combustion, Inc. (PCI) has continued its development of a compact, vacuum-regenerable sorbent bed for effectively removing a broad range of trace contaminants, including ammonia, meeting NASA's target performance requirements, which can be integrated with the xPLSS CO₂/H₂O removal system. Both the primary trace contaminants as well as other species that threaten to exceed the 7-day Spacecraft Maximum Allowable Concentration (SMAC) levels are addressed. PCI's proven sorbent nanomaterials have high surface area on a structured support, enabling a compact, modular, and vacuum-regenerable TCC device. Current development efforts have focused on design optimization to reduce the pressure drop of the TCC canisters and extend their protection periods for the contaminants of concern. In this paper, performance data will be presented for the second generation TCC hardware prototypes integrated with a CO₂/H₂O removal system in a closed-loop ventilation test rig. Additionally, results from sorbent testing with multiple trace contaminants under PLSS operating conditions will be presented, including capacity, regenerability, and multi-cycle performance. The performance of an integrated, vacuum-regenerable TCC bed for multi-contaminant adsorption will be presented along with future maturation steps.

Nomenclature

°C	=	degree Celsius
°F	=	degree Fahrenheit
AC	=	activated carbon
ACFM	=	actual cubic foot per minute
BTU	=	British thermal unit
CFD	=	Computational Fluid Dynamics
CH ₂ O	=	Formaldehyde
CH ₄ S	=	Methyl mercaptan; i.e., methanethiol
EMU	=	Extravehicular Mobility Unit
EVA	=	Extravehicular Activity

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<i>g</i>	=	gram
<i>GSA</i>	=	geometric surface area
<i>hr</i>	=	hour
<i>in</i>	=	inch
<i>kPa</i>	=	kilopascals, absolute
<i>m</i>	=	meter
<i>mg</i>	=	milligram
<i>min</i>	=	minute
<i>mL</i>	=	milliliter
<i>mm Hg</i>	=	mm of mercury
<i>NASA</i>	=	National Aeronautics and Space Administration
<i>NH₃</i>	=	Ammonia
<i>PCI</i>	=	Precision Combustion, Inc.
<i>ppm_v</i>	=	parts per million by volume
<i>psia</i>	=	pound per square inch, absolute
<i>RCA</i>	=	Rapid Cycle Amine
<i>RH</i>	=	relative humidity
<i>slpm</i>	=	standard liters per minute (at 14.7 psia and 21°C)
<i>SMAC</i>	=	Spacecraft Maximum Allowable Concentration
<i>TC</i>	=	trace contaminant
<i>TCC</i>	=	Trace Contaminant Control
<i>TRL</i>	=	Technology Readiness Level
<i>wt.%</i>	=	percent by weight
<i>xPLSS</i>	=	Exploration Portable Life Support System

I. Introduction

COMPACT, low pressure drop, vacuum-regenerable Trace Contaminant Control (TCC) systems would be of great utility to NASA for their Exploration Portable Life Support System (xPLSS) applications. A broad range of trace contaminants (TCs) tend to build up in the ventilation loop of a spacesuit due to crew metabolic processes, off-gassing of spacesuit materials, and by-products of the processes contained within the suit. For example, the Rapid Cycle Amine (RCA) swing bed used for CO₂/H₂O removal in the xPLSS off-gases NH₃ from its amine sorbents which must be removed to ensure the astronaut's health and comfort. TCs are traditionally removed from the spacesuit ventilation loop via an activated carbon (AC) bed. While AC is an effective solution, it has a high associated life cycle operating cost due to its limited regeneration capability which necessitates larger cartridges, a challenge for the space restricted xPLSS, and replacement cartridges which are expensive to launch and are not feasible for long duration space missions. A vacuum regenerable TCC sorbent system would overcome many of these shortcomings. According to a review paper recently published by NASA, the minimum objective for TCC would be a vacuum-regenerable unit capable to remove all of the significant compounds that threaten to exceed the 7-day Spacecraft Maximum Allowable Concentration (SMAC) during Extravehicular Activity (EVA) and for Extravehicular Mobility Unit (EMU) applications with the optimal objective to also enable removal of less significant compounds.¹ Rapid vacuum regenerability permits low power, real-time, on-the-suit TCC bed regeneration and significantly reduces the size of the sorbent bed. To simplify the xPLSS operation, a TCC which operates with the same half cycle times as the RCA unit (i.e., 1-to-3 minutes nominally, up to 15-min during low metabolic rate periods) would be preferred. This would permit simultaneous regeneration of the new TCC hardware with NASA's RCA module and eliminate the need for a second vacuum switching valve assembly. Additional requirements necessitate minimizing pressure drop across the TCC unit (i.e., sorbent bed and housing) to reduce power consumption by the xPLSS fan, to a target of 0.3 in H₂O across the sorbent bed. Based on the TC generation rates provided by NASA and the air exchange rate within the xPLSS corresponding to an air flow rate of 6 actual cubic feet per minute (ACFM) at 4.3 psia (29.6 kPa) and 60°F (15.6°C),² the concentration of each contaminant of interest can be determined and compared to the 7-day SMAC limit.³ The concentrations for three TCs most likely to exceed their 7-day SMAC levels without a TCC device are summarized in Table 1.

Several different sorbent materials are currently under development for TCC applications by other groups. For example, NUCON International, Inc. has developed impregnated carbon treated absorbents pellets for the removal of low to high concentrations of NH₃, with an adsorption capacity in the range of 5 – 8 wt.% over the range of 0 – 95%

Table 1. Selection of contaminants in the xPLSS ventilation loop that are expected to exceed their 7-day SMAC limits without TCC and are therefore considered as the design drivers for the TCC bed development effort. ^{2,3}

Trace Contaminant	Total Generation Rate (mg/day)	7-day SMAC limit (ppm _v)	Concentration with no TCC (ppm _v)
Ammonia	80.	3.0	770
Formaldehyde	0.42	0.10	2.3
Methyl Mercaptan	0.83	0.50	2.8

relative humidity (RH).⁴ However, a guard bed or increased residence time is recommended to protect the sorbents from other contaminants. Moreover, the spent adsorbent must be thermally regenerated which adds operating cost in the form of power

consumption and necessitates a larger bed to account for the lack of real-time vacuum regeneration capability. Molecular sieves and zeolites have also been well researched for regenerative trace contaminant control.⁵ The adsorption capacity of pelletized adsorbents is, however, limited by diffusion to the core of the pellet, restricting access to available adsorption capacity. Channeling, or the formation of preferential flow channels, is also common in pellet beds and results in similar capacity penalties while also increasing pressure drop. Pellet beds also suffer from fines generation when exposed to vibrational loads which adds additional complexities and hazards for downstream system components. Adsorbents immobilized on monoliths avoid this fines generation challenge, however they have much lower geometric surface area (GSA) per unit volume, thus limiting adsorbent loading capacity. While zeolite materials have shown higher capacity when coated on high surface area substrates, they still demonstrate reduced cyclic capacity when regenerated via vacuum alone; often requiring the addition of heat to be fully regenerated which is not feasible for real-time, on suit regeneration due to the increased energy load and safety concerns in the oxygen environment of a spacesuit. Finally, some zeolites are hydrophilic and their trace contaminant removal efficiency can be negatively affected by humidity.

As previously reported, PCI has been developing functionalized sorbent nanomaterials with high surface area – up to 2600 m²/g; more than five times that of activated carbon – and high capacity for trace contaminants.^{6,7,8,9} These high surface area nanomaterials offer specific surface characteristics onto which various chemical functionalities can be easily attached, thus offering the possibility of tailoring a sorbent system to specific needs. Through its studies, the developed functionalized nanomaterials were further applied on high surface area Microlith[®] substrates (Microlith[®] is a patented PCI's product) for improved bed utilization and working/sorption capacity during adsorption. The use of Microlith[®] mesh substrate also provides the potential for low-pressure-drop system, resulting from its high void fraction, as well as avoidance of boundary layer formation, maximized surface area, and high tolerance to mechanical vibration due to strongly adhesive and cohesive coating formulations developed over the years at PCI.

In this paper, we will summarize the development effort and evaluation of a second-generation TCC housing, to optimize pressure drop during integration and operation with CO₂/H₂O removal system within a xPLSS unit. We will discuss the TCC bed engineering and the resulting specifications, including life cycle and environmental testing at the anticipated operational conditions when integrated with a CO₂/H₂O removal system in a closed-loop ventilation test rig. We will also summarize our development effort and evaluation of sorbent nanomaterials for the competitive removal of trace contaminants, with a focus on ammonia, formaldehyde, and methyl mercaptan, which drive the bed sizing and other required metrics. Demonstrating adsorption under the presence of all three specified contaminants creates a more realistic environment to better evaluate sorbent performance. Future optimization based on the test data and sorbent performance will also be summarized.

II. Microlith[®] Substrate Technology

The TCC adsorber units described in this paper implement PCI's patented Microlith[®] substrate technology (trademarked by PCI) for supporting the sorbent materials.¹⁰ Microlith substrate consists of a series of ultra-short-channel-length, sorbent coated meshes (sorbents could be zeolites, functionalized nanomaterials, metal oxides, etc.) with very small channel diameters (Fig. 1). The meshes can also be coated with active metal catalysts, such as base metals, transition metals, platinum group metals, and noble metals. The very short channel lengths allow the avoidance of boundary layer buildup and hence achieve improved heat and mass transfer coefficients, along with the benefits of low thermal mass and extremely high reaction rates. These attributes increase the efficacy of the adsorbers and/or chemical reactors as well as reduce their weight and volume. The use of these kinds of systems, where the reacting stream is passed through the sorbent materials or catalysts at extremely high space velocity, is generically termed a *short contact time* approach. In conventional honeycomb monoliths, foams, and pellets, a fully developed boundary layer is present over a considerable length of the device, resulting in high resistance for heat and mass transport thus

limiting the rate of reactant or contaminants transport to the surface of active sites (e.g., metal catalysts or sorbent materials). This is avoided when short channel length screens are used. A Computational Fluid Dynamics (CFD) analysis (Fig. 1) using Fluent illustrates the difference in boundary layer formation between a long honeycomb monolith and Microlith screens.

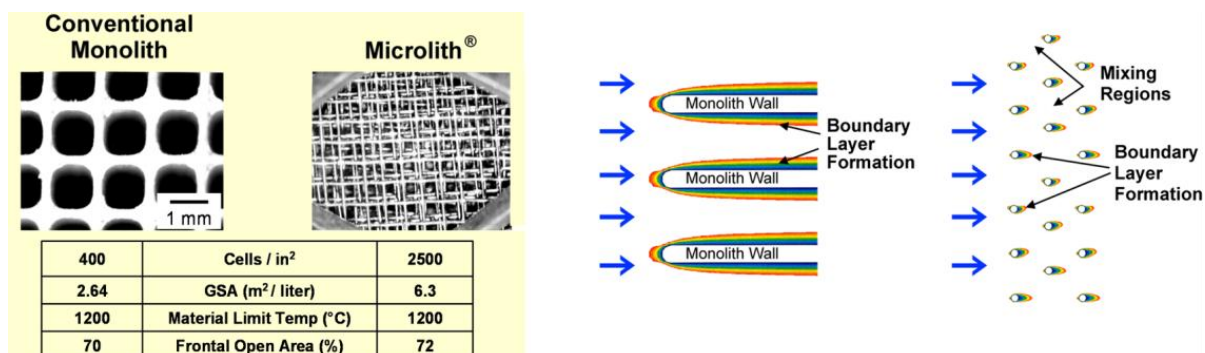


Figure 1. Physical characteristics of conventional, long honeycomb monolith and Microlith substrates (left), and CFD analysis of boundary layer formation for a honeycomb monolith and three Microlith screens (right), showing the minimization of boundary layer build up. The geometric surface area was calculated based on the ratio of the surface area of the strands to the volume of the substrates.¹¹

Microlith substrate provides about 3× higher geometric surface area over conventional monoliths with equivalent volume and open frontal area, resulting in a lower pressure drop while maintaining high surface area of active sites, which is beneficial for many applications requiring low pressure drop. Furthermore, Microlith substrate can pack more active surface area into a smaller volume, providing a substantial increase in adsorption area for a given pressure drop.

The use of Microlith substrates with high heat and mass transfer rates, high surface area, and low pressure drop has a significant impact on adsorber and reactor performance, weight, and size as compared to conventional pellet or monolith-based units. The effectiveness of the Microlith substrate technology and long-term durability of PCI's proprietary coatings have been systematically demonstrated in different applications. These include exhaust aftertreatment,¹² trace contaminant control,^{13,14} adsorption of Toxic Industrial Chemicals and sulfur,¹⁵ Sabatier reactors,¹⁶ catalytic combustion,¹⁷ partial oxidation of methane,^{18,19} liquid fuel reforming,²⁰ CO preferential oxidation, and water gas shift reactors.²¹

For xPLSS applications, utilizing Microlith substrate as the support structure for the sorbent nanomaterials provides the benefits of maintaining a low pressure drop at the high process air flowrate while keeping the TCC bed compact⁶ as well as improving the bed utilization and heat transfer. The improvement in bed utilization compared to the pellet beds resulted in higher gravimetric (wt.%) and volumetric (g/mL bed) sorption capacities in spite of the lower sorbent density in the Microlith support mesh compared to the bulk density of the sorbent material (e.g., pellets).²²

III. Experimental Setup

Sorbent preparation methods and the proof-of-concept prototype housing (first-generation) used for competitive sorbent performance evaluation in this paper have been described in previous International Conference on Environmental Systems (ICES) manuscripts.^{6,8,9} In short, the first-generation TCC assembly consisted of a round flanged sorbent canister containing approx. 20 mL of sorbent nanomaterial coated Microlith® substrate. The flexibility of the Microlith substrate enables the bed to be constructed with many different sorbents tailored for removing different TCs. To simultaneously remove each of trace contaminant of interest, a sorbent bed was assembled which contained some of each sorbent nanomaterial developed and optimized by PCI, in proportions corresponding to their effectiveness and the expected generation rate of each contaminant in the xPLSS. In general, the sorbent bed would need to be tailored for the trace contaminants of interest for the optimal performance. PCI sized the sorbent bed for this testing to provide 2- to 5-minutes of protection from each of the contaminants of interest (meeting current NASA full-scale protection period targets). Ammonia remains the contaminant with the highest generation rate in the xPLSS, and therefore its sorbent made up the dominant proportion of PCI's TCC bed, followed by the sorbents for removing methyl mercaptan and formaldehyde. The newly assembled TCC housing for competitive testing was installed on PCI's existing rapid cycle test rig which had been modified to permit measurement of multiple trace contaminants simultaneously.

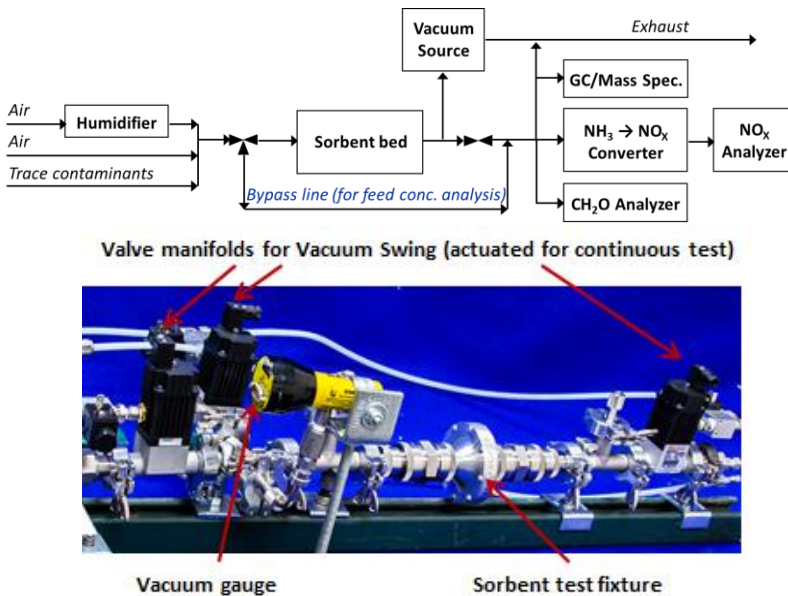


Figure 2. Schematic diagram and photo of PCI’s vacuum test rig and first-generation TCC housing, equipped with pneumatic valve manifolds, vacuum gauge, and sample ports, which allow for continuous, unattended vacuum swing operation.

Vaisala). Analysis of the NH_3 concentration was performed via a chemiluminescence detector from Thermo Scientific after converting NH_3 to NO_x in a standard catalytic oxidizer located immediately after the sorbent test fixture. Analysis of the CH_2O concentration was performed using an electrochemical voltametric formaldehyde sensor (RM Series, Interscan Corporation). Analysis of the methyl mercaptan concentration was measured with a gas chromatogram / mass spectrometer (Clarus 590/SQ8T, Perkin Elmer). Each analytical instrument was frequently calibrated and verified to ensure that the correct concentration of TCs was recorded during sorbent evaluation.

Table 2: Test matrix for competitive (i.e., co-adsorption) testing of PCI’s vacuum regenerable TCC module.

Parameters	Ammonia	Formaldehyde	Methyl Mercaptan
Adsorption Temperature	20 – 25°C (room T)		
Adsorption Pressure	1 atm		
Adsorption Atmosphere	Air		
Regeneration	Vacuum (0.3 to 6 mm Hg) @ room T		
Adsorption air flow rate	50 slpm (equivalent to the full-scale xPLSS mass flowrate of 6 ACFM at 4.3 psia and 17°C or 60 g/min)		
Relative humidity	20% (nominal humidity expected downstream of the RCA in the xPLSS)		
Bed Volume	~20 – 25 mL		
Inlet concentration	3.0 ppm _v (7-day SMAC)	0.2 ppm _v (2× 7-day SMAC)	1.0 ppm _v (2× 7-day SMAC)

PCI developed a modified protocol for competitive sorbent testing as summarized in Table 2. The process flow rate was maintained at the nominal rate of 50 slpm utilized throughout all sorbent testing (i.e., equivalent to the xPLSS full-scale *mass* flowrate of 6 ACFM at 4.3 psia and 17°C or 60 g/min) and the relative humidity was maintained at 20% (i.e., the nominal humidity expected downstream of the RCA in the xPLSS). Concentrations of 3.0 ppm_v NH_3 (the 7-day SMAC), 1.0 ppm_v methyl mercaptan (2× the 7-day SMAC, selected to be 2× the instrument’s detection limits) and 0.2 ppm_v formaldehyde (2× the 7-day SMAC, selected to be 2× the instrument’s detection limits) were utilized throughout the competitive testing. Due to cross sensitivity when measuring formaldehyde in the presence of methyl mercaptan, formaldehyde performance was only quantified periodically over the course of the competitive testing. During these periodic cycles (i.e., about every 20 cycles), methyl mercaptan was not included in the feed and only ammonia and formaldehyde performance was evaluated.

PCI used the same automated, vacuum test rig, described previously, during its recent sorbent nanomaterial evaluations.^{8,9} This rig could achieve vacuum levels ranging from 6 mm Hg down to 0.3 mm Hg during sorbent regeneration. Fig. 2 shows a schematic diagram and photo of the automated vacuum test rig. This rig includes an Edwards nXDS10iC dry scroll pump and Edwards IPV16PKAO pneumatic inline vacuum valves to perform vacuum regeneration. Swagelok Ultra-Torr® fittings were used to interface between the sorption bed canister and the rest of the test rig which uses ISO-KF flanges. Control and data acquisition is performed using a Raspberry Pi 4 Model B equipped with data acquisition and relay switching circuit boards to enable automated, unattended long term cyclic testing of PCI’s sorbent nanomaterials. Relative humidity (RH) was measured using a handheld RH detector (HMC 20,

IV. Experimental Results

A. Competitive Testing:

PCI completed approximately 25 hours (i.e., 150 cycles consisting of a fixed 5 minutes of exposure to contaminants followed by a fixed 5 minutes of vacuum regeneration at ~ 6 mm Hg) of competitive (i.e., co-adsorption) testing according to the matrix outlined in Table 2. A selection of the outlet concentration vs. time profiles obtained for ammonia, formaldehyde, and methyl mercaptan during competitive testing are provided in Figures 3, 4, and 5, respectively. The missing cycles were only omitted for clarity of the figures. As shown in Figure 3, the outlet concentration of ammonia never exceeded the breakthrough threshold of 1.5 ppm_v (i.e., 50% of the 7-day SMAC) and no performance degradation was observed over the 25 hours of 5-minute half cycle testing. The standard deviation for the maximum outlet NH₃ concentration across all 150 cycles was 0.1 ppm_v. This performance demonstrates that the selected bed configuration can effectively control the NH₃ concentration, even in the presence of other trace contaminants.

As shown in Figure 4, PCI's TCC module was found to consistently control the outlet concentration of formaldehyde below the 7-day SMAC (i.e., 0.1 ppm_v or 1/2 feed for this testing) throughout the 150 five-minute half cycle tests. An initial period of sorbent break-in was captured during cycle 20; however, all subsequent cycles showed consistent performance with minimal variation in the outlet concentration. The consistency of the formaldehyde sorption curves (standard deviation of 0.004 ppm_v) suggests that PCI's TCC can effectively control CH₂O under

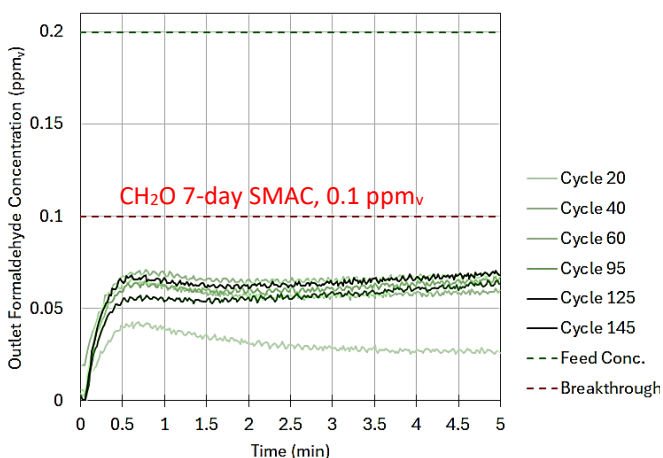


Figure 4: Outlet CH₂O concentration vs. time from 25 hours of competitive adsorption-regeneration cycle testing (using 50 lpm process air inlet with 3.0 ppm_v NH₃, 0.2 ppm_v CH₂O, 1.0 ppm_v CH₄S and 20% RH, 5-min vacuum regeneration). PCI's TCC consistently maintained formaldehyde below its 7-day SMAC.

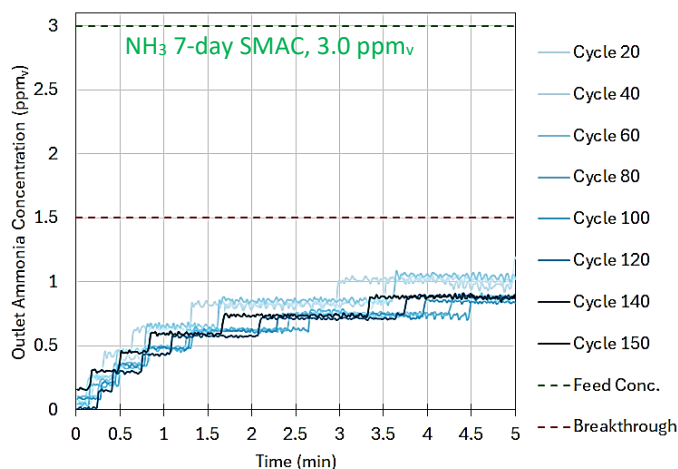


Figure 3: Outlet NH₃ concentration vs. time from 25 hours of competitive adsorption-regeneration cycle testing (using 50 lpm process air inlet with 3.0 ppm_v NH₃, 0.2 ppm_v CH₂O, 1.0 ppm_v CH₄S and 20% RH, 5-min vacuum regeneration). PCI's TCC consistently maintained ammonia below 50% of the 7-day SMAC.

competitive sorption conditions and is fully vacuum regenerable even with a weaker vacuum (compared to space vacuum) of 6 mm Hg.

Finally, as shown in Figure 5, PCI's TCC module was found to provide the required 2-minutes of protection (defined for this test as maintaining the outlet concentration below 95% of the feed) from methyl mercaptan under competitive sorption conditions and did not exhibit any degradation over 150 cycles of adsorption/vacuum regeneration. The standard deviation of the outlet methyl mercaptan concentration across all 150 cycles was 0.03 ppm_v. In light of the fact that the methyl mercaptan sorbent reaches saturation (i.e., outlet concentration equals inlet concentration) under these test conditions while the other two contaminants remain below their 7-day SMAC level, further optimization of the bed configuration should be possible. By exchanging some of the ammonia and/or formaldehyde coated substrate layers with additional layers of mercaptan removal sorbent,

it should be possible to extend the protection period for methyl mercaptan while maintaining the concentrations of ammonia and formaldehyde below the SMAC limits. This is proposed as future work for PCI's TCC module.

With the completion of 150 cycles of competitive contaminant testing (i.e., over ~25 hours of EVA equivalent operation), PCI's TCC module demonstrates the ability to maintain NH_3 , CH_4S and CH_2O below the required maximum limits for at least NASA's target of 2-minutes while demonstrating full vacuum regenerability with no signs of degradation. Performing additional cycling to assure durability over NASA's full 150 EVA hour target operating life and investigating the impact from other trace contaminants in the xPLSS ventilation loop, including those that are not expected to exceed their 7-day SMAC levels are proposed as potential future work for this technology.

B. Pressure Drop Optimization:

In addition to displaying progress in the area of competitive testing, PCI has also worked to minimize the pressure drop of the TCC module to reduce both its impact on the RCA performance and the power consumption of the xPLSS fan. Toward this goal, PCI developed a second-generation TCC canister design with a reconfigured bed geometry which fully utilizes the available space in NASA's Rapid Cycle Amine (RCA) $\text{CO}_2/\text{H}_2\text{O}$ removal module. Not only does this second-generation design have less pressure drop than the first-generation design,

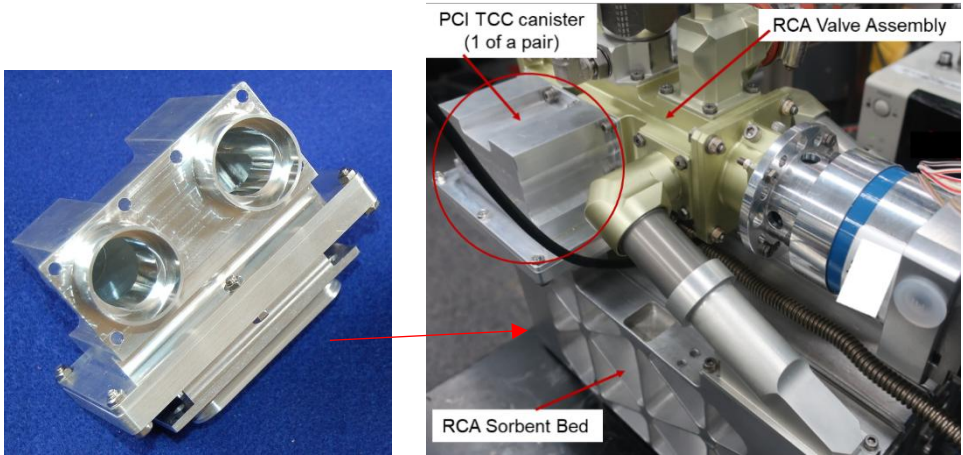


Figure 6: PCI's second generation TCC canister integrated with the RCA system for performance validation testing. The TCC canister takes the place of the RCA flow manifold. Note: The flange on this TCC canister prototype would not be present in finalized designs, allowing for flush integration.

TCC modules to capture ammonia released by the RCA before it enters the spacesuit and enables the TCC to utilize the RCA valve for simultaneous vacuum regeneration with the RCA bed. After multiple design-fabrication-test iterations involving CFD analysis and testing at PCI, a pair of second-generation housings were assembled and integrated with a $\text{CO}_2/\text{H}_2\text{O}$ removal module for performance evaluation under spacesuit relevant conditions. Note that a flanged approach was utilized for this prototype second-generation TCC design to facilitate sorbent installation/replacement/iteration during their development and optimization prior to integration with the $\text{CO}_2/\text{H}_2\text{O}$ removal module. PCI plans to eliminate this flange for future development efforts of production/flight hardware (e.g.,

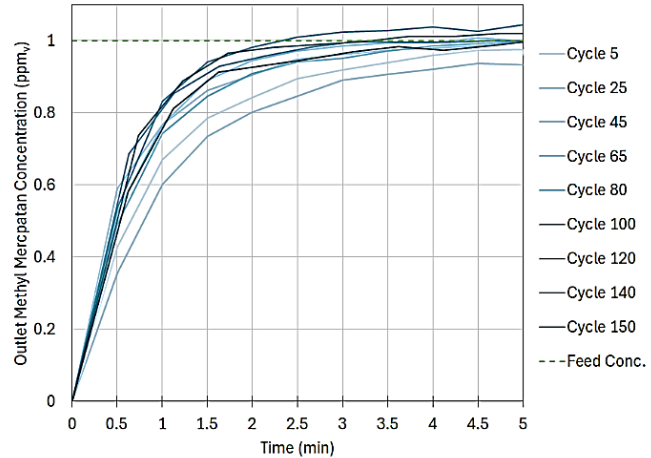


Figure 5: Outlet CH_4S concentration vs. time from 25 hours of competitive adsorption-regeneration cycle testing (using 50 lpm process air inlet with 3.0 ppm_v NH_3 , 0.2 ppm_v CH_2O , 1.0 ppm_v CH_4S and 20% RH, 5-min vacuum regeneration). PCI's TCC was able to control CH_4S for NASA's target of 2-minutes when operated in the presence of competing trace contaminants.

it also more intimately integrates with the RCA and does not exceed the space claim allotted to just the RCA in the spacesuit backpack. As shown in Figure 6, this second-generation design takes the place of the outlet flow header which connects the RCA sorbent bed with the integrated ball valve assembly used for vacuum swing regeneration. This integration location enables the

by using brazing). It should also be noted that during the integration testing with RCA, some of the TC analytical instruments were unavailable, and therefore only ammonia removal performance could be evaluated. To isolate the performance of the ammonia removal sorbent material during this testing, the materials for removing other trace contaminants were not included during assembly of these second-generation housings.

Table 3. Pressure drop analysis of PCI TCC prototypes.

Flow (ALPM)	Pressure Drop (inches of H ₂ O; [Pa])	
	First-generation prototype	Second-generation prototype
20	0.07 [17.4]	0.02 [5.0]
50	0.27 [67.2]	0.05 [12.4]
90	0.59 [146.8]	0.12 [29.9]
120	0.91 [226.4]	0.18 [44.8]
150	1.27 [316.0]	0.25 [62.2]
170 (equivalent to 6 ACFM at 4.3 psia and 17°C)	1.54 [383.2]	0.30 [74.7] (met NASA target)

After integration with the RCA module, the pressure drop of the second-generation TCC prototypes was evaluated to validate its improvement over the first-generation prototype. The pressure drop performance of the first and second-generation TCC prototypes at various flow rates are summarized in Table 3. As shown, the second-generation TCC showed significant pressure drop improvement compared to the first-generation design and met NASA's

target of <0.3 inches of water with 6 ACFM process air flow at 4.3 psia and 17°C.

After validating its pressure drop, the integrated RCA + TCC assembly was prepared for performance evaluation with CO₂, H₂O, and NH₃. It is important to note that the RCA CO₂/H₂O removal module used for this integrated testing had been in storage for an extended period of time prior to initiating this TCC testing. Therefore, the half cycle times for a given metabolic rate, automatically controlled by the CO₂ concentration exiting the RCA module, were shorter than they would have been for an assembly with fresh sorbents. In spite of this, meaningful data could still be collected during this testing and demonstrated that the integrated TCC + CO₂/H₂O module will meet NASA's performance requirements under xPLSS operating conditions.

Table 4. Test matrix used to evaluate PCI's second-generation TCC prototypes integrated with a CO₂/H₂O removal module.

Simulated metabolic rate (BTU/hr; [kW])	CO ₂ Flow Rate (slpm)	H ₂ O Flow Rate (g/min)	System Pressure (psia; [kPa])	System Flow Rate (ACFM; [LPM])	Instantaneous Cycle Threshold (mm Hg CO ₂ ; [kPa])	Fresh NH ₃ Injection Concentration (ppm)	Fresh NH ₃ Injection Rate (mg/min)
350 [0.1]	0.271	0.60	14.7 [101.3]	6 [170]	3 [0.4]	1.5	0.2 (total source rate)
350 [0.1]	0.271	0.60				3.0 (7-day SMAC)	0.4
1195 [0.35]	0.925	1.47				1.5	0.2 (total source rate)
1195 [0.35]	0.925	1.47				3.0 (7-day SMAC)	0.4
350 [0.1]	0.271	0**				1.5	0.2 (total source rate)
1195 [0.35]	0.925	0**				1.5	0.2 (total source rate)

** Dry test conditions to permit closed loop NH₃ recirculation to evaluate the steady state NH₃ conc.

The test matrix utilized to evaluate the integrated RCA and TCC systems is summarized in Table 4. This test matrix was developed to challenge the integrated system with process air loaded with carbon dioxide and humidity simulating a nominal metabolic rate of 1195 BTU/hr and a reduced metabolic rate of 350 BTU/hr. Two different levels of ammonia injection were tested: (i) 0.2 mg/min (83 mg/8-hr EVA) to simulate the total source generation rate expected within the xPLSS and (ii) 0.4 mg/min to simulate the worst-case scenario where the ventilation loop is being maintained at the 7-day SMAC (3 ppm NH₃). The concentration of ammonia exiting the TCC was continuously monitored and recorded using a chemiluminescence detector. Based on the capabilities of the test rig, all adsorption

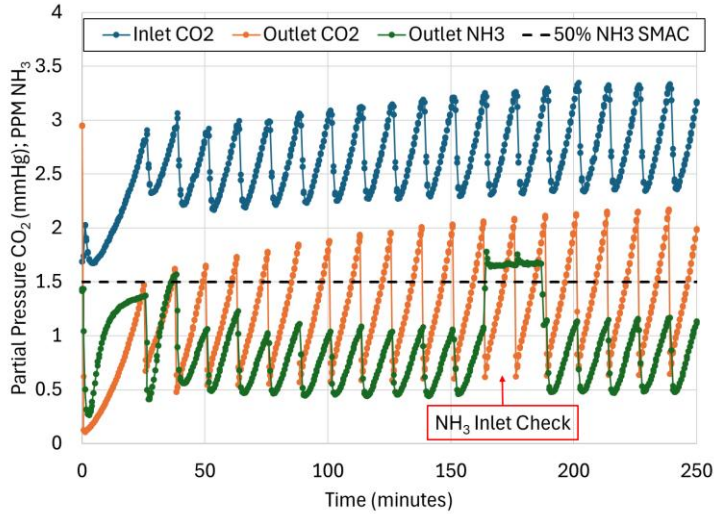


Figure 7. CO₂ partial pressure entering and exiting the RCA bed and NH₃ concentration exiting the RCA with integrated TCC under simulated 350 BTU/hr metabolic conditions with the total source NH₃ generation rate of 83 mg/8-hr EVA. PCI's TCC reduced the NH₃ concentration by ~43% for the full 4-hr. test under these full-scale spacesuit operating conditions.

laboratory which had the inadvertent effect of stripping out water soluble contaminants (e.g., NH₃). Therefore, it was not possible for the NH₃ concentration to build up in these tests and the feed was constant at the indicated level. For the final two conditions, humidity was omitted from the feed to eliminate the need for the humidity control system and to enable NH₃ recirculation. These final tests enabled the equilibrium, steady state NH₃ concentration in the suit to be examined.

The integrated RCA + TCC system was first tested with 350 BTU/hr equivalent amounts of CO₂ and humidity and with the total source (suit + metabolic) NH₃ generation rate of 83 mg/8-hr EVA (i.e., 0.2 mg/min NH₃ or 1.5 ppm_v). The results from cycling the combined system for ~4 hours at these conditions are provided in Figure 7. Unlike subsequent testing, the adsorption cycles were interrupted after 12.5 minutes rather than operating until the outlet CO₂ concentration exceeded 3 mmHg. This procedure change was implemented for this low metabolic rate test to enable more cycles to be completed in the allotted time. As shown, PCI's TCC consistently reduced the NH₃ concentration by at least ~43% and maintained it below 1.0 ppm_v for the duration of the test. The TCC also matched its design point by providing the required level of protection for up to ~10 minutes. More importantly, the ability of the RCA to maintain the CO₂ partial pressure below 2 mmHg for longer than 10 minutes while integrated with the TCC is a distinct improvement over previous testing with the first-generation TCC prototype.⁹ During the previous testing, the higher pressure drop first-generation TCC prototype restricted the vacuum level for the RCA regeneration and reduced its performance, such that it cycled every ~5 minutes (in response to the CO₂ partial pressure exceeding 3 mmHg). In fact, while not presented here, the RCA was found to operate with up to 20-minute half cycle times under simulated 350 BTU/hr conditions with and without an integrated second generation TCC. This result suggests the second generation TCC no longer has a negative effect on the RCA CO₂ removal performance.

Next, the simulated metabolic rate was increased to a nominal level of 1195 BTU/hr. The integrated RCA + TCC system was tested with ammonia injection rates of 0.2 mg/min (1.5 ppm_v) and 0.4 mg/min (3 ppm_v or the worst case, highest permissible NH₃ load in the xPLSS); however, for brevity, only a chart from the 0.4 mg/min testing is presented here because it was the most challenging condition. This level of ammonia in the suit is not expected to occur; however, it demonstrates the performance of PCI's TCC under the most strenuous operating conditions. As shown in Figure 8, the RCA half cycle time decreased to ~40 seconds as a result of the increased CO₂ load on the aged CO₂ sorbent beds. Under these conditions, the TCC removed at least 83% of the inlet ammonia and maintained the outlet concentration below 0.6 ppm_v. A similar removal efficiency was observed when NH₃ was injected at 0.2 mg/min (1.5 ppm_v) and the TCC consistently maintained the outlet concentration below 0.15 ppm_v. This increase in removal efficiency at higher metabolic rates is consistent with the results from previous testing and indicates that

cycles were performed at atmospheric pressure (i.e., 14.7 psia). The test rig utilized 1 to 2 mm Hg of vacuum to regenerate both the RCA and TCC modules simultaneously. Unlike the competitive testing presented earlier which used a fixed half cycle duration, this test rig had the capability to automatically switch between adsorption and regeneration as needed to maintain the outlet CO₂ partial pressure below 3 mm Hg. This resulted in a dynamic half cycle time based on the CO₂ removal performance of the RCA sorbent which is more representative of the actual application. The process flow rate was maintained at 6 ACFM (i.e., the expected full scale xPLSS ventilation loop flow rate) for all testing. It is important to note that in the first four test conditions, the ammonia exiting the TCC could not be recirculated back to the inlet due to the nature of the humidity control system in the closed loop test rig of the test

shorter half cycle times are beneficial for PCI's TCC sorbent due to there being less total NH_3 to remove per cycle. This further confirms that, in contrast to the RCA amine sorbent, PCI's TCC sorbent is adsorption limited and that it regenerates rapidly to vacuum regardless of vacuum pressure or duration of vacuum exposure.

As mentioned earlier, the final tests on the RCA with integrated TCC involved not injecting water to enable recirculation of the NH_3 exiting the TCC. This recirculation more closely approximates the NH_3 concentrations that will be encountered in the spacesuit xPLSS by allowing the concentration of NH_3 to build above the total source concentration and even surpass the 7-day SMAC. Data from testing the system at 350 BTU/hr equivalent amounts of CO_2 (without humidity) and the total source NH_3 generation rate of 83 mg/8-hr EVA is provided in Figure 9. The inlet concentration to the TCC, which includes the total source (suit + metabolic) NH_3 generation rate of 83 mg/8-hr EVA and any ammonia not captured by the TCC, never exceeds 2.5 ppm_v under these operating conditions. As shown, PCI's TCC module maintained the suit concentration below the 7-day SMAC level even with a dynamically changing feed concentration. It appears that the equilibrium outlet NH_3 concentration under these conditions will be ~1 ppm_v (i.e., ~33% of the 7-day SMAC). The average half cycle time at these conditions was ~8.3 minutes. This ability to control the ammonia concentration in the suit to below the 7-day SMAC while operating

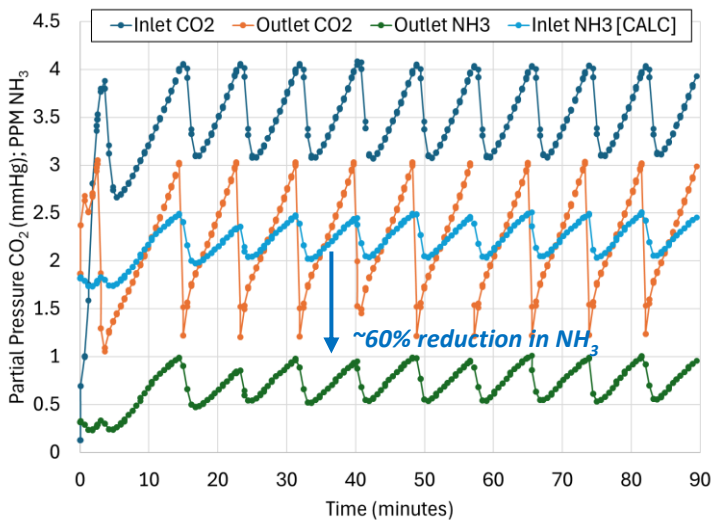


Figure 9. CO_2 partial pressure entering and exiting the RCA bed and NH_3 concentration exiting the RCA with integrated TCC under simulated 350 BTU/hr. metabolic conditions with the total source (suit + metabolic) NH_3 generation rate and recirculation of the outlet NH_3 . The TCC maintained the equilibrium NH_3 concentration within the suit below 33% of the 7-day SMAC when operated with >8-minute half cycles.

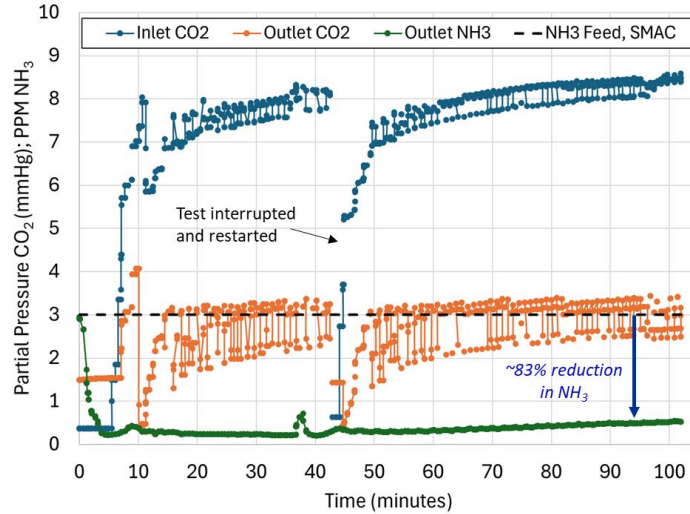


Figure 8. CO_2 partial pressure entering and exiting the RCA beds and NH_3 concentration exiting the RCA with integrated TCC prototypes under simulated 1195 BTU/hr. metabolic conditions with 3 ppm_v NH_3 in the feed (7-day SMAC). PCI's TCC beds consistently reduced the NH_3 concentration by at least 83%, maintaining the suit below 0.6 ppm_v NH_3 .

with half cycle times in excess of the 2- to 5- minute half cycle target, in conjunction with all the findings discussed above, suggests that PCI's second-generation TCC prototypes are correctly sized to provide the required protection for the xPLSS with some safety margin. When the simulated metabolic rate was increased to 1195 BTU/hr while maintaining the ammonia injection rate at 83 mg/8-hr EVA, the inlet concentration to the TCC never exceeded 2.0 ppm_v and the outlet NH_3 concentration appeared to stabilize at ~0.45 ppm_v. This improvement in performance was once again attributed to the shorter half cycle times at the higher metabolic rate and the adsorption limited nature of our TCC sorbents. This result further confirms the sizing of PCI's second-generation TCC prototypes for this xPLSS application.

In summary, the results from this integrated system testing validated the performance of PCI's second-generation TCC prototypes under full scale xPLSS

operating conditions. Altogether, PCI’s second-generation TCC modules underwent a total of 175 cycles at 6 different operating conditions representative of those present in the full-scale spacesuit xPLSS ventilation loop. This amounted to approximately 15 hours of operation while integrated with NASA’s RCA CO₂/H₂O removal module with minimal signs of performance degradation. The results of this testing confirm the capability of PCI’s TCC modules to provide NASA’s desired level of protection under representative full scale xPLSS operating conditions. The test results are encouraging and show the potential implementation of PCI’s TCC beds for effectively removing NH₃ and other trace contaminants of interest from the space suit ventilation loop, being able to maintain their concentrations below the 7-day SMAC, and demonstrated vacuum-regenerability (without any heat requirements). PCI’s second-generation TCC module provides a regenerable solution for TCC that is smaller, lighter, and less reliant on consumables than the state-of-the-art non-regenerable activated carbon solution currently utilized in the PLSS. A size and weight comparison between PCI’s TCC and the state-of-the-art carbon bed is provided in Table 5.

Table 5. Size and weight comparison between PCI’s regenerable TCC and the state-of-the-art non-regenerable TCC solutions.^{23,24,25}

	State-of-the-Art Activated Charcoal Bed	PCI Second-Generation TCC Prototype (flanged)	Estimate for Flight Hardware Design (without flange)
Weight (lbm; [kg])	1.9 [0.9]*	1.3 [0.6] (total 2-bed solution)	0.88 [0.4] (total 2-bed solution)
Volume (in ³ ; [mL])	87 [1428]*	76 [1245] (total 2-bed solution)	48 [787] (total 2-bed solution)
Operating Life (EVA hr)	150	>220 (demonstrated to date with PCI sorbent)	
*Estimate only, based on literature data			

V. Conclusion

In summary, this paper describes the results of PCI’s development and testing efforts for a compact, low pressure drop, vacuum regenerable TCC module for xPLSS applications. PCI’s sorbent optimization efforts have resulted in new formulations capable of providing the requisite protection from a broad range of trace contaminants, including simultaneous removal of ammonia, formaldehyde, and methyl mercaptan under both dry and humid conditions. The recently developed formulations have improved humidity tolerance and have demonstrated no cycle-to-cycle variability or loss of ability to maintain trace contaminants below their respective 7-day SMAC levels when operated for greater than 150 two-to-five-minute half cycles (i.e., ~25 hours of EVA equivalent operation) with regeneration to vacuum. Potential future work could include bed optimization to identify the necessary amount of methyl mercaptan sorbent to provide 5-minutes of protection, testing with additional trace contaminants, and further cycling to confirm durability for the requisite 150-hour operating life.

Additionally, PCI developed a second-generation TCC housing which achieves NASA’s pressure drop target of <0.3 inch of H₂O at the xPLSS ventilation loop conditions of 6 ACFM process air at 4.3 psia and 15.6°C while meeting NASA’s weight targets and integrating with the CO₂/H₂O removal module without exceeding its volumetric footprint. This second-generation TCC prototype was integrated with one of NASA’s CO₂/H₂O removal modules and demonstrated capability to maintain NH₃ below its 7-day SMAC for up to 10 minutes (at least double NASA’s target half cycle time of 2- to 5-minutes). These TRL-5 TCC beds were delivered to NASA for further performance evaluation and are ready for TRL-6 testing in a relevant environment.

These breakthroughs enable a compact, low pressure drop, and vacuum-regenerable TCC device for efficient removal of trace contaminants in PLSS, thus offering the potential for real-time, on-the-suit sorbent regeneration, reduced logistical burden associated with the bed replacement or thermal regeneration, and further volume and weight reduction of the TCC module. This effort is valuable to NASA as it significantly reduces the current PLSS technical risks and increases mission capability/durability/efficiency while at the same time increasing the TRL of the novel vacuum regenerable TCC system.

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