Regenerable Trace Contaminant Control for Advanced Portable Life Support System

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Christian Junaedi, Kyle Hawley, Codruta Loebick, and Nicholas Cameron

Precision Combustion, Inc. (PCI), North Haven, CT, USA

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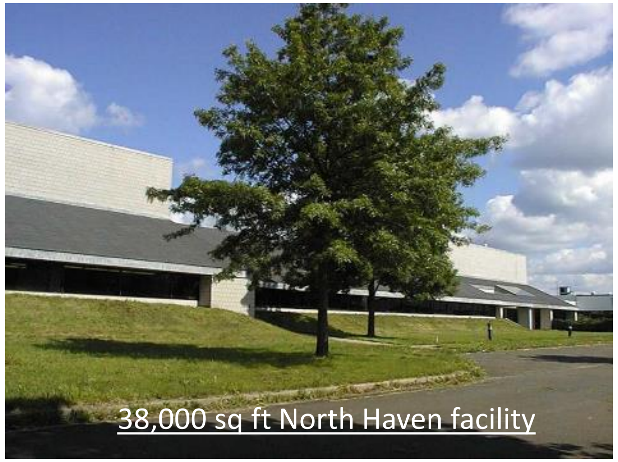
Presenter e-mail address: cjunaedi@precision-combustion.com

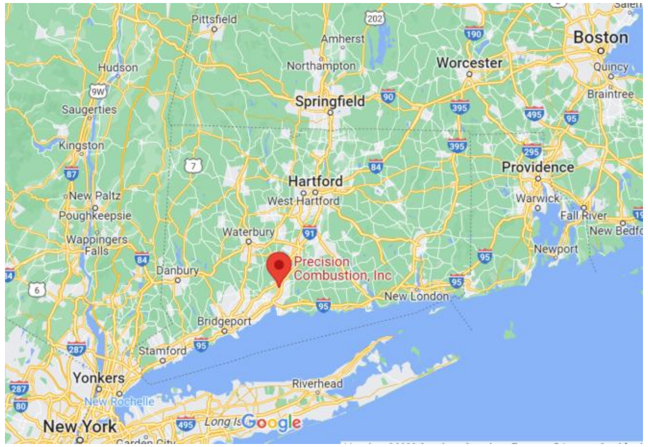
PCI's website: www.precision-combustion.com

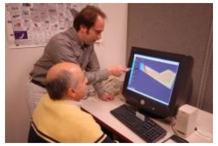
Precision Combustion, Inc. (PCI)

- Privately-held small business; established in 1986; located in North Haven, CT
- 48 employees: 14 PhDs, 34 engineers/technicians/admin.
- Core technologies: *Materials, Reactors, & Systems* for Energy, Power Generation & Environmental sectors
- Focus on *innovation & product development*
- Customers/partners: U.S. Govt., large & small companies, universities



38,000 sq ft North Haven facility







From Concept to Fielded Prototypes; multiple patents & IP's

R&D Background – Problem Statement for Vacuum Regenerable TCC

Trace Contaminant Control (TCC) for Advanced Spacesuit Applications

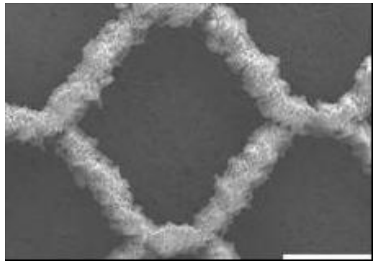
- A new TCC technology is needed, specifically considering power requirement, pressure drop, size & capacity.
- Implementation of Rapid Cycle Amine (RCA) bed for CO_2 removal in advanced spacesuit PLSS has created the need for removing trace contaminants due to ammonia off-gassing.
  - Earlier NASA report predicted that ammonia, formaldehyde, & methyl mercaptan will exceed SMAC w/o TCC device in the PLSS ventilation loop.
  - *Non-regenerable activated carbon* is the *current SOA* for TCC, resulting in logistical challenges, bulky system & reliance on consumables.
- Preference is for a TCC technology that can be regenerated in real time on-the-suit using a vacuum swing with about 3 min cycle (can be up to 15 min during low metabolic periods):
  - 3-min swing is the typical operation for the RCA bed.
  - Sorbent regenerability reduces bed size & eliminates filter change-out requirements.
  - Vacuum regeneration capability reduces power consumption.

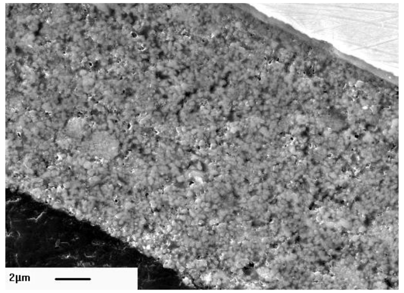
Focus of our R&D effort: evaluation of low pressure drop, *vacuum-regenerable sorbents* that could be integrated with the PLSS CO_2/H_2O removal system

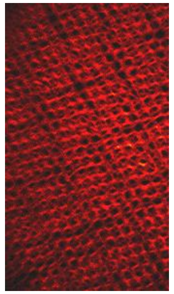
Outline

- *Approach*: Combination of PCI's Microlith® Substrate Technology & Novel Sorbent Materials
- *Design* of Trace Contaminant Control (TCC) Hardware
- *Evaluation* of PCI's TCC Modules:
  1. Experimental setup at PCI
  2. First generation prototype under competitive sorption conditions
  3. Second generation prototype integrated w. CO_2/H_2O removal module (simulated ventilation loop)
- *Concluding remarks*

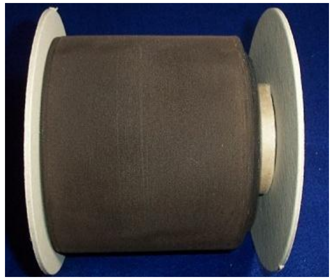
PCI Microlith® Substrate Technology

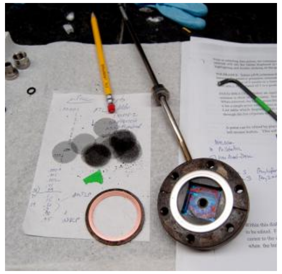






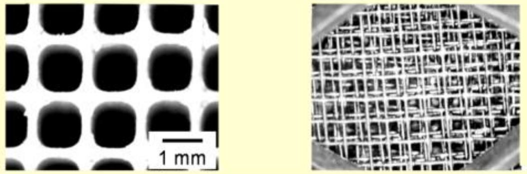
Thin, durable, metal mesh w. *very high surface area* that can be coated with sorbents or catalysts





Continuous sorbent & catalyst coating line with batched furnace & rigorous QA, QC in place

Conventional Monolith | Microlith®



400	Cells / in^2	2500
2.64	GSA (m^2/ liter)	6.3
1200	Material Limit Temp (°C)	1200
70	Frontal Open Area (%)	72

Microlith® Reactors or Adsorbers

- Low cost
- Compact
- Rapid thermal response
- High heat & mass transfer
- High surface area per unit volume
- Low pressure drop vs. pellet beds
- Design flexibility (e.g., planar, radial)

U.S. Patents 10,994,241 & 11,691,103 – Sorbent system for removing ammonia and organic compounds from a gaseous environment

Combining Microlith substrate w. functionalized sorbent nanomaterials for TCC applications

TCC Canister Design/Sizing

PCI continues to advance its TCC canister designs for demonstration in xPLSS applications

A pair of Gen-1 TCC canisters developed for vacuum-swing operation (used for sorbent evaluation/development)

Flow (ALPM)	Pressure Drop (inches of H_2O)	
	Gen-1 prototype	Gen-2 prototype
20	0.07	0.02
50	0.27	0.05
90	0.59	0.12
120	0.91	0.18
150	1.27	0.25
170 (equivalent to 6 ACFM at 4.3 psia and 17°C)	1.54	0.30 (met NASA target)

A pair of Gen-2 TCC canisters developed for low pressure drop & integration with a CO_2/H_2O removal module

- PCI's first-generation prototypes met the required TC removal efficiency & vacuum regenerability, but exhibited high pressure drop.
- The second-generation prototypes were modified to:
  - Meet NASA's pressure drop target of 0.3 in H_2O with 6 ACFM flow (at 4.3 psia and 17°C).
  - Intimately integrate with the RCA CO_2/H_2O removal module without exceeding its allotted space claim in the xPLSS backpack.

PCI has developed a TCC module which now meets all of NASA's performance and design targets

PCI Precision Combustion, Inc.

TCC Canister Design/Sizing (Cont.)

Flange & bolts utilized for prototype to aid sorbent iteration for validation testing.

These features can be eliminated during future development of production hardware (e.g., use brazing).

Second generation TCC test prototype

Size parameters for PCI's second generation TCC modules

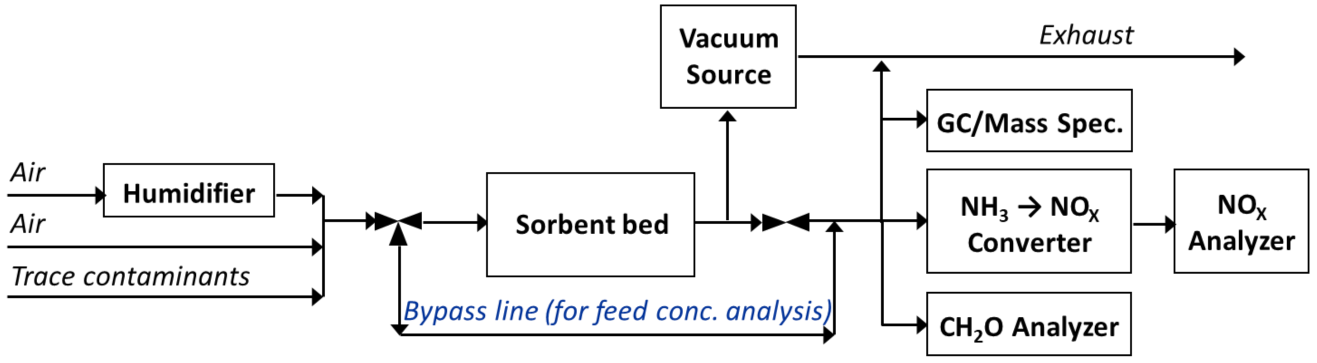
	State-of-the-Art Activated Charcoal Bed	PCI Second-Generation TCC Prototype (flanged)	Estimate for Flight Hardware Design (without flange)
Weight (kg)	0.9*	0.6 (total 2-bed solution)	0.4 (total 2-bed solution)
Volume (mL)	1428*	1245 (total 2-bed solution)	787 (total 2-bed solution)
Operating Life (EVA hr)	150	>220 (demonstrated to date with PCI sorbent)	

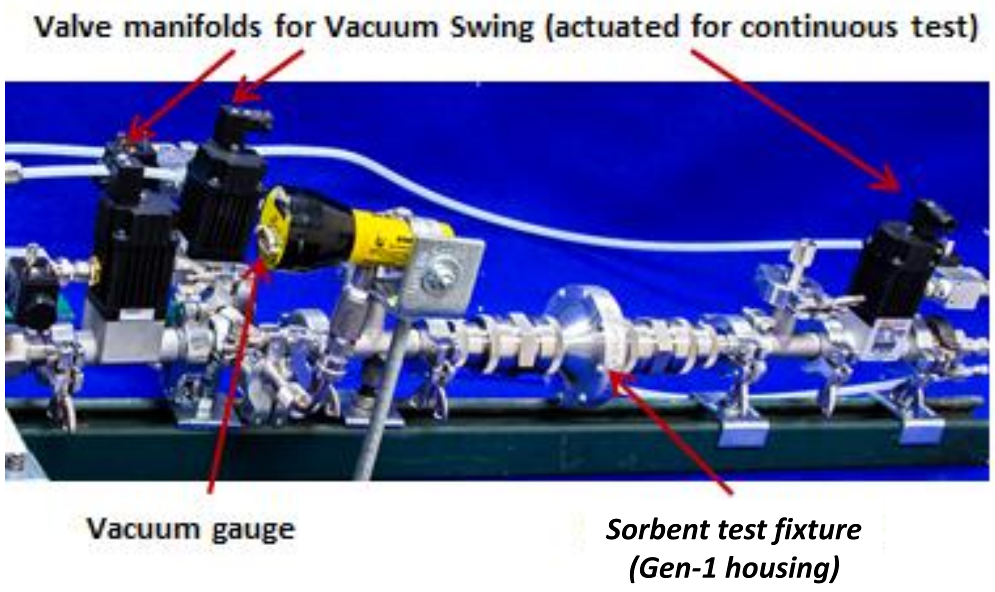
*Estimate only, based on published literature data.

A pair of PCI's second-generation prototypes are projected to achieve roughly 50% volume & weight savings compared to the state-of-the-art, non-regenerable activated charcoal bed

Experimental Setup at PCI

Test setup at PCI to facilitate automated vacuum-swing operation (adsorption – desorption)





Evaluation criteria for sorbent materials:

- Protection time (NASA targets a 2 to 3-min vacuum swing)
- Contaminant removal effectiveness
- Regenerability (*vacuum @ room T using vacuum pump*)
- Humidity tolerance

Test rig utilizes actuated valve manifolds for automated vacuum swing operation

Test Matrix for Sorbent Screening at PCI (Gen-1 Housing)

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)

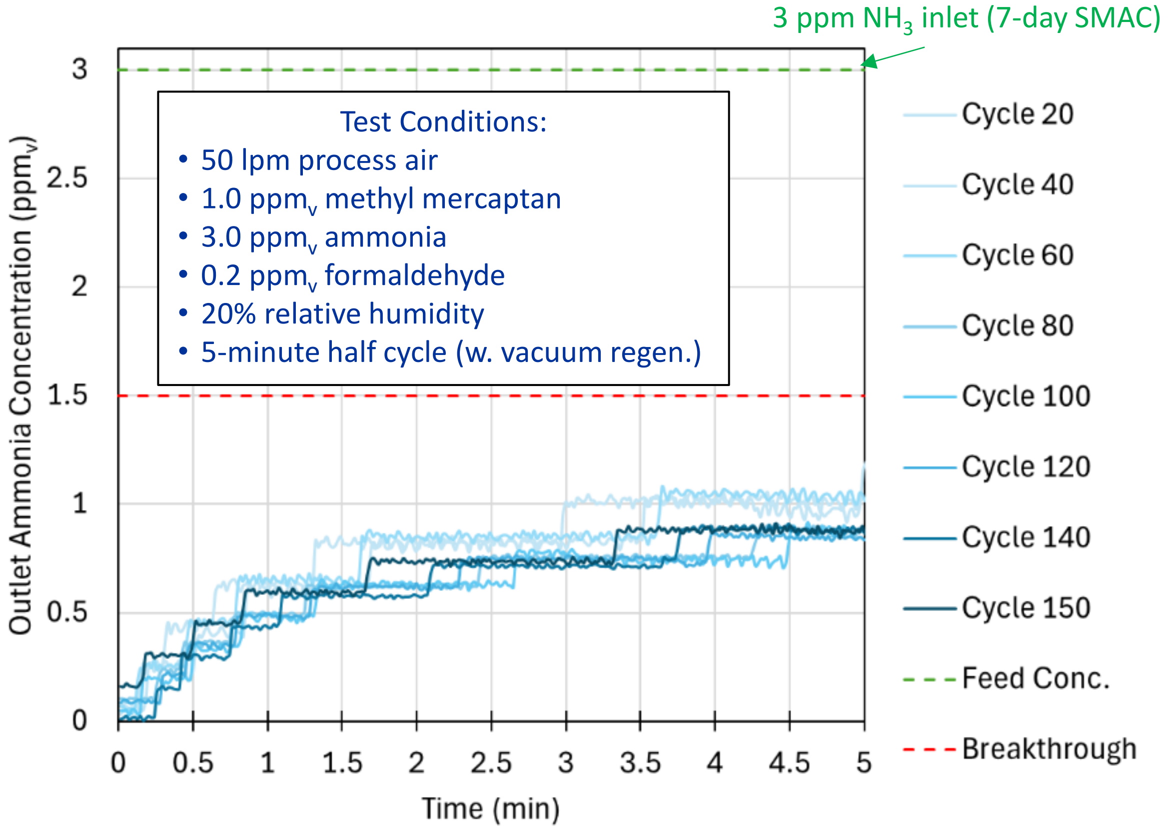
- Test conditions selected to simulate expected environment in xPLSS ventilation loop.
- Test matrix permits sorbent performance evaluation under competitive sorption conditions (i.e., simultaneous exposure to multiple contaminants).
- Formaldehyde and methyl mercaptan inlet conc. set at twice their SMAC levels to avoid PCI's sensor detection limits.

PCI's functionalized TC removal sorbent nanomaterials were evaluated under competitive sorption conditions

Competitive Sorption Testing: w. Ammonia, Methyl Mercaptan & Formaldehyde

- Prepared first generation TCC housing containing sorbents for ammonia, methyl mercaptan & formaldehydes removal:
    - Bed was tailored for a 5-min half-cycle.
- Inlet process air w. a cocktail of trace contaminants consisting of:
    - 3.0 ppm_v ammonia (i.e., 7-day SMAC)
    - 1.0 ppm_v methyl mercaptan (2× SMAC to avoid sensor detection limits)
    - 0.2 ppm_v formaldehyde (2× SMAC to avoid sensor detection limits)
    - 20% relative humidity
- Operated the TCC bed with 5-minute half cycle & regeneration under vacuum.

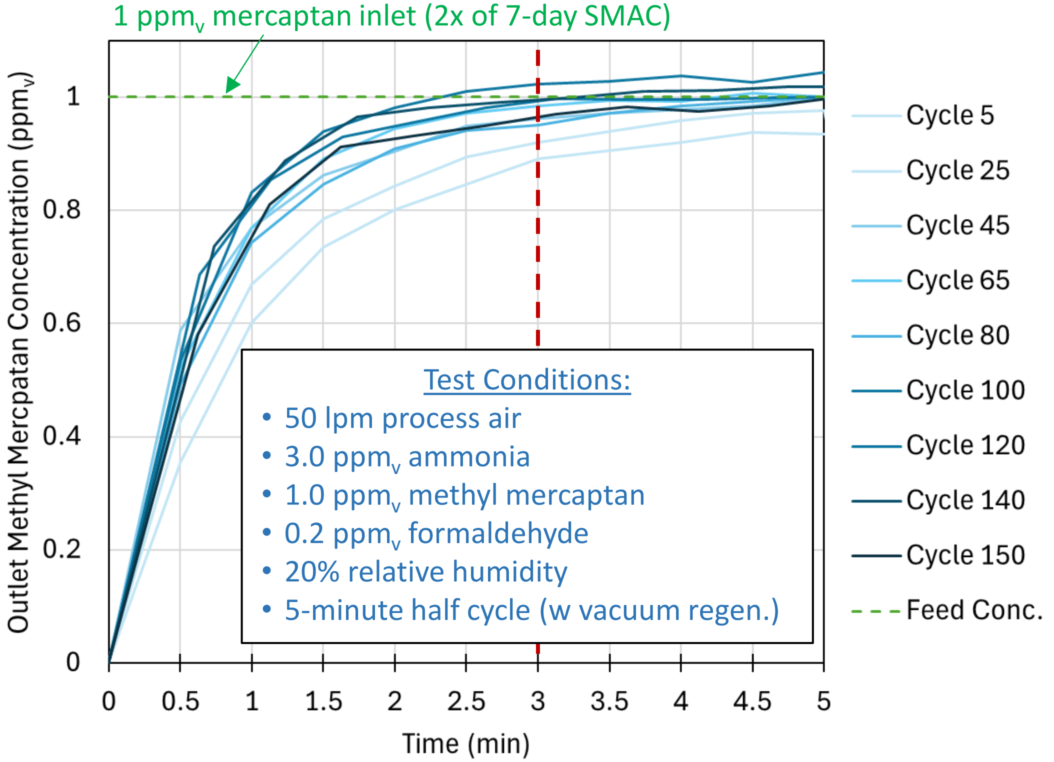
Outlet NH_3 conc. vs. time during competitive sorption test

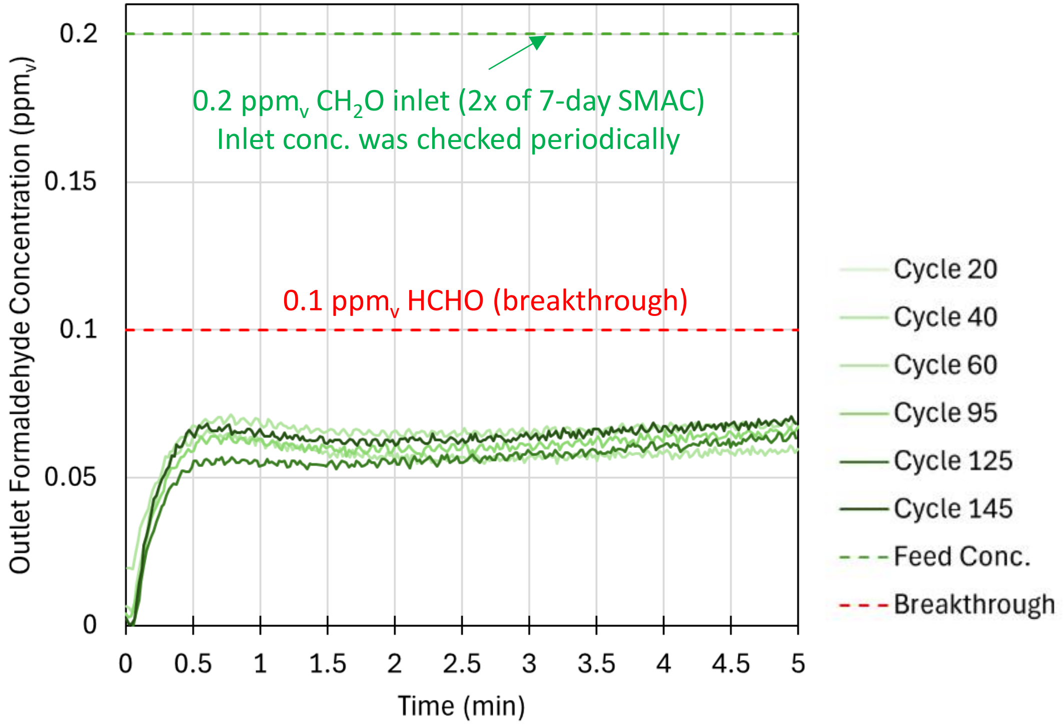


PCI's TCC module maintained NH_3, as well as other trace contaminants of concern, below safe levels for the designed 5-min half-cycle with vacuum regenerability

Competitive Sorption Testing: w. Ammonia, Methyl Mercaptan & Formaldehyde (Cont.)

Outlet methyl mercaptan (left) & formaldehyde (right) conc. vs. time during competitive adsorption testing





- Due to sensor interference with methyl mercaptan, formaldehyde sorbent performance could only be confirmed periodically (every 20 cycles).
  - Formaldehyde was injected into the TCC bed with ammonia & methyl mercaptan throughout all cycles.

PCI's TCC module showed stable performance for all trace contaminants over a total of 150 cycles (~25 hours of EVA equivalent operation)

Gen-2 TCC Canister Integration w. CO_2/H_2O Removal System

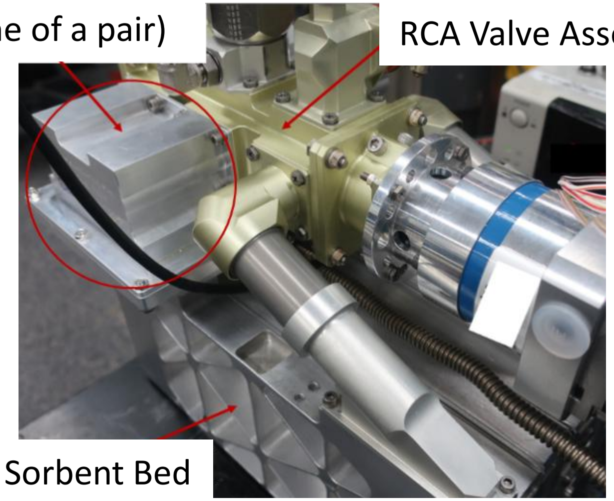
Integration of PCI's Gen-2 TCC canisters (two beds) into rapid cycle amine CO_2/H_2O removal module (test performed at a 3rd party facility):

- TCC beds integrated downstream of amine bed, within RCA valve assembly; utilizing the RCA valve manifold for simultaneous vacuum swing operation.
  - TCC beds & RCA will have the same half-cycle time.
  - This placement takes advantage of CO_2 & water vapor exposure during bed regeneration (beneficial to maintain sorbent performance).
- TCC canisters directly replace the stock RCA outlet flow manifolds & fit within the xPLSS backpack space claim.

PCI's second generation TCC canisters integrated with RCA

The flange in this canister was only for this integration effort (sorbent iteration at PCI) & can be eliminated in future canister development for flush integration.

PCI's second generation, low pressure drop TCC canister developed for intimate integration with RCA



Integration of PCI's second generation TCC canisters in the flow manifold of RCA for proof-of-concept performance evaluation

Test Matrix for TCC Evaluation Integrated w. CO_2/H_2O Removal System

- Objective: to challenge integrated system with **6 ACFM process air** loaded with CO_2, humidity, & ammonia.
  - Tested at nominal (1195 BTU/hr) & reduced (350 BTU/hr) metabolic rates for CO_2 & water inlet flows.
  - Varied NH_3 injection to simulate:
    - Case 1: The total suit + metabolic generation rate (83 mg/EVA or 0.2 mg/min)
    - Case 2: Operating at the 7-day SMAC of 3 ppm (*worst-case scenario*)
- *Use only vacuum to regenerate* both the CO_2/H_2O Removal System & TCC sorbents simultaneously (utilize existing valve manifold).
- The valve actuates (i.e., swing between sorbent beds) automatically based on outlet CO_2 partial pressure of 3 mm Hg.

Test #	Simulated Metabolic Rate	CO_2 Flow	H_2O Flow	System Flow Rate	Fresh NH_3 Injection Conc.	Fresh NH_3 Injection Rate	Notes
	[BTU/hr]	*[slpm]*	*[g/min]*	*[ACFM]*	*[ppm_v]*	*[mg/min]*	--
1	350	0.271	0.6	6	1.5	0.2 (total source rate)	Suit + metabolic
2	350	0.271	0.6	6	3.0 (7-day SMAC)	0.4	Worst case
3	1195	0.925	1.47	6	1.5	0.2	Suit + metabolic
4	1195	0.925	1.47	6	3.0 (7 day SMAC)	0.4	Worst case
5	350	0.271	0	6	1.5	0.2	Dry inlet to permit NH_3 recirculation
6	1195	0.925	0	6	1.5	0.2	

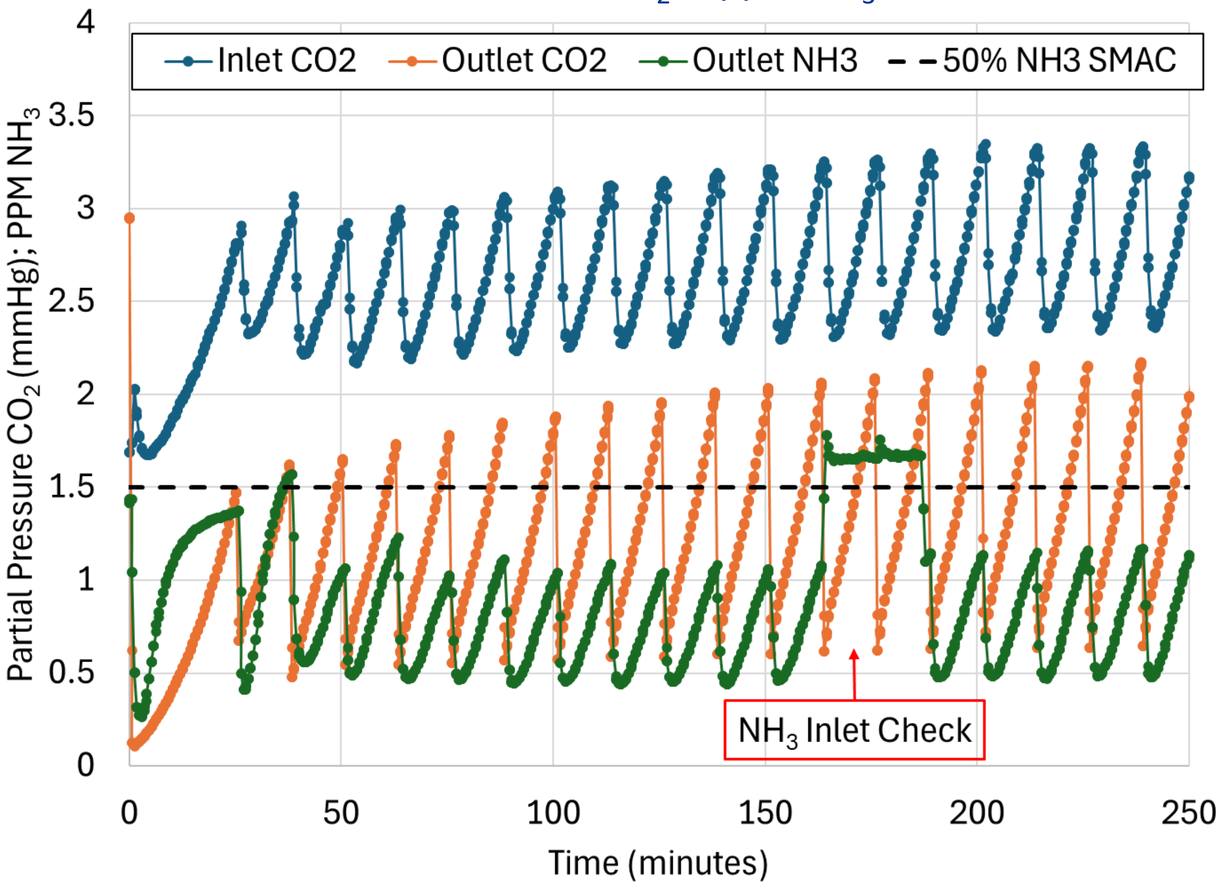
Established test matrix for evaluation of PCI's proof-of-concept TCC prototype under suit relevant flows & operating conditions

Test #1: 6 ACFM; 350 BTU/hr; 1.5 ppm NH_3

Test #	Simulated Metabolic Rate	CO_2 Flow	H_2O Flow	Fresh NH_3 Injection Conc.	Fresh NH_3 Injection Rate
	[BTU/hr]	*[slpm]*	*[g/min]*	*[ppm_v]*	*[mg/min]*
1	350	0.271	0.6	1.5	0.2 (total source rate)

- Injected NH_3 to match expected total suit + metabolic generation rate (83 mg/EVA; 0.2 mg/min).
- RCA operated with 12.5-minute cycle time (fixed) – exceeding NASA solicitation target of 2-min half cycle.
- TCC provided stable protection throughout the 4-hour test.
  - TCC removed 43% of NH_3 fed
  - Outlet NH_3 conc. was maintained at ~1 ppm

Partial Pressure of CO_2 & ppm NH_3 vs. time

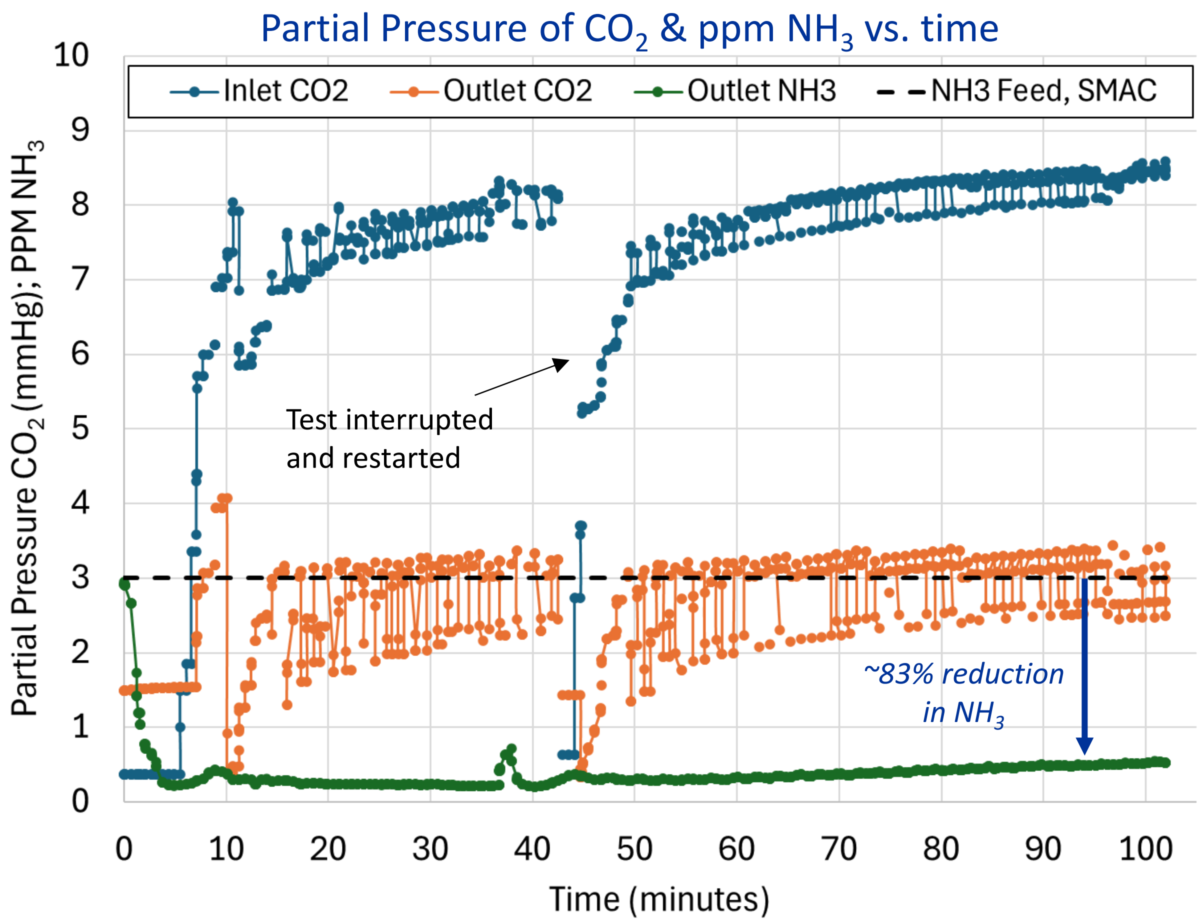


PCI's TCC prototype maintained the required NH_3 removal & NH_3 conc. in the suit throughout the test

Test #4: 6 ACFM; 1195 BTU/hr; 3 ppm NH_3 (Most Aggressive Case)

Test #	Simulated Metabolic Rate	CO_2 Flow	H_2O Flow	Fresh NH_3 Injection Conc.	Fresh NH_3 Injection Rate
	[BTU/hr]	*[slpm]*	*[g/min]*	*[ppm$_v$]*	*[mg/min]*
4	1195	0.925	1.47	3.0	0.4

- Increased to nominal metabolic rate of 1195 BTU/hr.
  - NH_3 feed conc. set to most aggressive scenario of suit operating continuously at 7-day SMAC (i.e., 3 ppm$_v$).
- RCA cycle times were shorter than nominal due to the age of this batch of RCA sorbent (i.e., <1 min. vs. nominal 2 – 5 min.).
  - Controlled by RCA CO_2 concentration (to maintain <3 mm Hg CO_2)
- Rapid cycling benefited TCC performance:
  - TCC removed ~83% of NH_3 fed
  - Outlet NH_3 conc. maintained below 0.6 ppm (< ½ SMAC)
- Results highlight that **PCI's TCC sorbent is adsorption limited**:
  - Sorbent rapidly regenerates when exposed to vacuum regardless of duration.
  - TCC bed size driven by NH_3 removal requirement.



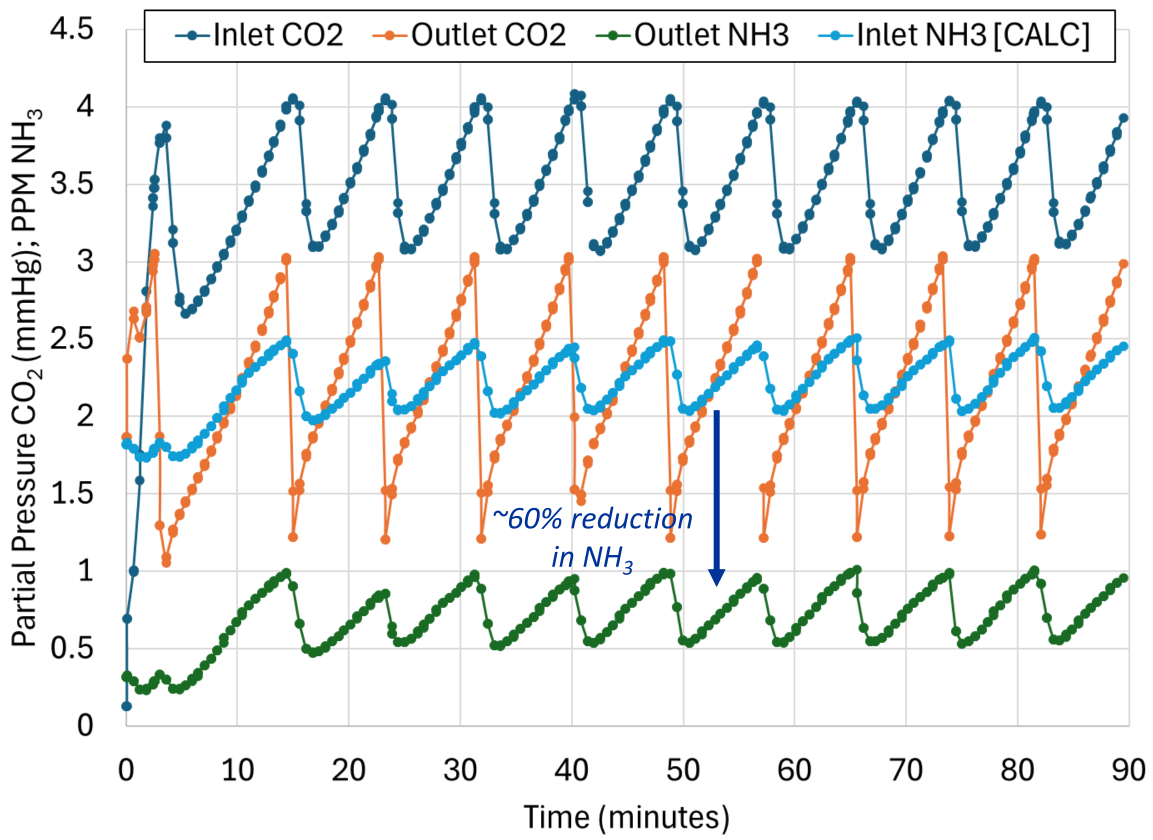
Test showed that TCC prototype achieved required NH_3 removal even with shorter half cycle times & 2× NH_3 generation rate (the beds maintained NH_3 outlet conc. with reduced vacuum regen. duration)

Test #5: 6 ACFM; 350 BTU/hr; NH_3 Recirculation (Closed Loop)

Test #	Simulated Metabolic Rate	CO_2 Flow	H_2O Flow	Fresh NH_3 Injection Conc.	Fresh NH_3 Injection Rate
	[BTU/hr]	[slpm]	[g/min]	[ppm_v]	[mg/min]
5	350	0.271	0	1.5	0.2

- Presence of H_2O in previous tests prevented recirculation of outlet NH_3 (limited by test system).
  - NH_3 was stripped out by inlet humidity control system.
  - Therefore, all NH_3 in feed was freshly injected.
- This test was performed dry to recirculate NH_3 exiting TCC along with freshly injected NH_3 (at 0.2 mg/min).
  - Simulates NH_3 conc. dynamics in suit.
  - Injected fresh NH_3 at the total source rate.
- TCC maintained outlet NH_3 conc. < 1 ppm_v, indicating TCC removed targeted NH_3 amount (suit + metabolic).

Partial Pressure of CO_2 & ppm NH_3 vs. time



TCC maintained NH_3 conc. below 33% of the 7-day SMAC under suit relevant operating conditions & flow rate (exceeding the target objective of half SMAC, 1.5 ppm_v).

Summary & Next Steps

- Evaluated novel functionalized nanomaterials under competitive sorption conditions:
  - Maintained all studied trace contaminants below SMAC levels for the solicitation required cycle time (≥3 minutes).
  - Demonstrated vacuum-regenerability (*without heating*).

Enables real-time, on-the-suit sorbent regeneration without additional power consumption

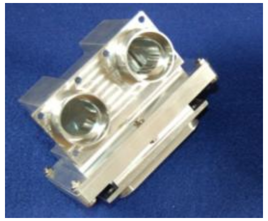
- Testing of TCC bed integrated w. RCA showed the following relevant features:
  - Compact TCC canister capable of meeting the target NH_3 removal (maintained suit conc. <50% of SMAC).
  - Ability to meet the 0.3" of H_2O requirement at 6 ACFM flow.
- The combination of Microlith support structures, durable and novel sorbent coating & an optimized housing design permits an efficient, low pressure drop, vacuum regenerable TCC bed.

Next steps:

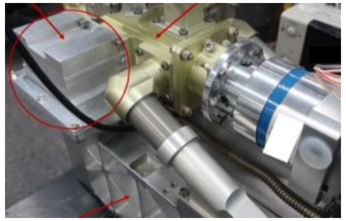
- Optimize TCC sorbent bed composition based on results from the competitive testing to achieve 5-minute protection for all contaminants.
- Perform additional cycling under competitive sorption conditions to confirm 150-hour operating life.
- Test the second-generation prototypes in a relevant environment (TRL-6; integrated with xPLSS).

Functionalized sorbent on high-surface-area support

Low-pressure drop, Microlith substrate-based TCC bed



CO_2/H_2O removal module with integrated TCC



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Contact: cjunaedi@precision-combustion.com

PCI's website: www.precision-combustion.com