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Endurance Testing of Microlith® Catalytic Oxidizer for Exploration Trace Contaminant Control

Saurabh A. Vilekar[1]
Precision Combustion, Inc., North Haven, CT, 06473, USA.

Matthew J. Kayatin[2] and Jennifer G. Williams[3]
NASA George C. Marshall Space Flight Center, Huntsville, AL, 35812

Precision Combustion, Inc. (PCI) and NASA Marshall Space Flight Center (MSFC) have been developing and testing high-temperature catalytic oxidizers (HTCO) based on PCI's patented Microlith® technology to meet the requirements of future spaceflight exploration missions. Multiple prototype units have been delivered to MSFC as part of continuous and iterative development process. These units have demonstrated benefits over competing technologies, including lower mass, lower volume, and reduced power consumption. The β-HTCO prototype was subjected to endurance testing (24,000+ hours) at MSFC to simulate catalyst ageing over a Mars transit mission duration. The end-of-life contaminant destruction efficiency was above the target metric of ≥90%. Microlith HTCO catalyst also demonstrated tolerance and recoverable performance after exposure to contaminants passed from the upstream guard bed. This feature could be utilized to monitor the health of the upstream guard bed and guide the maintenance schedule. The endurance testing, which included periodic reactor health testing, indicates that our approach results in a robust contaminant control solution for exploration missions beyond low earth orbit. Based on the β-HTCO test data, we designed a flight compatible, lightweight γ-LW HTCO that outperformed the traditional ISS HTCO unit with respect to volumetric and gravimetric density. PCI's γ-LW HTCO prototype was delivered to MSFC and is currently being integrated with other exploration-compatible trace contaminant control (TCC) components and devices. This will be followed by shakedown and life endurance testing at MSFC. This paper provides details on the test setup, test matrix, durability evaluation of the β-HTCO reactor, and future test plan for γ-LW HTCO reactor at MSFC.

Acronyms and Nomenclature

°C = degree Celsius
CH_4 = Methane
C_3F_8 = Octafluoropropane
ECLSS = Environmental Control and Life Support System
EDU = Engineering Development Unit
HTCO = High-temperature Catalytic Oxidizer
ISS = International Space Station
LW = Lightweight
LPM = Liter per minute
MSFC = Marshall Space Flight Center
OHS = Oxidation Health Sensor
ppm = Parts per million, by volume
PCI = Precision Combustion, Inc.
psia = Pound per square inch pressure, absolute

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[1] Senior Manager & Principal Scientist, PCI, 410 Sackett Point Rd., North Haven, CT 06473, USA.
[2] Senior Consultant & SME, CGI Federal, ECLSS Systems Team, Space Systems Dept., NASA MSFC, ES62.
[3] AST, Technical Mgmt./ECLSS Chemist, NASA, ECLSS Systems Team, Space Systems Dept., NASA MSFC, ES62.

SF_6 = Sulfur Hexafluoride
TCC = Trace Contaminant Control
TCCS = Trace Contaminant Control Subassembly (ISS)
VOC = Volatile organic chemical

I. Introduction

SPACECRAFT cabin air quality control is traditionally accomplished via physical and chemical adsorption of contaminants on granular adsorption media such as activated carbon or pelletized zeolites. While these methods are effective at removing a variety of semi-volatile organic compounds, they are often less effective at removing light hydrocarbons (e.g., methane), light alcohols and aldehydes, and carbon monoxide.[1] To overcome these challenges, thermal catalytic oxidation has been demonstrated and currently represents state-of-the-art in closed loop Environmental Control and Life Support System (ECLSS) Trace Contaminant Control (TCC). In this approach, a contaminated process gas stream is contacted with solid catalyst media at high temperature to oxidize various species, ideally producing carbon dioxide and water. The product gas stream may then be returned to the cabin where these less toxic reaction products are recovered in other ECLSS or Temperature and Humidity Control subsystems. This TCC process architecture has been successfully implemented within the International Space Station (ISS) Trace Contaminant Control Subassembly (TCCS) since 2001.[2] The continuous operation of TCCS onboard ISS throughout dynamic crew complements with varied visiting vehicle providers has proven the robustness and reliability of an High-temperature Catalytic Oxidizer (HTCO) for exploration TCC, as evidenced by measured trends in archival cabin air quality.[3]

Existing TCCS components are neither optimized nor right sized for contemporary contaminant loads expected over exploration mission durations.[4] Thus, the repurposing of ISS hardware is neither prudent nor sensible for TCC application within exploration missions. The current ISS TCCS Catalytic Oxidizer Assembly comprises two components; a regenerative heat exchanger assembly and a heated, fixed packed bed of pelletized catalyst.[5] Reproduction of the regenerative heat exchanger involves fabrication of a complex path, brazed-fin heat exchanger design, separated from the catalyst bed, and may no longer be an ideal or practical design solution as compared to closely coupled components. Furthermore, exact reproduction of the catalyst bed is impossible due to commercial obsolescence of the legacy pelletized catalyst media. These challenges represent not only potential points of departure from the legacy design but also opportunities for improvements in process efficiency, weight, volume, and performance. Precision Combustion, Inc. (PCI) with support from the National Aeronautics and Space Administration (NASA) has developed a close-coupled catalytic oxidizer and recuperator assembly, with demonstrated benefits of reduced weight and volume, featuring ease of manufacturability without compromising performance.[6,7] Further advances to scale the technology to provide sufficient margin to accommodate varied future mission profiles and new spacecraft designs, while transitioning the previously demonstrated hardware to a flight compatible design whilst maintaining performance, have been recently reported.[8,9] An exhaustive description of the development history and funding sources for this technology has been chronicled elsewhere.[4] In the present effort, we leverage the scaled up, flight compatible, heat recuperator integrated, HTCO recently advanced to a robust γ-lightweight (LW) flight compatible design iteration. This γ-LW design was recently incorporated into a Mars transit TCC design solution and coupled with an exploration adsorption guard-bed prototype.[10] Details of the hardware development, including lessons-learned from endurance testing of the prior generation β-HTCO design, are discussed herein. Additionally, the proposed shakedown and life endurance test plans for continued advancement of the γ-LW at MSFC are presented.

II. Life Endurance Testing, β-HTCO

Figure 1. A photograph of the β-HTCO EDU prototype.

PCI has previously designed and delivered to NASA MSFC, a β-HTCO engineering development unit (EDU) prototype (Figure 1) which served as the design basis for the γ-LW HTCO prototype. The β-HTCO was subsequently integrated within the MSFC Catalytic Oxidizer Assembly Life Test Stand (Figure 2), located within the north high-bay of MSFC Building 4755.

A. Endurance Test History

Endurance testing of β-HTCO was initiated during calendar year 2019. The test stand design allowed for continuous and near-autonomous operations of the control software via remote user access around the clock. This enabled uninterrupted endurance testing throughout scheduled federal holidays, lapses in appropriations, and the COVID-19 pandemic. Under nominal conditions, the test stand provided 25 liter per minute (LPM) of ambient laboratory air to the β-HTCO having a reactor core operating temperature of 400°C. Influent process air was pre-cleaned by a guard-bed (8 inch diameter and 18 inch packed length), packed with 52.8 lb of Cabot Norit® GCA48 (4 x 8 mesh) activated carbon. Process flow was feedback controlled using a calibrated TSI 4040 mass flow meter. The test flow setpoint was controlled in standard LPM mode until approximately 462 test days had elapsed. After this time, setpoint control was switched to operate in actual volumetric flow mode which stabilized the day-to-day measured reactor performance. The pressure drop across the reactor was monitored throughout the test duration. Periodic air monitoring in the 4755 north high-bay has shown that persistent background levels of trace acetone, ammonia, and ethanol were present. It is understood that these trace contaminants, plus any other contaminants generated by other light industrial operations (forklift operation, chemical handling, adjacent urine processing), were being introduced to the reactor as an incidental challenge load. Reactor health was benchmarked periodically by introducing 100 parts per million by volume (ppm) methane (CH_4) from a certified gas bottle via mass flow controller. This contaminant load was selected to represent a nominal molar equivalent spacecraft volatile organic compound contaminant load. The selection of CH_4 as the test species was intended to provide a simple, yet challenging thermal oxidation load from which to measure the catalyst performance over elapsed time. Using CH_4 as a challenge contaminant was also a strategic choice, setting a rigorous standard for the oxidizer's contaminant destruction efficiency because CH_4 is usually one of the more difficult contaminants to oxidize. A CH_4 destruction efficiency target of ≥90% was selected which, if achieved, should ensure the unit's ability to exceed the desired volatile organic chemical (VOC) destruction efficiency target of ≥90%. Reactor inlet and outlet CH_4 concentrations were measured using a Gasmet DX4040 Fourier-transform infrared spectrometer for multiple samples and from this data the average single-pass oxidation efficiency was calculated. Figure 3 provides the experimentally observed CH_4 oxidation efficiency for the

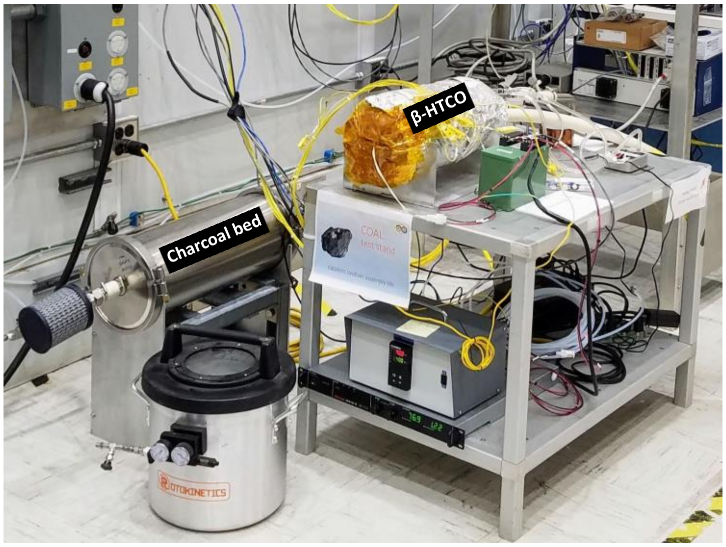


Figure 2. β-HTCO integrated within the MSFC Catalytic Oxidizer Assembly Life Test Stand.

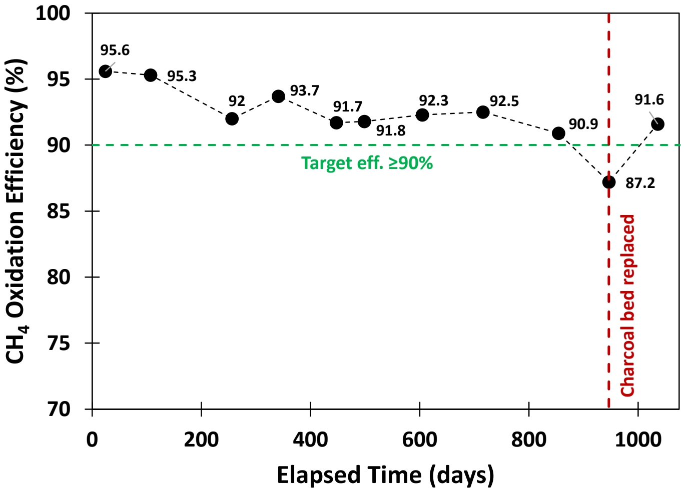


Figure 3. β-HTCO prototype oxidation performance against 100 ppm CH_4 (tested at NASA-MSFC).

β-HTCO over the complete 1,036 days (i.e., 24,867 hours) of reactor endurance life testing.

B. Endurance Test Discussion

The CH_4 oxidation performance data demonstrates the long-term thermal stability of PCI's HTCO catalyst. As shown in Figure 3, the end-of-life single-pass contaminant oxidation efficiency for CH_4 was measured to be ~92% over a simulated Mars transit test duration. From this dataset, end-of-life CH_4 oxidation efficiency was conservatively assumed to be ~90% for exploration TCC hardware design.[10] As mentioned earlier, the expected destruction efficiency for other VOCs is expected to be higher.

There were three observed subtleties within the endurance dataset that deserve more discussion herein. First, as mentioned previously, it was observed that data scatter could be reduced by operating the process mass air flow controller in actual volumetric flow mode wherein the internal temperature and pressure sensors compensate for the measured gas density in real time. This resulted in the steady observed data as recorded between 400 to 800 test days in Figure 3 wherein the average efficiency and standard deviation was stable at 92.1 ± 0.4%. Next, an apparent downward trend was observed from test day 716 to test day 854 and 946 wherein the measured CH_4 oxidation efficiency decreased from 92.5% to 90.9% and 87.2%, respectively. This was a curious trend and initially was believed to be a result of some irreversible change within the HTCO catalyst itself (i.e. ageing, fouling, sintering, or poisoning). To this end, it was decided to refresh the guard-bed by replacing the expended activated carbon with a fresh bed fill to prevent further degradation of the catalyst. Remarkably, the measured oxidation efficiency recovered to a level consistent with that observed prior to any observed performance degradation. This result indicated that measured performance was not inherent to the catalyst itself but instead related to the upstream guard-bed's end-of-life condition. This behavior was reminiscent of a phenomenon observed during testing of one of the earliest PCI reactor prototypes, i.e., the α-HTCO.

Testing of the early generation PCI reactor-core integrated with an existing ISS TCCS-like plate-fin heat recuperator was completed at MSFC in 2005.[11] One of the α-HTCO test phases involved challenging the reactor with sulfur hexafluoride (SF_6), a common dielectric gas, to simulate the impact of certain payload leaks. Overall, the injection of SF_6 had no negative impact on the measured CH_4 oxidation performance as indicated by separate pre and post-test challenges with CH_4. Remarkably though, when equimolar SF_6 and CH_4 were injected simultaneously, a 26% reduction in CH_4 efficiency was observed. Furthermore, the CH_4 performance completely recovered when SF_6 was removed from the stream. Since there was no evidence that SF_6 was degraded or oxidized, it was postulated that a reversible interaction with the catalyst surface, such as physical adsorption, was inhibiting CH_4 oxidation instead.

A separate α-HTCO test phase examined the effect of the halocarbon octafluoropropane (C_3F_8) on the catalyst. The specific concern was whether this refrigerant, ubiquitous on ISS, would poison the PCI HTCO catalyst. Prior testing with the legacy TCCS catalyst indicated that CH_4 performance loss was correlated to free-halogen partial pressure within the reactor itself.[5] This result indicates that in general, a catalyst must first be capable of degrading a halocarbon to exhibit CH_4 performance losses. While the legacy TCCS catalyst did not oxidize C_3F_8, the PCI catalyst was shown to be much more active towards this refrigerant.[11] The enhanced activity of the PCI catalyst is particularly interesting when juxtaposed against the reversible observation of the SF_6 challenge test. While it was assumed that oxidation or degradation of the halocarbon would result in poisoning, CH_4 challenge testing post-C_3F_8 injection indicated evidence of poisoning resistance within the PCI catalyst.[11] To this end, a hypothesis towards cause of the observed and recoverable performance losses within Figure 3 data is proposed.

The distribution of trace contaminants on activated carbon is dictated by complex competitive adsorption phenomenon. In this manner, compounds adsorbed within a guard-bed may be displaced and pushed deeper within the bed depth by stronger adsorbing species. This is most readily seen by observing the breakthrough waves of lighter, weakly adsorbed compounds throughout the bed service life. In fact, operating an adsorption bed on-stream too long can result in higher effluent partial pressures than one influent. This behavior is known as compound roll-off. The descriptions of such phenomena are intended to set the stage for our hypothesis, wherein the original bed fill of activated carbon became saturated with some unknown contaminants that temporarily impacted the β-HTCO CH_4 oxidation performance. In this manner, it is possible that a contaminant was rolling-off the adsorption bed with enhanced partial pressure relative to the nominal MSFC laboratory background concentration. The timely refurbishment of the guard-bed with fresh activated carbon relieved that elevated influent partial pressure condition such that reactor performance was restored. The difficulty in this hypothesis lies within identifying a clear smoking gun. High-bay trace contaminant surveys in 2021 and 2023 have only ever identified acetone, ethanol, methanol, isopropanol, propane, acetaldehyde, and ammonia as contaminants present in the facility air. While no species of concern were noted, it is possible that halocarbons were present from fugitive emissions and leaks at background levels below detection limits. These trace levels of contaminants could still be concentrated within the adsorption bed and released downstream over time, fitting our hypothesis well. In absence of quantitative data, MSFC personnel were interviewed about known events that occurred within the test facility around the time of the initial CH_4 performance loss. Notable candidate events included: a) Chiller compressor refurbishment nearby utilizing an R-410a (H-32/R-125 mixture) refrigerant, b) adjacent usage of precision cleaner containing HFO-1233zd/R-1234ze, and c) adjacent compressor loop with possible fugitive R134a emissions (not proven to be leaking with certainty, however). The potential release of halocarbon containing refrigerants, capture on the test stand guard-bed, and subsequent saturation or release of the unknown contaminant to the β-HTCO is a plausible explanation for the recoverable CH_4 performance as observed within the present and historical testing at MSFC.

C. In-situ Process Monitoring

Based on demonstrated performance impacts to the PCI catalyst from the presence of certain compounds, it would be advantageous to determine or anticipate end-of-life for the upstream guard-bed so that compounds normally controlled by the HTCO do not increase in concentration to an extent that air quality is negatively impacted. This could be achieved by relying on real-time onboard instrumentation or process air sensors. While both approaches provide insight into the cabin atmosphere, the selection of a monitoring strategy may depend on the mission or vehicle design with unforeseen complexity added to the ECLSS operations concept.

Historically, ISS air quality has been trended via Crew Health Care System's Air Quality Monitor and more recently by the Analyzing Interferometer for Ambient Air managed by the European Space Agency.[3] Based on the air quality dataset, performance of the guard-bed may be inferred based on cabin-level mass balances.[12] Alternatively, information may be fed as a time-resolved dataset to a validated digital twin[13] or dynamic model[14] from which lifetime projections may be made. While it is assumed that some air quality monitor(s) will be implemented for exploration, there is no guarantee that Crew Health and Performance hardware will be ground-ruled for flight operation's decision making. Furthermore, analysis time is needed to fully interpret and understand the implication from this dataset.

In-line process monitoring of the HTCO provides possibly the most direct approach to process health. While we cannot determine the permitted use-cases for the Crew Health monitors, utilization of integrated air sensors can be advantageous to independently monitor TCC System performance. In this implementation, the HTCO inlet and/or outlet may be monitored to look for specific contaminants or trends in some measurement to correlate with oxidation performance. This approach is conceptually analogous to that of the ISS Water Processor's Reactor Health Sensor.[15] The selection of the HTCO Oxidation Health Sensor (OHS) is difficult without knowledge of the vehicle thermal working fluids and potential payloads with myriad emissions that may need to be interpreted by the OHS. As such, the most practical approach may be to baseline the design with non-speciated organic vapor sensors such as a photoionization[16] or metal oxide-based sensors[17] capable of tracking refrigerants. Selection of a sensor type would also be dependent on whether direct reactor performance for a tracer species such as CH_4 is desired or practical. Existing fenceline emission monitoring programs for the chemical and manufacturing industries may also be utilized for insight into non-specific sensor utilization strategies herein.[18] The optimal sensor strategy may be mission dependent or require modification to mitigate radiation susceptibility and is therefore beyond the scope of this study.

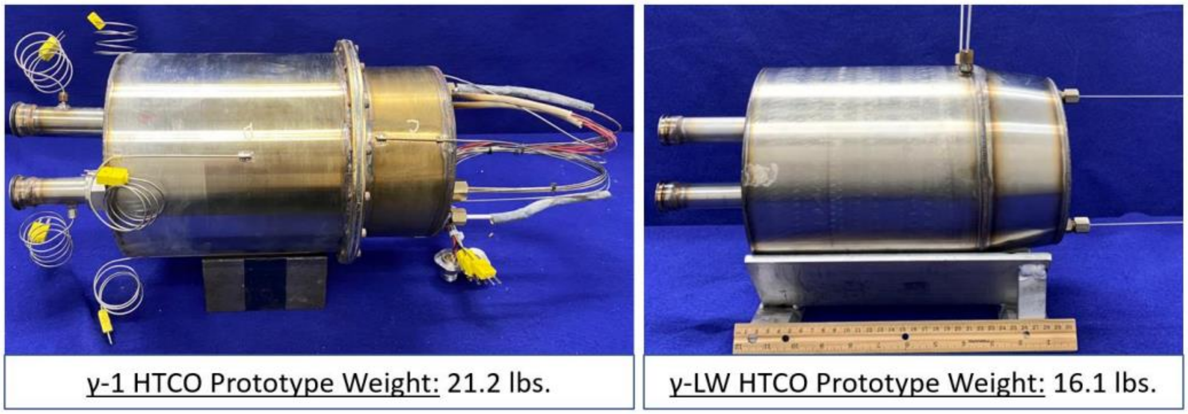
III. Proposed Testing, γ-LW HTCO

The introduction of flight-compatible interfaces and sensors brings new potential failure modes to the reactor design that must be understood by shakedown and endurance testing.

A. Launch Vibration Testing

Shakedown testing of the α-HTCO reactor core included random vibration launch load survivability at 3.1-g root-mean-square as was specified for TCCS launch and landing at the time of development.[19] Both visual inspection and functional performance testing were utilized to check the α-HTCO post-vibration. MSFC proposes to subject the γ-LW HTCO prototype (Figure 4) to random vibration testing with mapping of the thermal, electrical, hydraulic, and oxidation performance pre and post vibration. The selection of test acceleration loads must be carefully considered and tailored to the anticipated launch vehicles utilized by the programs in consideration. This requires further internal review with NASA personnel, in particular for various program-dependent interface requirements or definition documentation. In lieu of specific programmatic requirements, the γ-LW HTCO will benefit from being vibration tested with a variety of exploration loads to simulate worst-case scenarios and improve flight readiness.

Unlike vibration testing with the α-HTCO where the reactor core could be separated from the heat recuperator and removed for direct mounting on the test fixtures, the γ-LW prototype cannot be non-destructively disassembled. During the γ-HTCO development process, NASA executed a contract option to streamline the 1st iteration γ-1 HTCO prototype by removing superfluous instrumentation and interfaces not necessary for flight operations. A side-by-side comparison between these two prototypes is shown by Figure 4. Additional thermocouples and instrumentation fittings, used for extensive benchmarking of the γ-1 HTCO, were eliminated in the γ-LW HTCO design. The γ-1 HTCO comprises a mated flange, allowing for the capability to disassemble the prototype (i.e., decouple the integrated heat recuperator and the HTCO) for evaluation and reassembly to facilitate development and performance testing efforts. Based on β-HTCO testing results, demonstrating long duration operation with ≥90% destruction efficiency, it was determined that a hermetically sealed and welded construction could be implemented within the γ-LW HTCO, as disassembly over the life of the hardware is not anticipated. This design change allowed for elimination of the flanges, gasket, and sealing hardware used to connect the recuperator to the HTCO housing assembly, as well as a 1 inch compression fitting on the exhaust process gas tubing. The recuperator housing was extended to support a change in the resistance temperature detector fitting location to improve sensor robustness, and the HTCO housing was reshaped for additional weight savings. The design change to a welded, flangeless housing will require new approaches to inspect the test article such as non-destructive evaluation. For example, computer-assisted tomography scanning may need to be utilized to diagnose any potential failure modes or identify any observable changes internal to the device such that the operability of the reactor can be preserved.


γ-1 HTCO Prototype Weight: 21.2 lbs.

γ-LW HTCO Prototype Weight: 16.1 lbs.

Figure 4. Side-by-side comparison of γ-1 and γ-LW HTCO prototype assemblies without insulation.

B. System Fluid Compatibility

The chemical compatibility between TCC equipment design and vehicle system-level working fluids, such as internal active thermal control system coolant loops, is often neglected until later within the design review cycle process wherein the vehicle design matures enough to make selections on loop volume and chemistry. As discussed previously for halocarbons and dielectrics, the selection of an internal vehicular working fluid can have tangible impacts on the TCC equipment lifetime and cabin air quality. The impact of both loss of containment and continuous fugitive background emissions to the cabin should be considered when mission planning for spares. To this end, we propose conducting a survey of common working fluids currently specified to various programs and vehicles to look for commonality and emerging trends in fluid chemistry. From this assessment, we may plan to screen HTCO reactor CH_4 oxidation performance in presence of these fluids or similar representative chemical classes to better understand the potential impacts to hardware life and air quality. Identification of potential partial oxidation and thermal degradation byproducts may also be necessary to understand associated risks for exploration.

C. Exploration Pressure Testing

Exploration vehicles and habitats are planning for reduced pressure operating environments to facilitate an increased frequency of extravehicular activity. By operating the cabin at absolute pressures below 1 atm, complex and time-consuming prebreathe protocols, typically required to prevent decompression sickness, can be simplified. An independent assessment of the impacts of operating trace contaminant control equipment in reduced pressure atmospheres was conducted by the NASA Engineering and Safety Center.[20] It was determined that both heat and mass transfer were impacted by reduced pressures such that mass transfer phenomena are enhanced while heat transfer is inhibited. To this end, we propose mapping of the thermal, hydraulic, and oxidation performance of the γ-LW HTCO at both 8.2 and 10.2 psia by utilizing the MSFC Variable Atmosphere Test Facility. By comparison against the existing datasets collected at 1 atm (14.7 psia), the impact of reduced pressure operations on time-transient periods can be understood. This will further our understanding on operational impacts while informing and guiding future design considerations.

IV. Conclusion

Precision Combustion, Inc. (PCI) and NASA Marshall Space Flight Center (MSFC) have shared a long history in developing and testing high-temperature catalytic oxidizers, based on PCI's patented Microlith® technology to meet the challenging requirements of exploration space missions. Recently, the β-HTCO EDU prototype completed long-duration endurance testing (24,000+ hours) at MSFC to simulate catalyst ageing over a Mars transit mission duration. PCI's Microlith HTCO catalyst demonstrated tolerance and recoverable methane oxidation performance after exposure to contaminants believed to have been concentrated within the upstream activated carbon guard-bed. Remarkably, the end-of-life contaminant destruction efficiency was above the target metric of ≥90%. The endurance testing outcomes indicate that our approach results in a robust contaminant control solution for exploration missions beyond low earth orbit. Based on the β-HTCO test data, we designed a flight compatible, lightweight γ-LW HTCO that outperformed the traditional ISS HTCO unit with respect to volumetric and gravimetric density.[9] Recommended shakedown testing, including vibration, chemical compatibility, and environmental test objectives, were discussed for proposed detailed test objectives to continue maturation of the technology towards exploration mission application.

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