Development of the Miniature Total Organic Carbon Analyzer

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Monitoring the Total Organic Carbon (TOC) in spacecraft potable water will be of major importance in long-duration human space exploration. In-flight analysis of potable water produced from a regenerative water processor provides immediate feedback on the quality of reclaimed water for crew health as well as water processing system health monitoring. This paper updates the progress in development of the next generation Total Organic Carbon Analyzer (TOCA) designed for the unique requirements of an exploration-class mission. The current objective is to design, build, certify, deliver, and operate a TOCA technology demonstration on the International Space Station (ISS). The next generation analyzer system technology was previously developed and selected among a feasibility study of other options. The new system provides primary advantages of reduced mass and volume through reduced system complexity and reduced need for consumables; therefore, the flight project is named MiniTOCA. The project has recently completed design of the tech demo instrument and assembled and tested a flight-like engineering development unit. The engineering unit has undergone performance testing and environmental testing which provides confidence for the project to move forward with flight unit production and certification activities. Test results are summarized in this paper. The flight unit is targeted for delivery to ISS in late 2025.

Nomenclature

ANCL	=	*Acoustics and Noise Control Laboratory*
CDR	=	*Critical Design Review*
CHeCS	=	*Crew Health Care System*
COTS	=	*Commercial off-the-shelf*
EDU	=	*Engineering Development Unit*
EMI/EMC	=	*Electromagnetic interference / electromagnetic compatability*
EMU	=	*Extravehicular mobility unit*
IRD	=	*interface requirements document*
ISS	=	*international space station*
KHP	=	*potassium hydrogen phthalate*
MCC	=	*mission control center*
MCO	=	*Mars Campaign Office*
MTLS	=	*miniature tunable laser spectrometer*
MiniTOCA	=	*Miniature Total Organic Carbon Analyzer*
PDR	=	*Preliminary Design Review*
PIRN	=	*Preliminary Interface Revision Notice*
PRVD	=	*Product Requirements and verification document*

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RSD	=	*relative standard deviation*
SDL	=	*System Development Laboratory*
SRR	=	*System Requirements Review*
SSP	=	*Space Station Program*
TIC	=	*total inorganic carbon*
TLS	=	*tunable laser spectrometer*
TOC	=	*total organic carbon*
TOCA	=	*Total Organic Carbon Analyzer*
UV	=	*ultraviolet*

I. Introduction

Monitoring the concentration of total organic carbon (TOC) in spacecraft employing regenerative potable water systems is a baseline requirement. TOC provides broad-spectrum analysis of product water quality to assess the potential toxicity of the water for crew health and the performance of the water processor system. While the International Space Station (ISS) successfully employs a Total Organic Carbon Analyzer (TOCA) to complete these tasks, the core technology does not lend itself to the mass, volume and consumable savings considered sufficient to support long-duration missions.[1] This paper updates the progress in development of the next generation TOCA designed for the unique requirements of an exploration-class mission, taking place as the Miniature Total Organic Carbon Analyzer (MiniTOCA) project. The current objective is to design, build, certify, deliver, and operate the MiniTOCA technology demonstration on the International Space Station (ISS) as part of demonstrating that the miniaturized implementation meets technology and performance goals for future exploration missions.

II. Total Organic Carbon for Exploration, EVA, and beyond

The long-term goal is to develop a small potable water quality monitor suitable for long-duration missions that can be integrated into the water processing system. The requirements levied on the hardware utilize a novel approach to system architecture to provide reliable data with minimal crew interaction. The functional objective of the hardware is to determine recovered water quality as a function of TOC by measuring carbon dioxide (CO_2) levels produced during oxidation of contaminants from a sample of water. Hardware development has a multitude of challenges that include producing hardware that not only functions with lack of gravity but must adhere to numerous safety requirements that potentially drive design. Thus, careful evaluation of available technologies was completed early in the project and breadboard architectures were developed and tested to provide the final design for the MiniTOCA.

Table 1. Requirements and characteristics of the MiniTOCA technology demonstration instrument.

Mass / Power / Volume & Environment Requirements		MiniTOCA Notes
Volume	<0.03 m^3	0.03 m^3
Mass	<20 kg	20 kg
Power	<175W average	<60W, average 45W as tested with EDU
Microgravity Performance	Not sensitive	To be tested on ISS
Ambient CO_2 compatability	Up to 0.52% CO_2	Tested to 3.9 mmHg ppCO_2 with EDU
Sampling Interface	Bag samples & interface to potable water system	Samples bags & continuous connection to potable water
Sample Compatibility Requirements		
Gas content	Up to 5% free gas in water	Uses a degasser
pH	4.5-9	Does not require buffer chemicals
Silver biocide	Up to 0.4 mg/L	Not used in US segment
Iodine biocide	Total iodine up to 4 mg/L	Verified through EDU performance testing
Total inorganic carbon (TIC) interference	Up to 2.5 mg/L TIC as dissolved CO_2	Verified through EDU performance testing
Performance Requirements		
TOC Analysis Range	0.6 ppm – 10 ppm TOC	Verified through EDU performance testing
TOC Accuracy	+/- 25% over TOC range	Verified through EDU performance testing
TOC Precision	+/- 25% RSD for a series of 3 replicates, over TOC range	Verified through EDU performance testing

A. Requirements Overview

Analytical requirements for TOC analysis in exploration missions draw heavily from the existing success of ISS missions and lessons-learned. The Crew Health Care System (CHeCS) Specification, SSP 50470 documents high-level requirements for in-flight TOC analysis. One key difference is the assumption that exploration TOC analysis will only be needed for recycled water rather than ground-supplied or Russian-supplied water. This is an advantage because recycled water is more pure. A lesson learned is that while crew health drives the criticality of water quality monitoring, other systems that utilize potable water are invested stakeholders in water quality data. The sublimator in the extravehicular mobility unit (EMU), for example, has shown sensitivity to organic chemicals.[2] The primary system stakeholder is the water processing system who must utilize TOC data to monitor changes in system performance and plan maintenance activities. Other users of potable water are the oxygen generation system and EMU. On ISS, these systems require TOC purity below the crew consumption limit of 5 mg/L. The exploration TOC system is requiring TOC detection as low as 0.6 mg/L in order to ensure water purity for extravehicular suits as well as providing early trending for other users. In summary, an exploration TOC analyzer is expected to provide TOC quantification in the range of 0.6 to 10 mg/L TOC.

B. System Challenges

Successful TOC analysis requires a system of mechanical, electrical, and control processes interacting to control the multi-phased chemical/analytical process. Developing this system presents multiple challenges. For spaceflight applications the obvious challenge is lack of gravity. This is significant for TOC analysis because nearly all terrestrial TOC systems utilize gravity for gas/liquid separation either in sample handling or for gases generated during sample oxidation. Other challenges are chemical interferents such as CO_2 and iodine. Since TOC analysis is performed by measurement of CO_2 it is critical to distinguish between CO_2 initially present in the water and the CO_2 generated from oxidation of organic chemicals. Trade study development of TOC oxidation techniques revealed that iodine interferes with oxidation efficiency because iodine is oxidized at the same time as organics, thus reducing the oxidation potential of the system. A successful system must produce excess oxidation radicals.

Design of a system for flight safety presents additional challenges. A primary challenge is to ensure that the system is designed to prevent water leakage when integrated with the vehicle potable water system and assuming two worst-case failures (two fault tolerant). This level of redundancy for safety imposes additional pressure controls that must not impair the analytical system. Another safety risk is hazardous chemicals. Most commercial TOC analyzers utilize acids for pH control and oxidizers for oxidation, but the commonly used chemicals require triple containment for spaceflight application which greatly increases complexity and lowers reliability of the instrument. It is preferred to design a system without the use of chemical reagents. Additionally, chemical additives have limited shelf-life and must be resupplied which is not suitable for long duration missions like Mars transit. For these reasons, an exploration TOC analyzer should not utilize consumable chemicals.

C. Exploration TOC System Selection

Selection of the TOCA system that best fits an exploration mission was conducted by narrowing a field of candidates. Based on prior research and development, the most favorable technologies were organized into appropriate system architectures and developed into breadboard systems.[3] Development of the breadboard units allowed demonstration of the integrated technologies, development of methods and concepts of operation, and the collection of system-level performance data. Following breadboard configuration and development work, the systems were

Table 2. Selected candidate architectures for an Exploration-Class TOCA, with the architecture for MiniTOCA in green. Breadboards were built for each of these and tested as part of the early work to select technologies for this next generation TOC instrument.

Technology Architecture Options for Sample Oxidation & TOC Determination			
Oxidation	Phase Separation	Carrier	Detection
Ultraviolet (UV) w/ recirculation + electrolyzer	Membrane	Deionized Water	Conductivity
UV w/ recirculation + electrolyzer	Membrane	Nitrogen Gas	Tunable Laser Spectroscopy (TLS)
UV w/ recirculation + electrolyzer	Membrane	Water Vapor	TLS
Catalytic Combustion	Evaporation on hot catalyst	CO2-free air / gas	“Hot” TLS

assessed for performance using known TOC standards. In total, nine breadboard architectures were conceived, built, and tested. Of the nine technologies, four systems were deemed to meet requirements and/or have sufficient data to be included in the subsequent trade evaluation. These systems are described in Table 2, and details of the technology downselect process is summarized in the 2020 ICES paper[3]. A technology downselect meeting with project stakeholders occurred in February of 2020. Overall, the stakeholder review team agreed multiple candidate system architectures were capable of meeting the proposed requirements for an exploration MiniTOCA system and the trade space between the technologies were considered extremely tight. The breadboard system architecture selected was UV oxidation with membrane transfer via nitrogen sweep gas with detection of CO_2 using a tunable laser spectrometer (TLS). Selection was based on the system's small sample volume, small overall size/mass, zero consumables, projected long-life, and reliable analytical performance.

D. MiniTOCA System Description

UV oxidation with recirculation followed by detection of evolved CO_2 using TLS was selected as the core technology for MiniTOCA. The simplified MiniTOCA system diagram is shown in Figure 1. Water is introduced into the analyzer *via* one of two paths. Sample bags can be connected to the instrument by Luer lock connection, and the water in the bag analyzed. This is useful for any grab sampling or analysis of stored water. MiniTOCA is also connected directly to the ISS potable water bus, and could sample the potable water at any time without disturbing the potable water system. The instrument requires an interface to vehicle nitrogen for use as the sweep gas during the gas / liquid separation step, prior to detection of CO_2 that was generated from the oxidation of the trace organic compounds present in the water.

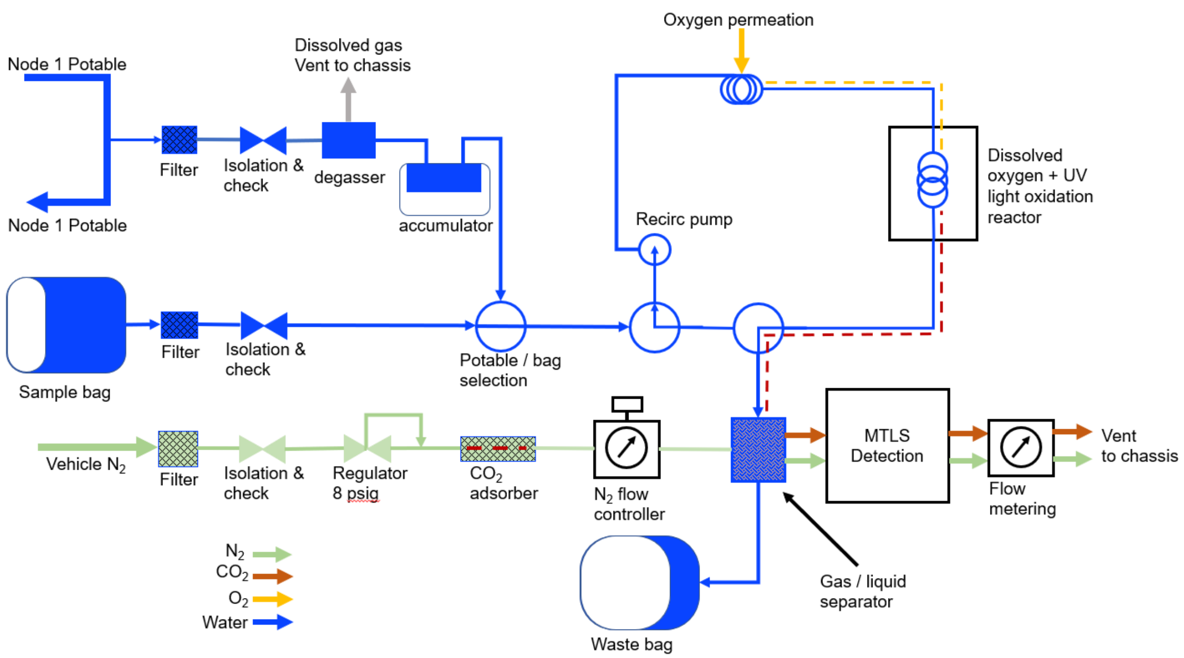

Figure 1. Simplified schematic of the MiniTOCA instrument. Water can be sampled from two different sources. The instrument is plumbed to the potable water bus or can accept bag samples. Oxidation of organics present in the water is achieved by activating the dissolved oxygen in the water with UV light which results in oxidation of the organic chemicals into carbon dioxide. The carbon dioxide is measured by the MTLS by sweeping it out of the water in the gas / liquid separator.

III. Flight Technology Demonstration Path

In October 2020, the Mars Campaign Office (MCO) kicked off the MiniTOCA project to develop a flight demonstration unit for operation on ISS. The project developed requirements and held the System Requirements Review (SRR) in January 2021. Some of the requirements were modified in this review and the project adapted to the revisions. The requirements for the flight project are documented in the Project Requirements and Verification Document (PRVD), JSC-67571. The project Preliminary Design Review (PDR) was held in April through June 2022. With successful PDR, the project was authorized to proceed with detailed design. A major undertaking which facilitated development of the detailed design was to design, build, and test a high fidelity, flight-like EDU. An EDU greatly reduces risk to the project certification phase by demonstrating the capability to fabricate, assemble, and test the flight design. The EDU was fully assembled in March 2023 and then entered a phase of first-time checkouts, modifications, and tuning. The system progressed with increasing levels of functional performance until the formal EDU testing program began. The project chose to subject the EDU to an engineering evaluation comprising the full suite of tests that would normally be performed for certification of the flight hardware, including vibration and EMI/EMC testing. At the Critical Design Review (CDR) in November of 2023, the detailed design of MiniTOCA was presented along with forward plans and schedules for manufacturing and assembly. Upon the successful completion of CDR the MiniTOCA project was approved to begin plans for manufacturing and is currently in the flight hardware build phase of the project.

IV. Engineering Development Unit Testing Results

Building a flight-like EDU provided the MiniTOCA project an opportunity to perform environmental testing of the hardware prior to building the flight hardware. This activity was focused on risk reduction and provided the project an opportunity to make necessary updates to the hardware design if necessary, prior to this being a concern or schedule driver *after* the flight hardware was assembled due to issues discovered during certification. There was also an opportunity to make corrections to or streamline the assembly process and certification testing plans & procedures of the flight hardware. Overall, the cost and schedule invested for buildup and testing of the high fidelity EDU results in confidence in the flight design and contributed to the successful CDR. The project does not expect any significant challenges in testing and certification of the flight hardware due to favorable results from EDU testing. Photos of the MiniTOCA EDU are show in Figure 2.

The EDU was subjected to a nearly complete environmental testing program that is analogous to what would be performed to certify the flight hardware. A list of tests performed on the EDU hardware, as well as brief information related to results and anything to be addressed for the flight build, is shown in Table 3. Further key details for each of the tests are given in sections following. Other testing, including proof and leak testing, and grounding and bonding testing, was performed as required to verify system safety and compliance with requirements in terms of preventing water leaks, and verifying design for hardware grounding, bonding, and isolation requirements. In one instance, the

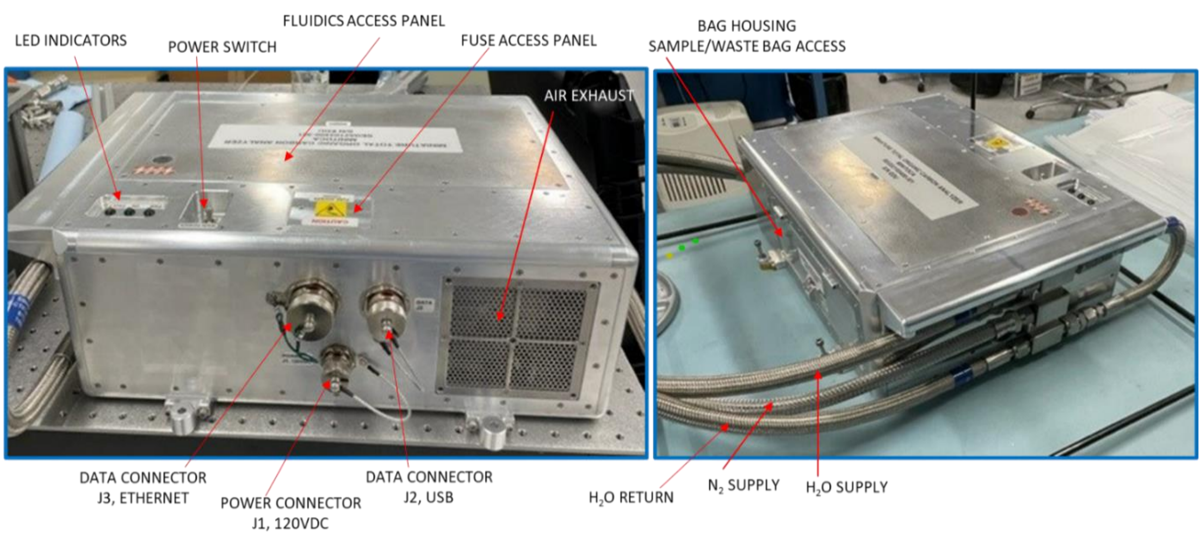

Figure 2. (Left) Side view of the MiniTOCA EDU, showing connectors and cooling air exhaust area. The MiniTOCA EDU is a fully functional instrument built to form / fit / function with respect to the flight hardware requirements. (Right) View of potable water hoses and nitrogen supply hose. The instrument interfaces to the vehicle with hoses to the potable water bus, which is connected in a flow-through arrangement with pressure drop requirements so that there is no disturbance in operation of the potable water system.

EDU evaluation resulted in identifying a location where the electrical bonding arrangement required a change. This was an opportunity to make this change in the EDU and verify the updated design prior to this becoming and issue and discrepancy during flight hardware certification.

A. Engineering evaluation of the EDU through environmental test

This section details the environmental testing performed for the MiniTOCA EDU in advance of the Critical Design Review (CDR) and the subsequent build of the flight unit to evaluate and validate that the current design can comply with the test requirements of JSC-67571, Project Requirements and Verification Document for the MiniTOCA.

Random Vibration Test: MiniTOCA random vibration testing was performed in the JSC B13 Structures Test Laboratory (STL). The MiniTOCA was tested in 3 orthogonal axes to vibration spectra taken from the latest Preliminary Interface Revision Notice (PIRN) to the Common IRD (50835-IRD-PIRN-0033, 3.1.1.2.1.2.2). The overall objective of this test was to demonstrate the ability of the MiniTOCA EDU to withstand the stresses and accumulated fatigue damage resulting from the maximum random vibration environment. The test is also intended to detect material and workmanship defects. The MiniTOCA EDU was powered on and monitored during each run and functioned as anticipated real-time. Post-axis inspections and abbreviated functionals were completed to determine the health of the unit under test, during testing. The vibration qualification test was successfully completed, and the target Grms in all three (3) axes were achieved.

EMI/EMC Test: MiniTOCA EMI/EMC testing was performed in the B14A JSC EMI Test Facility (JETF). The MiniTOCA was tested per SSP 30237 rev T and SSP 57000 rev T, performing tests for Conducted Emissions, Conducted Susceptibility Radiated Emissions, Radiated Susceptibility, and Static Magnetic fields. The MiniTOCA was found to be in compliance with all the tests and also functioned nominally before and after testing, providing confidence in the electrical design.

Acoustic Emissions Test: MiniTOCA acoustic emissions testing was performed in the B241 Acoustics and Noise Control Laboratory (ANCL). During this evaluation, the ANCL collected sound pressure level data produced by the MiniTOCA during all unique, noise-producing modes of operation that are representative of nominal, on-orbit operating modes to demonstrate the ability of the MiniTOCA EDU to operate in compliance with noise limits per SSP 50835. The test was completed successfully and met all test objectives for intermittent noise requirements. The MiniTOCA EDU exceeded continuous noise limit requirements during this test, but the exceedance was very slight. Given that noise from fans can vary from unit to unit, the project is moving forward with the design as-is and documenting an acoustic exceedance as a project risk to track.

Power Quality Test: MiniTOCA power quality testing was performed in JSC B361. During this evaluation, the JSC Power Quality Facility introduced all the possible ISS provided normal and abnormal power quality conditions specified in SSP 52051, ISS User Electric Power Specifications and Standards, Volume 1. All required test runs completed successfully and met all test objectives with no failures (Pass). The MiniTOCA EDU was powered on and monitored during each run and functioned as anticipated. Post-test inspections were performed and no physical damage of the unit under test was observed during testing. The MiniTOCA EDU completed a post-test functional test at KBR and functioned nominally.

Table 3. Engineering evaluation tests performed on the EDU and notes regarding results.

Test	**Results of EDU Engineering Evaluation**	**Notes related to flight build**
Random vibration	Pass	Identified some COTS assemblies that need fasteners staked
EMI / EMC	Pass	Test identified some required schematic changes to power board
Acoustic Emissions	Intermittent: Pass Continuous: Fail	Test identified continuous noise due to cooling fan is a concern, but magnitude of exceedance plus variability in fans is considered in forward risk assessment
Power Quality	Pass	None
Arcturus	Pass	None
Thermal Testing	Pass	None; thermal cycling not performed on EDU but thermal testing shows that experiment agrees with modeling and that component temperatures do not exceed limits, with margin.

Arcturus Testing: The Arcturus data system was connected to the MiniTOCA via the MiniTOCA USB data cable. A software update was transferred to MiniTOCA via the Arcturus system. Following installation of the software update, a sample analysis was commanded from MCC via the Arcturus system. This sample analysis used a sample bag and waste bag for water management. Following analysis completion, the MiniTOCA transferred the data via the Arcturus system to the MCC. Primary objective was to verify that MiniTOCA was capable of interfacing with the Arcturus system by transferring data between MiniTOCA and MCC. The secondary objective was to determine any software changes that may be required for flight unit build. The test was completed successfully and met all test objectives. MiniTOCA control oftware was successfully able to send and receive data through the Arcturus network. This included an analysis transfer and retrieve file test as well as an update configuration and software test. Post-test inspections were performed to determine functionality and no physical damage to the unit under test during testing was observed.

Burn-in Test: The purpose of the burn-in test is to detect material and workmanship defects which can result in early component failure. This test is usually performed at the end of the Certification test series. Only the remainder of the 300 hours that were not completed during Certification testing will be completed in this test. The MiniTOCA EDU Test Log indicated that the EDU has well over the 300 hours of run time needed to fulfill this requirement and therefore no further burn-in testing was required.

B. EDU Performance and Functional Testing Results

Performance and functional testing of the MiniTOCA EDU was conducted in the KBR System Development Laboratory (SDL) to verify that the MiniTOCA is fully functional and meets the specified operational requirements. The test results are summarized in Table 4. The ambient environment in which the instrument operated was controlled, and the MiniTOCA EDU was placed in an atmospheric chamber containing up to 3.9mmHg partial pressure CO_2 ($ppCO_2$) to mimic the maximum elevated CO_2 levels that might ever be encountered while operating on ISS, although typical practice is to maintain ambient CO_2 levels below 3.0 mmHg.

The purpose of this performance testing campaign is to verify the performance and functional requirements of the MiniTOCA using a 7-point analytical matrix as well as verifying the calibration stability. Calibration stability is

Table 4. Performance testing results obtained from the MiniTOCA EDU. Tests were performed over the requirements range for TOC and TIC, and tests were performed with oxidation challenges and in the presence of biocide (iodine). The results obtained from the EDU validate the architecture, technology and flight design planned for the technology demonstration on ISS. Requirements for both accuracy and precision are +/-25% across the requirement range of 0.6-10 mg/L TOC.

Test	Sample TOC (mg/L)	Sample TIC (mg/L)	Constituents	Test Purpose	Date Completed	EDU Accuracy (% Error)	EDU Precision (%RSD)
1	10	<0.5	TOC as KHP	Max TOC Range	8/24/23	-1%	+6%
					8/29/23	0%	4%
					9/6/23	2%	-7%
2	0.6	<0.5	TOC as KHP	High/Low Carryover, Minimum range	8/25/23	-27%	+22%
					8/30/23	+22%	+11%
					9/7/23	-4%	+11%
3	0.6	2.5	TOC as KHP	Minimum requirement range with max TIC	10/10/23	+17%	+12%
					10/12/23	+21%	+12%
4	1	2.5	TOC as KHP	Minimum CHeCS TOC range with max TIC	10/9/23	+9%	+5%
5	5	<0.5	TOC as KHP	Flight rule health limit TOC	8/17/23	+4%	+5%
					9/7/23	0%	+3%
					10/12/23	-10%	+4%
6	3	<0.5	TOC as Ethanol	Calibration check TOC with ethanol + iodine	8/30/23	+21%	+5%
					9/5/23	+12%	+11%
7	10	<0.5	TOC as Ethanol	Max TOC range with ethanol and iodine oxidation challenge	8/25/23	0%	+8%
8	0.3	<0.5	TOC as KHP	Engineering evaluation at 0.3	8/29/23	+8%	+6%
					10/13/23	-19%	+6%

evaluated by how long the instrument holds calibration after initial calibration – the need to recalibrate or not. Calibration conditions were found to be significant with respect to accuracy. The MiniTOCA EDU was initially calibrated at ambient CO_2 conditions of approximately 500 ppm CO_2, which led to some results on water samples reporting high during performance testing. The instrument was re-calibrated in a CO_2 environment more representative of what is expected on ISS and closer to the high test case of 3.9 mmHg CO_2, and after re-calibration the reported results are accurate. Calibration stability results also show that the MiniTOCA EDU calibration was stable over 4 months and 16 samples.

Each MiniTOCA measurement is an average of three successive determinations of the TOC value of a sample (3 replicates). The percent relative standard deviation (%RSD) is examined for the three determined values, and must be less than 25% for the average of 3 replicates. This is a measure of the stability and repeatability of the instrument and the values it reports. Accuracy is determined relative to a certified value obtained for the sample being tested. Accuracy is calculated by determining the difference in the single reported value (average of 3 replicates) versus the true TOC value of the sample. Samples were sent to the Toxicology and Environmental Chemistry Laboratory at JSC to obtain the true TOC value of the sample. MiniTOCA shall report the value of TOC for a sample to within +/- 25% of the sample's true TOC value.

With respect to the results reported in Table 4, all tests showed MiniTOCA meets requirements except for one run of carry-over testing. Sample 1, followed by Sample 2, is a carry-over test in that a low concentration sample is measured right after a high concentration sample. This test is challenging, because the first replicate of Sample 2 following the last replicate of Sample 1 may be biased if there is any carry-over from one sample to the next. After 2 retests, we were confident that the MiniTOCA design will meet carry-over requirements. Other tests identified as "oxidation challenge" use ethanol instead of KHP to verify that the system effectively oxidizes a compound which is known to be less susceptible to UV light oxidation. Addition of iodine additionally "challenges" the instrument because iodine is photochemically active, but the EDU results suggest the system has a robust oxidation system capable of performing under various conditions.

Additional functional testing included verifying the performance of the MiniTOCA at the high and low operational water bus pressures, using the potable water bus sampling loop and accumulator.

V. Conclusion

The MiniTOCA project has successfully transitioned from technology development effort to flight technology demonstration project. We have demonstrated that the selected architecture for a miniature TOC analyzer based on UV oxidation and tunable laser spectrometry detection of the produced CO_2 is both feasible and an excellent performer. The choices of technologies lend themselves to miniaturization, minimize the use of consumables, and overall are forward-leaning for exploration applications. The planned technology demonstration on ISS will validate the implementation and provide operational experience and testing results that will be useful to Exploration.

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