

Second Generation International Space Station (ISS) Total Organic Carbon Analyzer (TOCA) Verification Testing and On-Orbit Performance Results

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The Total Organic Carbon Analyzer (TOCA) is designed to autonomously determine recovered water quality as a function of TOC. The current TOCA has been on the International Space Station since November 2008. Functional checkout and operations revealed complex operating considerations. Specifically, failure of the hydrogen catalyst resulted in the development of an innovative oxidation analysis method. This method reduces the activation time and limits the hydrogen produced during analysis, while retaining the ability to indicate TOC concentrations within 25% accuracy. Subsequent testing and comparison to archived samples returned from the Station and tested on the ground yield high confidence in this method, and in the quality of the recovered water.

Nomenclature

μg = micrograms

I. Introduction

In support of a crew of six onboard the International Space Station (ISS), wastewater and urine is reclaimed and reprocessed into potable water. The ISS Regenerative Water Processing Assembly (WPA) purifies wastewater into potable water for consumption and oxygen generation on ISS. The Total Organic Carbon Analyzer (TOCA) is supporting hardware that takes samples of WPA water and analyzes for the presence of Total Organic Carbon (TOC). Measuring TOC in the water provides a general indication of overall water quality by indicating the potential presence of hazardous organic load. Low TOC indicates that the water processor is functioning properly. The WPA also contains conductivity sensors to monitor water quality and directs re-processing if water quality parameters are not met. These conductivity sensors are the first and second controls for water quality on ISS, and TOCA serves as the third control to out of specification potable water for ISS. The TOCA was designed to be a stand-alone, redeployable piece of hardware that performs off-line sampling of the regenerated potable water from the WPA to verify water quality on ISS.

To accomplish the required functions, TOCA is located close to the Water Recovery System (WRS) rack, to enable direct sampling from the WPA. TOCA can also receive samples via a sample bag, which is normally filled from the Potable Water Dispenser (PWD) on-orbit. Several principles of TOC analysis are listed below:

- Total Carbon (TC) = Total Inorganic carbon (TIC) + Total Organic Carbon (TOC)
- TIC interferes with the direct measurement of TOC and must be removed prior to measuring TOC

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- TOCA converts and removes TIC by creating a pH shift with a slightly acidic buffer which forces inorganic carbon species into carbon dioxide (CO₂) gas
- TOCA oxidizes organic carbon species remaining in the sample water to CO₂ gas
- TOCA produced CO₂ gas is measured using a very precise Infra-red CO₂ detector
- General process steps for the on-orbit instrument include:
 1. Flush and purge liquid and gas loops
 2. pH shift and convert and remove TIC
 3. Detect CO₂ from TIC
 4. Oxidize TOC
 5. Detect CO₂ from TOC
 6. Analyze and report

Terrestrially, TOC analyzers employ various methods to determine TOC. Most notably, ground based units involve wet chemistry techniques that use hazardous reagents or high temperature combustion. The first generation¹ ISS TOC analyzer employed phosphoric acid (H₃PO₄) and ammonium persulfate ((NH₄)₂S₂O₈), both designated as toxicity level 2 chemicals. The reagents resulted in design complexity and required two verifiable levels of containment. The lessons learned from the first generation TOCA resulted in the ISS Program requesting the new unit not utilize hazardous liquid reagents.

The analytical range also posed a challenge. The ISS Program required a 1,000 to 25,000 µg/L range with an accuracy of ± 25% for TOC. The need for a large analytical range was driven by the various international supplies of potable water. The go/no-go decision on water potability is 3,000 µg/L for WPA supplied water and 20,000 µg/L for Russian Segment Water.

Taken together, these constraints resulted in an initial design concept for a non-toxic TOC analyzer to be deployed in a flight-ready package that is robust to the special considerations of microgravity and the on-orbit cabin environment. This basic concept was developed through laboratory component level testing, two moderate fidelity integrated system breadboard prototypes, a flight-like full scale prototype, and the final TOCA unit.

The overall TOCA layout consists of a custom integrated chassis with an electronics and fluids module as shown in Figure 1. Commercial-Off-the-Shelf (COTS) components for internal hardware were selected where appropriate and integrated based on lessons learned during development. The electronics module contains power supplies, power distribution circuits, a computer, and a touchscreen interface. With the exception of wire feed throughs, the fluids module is separate in terms of structure and chassis cooling. The fluids module chassis consists of front and rear compartments separated by a mid-plane bulkhead. Access to both compartments is made via large access panels where the front panel allows access to several components that are on-orbit replaceable units (ORUs) that require servicing throughout nominal operations [e.g., sample bag, acidic buffer container, waste bag, an oxidizer reactor, and a gas-liquid-separator (GLS)].

The major elements of the TOCA schematic are shown in Figure 2. Several underlying core technologies of the analyzer including the Boron Doped Diamond (BDD)-coated electrode oxidizer^{2, 3} Volume Compensation Assembly (VCA), and CO₂ detector⁴ were developed by OI Analytical, College Station, TX. The analyzer uses an innovative BDD-based electrolysis reaction to oxidize the organic carbon. The TOCA consists of two loops that perform the TOC analysis: a water sample loop and a gas loop. The water sample loop's primary function is to introduce and prepare the water sample for analysis. The sample loop contains valves and a pump to direct sample flow, a gas/liquid separator to remove gas from the water sample, a Volume Compensation Assembly (VCA) to provide for sample loop volume adjustability as gas is eliminated (either by de-bubbling the initial sample draw or as TIC and TOC conversion are accomplished), an acid buffer solution container for TIC conversion, and an oxidizer reactor to oxidize TOC.

The gas loop requires the use of ISS Nitrogen (N₂) as a sweep gas. It contains valves and orifices to control and direct gas flow, an adsorber to remove residual CO₂ from

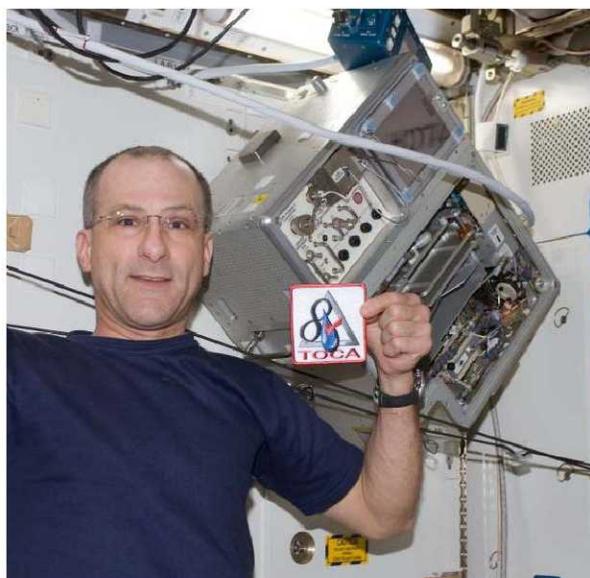


Figure 1. TOCA on the International Space Station with Astronaut Don Pettit prior to installation. Electronics module shown in top section, and fluids module with cover removed shown on bottom.

the ISS N₂, a hydrogen catalyst to reduce Hydrogen (H₂) and Oxygen (O₂) byproducts from the oxidizer's electrolysis process, a Nafion® tube based moisture exchanger to remove moisture within the gas loop, and a Non-Dispersive – Infrared (ND-IR) detector to detect CO₂ from the TIC and TOC conversions.

A more in-depth review of the design of TOCA is discussed in a 2009 paper from this conference⁵.

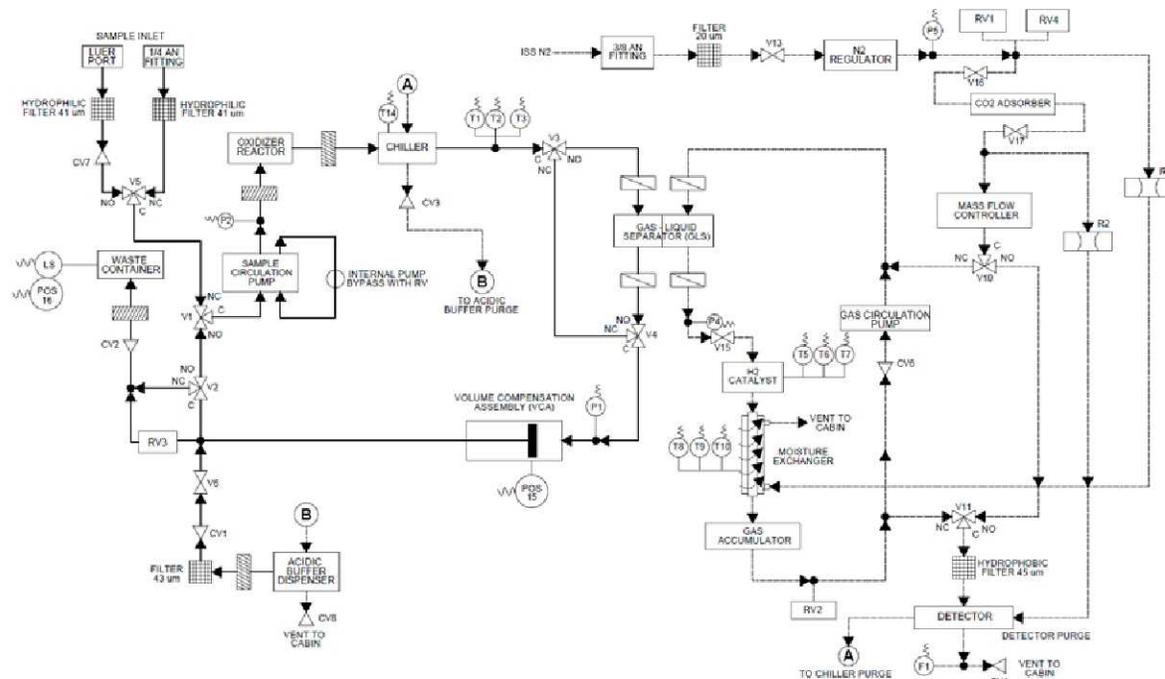


Figure 2. TOCA Protoflight Unit #1 Process Schematic

II. On-orbit Activation and Checkout Timeline

The TOCA Protoflight Unit #1 (PFU1) arrived on the ISS in November 2008. The unit was installed in the ISS laboratory, on the front of the WRS2 rack. Prior to being considered fully operational, both the WRS and TOCA had to demonstrate successful activation and checkout (A/CO), followed by successful 90-day on-orbit operation. The requirements for the TOCA A/CO and 90-day checkout included:

- System “calibration check” values must be consistent with calibrated values.
- Review of engineering data must indicate that unit is operating as designed.
- Initial water sampling from the WPA had to be performed to ensure the TOCA sample values were consistent with archival sample values (ground based analysis).
- Water sampling during 90-day checkout period to ensure TOCA values continue to match well with archive sample analysis performed on the ground.
- Operate TOCA for a time period sufficient to require the waste bag ORU changeout).
- Operate TOCA for a minimum of 12 analyses to verify no degradation of the gas-liquid separator (GLS) and provide overall trend data on the system.

The initial activation was performed on Nov 21, 2008 followed by a calibration check. The first analysis after check-out was terminated because of a GLS failure. It was repaired within three days by replacing the GLS, which is an ORU. The first waste bag changeout was completed on Dec 3, 2008, meeting another 90-day checkout requirement. Before completing the last 90-day checkout requirement of completing 12 analyses, TOCA experienced an unexpected shutdown during analysis attributed to high pressure in the gas loop. Several troubleshooting runs were performed on-orbit and in the TOCA Engineering Development Unit (EDU) on the ground in an effort to identify the root cause of the problem. The engineering team determined that the hydrogen catalyst in the unit had stopped functioning and was allowing hydrogen and oxygen gas to build up within the gas loop, causing the early shutdown. An operational workaround was developed to allow continued operation of TOCA with the failed catalyst still in place. The “React TOC” phase of the analysis was reduced from 10 minutes to 2

minutes to allow operation without exceeding the pressure limit, and the hydrogen and oxygen generated during the run are vented and dispersed into the cabin rather than utilizing the TOCA catalyst to recombine the gases into water. The development of this process is described later in this paper.

Calibration to the new method was completed and subsequent analyses demonstrated good repeatability and no additional anomalies occurred during this period (dates). A replacement catalyst and other repair items were delivered on flight 15A as a contingency, but replacement of the catalyst is unplanned given the success of the new method. Implementation of this new method allowed TOCA operation to continue and the last operational requirement was met on 3/4/09. TOCA operation was deemed acceptable for six person crew onboard ISS once this last operational requirement was met.

III. On-Orbit Performance

TOCA PFU1 was originally certified for a one year life but was extended to two years based on data obtained using the Engineering Development Unit (EDU) on the ground. Following is a summary of the on-orbit results and further discussion of on-orbit failures experienced to date.

A. Analysis Results

To date, TOCA PFU1 has completed over 125 samples. An assessment of TOCA performance can be made by comparing the in-flight results to two categories of ground-based analyses, (1) archived potable water samples analyzed in a laboratory after return to the ground, and (2) prepackaged on-orbit calibration standards.

The TOCA PFU1 meets analytical accuracy requirements of $\pm 25\%$ within a range of 1,000 to 5,000 $\mu\text{g/L}$ TOC. This range is required to envelope the go/no-go decision for water potability of 3,000 $\mu\text{g/L}$ for U.S. Segment potable water sources. When TOCA analyzes water above or below the calibrated values the instrument must extrapolate the linear equation which may exaggerate errors. The current TOCA instrument tends to underpredict values below 1000 $\mu\text{g/L}$ as demonstrated by the in-flight data. When the analytical response is exceedingly low, the TOCA will calculate negative values for TOC.

In addition, the TOCA has a calculated Method Detection Limit per the procedure dictated by Environmental Protection Agency regulations 40 CFR Part 136, Appendix B. [Appendix B to Part 136—Definition and Procedure for the Determination of the Method Detection Limit—Revision 1.11]. The method detection limit (MDL) represents the minimum concentration that can be measured and reported with 99% confidence that the TOC concentration is greater than zero. As shown in Figure 1, the TOCA MDL was 200 $\mu\text{g/L}$ from activation until Jan 2009 when it was increased to 475 $\mu\text{g/L}$ because of the new analytical method. Strict comparisons between in-flight and archive results are difficult since the majority of samples are below the TOCA MDL.

As shown in Figure 3, the TOCA and archive results are in agreement that the ISS potable water is consistently well within the acceptable TOC range for crew health. More specifically, both data sets indicate that the water is consistently below the minimum detection limit for TOCA (475 $\mu\text{g/L}$ TOC). A notable difference in the values is that the TOCA data is reporting negative values for TOC. This effect is caused by the limited calibration range of the TOCA.

The TOCA preventative maintenance plan calls for analysis of a calibration check standard once every three months. The calibration check solution is nominally 3000 $\mu\text{g/L}$ TOC to provide a test of the instrument accuracy at the health limit. Figure 3 also shows results of five calibration checks that have been performed in-flight and all show good correlation with the ground-certified value. In addition, a 1000 $\mu\text{g/L}$ TOC standard was analyzed on 2/10/2010 and again showed excellent correlation.

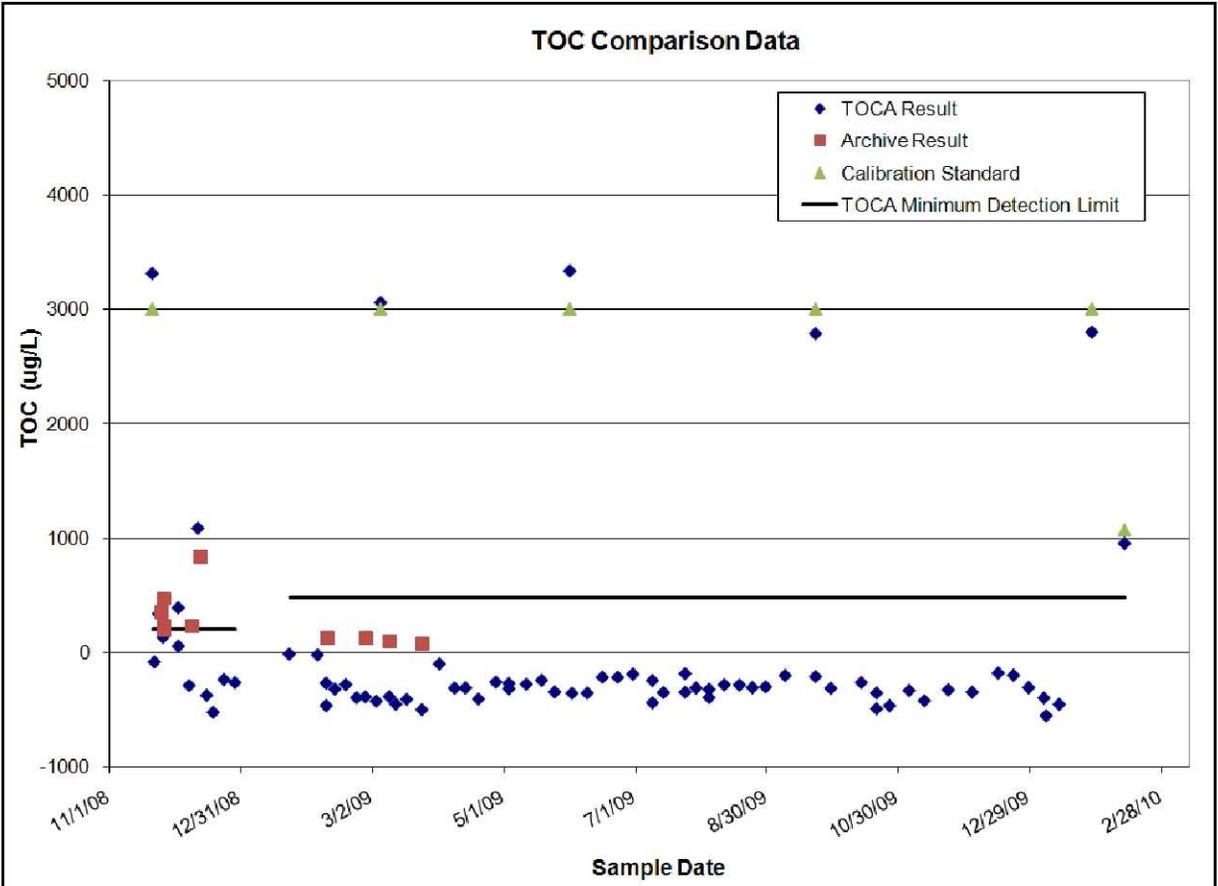


Figure 3. TOC analysis data for on-orbit TOCA results, comparison to archived samples analyzed on the ground, and given calibration standards.

B. Failures

On November 23, 2008 a water sample analysis was automatically terminated due to off-nominal system health values. Review of the data indicated a potential leak in the internal water circulation loop or lack of the system to fully prime and remove gas from the sample water. The crew was instructed to inspect for leakage and following multiple troubleshooting activities over a three day period, the crew found only a small droplet of water on the external housing of the gas-liquid separator (GLS). The GLS is replacement unit, so it was removed and replaced and the subsequent TOCA analysis attempt was successful. The replacement GLS has performed without issue for over 15 months.

The leaking GLS was returned on space shuttle flight STS-126 and returned to Houston for analysis and testing. Post-flight testing confirmed a leak on the water side of the GLS. Unfortunately, a root cause of the leak has not been determined, but any other in-flight or ground-based operation of the GLS assembly has not produced similar failures, and the failure seems isolated. Should future leakage occur at the GLS, this in-flight experience demonstrates the TOCA's capability for proper detection of an anomaly and simple in-flight replacement of the GLS unit.

On December 24, 2008, the TOCA automatically terminated an analysis by detecting high pressure in the gas loop during the "React TOC" phase. In an attempt to recover operations the pressure limit was increased through an uplinked software change. The next analysis on December 30 was also terminated prior to completion due to high pressure in the gas loop. Initial root cause analysis revealed multiple potential causes. To provide more data a troubleshooting activity was executed to dry the TOCA gas lines to evaluate moisture blockage as a potential cause of the increased pressure; however, data collected during troubleshooting indicated no improvement in performance.

All legs of the fault tree were eliminated except for "catalyst failure". The catalyst fails to recombine hydrogen and oxygen gas products in the TOCA gas loop thus allowing accumulation of these gases and results in increased

pressure. The root cause of the catalyst failure has not been determined and is not possible without return and detailed analysis of the catalyst assembly. The catalyst assembly was not designed for on-orbit replacement; however, a spare catalyst was flown on STS-128 for in-flight maintenance in case a software workaround was not possible. The resolution of this issue is described in the following section.

In June 2009 a failure was experienced in a mass flow controller unit during ground-based testing. Upon power up, the valve within the mass flow controller failed to open and allow flow through the unit. The mass flow controller is used in TOCA to precisely regulate the flow of nitrogen through the TOCA gas loop and CO₂ detector. The nitrogen flow rate is directly related to CO₂ detection and therefore is critical for accurate determination of TOC in water samples. Additional testing revealed that other ground units were also failing to allow flow. Further inspection and discussion with the manufacturer indicated that the internal valves could experience compression set if allowed to sit without being cycled for extended durations. Although the on-orbit mass flow controller component was functioning properly, a risk mitigation plan was initiated to ensure regular cycling of the component to minimize the risk of sticking. The plan requests operation of TOCA at least once every 14 days. This is typically not a planning constraint since the TOCA nominal operation schedule is to perform a water sample at least once per week.

On September 8, 2009, the in-flight TOCA pressure sensors detected off-nominal pressures resulting in an automatic termination of the sample analysis and the TOCA was powered down in a safe state. The TOCA sensor data was then downlinked per nominal procedures. The regularly scheduled sample analysis on 9/15 was cancelled to allow time for data evaluation. Evaluation of the downlinked pressure data confirmed that the liquid loop pressure exceeded the software-defined limits and terminated the analysis. The data also confirmed that the liquid loop relief valve opened and protected the system as designed. Other troubleshooting revealed the cause of the pressure increase to be a brief loss of VCA control due to temporary noise in the control lines. The VCA (volume compensation assembly) is a pair of flow-through syringes that actively control pressure in the TOCA liquid loop based on pressure sensor feedback.

Based on understanding of the cause of the pressure increase and the data that indicated a nominal system, two analyses were scheduled and successfully performed on 9/22. To date, this was an isolated occurrence of the anomaly, so it is confirmed as intermittent with a low recurrence frequency.

On October 20, 2009 the TOCA detected off-nominal high water temperature as detected by thermistors T1, T2, and T3. The downlinked data confirmed agreement between all three sensors, and also indicated off-nominal high ambient temperatures at the start of analysis. Further investigation into the ISS cabin temperatures indicated that the cabin was indeed higher than any previous TOCA run. The TOCA water temperature is controlled by a thermoelectric chiller using feedback from thermistors T1, T2, and T3. Temperature control is necessary for TOCA because variability in the water temperature impacts the accuracy of the TOC analysis. TOCA thermal testing demonstrated that the TOCA chiller is limited and ambient temperature variation can still impact the water temperature and the TOC results. The high water temperature message has not repeated in any subsequent analysis. Should the nominal ISS temperature range change in the future the TOCA fault limits could be adjusted through a software update to avoid loss of analysis data.

IV. Hydrogen Catalyst Failure and Recovery

As a result of the TOCA H₂ catalyst failure, the TOCA no longer recombined H₂ and O₂, resulting in a gas build-up in the TOCA gas collection loop followed by automatic out-of-limit shutdown of the fault detection program. The catalyst is not required for functionality but was implemented as a safety control. Safety impacts associated with the failure were a higher gas collection loop pressure, higher H₂ concentration, and more H₂ gas vented. To allow continued TOCA operation without producing increased H₂ safety issues, a two minute oxidation process was adopted in place of 10 min oxidation method. This lowered oxidization time reduced the gas collection loop pressure buildup which is a key parameter in the hydrogen hazard analysis. The H₂ vented is an insignificant amount.

Testing conducted on the ground using the TOCA EDU showed that while the TOC integrated area counts are significantly reduced with the two versus ten minute approach, linearity and repeatability still exist. Data indicates that with this new method, TOCA still meets performance requirements levied for the Project Technical Requirements Specification for WPA operation primarily to meet an accuracy of +/-25% of TOC levels from 1000-5000 ppb with TIC levels up to 500 ppb TOC. See Figure 4.

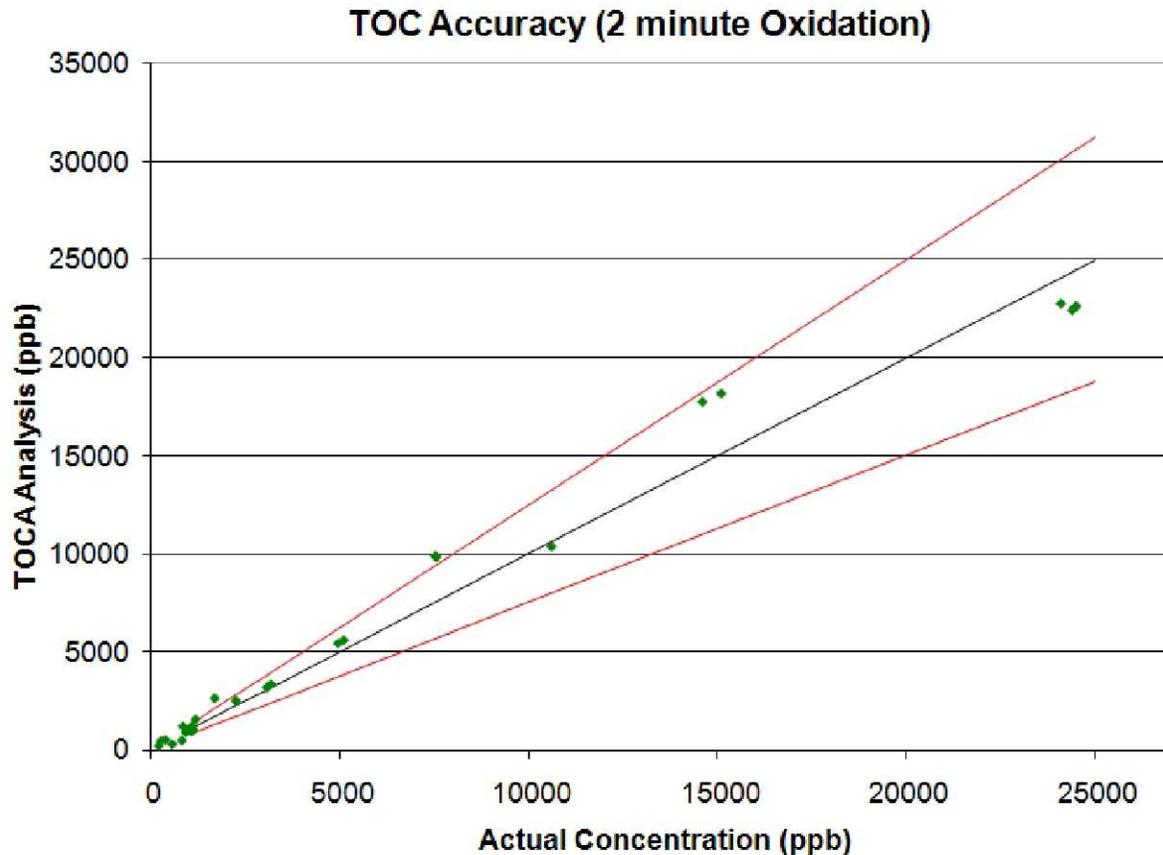


Figure 4. 2 Minute Oxidation Process Accuracy. Samples shown as scatter plot, with linear trend and statistical upper and lower bounds shown.

The following limitations were highlighted through developing this process:

- Sensitivity of the TOCA is reduced, errors are magnified
- TIC carryover becomes significant between TIC 5000-10000 ppb
- TOCA appeared to over predict ISS representative samples
- Method Detection Limit increased from 200 ppb to 475 ppb
- Calibration through 1000 ppb is essential to meeting accuracy requirements at the low end of the detection range.

TIC at higher values were found to be an issue because of the greatly reduced TOC area counts associated with two minutes of oxidation. A small amount of TIC carryover into the TOC process adds interference CO₂ and causes an indicated increase in TOC.

V. TOCA Oxidation Efficiency

The TOCA uses a stable TOC species of Potassium hydrogen phthalate (KHP) for certification testing and calibration. Some early testing had indicated that the TOCA was relatively insensitive to different species of TOC compounds. Following the change of the TOCA process from ten minutes of oxidation time to two minutes of oxidation, TOCA was retested with a set of International Space Station (ISS) representative compounds. A mistake in producing the sample compound produced a solution with a relatively high methanol concentration. When this solution was analyzed in TOCA EDU it over-predicted this value relative to the commercial TOC analyzers used in the JSC Water and Food Analysis Laboratory (WAFAL).

The TOCA CO₂ detector measures the by-product of oxidation (CO₂). Detector counts are integrated over time to produce a term called area counts which then is converted to TOC with a calibration curve. The plateaus were found to be high relative to KHP if the solution contained minimal carbon bonds (interpreted by the team to be compounds that were easier to oxidize). Large carbon chain molecules or multiple double bonded carbons seemed to

have the effect of being harder to oxidize and therefore less CO₂ by-product until those chains were broken down. Several example compounds are shown in Figure 5.

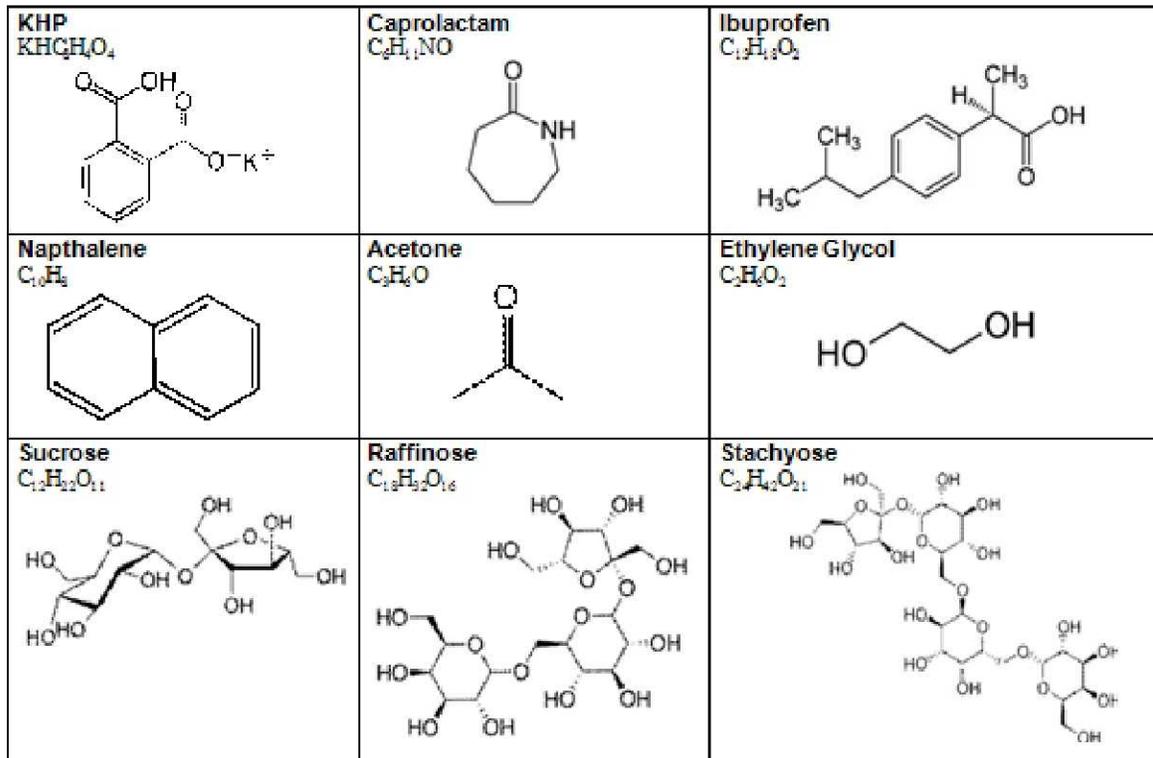


Figure 5. Example organic carbon compounds tested in TOCA

In order to test the relative efficiencies of oxidation of these compounds and TOCA control parameters, the ratio of Error Relative to KHP area counts was used. The term assumes the KHP value for a given amount of TOC is correct and the TOC value, either lower or higher of another species of TOC, as the error from that baseline. When this term is negative the TOCA is under predicting TOC; when it is positive it is over predicting.

The data indicated that higher current density and increased time could reduce the effect of oxidation efficiency differences. The difficult compounds became the driving requirements for increasing oxidizer current and time for subsequent TOCA iterations, as the easier compounds always completed oxidation compared to KHP when oxidation time was extended. To approximate the values for current and time the target area count error was set at 10% or less and solutions of 5000 ppb and 25000 ppb were analyzed. The target current and time were identified as 460 mA and 30 minutes. The 460 mA was limited by the TOCA's 24 volt power supply and the resistivity of the oxidizer.

All of the selections except the starches are considered compounds of toxicological concern that have been previously detected in some ISS archival potable water samples. The starch is a large chain compound and thus a good test of the number of carbons/carbon bonds hypothesis. Ibuprofen is a pharmaceutical and has 13 carbons (only 1 more carbon than sucrose). Benzene and caprolactam both have 6 carbons, so using both would test the benzene ring effect; however, benzene is very volatile and could confound data interpretation. Formaldehyde was selected over ethylene glycol as a small compound for comparison to methanol, because formaldehyde was the limiting compound for the new TOC drinking water limit of 3000 ppb.

Representative TOCA oxidation efficiency results are shown in Figure 6, indicating effective and accurate oxidation for the range of organic carbon constituents tested. This new oxidation method will be implemented on future TOCA flight units.

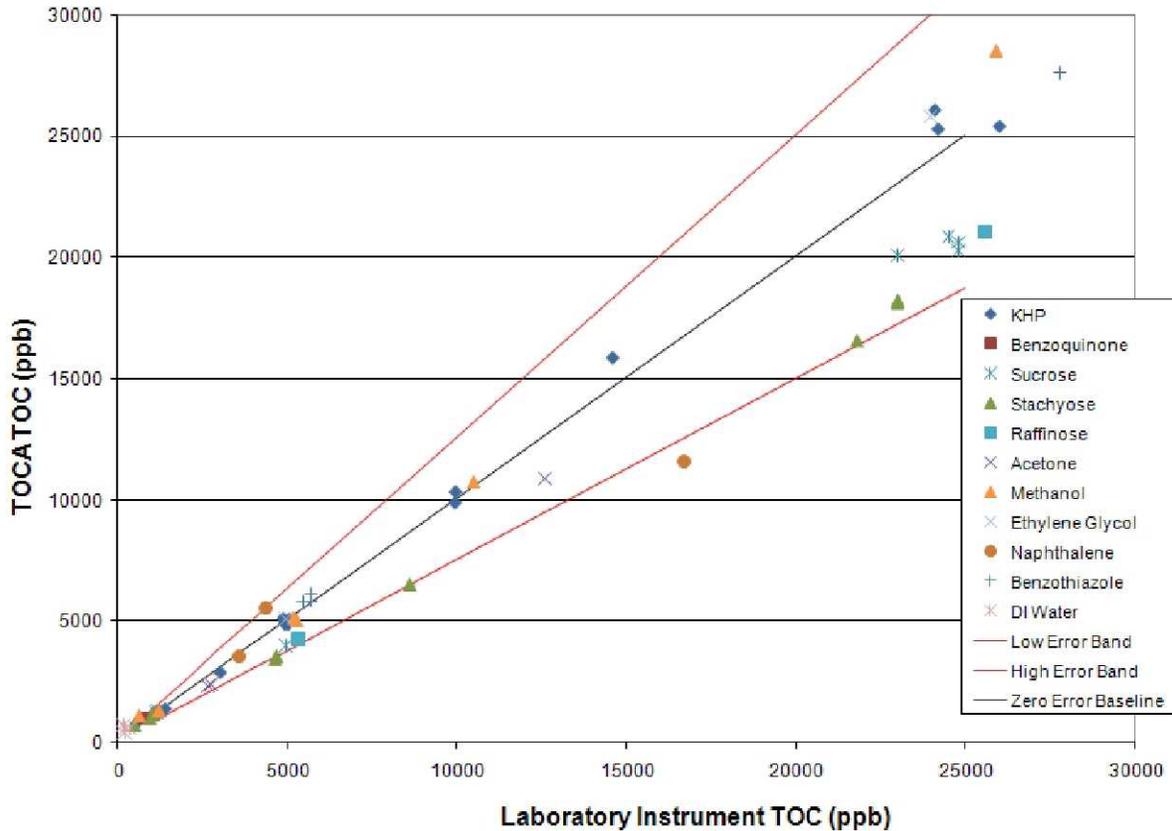


Figure 6. Representative TOCA oxidation efficiency results with a range of tested constituents.

VI. Further Development and Recommendations

In order to meet the required delivery timeline, the operational requirements for PFU1 were reduced from the original set of requirements. Two more TOCA protoflight units are being built (PFU2 and PFU3) to support on-orbit sampling of reclaimed water for the life of the ISS. These two units will meet the original requirements and will have longer life, design and operational improvements over the original flight unit. The design improvements are based on experience with the first flight unit. The requirements for PFU2 and PFU3 will enhance accuracy and range performance of TOCA, add the capability to analyze water from Russian-owned sources on-orbit, and certifies life to 5 years. The difference in performance requirements for TIC and TOC detection between PFU1 and PFU2/3 are shown in Figure 7.

Electronics improvements are being made to the power system to reduce the susceptibility of TOCA to power transients and to improve power distribution throughout the TOCA electronics module. Changes recommended in a GIDEP alert for a voltage reference that is used at multiple locations in the TOCA electronics will also be implemented.

Mechanical improvements include a redesign of the Oxidizer cable to be more user-friendly and add strain relief. Oxidizer is an ORU which requires the ability to mate/de-mate the cable numerous times without damage. The hydrogen catalyst function is also being removed and the routing for the gases produced during analysis is being reworked to allow all gases to be diluted with nitrogen flow prior to venting into the cabin.

Some technology improvements are also being made, one to incorporate a redundant IR filament in the TOCA detector to increase reliability, and one to add a second USB port for keyboard/mouse control of the TOCA. The next two flight units are also adding the capability to be controlled wirelessly if this functionality should be desired in the future.

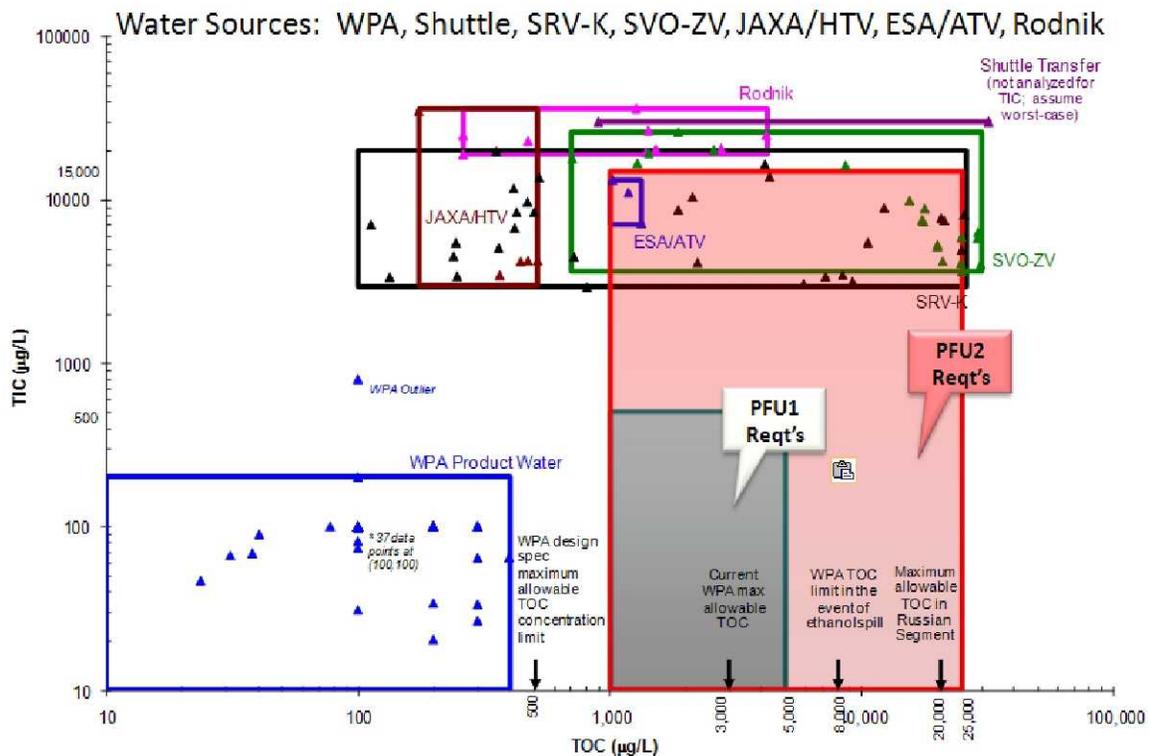


Figure 7. Range of expected TOC and TIC contaminants in various water sources on the ISS; PFU1 versus PFU2 requirements.

Beyond the development of PFU2 and PFU3, there are several other lessons learned that may improve a similar technology in the future. Continued development of TOCA systems is treated here as the potential set of redesign opportunities for TOCA-like systems that would serve potential needs of ongoing ISS and future manned spaceflight programs. We consider this a brief advance planning exercise to explore development approaches for future TOCA systems.

The TOCA protoflight units (PFU1,2,3) serve as a well-defined baseline of a flight-rated TOCA system. Other potential redesign opportunities with respect to this baseline system by considering four basic classes of development strategies are shown in Table 1. These development options serve as a framework for future trade studies. These are useful for manned spaceflight stakeholders to consider when generating plans for future projects.

Table 1. General recommendations for further development of TOCA-like systems

Development Strategy	Engineering Approach	Potential Features & Benefits
More Robust	Perform more exhaustive characterization of the TOCA system to identify sensitivities and define performance limits	More durable Longer life rating Improved environmental operating capability
More Effective	Conduct studies to determine process methods that will oxidize a larger set of challenge solutions	Increased capability of monitoring broader range of samples and contaminants
More Efficient	Perform parametric exercises to identify more optimal design solutions for the same nominal requirement set	Smaller Lighter Less power, water, time to operate
More Innovative	Systematically identify and perform feasibility analyses of TOCA variants along the following perspectives ⁶ Functionality Architecture Interfaces User interactions (Con-Ops)	Use oxidation to treat water, not just monitor water quality (TOCA recycler) Real-time quality measurements during water recycling (TOCA within WPA) Radically different system layout and packaging (TOCA in a can or as part of the drinking dispenser)

VII. Conclusion

The second generation TOCA has been on the International Space Station for over a year and a half, and has served as a valuable and reliable tool for analysis of reclaimed wastewater. The TOCA results correlate well to archived ground-analyzed samples, and calibration check standards.

Several failures and other issues during checkout and operational use of TOCA were resolved and provided valued design information for further improvements to subsequent TOCA units. In particular, an improved oxidation method was developed that precluded the need for a H₂ catalyst, and still resulted in data within accuracy requirements.

The TOCA PFU1 has demonstrated the viability of a reagent-free, autonomous analytical instrument for microgravity spaceflight, and lends credibility to the continued development of similar instruments.

Acknowledgments

The authors recognize and thank the dedication and skill of the TOCA team, including Bioastronautics contract personnel project manager Steve Maignaud, deputy project manager Jeff Milstead, Eric Warren, Mike Romell, Daniela Monterrubio, Heather Mera, Gary Jones, Ben Shoemaker, Scott Flechsig, and Alan Ruter. At NASA, Dr. Karen Pickering, Nik Adam, Carolyn Krumrey, Dale Roberts, and Dave Mathers.

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