

Design, Certification, and Deployment of the Colorimetric Water Quality Monitoring Kit (CWQMK)

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In August 2009, an experimental water quality monitoring kit based on Colorimetric Solid Phase Extraction (CSPE) technology was delivered to the International Space Station (ISS) aboard STS-128/17A. The kit, called the Colorimetric Water Quality Monitoring Kit (CWQMK), was flown and deployed as a Station Development Test Objective (SDTO) experiment on the ISS. The goal of the SDTO experiment is to evaluate the acceptability of CSPE technology for routine water quality monitoring on the ISS. This paper provides an overview of the SDTO experiment, as well as a detailed description of the CWQMK hardware and a summary of the testing and analysis conducted to certify the CWQMK for use on the ISS. The initial results obtained from the SDTO experiment are also reported and discussed in detail.

Nomenclature

<i>AEMC</i>	=	Advanced Environmental Monitoring and Controls
<i>Brij-30</i>	=	polyoxyethylene (4) lauryl ether
<i>COTS</i>	=	commercial orbital transportation services
<i>CSPE</i>	=	colorimetric solid phase extraction
<i>CWQMK</i>	=	colorimetric water quality monitoring kit
<i>EMI</i>	=	electromagnetic interference
<i>DMABR</i>	=	5-(<i>p</i> -dimethylaminobenzylidene)rhodanine
<i>DRS</i>	=	diffuse reflectance spectrophotometer
<i>IP</i>	=	International Partners
<i>ISS</i>	=	International Space Station
<i>JPL</i>	=	Jet Propulsion Laboratory

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<i>JSC</i>	= Johnson Space Center
<i>MORD</i>	= medical operations requirements document
<i>PVP</i>	= polyvinylpyrrolidone
<i>PWD</i>	= potable water dispenser
<i>RAM</i>	= random access memory
<i>SDTO</i>	= Station Development Test Objective
<i>SVO-ZV</i>	= Russian system for dispensing ground-supplied water
<i>US</i>	= United States

I. Introduction

Representative water quality data must be collected in order to ensure that a safe supply of potable water is available to the crew on the International Space Station (ISS). Currently, most of the water quality data for the environmental control and life support systems on the ISS are obtained by collecting archive water samples in-flight and returning them for ground analysis. This archival approach to water quality monitoring has several limitations, most notably the time lapse between sample collection and ground analysis. Samples collected on-orbit must be stored until they can be manifested for return aboard the Shuttle or a Soyuz vehicle. Typical on-orbit storage times for water samples can range from 1-4 months, but in certain situations it can be much longer due to launch delays and limited return payload capacity. This time lapse between sampling and analysis precludes implementation of real time adjustments or corrective actions when water does not meet specifications. Sample integrity is another concern associated with relying on archival samples for water quality monitoring. The storage and transport conditions that archive water samples are subjected to are not ideal, and, as a result, the concentrations of certain compounds in the samples may be affected. This can complicate data interpretation and raise questions about whether or not the results from ground analyses are representative of the water quality on-orbit.

The upcoming retirement of the Shuttle Orbiter fleet creates additional challenges for sample return and makes continued reliance on archival samples for water quality monitoring on the ISS impractical. Following the final Shuttle mission, there will be a sharp decrease in available return payload mass. This will limit both the volume and total number of samples that can be returned and may significantly increase the time lapse between sample collection and analysis. Additionally, since the US will be forced to rely on Commercial Orbital Transportation Services (COTS) and International Partner (IP) vehicles, the process for transporting archive samples to the laboratory once they are returned to the ground may become more difficult.

Following Shuttle retirement, it will no longer be feasible to rely solely on archive water samples to collect such data. Since the vast majority of analytical instruments used to analyze water samples in ground laboratories are not suitable for deployment in-flight, new hardware systems capable of monitoring water quality in-flight on the ISS

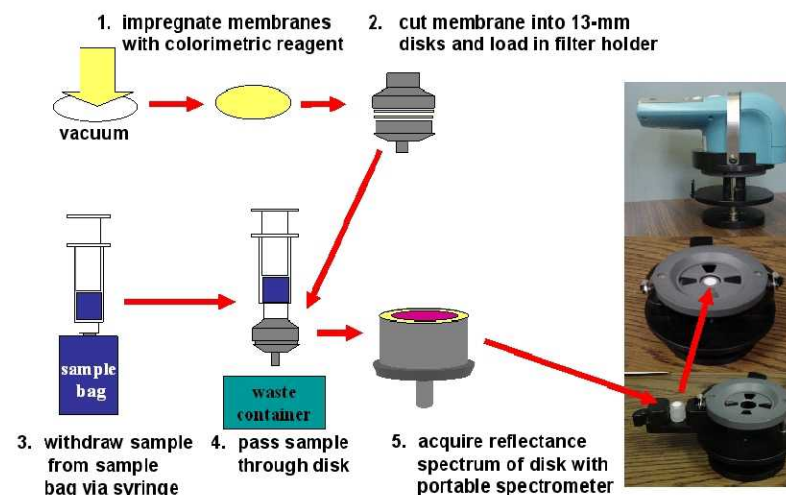


Figure 1. Overview of CSPE Process

need to be developed. The unique operational environment on the ISS and the rigorous safety regulations applied to hardware deployed on the ISS dictate that any water quality monitoring system developed for use in-flight possess several key characteristics. The “ideal” water quality monitoring system for the ISS must be small, light weight, provide direct real time readout of results, minimize waste, contain no hazardous materials, meet strict storage and power guidelines, and function effectively in zero gravity. Further, the system must also be rapid and easy to use so that the crew time required for training and operation is minimal.

One candidate technology that embodies many of the characteristics of the ideal water quality monitoring platform for the ISS is Colorimetric Solid Phase Extraction (CSPE). CSPE is a sorption-spectrophotometric technique that combines colorimetric reagents, solid-phase extraction, and diffuse reflectance spectroscopy to quantify trace analytes in water samples. In CSPE, a syringe is used to meter a known volume of sample through an analysis cartridge that contains a membrane disk impregnated with an analyte-specific colorimetric reagent and any additives required to optimize the complexation of the reagent and analyte. As the sample is passed through the analysis cartridge, analytes are selectively extracted and complexed on the membrane. Formation of the analyte-reagent complex causes a detectable change in the color of the membrane disk that is proportional to the analyte concentration. The analyte is then quantified by measuring the color of the membrane disk surface using a hand-held diffuse reflectance spectrophotometer (DRS). This entire process is illustrated in Fig. 1.

To date, CSPE methods have been developed for a variety of analytes. Current methods include ionic silver,^{1,2} total silver,³ iodine and total iodine,^{2,4,5} nickel(II),^{6,7} chromium(VI),⁷ copper (II),⁷ iron (III),⁷ and formaldehyde.⁸ These methods have all been tested extensively in a terrestrial environment to assess analytical performance and identify potential interferences. Additionally, several of the methods (silver, iodine, and total iodine) were successfully tested on a series of parabolic flights to assess their performance in microgravity.^{2,9,10,11,12,13} Those microgravity experiments clearly demonstrated the potential for a CSPE based system to address some of the water quality monitoring requirements on the ISS. As a result, a Station Development Test Objective (SDTO) experiment was designed to evaluate the suitability of CSPE technology for routine use on the ISS. The experiment, SDTO #15012-U “Near Real-time Water Quality Monitoring Demonstration for ISS Biocides Using Colorimetric Solid Phase Extraction (CSPE)” was launched on STS-128/17A in August of 2009 and is currently deployed on the ISS. The following sections of this paper provide an overview of the SDTO experiment and describe the experiment hardware, the Colorimetric Water Quality Monitoring Kit (CWQMK), in detail. Also discussed are the certification process for the experiment hardware and initial results from deployment of the CWQMK on the ISS.

II. Overview of SDTO Experiment

A. Purpose

The primary purpose of the SDTO experiment is to demonstrate the capability to collect in-flight water quality data on the ISS using CSPE technology. This will be accomplished by using the CWQMK hardware to measure ionic silver and molecular iodine concentrations in ISS water samples on-orbit. Secondary objectives of the experiment include evaluating the accuracy and reproducibility of the CSPE methods, assessing the long-term stability of the DRS and consumable items included in the kit (standard solutions and analysis cartridges), examining approaches to manually manipulate sample bags to isolate and collect bubble-free aliquots of samples, and utilizing downlinked data to verify the results of on-orbit analyses and facilitate real-time troubleshooting, if needed.

B. Analytes

Silver and iodine were selected as test analytes for the SDTO experiment because they are the biocides used in the potable water storage and distribution systems on the ISS. Biocides are added to the potable water systems on spacecraft to inhibit microbial growth. On the United States (US) segment of the ISS molecular iodine serves as the biocide, while the Russian space agency utilizes silver as a biocide in their systems. In both cases, the biocides must be maintained at a level sufficient to control bacterial growth, but low enough to avoid any negative effects on crew health. The presence of high levels of iodine in water can cause taste and odor issues that result in diminished water consumption. There are also concerns about potential impacts on thyroid function following exposure to high levels of iodine. Long term consumption of water containing high levels of silver may result in an irreversible blue-gray discoloration of the skin, a condition known as argyria. Ensuring biocides are maintained at safe, effective levels is one application that clearly illustrates the need to develop in-flight water quality monitoring systems to ensure the health and safety of spaceflight crews.

In addition to illustrating the need to develop in-flight water quality monitoring systems, there is also an unmet requirement to perform in-flight iodine monitoring on the US segment of the ISS. The requirements for in-flight water quality assessment on the ISS are listed in the ISS Medical Operations Requirements Document (MORD). Section B of requirement 7.2.2.2 in the MORD contains the list of parameters that should be monitored in water samples collected from the US segment.¹⁴ Based on recommendations from the NASA Aerospace Medicine Board, iodine, iodide, and iodine compounds are included in that list. At present time, no flight-certified hardware exists

that is capable of measuring iodine, iodide, or iodine compounds in water samples on-orbit. Therefore, successful execution of the SDTO experiment will not only demonstrate the ability to collect in-flight water quality data with CSPE technology, it may also serve as the first step towards developing a system that will address an unmet MORD requirement

C. Approach

The capability to collect water quality data in-flight on the ISS will be demonstrated through a combination of in-flight analyses performed with the CWQMK hardware and experiments conducted on the ground. The SDTO experiment will consist of six in-flight analysis sessions. During each session, ground supplied standard solutions and water samples collected from different points on the ISS will be analyzed with the CWQMK hardware. Water samples will be analyzed in triplicate to assess the reproducibility of the CSPE methods. Samples will be collected from both the US and Russian segments of the ISS. In the US segment, samples will be collected from two different points; the US Potable Water Dispenser (PWD) dispensing needle and the PWD auxiliary port. Multiple sampling points are being utilized in the US segment because the MORD requires the total iodine concentration in potable water to be <0.2 mg/L at the point of consumption. Initially, samples for the SDTO experiment were only going to be collected from points of crew consumption. However, since the most of the iodine is removed from the US potable water before it reaches the PWD dispensing needle there were concerns that analysis of samples collected at the dispensing needle would not fully demonstrate the ability to measure iodine concentrations on the ISS with CSPE. To ensure that the objectives of the SDTO experiment were being met, the PWD auxiliary port was added as an additional sampling point. The PWD auxiliary port is upstream of the iodine removal hardware in the PWD, so these water samples should contain the nominal biocidal concentration of molecular iodine (1-4 mg/L). In the Russian segment, samples will be collected from the system for dispensing ground-supplied water (SVO-ZV). Samples from the US segment will be analyzed for molecular iodine and samples from the Russian segment will be analyzed for ionic silver. The unused portions of the water samples analyzed in each session will be temporarily stowed in the CWQMK until they can be returned to ground for laboratory analysis. At the conclusion of the analysis session, data will be downloaded from the DRS onto the ISS network using an Excel[®] spreadsheet. The data will then be downlinked from the ISS network and transferred to the project team on the ground.

Ground experiments will be conducted in parallel with the in-flight analysis sessions to serve as a control for the in-flight analyses. The ground studies will be performed with the same standard solutions used in the kit and analysis cartridges that were prepared at the same time as those launched in the CWQMK. The standard solutions will be analyzed with standard laboratory methods to assess their stability. The analysis cartridges will be exposed to test solutions containing known concentrations of either molecular iodine or ionic silver. The iodine and silver concentrations obtained from the analysis cartridges will be compared to results from standard laboratory methods and percent recoveries will be calculated for each analyte. The ground experiments will be used to assess the stability of the standard solutions and analysis cartridges as well check for any degradation in performance that may occur as a result of exposure to launch environments and storage conditions on the ISS.

The ultimate determination of the suitability of CSPE technology for routine use in a water quality monitoring system for the ISS will be based upon reproducibility of the measurements and agreement between the results from the in-flight analyses and the results from ground analyses performed on the unused portions of the samples. As described above, the unused portions of the water samples collected in support of the SDTO experiment will be stowed in the CWQMK until they can be returned to ground. Once returned, the unused samples will be analyzed with standard laboratory methods and the results will be used to evaluate the performance of the CWQMK.

III. Hardware Description

The components of the CWQMK were launched in a 16" x 6" x 9" pouch made of natural Nomex fabric. The kit contains all the consumables and equipment required to analyze water samples for molecular iodine and ionic silver. Items provided in the CWQMK include two DRSs with attached sample locators (one primary, one spare), two data transfer cables, calibration standards for both DRS units, a staging/work pallet with iodine standard syringe and drying syringe, six monthly analysis packs, a water pack, and a spares pack. The only consumables from the ISS inventory needed to support the SDTO experiment are batteries (qty. = 8, AA alkaline) from the ISS battery pantry that are used to power the DRS units and the water that is collected for analysis (600 mL total). Additional details about the major components of the CWQMK and how they are used in the SDTO experiment are provided in the following sections. A picture of CWQMK S/N 1001 configured for flight is shown in Fig. 2.



Figure 2. CWQMK S/N 1001 configured for flight.

A. Diffuse Reflectance Spectrophotometers

The instruments used to measure molecular iodine and ionic silver concentrations during the CWQMK in-flight analysis sessions and the ground experiments are BYK-Gardner spectro-guide sphere (d/8° spin) diffuse reflectance spectrophotometers (Model 6839). These hand-held instruments are small, lightweight, battery operated, and collect spectral data over the visible range (400 nm - 700 nm) with 10-nm resolution. They are simple to operate and collect data rapidly; acquisition of a single reflectance spectrum requires only 2 sec. The instruments are also very rugged. They use LED light sources and require no optical alignment. The aperture of the instrument is 11-mm, which readily accommodates the 13-mm diameter membrane disks loaded in the analysis cartridges.

The only differences between the DRSs used in the SDTO experiment and the commercially available units sold by BYK-Gardner are the addition of a sample locator and modification of the instrument firmware. The sample locator was fabricated in-house and allows membrane disks to be positioned reproducibly at the instrument aperture. The instrument firmware was modified by the manufacturer to calculate analyte concentrations and provide direct readout of the concentrations on the instrument screen. Analyte concentrations are calculated using response functions that were determined experimentally. The response functions are based on the value of the Kubelka-Munk function at the wavelength of detection for each analyte. The Kubelka-Munk function is shown in Eq. 1,

$$F(R) = \frac{(1-R)^2}{2R} \quad (1)$$

where R is the relative reflectance of the membrane disk surface at a given wavelength. $F(R)$ is directly related to the concentration of the complexed analyte, C , by Eq. 2,¹⁵

$$F(R) = \frac{2.303\varepsilon C}{s} \quad (2)$$

where ε is the absorptivity of the analyte-reagent complex and s is the scattering coefficient of the membrane surface. The experimentally derived response functions for molecular iodine and ionic silver are shown in Eqs. 3 and 4, respectively.

$$[I_2] = \frac{F(R)_{440nm} + 0.0149}{0.2393} \quad (3)$$

$$[Ag^+] = \frac{-0.1394 + \sqrt{0.1394^2 - (4 \times 0.0367 \times (0.0011 - F(R)_{580nm}))}}{2 \times 0.0367} \quad (4)$$

The subscripts to $F(R)$ in equations 3 and 4 denote the detection wavelengths for the analytes.

B. Calibration Standards

Calibration standards were included in the CWQMK to perform initial calibration of the DRSs detector response and run periodic calibration checks. The standards consist of a black standard, two white standards, and two green standards. The black standard is a felt lined cup that functions as a light trap. It is used to set 0% reflectance on the instruments. The white standards are painted metal coupons that approximate ideal diffuse reflectors. They are used to set 100% reflectance on the instruments. The green standards are painted metal coupons that are used to check the calibration of the DRSs. Green standards are used because the reflectance spectrum of the standards spans the entire spectral range of the instruments (400 nm - 700 nm). A single black standard was included in the CWQMK.

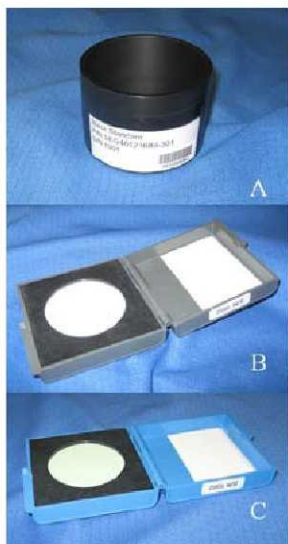


Figure 3. Calibration standards included in the CWQMK. Black standard (A), white standard (B), and green standard (C).

Once calibrated, the DRSs should not need to be recalibrated for the duration of the SDTO experiment. The calibration of the DRS used to perform analyses will be checked at the beginning of each in-flight analysis session by analyzing the green standard. If the DRS is out of calibration, the calibration procedure will be repeated with the black and white standards before continuing the session.



Figure 4. Green standard positioned at the aperture of the DRS.



Figure 5. Staging/work pallet populated for analysis.

C. Staging/Work Pallet

The staging/work pallet is a 7" x 8½" rectangle of reinforced natural Nomex fabric that is used to organize the samples, standards, and consumables while conducting in-flight analyses with the CWQMK. The front side of the pallet contains pockets to hold the analysis cartridges and silver syringes and elastic straps to hold the sample bags, standard bags, waste bag, iodine syringes, and drying syringe. Velcro squares are stitched onto the backside of the pallet so that it can be affixed to the interior surfaces of the ISS in a position that is convenient for the crew member. Two items are permanently stowed on the staging/work pallet; the iodine standard syringe and the drying syringe. The iodine standard syringe is a 10 mL glass-barrel syringe (SGE Analytical Science, P/N 008962) that is covered with transparent heat shrink tubing. The heat shrink tubing is used to prevent shards of glass from escaping into the ISS atmosphere in the event of the barrel breaking. The drying syringe is a 60 mL plastic syringe (Becton Dickinson, P/N 301035) that is used to remove residual water from the analysis cartridges and dry the membrane disks prior to analysis. These two items are stored on the staging/work pallet because they are reused during each analysis session. All other items are either consumed during the analysis session or removed at the conclusion of the session and either discarded or stowed elsewhere in the CWQMK. A photo of the staging/work pallet populated to conduct analyses is shown in Fig. 5.

D. Monthly Analysis Packs

Six monthly analysis packs are provided in the CWQMK. The monthly analysis packs are 12" x 6" Ziploc bags that contain the consumables used to perform in-flight silver and iodine measurements. Each pack includes silver syringes, an iodine sample syringe, a waste bag, and silver and iodine cartridge pack assemblies. Two silver syringes are included in each monthly analysis pack; one standard syringe and one sample syringe. These syringes are 1 mL plastic syringes (Henke-Sass, Wolfe P/N 4010-200VD) that are discarded after analyses are complete. The iodine sample syringe is identical to the iodine standard syringe described above. The iodine sample syringe is also discarded at the end of each analysis session. The waste bag is a 2-port, 1 L Teflon bag (American Fluoroseal, P/N 2PF-0270) that is fitted with a reflux valve (B.Braun, P/N 415068) and tethered cap (Qosina, P/N 65455) on one of the ports. The second port is fitted with a plug that is supplied by the manufacturer and held in place with heat-shrink tubing. The waste bag is used to collect the effluent from the analysis cartridges during analyses. The silver and iodine cartridge pack assemblies contain the analysis cartridges used to perform silver and iodine analyses. Each cartridge pack assembly consists of 4 analysis cartridges (either silver or iodine) that are vacuum sealed in a pouch made of CadPack-P (Cadillac Products) laminate film. The analysis cartridges are loaded with membrane disks that were prepared according to established procedures.¹⁶ Membrane disks in the silver analysis cartridges are treated with 5-(*p*-dimethylaminobenzylidene)rhodanine (DMABR) and polyoxyethylene (4) lauryl ether (Brij-30). Iodine sensitive membrane disks are treated with polyvinylpyrrolidone (PVP). At the conclusion of each analysis session, the Ziploc bag that contained the consumables is used as a trash container. All discarded items are placed in the Ziploc bag and it is discarded in the ISS trash.

E. Water Pack

The water pack is a 5½" x 9" x 2" soft goods pouch made of natural Nomex fabric. The pack is used to store empty sample bags, samples waiting to be analyzed, archive samples waiting to be returned to the ground, and the ground supplied standard solutions. A photo of the water pack is shown in Fig. 6. Empty silver and iodine sample bags are stored in the "Empty Sample Bags" pocket in the pack. Silver sample bags are 30 mL Teflon bags (American Fluoroseal, P/N 1PF-0030) that are fitted with the same reflux valves and tethered caps used on the waste bags. The iodine sample bags are 70 mL Teflon bags (American Fluoroseal, P/N 1PF-0071-CTRV) that come from the vendor with a reflux valve and tethered cap installed. Once the sample bags are filled, they are temporarily stowed under elastic straps in the water pack until they can be analyzed. Once analysis is complete, the sample bags with the unused portion of the samples are stored in the "Archived Sample Bags" pocket of the water pack until they are returned to the ground.



Figure 6. Water pack opened showing silver and iodine standard solutions.

The ground supplied silver and iodine standard solutions are also stored in the water pack. These standard solutions serve as a control for the in-flight analysis sessions; they will be used to assess potential degradation of the analysis cartridges during storage on the ISS. A solution of silver fluoride containing $400 \pm 40 \mu\text{g/L}$ of ionic silver is used as the silver standard. The iodine standard is a dilute solution of molecular iodine that contains $2.0 \pm 0.2 \text{ mg/L}$ iodine as molecular iodine. Both standard solutions are stored in 2-port Teflon bags that are configured similar to the waste bag. One port is fitted with a reflux valve and tethered cap and the other port is fitted with a plug supplied by the manufacturer and held in place with heat-shrink tubing. The silver standard bag is a 55 mL bag (American Fluoroseal, P/N 2PF-0055) that was filled with $50 \pm 5 \text{ mL}$ of standard prior to launch. The iodine standard bag is a 150 mL bag (American Fluoroseal, P/N 2PF-0150-K) that was filled with $150 \pm 15 \text{ mL}$ of iodine standard.

F. Spares Pack

A spares pack was included in the CWQMK for contingency situations. The pack is a 3½" x 1½" x 6⅜" soft goods pouch made of natural Nomex fabric that contains an extra set of consumables, extra sample bags, sampling adapters, a silver syringe, an iodine syringe, and a drying syringe. In the event that any of the items in the monthly analysis packs are damaged or either of the syringes stored on the staging/work pallet are inadvertently discarded, a replacement can be taken from the spares pack and the analysis session can be completed.

Sampling adapters for the US PWD and the SVO-ZV were included in the spares pack to simplify scheduling the CWQMK in-flight analysis sessions. Under normal circumstances, the water samples analyzed as part of the SDTO experiment will be collected during the routine monthly water sampling sessions using the adapters provided in the water sampling kit. While this minimizes the crew time associated with the SDTO experiment and reduces the overall mass of the CWQMK, it also adds constraints to scheduling the in-flight analysis sessions. Since samples must be collected prior to the analysis, the in-flight analysis sessions with the CWQMK must be scheduled after the monthly water sampling activity. If the monthly water sampling sessions are cancelled due to conflicts on crew timelines or issues with the ISS systems then no in-flight analysis session can be conducted. Providing sampling adapters in the spares pack allows the samples for the SDTO experiment to be collected independent of the nominal archive water samples and adds the capability to perform in-flight analysis sessions when monthly water sampling sessions are cancelled.

IV. Certification of the CWQMK

The CWQMK is experimental hardware that was launched as a SDTO experiment for ISS. Typically, the only requirements levied against experimental hardware are those related to safety. After being reviewed by the Johnson Space Center (JSC) Safety Review Panel, it was determined that no new or unique hazards were associated with deployment of the CWQMK hardware on the ISS. As such, certification of the CWQMK for use on the ISS consisted of addressing the generic hazards listed in the ISS Flight Hardware Standardized Hazard Control Report, form # JSC 1366.

In addition to verifying that all safety requirements were met, the CWQMK hardware also underwent functional testing prior to delivery. There were two elements to the functional testing; radiation susceptibility and performance validation. The radiation susceptibility test evaluated the impact of ionizing radiation on several of the CWQMK components. Performance validation testing was conducted to ensure that the DRSs and analysis cartridges launched in the CWQMK met the performance requirements established for the hardware. Additional details pertaining to the safety certification and functional testing of the CWQMK are provided in the following sections.

A. Safety Certification

As stated above, the safety certification of the CWQMK consisted of addressing the generic hazards listed in the ISS Flight Hardware Standardized Hazard Control Report, form # JSC 1366. There are 17 individual hazards listed in the report, 9 of which were deemed to be applicable to the CWQMK hardware. The applicable hazards were structural failure, sharp edges, shatterable material release, flammable materials, material off-gassing, non-ionizing radiation, battery failure, touch temperature, and pinch points. For 6 of the applicable hazards, verification of control was accomplished through analysis of the materials used to fabricate the hardware and inspection of the as-built hardware. Only the hazards associated with material off-gassing, non-ionizing radiation, and touch temperature required testing for verification of the hazard control.

Off-gas testing of the CWQMK hardware was conducted at the White Sands Test Facility. The test focused on the unique components of the CWQMK that are not commonly used in the ISS environment. As such, no soft goods assemblies or packing foam were included in the test. Two separate off-gas tests were performed with the components of the kit. The first test was conducted with the DRS (including batteries), the calibration standards, and the data transfer cable. The second test was performed with the silver and iodine cartridge pack assemblies and the silver and iodine standard solutions. Both tests were conducted using standard test procedures. All components were tested for 72 hours at a temperature of 120° F (49° C). In both tests, the Toxic Hazard Indices (T-values) for all detected compounds were well below acceptable levels for the ISS environment.^{17,18}

Non-ionizing radiation testing was performed in the Electromagnetic Interference (EMI) laboratory at the Johnson Space Center. This testing was focused on the electromagnetic emissions from the DRS when it is connected to a laptop computer, similar to the configuration that will be used during data transfer on the ISS. During the test, the configuration exceeded the acceptable emission limits at frequencies below 30 MHz, at 264.15 MHz, and at 312.2 MHz. The test results were reviewed by the ISS Electromagnetic Effects Panel and a Tailoring/Interpretation Agreement was prepared to address the exceedances.¹⁹ The panel determined that none of the exceedances posed a concern for the ISS and no operational constraints were imposed on the CWQMK hardware. Exceedances below 30 MHz do not affect any ISS hardware because no ISS or visiting vehicle intentional receivers operate at frequencies that low. The higher frequency exceedances were also deemed to pose

minimal risk to ISS hardware. While 264.15 MHz is close to a downlink frequency used by the Shuttle vehicles, the emission from the DRS will be attenuated by the shielding of the ISS and the exceedance is not expected to interfere with Shuttle communications.

Touch temperature testing was conducted in the flight hardware laboratories of the Wyle Integrated Science and Engineering Group. Testing verified that the DRS does not exceed the 113 °F (45 °C) touch temperature limit for continuous contact specified in the ISS Safety Requirements Document.²⁰ As a result, no operational controls or hardware modification was required for operating the DRS on the ISS.

B. Functional Testing

The functional tests performed on the CWQMK hardware focused on assessing the susceptibility of the CWQMK components to ionizing radiation and evaluating the performance of the DRSs and analysis cartridges launched in the kit. Radiation susceptibility testing examined the impacts of ionizing radiation on the DRSs, analysis cartridges, and standard solutions. The performance evaluation was conducted to verify that the DRSs and analysis cartridges met performance requirements established for the CWQMK.²¹

Radiation susceptibility testing was performed at the Indiana University Cyclotron Facility in Bloomington, Indiana. A DRS, silver and iodine analysis cartridges, and silver and iodine standard solutions were irradiated with a proton beam to simulate the worst-case ionizing radiation environment on the ISS. The test articles were exposed to a proton beam with a minimum proton density of 1×10^{10} protons/cm², which equates to a radiation dose of 600 rad. This is the standard radiation exposure used when testing hardware for use on the ISS. The susceptibility of the DRS to the radiation beam was examined by remotely commanding the DRS to collect data while the instrument was being irradiated. Additionally, data stored in the instrument memory was periodically downloaded to a computer to check for corrupt data files. To assess the impact of the radiation dose on the analysis cartridges, irradiated analysis cartridges were exposed to solutions with known silver or iodine concentrations. The results obtained from the irradiated cartridges were then compared to results obtained with non-irradiated cartridges that had been exposed to the same solutions. The effect of the radiation dose on the standard solutions was evaluated by comparing silver and iodine concentrations measured by CSPE in the standard solutions before and after irradiation.

Two main issues were observed with the DRS during the radiation susceptibility test. The first issue was corruption of data files that resulted from exposure to ionizing radiation. Potential corruption of the data files on the DRS was a known risk prior to the test. Data files are stored in static random access memory (RAM) on the instrument, which is known to be susceptible to ionizing radiation. In order to mitigate the risk, the instrument manufacturer modified the firmware on the DRS to store data in multiple memory locations; each data file is written in three distinct locations on the static RAM. An error checking and data recovery protocol was also added to the firmware that allows the instrument to detect corrupt data files and repair them by comparing the data values stored in the different memory locations. This mitigation strategy proved to be very effective; all corrupt data files that occurred as a result of irradiation were successfully detected and repaired by the instrument.

The second issue was a lock up of the DRS that led to early termination of the test. The test plan called for the radiation beam to be focused on six different areas of the instrument. Irradiation of the first two areas on the instrument, on the back side of the display screen, resulted in corrupt data files (described above). When the focal point of the radiation beam was moved closer to the integrating sphere, the instrument froze and could not be recovered. At this point the test was terminated. The result from this test was very surprising because the operational code for the instrument is stored in a flash memory module. Previous testing performed on other flight hardware has shown that flash memory is very robust and is not affected by ionizing radiation. After discussing the test results with the instrument manufacturer, it was discovered that while the operating code for the DRS is stored in the flash memory, several variables used by the code are written to the static RAM. The manufacturer confirmed that corruption of these variables would cause an error that could cause the instrument to lock up. Fortunately, two reset functions were incorporated in the instrument firmware that allows these variables to be reset and makes it possible to recover from this type of error. The 'soft' reset function resets the operating variables stored in the static RAM but leaves all other data intact. The 'hard' reset function deletes all data stored in the static RAM then resets the operating variables to their default values. If either reset function is used, the instrument must be recalibrated before performing any analyses. Once the DRS was returned from the test facility, operation was successfully restored using the 'soft' reset function. While the possibility for a radiation incident to lock up the DRS is not ideal, it should be noted that the test conditions represented a worst-case scenario for radiation exposure. The nominal

radiation levels on ISS are significantly lower than the test dose. Also, the risk of a radiation induced instrument lock up preventing data collection is sufficiently mitigated by the capability to restore operation using the reset functions and the fact that two DRSs are provided in the kit.

Exposure of the analysis cartridges and standard solutions to the radiation beam had no observable effect on the performance of the analysis cartridges or the analyte concentrations in the standard solutions. Mean analyte concentrations (N=3) obtained from the irradiated analysis cartridges were within 15% of the pre-test value. The silver concentration in the standard solution following radiation exposure was within 15% of the pre-test value measured by CSPE. Iodine concentrations measured in the standard solution with CSPE before and after irradiation differed by less than 5%.

The performance evaluation was conducted to verify that the DRSs and analysis cartridges in the CWQMK met the performance requirements listed in JSC-64149, Science Requirements Document for the Colorimetric Solid Phase Extraction (CSPE) Technology Demonstration. This document lists the top level science and performance requirements that must be met by the CWQMK hardware. It states that the CWQMK must provide the capability to measure ionic silver and molecular iodine in water samples collected on ISS. Analytical ranges for the two analytes are also specified in this document. The CWQMK hardware must be capable of measuring ionic silver in the range from 0.1 – 1.0 mg/L and molecular iodine in the range from 0.2 – 4.0 mg/L.

In order to verify that these performance requirements are met by the CWQMK, tests were conducted with the DRSs that were launched in the flight kit and analysis cartridges prepared at the same time as those launched in the kit. During the test, analysis cartridges were exposed to solutions with known concentrations of either ionic silver or molecular iodine that spanned the required analytical range for each analyte. The cartridges were then analyzed with the flight DRSs to measure silver and iodine concentrations that were compared to reference concentrations by calculating a percent recovery for the analysis cartridges. The percent recovery was calculated by dividing the analyte concentration obtained with CSPE by the reference concentration measured with standard laboratory methods and multiplying by 100. Silver reference concentrations were measured using inductively coupled plasma mass spectrometry. Iodine reference concentrations were measured using the leuco-crystal violet spectrophotometric method. Results from this test are summarized in Table 1. The overall agreement between the silver and iodine concentrations measured by CSPE and the standard laboratory methods was excellent. The percent recoveries for silver ranged from 77 to 113.5 and the iodine recoveries ranged from 108.5 to 115.0. These data clearly demonstrate that the CWQMK hardware provides the capability to measure ionic silver and molecular iodine in the required analytical ranges.

Table 1. Data from the performance evaluation of the DRS units and analysis cartridges.

Analyte	Reference concentration (mg/L)	DRS S/N 1001		DRS S/N 1002	
		Mean concentration measured by CSPE (mg/L), N=3	% Recovery	Mean concentration measured by CSPE (mg/L), N=3	% Recovery
Silver	0.140	0.108	77.1	0.118	84.3
	0.540	0.533	98.7	0.613	113.5
	1.040	1.001	96.3	1.057	101.6
Iodine	0.20	0.22	110.0	0.22	110.0
	1.93	2.22	115.0	2.11	109.3
	5.03	5.46	108.5	5.67	112.7

V. Initial Results

To date, four of the six planned in-flight analysis sessions with the CWQMK have been completed. During the first three sessions, samples from the PWD dispensing needle and the SVO-ZV system were analyzed. The plan for the SDTO experiment was to alternate the sampling point in the US segment between the PWD dispensing needle and the PWD auxiliary port. However, due to delays preparing the procedure for sampling the PWD auxiliary port no samples could be collected from that point until the fourth analysis session. The first sample from the PWD auxiliary port was analyzed during the fourth session, along with another sample from the SVO-ZV system. Issues

were also encountered downloading spectral data from the DRS. Delays deploying new laptops on the ISS prevented data from being downloaded to the ISS network until after the fourth analysis session. Prior to the initial download, the only data available were the silver and iodine concentrations obtained during the analyses. These concentrations were recorded in the crew notes and called down to Mission Control. Results obtained during the in-flight sessions and descriptions of the difficulties encountered during the sessions are discussed below along with the results from the first four sessions of the parallel ground experiments.

A. Results from In-flight Analysis Sessions

The first in-flight analysis session with the CWQMK occurred on September 23, 2009. This session was scheduled immediately after activation and checkout of the experiment hardware. Samples from the PWD dispensing needle and the SVO-ZV system were analyzed during the session. Due to conflicts on the crew timeline, the crew member on the ISS that was trained to work with the CWQMK was not available to perform these activities. As a result, the crew member that performed the activation and check out of the hardware and conducted the first analysis session had never seen the CWQMK hardware prior to working with it. Despite their lack of familiarity with the hardware, the crew member successfully completed the activation and check-out, which included calibration of the DRS. Unfortunately, several issues arose during the analyses and the data set collected during the session was incomplete.

The first issue that was reported during the session was caused by failure of one of the silver analysis cartridges. During analysis of the silver standard solution, the membrane disk stuck in the upper portion of the analysis cartridge when it was disassembled. Normally, the membrane disk remains on the lower portion of the cartridge, which is loaded in the sample locator on the DRS so that the reflectance spectrum of the disk can be collected. Since the crew member was never trained to perform the analyses, they did not recognize this failure and proceeded to collect the reflectance spectrum of the screen that supports the membrane disk in the lower portion of the analysis cartridge. The crew member then moved on to analyze the water sample from the SVO-ZV system. When they disassembled the first analysis cartridge exposed to the sample, the cartridge functioned as expected and the membrane disk stayed in place on the lower portion of the cartridge. This led to some confusion because the cartridge looked very different than the one used with the standard solution. Eventually, members of the project team were able to explain what had happened and clarify how the analysis cartridges are supposed to function. By this time, however, the analysis session had exceeded its expected duration and the crew member needed to move on to their next assigned task. The crew member eventually returned to complete the analysis session during their pre-sleep period, but due to the earlier difficulties there were only two silver analysis cartridges remaining. They re-ran the silver standard solution and the SVO-ZV sample with the remaining cartridges. No replicate analyses were run on the SVO-ZV sample. The measured silver concentrations in the standard solution and sample were 0.404 mg/L and <0.100 mg/L, respectively. The silver concentration measured in the standard solution was consistent with results from pre-flight sampling. The result from the SVO-ZV sample was lower than anticipated, but it was not outside the range of silver concentrations previously measured in archive samples.

There also seems to have been some misunderstanding during analysis of the iodine standard and the sample from the PWD dispensing needle. While no difficulties or issues were documented in the crew notes, the replicate analyses were not run on the sample. The downloaded data and crew notes show that the iodine standard was analyzed followed by a single analysis of the PWD sample. The iodine concentration measured in the standard solution was 2.79 mg/L and the sample contained <0.20 mg/L iodine. The iodine concentration measured in the standard solution was higher than expected, but it is suspected that this may have resulted from low flow rate during analysis of the standard. Previous testing has shown that the iodine concentrations measured by CSPE can be affected by the rate at which the sample is passed through the analysis cartridge. If the flow rate is significantly less than 1 ml/s the iodine concentration measured in the sample will be higher than the actual concentration. Given the fact that the crew member performing the analyses was not familiar with the hardware and other elements of the session were missed (replicate analyses), it is possible that they did not notice the target flow rate called out in the procedure. The iodine concentration detected in the sample from the PWD dispensing needle is consistent with the expected concentration downstream of the iodine removal hardware in the PWD. Based on the issues encountered during the first analysis session, the crew procedures were updated to emphasize the target flow rate for the iodine analysis and the need to analyze the samples in triplicate.



Figure 7. ISS crew member performing the second in-flight analysis session with the CWQMK.

the concentration measured during both runs was <0.100 mg/L. The iodine concentration measured in the standard solution was 1.95 mg/L. This result is closer to the initial concentration loaded in the standard bag (1.83 mg/L), but still slightly elevated. All three analyses performed on the sample from the PWD dispensing needle yielded the expected iodine concentration of <0.20 mg/L. A photo of the crew member conducting the second analysis session on the ISS is shown in Fig. 7.

At the conclusion of the session, the DRS indicated that a memory check had failed and data files had been repaired. A comparison of the downlinked data with the results reported during the first two sessions confirmed that the data files were successfully repaired. This incident is the same type of memory failure that occurred during radiation susceptibility testing. Recovery of the corrupt data files proves that the memory check and file repair protocols incorporated in the DRS firmware effectively mitigate the risk associated with storing data in the static RAM on the instrument.

The third in-flight analysis session with the CWQMK was conducted on November 11, 2009. Once again, samples from the PWD dispensing needle and the SVO-ZV system were analyzed. This was the final planned sample from the PWD dispensing needle; all future samples from the US segment will be collected from the PWD auxiliary port. A trained crew member performed the session and while no issues were recorded in the crew notes, only two analyses were reported for the PWD sample. No explanation was provided for the missing data point. The silver concentration measured in the standard solution continued to decrease. It was only 0.277 mg/L during this session. As this was the second session that showed a decrease in the silver concentration in the standard with no reported anomalies, the results seems to indicate that some degradation is occurring in either the standard solution or the analysis cartridges. All three analyses run on the SVO-ZV produced silver concentrations <0.100 mg/L, consistent with the previous sessions. The iodine concentration in the standard solution was 1.33 mg/L. This result is lower than the initial concentration measured when the standard bag was filled, but some decrease in the iodine concentration was anticipated. The iodine concentrations measured during the two analyses of the PWD sample were both <0.20 mg/L.

The fourth in-flight analysis session was run on January 7, 2010. No analysis session was scheduled in December 2009 because issues with the US water recovery system limited the availability of water and all water sampling was canceled. The same crew member conducted both the third and fourth sessions. Samples from the PWD auxiliary port and the SVO-ZV system were analyzed during the activity. This was the first analysis of a water sample from the auxiliary port, which is upstream of the iodine removal hardware in the PWD. A complete data set was collected during the session and no issues or anomalies were reported. The silver concentration in the standard solution was measured as 0.236 mg/L and all three analyses run on the SVO-ZV samples returned concentrations <0.100 mg/L. The iodine concentration in the standard solutions was measured to be 1.49 mg/L, which is slightly higher than the last time the standard was analyzed. This difference is larger than what would be expected due to variability in the analysis cartridges and may be indicative of low flow rate during analysis of the

standard. The mean iodine concentration detected during the three analyses of the auxiliary port sample was 0.76 mg/L. As expected, iodine was detected in the sample, but the measured concentration is approximately half the expected concentration for water collected from the auxiliary port. Since this was the first time samples were collected from the auxiliary port, it is not clear if the flush volume specified in the collection procedure was adequate to purge stagnant water from the PWD plumbing. It is possible that the collected sample included some water that had been stagnant in the PWD plumbing for several months, which could explain the reduced iodine concentration. The results from all four in-flight analysis sessions are summarized in Table 2.

Table 2. Results from in-flight analysis sessions.

Solution	Session 1 Measured concentration (mg/L)	Session #2 Measured concentration (mg/L)	Session 3 Measured concentration (mg/L)	Session 4 Measured concentration (mg/L)
Silver standard	0.404	0.315	0.277	0.236
Sample from SVO-ZV	<0.100	<0.100	<0.100	<0.100
	-	<0.100	<0.100	<0.100
	-	-	<0.100	<0.100
Iodine Standard	2.79	1.95	1.33	1.49
Sample from PWD dispensing needle	<0.20	<0.20	<0.20	-
	-	<0.20	<0.20	-
	-	<0.20	-	-
Sample from PWD auxiliary port	-	-	-	1.04
	-	-	-	0.63
	-	-	-	0.63

B. Results from Ground Experiments

Ground experiments were conducted in parallel with each in-flight analysis session to serve as controls for the in-flight analyses. The experiments are used to assess the stability of the standard solutions and analysis cartridges and check for any degradation in performance that may have occurred as a result of exposure to the Shuttle launch environment and storage conditions on the ISS. All experiments were performed with bags of the same standard solutions used in the flight kit and analysis cartridges that were prepared at the same time as those used to perform the in-flight analyses. All test items were processed and configured as Class I flight hardware before being downgraded for use in the ground tests. The standard solutions and analysis cartridges were stored at ambient temperature and protected from direct exposure to light until used.

Samples of the standard solutions used in the CWQMK were collected after each in-flight analysis session and analyzed with standard laboratory methods. Silver concentrations were determined using inductively coupled plasma-mass spectrometry and iodine concentrations were measured using the leuco crystal violet spectrophotometric method. The results from these analyses are plotted in Fig. 8. Initial analyses performed when the standard solutions were prepared in April, 2009 showed that the silver standard solution contained 0.403 mg/L of silver and the iodine standard contained 1.83 mg/L of iodine.

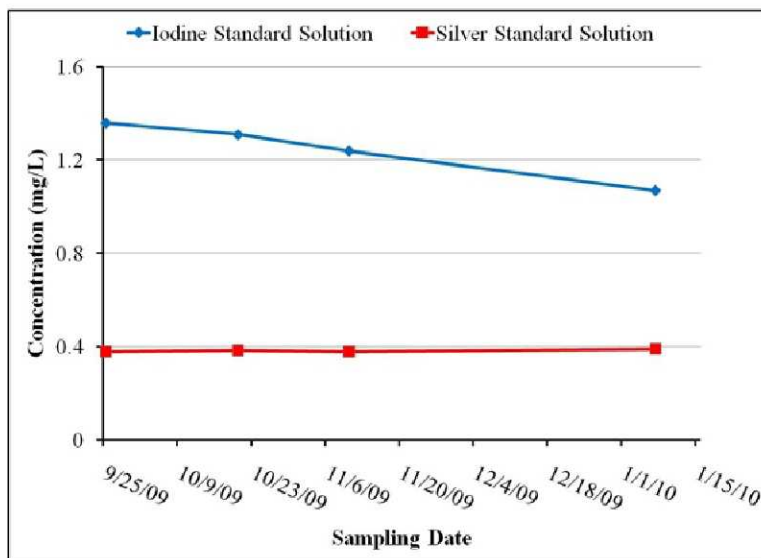


Figure 8. Results from ground experiments performed with the standard solutions launched in the CWQMK.

Thus far, all analyses performed on the silver standard solution indicate that the silver standard is very stable. The measured silver concentrations have remained within 5% of the initial concentration measured in the standard solution for 9 months. The iodine standard solution, however, has shown signs of significant degradation during the ground experiments. By the time the initial in-flight analysis session had occurred, the concentration of iodine in the standard solution had already decreased to 1.36 mg/L. The measured iodine concentration continued to decrease in the subsequent ground experiments, falling to 1.07 mg/L by the time the fourth in-flight analysis session was complete.

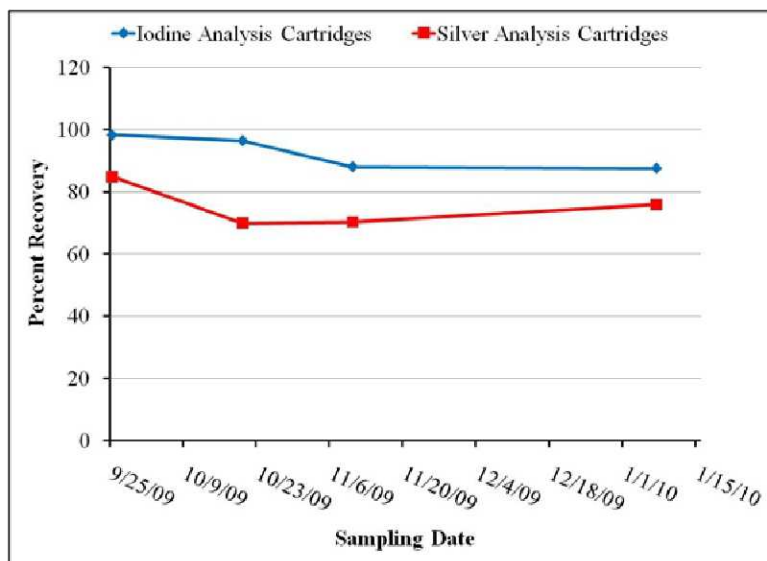


Figure 9. Results from ground experiments performed with silver and iodine analysis cartridges prepared at the same time as those used during in-flight analysis sessions.

The ground experiments also evaluated the stability of the analysis cartridges that were launched in the CWQMK. Following each in-flight session, analysis cartridges prepared at the same time as those used in the in-flight analyses were exposed to solutions with known silver and iodine concentrations. The silver and iodine concentrations measured with the analysis cartridges were used to calculate percent recoveries in the same manner described above. The mean percent recoveries calculated during each set of ground experiments are plotted in Fig. 9. All data points in the figure represent mean recovery values calculated from at least 3 replicate analyses.

The silver analysis cartridges demonstrated a definite decrease in percent recovery as storage times increased in the ground experiments. The silver solutions used to test the analysis cartridges on the ground contained approximately 0.400 mg/L of silver. During the performance verification test, other analysis cartridges that were prepared at the same time exhibited a 98.7% recovery when challenged with a similar solution (see Table 1). The initial set of ground experiments were conducted 5 months after the performance verification test. By that time, the percent recovery obtained from the silver analysis cartridges had dropped to 84%. The percent recovery decreased another 15% in the second set of ground experiments, before stabilizing between 70% and 75% in the third and fourth set of experiments. The iodine analysis cartridges also displayed some decrease in the calculated percent recoveries, but the decrease was not as severe as that observed with the silver cartridges. The percent recoveries were greater than 96% during the first two sets of experiments. These values are similar to values obtained during the performance verification test. The recoveries decreased slightly during the third and fourth set of experiments, but remained above 87%.

C. Discussion

While the ultimate evaluation of CSPE technology will be based upon agreement between the results from the in-flight analyses and the ground analyses performed on the unused portions of the samples, the results obtained from the ground experiments provide interesting insight into some of the data collected during the first four in-flight analysis sessions. The silver concentrations measured during in-flight analyses of the standard solution decrease by 42% over the four analysis sessions. Results from the ground experiments indicate that silver standard solution should be very stable, which would suggest the performance of the analysis cartridges is degrading. This is supported by the ground experiments with the silver analysis cartridges which showed a 25-30% decrease in calculated percent recoveries when compared to results from the performance verification test. Additional ground testing will be needed to determine if the apparent stabilization of the analysis cartridges is real or if the percent recoveries will continue to decrease. If the performance of the silver analysis cartridges does stabilize, it may be possible to compensate for the degradation in response by aging cartridges prior to use and adjusting the coefficients in the response function used to calculate the silver concentrations.

The ground experiments also substantiate the hypothesis that the flow rates used during in-flight analyses of the iodine standard solution were too slow. The marked decrease in the iodine concentration measured in the standard solution coupled with the consistent percent recoveries observed with the iodine analysis cartridges indicate that the results reported during 3 of the 4 in-flight analysis sessions are likely higher than the actual iodine concentration in the standard solution. This is further evidenced by the results of the standard analyses from the first two analysis sessions. The iodine concentrations measured during these sessions (2.97 mg/L and 1.95 mg/L) were both higher than the concentration measured when the standard bag was filled (1.83 mg/L). The only result that agrees with the ground experiments is the concentration reported during the third session. In-flight analysis measured 1.33 mg/L iodine as compared to the 1.24 mg/L measured in the ground experiments. The difference between these two values is less than 10%, which is within the experimental error of the measurement. Despite earlier efforts to emphasize the flow rate required to accurately perform iodine analyses, it appears as though this important point is still being missed during the in-flight analyses. Further modification of the analysis procedures is underway to address this issue.

The first set of archive water samples from the SDTO experiment are manifested for return on STS-131/19A. Until the ground analyses are completed on those samples it will not be possible to fully assess the results from in-flight analyses run on the samples. The silver concentrations measured in the samples were all lower than anticipated, but not outside the range of concentrations previously detected in archive water samples from the SVO-ZV system. This may be partially due to the apparent decrease in percent recoveries observed with the silver analysis cartridges in ground experiments. The iodine concentrations measured in the samples from the PWD dispensing needle have all been within the expected range of <0.20 mg/L. If reduced flow rate is responsible for the elevated iodine concentrations measured during analysis of the standard solution, it is reasonable to assume that the same flow rates would have been used during analyses run on the samples. This would mean that the iodine concentrations measured in the samples would also tend to be elevated. Unfortunately, the measured concentrations are below the detection limit for the method, so it may not be possible to verify this assumption, even with the results from ground analyses. Analysis of the one sample collected from the PWD auxiliary port showed that iodine was present upstream of the iodine removal hardware the PWD, as expected. However, due to questions about the flush volume specified in the collection procedure, it may be difficult to access the accuracy of this result. The sample collection procedures are being modified to ensure that representative samples of the WPA product water are collected during future sessions.

VI. Conclusions and Future Work

Despite the difficulties encountered during the first four in-flight analysis sessions with the CWQMK, the initial results from the SDTO experiment are promising. The CSPE based kit has demonstrated the ability to measure ionic silver and molecular iodine in water samples on the ISS and the error checking and data recovery protocols have proven to effectively mitigate the risk of data loss as a result of ionizing radiation. The accuracy of the measurements remains in question, but the upcoming return and ground analysis of the archived water samples will address this topic. The ground experiments have proven to be a useful tool for evaluating the performance of the flight kit. Several issues were identified in the ground experiments and the project team is working to resolve them through extended shelf-life studies, evaluation of alternate hardware processing, and improvement of the in-flight analysis procedures.

Due to the incomplete data sets collected during the first four in-flight activities, the decision was recently made to resupply the CWQMK. By launching additional monthly analysis packs, the incomplete sessions can be repeated. This will extend the duration of the SDTO experiment and ensure that adequate data is collected to effectively evaluate CSPE technology. Work is also underway to expand the capabilities of the CWQMK by adding the consumables needed to perform total iodine analyses. The project team is also exploring the possibility of converting the CWQMK to an operational flight system, pending the outcome of the SDTO experiment.

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