

Monitoring of the Atmosphere on the International Space Station with the Air Quality Monitor

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During the early years of human spaceflight, short duration missions allowed for monitoring of the spacecraft environment to be performed via archival sampling, in which samples were returned to Earth for analysis. With the construction of the International Space Station (ISS) and the accompanying extended mission durations, the need for enhanced, real-time monitors became apparent. The Volatile Organic Analyzer (VOA) operated on ISS for 7 years, where it assessed trace volatile organic compounds in the cabin air. The large and fixed-position VOA was eventually replaced with the smaller Air Quality Monitor (AQM). Since March 2013, the atmosphere of the U.S. Operating Segment (USOS) has been monitored in near real-time by a pair of AQMs. These devices consist of a gas chromatograph (GC) coupled with a differential mobility spectrometer (DMS) and currently target detection list of 22 compounds. These targets are of importance to both crew health and the Environmental Control and Life Support Systems (ECLSS) on ISS. Data is collected autonomously every 73 hours, though the units can be controlled remotely from mission control to collect data more frequently during contingency or troubleshooting operations. Due to a nominal three-year lifetime on-orbit, the initial units were replaced in February 2016.

This paper will focus on the preparation and use of the AQMs over the past several years. A description of the technical aspects of the AQM will be followed by lessons learned from the deployment and operation of the first set of AQMs. These lessons were used to improve the already-excellent performance of the instruments prior to deployment of the replacement units. Data trending over the past several years of operation on ISS will also be discussed, including data obtained during a survey of the USOS modules. Finally, a description of AQM use for contingency and investigative studies will be presented.

Nomenclature

AQM = Air Quality Monitor
DMS = Differential Mobility Spectrometer
DMSD = dimethylsilanediol

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<i>ECLSS</i>	= Environmental Control and Life Support Systems
<i>GC</i>	= Gas Chromatograph
<i>GC/MS</i>	= Gas Chromatograph/Mass Spectrometry
<i>HMCTS</i>	= Hexamethylcyclotrisiloxane
<i>ISS</i>	= International Space Station
<i>MCA</i>	= Major Constituent Analyzer
<i>MDL</i>	= Minimum Detection Limit
<i>MQL</i>	= Minimum Quantitation Limit
<i>RF</i>	= Radio Frequency
<i>SVT</i>	= Science Verification Testing
<i>TMS</i>	= trimethylsilanol
V_c	= Compensation Voltage
<i>VOCs</i>	= Volatile Organic Compounds

I. Introduction

Monitoring of the spacecraft environment is required to ensure the safety of the crew and the health of the vehicle systems. In the case of the spacecraft atmosphere, this monitoring has long relied on the collection of archival samples and their return to Earth for analysis.^{1,2} For short duration missions, such as those of early missions or the Space Shuttle, this type of monitoring was sufficient. However, with expeditions on the International Space Station (ISS) lasting up to 6 months or a year, it became necessary to develop real-time methods for determining the concentrations of both major constituents (O₂, CO₂, N₂, etc.) and trace contaminants (e.g. volatile organic compounds (VOCs) or combustion products). A recent review has summarized some of the real-time monitoring on the ISS.³ Briefly, the major constituent concentrations aboard the ISS are currently measured using a magnetic sector mass spectrometer, the Major Constituent Analyzer (MCA).^{4,5} Trace VOCs are measured using the Air Quality Monitor (AQM),⁶⁻¹¹ which combines gas chromatography (GC) with differential mobility spectrometry (DMS), a variant of ion mobility, which was used successfully for trace VOC detection for 8 years on the ISS.¹²⁻¹⁶ Two AQMs, containing different GC columns, are currently used to monitor for 22 target compounds, with the targets consisting of 1) those routinely detected in the ISS atmosphere at measureable concentrations, 2) those that would represent a significant hazard if detected, and 3) those that could potentially affect Environmental Control and Life Support Systems (ECLSS). Examples of these classes are 1) acetone, xylenes, and 2-butanone, 2) benzene and acrolein, and 3) ethanol, 2-propanol, and cyclic siloxanes. The current target list is shown in **Table 1**.

Table 1 – Target Compounds for the ISS Air Quality Monitor

Target Compounds	
Acetaldehyde	2-Butanone
Methanol	Hexane
Ethanol	Benzene
Acetone	Hexamethylcyclotrisiloxane
2-Propanol	Octamethylcyclotetrasiloxane
n-Butanol	Decamethylcyclopentasiloxane
Ethyl Acetate	Trimethylsilanol
Dichloromethane	Hexanal
Toluene	Propenal
Xylenes (m,p)	1,2-Dichloroethane
Xylene (o)	Ammonia

II. Experimental

The AQM, the principles behind its operation, and its essential components have been described in detail previously.^{17,18} Each unit, built by Draper Labs, integrates a preconcentrator containing Carboxen 1000, one of 2 GC columns (DB5-ms or VF-624ms, 15 m x 0.25 mm), a microDMx sensor, and a Windows XP computer in a 3kg, 25.4 cm x 15.2 cm x 13.2 cm size package with a nominal peak power demand of 72 W. The instrument is controlled by Draper Expert software using customized methods for sampling times and heating rates. The gas used for both carrier gas and detector make-up gas is ambient air that is drawn into the unit upon power up. This recirculated gas is scrubbed of moisture and VOCs using 2 cartridges containing molecular sieve/activated carbon and Carboxen 549. An additional cartridge containing the same materials is used to flush (purge) the preconcentrator after sampling to remove excess moisture. During operation, an air sample is pulled over the preconcentrator to trap and concentration the VOCs. The preconcentrator is then ballistically heated to transfer the VOCs to the GC column for separation. As the analytes enter the DMS detector cell, they are ionized and exposed to asymmetric radio-frequency (RF) fields that displace the ions perpendicularly to the gas flow. To prevent the neutralization of the ionized analyte on the cell walls, a small DC voltage (compensation voltage, V_c) is applied to steer the ions away from the walls and towards the detector. The value of V_c for an individual compound is related to the interaction of the ion with the RF field.^{19,20}

For preparation of the AQMs prior to flight, the retention times and DMS detector values (RF and V_c) of each of the target compounds were determined to ensure proper peak identification. Typical data obtained from a scan run at an RF voltage of 900 V are shown in **Figure 1**. As the AQM are nominally commercial items, the retention times should be approximately the same from unit to unit, as long as the same type of GC column is being used. Similarly, the V_c required to steer a particular ion towards the detector at a specific RF voltage should not change significantly. However, slight differences are often seen, requiring at least a confirmation prior to calibration. Following this “compound characterization” process, methods are prepared that will allow for automated detection and quantification of the target compounds. These GC methods establish windows in GC retention time where the RF voltage and compensation voltage are set for each target compound to increase selectivity and sensitivity (**Figure 2**).

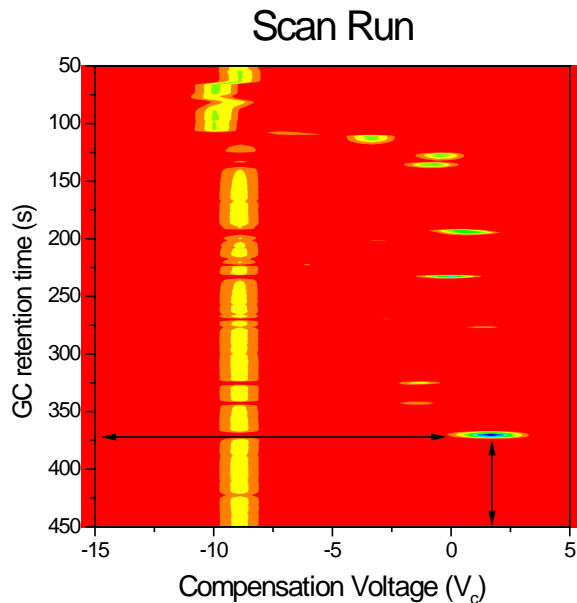


Figure 1 – Scan run of Air Quality Monitor, in which the compensation voltage (V_c) is scanned at a specific RF voltage. This data is used to determine the appropriate RF voltage, V_c , and retention time window to obtain the ideal sensitivity for specific compounds

When the automated methods are set, calibration of the AQMs can begin, with the calibration ranges being specified by the AQM Science Requirements.²¹ As the different GC columns in the 2 AQMs are used to prevent issues with coelutions (as seen previously^{6,8}), calibration is performed with three separate sets of calibration compounds: 1) those separated with the VF-624 column, 2) those separated with the DB5 column, and 3) those that are detected on both units. Calibrations are performed using five concentration levels of each compound (with the exception of ammonia), and at least four runs of each are performed to develop an average response. The lowest concentration mixes are run seven times in order to develop a minimum detection limit (MDL) and minimum quantitation limit (MQL). After obtaining the responses for each target compound at each concentration level, calibration curves are developed, and the look-up tables developed from the curves are loaded onto the AQMs. As the final step of the calibration process, the AQMs must undergo verification testing that validates their accuracy and precision. During this science verification testing, the AQMs are exposed to mixes of unknown concentrations (to the analyst) that mimic the ISS air in that they contain typical levels of major constituents as well as relevant concentrations of the target compounds. Following the analysis of the mixes, the automated results from the AQM are compared to the actual concentrations of the mixes as determined by gas chromatography/mass spectrometry (GC/MS).

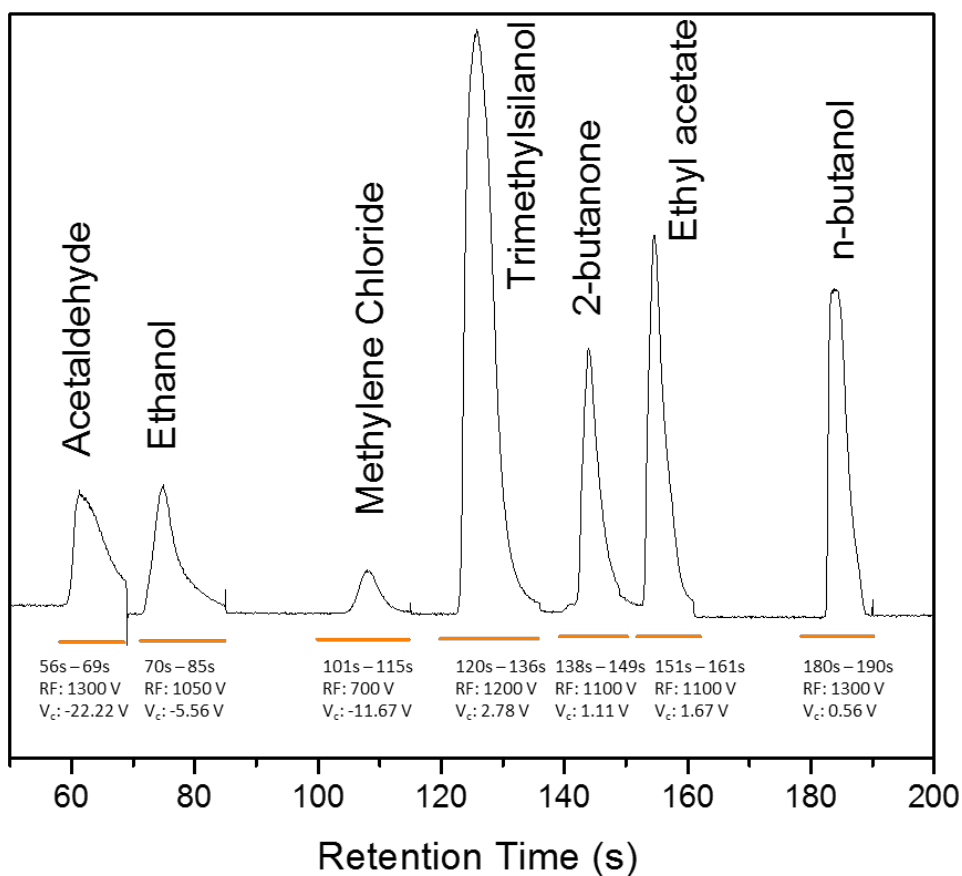


Figure 2 – GC trace from a calibration run showing the detection of 7 polar compounds in a single run. Below each peak are the retention time windows, in which a specific RF voltage and V_c are set for the individual compounds.

III. Results and Discussion

During preparation of the first set of AQMs delivered to the ISS in March 2013, issues arose with the calibration and verification of several compounds.⁹ First, due to its extremely polar nature, calibration of the AQMs for ammonia detection was problematic, especially over a wide range. However, as seen in **Figure 3**, when ammonia is present, it produces a visible change in the output of the AQM. As such, ammonia was listed as a “trending” compound. Even so, this ability to determine present/not present was shown to be useful in an operational situation. On January 14, 2015, an ammonia release alarm occurred on the ISS. This alarm resulted in the crew donning masks, evacuating to the Russian segment, and closing the hatches. While it was later confirmed by the use of Drager tubes that there was no ammonia present, the AQM was one of several on-board, real-time instruments that were used during the day to confirm that no ammonia was present.²² With the extreme sensitivity of the AQM to ammonia and the fact that any significant increase of the ammonia concentration would be detected, the ability to determine the absolute low-level concentration is not necessary; therefore, this compound has remained qualitative-only for the current AQMs.

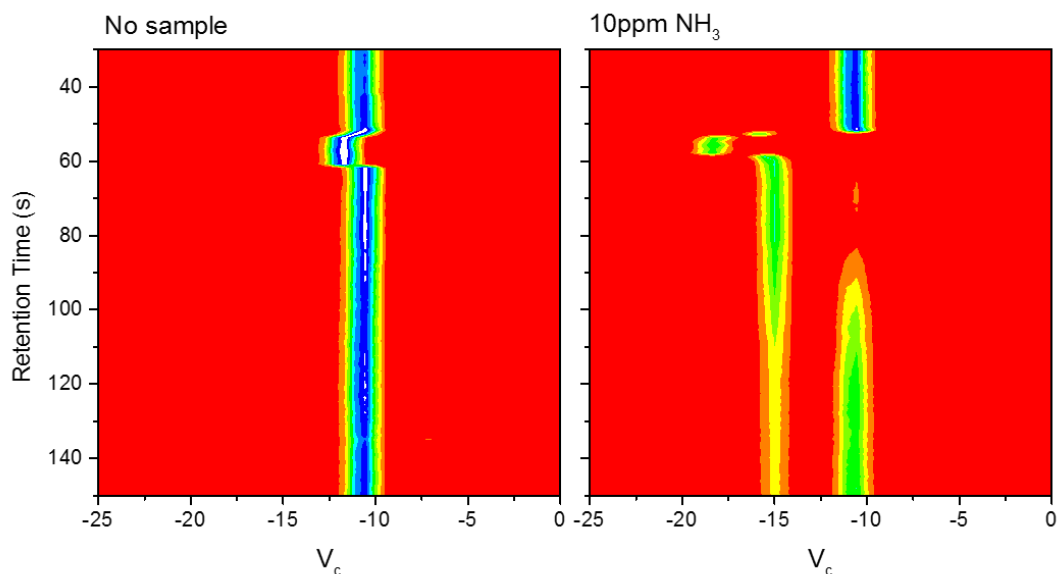


Figure 3 – Change in AQM scan run observed with the addition of 10 ppm ammonia. The addition of ammonia produces a clear change in the spectrum, allowing for the trending of this compound.

The second compound to cause an issue during calibration and ground verification of the first set of AQMs was hexamethylcyclotrisiloxane (HMCTS). As with ammonia, this compound is extremely sticky, and losses in both the mix canisters and sample lines are to be expected. Additionally, the method used for preparation of the mixes (permeation tubes) was unreliable, leading to questions as to the actual concentrations in the canisters. Again, as with ammonia, though, it was shown that HMCTS could be trended. For the most recent delivery, however, new methods of standard preparation and exposure of the instruments to HMCTS led to their successful ground verification for that compound. In the case of the standard preparation, the permeation tube method was replaced by a method that sampled the headspace above the solid compound at different temperatures.²³⁻²⁵ Taking advantage of the change in vapor pressure with temperature resulted in more reproducible concentrations in the standard canisters. During both calibration and science verification testing, a new process for sample introduction was also used. Previously, when exposing the AQMs to sample, the canister would be opened and the sample pump of the AQM would be started. After a 10 second delay, the inlet valve would open to pull in a sample. It was noticed, however, that the response of several compounds (specifically the cyclic siloxanes) would increase with each additional run, even though the concentrations were not changing. For preparation of the current units, however, when changing

standard concentrations, the standard was allowed to flow through the lines for at least 1 minute prior to starting sample collection. **Figure 4A** shows the calibration runs of the 2nd lowest concentration of HMCTS for AQM 1004 (1st delivery) while **Figure 4B** shows the calibration runs of the same concentration of HMCTS for AQM 1018 (2nd delivery). These figures show 2 important points. First, the passivation of the sample lines allows for much more reproducible detection of HMCTS. Secondly, while sensitivities between units are expected to vary somewhat, the substantial difference between the HMCTS intensities confirmed that the permeation tube method of standard preparation was not providing the theoretical concentration, as later confirmed by GC/MS analysis.²³

The final compound to cause difficulties during the initial flight preparation was acetaldehyde. During calibration, this compound performed very well, with acceptable accuracies and precision. However, during the final check of the units prior to delivery to the ISS, Science Verification Testing (SVT), the accuracy requirement was not met using the automated methods due to movement of the acetaldehyde peak.⁹ However, manual integration of the acetaldehyde peak following a recalibration showed that, at a minimum, the concentrations of acetaldehyde could be trended. With this information, a waiver was obtained for the acetaldehyde accuracy requirement for delivery,²⁶ and the compound was declared as “trending.” Once on the ISS, the position of the acetaldehyde peak stabilized and passed the validation process,¹⁰ thereby allowing the waiver to be retired and the AQM to be declared validated for this compound.

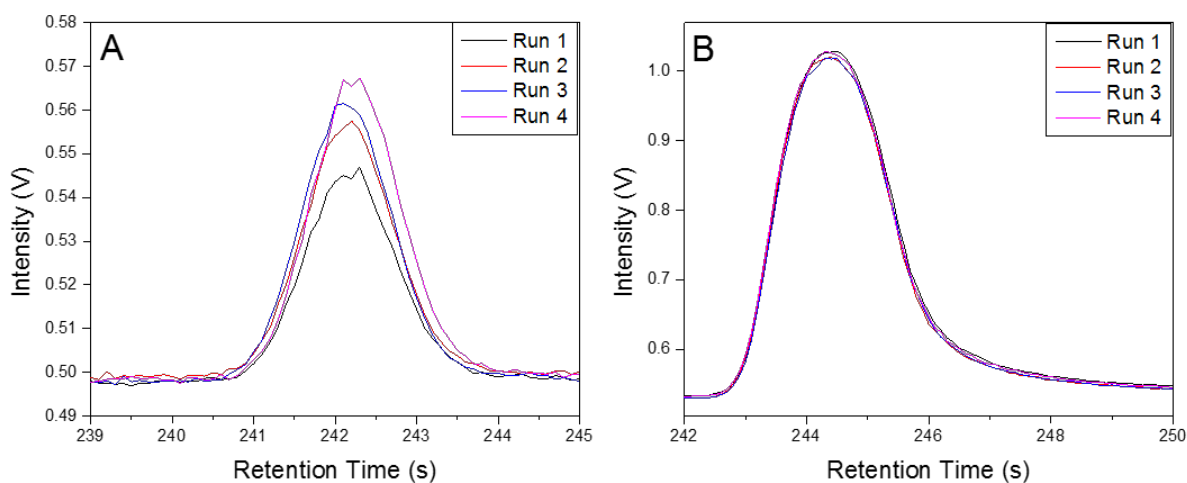


Figure 4 – Calibration data for HMCTS obtained on A) AQM 1004 and B) AQM 1018 showing the effects of changes in processing of the units and standard preparation

The challenges experienced during the initial AQM delivery provided insights into potential improvements in the future flight processing of the units. During this delivery, the calibration/ verification of the units occurred in the Toxicology Laboratories at NASA Johnson Space Center, while other processing occurred off-site at the Wyle (now KBRwyle) Fabrication and Testing Lab. This led to a number of transfers of the AQMs, which could have exposed the units to contamination, thereby affecting the detection of specific compounds (e.g. acetaldehyde). For the second delivery of the AQMs, all processing, including calibration/verification, was performed in the off-site laboratories. In this case, while the instruments were not moved from site to site during processing, though, the surroundings were not necessarily “controlled,” as other hardware was being processed in the same (large) area. Processing of the other hardware for flight often involved cleaning with isopropanol, which, while not noticeable to the AQM team, caused there to be a background of this compound during calibration. During SVT, when all other work in the lab had been stopped for the testing and the background was removed, the isopropanol results were below the required accuracy limit. The resulting investigation showed the presence of the background and the AQM was recalibration for isopropanol, after which it passed its verification requirement.

A final change for the second delivery of AQMs to the ISS regarded the calibration range for ethanol. The upper level requirement for ethanol detection by the AQM is 7 mg/m³. While this value is well below the Spacecraft

Maximum Allowable Concentration (2000 mg/m^3)²⁷ and the historical average ISS ethanol concentration, consistent atmospheric ethanol concentrations at this level could adversely affect the Catalytic Reactor of the U.S. Water Processor Assembly (WPA).²⁸ This calibration range was sufficient until mid-2014, at which point it began to trend upward with occasional spikes above the upper calibration point of the AQM ($\sim 7.5 \text{ mg/m}^3$). Eventually, the ethanol concentration was consistently above this calibration point, and manual integration of the peak was required to estimate the actual concentrations due to changes in peak shape/position (**Figure 5**). To prevent a reoccurrence of this, a secondary ethanol calibration was performed during the processing of the second set of AQMs. While the “normal” calibration covered the range of $\sim 0.2 \text{ mg/m}^3 - 8.0 \text{ mg/m}^3$, the secondary calibration covered a range of $\sim 6.3 \text{ mg/m}^3 - 13.4 \text{ mg/m}^3$. To not overload the AQM, the normal 5 second sample length was shortened to 3 seconds. The other parameters were very similar. Since the delivery of the new AQMs to the ISS in February 2016, the ethanol concentration has generally trended downward, with the levels now well into the range of the regular calibration (**Figure 6**).

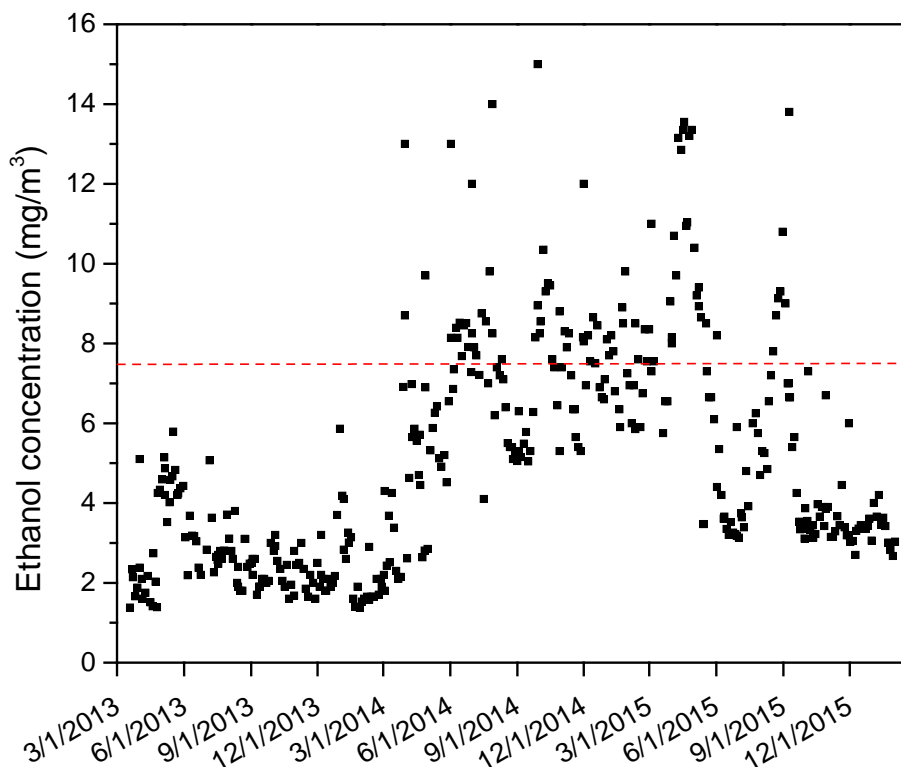


Figure 5 – ISS atmospheric ethanol concentration as measured by AQM 1004. The dashed red line indicates the highest calibration point of AQM 1004.

As discussed previously,¹⁰ once on board the ISS, the first AQMs underwent a validation process in which the results obtained from the units was compared to those from archival samples obtained simultaneously. At the end of the validation period, eighteen compounds met the validation criteria, while three (n-butanol, trimethylsilanol, and decamethylcyclopentasiloxane) did not. After analysis of the data, it was determined that there was an offset of a factor of two for n-butanol and trimethylsilanol, so the AQM measurements could be used for the assessment of air quality. For decamethylcyclopentasiloxane, however, there was no simple explanation for its behavior in comparison to the archival samples, so it was used only for trending. With the delivery of the second set of AQMs, the improvements in calibration processes and standard preparation described for HMCTS allowed for the correction of these issues, and the AQM results for all target compounds are being used for air quality assessment on the ISS.

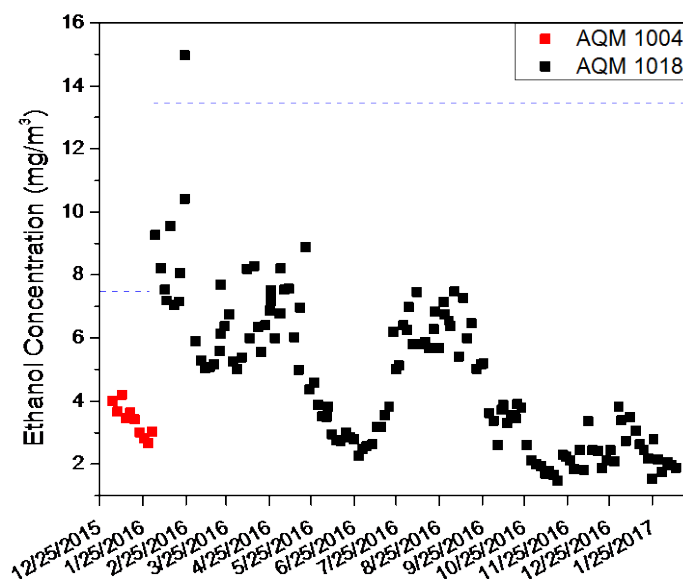


Figure 6 – ISS atmospheric ethanol concentration measured by AQM 1004 (red) and AQM 1018 (black). The dashed blue lines represent the highest calibration points for the units. Note the much higher calibration range for AQM 1018 obtained by adding a secondary calibration for high ethanol.

Over the lifetime of the ISS, the air has appeared to be fairly well mixed based on concurrent archival sampling in different modules. However, this determination was generally based on monthly snapshots of the VOC load. With the validation of the AQM, a survey of the U.S. segment was undertaken to confirm the mixing over an extended period. During this survey, one AQM remained in the U.S. Laboratory while the other unit was moved to the Columbus and Japanese Pressurized Modules for 60 days each. This process allowed for a longer semi-weekly assessment of these modules and also allowed for an archival sample to be obtained for comparison with the AQM results. The concentrations of acetaldehyde and 2-propanol obtained over the course of the survey are shown in **Figures 7 and 8**, respectively, along with the corresponding archival sample values. As can be seen, there are no significant changes upon movement of the units to different areas of the U.S. segment of the ISS. The results for other compounds greater than trace levels are similar, confirming that there is little deviation in contaminant concentrations between modules. Based on these results, it was determined that no further AQM surveys were needed.

While the use of the AQMs in a contingency situation was described above regarding the potential ammonia leak on the ISS, the AQMs have also successfully provided information to engineers in an ongoing ECLSS investigation aimed at lowering the concentration of dimethylsilanediol (DMSD) in the humidity condensate of the WPA to extend the life of the on-board multifiltration beds. It has been proposed that silicon-based compounds in the atmosphere are converted to DMSD on the condensing heat exchangers.²⁹ As such, air scrubbers containing activated charcoal were installed in Node 1 of the ISS on May 12, 2015 to remove these organo-silanes.³⁰ Using only archival samples, it could have taken up to 6 months for any information to be gained as to the success of the filters. However, with the AQMs collecting data twice a week and the data being downloaded and analyzed weekly, ECLSS engineers were able to determine very quickly that the filters were successfully removing siloxanes and trimethylsilanol (TMS) from the air. **Figure 9** shows the dramatic change in TMS concentration following the installation of the filters, a change that was observed for the other organo-silicon compounds targeted by the AQM. This testing has continued with the current AQMs. The installation of the air scrubbers and the subsequent reduction in atmospheric siloxanes did not have a corresponding decrease in the DMSD concentration in the humidity condensate, which led to a suggestion that there were perhaps higher localized siloxane levels near the condensing heat exchanger. On May 2, 2016, an AQM was moved to Node 3 to analyze for siloxanes while a dry-

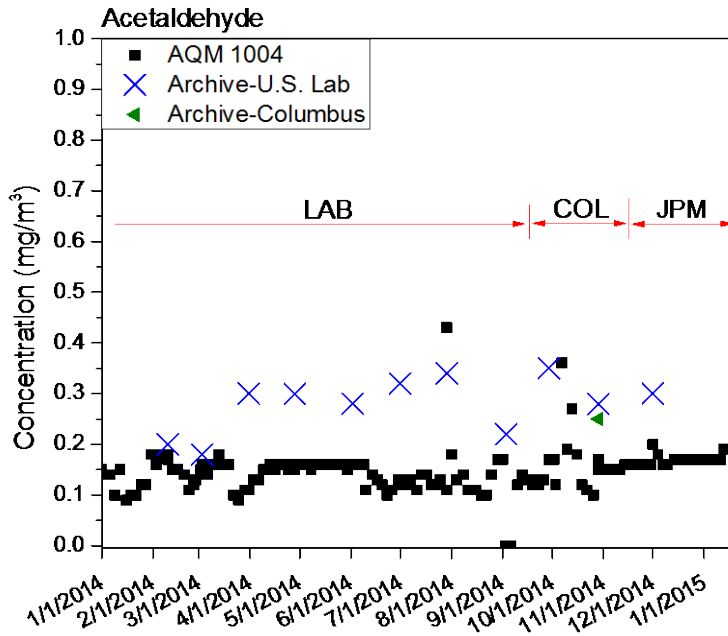


Figure 7 – Acetaldehyde concentrations measured by AQM 1004 during the survey of the U.S. Operating Segment of the ISS

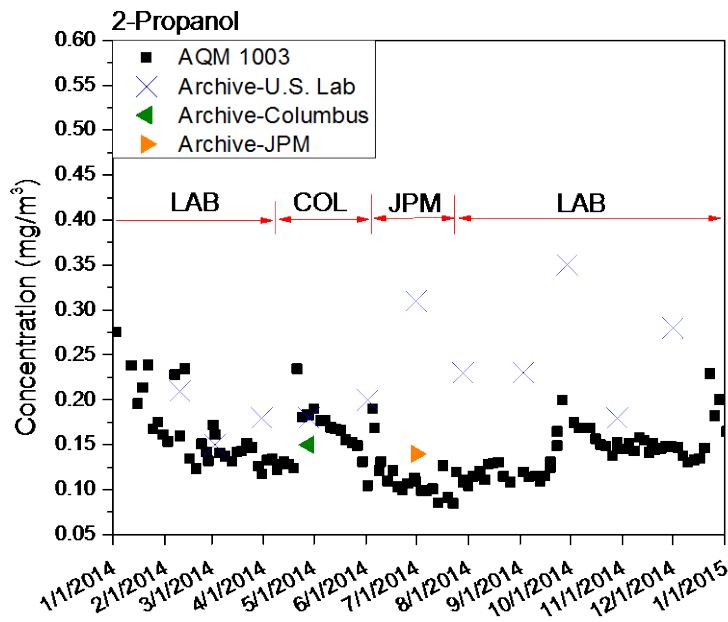


Figure 8 – 2-propanol concentrations measured by AQM 1003 during the survey of the U.S. Operating Segment of the ISS

out cycle was being performed on the heat exchanger. However, the siloxane concentrations in Node 3 were actually found to be lower than those in the U.S. Lab. Again, though, the ability to gain near real-time insight into the atmosphere allowed the ECLSS engineers to begin to think about other possibilities.

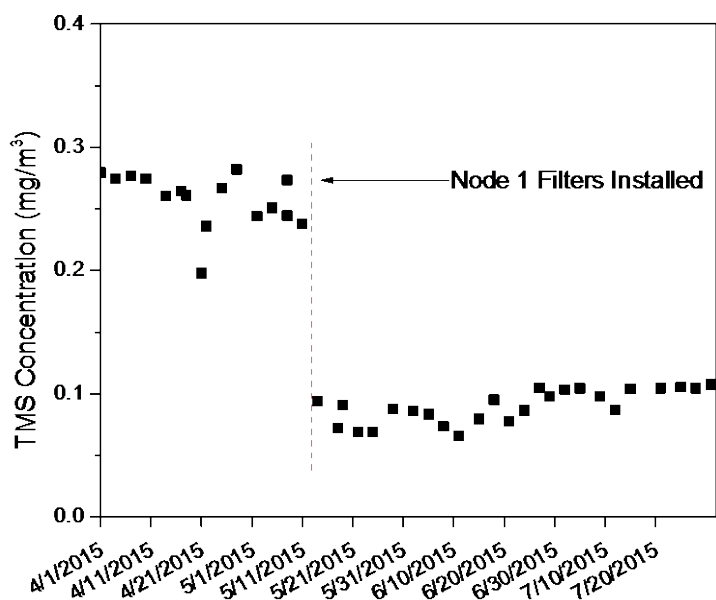


Figure 9 – Trimethylsilanol concentrations measured by AQM 1004. Air scrubbers containing activated carbon were installed in Node 1 to remove organo-silicon compounds on May 12, 2015. This figure shows the dramatic effects on the TMS concentrations, an effect that was also seen for the other siloxane compounds.

IV. Conclusion

The operational Air Quality Monitor was delivered to the ISS in early 2013. During 3 years of operation, the units were successfully validated and showed their usefulness in nominal, contingency, and investigative situations. While the AQMs performed well during their initial deployment, there were some lessons learned that were applied during the preparation of the second set of AQMs that were activated on-board in early 2016. These changes included an expanded calibration for ethanol and improved calibration standard preparation and processes. The new units continue to provide high-quality data on the atmosphere of the ISS and have proven to be reliable, always available to address issues related to the atmosphere.

Acknowledgments

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