Further Characterization of Aerosols Sampled on the International Space Station

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Spacecraft cabin air quality is of fundamental importance to crew health, with concerns encompassing both gaseous contaminants and airborne particles. Quantification of spacecraft indoor aerosols will increase our understanding of crew exposure and cabin cleanliness. Aerosols on the International Space Station (ISS) have been sampled and brought back to Earth for analysis to characterize the airborne particulate matter in the cabin. Microscopic analyses have been performed to determine morphology and particle size information, and Energy Dispersive X-ray Spectroscopy (EDS) provides information on the chemical elements present in the particles. With the use of IntelliSEM software[1] for computer-controlled scanning electron microscopy (CCSEM), this data provides particle size distribution information and statistics on particle materials. Many of the particles collected were made up of multiple elements and had uncommon morphologies compared to typical indoor aerosols on Earth. These characteristics are thought to be from unique formation mechanisms in the microgravity environment. Several notable particle types are examined further in this work. Bromine-containing particles and cadmium-containing particles are discussed, as they constitute a health hazard to crew members. Humans in indoor living and working spaces are typically the single largest particle emission source, and this was observed in the sampled aerosols in ISS as well.

Nomenclature

AAS	=Active Aerosol Sampler
ACGIH	=American Conference of Governmental Industrial Hygienists
ARED	=Advanced Resistive Exercise Device
CCSEM	= computer controlled scanning electron microscopy
COTS	= commercial-off-the-shelf
DUST	= Divert Unwanted Space Trash, experiment on ISS
EDS	= energy dispersive x-ray spectroscopy
HEPA	= high efficiency particulate air
ICP-MS	= inductively coupled plasma mass spectrometry
ISS	= International Space Station
PAS	= Passive Aerosol Sampler
PMM	= Permanent Multipurpose Module
SEM	= scanning electron microscope
TEM	= transmission electron microscope

I. Introduction

THE Aerosol Sampling Experiment took place in Increment 50/51 on the International Space Station (ISS) in December 2016. A repeat of the experiment has been completed on ISS in Increment 56, and the analysis of the second set of samples is ongoing. The complete description of the sampling hardware and experiment protocol is

[1] Certain commercial software, equipment, instruments, or materials are identified in this paper to foster understanding. Such identification does not imply recommendation or endorsement by the National Aeronautics and Space Administration (NASA), nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

provided in references 1 and 2. The Passive Aerosol Sampler (PAS) design created for this experiment was based on placing sampling substrates in the flow path of "dirty" air flowing into the ISS vents, to collect particles for up to five different sampling durations in the same location. Particles landed on five aluminum substrates covered with double coated black carbon conductive tape (Ted Pella, Inc., P/N 16084-8). The optimal sampling duration to get particle coverage appropriate for microscopic analysis varies by ISS location, so multiple substrates in one sampler provided different particle loadings to choose from as well as redundancy to reduce risk. The operations in Increment 50/51 collected particles for 2, 4, 8, 16 and 32 days with the ideal samples mostly in the 16 and 32-day durations. PAS J is shown in Figure 1 in the US Lab on the HEPA filter, with all five sampling substrates exposed.

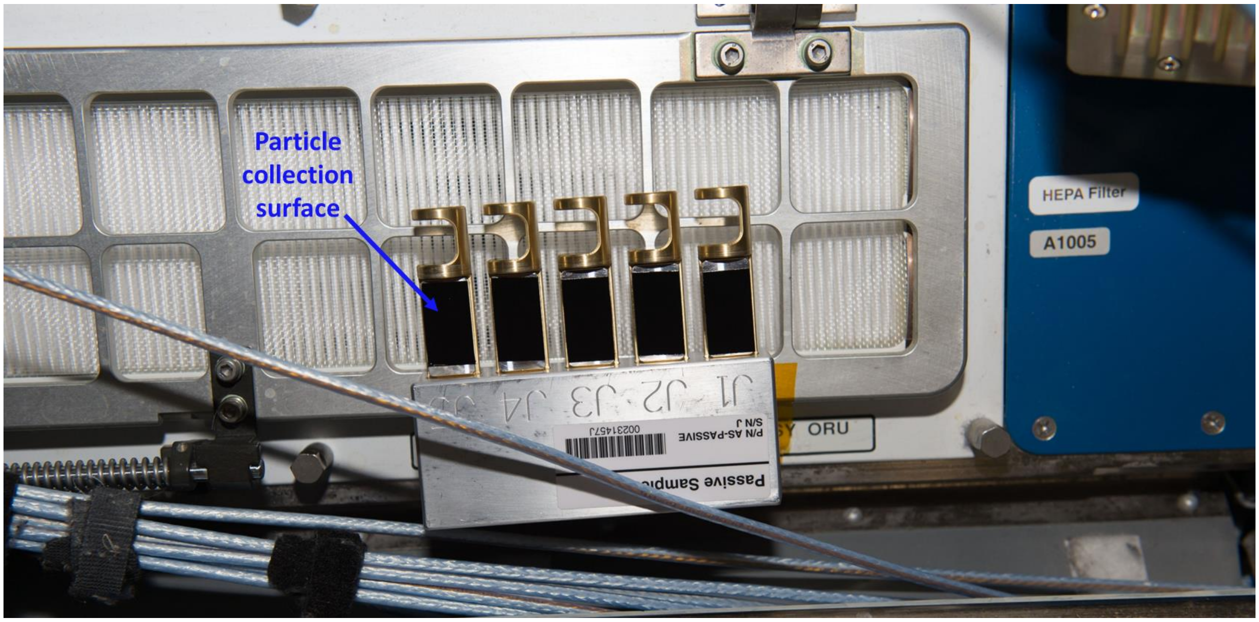

Figure 1. Passive Sampler J is mounted on the edge of the HEPA filter in the US Lab in ISS during the Aerosol Sampling Experiment in Increment 50/51. Note that a Nomex screen covers the filter pleats to protect against damage that may occur when the crew vacuums ISS vents.

The PAS collected larger airborne particles in the size range from tens of micrometers to hundreds of micrometers in diameter, which approach the filter with velocities on the order of 0.5 to 1.0 m s^{-1}. Each substrate acts as an impactor, taking advantage of the inertia of larger particles which collide with the substrate and are held in place by the tape adhesive. Smaller particles in the nanometer range do not deposit on the substrates, but follow the air stream around the substrates, and enter the filter. A different collection method is necessary for the smallest size range of particles because they do not impact on surfaces, but rather, follow the streamlines of the air flow around obstructions. Therefore a commercial-of-the-shelf (COTS) thermophoretic aerosol sampler was used, which can capture particles as small as 5 nm. The Active Aerosol Sampler (AAS) was a TPS100 Personal Thermophoretic Sampler[3] (RJ Lee Group) adapted for low gravity, which sampled for up to 7 hours at 5 ml/min in ISS locations adjacent to the PAS vent locations. The experiment did not provide any real-time information on particle concentrations or characteristics, only samples, which were returned to Earth for subsequent analysis. AAS results are covered elsewhere.[2] A subset of the most recent characterization efforts are included in this work.

II. Unique Particle Morphology

Unique particle morphologies were observed in the particles collected by the PAS. Multi-component particles were abundant, consisting of individual metal particles embedded in a carbonaceous matrix. This complexity presented challenges for common image analysis methods for counting particles, which determine the edges to outline individual particles based on contrast thresholds in the microscopic image.

A. Particle Aggregation in Microgravity

It is unknown whether many individual particles agglomerated after becoming airborne on ISS, or whether the parent materials that generated the particles were composites to begin with. Some insight can be gained from informal aggregation experiments performed in microgravity.[4] These experiments were intended to shed light on planet formation mechanisms, and were performed on ISS during two different Space Shuttle expeditions. Several types of particle materials were mixed in different types of bags in microgravity. Particle aggregation was observed to different degrees under different conditions. The sizes of the particles were between 100 and 7000 micrometers (7 mm). The lower end of this size range matches particles that were collected on ISS in the Aerosol Sampling Experiment, so results can be considered relevant and can shed light on possible particle formation mechanisms on ISS. Particle materials in the aggregation experiments were at ambient pressure (the ISS indoor environment) and included the materials NaCl, Coffee, Sucrose, which are crystalline and noncrystalline solids. Some particles were observed to aggregate with strong cohesion, even at low number densities at time scales on the order of seconds. It was observed that smaller particles aggregated more quickly and with higher cohesive strength. In terms of morphology, angular particles aggregated readily whereas round smooth particles did not. Overall, it was concluded that electrostatic forces dominate the process. Mechanically generated particles are more likely to have high levels of charge. Sodium chloride (which is apparent in many of the captured aerosols) and polymeric aerosols are also more likely to exist in a charged state than conducting materials.[5,6] The conclusions from these informal experiments indicate that the formation mechanism of many sampled ISS aerosols is likely agglomeration of primary particles in the diameter range from sub-micrometer to tens of micrometers, by electrostatic forces.

An example of a complex particle captured in the US Lab is shown in Figure 2. This particle is an agglomerate of four distinct material classes, with metals (high atomic number elements) appearing brighter than carbon and other low atomic number materials. The complex structure is dominated by a metal wear particle, which is cadmium-rich.

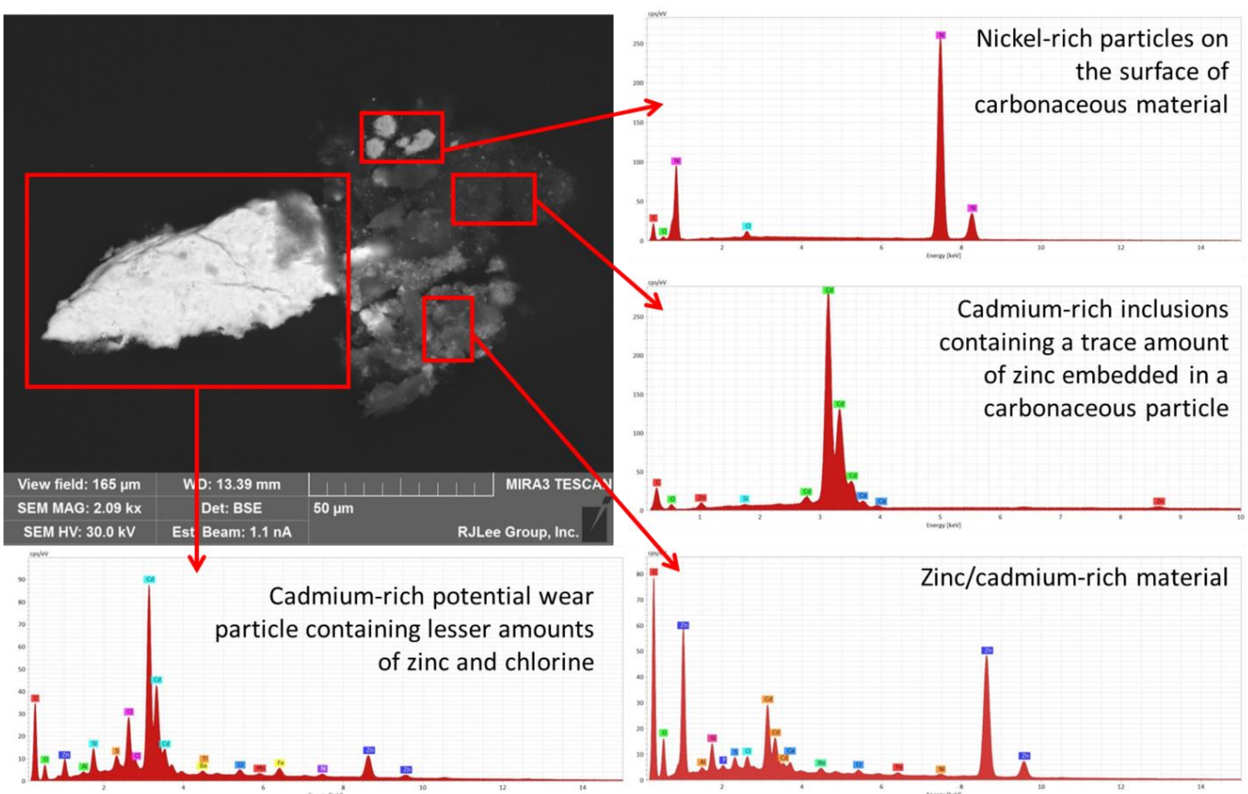

Figure 2. A complex structure consisting of a metal wear particle (predominantly cadmium) attached to three different types of particle materials, including nickel-rich particles, cadmium-rich inclusions in a carbonaceous matrix, and zinc-cadmium-rich material. This particle was captured in Node 3.

Three distinct metal particles at the top of the micrograph are nickel-rich (upper EDS spectrum) and are on the surface of a carbonaceous material. The EDS spectrum of adjacent material shows very small cadmium-rich inclusions in a carbonaceous matrix with a trace amount of zinc. The lower right EDS spectrum shows zinc- and cadmium-rich material below the carbonaceous portion. This complex morphology may be explained in part by the microgravity aggregation experiments.

B. Electron Microscopy Analysis Technique

High-resolution micrographs from manual microscopy on the PAS samples were augmented with computer-controlled scanning electron microscopy (CCSEM). This technique uses scanning electron microscopy (SEM), energy dispersive x-ray spectroscopy (EDS) and the IntelliSEM software (RJ Lee Group) to locate size and characterize particles efficiently. The individual bright metal particles within complex structures were analyzed individually for two reasons. First, if analyzed as a single particle, the agglomerate in Figure 2 would be very difficult to categorize in terms of materials. Second, the edges of high atomic number materials have a very high contrast to the grey-scale of the carbon materials and the black carbon tape, and are well-suited for edge-detection algorithms. The initial IntelliSEM analysis approach for these samples included every separate metal particle or inclusion observed with atomic number of titanium and higher, with the lower size limit of the analysis set to 1 micrometer. When these particles were sized and counted in IntelliSEM, the result was not total particle counts, but rather an analysis of individual metal particles present in larger multi-component particles.

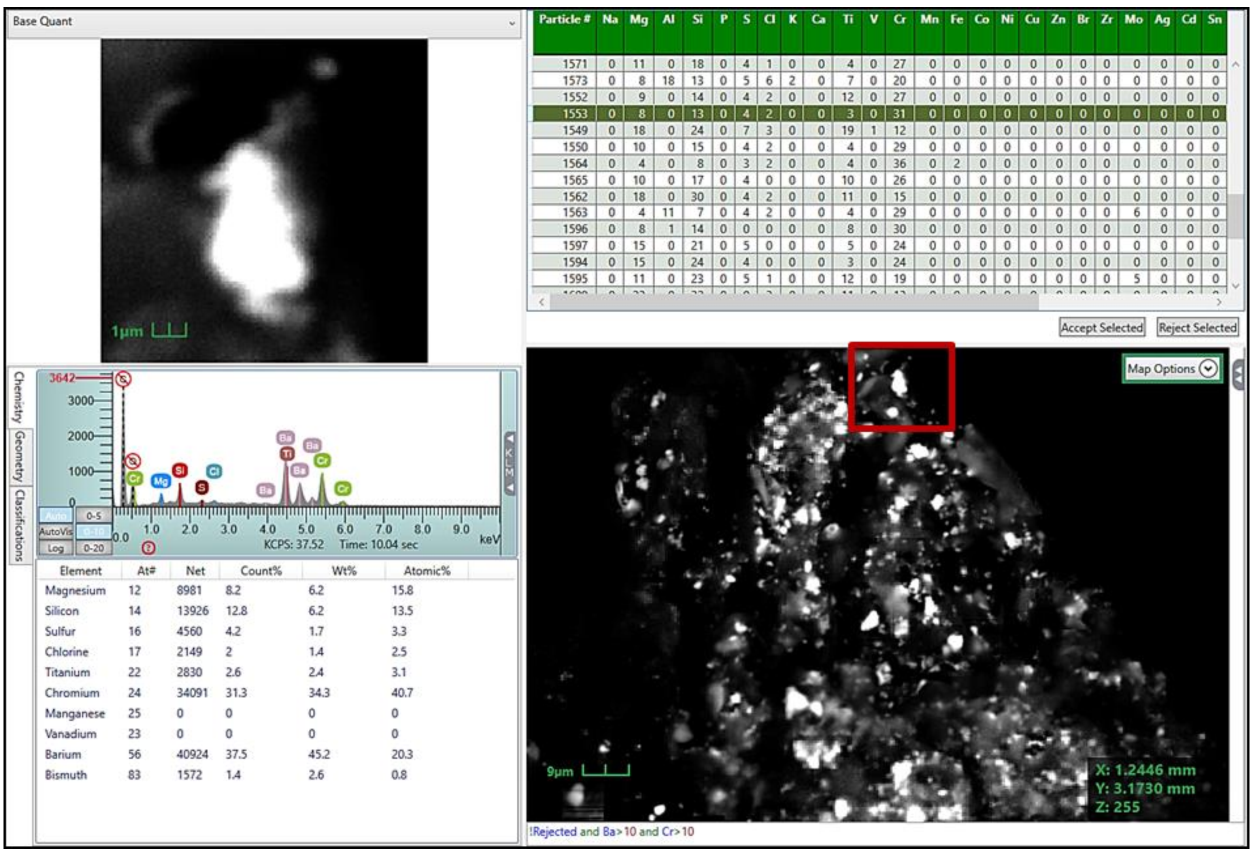

Figure 3. One view of the IntelliSEM graphical user interface, showing the particle statistics of the metal particle analysis (upper right), a multi-component particle with many individual metal particles in a carbon matrix (lower right), a barium chromium-rich particle which was analyzed in the IntelliSEM size distribution analysis (upper left), and the EDS spectrum of the barium chromium-rich particle (lower left).

An example of the IntelliSEM graphical user interface for one metals analysis is shown in Figure 3, which has a low-resolution image of a particle in the upper left quadrant. The table in the upper right quadrant has particle statistics of elements present in each particle (x-ray counts from EDS) with the dark row highlighting the chemistry of the particle to the left which is assigned number 1553. The EDS spectrum of this particle is shown in the lower left quadrant and the larger field of view of the multi-component particle is in the lower right quadrant, with particle number 1553 contained in the red box. The coordinates of this view are in the lower right hand corner of the image, which allows the particle to be relocated in the microscope to obtain a high-resolution image at a later time, or for some other analysis to be performed, such as Raman spectroscopy.

The metals analysis relies on user-defined classes of element combinations, which are based on frequent element combinations observed over all the particles analyzed. The total number of particle material classes must be kept to a reasonable amount, for example, the cadmium-rich inclusions shown in Figure 2 may be placed in the material class 'cadmium-rich' rather than one called 'cadmium-zinc' so the trace amount of zinc embedded in the carbon matrix is omitted from the category name. This methodology for characterizing metal particles is the basis for the next section, which investigates some notable particle types.

III. Notable Particle Types

Several notable particle types are explored further because they are of interest from a human health perspective, or they are of interest because of their abundance. Particles containing bromine and cadmium fall into the first category. Particles associated with human biology are plentiful and indicate that the crew members themselves account for the largest single emission source on ISS.

C. Bromine Particles

Bromine is an element primarily used as a fire retardant. It is incorporated into materials in the form of organohalogenated compounds that can contain chlorine or bromine, and can also be used in combination with inorganic fire retardants containing aluminum, magnesium or antimony.[7] Brominated flame retardants are added to many classes of consumer products, such as upholstery foam, carpet padding, consumer electronics, and throughout the interiors of automobiles, etc. The environmental persistence and bioaccumulation of halogenated fire retardants in the food chain on Earth is well documented, and can result in human endocrine disruption, reproductive system toxicity, cancer and other health effects.[7,8,9]

Some fire retardants can volatilize off plastic surfaces, a process that is accelerated for heated surfaces, such as laptops. These vapors can condense on nearby surfaces and in the dust which accumulates on the surfaces. Mechanical abrasion generates fire retardant-containing particles with normal handling, and weathering or environmental effects, such as thermal cycling or aging, and even radiation, can increase emission rates. On Earth, these particles would settle on furniture or carpets, but in low gravity, they can agglomerate with other particles that swirl around with the ventilation flow and land on the air intakes. Vacuuming dust from ISS vents (or carpets on Earth) disturbs the particles and some will be re-aerosolized. Thus, dust is a routine human exposure route for fire retardants both on Earth and in a spacecraft cabin. Literature studies with material from vacuum cleaner bags show that bromine is consistently found in dust from living spaces.[7,8,9,10] Concentrations varied widely between households. Wagner et al. performed microscopy and found multiple types of discrete bromine particles within dust samples, some as uniform mixtures within a parent material and others as multi-sourced agglomerates.[7] Morphologies included both smooth and angular features, and diameters of individual bromine inclusions ranged from 20 to 100 micrometers.

Two micrographs of bromine-containing particles are shown in Figures 4 and 5 with their corresponding EDS spectra. Figure 4 appears to have randomly oriented rod-like particles and/or flakes containing bromine distributed over a carbon matrix. Figure 5 has a 5 micrometer round bromine particle adhered to what appears to be a skin flake. In both examples, the size of the bromine particles fall within the size range of inclusions in Wagner et al. The angular edges of the approximately 300 micrometer particle in Figure 4 indicate that it could have been abraded or fractured from a plastic parent material that contains bromine fire retardant dispersed in the carbon matrix. The single bromine-rich particle in Figure 5 appears separate from any parent material. Its small size (approximately 5 micrometers) and round morphology does not indicate mechanical abrasion as a generation mechanism, so it may be representative of a homogeneous solid-phase fire retardant that was added to a raw material along with other additives.

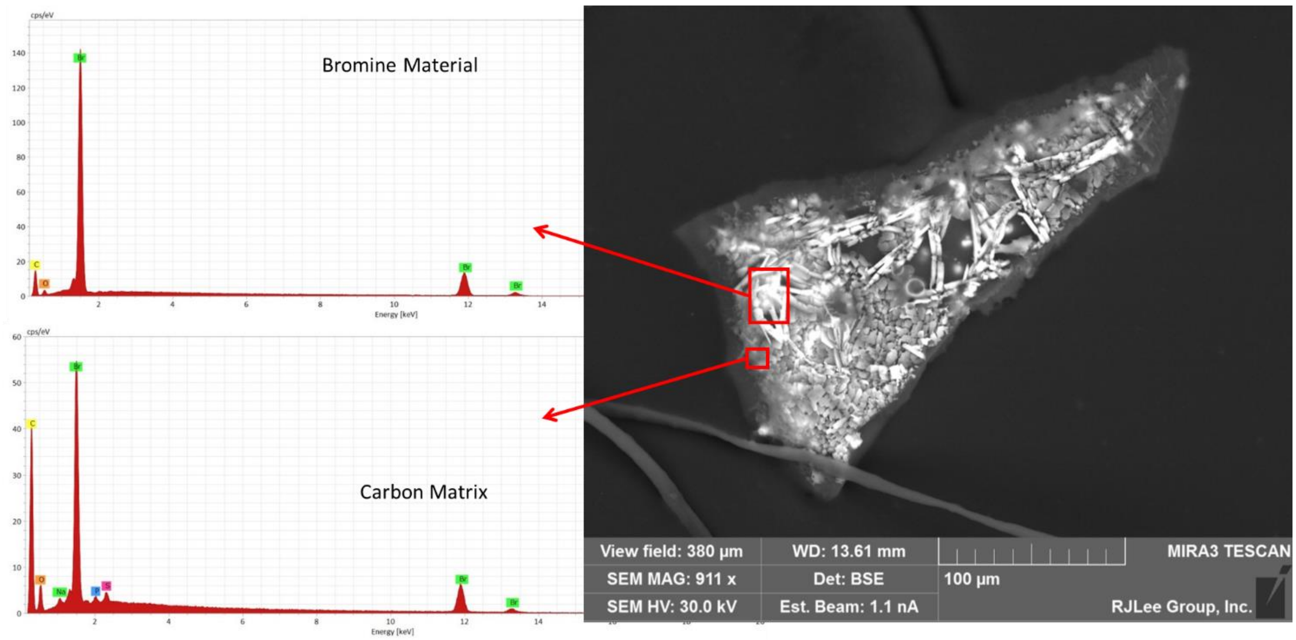

Figure 4. Bromine material associated with a bromine-enriched carbon matrix, captured in Node 3.

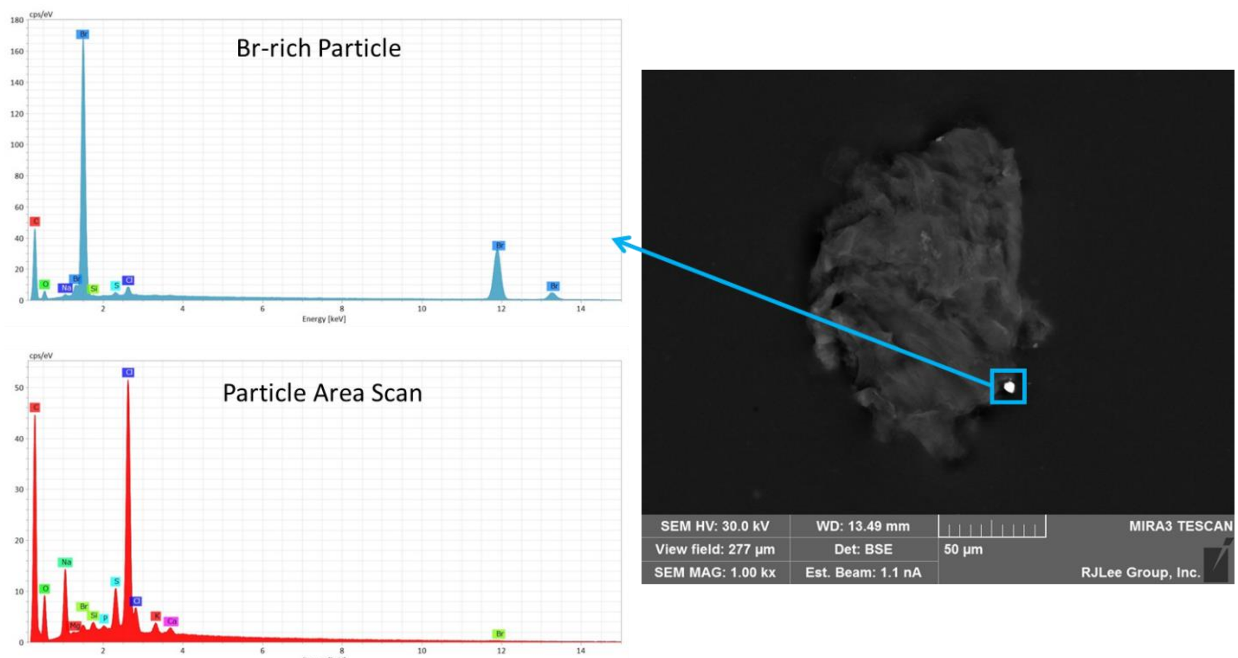

Figure 5. A bromine-rich particle adhered to a carbon particle enriched with NaCl, captured in the Permanent Multipurpose Module.

In the IntelliSEM metals analyses performed for every passive sampling location, the bromine-rich and bromine-bearing particle material classes are summed to give an estimate of the quantity of bromine in the ISS airborne particulate matter. Table 1 shows the relative abundance of bromine as a percentage of the total number of particles collected in all sampling locations during the first Aerosol Sampling Experiment (December 2016).

Table 1: Bromine-containing Particles Sampled on ISS

Passive Sampler	Location	Sample Duration, days	% Bromine Particles, by Number
B-16	Node 1 Deck 1	16	0.5
D-16	Node 3 Deck 3	16	8.1
E-32	PMM	32	0
F-32	Node 2 Deck 2	32	9.3
G-8	Node 3 Forward 3	8	1.7
J-32	US Lab Bay 1	32	0
K-16	US Lab Bay 3	16	1.2

At the the Node 3 Deck 3 and the Node 2 Deck 2 sampling locations, bromine-containing particles made up 8.1% and 9.3% of all particles counted in the IntelliSEM metals analysis. There are most likely items containing fire retardants that are sources in the vicinity of these sampling locations that are regularly manipulated, resulting in particle emissions. In the two locations on ISS with the least amount of total particle deposition, the Permanent Multipurpose Module and the US Lab filter at Bay 1, zero bromine particles were collected during the 32-day sampling duration. The remaining ISS sampling locations had bromine in 0.5 to 1.7 percent of all the particles analyzed. Similar to Earth-based living spaces, the concentration of bromine particles in ISS air is nonhomogeneous, and future sampling results are expected have better statistics for many of the same locations. These results show that the bromine particle emission rates on ISS are relatively low compared to the abundance of other particle material classes in the analysis.

D. Cadmium Particles

Cadmium is a metal used in pigments, platings, and coatings, and was the electrode material for nickel-cadmium batteries before lithium-ion batteries became prevalent. Its use has decreased significantly as it bioaccumulates and long-term exposure causes serious health effects in the lungs, bones and kidneys and it is a probable human carcinogen.[11,12,13] Minor exposure to cadmium dust can cause local skin or eye irritation. Workers mainly in manufacturing and refining are at risk, but cigarette smoking and indoor dust can be a source of daily exposure.[14] Several studies have shown that cadmium levels are higher in smaller size fractions of dust. One study of office and household dust from domestic vacuum cleaner bags showed that cadmium levels were higher in particles less than 75 micrometers in diameter, compared to larger particles collected in the study (up to 1000 micrometers).[15] Another study had higher similar results for particles less than 63 micrometers (versus particles up to 150 micrometers).[16] Older buildings had higher cadmium concentrations than newer construction.[15] The cadmium metal wear particle in Figure 2 is consistent with the small particle size bins of the previously mentioned studies. It was captured in Node 3 of ISS near the Advanced Resistive Exercise Device (ARED). Each crew member typically uses this equipment for up to 2 hours daily and metal particles are inevitably generated with mechanical wear. The source of the particle in Figure 2 may be one or more ARED components with cadmium plating.

In the IntelliSEM metals analyses performed for every passive sampling location, the cadmium-rich and cadmium-bearing particle material classes are summed to give an estimate the quantity of cadmium in the ISS airborne particulate matter. Table 2 shows the relative abundance of cadmium as a percentage of the total number of particles collected in all sampling locations during the first Aerosol Sampling Experiment (December 2016).

No cadmium-containing particles were collected in Nodes 1 and 2 and in the PMM. Both PASs in Node 3 had the highest number of cadmium particles, which can be explained by the exercise equipment. PAS J and K in the US Lab had about half the number of cadmium-containing particles as those in Node 3. The American Conference of Governmental Industrial Hygienists (ACGIH) exposure guideline to cadmium is an airborne mass concentration of 0.002 mg/m^3 (respirable fraction), and ISS is nowhere near this limit. Similar to Earth-based living spaces, the concentration of cadmium-containing particles in ISS air is nonhomogeneous, and future sampling results are expected have better statistics for many of the same locations. These results show that the cadmium particle emission rates on ISS are extremely low.

Table 2: Cadmium-containing Particles Sampled on ISS

Passive Sampler	Location	Sample Duration, days	% Cadmium Particles, by Number
B-16	Node 1 Deck 1	16	0
D-16	Node 3 Deck 3	16	1
E-32	PMM	32	0
F-32	Node 2 Deck 2	32	0
G-8	Node 3 Forward 3	8	1.4
J-32	US Lab Bay 1	32	0.8
K-16	US Lab Bay 3	16	0.5

Vacuum bag debris from ISS, which consists of airborne particulate matter that accumulates on the screens covering the filters, was also analyzed for metals, including cadmium, as part of the Divert Unwanted Space Trash (DUST) project. Inductively coupled plasma mass spectrometry (ICP-MS) was performed on three samples of dust from ISS vacuuming activities on or before June 5, 2017 (the date the bag was removed from the vacuum cleaner). Samples were prepared with acid digestion and both deionized water and nitric acid extraction, and analyses were performed in duplicate with separate aliquots. Cadmium was detected in each sample ranging from an average of 24 µg/g for the water-soluble analysis to 58 µg/g and 59 µg/g for the acid digested and acid soluble analyses, respectively. These volumetric ratios are very small compared to other metals detected in this analysis, with aluminum, iron, zinc and nickel more abundant (anywhere from a factor of four to two orders of magnitude more abundant).

The two different methods of sampling airborne particulate matter and the different analysis techniques show that cadmium particles have been present in ISS air. However, the quantities of cadmium are extremely small compared to quantities of other particle materials sampled and analyzed by each respective method.

E. Human-generated Particles

Skin flakes are generated by movement of the human body and rubbing of limbs and clothing.[16] The average size of skin flakes emitted during exercise varies by person but one study documented the average skin flake at approximately 33 µm by 44 µm and between 1 and 5 µm thick.[17,18] Another study compared occupied and unoccupied school classrooms and dust generation rates. The dust collection rate during occupied sampling times were an order of magnitude higher than sampling in unoccupied rooms. Furthermore, the most abundant protein in all of the samples (occupied and unoccupied) was identified as human epithelial keratin, indicating that skin flakes comprised the bulk of all the particles collected.[19] A similar trend is seen from sampling on ISS, shown in Figure 6 (a) and (b), where the PAS substrates from two different locations are compared after 32 days of particle collection. In the PMM storage area, there was almost no human traffic (at the time of the sampling), which resulted in a nearly clean sampling substrate (a). In contrast, Node 3, which is the exercise and hygiene area, has a very high concentration of particles seen in image (b) due to human presence and vigorous activities during waking hours.

The IntelliSEM analysis on metals previously presented took advantage of the bright appearance of metals and high contrast particle edges in SEM images. This technique, while straightforward, excluded carbonaceous particles for lack of definition. As an example, Figure 5 has a bright metal particle which is easily distinguished, however, the skin flake underneath has very low contrast against the carbon tape background. The presence of lint fibers presented difficulties, as they often landed on top of particles, obstructing the true edges of particles, and they were observed to curve around and cross other fibers, which also confuses edge detection algorithms. With some adjustment, the IntelliSEM technique was adapted to analyze all particles captured on the PAS K substrate that was deployed in the US Lab for 16 days. Only particles larger than 5 µm were included, and fibers were omitted from the analysis. This was necessary because edges of individual fibers cannot be easily resolved since they often are curved with varying radii or overlap with other fibers.

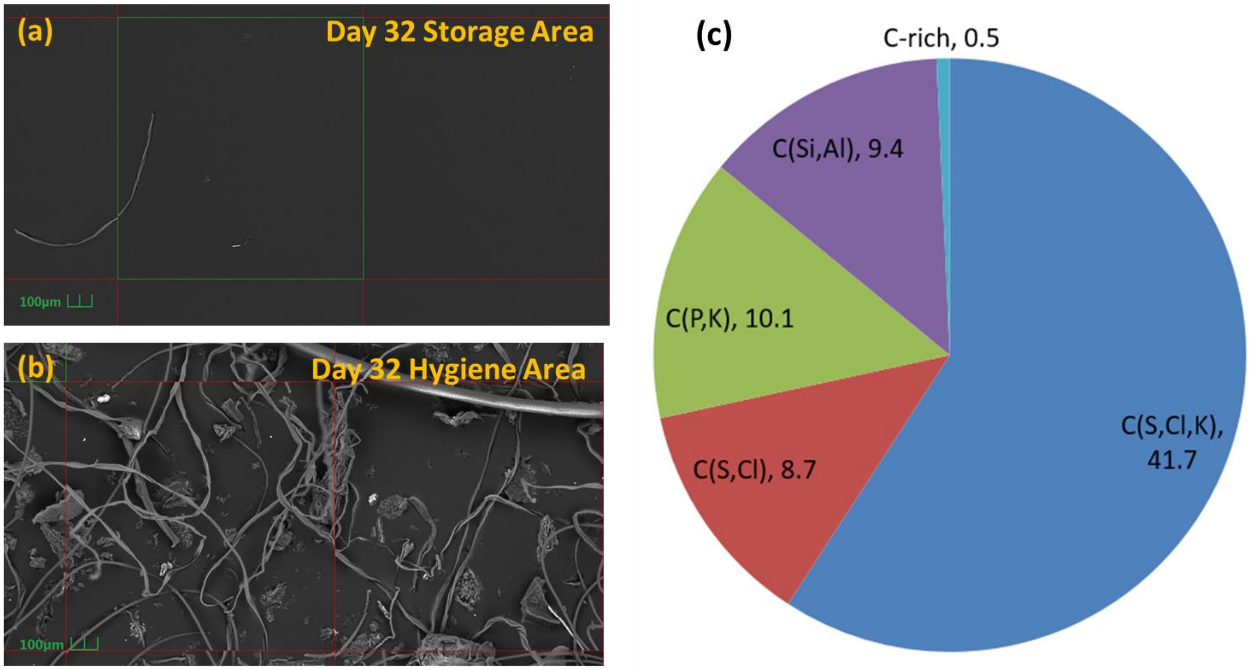

Figure 6. Passive Aerosol Sampler particle loading comparison (left), showing the PMM storage area 32-day sample (a) above the Node 3 32-day sample (b) where the crew performs exercise and hygiene. Right image: The PAS K 16-day sample was analyzed with IntelliSEM, and the pie chart (c) shows various carbonaceous particle classes (totaling 70.4% of all particles analyzed), with the group containing carbon with sulfur, chlorine and potassium (in blue) indicative of human biology, and the group containing carbon with phosphorus and sulfur (in green) indicative of plant biology.

Out of 1306 particles detected, the minimum diameter was 5.0 µm, the maximum diameter was 556.3 µm, and the average of 36.5 µm. Heavier metals comprised only 5.4% of the weight of all particles collected, other particle classes consisted of different combinations of aluminum, silicon and calcium (combined at 16.3% by weight) and 7.9% were classified as miscellaneous (having no consistent material categories). The remaining 70.4% of the weight of the analyzed particles were classified as carbon-rich. These carbon-rich particles were further classified into categories, as shown in the pie chart in Figure 6. The blue segment accounts for carbon-rich particles containing sulfur, chlorine and potassium, which is a combination indicative of human biology. Figure 7 shows an example of a human skin flake, which has a prominent carbon peak with S, Cl and K peaks in the EDS spectrum. The green segment of the pie chart in Figure 6 (c) accounts for carbon-rich particles containing phosphorus and sulfur, which is a combination indicative of plant biology. The airborne plant material on ISS could be attributed to paper, food, wood, or the Veggie plant growth facility. Quantitatively, it is important to emphasize that the results of this analysis of all types of particles (metals and non-metals) show that humans are the single largest source of aerosol emissions on ISS. From the PAS K results, humans produced 41.7% of all particles analyzed. Airborne plant material accounted for 10.1% of all particles analyzed, and heavier metals accounted for only 5.4% of the particles.

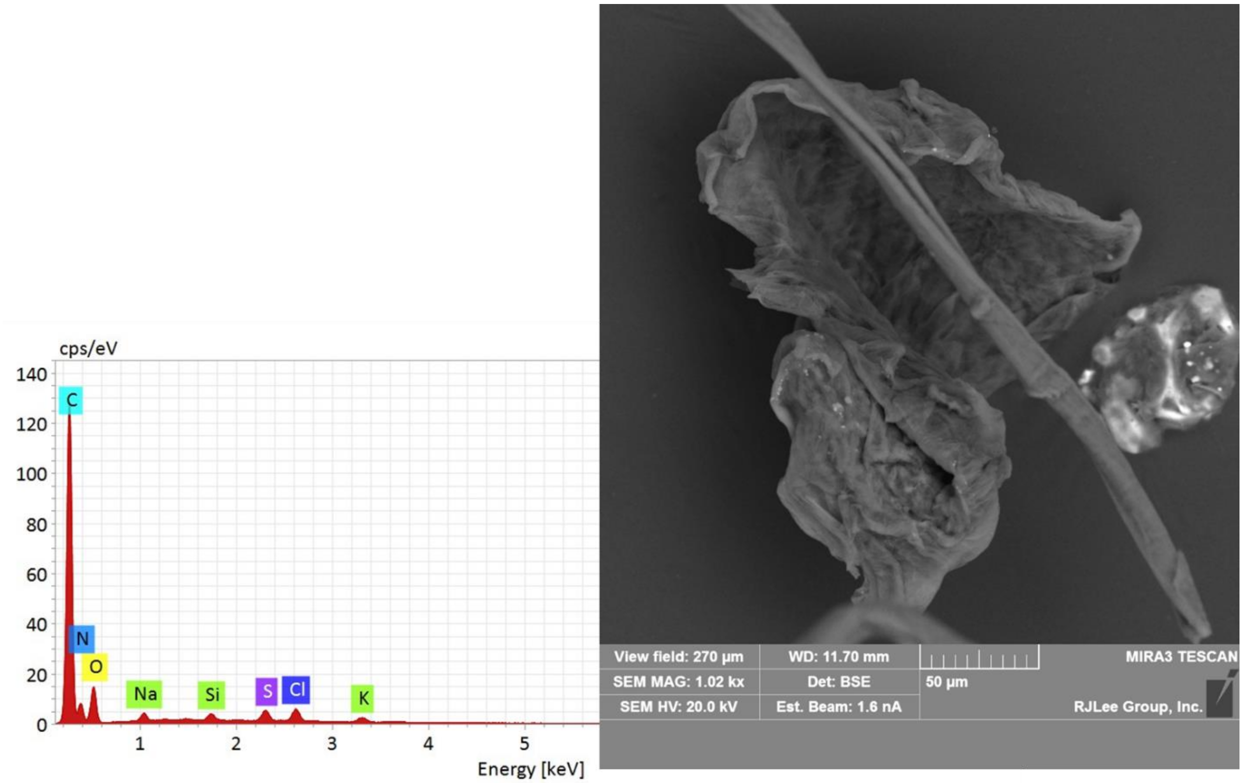

Figure 7. Skin flake captured in Node 3, a cotton lint fiber deposited across the skin flake and a brighter metal particle is adjacent on the right.

IV. Conclusion

The Aerosol Sampling Experiment on ISS provided a large data set on airborne particles that can be investigated further. Many particles have unique morphologies unlike what is typically found on Earth. A reasonable explanation for the complex multi-material agglomerates can be found in previous experiments in low gravity, designed to demonstrate planet formation mechanisms. Particles with diameters on the order of tens of micrometers can aggregate by electrostatic forces and produce some of the complex, multi-component particle morphologies seen in the PAS samples. Several particle types were highlighted in this work and the IntelliSEM methodology for analyzing particle quantities and elemental composition was outlined. The metals analyses are important for understanding quantities and types of airborne particles that may be eye hazards or may be hazardous to breathe or ingest. However, in the analysis of all particles larger than 5 micrometers, these metals made up less than 6% of all particles analyzed. The bulk of the aerosol emissions on ISS are carbonaceous particles, and human skin flakes are the largest proportion. Bromine- and cadmium-containing particle emission sources are confirmed to be present, though very small. Future work in ISS aerosols includes the completion of a full IntelliSEM analysis of the re-flight samples collected in July 2018. Both sets of experiment results will be combined into a database that can be used for further inquiries, including estimating emission rates of various particle classes with improved statistics. A fiber analysis will be completed to estimate the burden of clothing lint and other fibers on ISS air. The DUST experiment will also provide information to cross-reference between airborne ISS particle samples and ISS vacuum bag debris.

Acknowledgments

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