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Analysis of Dust Samples Collected from an In-Service Interim Storage System at the Maine Yankee Nuclear Site

Charles R. Bryan and David G. Enos

Prepared by
Sandia National Laboratories
Albuquerque, New Mexico 87185 and Livermore, California 94550

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Charles R. Bryan
Storage and Transportation Technologies Department
Sandia National Laboratories
P.O. Box 5800
Albuquerque, New Mexico 87185-MS0779

David G. Enos
Materials Reliability Department
Sandia National Laboratories
P.O. Box 5800
Albuquerque, New Mexico 87185-MS0888

Abstract

In July, 2016, the Electric Power Research Institute and industry partners performed a field test at the Maine Yankee Nuclear Site, located near Wiscasset, Maine. The primary goal of the field test was to evaluate the use of robots in surveying the surface of an in-service interim storage canister within an overpack; however, as part of the demonstration, dust and soluble salt samples were collected from horizontal surfaces within the interim storage system. The storage system is a vertical system made by NAC International, consisting of a steel-lined concrete overpack containing a 304 stainless steel (SS) welded storage canister. The canister did not contain spent fuel but rather greater-than-class-C waste, which did not generate significant heat, limiting airflow through the storage system. The surfaces that were sampled for deposits included the top of the shield plug, the side of the canister, and a shelf at the bottom of the overpack, just below the level of the pillar on which the canister sits. The samples were sent to Sandia National Laboratories for analysis. This report summarizes the results of those analyses.

Because the primary goal of the field test was to evaluate the use of robots in surveying the surface of the canister within the overpack, collection of dust samples was carried out in a qualitative fashion, using paper filters and sponges as the sampling media. The sampling focused mostly on determining the composition of soluble salts present in the dust. It was anticipated that a wet substrate would more effectively extract soluble salts from the surface that was sampled, so both the sponges and the filter paper were wetted prior to being applied to the

surface of the metal. Sampling was accomplished by simply pressing the damp substrate against the metal surface for two minutes, and then removing it. It is unlikely that the sampling method quantitatively collected dust or salts from the metal surface; however, both substrates did extract a significant amount of material. The paper filters collected both particles, trapped within the cellulose fibers of the filter, and salts, while the sponges collected only the soluble salts, with very few particles.

Upon delivery to Sandia, both collection media were analyzed using the same methods. The soluble salts were leached from the substrates and analyzed via ion chromatography, and insoluble minerals were analyzed by scanning electron microscopy and energy dispersive X-ray spectroscopy. The insoluble minerals were found to consist largely of terrestrially-derived mineral fragments, dominantly quartz and biotite. Large (mm-sized) aggregates of calcium carbonate, calcium silicate, and calcium aluminum silicate were also present. The aggregates had one flat, smooth surface and one well crystallized surface, and were interpreted to be efflorescence on the inside of the overpack and in the vent, formed by seepage of cement pore fluids through joints in the steel liner of the overpack. Such efflorescence was commonly observed during the boroscope inspection of the storage system at the site. The material may have flaked off and fallen to the point where the dust was collected, or may have brushed off onto the sponges when the robot was retrieved through the inlet vent.

Chemical analysis showed that the soluble salts on the shield plug were Ca- and Na-rich, with lesser K and minor Mg; the anionic component was dominated by SO_4 and Cl, with minor amounts of NO_3. The cation composition of the soluble salts from the overpack shelf and the canister surface was similar to the filter samples, but the anions differed significantly, being dominantly NO_3 with lesser Cl and only trace SO_4. The salts appear to represent a mixture of sea-salts (probably partially converted to nitrates and sulfates by particle-gas conversion reactions) and continental salt aerosols. Ammonium, a common component in continental aerosols, was not observed and may have been lost by degassing from the canister surface or after collection during sample storage and transportation.

The demonstration at Maine Yankee has shown that the robot and sampling method used for the test can successfully be used to collect soluble salts from metal surfaces within an interim storage system overpack. The results were consistent from sample to sample, suggesting that a representative sample of the soluble salts was being collected. However, it is unlikely that the salt samples collected here represent quantitative sampling of the salts on the surfaces evaluated; for that reason, chloride densities per unit area are not presented here. It should also be noted that the relevance to storage systems at the site that contain SNF may be limited, because a heat-generating canister will result in greater airflow through the overpack, affecting dust deposition rates and possibly salt compositions.

ACKNOWLEDGMENTS

This work was carried out in coordination with the Electric Power Research Institute (EPRI) and Maine Yankee site personnel. Jeremy Renshaw and Paul Plante played important roles in directing sample collection and in providing the samples to Sandia; they also provided technical reviews of this document. At Sandia, Jessica Kruichak and Skyler Castillo-Wilson helped with sample preparation and analysis.

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NOMENCLATURE

BSE	backscattered electron [SEM image]
DOE	Department of Energy
EDS	energy dispersive [X-ray] spectroscopy
EPRI	Electric Power Research Institute
IC	ion chromatography
ISFSI	independent spent fuel storage installation
SE	secondary electron [SEM image]
SEM	scanning electron microscope
SNL	Sandia National Laboratories
SS	stainless steel
XRD	X-ray diffraction
XRF	X-ray fluorescence

1. INTRODUCTION

In July, 2016, the Electric Power Research Institute (EPRI) and industry partners performed a field test at the Maine Yankee Nuclear Site, located near Wiscasset, Maine. The primary goal of the field test was to evaluate the use of robots in surveying the surface of in-service an interim storage canister within an overpack; however, as part of the demonstration, dust and soluble salt samples were collected from horizontal surfaces within the interim storage system. The storage system is a vertical system made by NAC International, consisting of a steel-lined concrete overpack containing a 304 stainless steel (SS) welded storage canister. The surfaces that were sampled for deposits included the top of the shield plug, the side of the canister, and a shelf at the bottom of the overpack, just below the level of the pillar on which the canister sits (Figure 1). The robot that sampled the dust entered through the rectangular channels that form the inlet vents, which provide a direct pathway to an open space near the canister pedestal, below the canister. The robot then moved up and outward through rectangular openings in the circular ring that helps support the canister, into the annulus and onto the shelf from which the dust was sampled.

The canister did not contain spent fuel but rather greater-than-class-C waste, which did not generate significant heat, limiting airflow through the storage system. The canister had been in service for over 14 years when the sampling occurred. Dust on the shelf and on the shield plug may have entered through the inlet vents, but also could have been blown into the outlet vents near the top of the overpack, as there was little or no thermally-driven air flow out of the vents. It should be noted that the amounts, and potentially even the composition, of deposited dusts on this unheated canister may not be relevant to canisters containing heat-generating waste at the site.

Because the primary goal of the field test was to evaluate the use of robots in surveying the surface of the canister within the overpack; collection of dust samples was carried out in a qualitative fashion, using paper filters and sponges as the sampling media. Two filters were used to collect dust by hand on the shield plug that rested on top of the canister. Using the robot, one sponge sample was taken from the side of the canister, and five sponges samples were taken from a shelf near the bottom of the overpack. The sampling focused mostly on determining the composition of soluble salts present in the dust. It was anticipated that a wet substrate would more effectively extract soluble salts from the surface that was sampled, so both the sponges and the filter paper were wetted with demineralized water prior to being applied to the surface of the metal. Sampling was accomplished by simply pressing the damp substrate against the metal surface for two minutes, and then removing it. It is unlikely that the sampling method quantitatively collected dust or salts from the metal surface; however, both substrates did extract a significant amount of material. The paper filters collected particles trapped within the cellulose fibers of the filter and salts dissolved in the moisture in the filter, while the sponges collected only the soluble salts, with very few particles.

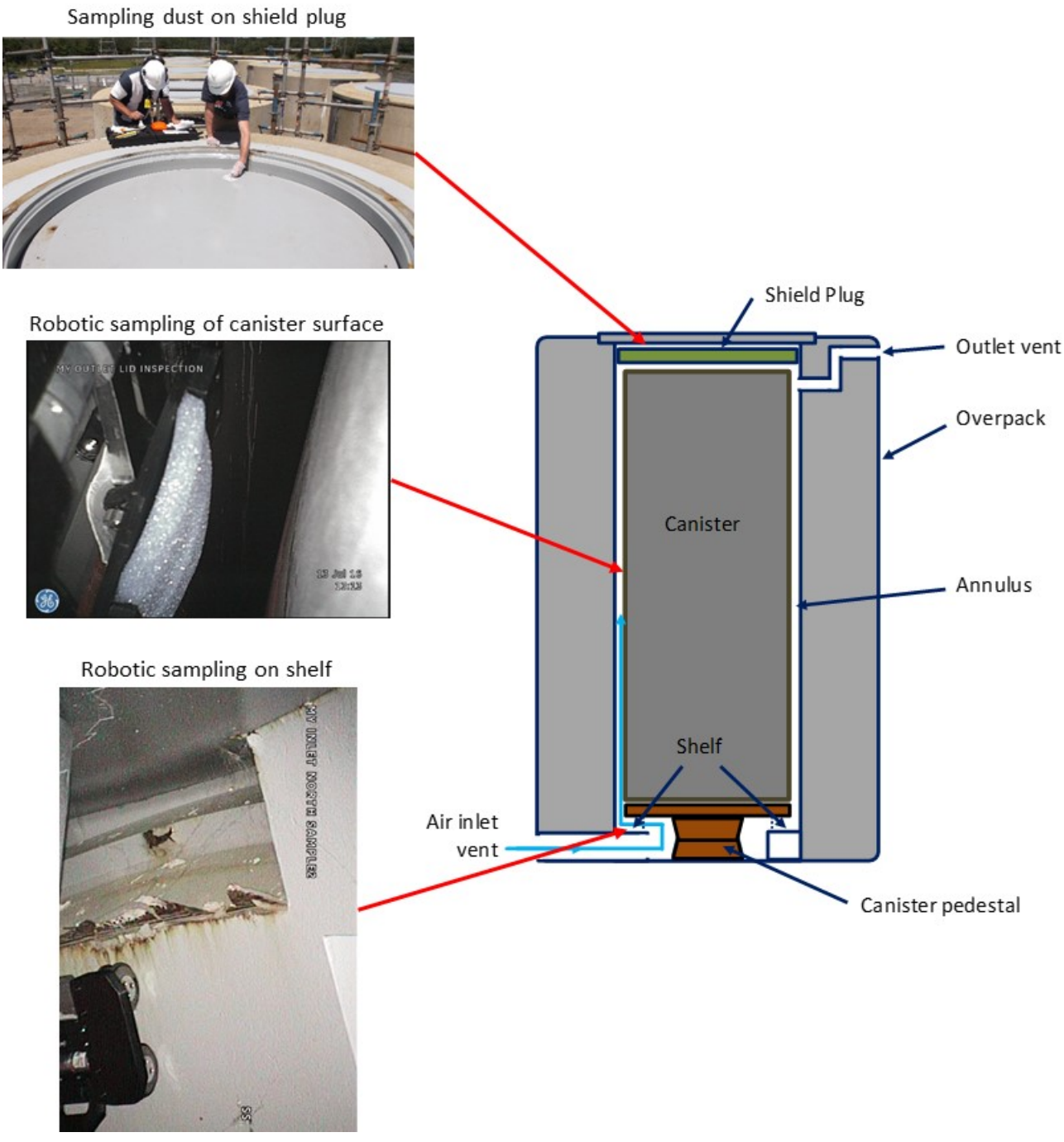


Figure 1. Storage system design (upper), and a close-up of the lower plate of the overpack (lower).

Upon delivery to Sandia, the samples were photographed, and then the soluble salts were leached from the substrates with deionized water and analyzed for anions and cations via ion chromatography (IC). The insoluble minerals on the filters, and those that were dislodged from the sponges during rinsing, were analyzed by scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS).

Sampling and analytical methods, and a list of the samples collected, are provided in Section 2 of this report. Section 3 summarizes the results of the SEM/EDS and chemical analyses, and Section 4 provides conclusions.

To summarize what is presented in the rest on this report, the insoluble minerals were found to consist largely of terrestrially-derived mineral fragments, dominantly quartz and biotite. Large aggregates (up to a few mm) calcium carbonate, calcium silicate, and/or calcium aluminum silicate were also present. These had one flat surface and one well crystallized surface, and were interpreted to be efflorescence on the inside of the overpack and in the vent, formed by seepage of cement pore fluids through joints in the steel liner of the overpack. The material may have flaked off and fallen to the point where the dust was collected, or may have brushed off onto the sponges when the robot was retrieved through the inlet vent. Chemical analyses showed that the soluble salts on the shield plug were Ca- and Na-rich, with lesser K and minor Mg; the anionic component was dominated by SO_4 and Cl, with minor amounts of NO_3. The cation composition of the soluble salts from the overpack shelf were compositionally similar, but the anions differed significantly, being dominantly NO_3 with lesser Cl and only trace SO_4. The salts appear to represent a mixture of sea-salts (probably partially converted to nitrates and sulfates by particle-gas conversion reactions) and continental salt aerosols. Ammonium, a common component in continental aerosols, was not observed and may have been lost by degassing from the canister surface, or after collection during sample storage and transportation.

The demonstration at Maine Yankee has shown that the robot and sampling method used for the test can successfully be used to collect soluble salts from metal surfaces within an interim storage system overpack. The results were consistent from sample to sample, suggesting that a representative sample of the soluble salts was being collected. However, it is unlikely that the salt samples collected here represent quantitative sampling of the salts on the surfaces evaluated; for that reason, chloride densities per unit area are not presented here. It should also be noted that the relevance to storage systems at the site that contain SNF may be limited, because a heat-generating canister will result in greater airflow through the overpack, affecting dust deposition rates and possibly salt compositions.

2. SAMPLES AND METHODS

2.1. Samples

Two months prior to the sampling at Maine Yankee, Sandia was queried as to the best type of materials to use for collecting soluble salts from surfaces within the overpack. SNL tested 4 different materials for sampling effectiveness and for levels of leachable salt components and recommended two materials for use. SNL suggested using 4.5 cm diameter Whatman #41 filter paper, which is ashless and contains virtually no soluble components. A blue polyurethane sponge made by Truclean ® that effectively cleaned a smooth surface and that leached little in terms of soluble salts, was also suggested. SNL provided these materials to EPRI for use. SNL also recommended a leaching procedure for the sponge, to minimize the leachable salt content. Full results of the testing and analysis of the candidate materials are provided in Appendix A. Prior to use, the sponge blocks provided by Sandia were cut to shape, and rinsed several times with deionized water. They were then soaked for approximately 30 hours, and then rinsed a final three times. This procedure significantly reduced the already low leachable salt content in the sponges.

On July 13, 2016, samples of dust and salt surface deposits were collected from flat surfaces within an in-service dry storage system at the Maine Yankee Nuclear Facility. The filter samples were collected by hand from the shield plug on top of the canister and the sponge samples were collected from the canister surface and from a shelf in the overpack, just above the inlet vents, using a robot with magnetic wheels. Following collection, the filter samples and the sponges, respectively, were placed in Ziploc® polyethylene plastic bags and sealed glass jars for shipping. The samples were delivered to SNL on July 15th. The samples were delivered in an ice chest containing several blocks of Blue Ice®. Upon delivery, the samples were placed in a refrigerator until analysis. Eight samples were delivered (Table 1); two moist filter samples that had been collected from the shield plug, one sponge sample from the canister surface, and five sponge samples that had been collected from the overpack itself. Prior to analysis, the samples were photographed (Figure 2). The filter samples had a heavy visible dust load; of the sponges, only Sample #8 had more than a few grains of coarse sand-sized dust on the surface. Note that all sponge samples with the exception of Sample #3 consisted of a single sponge, shaped to fit into a holder on the robot. Sample #3 was the sample from the canister surface itself and consisted of two rectangular rods of sponge material; no visible dust particles were present on this sample.

Solid particles that washed off the sponges during the rinsing process were retained for later examination. These were mostly sand-sized materials; however, in several samples, one or two large white or grey flaky aggregates were also present; these had usually fallen off of the sponges, and remained in the jars. Unlike the sponges, the dust solids on the filter paper samples did not rinse off, but were trapped in the fibers of the paper and were retained on the paper. The filters with the trapped dust particles and the insoluble dust particles from the sponges were kept for later imaging and analysis by SEM/EDS analysis.

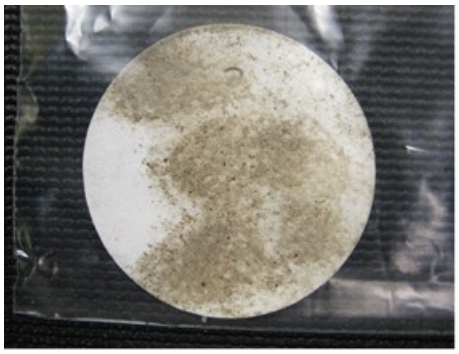
Sponge blanks were not delivered with the original samples, but were provided upon request, several weeks after the initial samples were delivered. EPRI and their team shipped 6 sponge samples to Sandia for use as blanks. Three of these had been cut and rinsed in preparation for

the sampling on 7/13/2016; the other three had been freshly cut and rinsed following the same procedure as the original sponges. The second set was included in case sponge degradation in the interval (perhaps due to oxidation of the freshly cut surfaces, or possibly biodegradation if the sponges were still damp during storage) increased the leachable salt content.

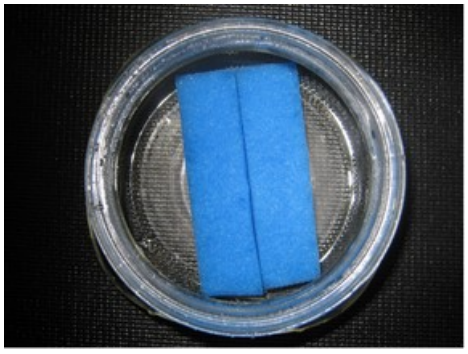
Table 1. Dust Samples Collected from a Dry Storage Canister at the Maine Yankee Nuclear Facility.

Sample #	Type	Description
1	Filter paper	Shield Plug #3 7/11/16
2	Filter paper	Shield Plug #4 7/11/16
3	Sponge	VCC2, South Outlet Vent, TSC-CS Contamination 7/13/16 (Sample from the canister surface)
4	Sponge	VCC2, Northwest Inlet Vent, North side shelf 7/13/16
5	Sponge	VCC2, Northwest Inlet Vent, West side shelf 7/13/16
6	Sponge	VCC2, Southeast Inlet Vent, East side shelf close to vent 7/13/16
7	Sponge	VCC2, Southeast Inlet Vent, South side shelf 7/13/16
8	Sponge	VCC2, Southeast Inlet Vent, South side shelf close to vent, attempted ~0.8" movement (one way) 7/13/16
Blanks		
7/13/2016-1	Sponge	Blank sample
7/13/2016-2	Sponge	Blank sample
7/13/2016-3	Sponge	Blank sample
8/2/2016-1	Sponge	Blank sample
8/2/2016-2	Sponge	Blank sample
8/2/2016-3	Sponge	Blank sample
Demin. water	Water	Water used to wet the sampling media prior to dust collection

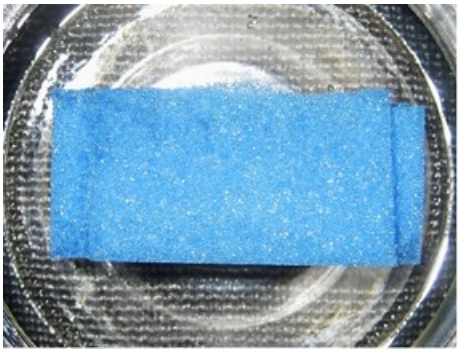
Sample 1



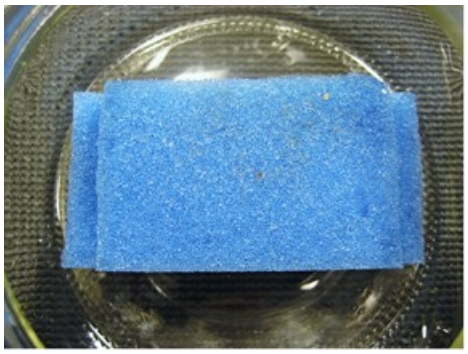
Sample 2



Sample 3



Sample 4



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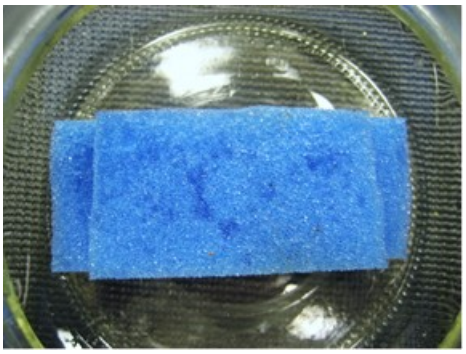
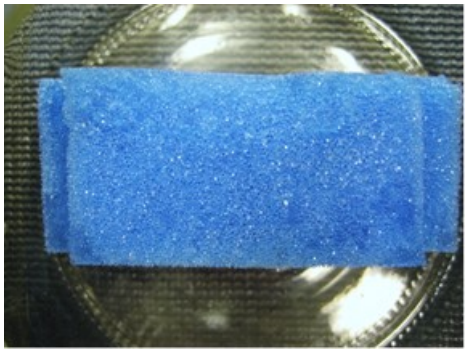
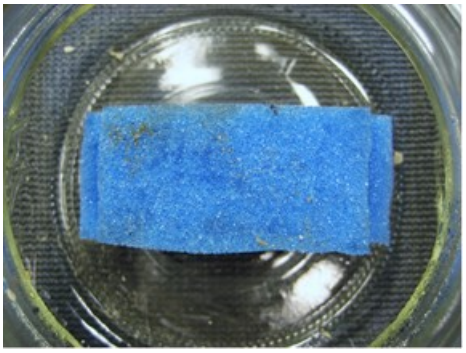


Sample 6



Sample 7



Sample 8

Figure 2. Filter and sponge samples collected at the Maine Yankee Nuclear Site.

2.2. Methods

The following methods and equipment were used to characterize the samples from Maine Yankee:

- *SEM imaging and EDS element mapping*. SEM/EDS analysis of the insoluble dust residues provided textural and mineralogical information on the insoluble phases, and allowed visual identification of organic matter (floral/faunal fragments).

- *Chemical analyses of the soluble salts in the dust.* Soluble salts were leached from the filters and sponges and analyzed to determine their concentration and composition.

Other methods have been used previously, including micro-X-ray fluorescence (XRF) analysis and X-ray diffraction (XRD). These methods were not used here for two reasons. First, the samples were damp when delivered, and leaching the soluble salts was the primary purpose of the sampling. The sponges were too thick for XRF or XRD analysis without cutting to extract a sample, and because they were damp, this would have risked contaminating the sample. Also, by maintaining the samples intact prior to leaching, the leached salts could be related to the sample size to produce a minimum salt surface load per m^2. Second, the soluble salts were dispersed throughout the sponge in the absorbed water, and were not on the surface; the XRF analysis would not have shown these. Most of the dust on the tops of the sponges and the filters was very coarse (sand-sized), and the XRF analysis would have been dominated by the coarse particles; the large sand-sized particles could be readily identified by SEM; therefore, XRD was not necessary.

SEM Imaging and EDS Analysis
SEM/EDS analysis of the samples provides textural and mineralogical information of the insoluble dust residues, and allows visual identification of organic matter (floral/faunal fragments). Sample fractions retained for SEM analysis were coated with carbon to reduce sample charging during analysis. Imaging and element mapping was done with a Tescan Vega3 SEM, equipped with an EDAX Element® EDS detector. An accelerating voltage of 15 keV was used, and working distances of 9 to 12 mm, with varying degrees of magnification. Images were obtained using both secondary and backscattered electron imaging. A relatively high beam current was used to produce a high count rate and facilitate rapid element mapping. However, the elevated beam current did enhance sample charging, resulting lower image quality in some cases.

Chemical Analysis
After being photographed, the samples were transferred to pre-weighed sterile 50 ml polypropylene sample tubes. Deionized water was used to rinse the sample jars and Ziploc® plastic bags and was transferred to the tubes containing the samples; additional water was added to bring the total to approximately 20 ml (the actual amount was determined by weight). The tubes were weighed, and the samples were allowed to equilibrate for approximately 1 hour, during which the sponge samples were periodically compressed and allowed to re-expand to rinse out the soluble salts. Then, sponges and filter paper were removed, compressing to drive

out retained water. The solutions were filtered through 0.45 µm syringe filters and 5 ml aliquots were immediately transferred to sample vials for analysis by ion chromatography (IC).

Anionic analytes were F^-, Cl^-, Br^-, NO_2^-, NO_3^-, SO_4^{-2}, and PO_4^{-3}; however, Br^- and NO_2^- were never detected, and are not included here. IC analyses were done with a Dionex ICS-1100 RFIC Ion chromatograph with a Dionex Ionpac AS-23 RFIC column and AG-23 guard column, and a Dionex AERS 500 suppressor. Blanks were run after every sample to minimize carryover, but little was observed. Five standards and a blank were made by dilution of stock Dionex IC anion standards. Sample concentrations were estimated using a subset of the standards (never less than three and a blank), excluding those which were higher than necessary to constrain the sample concentration. This was done because the calibration curves were based on the least squares method, which over-weights higher-concentration standards and results in larger errors for values in the lower part of the range.

Cationic analytes were Li^+, Na^+, NH_4^+, K^+, Mg^{2+}, and Ca^{2+}. IC analyses were done with a Dionex Ionpac CS-12A column and CG-12A guard column, and a CERS 500 suppressor, all 4 mm in diameter. Blanks were run after every sample to minimize carryover, but little was observed. The standards were made by dilution of stock Dionex IC cation standards. As with the anions, five standards and a blank were made, but only a bounding subset of the standards (never less than three and a blank) was used to determine the sample concentrations.

3. RESULTS

3.1. SEM/EDS Analysis of Insoluble Mineral Residue

SEM/EDS analysis of the insoluble dust residues was carried out to determine the composition and morphology of the insoluble materials. Analyzed samples include small pieces cut from both of the filter samples, which had abundant large (sand-sized) dust particles trapped in the fibers, and a single SEM mount of particles that had dislodged from the sponges during leaching to extract the soluble salts. Results are summarized here with typical images from samples; a complete suite of analyses is provided in Appendix A, and allows the reader to better assess the representativeness of the results provided here.

3.1.1. Filter Samples

The insoluble mineral grains on the two filter samples were not dislodged during the leaching to extract soluble salts, because they were entangled by the fibers of the filter. After leaching, the filters were dried and sections were cut for SEM/EDS analysis. The minerals on the filters were mostly coarse, sand-sized grains, visible to the naked eye. The minerals are identical on both filters. Figure 3 is a backscattered electron (BSE) SEM image of minerals on Sample #1. In BSE images, the relative brightness of the different phases present indicates the average atomic number (Z). The filter fibers are mostly carbon and oxygen, with a low average Z, and are dark gray; the minerals comprise mostly angular quartz grains (Si, O) (grey) and platy flakes of biotite (Si, Al, Fe, K, Mg, O) (pale grey). A few spherical, bright grains of iron oxide are also present in Figure 3; a magnified image of one such sphere, with its X-ray spectrum, is provided in Figure 4. Iron oxide spheres are common in dusts, and here, may represent fly ash. In some storage canister dusts that have been sampled (Bryan and Enos 2014, 2015b), similar spheres represent welding condensate or spatter and contain Ni or Cr, but the lack of Cr and Ni indicate that the spheres here were probably derived from fly ash.

Figure 5 shows minerals on Sample #2, and a secondary electron (SE) image and element map of an area on Sample #2 is shown in Figure 6. Note that secondary electron images show surface topography; shades of grey do not coincide to differences in Z, so compositional information is not provided.

Other terrigenous minerals such as potassium feldspar (K-Al-Si-O) are present in much smaller amounts. A single several millimeter fiber of plant matter was also observed. No salt phases were observed, but the samples were leached with deionized water prior to SEM analysis, so the lack of soluble minerals was expected.

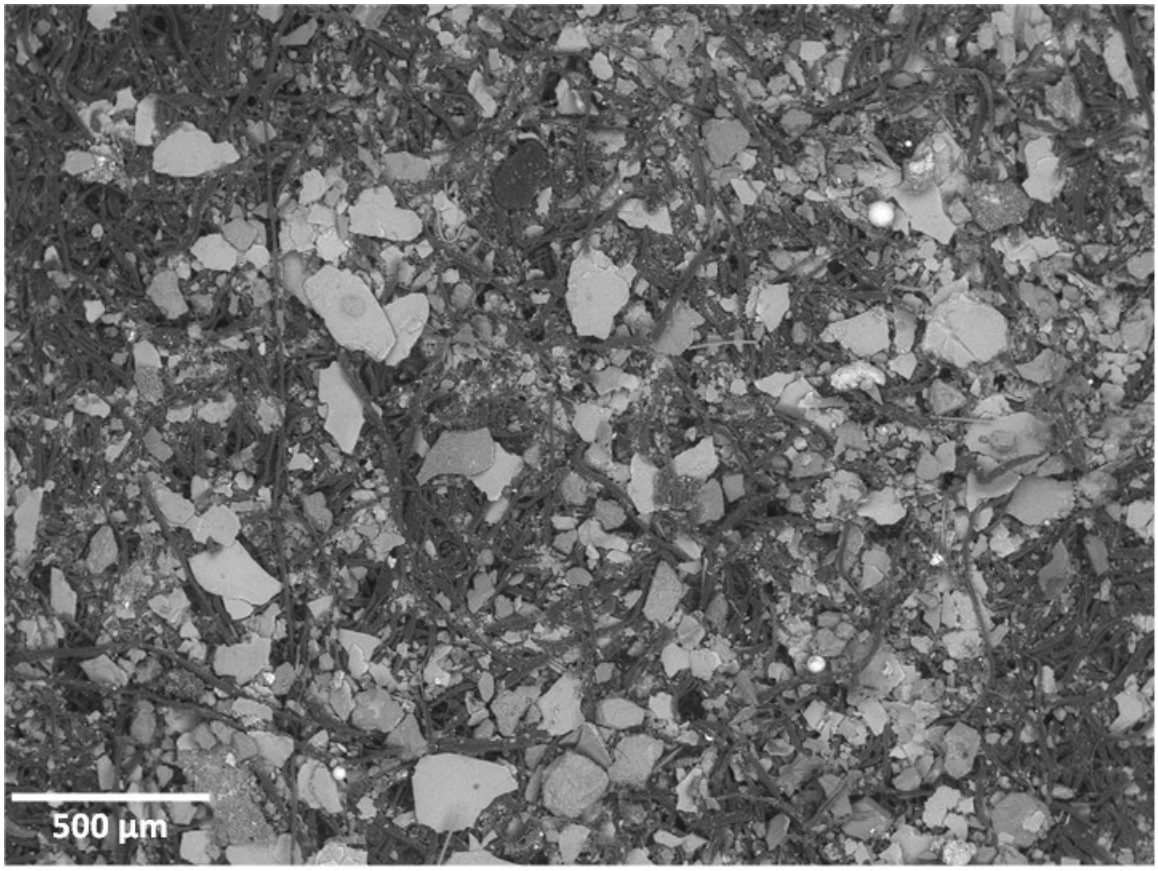


Figure 3. SEM BSE image of mineral grains embedded in the Sample #1 filter from the shield plug. Platy biotite flakes (pale gray); angular quartz grains (darker gray).

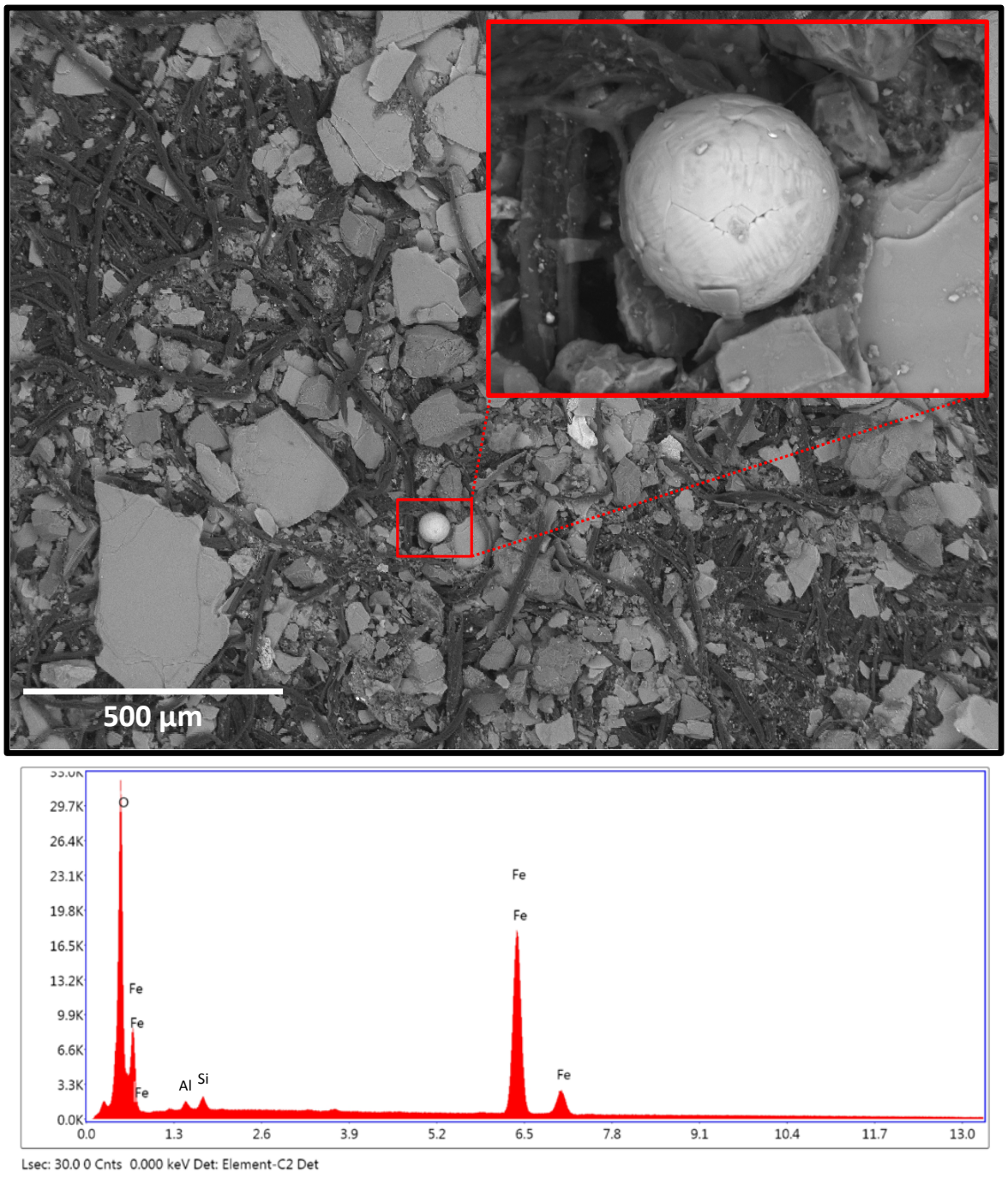


Figure 4. SEM BSE Image and X-ray Spectrum for Spherical Iron Oxide Grain on the Sample #1 Filter from the shield plug.

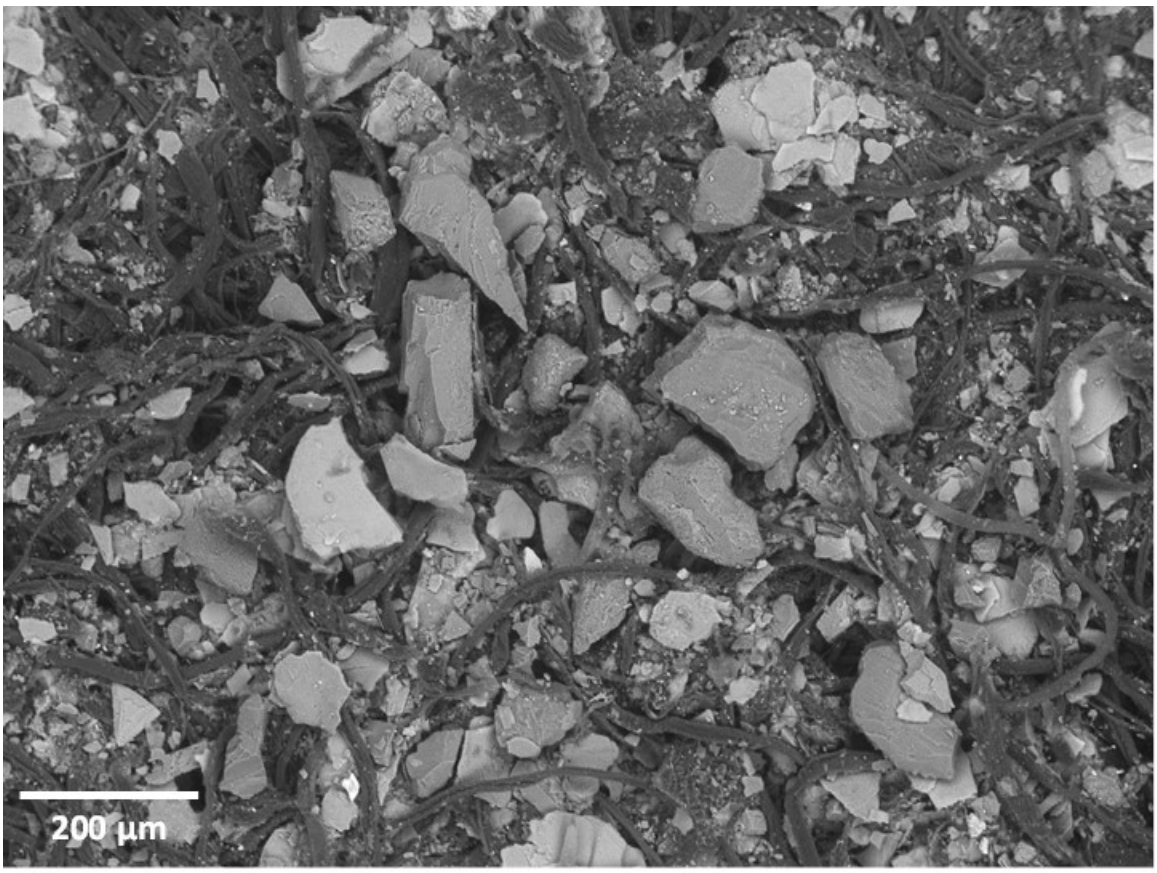
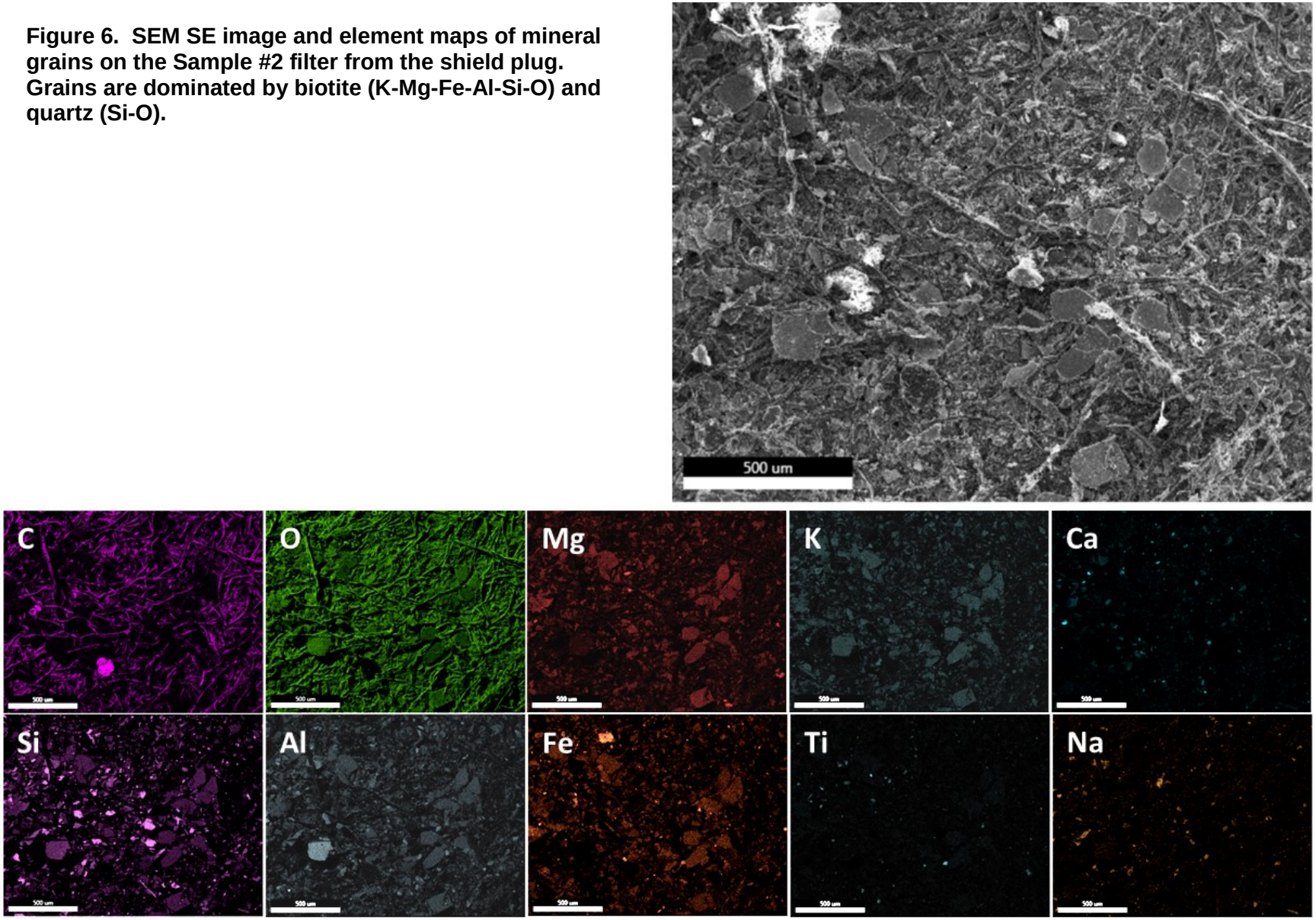


Figure 5. SEM BSE image of mineral grains embedded in the Sample #2 filter from the shield plug. Platy biotite flakes (pale gray); angular quartz grains (darker gray).

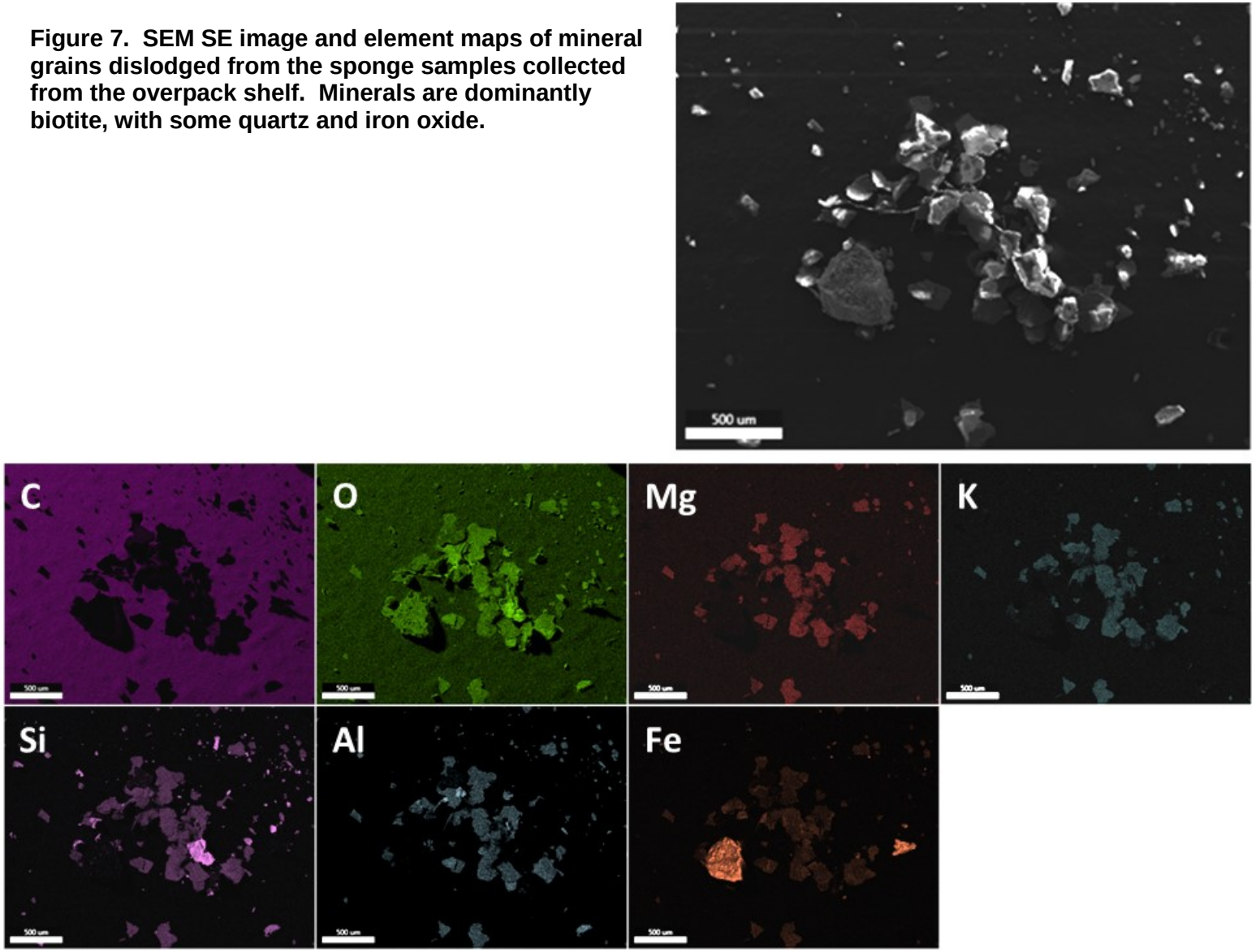
Figure 6. SEM SE image and element maps of mineral grains on the Sample #2 filter from the shield plug. Grains are dominated by biotite (K-Mg-Fe-Al-Si-O) and quartz (Si-O).



3.1.2. Insoluble Particles from the Sponges

In most cases, only a few sand-sized mineral grains were present on the sponge samples, and they dislodged during rinsing. The particles were collected and placed on carbon tape for analysis by SEM/EDS; the grains matched those observed on the filter samples, consisting mostly of biotite, with some quartz and iron oxide grains (Figure 7). However, a few larger grains were also present, up to a few mm in diameter. These were mostly dislodged during transportation and were recovered from the jars in which the sponges were shipped. These larger grains had a distinctive morphology, consisting of platy sheets of well-developed crystals with a flat bottom (See Figures 8 through 11). The individual crystals display several forms, including dodecahedrons and trigonal prisms. EDS X-ray analysis determined that the plates consist of calcium carbonate, calcium (aluminum) silicate, or a mixture of both. There can be little doubt that these plates represent efflorescence that formed by evaporation of concrete pore fluids. The well-formed crystals indicate that these crystals formed on an open surface, and given the very flat, even bottom, they probably represent efflorescence that formed on the steel inner surfaces of the overpack by concrete fluids seeping through gaps in the steel shell. Such efflorescence was observed in abundance during the canister inspection, as white stains and crusts on the inner surfaces of the overpack (Figure 12), which apparently formed by concrete fluids seeping out between joints in the metal shell (Fales, 2016). The particles may have flaked off the metal and accumulated on the shelf in the overpack, or may have brushed off onto the sponges as the robot was retrieved through the overpack vents. Finally, a single large zinc-rich grain was found, probably a fragment of the Zn-rich protective paint on the overpack outer surface.

Figure 7. SEM SE image and element maps of mineral grains dislodged from the sponge samples collected from the overpack shelf. Minerals are dominantly biotite, with some quartz and iron oxide.



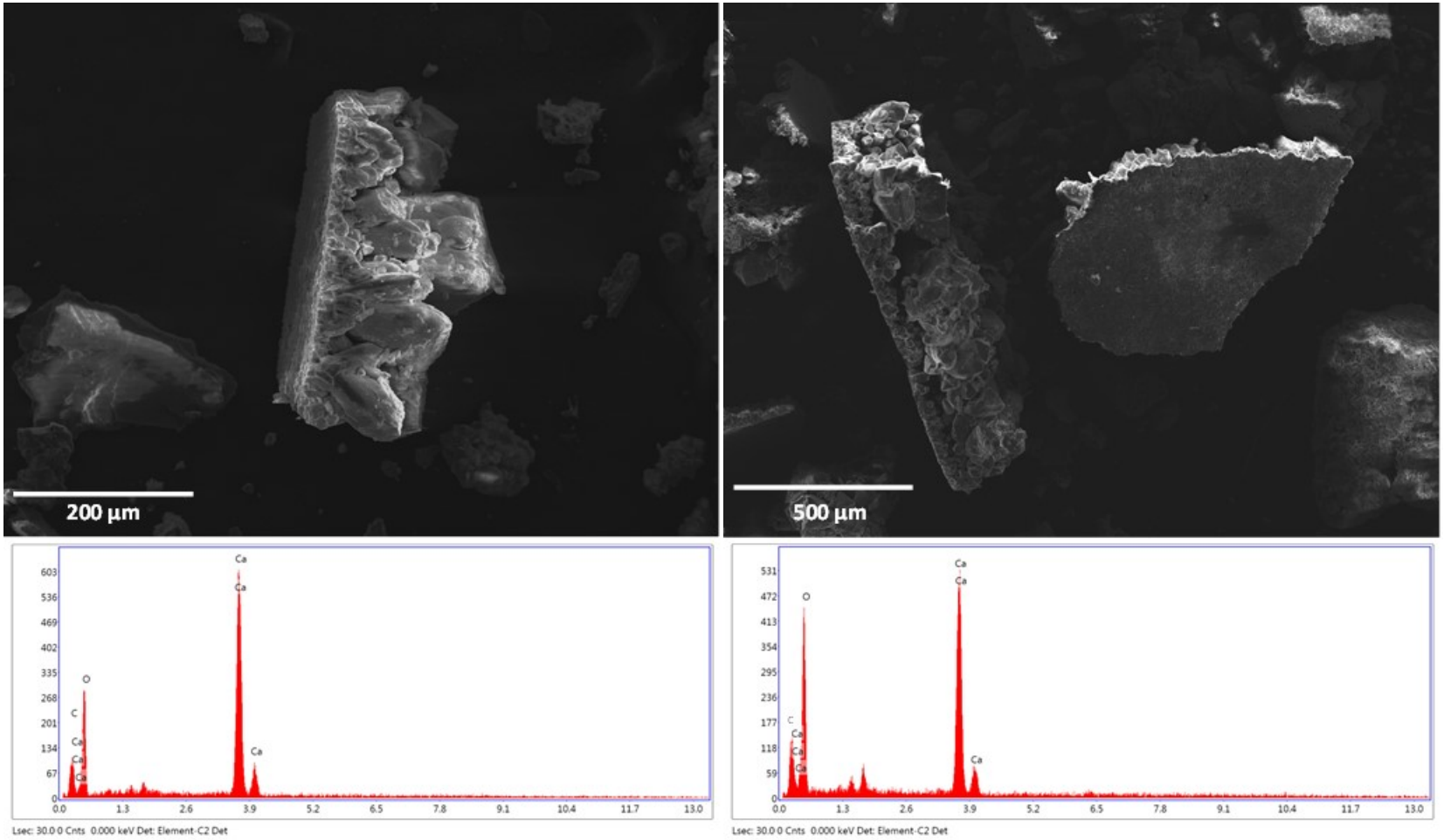


Figure 8. SEM SE images and EDS spectra of calcite plates found in the sponge insoluble residues from the overpack shelf.

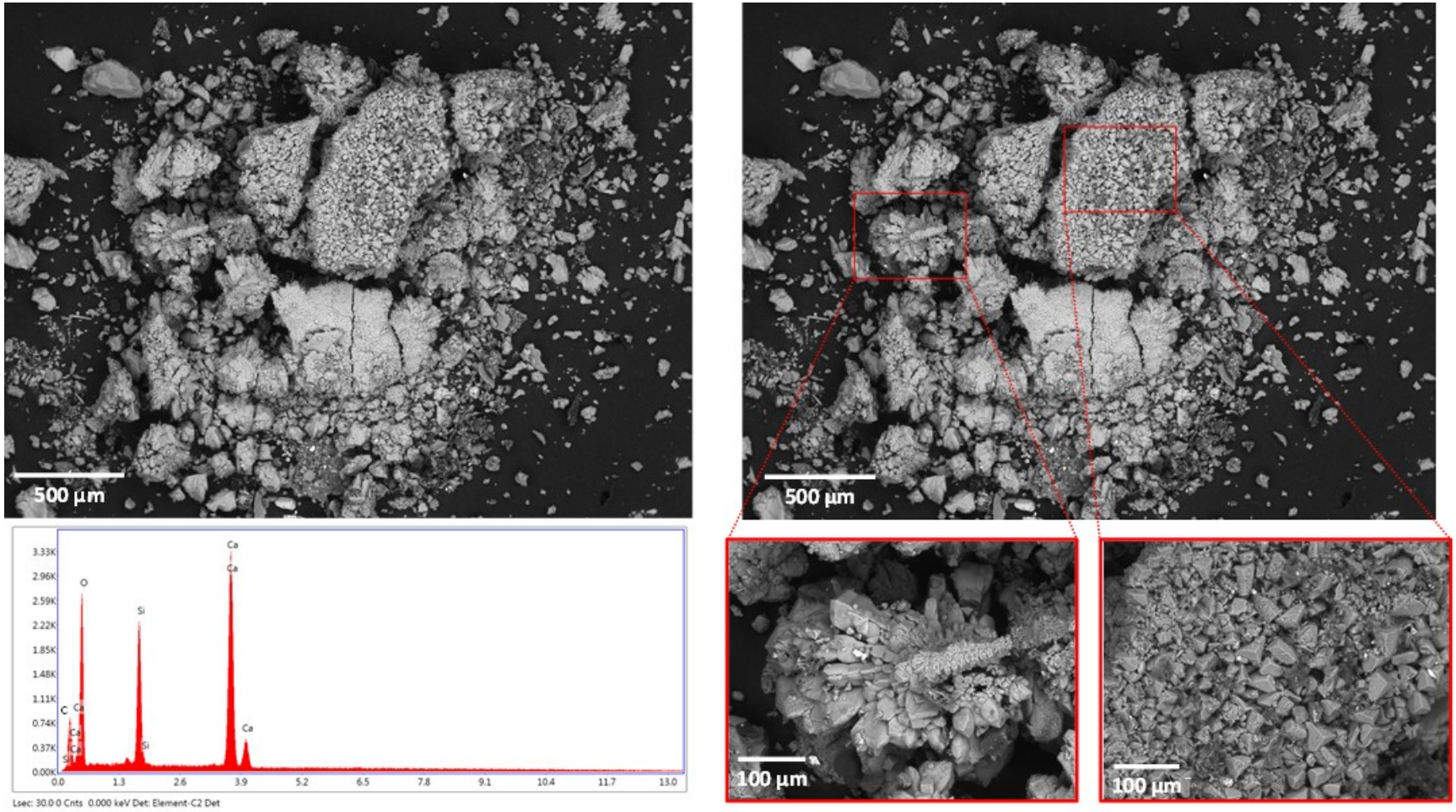
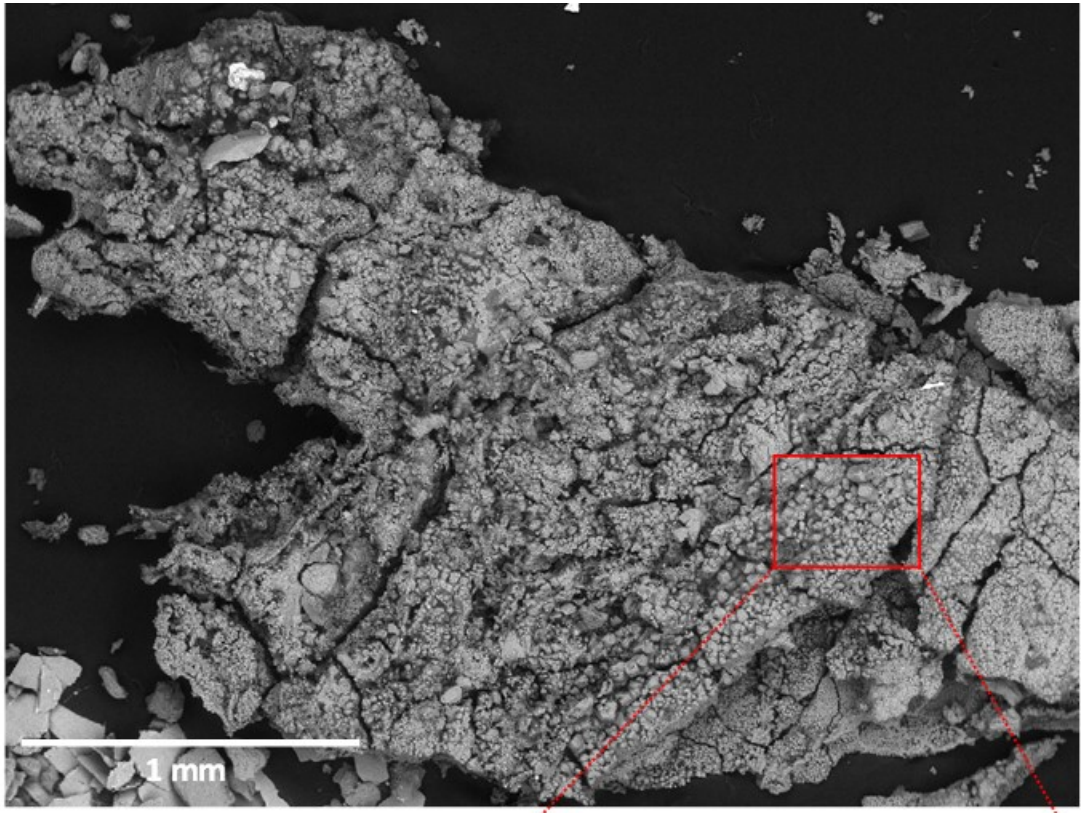
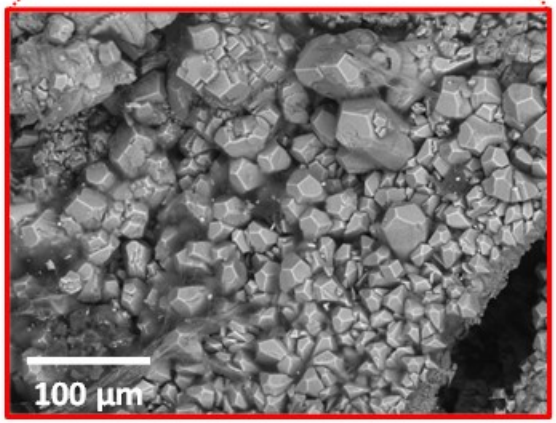
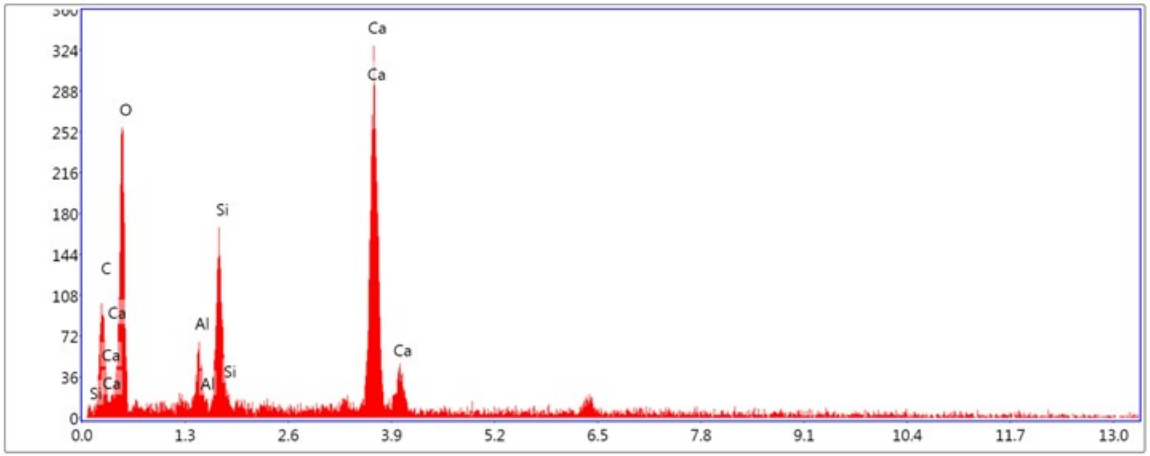


Figure 9. SEM SE image and EDS X-ray spectrum of a large plate of well-crystallized calcium silicate in the sponge insoluble residues from the overpack shelf.

Figure 10. SEM SE image and EDS X-ray spectrum of large plate of calcium aluminum silicate in the sponge insoluble residues from the overpack shelf.







Lsec: 30.0 0 Cnts 0.000 keV Det: Element-C2 Det

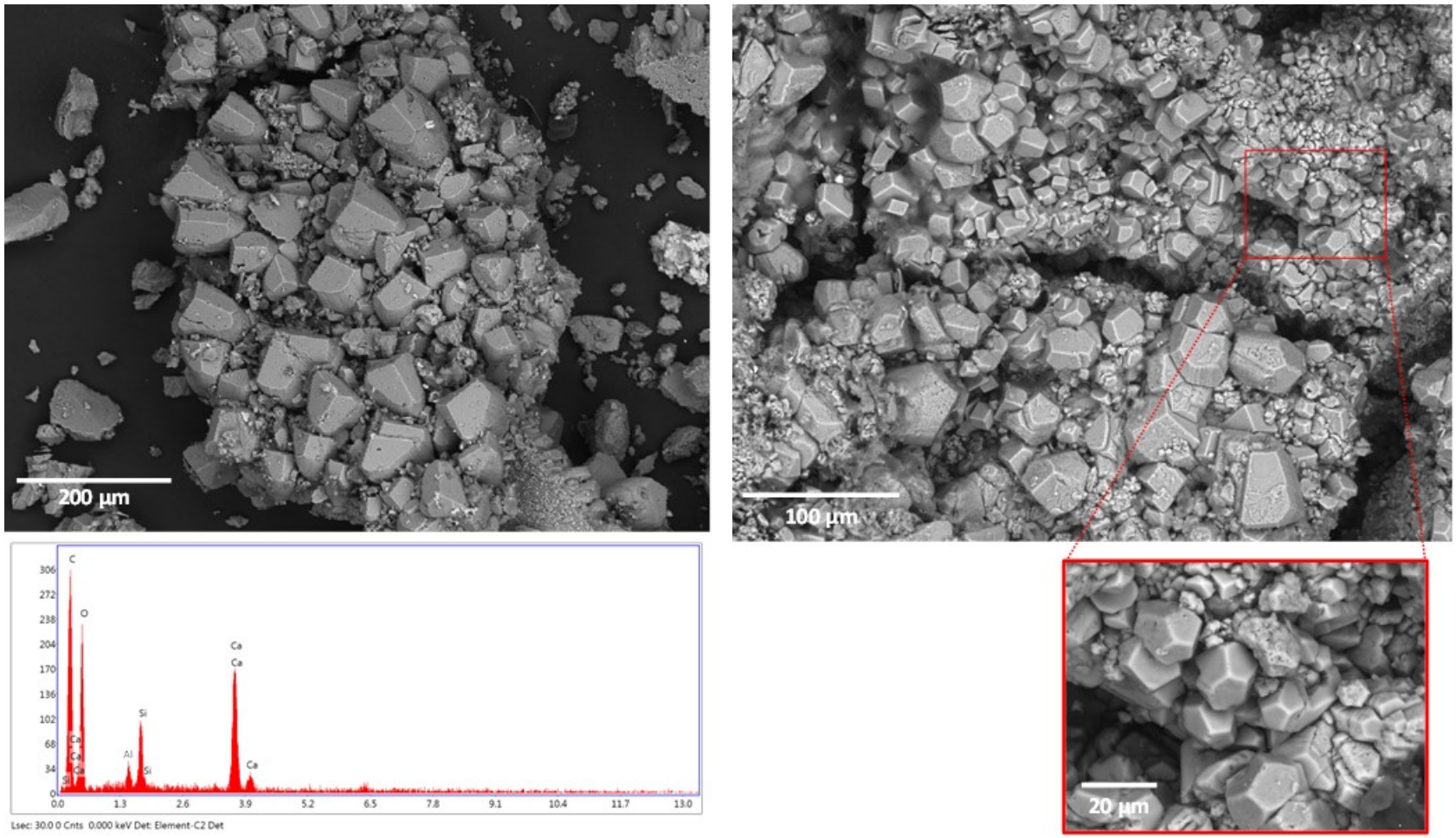


Figure 11. SEM SE images and an EDS X-ray spectrum of well-crystallized calcium aluminum silicate in the sponge insoluble residues samples from the overpack shelf.

Figure 12. Video image of white crusts of cementitious material on the inner surface of the overpack, located near an outlet vent.

3.1.2. Summary of SEM/EDS Analyses

To summarize, the insoluble residues retained by the filters and those dislodged from the sponges after rinsing were analyzed by SEM/EDS. In general, the dust particles were quite coarse, generally in the range of tens to hundreds of microns in diameter, in the sand-sized range. The grains are largely terrestrial in origin, and are dominated by biotite flakes perhaps 2/3 of the total, and angular quartz grains (~1/3). Other detrital terrestrial minerals are also present in small amounts, as well as iron oxide spheres, probably derived from fly ash. A few large plates (up to a few mm) of well crystallized calcite and calcium (aluminum) silicate are present and represent efflorescence formed by concrete or cement pore fluids; that crystallized on the inner metallic surface of the overpack. Such efflorescence was observed in abundance during the canister inspection, as white stains and crusts on the inner surfaces of the overpack, which apparently formed by concrete fluids seeping out between joints in the metal shell (Fales, 2016).

3.2. Chemical Analysis of Soluble Salts

The actual samples collected from the interim storage system were collected on 7/13/16, and delivered to Sandia on 7/15/16, but blank samples were not included. Sponge blanks were supplied upon request afterwards. Two sets of blanks were sent. The first set consisted of blank sponges that were rinsed and prepared with the actual samples on 7/13/16, but never used. The second set of blanks consisted of sponges that were prepared in an identical manner, but just prior to being sent to Sandia on 8/2/16. Two sets were provided so that it was possible to evaluate whether ageing of the sponges after cutting had any effect on the leachable salt load. No blanks were sent for the Whatman#41 filters; however, the filters are "ashless" meaning that they are nearly pure cellulose and contain virtually no inorganic materials. Previous analyses have shown that they contain no measurable soluble salts.

The soluble ion concentrations in the sponge blanks, in µg/sample, are shown in Table 2. Values shown in italicized gray type had clear IC peaks, but fell between the lowest standard and the blank, and are semi-quantitative. Measurement uncertainties are generally 5% or less, but may be as high as 20% for low values, near the quantitation limit. For comparison with data in Appendix A, it is noted that each sponge weighed approximately 0.8 g. The soluble salts that were leached from the sponges were quite low compared to the amount leached during sponge testing, indicating that the more rigorous pre-rinsing procedure was effective in removing more soluble salts from the sponge media. There is a significant difference between the 7/13/16 set of blanks and the 8/2/16 set of blanks; the 7/13/16 blanks had significantly higher leachable salts. This may indicate that the leachable salt content does increase with ageing; alternatively, perhaps the 7/13/16 blank sponges were simply not rinsed as thoroughly as the 8/2/16 blank samples. When compared to the soluble salt concentrations in the samples, the soluble salt concentrations in all of the blanks were very low, sufficiently low that the potential contribution from the sponges can be ignored. The blanks will not be considered further.

The soluble ion concentrations in the samples are shown in Table 3 (see Table 1 for a full description of each sample). The soluble salt compositions of the samples from both the shield plug and the overpack shelf were readily detectible. Although the soluble salt concentrations leached from the filters and sponges were more or less commensurate, the compositions differed significantly. In both sets of samples, the cations are dominantly Ca and Na, with moderate levels of K and minor Mg. However, the shield plug samples are sulfate- and chloride-rich, with minor NO_3; the overpack shelf samples and the canister sample are NO_3-rich, with moderate Cl but very little SO_4. Ammonium was not observed on any of the samples; however, there are several possible explanations for this. The absence of ammonium is consistent with experimental evidence that it does not persist in the presence of nitrate or chloride, because it is lost from the surface via a coupled degassing reaction (Bryan and Enos 2015a,b; Enos *et al.* 2016). Moreover, if unreacted cement phases were present in the dust and in contact with the deliquesced salts, then the resulting high pH would have promoted conversion of NH_4^+ to NH_3, further driving degassing. Finally, ammonium may have been lost by degassing from the moist sampling media during sample storage and transportation.

The salt concentrations in micro-equivalents (µEq) per sample are provided in Table 4. The number of µEq is equal to the number of µmoles multiplied by the charge of the ionic species;

this eliminates the effect of different masses for the ionic species, providing a clearer understanding of the relative importance of each species with respect to the total ion load. The charge balance error is calculated from the total cationic and anionic µEq present using:

$$Charge\ balance\ error, \% = \frac{(Sum\ of\ cations - Sum\ of\ anions)}{(Sum\ of\ cations + Sum\ of\ anions)} \times 100$$

As has been previously observed for soluble salt analyses at Calvert Cliffs, Hope Creek, and Diablo Canyon (Enos *et al.* 2013; EPRI 2014; Bryan and Enos, 2014; Bryan and Enos 2015a), the charge balance is poor; measured anion µEq are much less than cation µEq. This has previously been attributed to carbonate species, both because carbonate cannot be measured in the IC analyses, and because carbonates have been commonly observed in SEM analyses of the dusts (Enos et al. 2013; EPRI 2014; Bryan and Enos, 2014; Bryan and Enos 2015a). Calcium carbonate in the form of cement efflorescence was observed in the insoluble dust residues, so carbonate is also likely to be responsible for the charge imbalance in the Maine Yankee samples.

The salt compositions from both sampled locations suggest that the soluble salts are a combination of both sea-salts (rich in Na, Cl, and Mg), and salts derived from continental sources, rich in NH_4, K, Ca, NO_3, and SO_4, although NH_4 was not observed here. It is reasonable to assume that most of the chloride was deposited as sea-salts; however, chloride is deficient relative to Na. If the chloride was deposited as sea-salts, then the salt particles partially underwent particle-gas conversion reactions prior to or after deposition. Such reactions have been discussed before (Bryan and Enos 2015b), and convert chloride-rich sea-salts to nitrate and sulfate minerals. By reducing the chloride load on the canister surface, these reactions reduce the risk of canister SCC.

Table 2. Soluble Ion Concentrations in Sponge Blanks, µg/sample.

Sample #	Na^+	NH_4^+	K^+	Mg^{2+}	Ca^{2+}	F^-	Cl^-	NO_3^-	SO_4^{2-}	Sum, µg
7/13/16-1	2.3	n.d.	1.4	*0.1*	9.8	n.d.	*1.8*	6.2	n.d.	21.5
7/13/16-2	3.6	n.d.	1.8	*0.1*	11.0	n.d.	4.3	9.9	n.d.	30.7
7/13/16-3	2.3	n.d.	1.7	n.d.	8.8	n.d.	*2.0*	10.6	*1.4*	26.7
8/2/16-1	*1.1*	n.d.	*0.5*	n.d.	*3.1*	n.d.	*2.7*	3.7	n.d.	11.0
8/2/16-2	*1.1*	n.d.	*0.3*	n.d.	*1.6*	n.d.	*2.4*	*0.4*	n.d.	5.8
8/2/16-3	*1.6*	n.d.	*0.4*	n.d.	*2.9*	n.d.	3.8	*2.0*	n.d.	10.7

Note: Values shown in italicized gray type had clear IC peaks, but fell between the lowest standard and the blank, and are semi-quantitative.

Table 3. Soluble Ion Concentrations in Maine Yankee Dust Samples, µg/sample.

Sample #	Na^+	NH_4^+	K^+	Mg^{2+}	Ca^{2+}	F^-	Cl^-	NO_3^-	SO_4^{2-}	Sum, µg
1 (filter) shield plug	86.6	0.1	34.2	12.2	66.1	0.7	84.0	8.7	179	472
2 (filter) shield plug	60.2	n.d.	13.8	6.4	52.7	0.6	48.6	3.2	102	287
3 (sponge) canister	80.9	0.6	26.6	2.2	50.7	n.d.	42.9	167	6.0	377
4 (sponge) overpack shelf	65.7	*0.1*	24.4	2.7	60.4	*0.2*	19.2	188	10.9	371
5 (sponge) overpack shelf	63.6	*0.1*	24.6	2.5	67.8	*0.3*	18.0	178	15.1	370
6 (sponge) overpack shelf	60.6	0.2	22.3	2.5	57.2	n.d.	14.8	171	11.8	340
7 (sponge) overpack shelf	93.4	0.2	48.0	4.2	97.5	n.d.	42.4	343	15.5	644
8 (sponge) overpack shelf	80.7	0.4	61.4	4.3	111.8	n.d.	28.7	166	15.6	469

Note: Values shown in italicized gray type had clear IC peaks, but fell between the lowest standard and the blank, and are semi-quantitative.

Table 4. Soluble Salt Concentrations in Maine Yankee Dust Samples, µEq/sample.

Sample #	Na^+	NH_4^+	K^+	Mg^{2+}	Ca^{2+}	F^-	Cl^-	NO_3^-	SO_4^{2-}	Sum cat.	Sum an.	CBE, %
1 (filter) shield plug	3.77	0.01	0.87	1.00	3.30	0.03	2.37	0.14	3.73	8.95	6.27	17.6
2 (filter) shield plug	2.62	n.d.	0.35	0.53	2.63	0.03	1.37	0.05	2.12	6.13	3.57	26.3
3 (sponge) canister	3.52	0.03	0.68	0.19	2.53	n.d.	1.21	2.69	0.13	6.95	4.03	26.6
4 (sponge) overpack shelf	2.86	*0.01*	0.62	0.22	3.01	*0.01*	0.54	3.02	0.23	6.73	3.81	27.7
5 (sponge) overpack shelf	2.76	*0.01*	0.63	0.21	3.38	*0.01*	0.51	2.87	0.31	6.99	3.70	30.8
6 (sponge) overpack shelf	2.63	0.01	0.57	0.21	2.86	n.d.	0.42	2.76	0.25	6.28	3.42	29.5
7 (sponge) overpack shelf	4.06	0.01	1.23	0.34	4.86	n.d.	1.20	5.53	0.32	10.51	7.05	19.7
8 (sponge) overpack shelf	3.51	0.02	1.57	0.35	5.58	n.d.	0.81	2.68	0.32	11.03	3.82	48.6

Notes: Values shown in italicized gray type had clear IC peaks, but fell between the lowest standard and the blank, and are semi-quantitative.
CBE = charge balance error, %.

4. CONCLUSIONS

In July, 2016, the Electric Power Research Institute and industry partners performed a field test at the Maine Yankee Nuclear Site. The primary goal of the field test was to evaluate the use of robots in surveying the surface of an in-service interim storage canister within an overpack; however, as part of the demonstration, dust and soluble salt samples were collected from horizontal surfaces, and one vertical surface, within the interim storage system. The storage system is a vertical system made by NAC International, consisting of a steel-lined concrete overpack containing a 304 SS welded canister. The canister did not contain spent fuel but rather non-heat generating greater-than-class-C waste. The surfaces that were sampled for deposits included the top of the shield plug; a shelf at the bottom of the overpack, just below the level of the top of the pillar on which the canister sits; and the vertical side of the canister itself. The samples were sent to Sandia National Laboratories for analysis. This report summarizes the results of those analyses.

Because the primary goal of the field test was to evaluate the use of robots in surveying the surface of the canister within the overpack; collection of dust samples was carried out in a qualitative fashion, using paper filters and sponges as the sampling media. The sampling focused mostly on determining the composition of soluble salts present in the dust. It was anticipated that a wet substrate would more effectively extract soluble salts from the surface that was sampled, so both the sponges and the filter paper were wetted prior to being applied to the surface of the metal. Sampling was accomplished by simply pressing the damp substrate against the metal surface for two minutes, and then removing it. It is unlikely that the sampling method quantitatively collected dust or salts from the metal surface; however, both substrates did extract a significant amount of material. The paper filters collected both particles, trapped within the cellulose fibers of the filter, and salts, while the sponges collected only the soluble salts, with very few particles.

Upon delivery to Sandia, the soluble salts were leached from the substrates and analyzed via ion chromatography, and insoluble minerals were analyzed by scanning electron microscopy and energy dispersive X-ray spectroscopy. The insoluble minerals were found to consist largely of terrestrially-derived mineral fragments, dominantly biotite and quartz. Large aggregates (up to a few mm) calcium carbonate, calcium silicate, and/or calcium aluminum silicate were also present. These had one flat surface and one well crystallized surface, and were interpreted to be efflorescence on the inside of the overpack and in the vent, formed by seepage of cement pore fluids through joints in the steel liner of the overpack. The material may have flaked off and fallen to the point where the dust was collected, or may have brushed off onto the sponges when the robot was retrieved through the inlet vent.

Chemical analysis showed that the soluble salts on the shield plug were Ca- and Na-rich, with lesser K and minor Mg; the anionic component was dominated by SO_4 and Cl, with minor amounts of NO_3. The cation compositions of the soluble salts from the overpack shelf were compositionally similar to the shield plug samples, but the anions differed significantly, being dominantly NO_3 with lesser Cl and only trace SO_4. The sample from the canister surface was similar to those from the overpack shelf, with only minor sulfate. The salts appear to represent a mixture of sea-salts (probably partially converted to nitrates and sulfates by particle-gas

conversion reactions) and continental salt aerosols. Ammonium, a common component in continental aerosols, was not observed and may have been lost by degassing from the canister surface, or after collection during sample storage and transportation.

The demonstration at Maine Yankee has shown that the robot and sampling method used for the test can successfully be used to collect soluble salts from metal surfaces within an interim storage system overpack. The results were consistent from sample to sample, suggesting that a representative sample of the soluble salts was being collected. However, it is unlikely that the salt samples collected here represent quantitative sampling of the salts on the surfaces evaluated; for that reason, chloride densities per unit area are not presented here. It should also be noted that the relevance to storage systems at the site that contain SNF may be limited, because a heat-generating canister will result in greater airflow through the overpack, affecting dust deposition rates and possibly salt compositions.

5. REFERENCES

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APPENDIX A: TESTING OF SPONGE MATERIALS FOR USE AS SAMPLING SUBSTRATES

Two months prior to the sampling at Maine Yankee, Sandia was queried as to the best type of sponge to use for collecting soluble salts from surfaces within the overpack. In response, SNL chose three high-quality sponges to evaluate for two properties: (1) the amount of soluble salts that leach out of the material; and (2) the ability to cleanly wipe a smooth surface.

Three different commercial sponges were evaluated. All three are synthetic and are not hard or stiff when dry. The sponges are shown in Figure A-1. The three sponges are:

- Verticlean sponge, by Contec®. This white sponge is ½" thick, and is made of polyester, with a Polynit fabric layer bonded to each side. Where exposed along the edges of the sponge, the center has a brownish discoloration; however, this appears to be cosmetic. The cloth covering means that this sponge is resistant to wear-and-tear.
- TruCLEAN polyfoam sponge blocks by Perfex®. These sponge blocks are 4" x 8" x 2" and made of polyurethane. They are blue in color, and are able to withstand temperatures up to 121ºC.
- TruCLEAN Hydro-Sorb sponge blocks by Perfex. These sponges are white in color and also made of polyurethane, and are temperature resistant to 121ºC. The sponges are somewhat denser than the TruCLEAN blue sponge blocks, and are treated to wet more readily and to effectively wick up liquid, leaving little residue.

Each sponge was evaluated in several ways. First, it was qualitatively noted how readily each sponge wetted, when exposed to water. The Hydro-sorb sponge, designed to be hydrophilic, wetted most easily, and the Verticlean, the least easily. Then, water retention was tested. After thoroughly wetting, each sponge was wrung out and the amount of water retained in the damp sponge, as a function the original dry weight of the sponge, was measured. The sponge was then immersed in a beaker of water and repeatedly squeezed until it was fully saturated and all air bubbles had been expelled. The sponge was then removed from the beaker and placed in a tray to drain. None of the sponges drained, each retaining the water in a fully saturated state. The saturated sponges were then weighed, and the amount of retained water determined. These results are shown in Table A-1. Although the dense Hydro-sorb sponge wetted most easily, it retained the least water, in both the damp and saturated conditions. The Tru-CLEAN blue sponge retained the most water in both conditions. The Verticlean was intermediate.

Table A-1. Water retention by sponges.

Sponge	Water retained, fraction of dry weight	
	Damp	Saturated
Verticlean	1.15	19.04
TruCLEAN – blue	1.68	32.72
Tru-CLEAN Hydro-sorb	0.66	13.78

Contec® Verticlean sponge

Perfex® TruCLEAN polyfoam sponge block

Perfex® TruCLEAN Hydro-Sorb sponge block

Figure A-1. Sponges that were evaluated for soluble salt sampling at Maine Yankee.

In the field, samples will be collected by wiping a damp sponge across the surface of the metal. To test how evenly and cleanly each sponge wiped a surface, damp sponges were wiped across a tabletop, and the resulting damp trail was examined. Results are shown in Figure A-2 below. Both of the Tru-CLEAN sponges worked well, but the Verticlean did not, leaving a streaky trail of water dots, and leaving a puddle when lifted off the surface—water that squeezed out during the wiping process was not readily reabsorbed when the sponge was lifted off the surface.

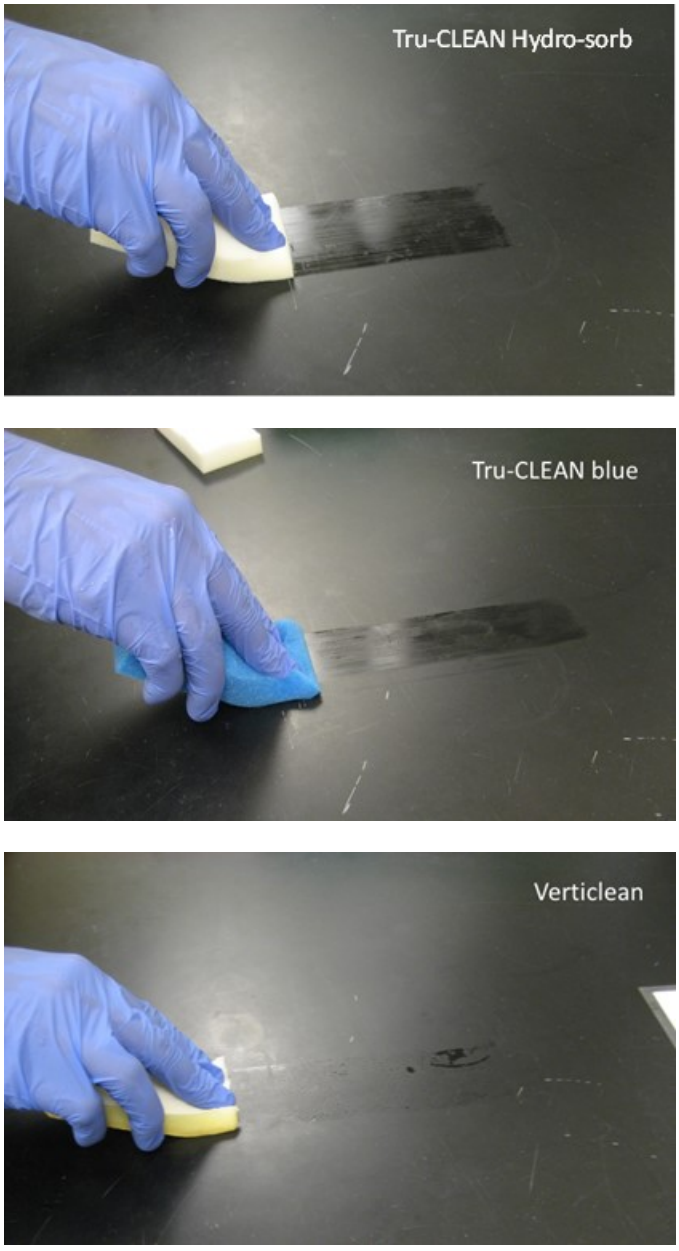

Figure A-2. Sponge wipe tests.

Finally, each sponge was evaluated for leachable salts. This was accomplished by taking pieces of the sponges and soaking them in deionized water overnight, repeatedly compressing them to rinse out the leachable salts. The soak water was then analyzed to determine what leached out of the sponges. The test was run using dry sponges (as delivered), and also using sponges that had been thoroughly rinsed and wrung out several times. This was to determine if rinsing the sponges reduced the soluble salt load. The results are shown in Table A-2, in micrograms leached per gram of sponge, and in Figure A-3. Values that are gray and italicized displayed well-formed IC peaks but were below the value of the lowest standard, and are semi-quantitative. Three different size sponge pieces were used, going from largest to smallest for each set of 3 samples; because of this, when normalized by the mass of the sponge, some samples will be italicized and gray even though they are higher than some black values. Based on values for standards run as unknowns, values within the quantitation range of the standards have uncertainties of ±10%, or ±20% for samples near the quantitation limit. Several trends are obvious in the data. The highest overall salt levels in the initial sponge material are present in the Verticlean sponge. Although there is some variability, the lowest initial salt levels are in the Hydro-sorb sponge. The Verticlean sponge benefits the most from rinsing; however, due to the initial high salt loads, it does not have the lowest salt loads after rinsing. The Hydro-sorb does not benefit as much from rinsing, possibly because of its dense structure, but even so, it has the lowest salt loads after rinsing, because of the low initial values. The Polyfoam blue has intermediate performance; initial salt loads are lower than the Verticlean, but higher than the Hydro-sorb. The Polyfoam blue sponge rinses effectively for most species; however, chloride does not appear to rinse out of the sponge well. For all sponges, the leachable salts are relatively low, and are further decreased by rinsing.

Table A-2. Results of Sponge Leaching Test

Sample #	Description	Concentraton, µg/g								
		Na^+	K^+	Ca^{2+}	Mg^{2+}	F^-	Cl^-	NO_2^-	NO_3^-	SO_4^{2-}
1	Verticlean -1	8.9	*1.9*	7.7	*1.1*	1.1	20.9	7.2	1.2	2.0
2	Verticlean -2	7.0	*1.8*	10.3	*0.8*	0.6	18.4	6.6	1.5	1.9
3	Verticlean -3	7.1	*1.6*	5.7	*0.8*	0.7	18.8	7.8	1.9	*1.2*
4	Verticlean "Trimmed"	6.9	*1.5*	6.5	*0.7*	0.6	18.5	8.5	1.4	*1.1*
5	Verticlean "Discolored"	6.9	*1.5*	4.3	*0.7*	0.6	17.5	5.9	1.5	*1.1*
6	Verticlean Rinsed -1	*1.7*	*0.6*	*1.9*	*0.1*	0.5	11.8	6.1	n.d.	n.d.
7	Verticlean Rinsed -2	*1.7*	*0.6*	*1.6*	*0.2*	*0.5*	12.3	6.4	n.d.	n.d.
8	Verticlean Rinsed -3	*1.9*	*0.7*	*1.9*	*0.2*	*1.0*	13.1	6.5	*2.3*	n.d.
9	Hydrosorb -1	5.4	*1.5*	2.3	*0.1*	5.5	12.6	n.d.	n.d.	n.d.
10	Hydrosorb -2	5.8	*1.7*	3.4	*0.2*	5.2	12.3	n.d.	n.d.	n.d.
11	Hydrosorb -3	5.4	*1.9*	*4.2*	*0.3*	6.6	13.2	n.d.	n.d.	n.d.
12	Hydrosorb Rinsed -1	3.9	*1.3*	*0.9*	n.d.	3.4	9.8	n.d.	n.d.	n.d.
13	Hydrosorb Rinsed -2	4.4	*1.4*	*1.2*	*0.1*	3.3	9.9	n.d.	n.d.	n.d.
14	Hydrosorb Rinsed -3	4.3	*1.5*	*1.3*	*0.1*	5.2	9.8	n.d.	n.d.	n.d.
15	Polyfoam -1	*1.8*	*1.5*	*2.7*	*0.1*	4.8	17.2	n.d.	2.6	*1.2*
16	Polyfoam -2	*2.3*	*1.7*	*4.0*	*0.2*	3.2	17.1	n.d.	5.6	n.d.
17	Polyfoam -3	*4.5*	*2.4*	*7.1*	*0.3*	4.9	17.9	n.d.	*4.7*	n.d.
18	Polyfoam Rinsed -1	*1.0*	*1.3*	*1.4*	*0.0*	1.8	15.1	n.d.	n.d.	n.d.
19	Polyfoam Rinsed -2	*1.2*	*4.1*	*1.9*	*0.1*	1.8	13.5	n.d.	n.d.	n.d.
20	Polyfoam Rinsed -3	*1.3*	*1.3*	*2.3*	*0.1*	*1.3*	14.4	n.d.	n.d.	n.d.

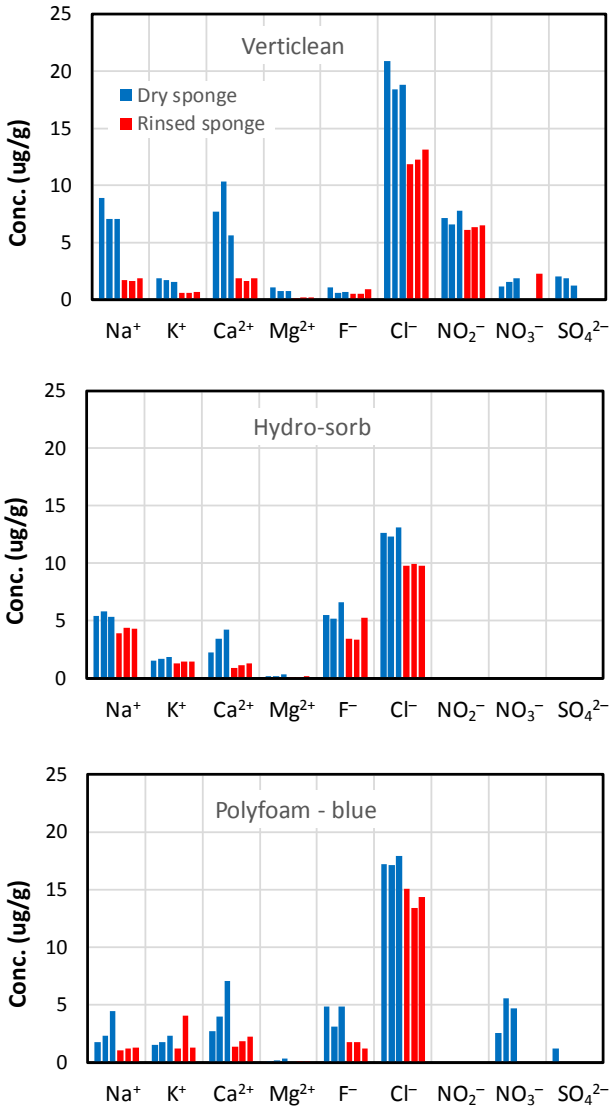

Figure A-3. Results of sponge leaching tests.

To summarize, rinsing the sponges with deionized water is effective at leaching out some of the soluble salts; a more thorough rinsing might have been more effective, and was recommended for the sponges used at Maine Yankee. The Verticlean sponge wipes surfaces poorly and has the highest initial salt loads; although it rinses effectively, it seems a poor choice, unless ability to withstand wear becomes important. Both the Hydro-sorb and Polyfoam-blue sponges wipe surfaces much more evenly than the Verticlean. The white Hydro-sorb sponge has the lowest initial salt load per unit mass, but is *more than twice as dense* as the Poly-foam blue; for a sponge of a given size, the Polyfoam-blue has a lower initial salt load, and also rinses more effectively. The Poly-foam blue was selected as the best choice, and was recommended for use at Maine Yankee. Even the relatively cursory rinsing done for these tests significantly lowered the soluble salt load of the sponges. A more thorough pre-rinsing procedure was recommended for the sponges to be used for sample collection at Maine Yankee.

Whatman #41 filter paper was recommended as an alternative to the sponges, for wet or dry sampling. This filter paper is ashless, meaning that is has no insoluble residue upon ashing (and hence has very low leachable components), and is tough enough to withstand abrasion when swiped across the metal surface. Paper consists of cellulose fibers, and retains dust well.
To summarize, testing suggested that Whatman #41 filter paper and the Tru-CLEAN polyfoam blue sponges were good choices for sampling, leaching little in the way of soluble components and effectively cleaning the surface.

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