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Stainless Steel Canister Surface Analysis: Process and Results

Charles Bryan and David Enos, Sandia National Laboratories
EPRI ESCP meeting, 3-5 December 2013, Charlotte



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Overview

- **Background**
- **Types of samples and sampling methods**
- **Analysis methods**
- **Calvert Cliffs**
 - Samples
 - Analysis results
 - Interpretation
- **Salt-Smart® preliminary data (efficiency)**

Background

- Stress corrosion cracking (SCC) of stainless steel due to deliquescence of chloride-rich salts on the metal surface is well-known, especially in near-marine environments.
- There is a concern that SNF interim storage canisters, in passively ventilated overpacks, could be subject of SCC.
- However, little is known about what is actually deposited on the packages, or how temperature, ventilation, and reactions between salt components might modify the composition of the deposited salts.
- **Goal of the EPRI Sampling Program:** Assess the composition of dust on the surface of in-service stainless steel SNF storage canisters, with emphasis on the composition of the soluble salts, that could deliquesce as the canisters cool.
- ISFSI locations to be sampled:
 - Calvert Cliffs: Transnuclear NUHOMS system, horizontal storage canister (samples analyzed and results presented here)
 - Hope Creek: Holtec HI-STORM system, vertical canister (samples currently being analyzed)
 - Diablo Canyon: Holtec HI-STORM system (scheduled week of Dec 2, CANCELLED)

Types of Samples Collected

- **Dry dust samples**
 - Used to characterize soluble/insoluble components (chemistry, mineralogy, texture); provide a sample, but cannot quantify amount per unit area
 - Calvert Cliffs
 - Transnuclear-designed sampling tool
 - Aggressive Scotch-brite® pad (coarse nylon fibers, alumina grains, trace titanium oxide, bonded together with resin)
 - Polyester fiber filter (rated 5 μm)
 - Vacuum suction pulls dislodged dust through the pad and onto the filter
 - Hope Creek/Diablo Canyon
 - Holtec designed sampling tool
 - Less-aggressive Scotch-brite® pad (no abrasive), no filter, no vacuum
- **Wet samples (soluble salts)**.
 - Used to characterize soluble salts (quantify amount per unit area)
 - Salt-smart® sensors

Analysis Methods

- **SEM imaging and energy dispersive system (EDS) element maps**
 - Provide textural and mineralogical information
 - Identification of floral/faunal fragments in dust
- **X-ray fluorescence**
 - Micro-analytical technique—allows chemical mapping of the filter/pad surfaces with a resolution of ~50 µm
 - Provides semi-quantitative chemical analyses—yields element ratios that can be used in mass balance calculations
 - Cannot detect elements lighter than sodium (e.g., oxygen, nitrogen)
- **X-ray diffraction**
 - Analysis of pads/filters for mineralogical information
- **Raman spectroscopy**
 - Microscopic technique provides mineralogical/compositional information of individual dust grains, if sufficiently large
- **GC-MS**
 - Stepwise thermal decomposition to drive off volatile, semi-volatile components in the dust
 - Only components driven off prior to decomposition of the filter/pad matrix can be observed
- **FTIR analysis**
 - Samples treated with solvents, and the solvents analyzed to determine volatile, semi-volatile organic components leached from the sample
- **Chemical Analysis**
 - Filter, pad, and Salt-smart® samples leached with DI water, and the leachate analyzed to determine soluble salts in the dust
 - Insoluble fractions digested and analyzed to determine bulk chemistry

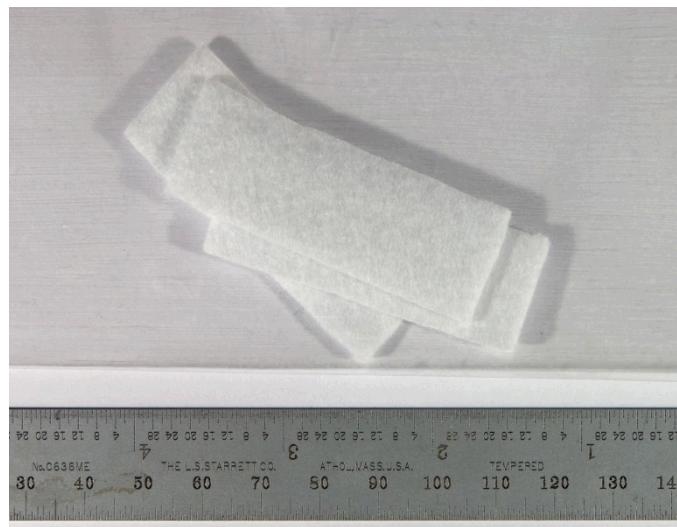
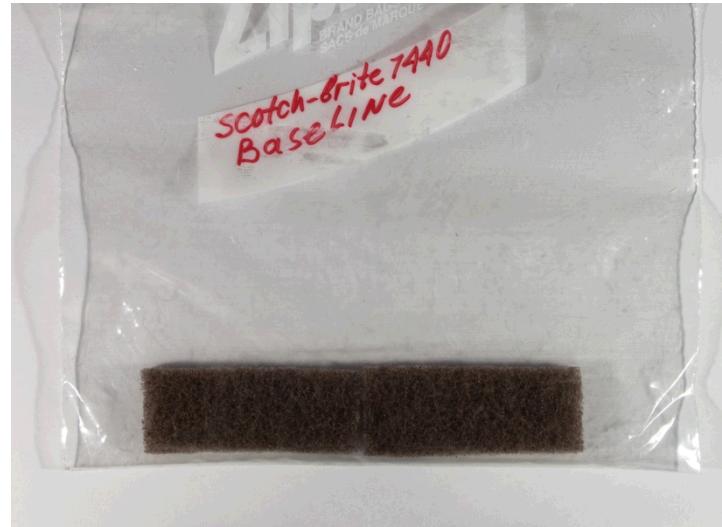
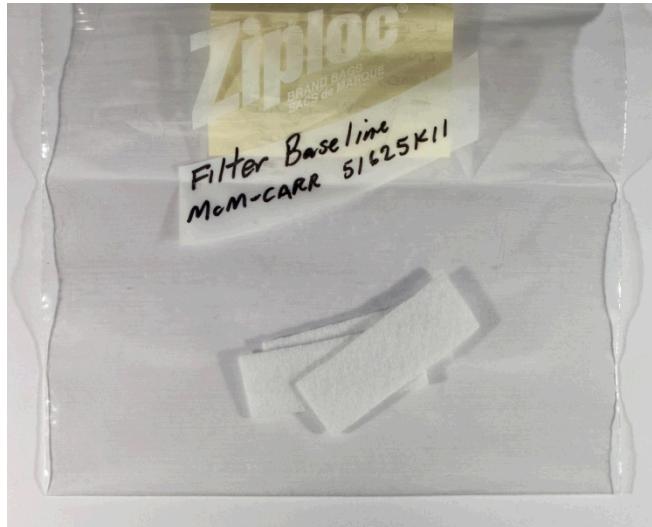
Analysis of Calvert Cliffs Samples

Samples stored for several months prior to analysis

- One dry dust sample and one Saltsmart® sample were analyzed by an external lab (Evans Analytical Group)
- Two dry dust samples were provided to Sandia
 - Filter and Scotch-Brite® pad on steel sampling head
 - Disassembled and pad and filter removed for analysis
 - EPRI #1 Sample: collected at 11 o'clock position
 - EPRI #1 filter
 - EPRI #1 pad
 - EPRI #2 Sample: collected at 7 o'clock position
 - EPRI #2 filter
 - EPRI #2 pad
 - Blanks provided
 - Filter blank
 - Pad blank

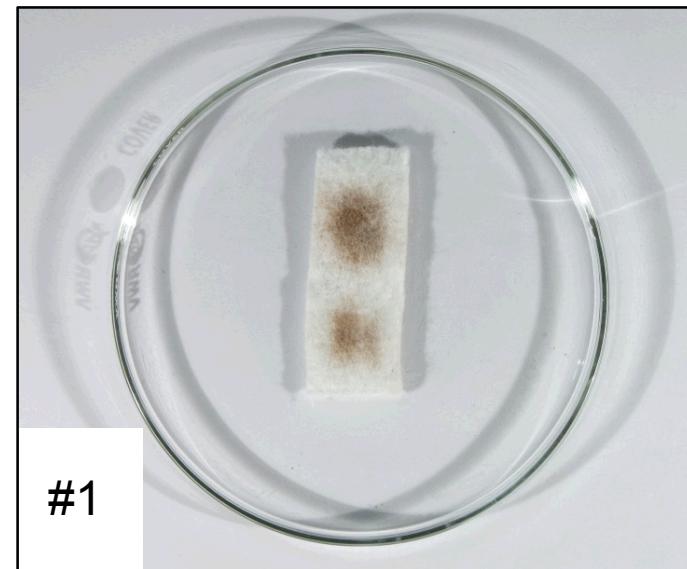
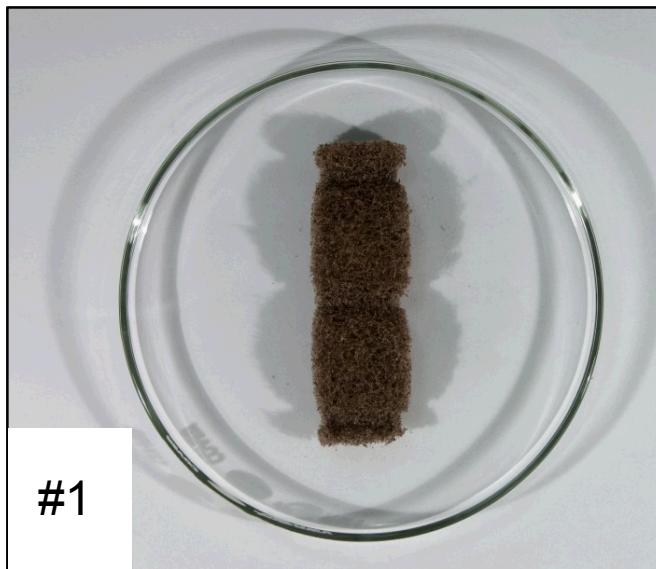
Photographs

Filter and Scotchbrite pad blanks:

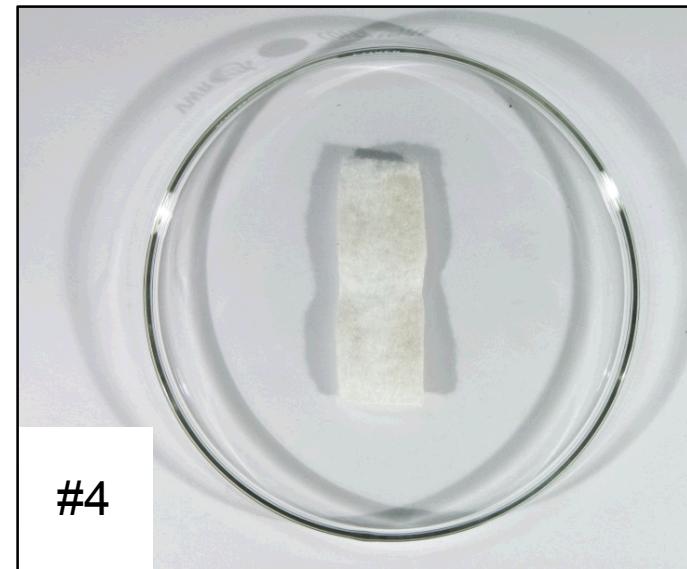
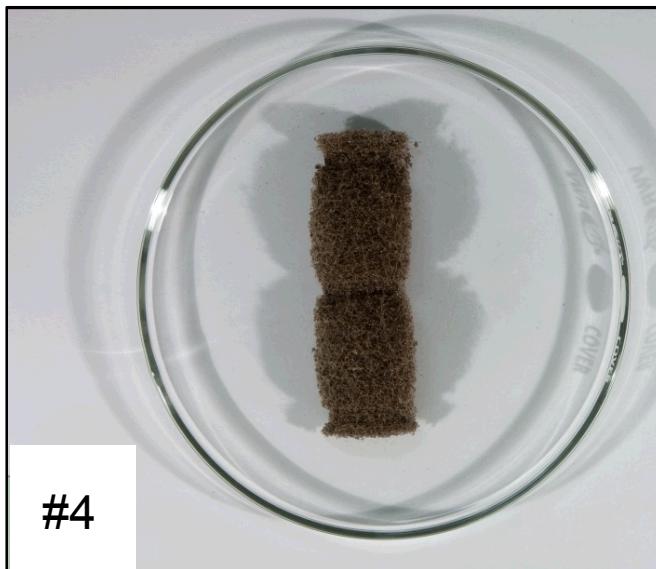


Pad and filter samples (after disassembly)

EPRI #1 sample
(11 o'clock)



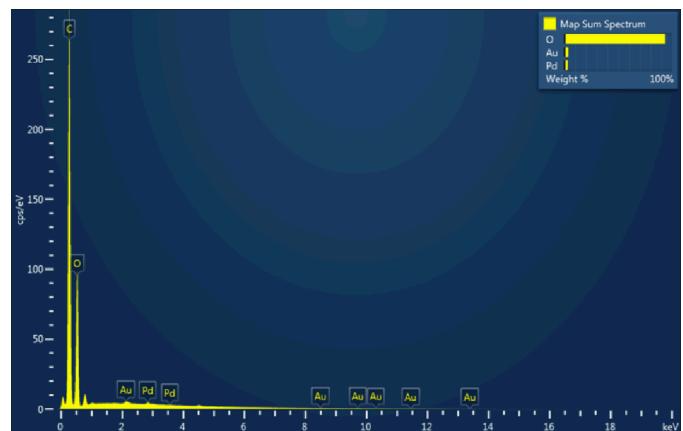
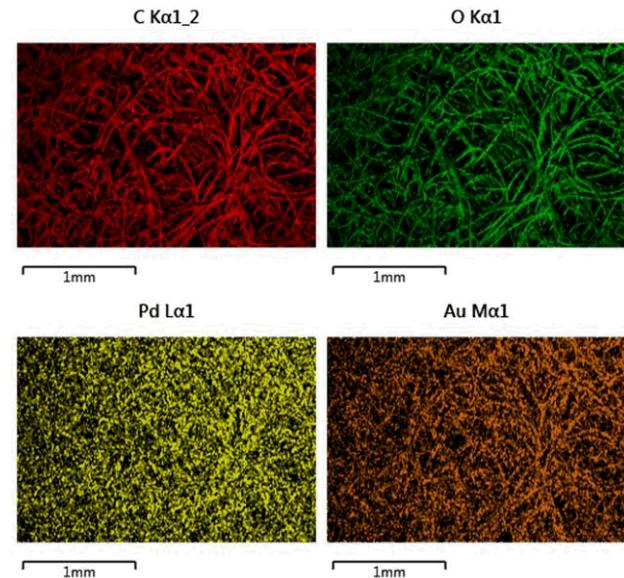
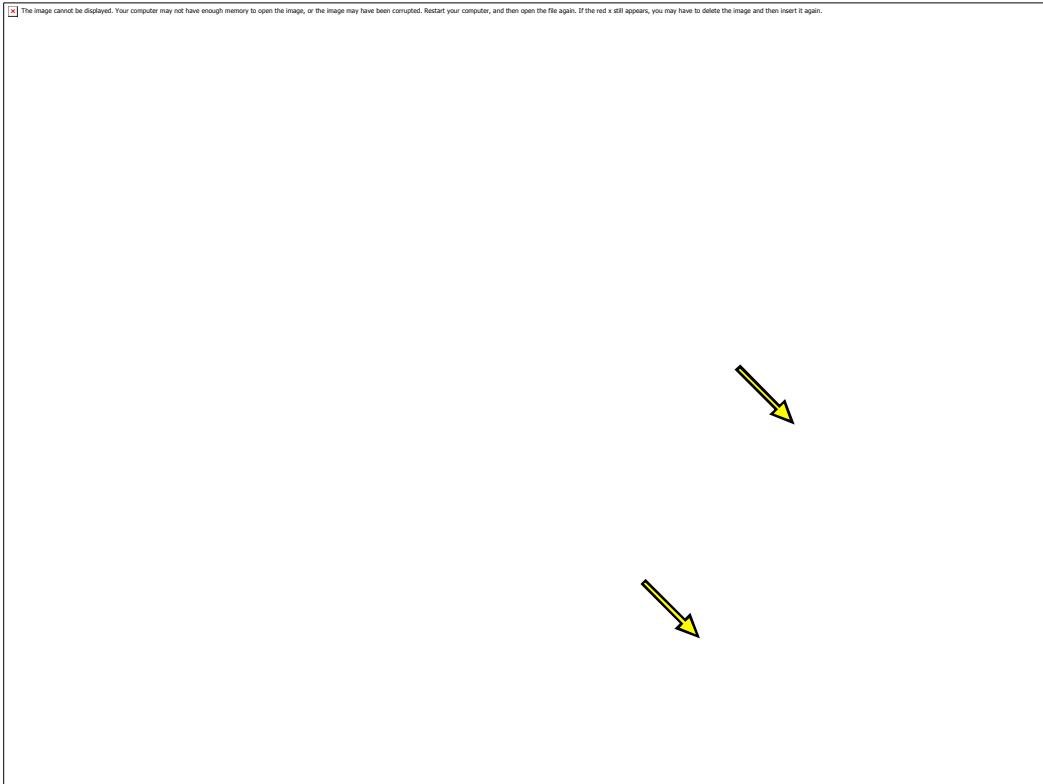
EPRI #4 sample
(7 o'clock)



SEM/EDS Analysis

EPRI filter blank

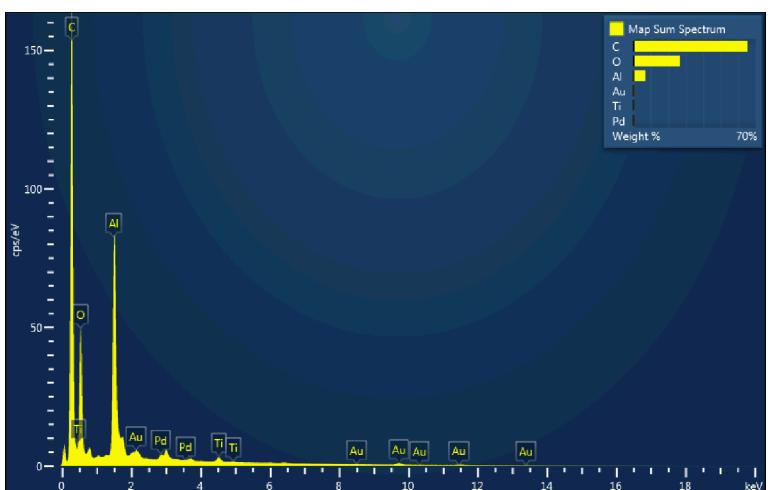
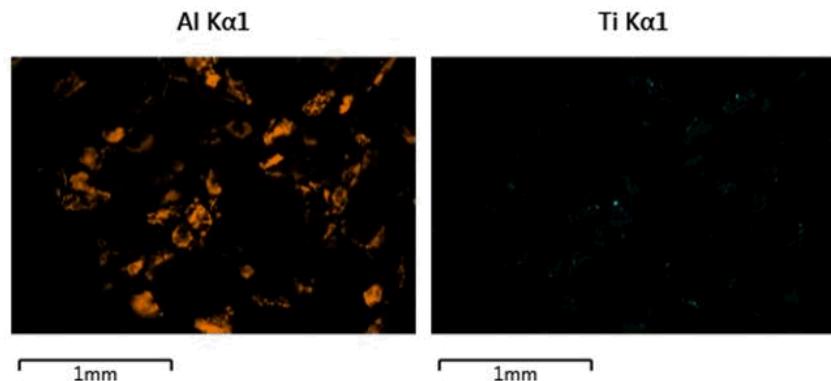
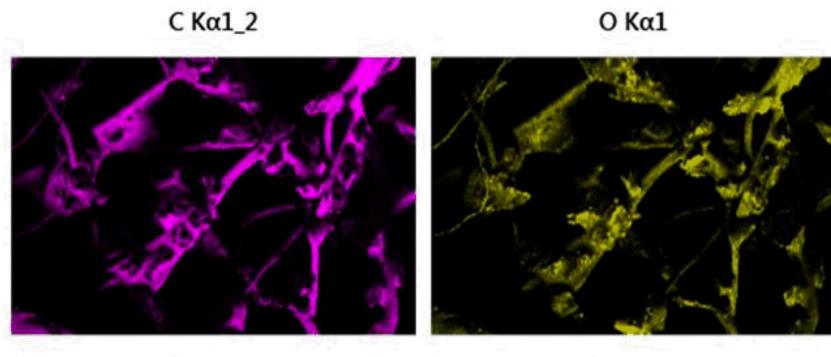
- Filter clean—sparse particulate contaminants
- Carbon, oxygen only significant components



SEM/EDS Analysis

EPRI Scotchbrite pad blank

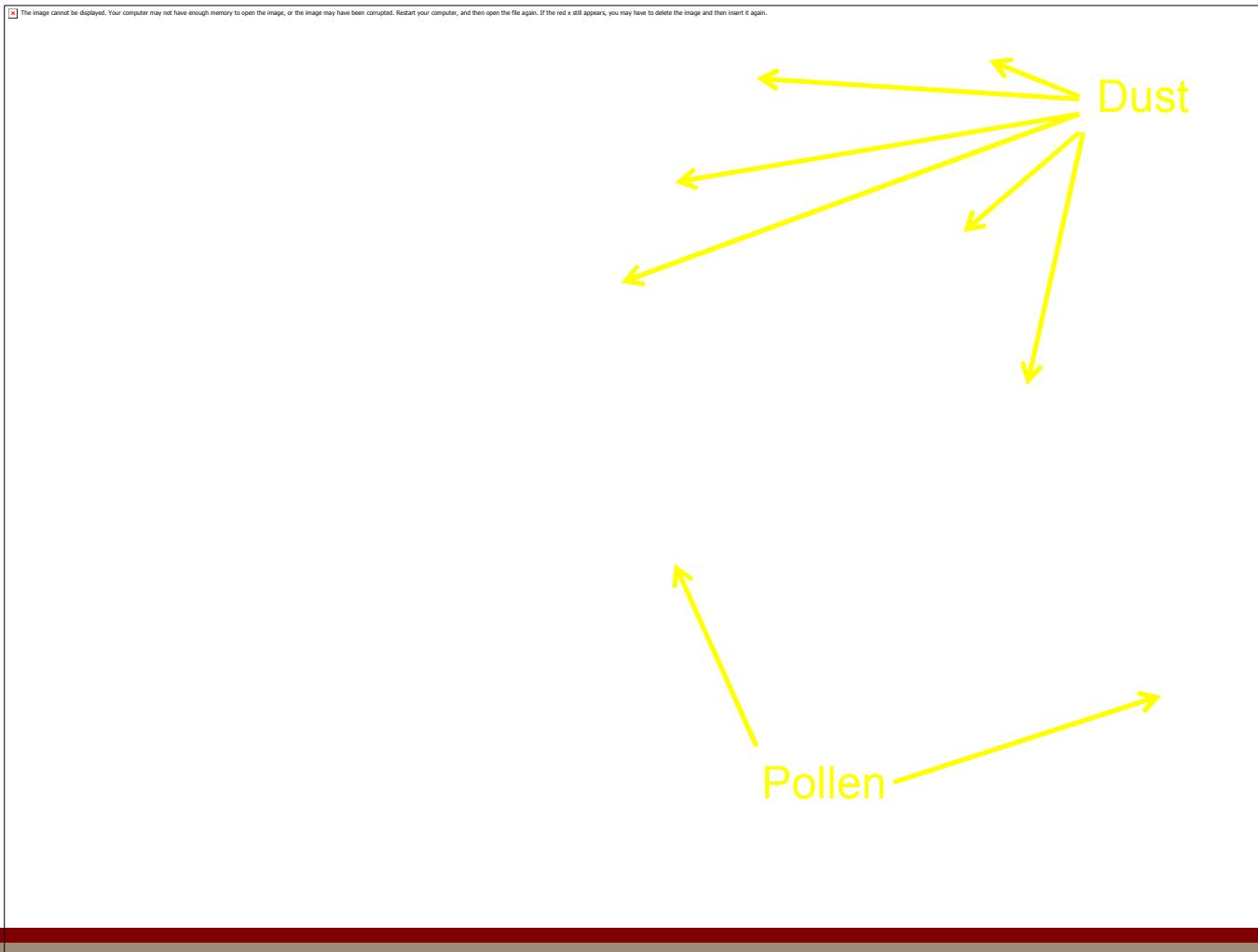
- Alumina grains and nylon fibers embedded in resin
- Trace titanium oxide particles
- Few contaminant particulates



SEM/EDS Analysis

EPRI #1 filter

- Major charging issues—hard to get good images.
- Fine dust particles adhering to fibers
- Coarse particles are dominantly pollen



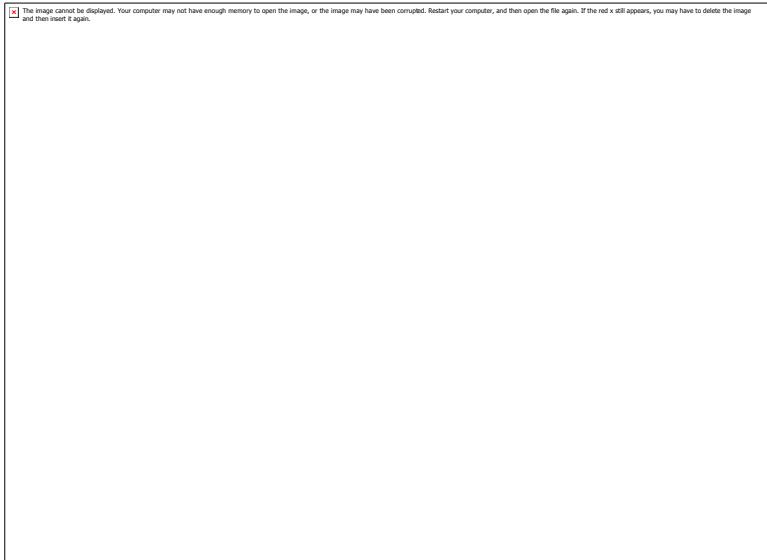
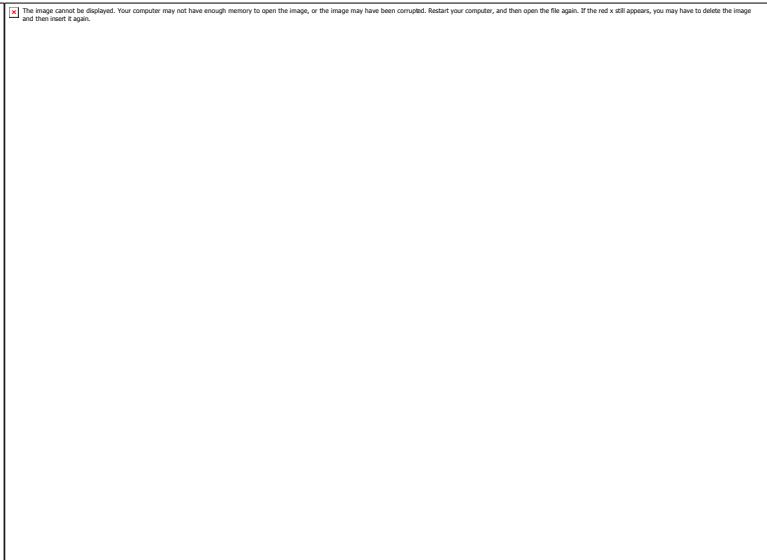
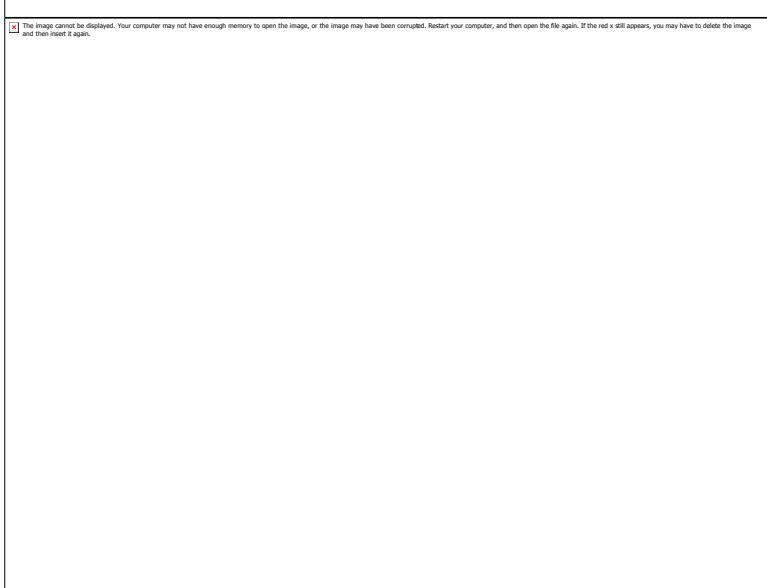
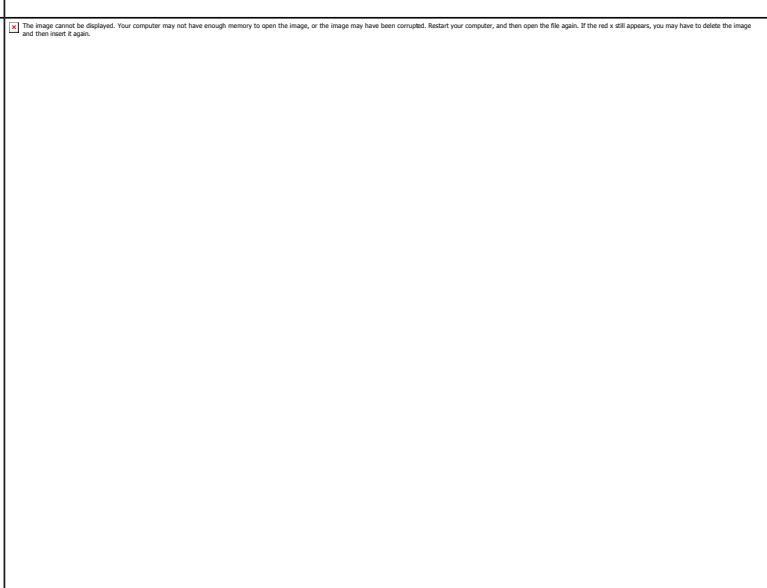
SEM/EDS Analysis

EPRI #1

filter

Plant, animal
matter.

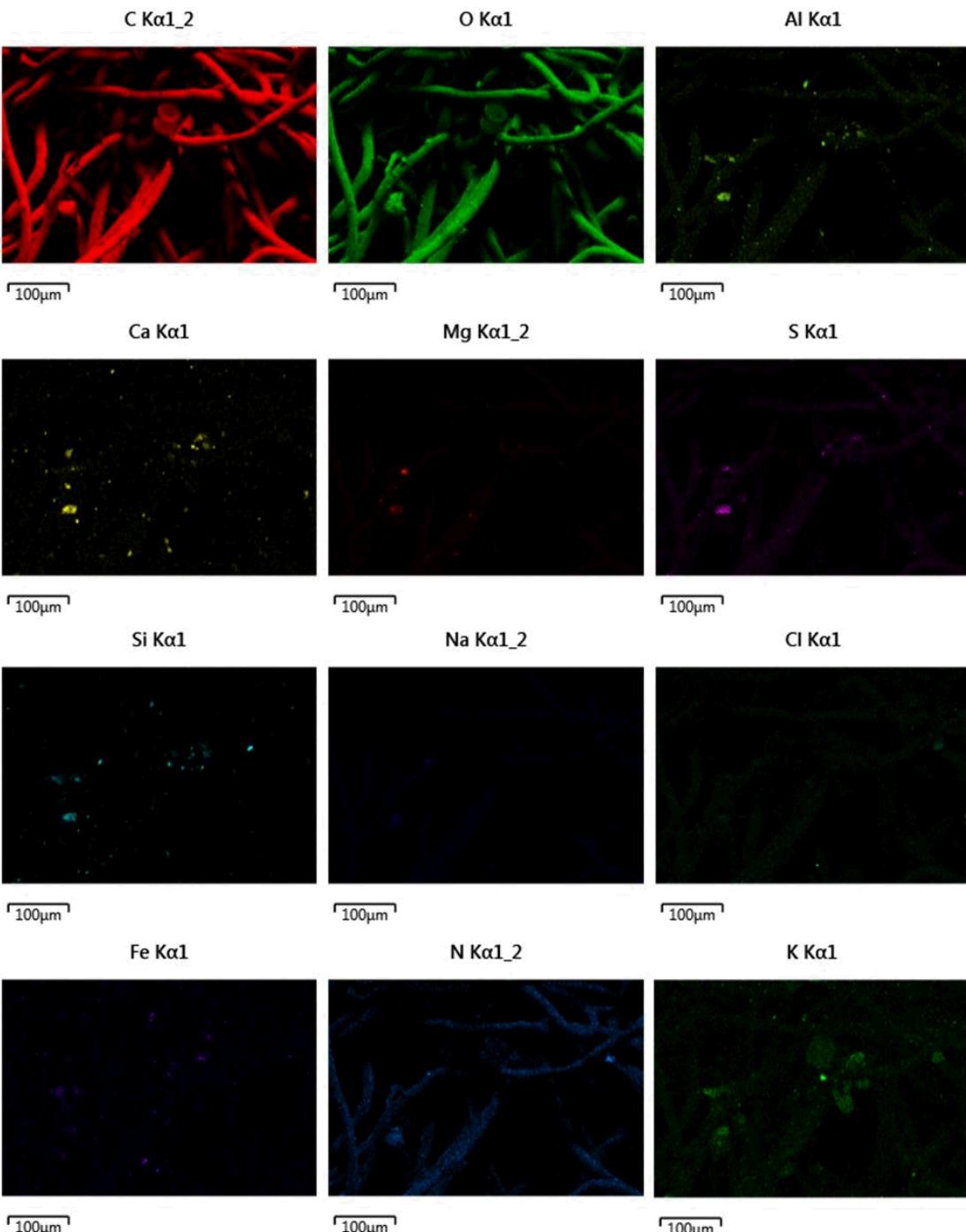
Pollen and
fibrous
materials

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SEM/EDS Analysis

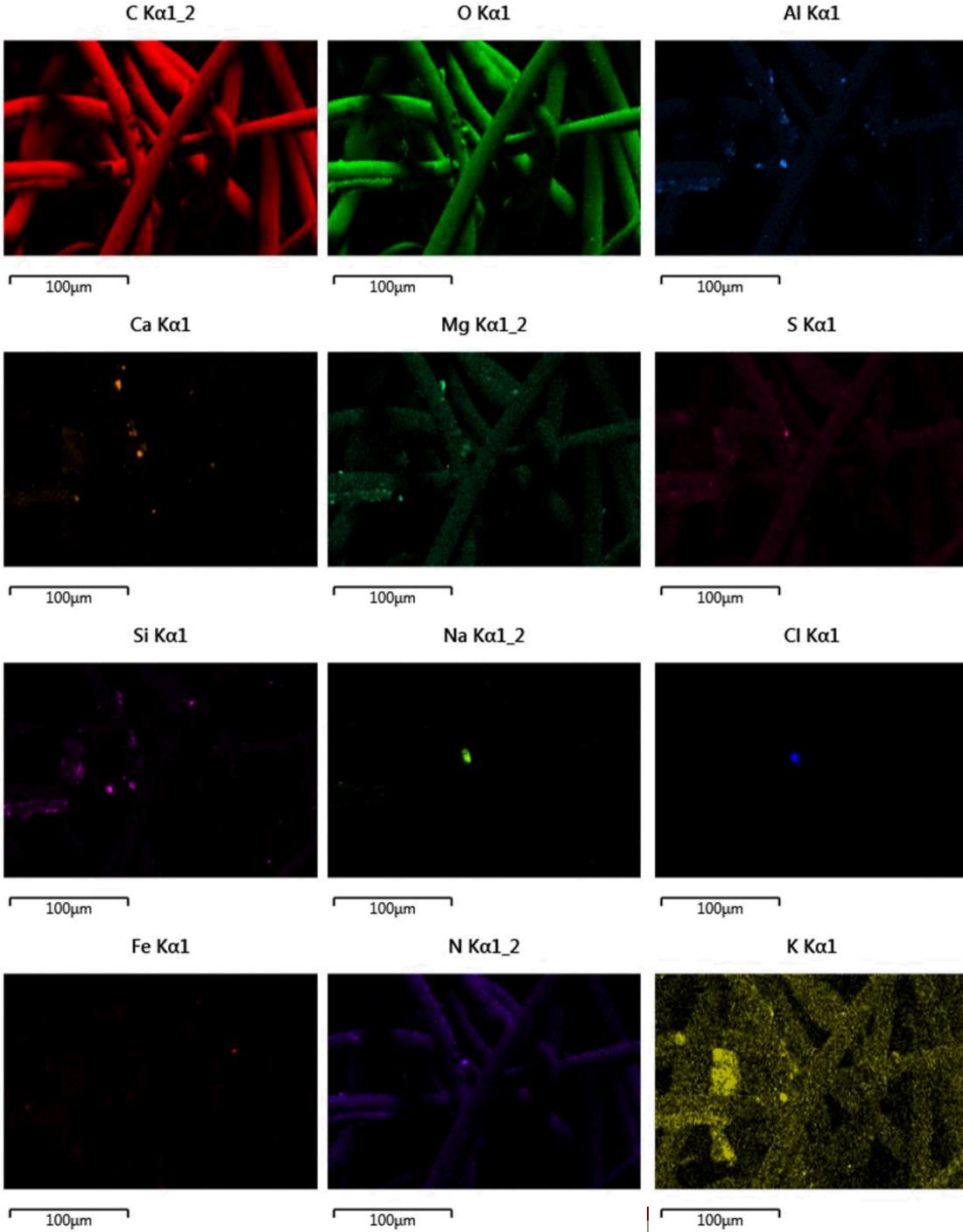
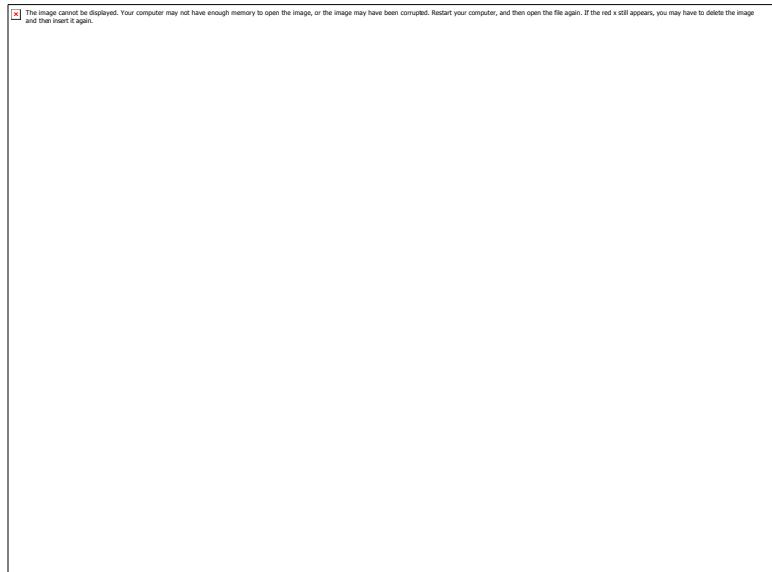
EPRI #1 filter

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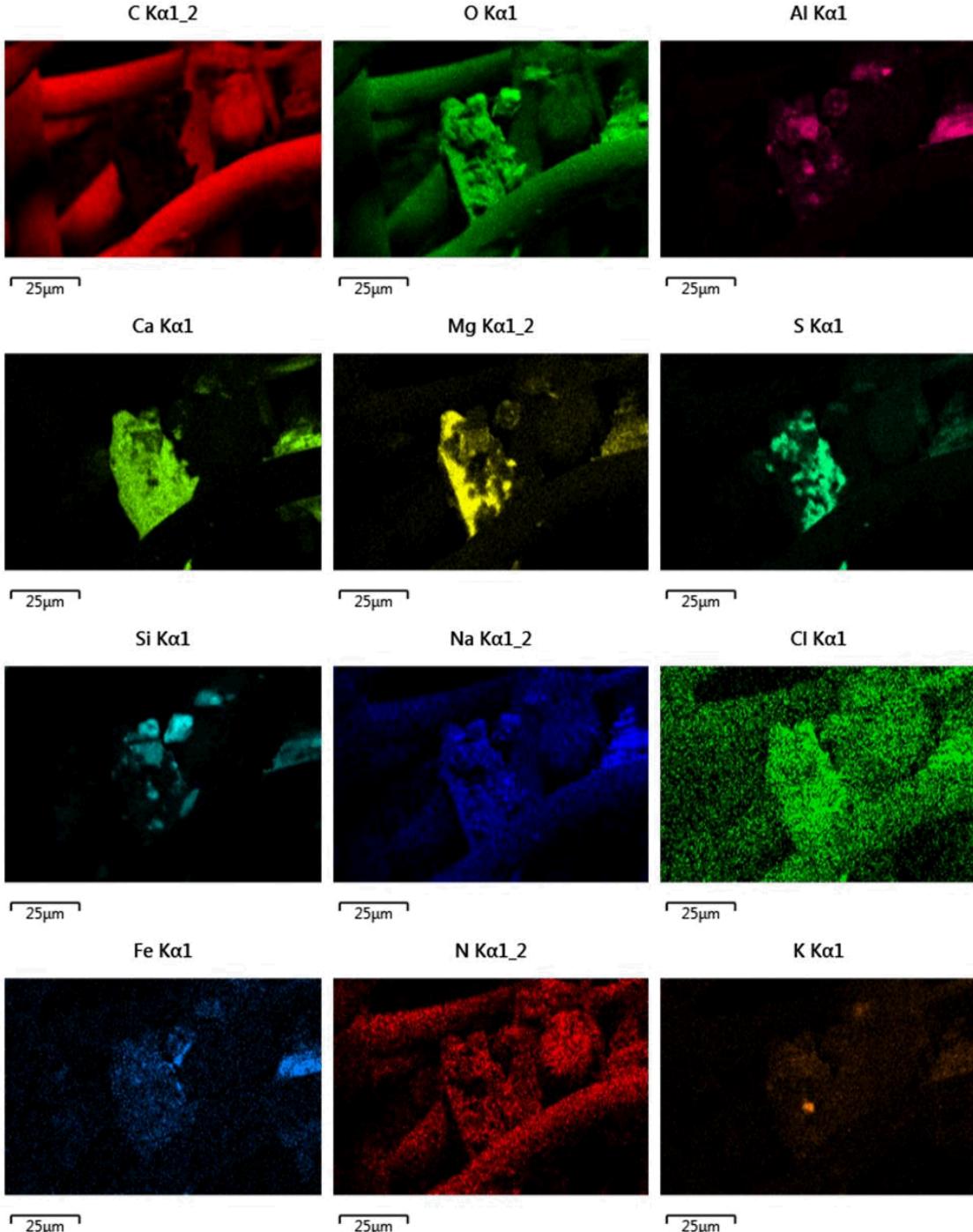
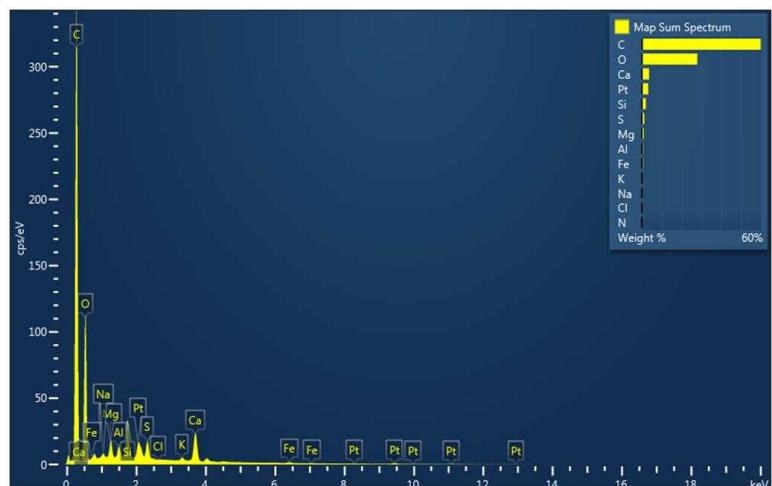
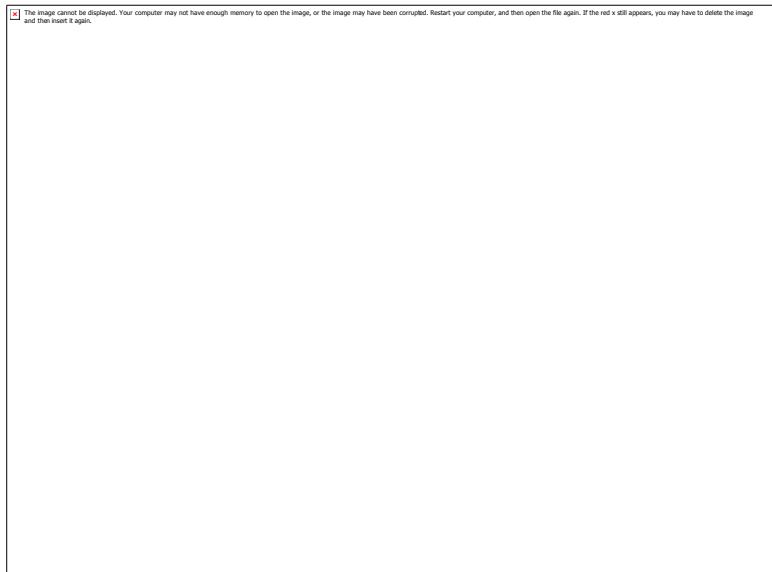
SEM/EDS Analysis

EPRI #1 filter



SEM/EDS Analysis

EPRI #1 filter



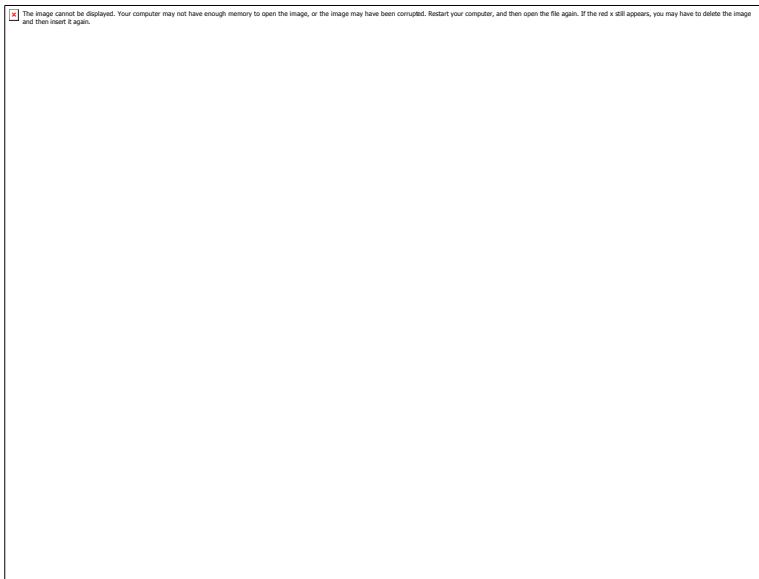
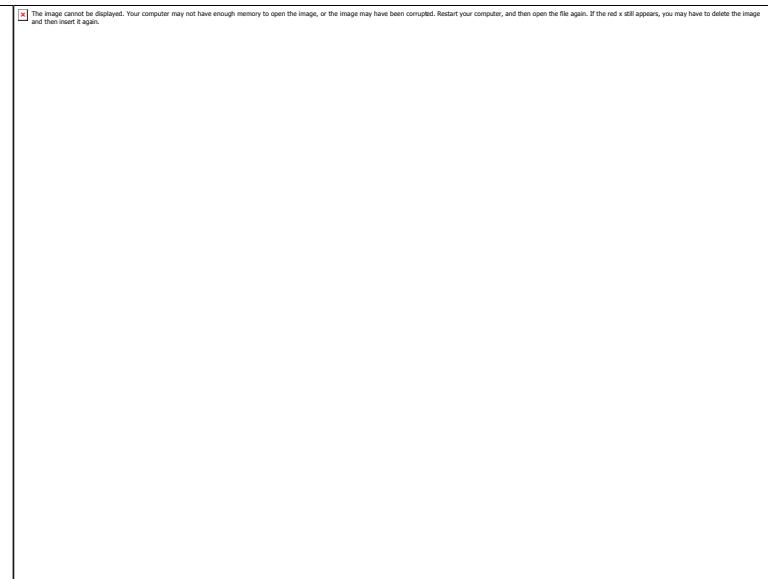
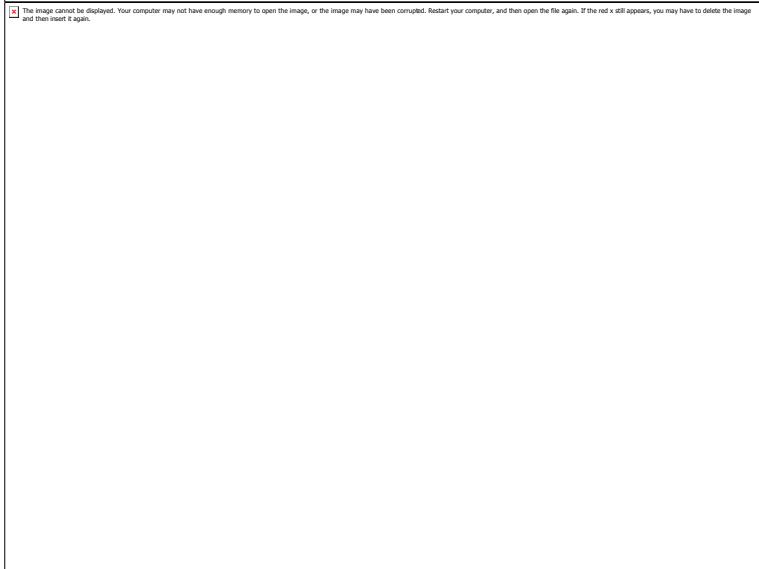
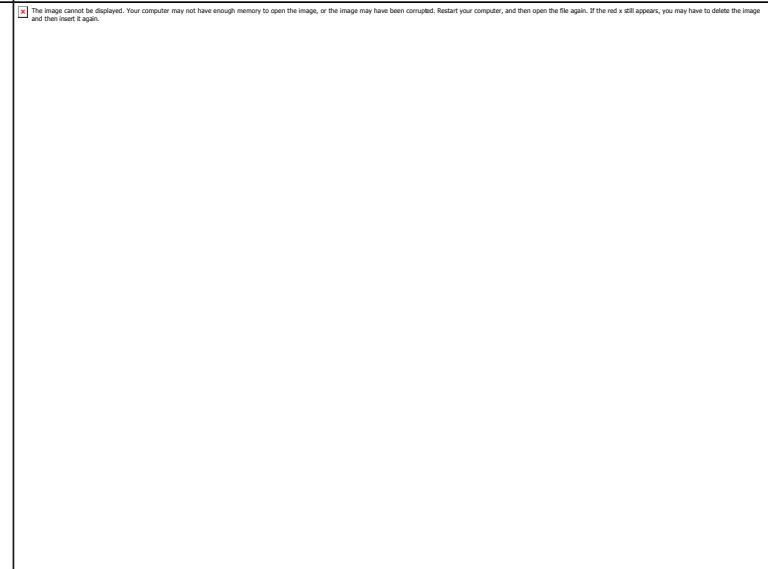
SEM/EDS Analysis

Dust particulates

EPRI #1 Scotchbrite

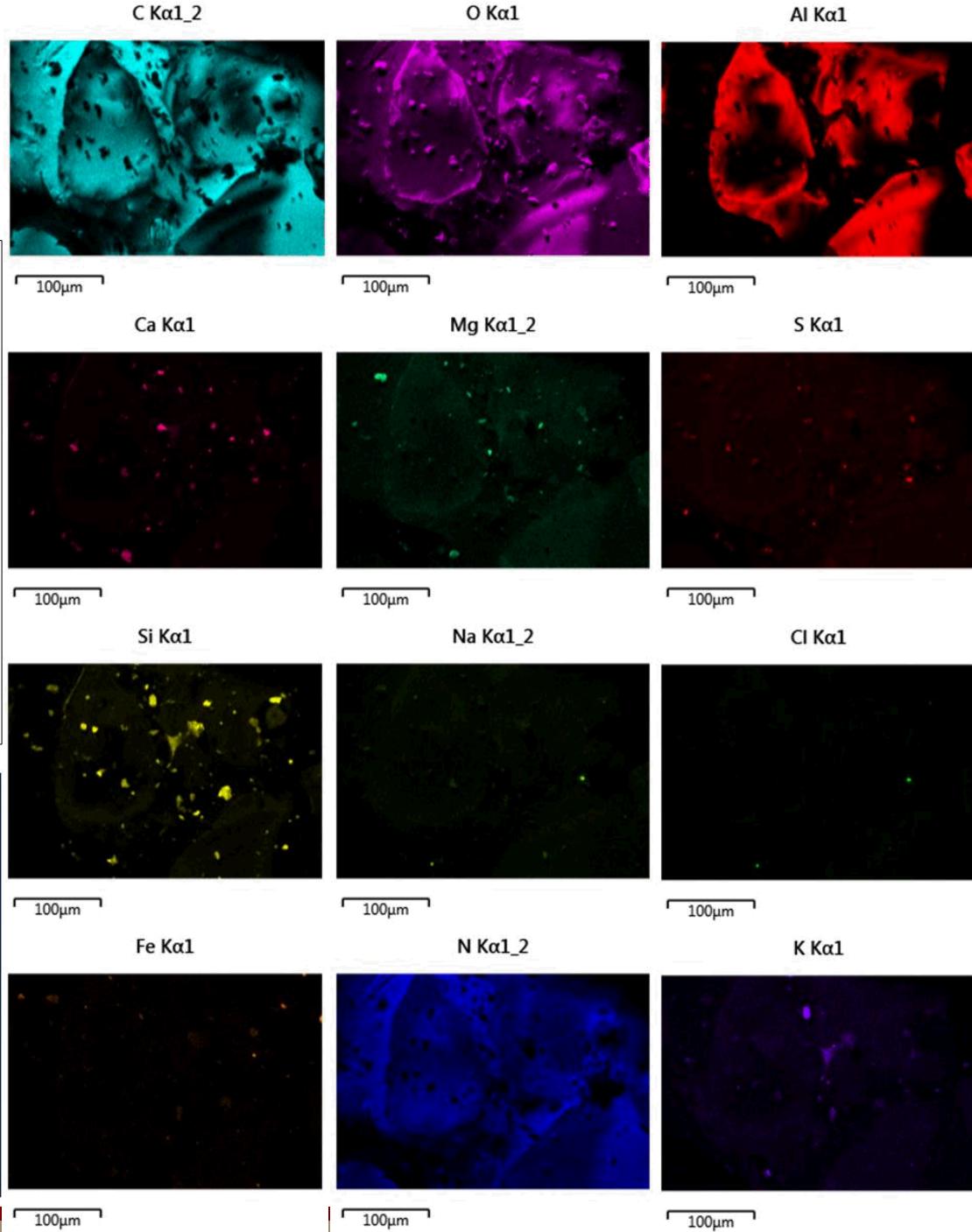
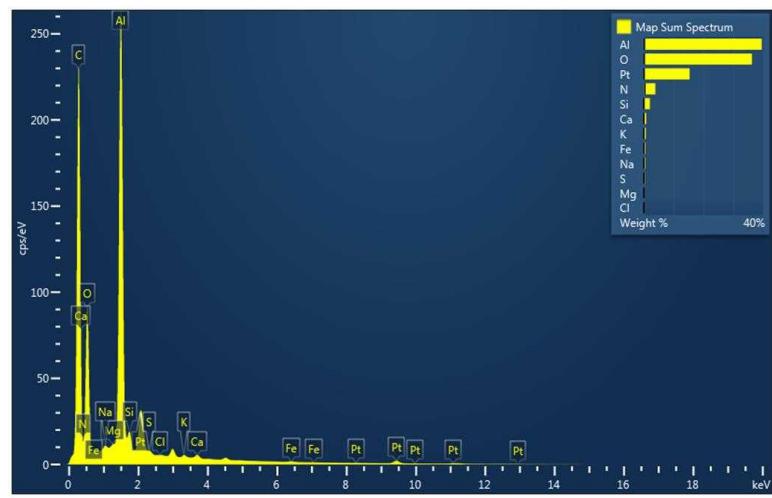
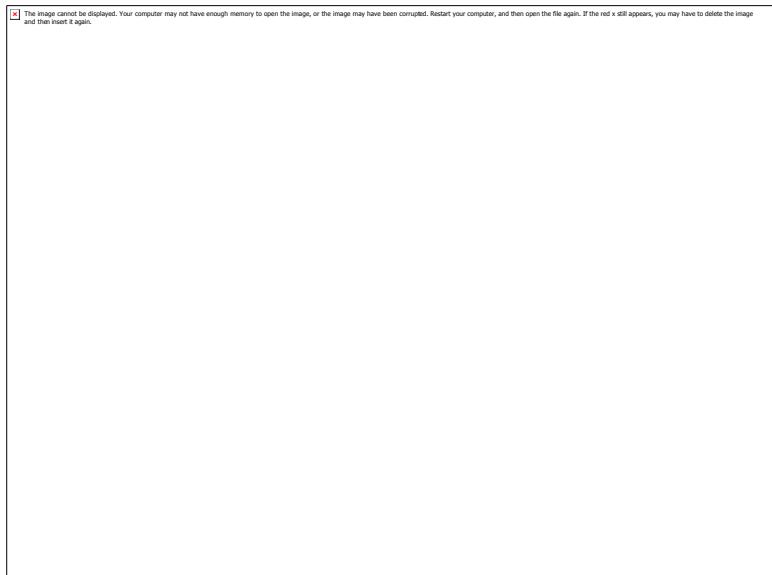
**Broken
alumina grain
with adhering
storage
container steel**

**Iron spheres—
fly-ash?**

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SEM/EDS Analysis

EPRI #1 Scotchbrite

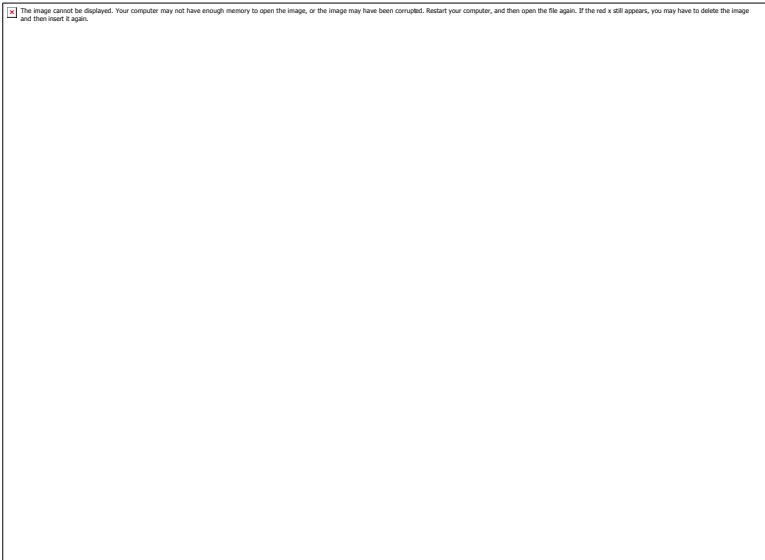


SEM/EDS Analysis

EPRI #4

filter

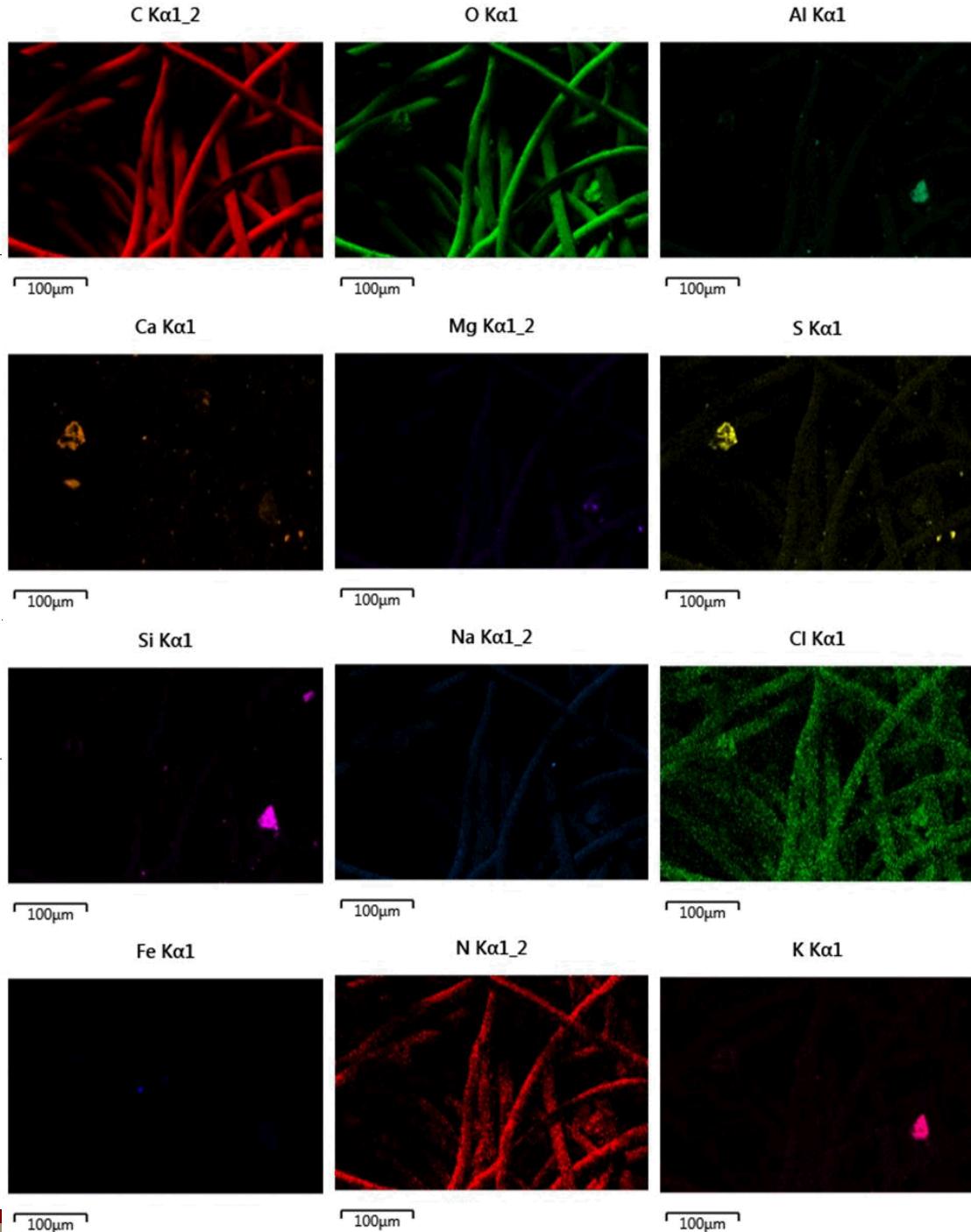
More lightly
loaded,
much less
pollen.

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SEM/EDS Analysis

EPRI #4 filter

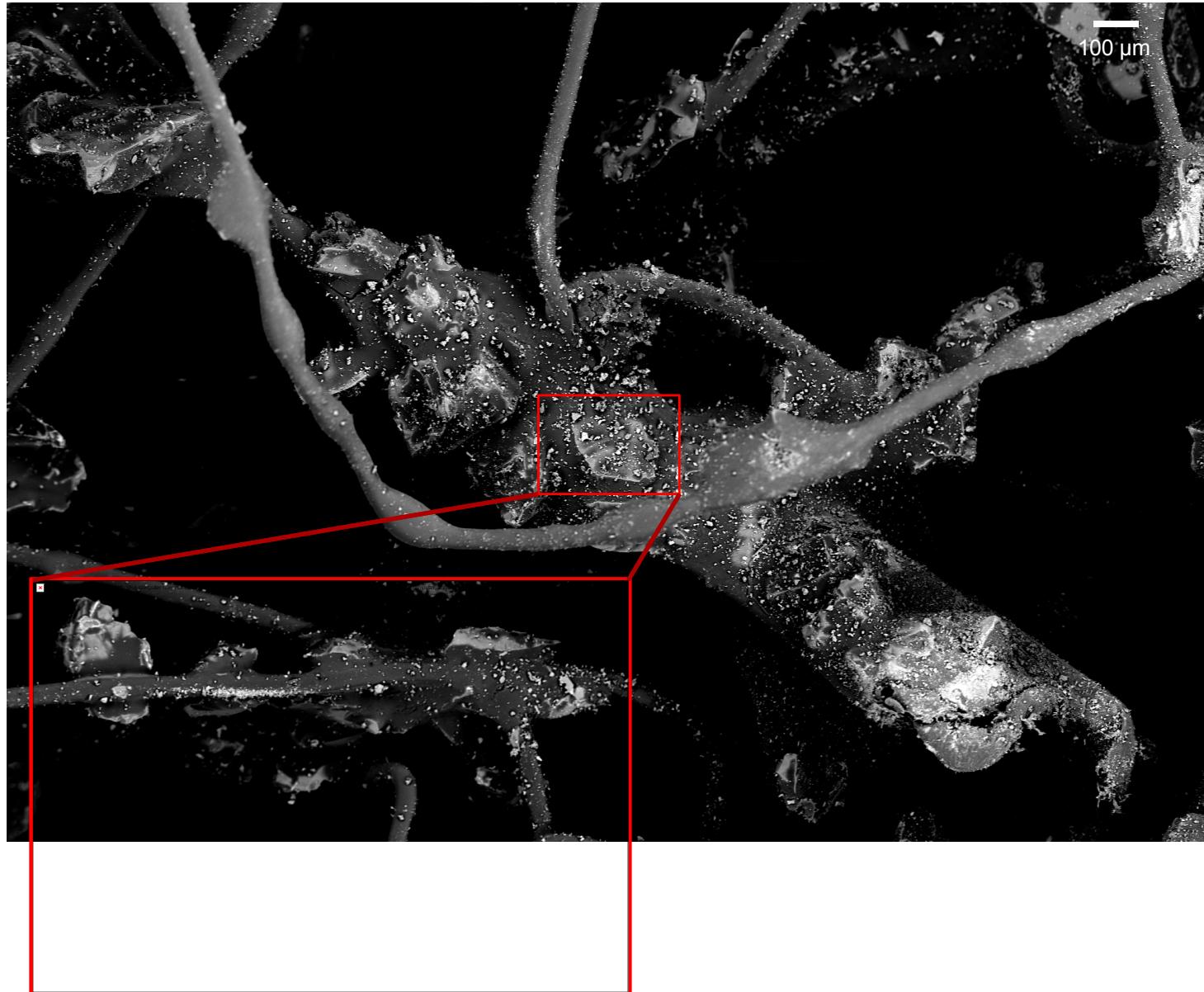
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SEM/EDS Analysis

EPRI #4 Scotchbrite

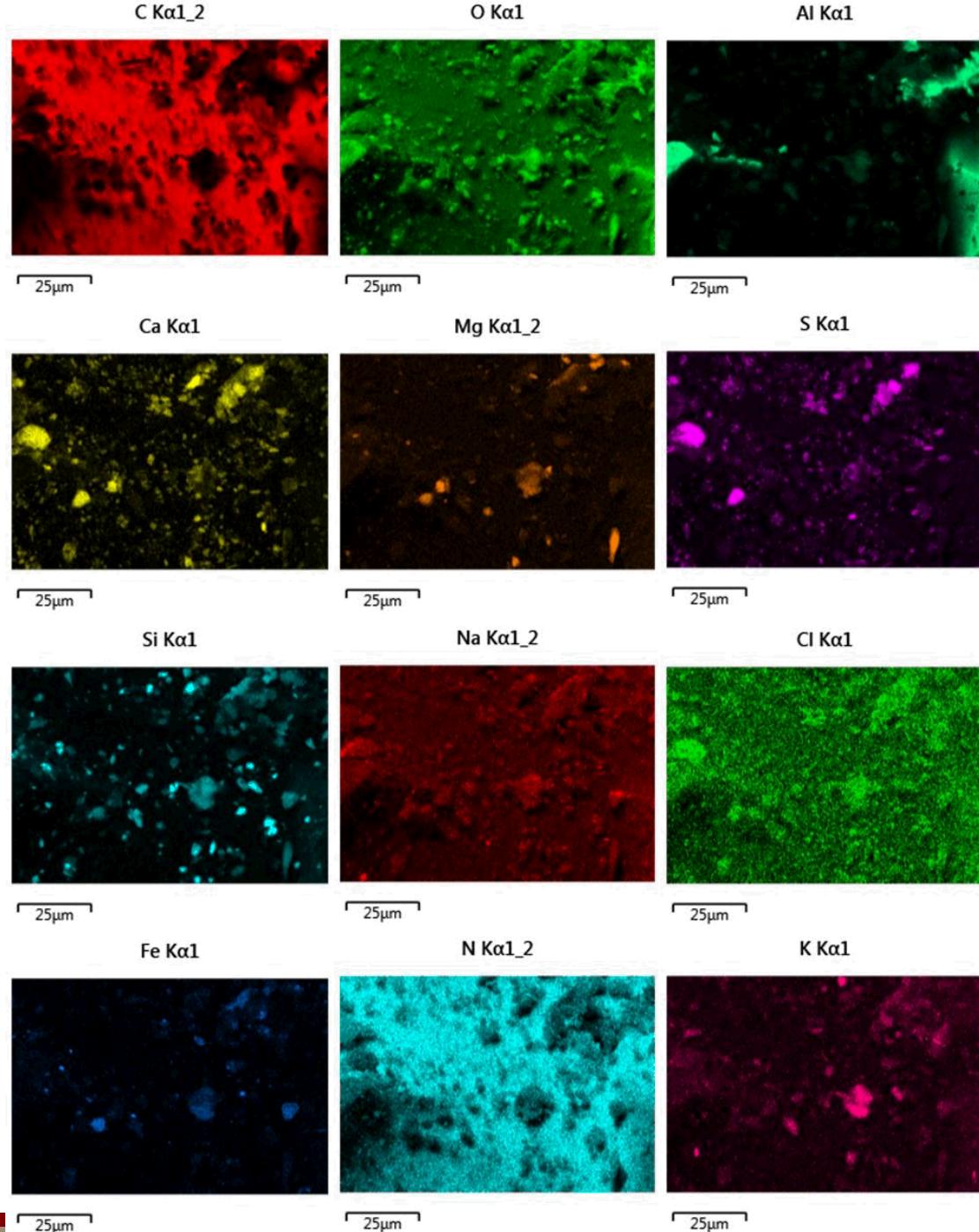
Heavily loaded
with dust
particulates



SEM/EDS Analysis

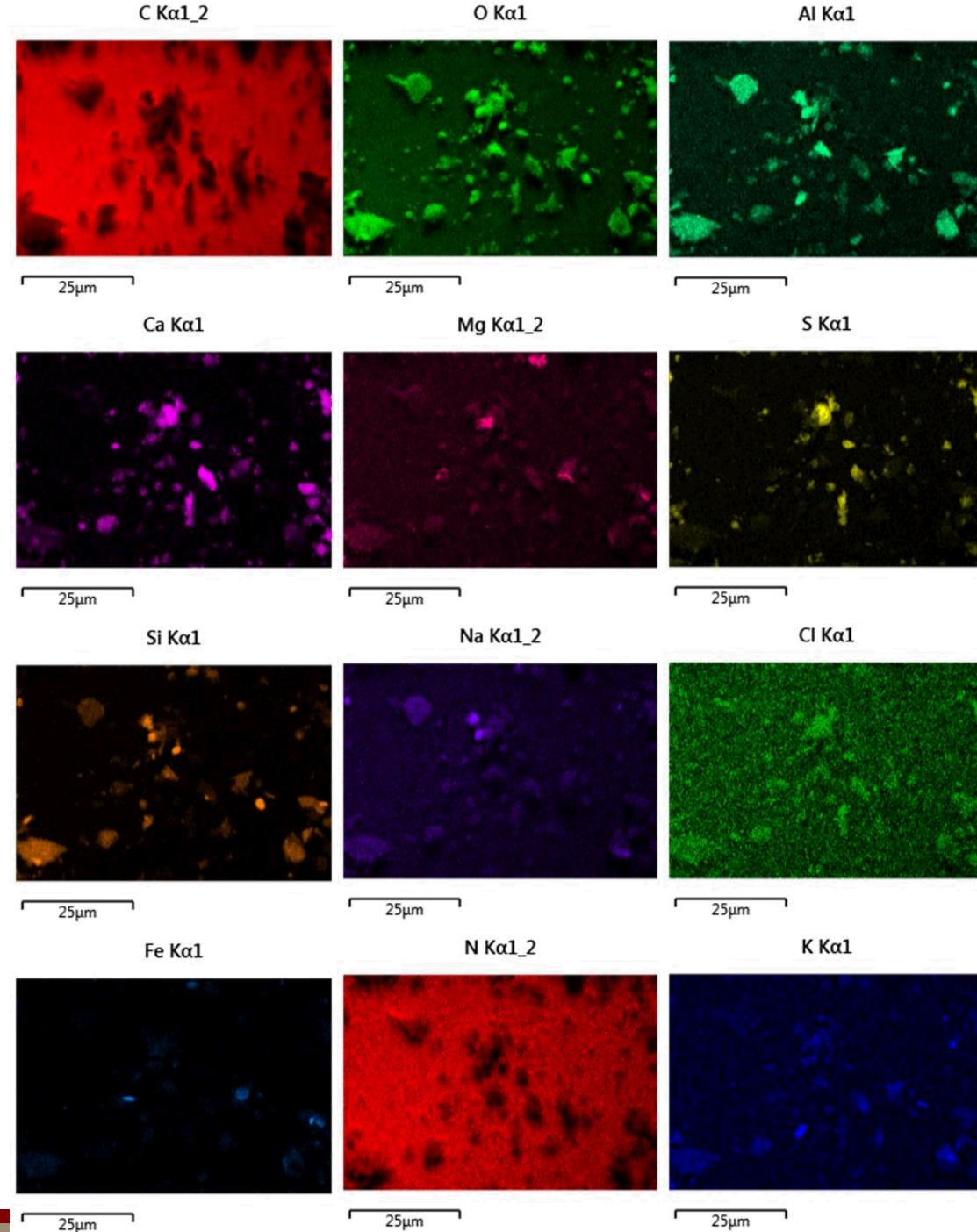
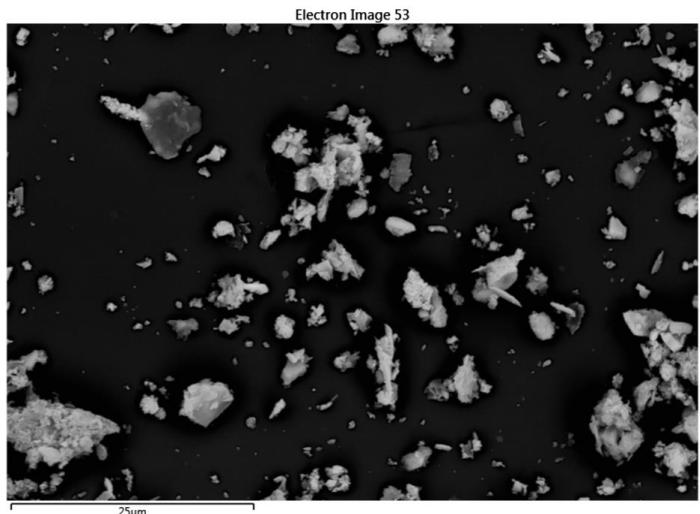
EPRI #4 Scotchbrite

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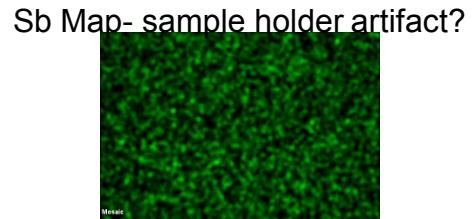
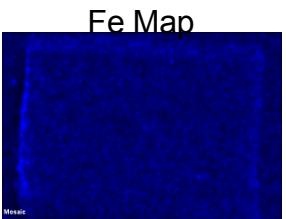
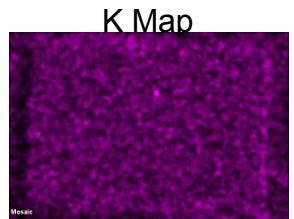
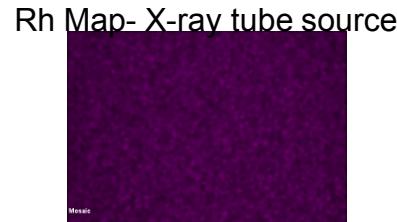
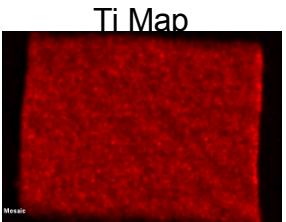
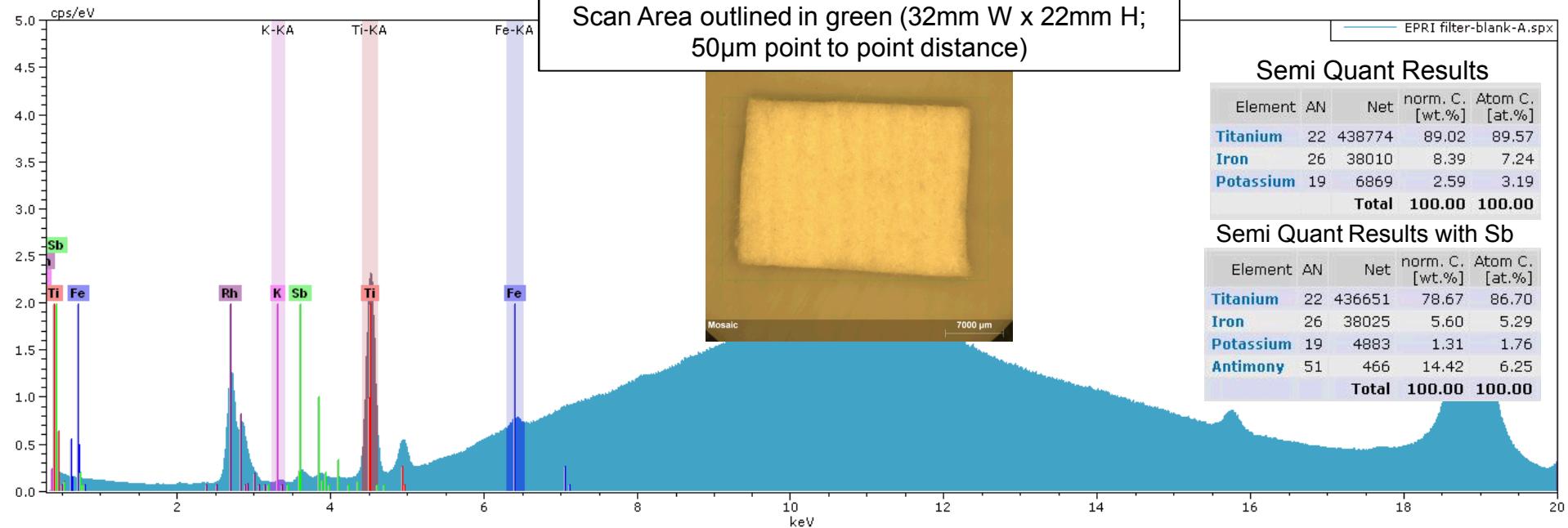


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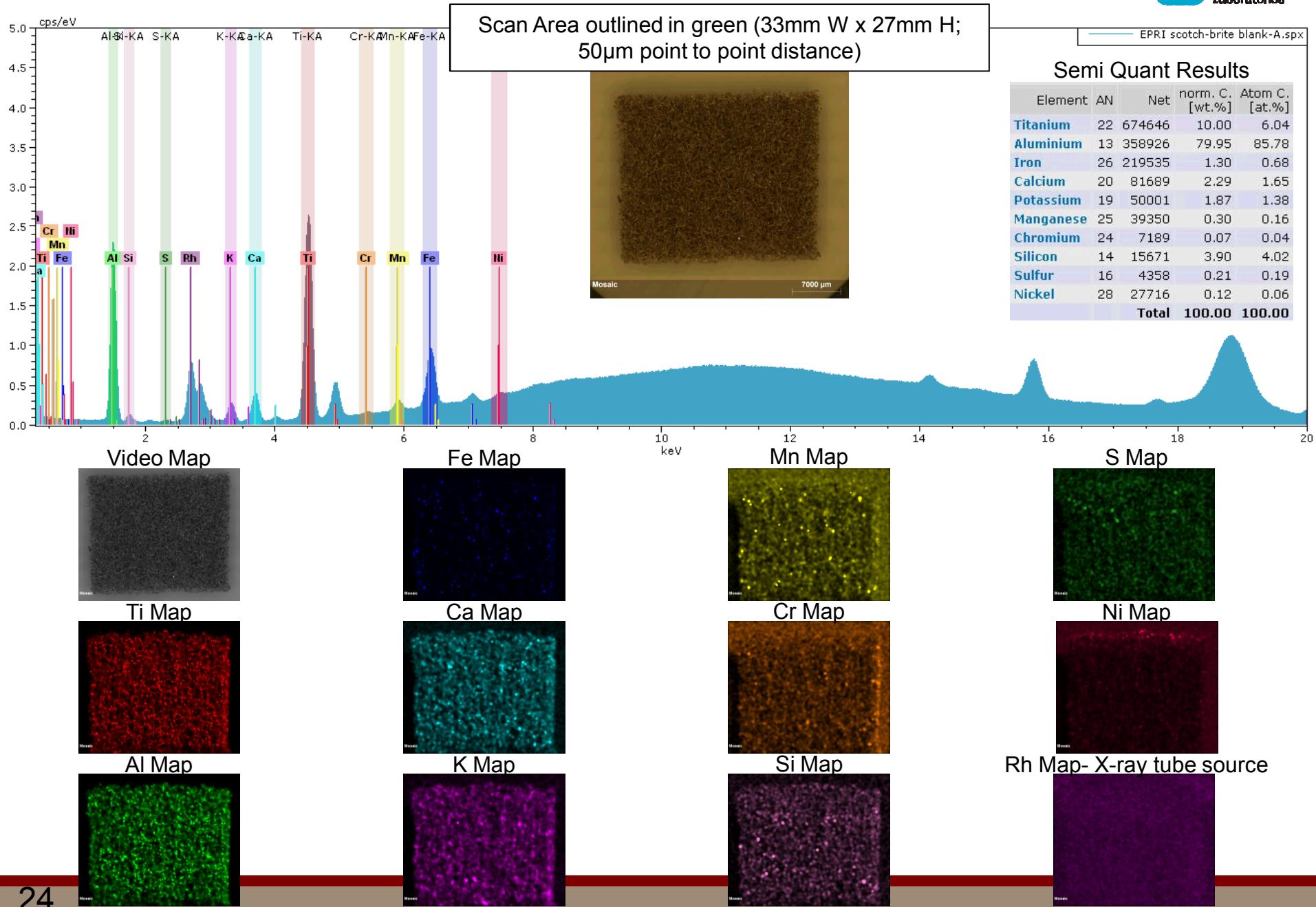
EPRI #4 Scotchbrite



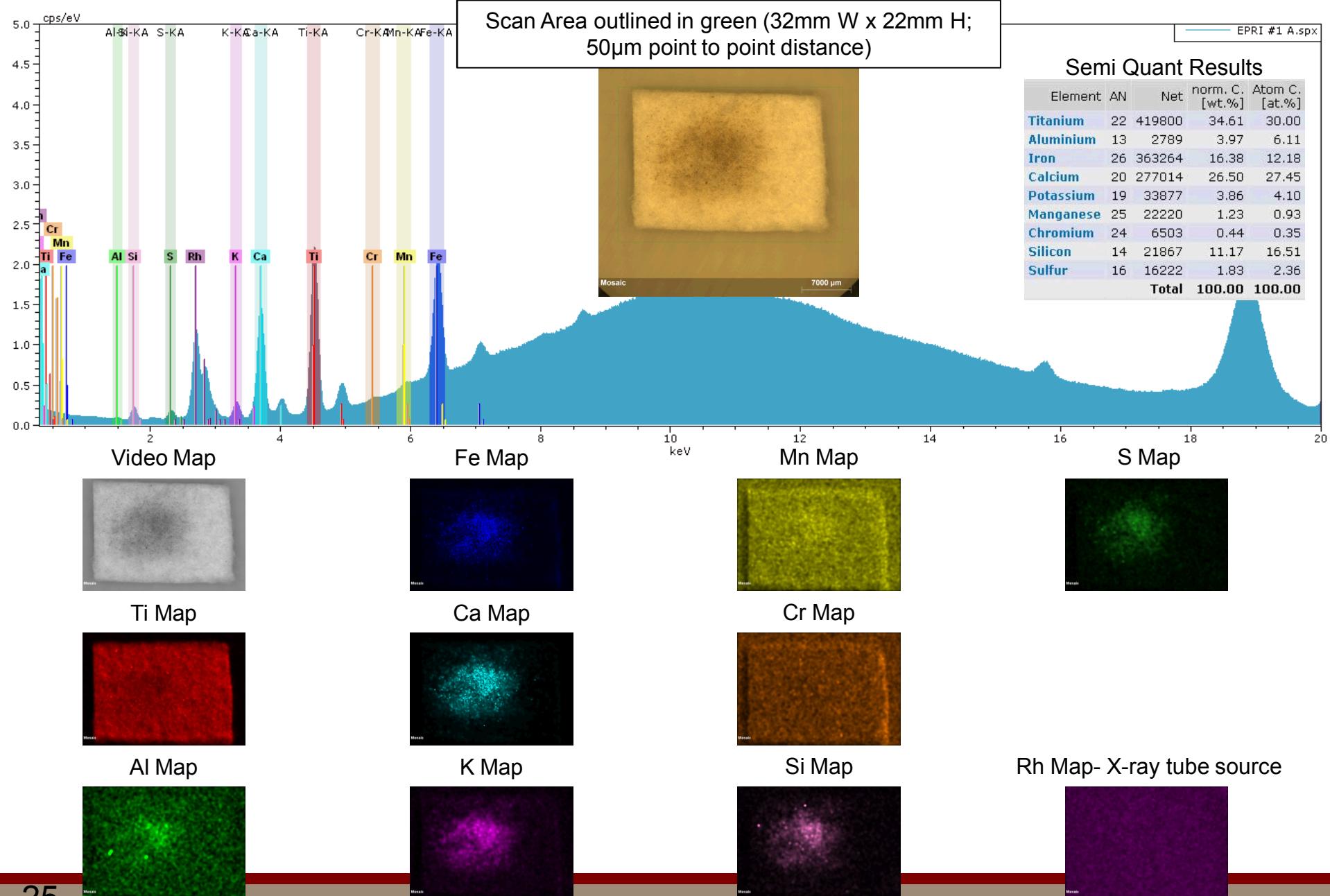
XRF Analysis: EPRI filter blank



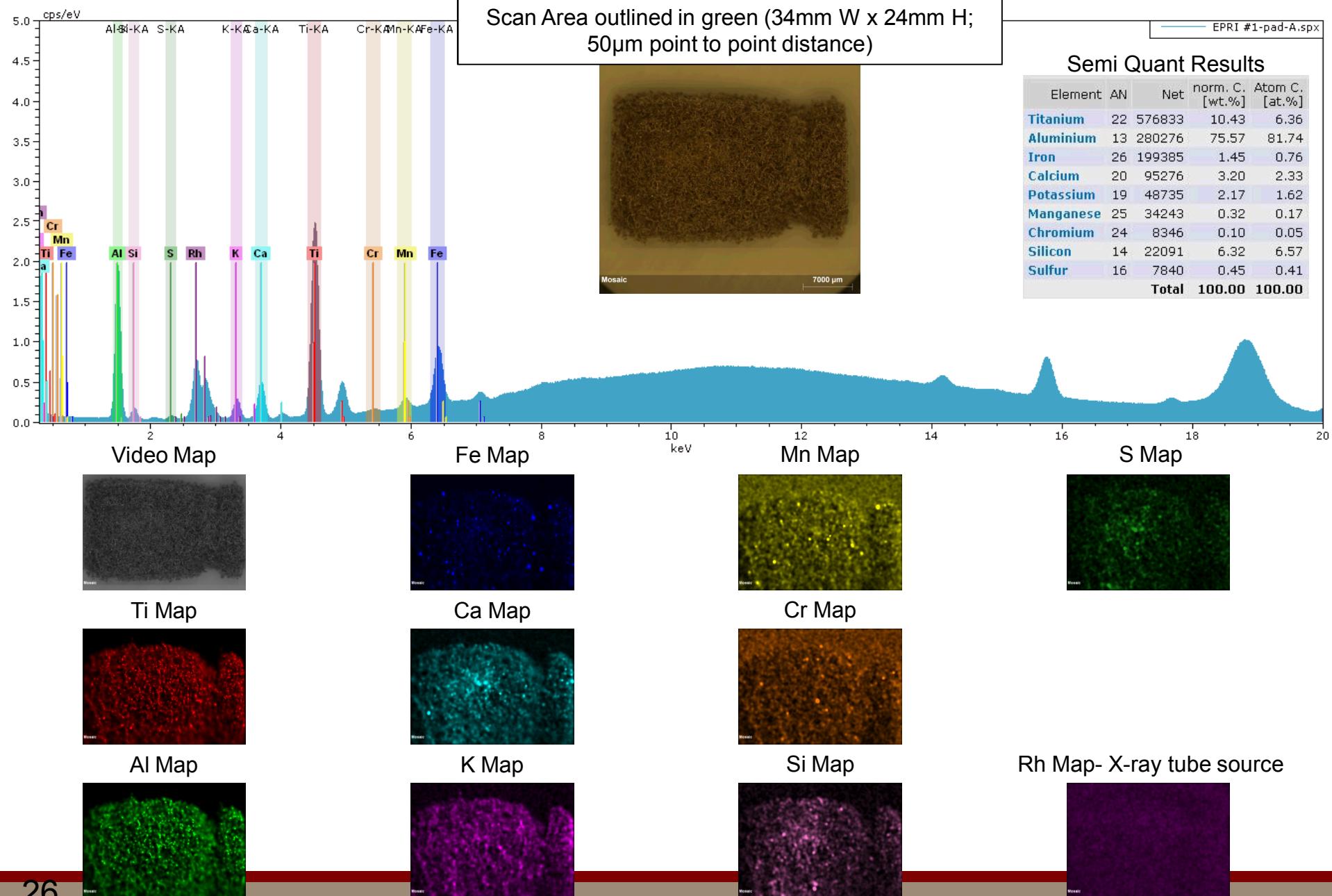
XRF analysis: EPRI scotch-brite blank



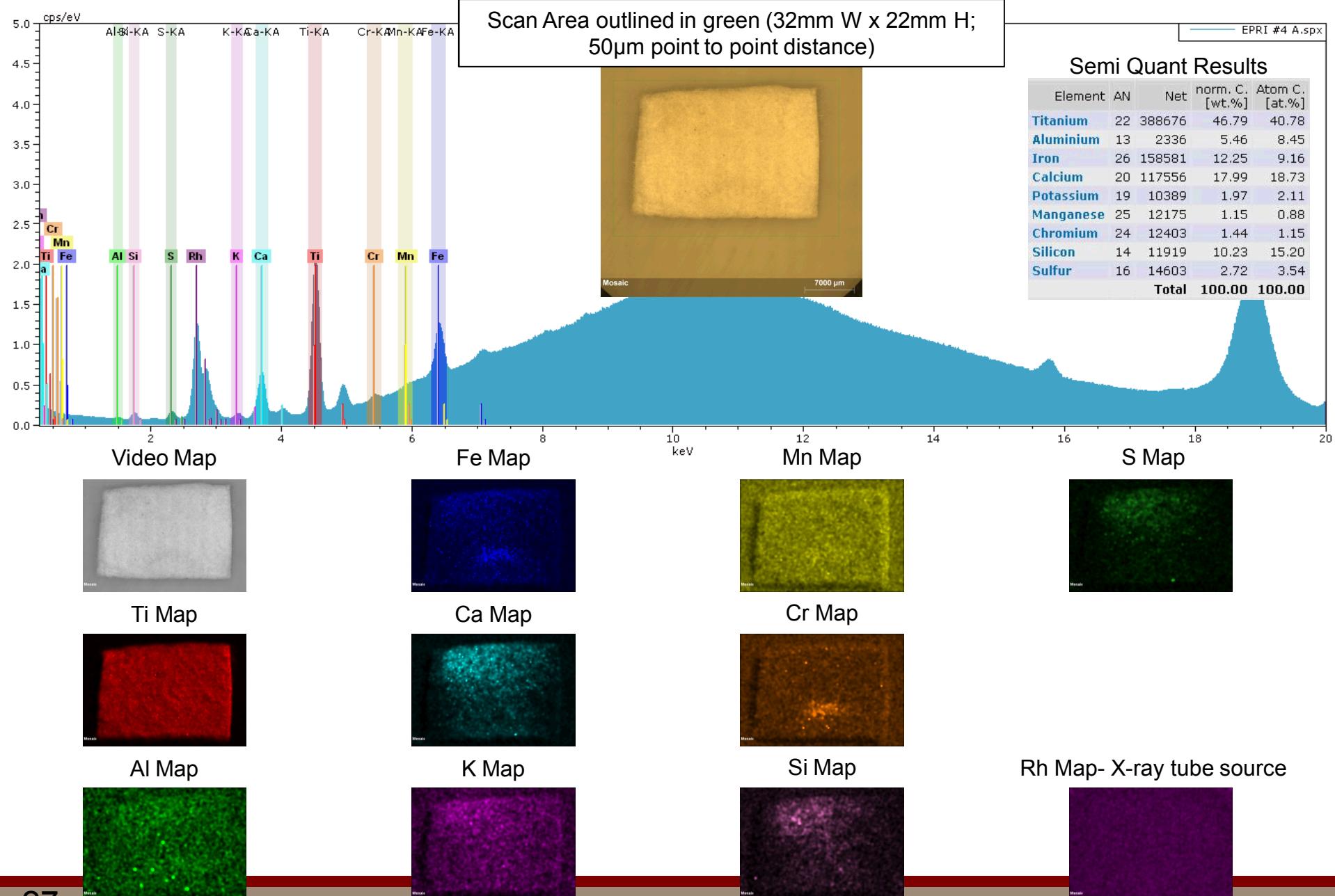
XRF Analysis: EPRI #1 filter



XRF Analysis: EPRI #1-pad



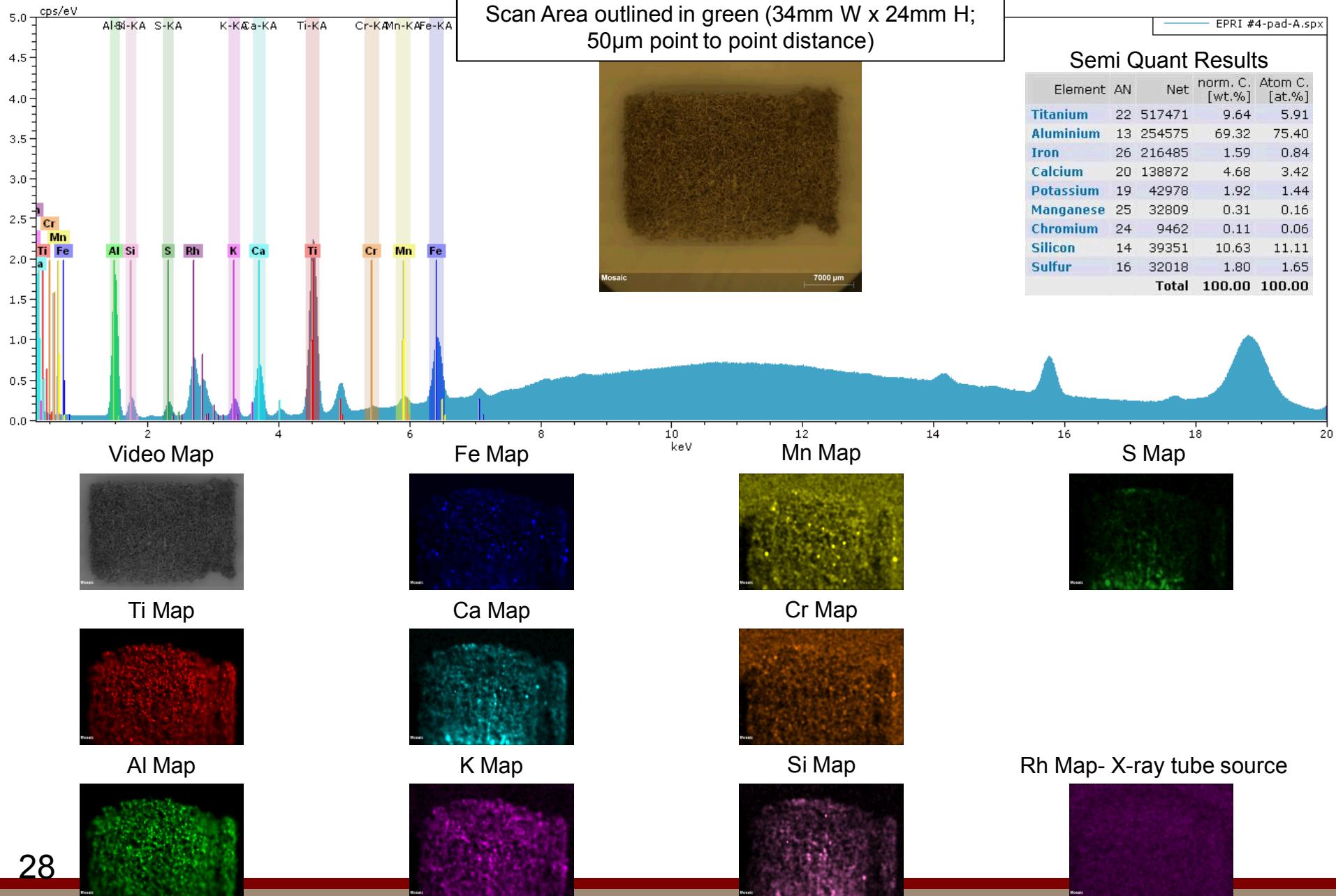
XRF Analysis: EPRI #4 filter



XRF analysis: EPRI #4-pad

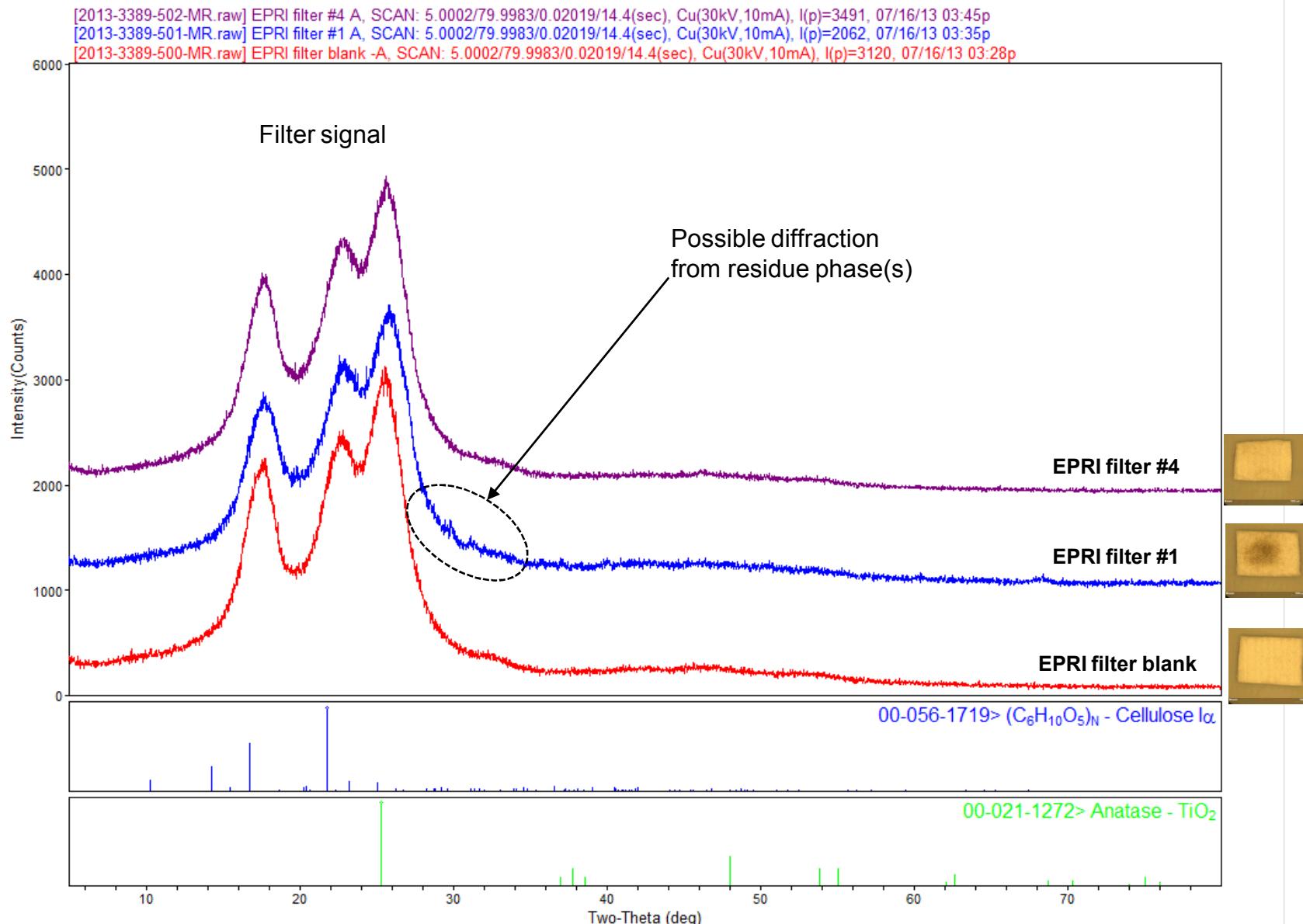
Scan Area outlined in green (34mm W x 24mm H;
50 μ m point to point distance)

EPRI #4-pad-A.spx



XRD Analysis: EPRI filter samples

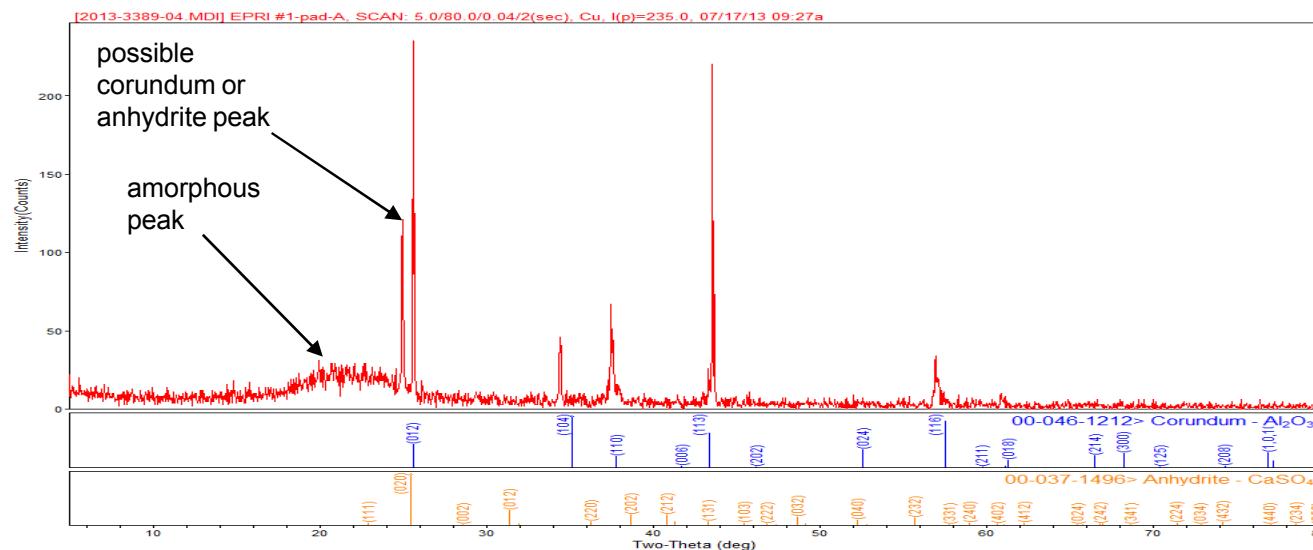
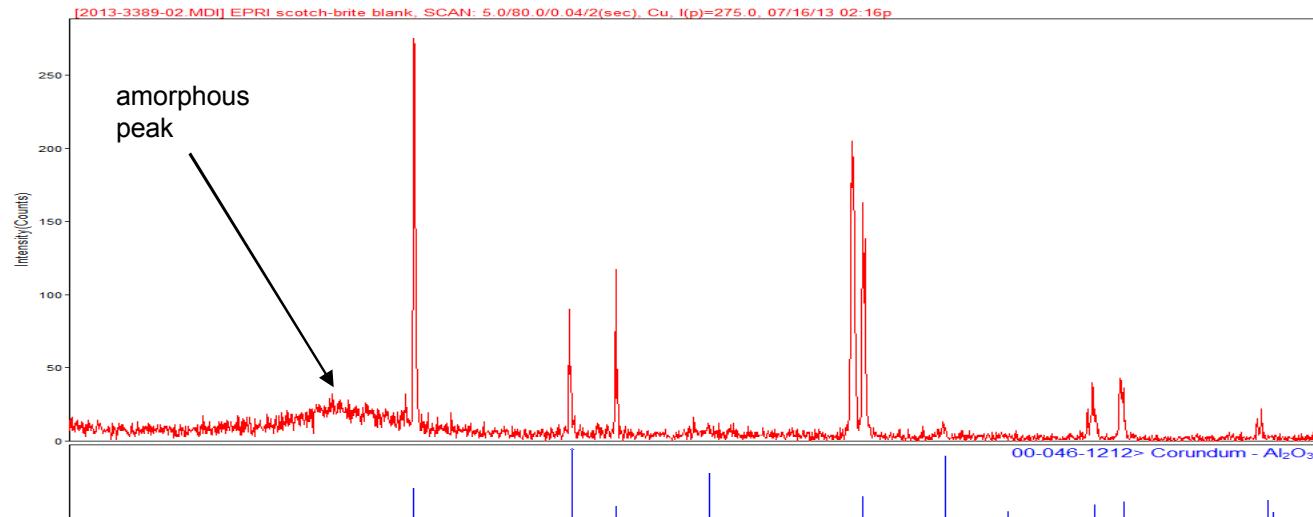
All the patterns are similar with little evidence of additional crystalline phases. There is a faint signal in the #1 pattern that could be indicative of an additional phase(s).



XRD Analysis: EPRI pads

The XRD pattern for the **EPRI Scotch-brite blank** shows a broad amorphous peak at $\sim 21^\circ$ 2 θ as well as sharp peaks for Corundum (Al_2O_3). The “splitting” and shifting of observed peaks is due to the dispersal of the phase at differing heights in the sample.

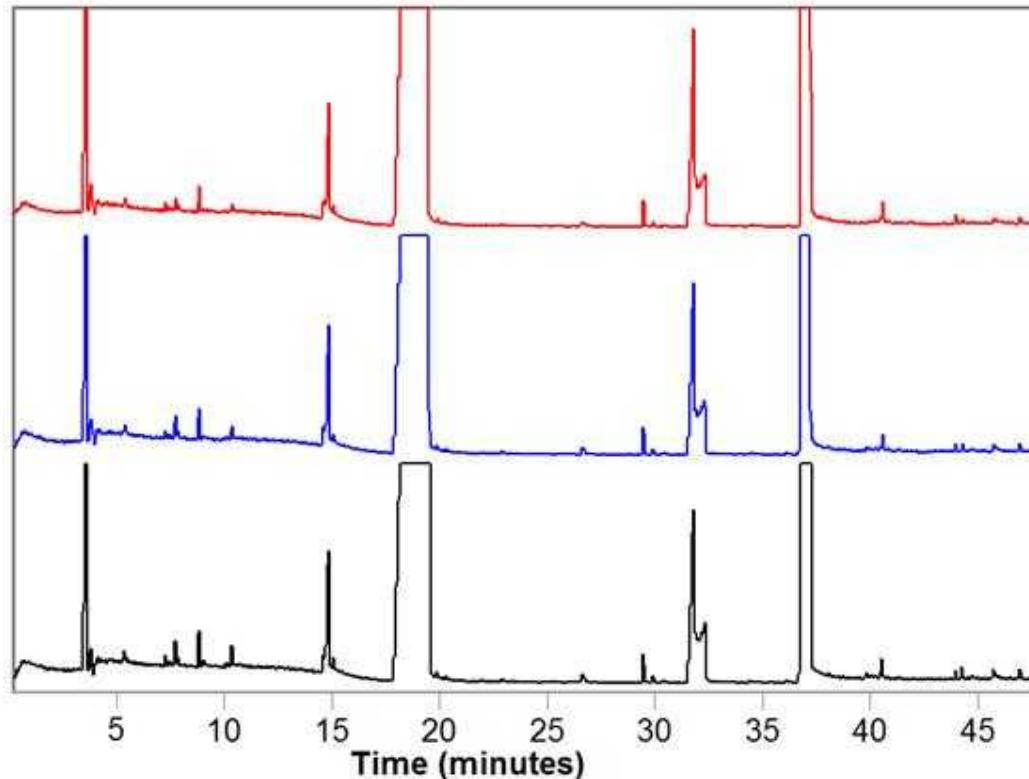
XRD results for the **EPRI #1-pad-A** again show a broad amorphous peak along with sharp diffraction peaks consistent with well crystallized corundum (Al_2O_3). Anhydrite (CaSO_4) is a possible match for some peaks, but the overlap between the peaks of Corundum and Anhydrite complicate the identification.



GC-MS Analysis

Filters and Scotchbrite pads

- Temperatures ramped to 100°C, 200°C and then 250°C. No difference was observed in decomposition patterns of the blanks versus the samples with time.



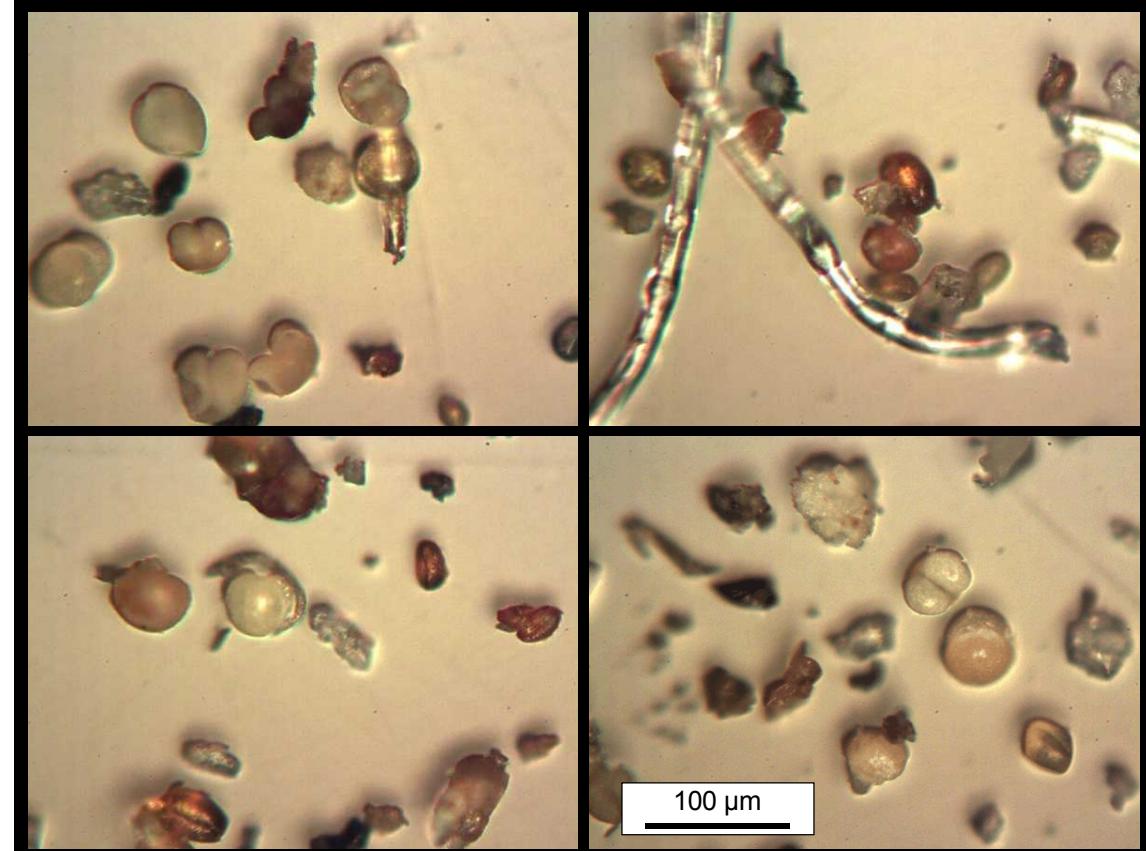
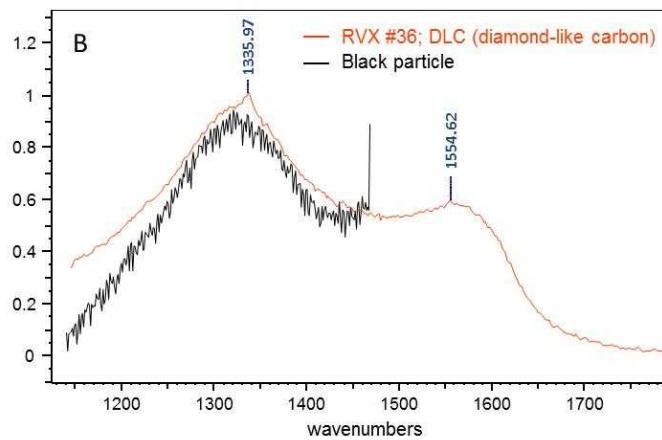
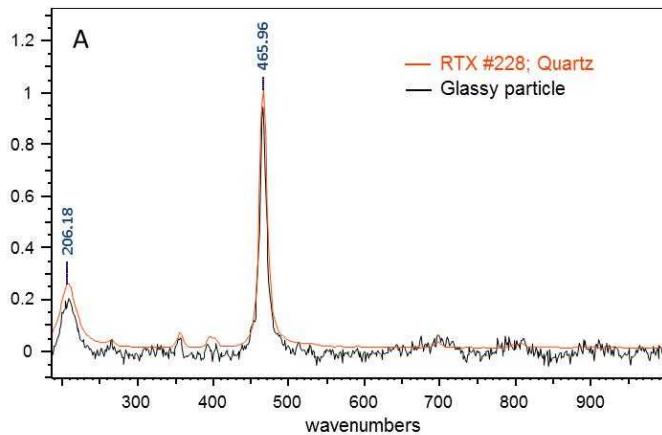
Total ion signal versus retention time for the Scotch-Brite® samples, blank, #1-D, #4-D (top to bottom). The y-axis is greatly expanded for viewing clarity of the minor peaks.

Micro-Raman Analysis

Photos taken during micro-Raman analysis show that dust coarse fraction is mostly pollen

Other phases identified:

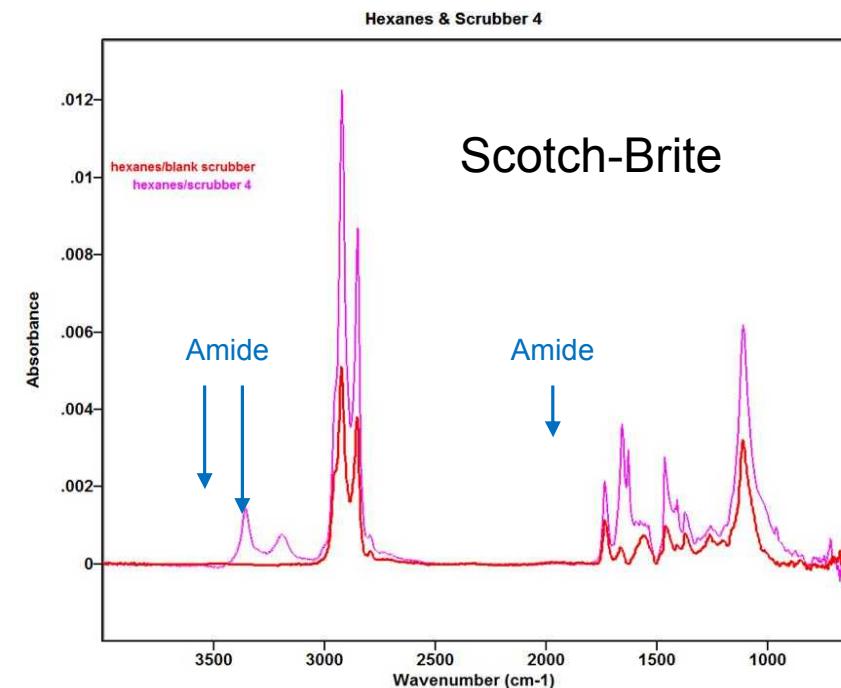
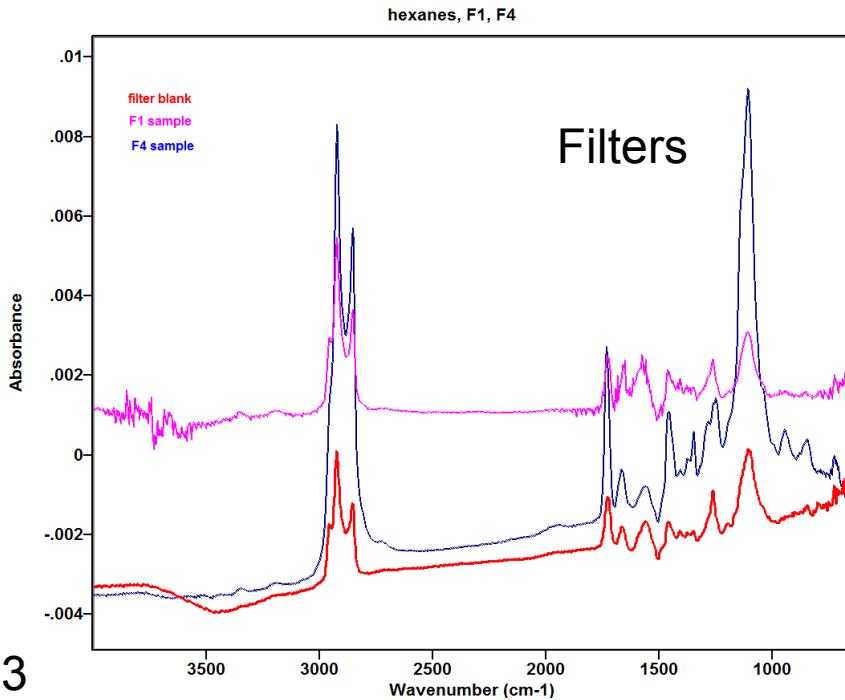
- Quartz
- Soot



FTIR Spectroscopy

Samples leached with isopropanol and hexane

- Isopropanol will leach polar organic molecules
- Hexane will leach non-polar organics (hydrocarbons)
- Filter samples did not vary significantly from blank
- Pad samples varied from blank: compound tentatively identified as a primary amide
 - Probable source: breakdown products of the scrubber, exposed by the abrasive action, allowing it to be extracted and not show up in the blank (e.g., the cured resin that is holding the abrasive to the nylon fiber; a slip agent on the nylon covered by the resin). Note that nylon itself is an amide.
 - Less likely: a protein or a degraded protein material in the dust



Chemical Analysis

Soluble salts

- Samples (pads and filters) placed in filter funnels (Whatman 541 ashless filter paper) and rinsed/leached with deionized water
- Cations analyzed by ICP-OES
- Anions analyzed by IC

Insoluble residue

- Filters—filter, residue, and Whatman filter paper ashed and digested with HNO_3 , HF
- Scotch-Brite® pads — dust rinsed from pad into Whatman filter, and filter/filtrate ashed and digested. Pads not included.
- Digested sample dried, redissolved in 2% HNO_3 , and analyzed by ICP-OES for major elements

Chemical Analysis: Soluble Salts

Water soluble salts:

- 30 minute leach with deionized water
- Cations: $\text{Ca}^{2+} >> \text{Na}^+, \text{Mg}^{2+}, \text{K}^+$
- Anions: $\text{SO}_4^{2-} >> \text{NO}_3^- > \text{Cl}^-$
- *Poor charge balances indicate analyses are deficient in anions (CO₃ and OH not measured)*

Salt phases observed by SEM:

- Calcium sulfate (gypsum) — dominant salt phase present, especially in EPRI #4 sample.
- Chlorides rare
 - *Halite (NaCl) — sparse*
 - *Sylvite (KCl) — single grain*
 - *Ca, Mg chlorides not observed*
- Ca, Mg, and Ca/Mg carbonates or hydroxides common.

Soluble salts, $\mu\text{g}/\text{sample}$

| Ion | Filter blank | Pad Blank | EPRI #1 filter | EPRI #1 pad | EPRI #4 filter | EPRI #4 pad |
|-----------------------------------|--------------|-------------|----------------|-------------|----------------|-------------|
| Na^+ | 4.80 | 3.38 | 23.6 | 18.4 | 3.66 | 14.7 |
| K^+ | 3.66 | 1.77 | 21.4 | 15.6 | 3.93 | 9.50 |
| Ca^{2+} | 0.92 | 0.46 | 78.0 | 21.1 | 24.9 | 154 |
| Mg^{2+} | 0.11 | 0.01 | 17.0 | 5.98 | 2.03 | 17.7 |
| F^- | 0.48 | 0.37 | 0.74 | 1.01 | 0.15 | 0.29 |
| Cl^- | 3.13 | 2.23 | 8.52 | 2.08 | 1.60 | 5.32 |
| NO_3^- | 1.37 | n.d. | 22.6 | 9.09 | 5.43 | 14.2 |
| SO_4^{2-} | 1.33 | 1.87 | 90.9 | 53.5 | 49.0 | 292 |
| PO_4^{3-} | 0.53 | n.d. | 7.17 | 2.05 | 0.87 | n.d. |
| Total mass, μg | 16 | 10 | 270 | 129 | 92 | 508 |
| Mass, minus blanks, μg | — | — | 255 | 118 | 80 | 498 |
| Charge Balance, % | | | 43.1 | 31.3 | 12.4 | 21.2 |

Chemical Analysis, Insolubles

Insoluble fraction, $\mu\text{g}/\text{sample}$

| Element | Filter blank | Pad Blank | EPRI #1 filter | EPRI #1 pad | EPRI #4 filter | EPRI #4 pad |
|--|--------------|------------|----------------|-------------|----------------|-------------|
| Na | 1.94 | 1.30 | 15.4 | 8.00 | 5.55 | 7.97 |
| K | 3.33 | 0.74 | 25.4 | 13.0 | 7.24 | 12.9 |
| Ca | 4.17 | 1.50 | 223 | 86.4 | 50.9 | 89.4 |
| Mg | 0.51 | 0.19 | 81.1 | 30.2 | 22.0 | 35.7 |
| Fe | 4.10 | 0.97 | 83.8 | 32.9 | 32.0 | 40.3 |
| Al | 0.91 | 0.67 | 104 | 42.4 | 22.6 | 48.0 |
| Total, μg | 15.0 | 5.4 | 533 | 213 | 140 | 234 |

Insoluble residue (EPRI#1) on filter paper



Insoluble fraction:

- Dominated by SiO_2 , CaO , MgO
- The large amount of SiO_2 implies *quartz* is a major phase (confirmed by SEM)
- Other phases observed (SEM):
 - *K-aluminosilicate, Na-aluminosilicate (feldspars)*
 - *Mg, Mg-Fe aluminosilicates (clays?)*
 - *Fe-oxide*
 - *Fe-metal (spherules – fly ash or welding residue?)*
- Many grains are composite or coated, making mineralogical identification difficult

Insoluble fraction, oxide wt%

| Element | EPRI #1 filter | EPRI #1 pad | EPRI #4 filter | EPRI #4 pad |
|-------------------------|----------------|-------------|----------------|-------------|
| Na_2O | 1.68 | 0.97 | 1.83 | 0.50 |
| K_2O | 2.48 | 1.61 | 1.88 | 0.81 |
| CaO | 28.2 | 13.0 | 22.5 | 6.8 |
| MgO | 12.3 | 5.43 | 12.1 | 3.27 |
| Fe_2O_3 | 10.5 | 4.97 | 13.9 | 3.12 |
| Al_2O_3 | 17.9 | 8.6 | 14.0 | 4.96 |
| SiO_2^* | 26.9 | 65.5 | 33.8 | 80.5 |
| Total | 100 | 100 | 100 | 100 |

* SiO_2 calculated from filter/pad XRF data

Data Interpretation

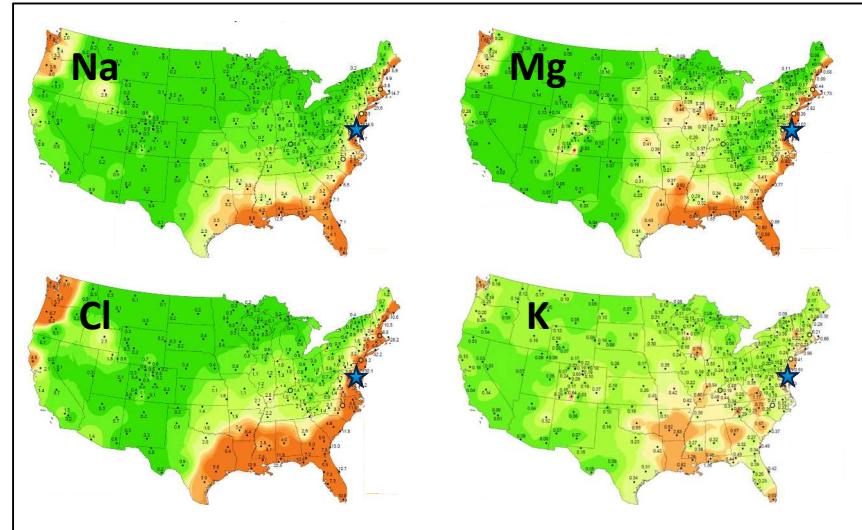
Composition of sea water

| | Conc., mg/L | |
|-------------------------------|---------------|-------------------------|
| Species | ASTM D1141-98 | McCaffrey et al. (1987) |
| Na ⁺ | 11031 | 11731 |
| K ⁺ | 398 | 436 |
| Mg ²⁺ | 1328 | 1323 |
| Ca ²⁺ | 419 | 405 |
| Cl ⁻ | 19835 | 21176 |
| Br ⁻ | 68 | 74 |
| F ⁻ | 1 | — |
| SO ₄ ²⁻ | 2766 | 2942 |
| BO ₃ ³⁻ | 26 | — |
| HCO ₃ ⁻ | 146 | — |
| pH | 8.2 | 8.2 |

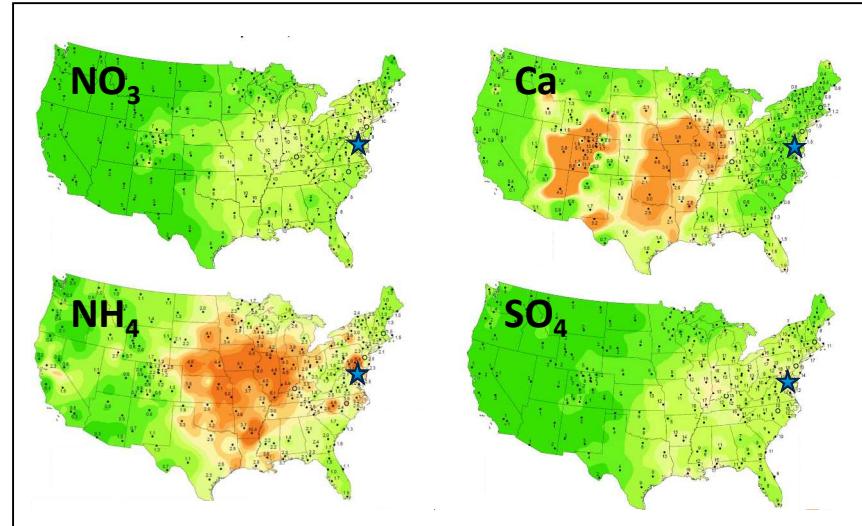
Inland salts—for example, data from an Iowa NADP site

| Species | Rain Conc., $\mu\text{eq/L}$ |
|-------------------------------|------------------------------|
| NH ₄ ⁺ | 29.22 |
| Na ⁺ | 2.05 |
| K ⁺ | 0.51 |
| Mg ²⁺ | 3.21 |
| Ca ²⁺ | 16.77 |
| Cl ⁻ | 1.98 |
| NO ₃ ⁻ | 15.57 |
| SO ₄ ²⁻ | 17.69 |

Salt components enriched in near-marine environments



Salt components enriched at inland sites



Data Interpretation

Salts do not appear to have a large marine component:

- Low Na^+ , Cl^- , high Ca^{2+} , SO_4^{2-}
- Sea salt deposition, followed by conversion via particle-gas conversion reactions? Does not explain low Na; cations (except ammonium) not affected by post-deposition reactions.
- Preferential deposition of adherent, deliquesced Ca-Cl salts, followed by conversion to sulfates and chloride-loss? Not likely—pollen-rich deposits on upper surface were clearly gravity deposited, and are also low in Na.

If salts are continental, why low ammonium (outside lab results), and low nitrate?

- Ammonium mineral decomposition—loss of ammonium, chloride, nitrate
- Particle-gas conversion reactions (e.g., SO_2 capture) preferentially incorporate sulfate, result in loss of nitrate, chloride)

Summary of Dust Sampling Results

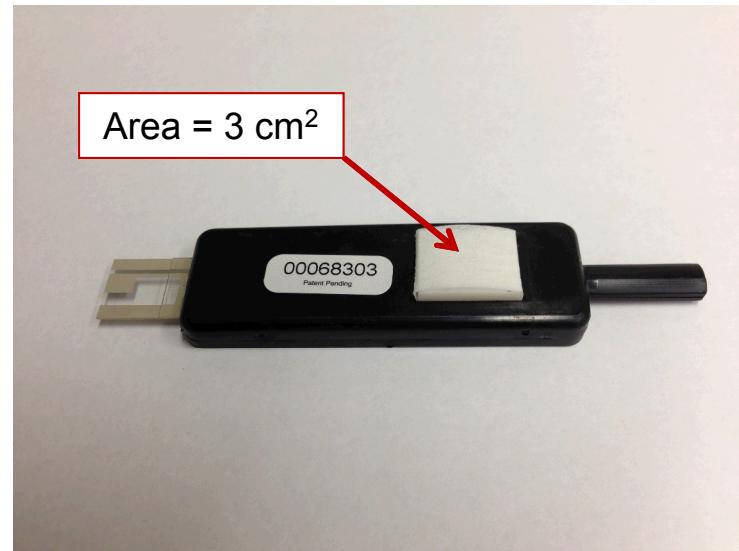
Dust samples were collected from the surface of an in-service interim storage container at Calvert Cliffs, MD, USA.

- The sample collected from the upper surface (11:00 location) consisted largely of pollen and other particles that settled gravitationally onto the package. The sample from the 7:00 position was finer-grained, and lacked the plant materials.
- The soluble salts in both samples are Ca and SO₄ rich. Gypsum is the dominant salt phase present.
- Chlorides comprise a small fraction of the total salt load, and are dominantly NaCl (a single grain of KCl was observed by SEM)

Despite the proximity to the coast and prevailing winds from the east, the dusts sampled from in-service containers at Calvert Cliffs do not appear to have a large sea salt component. Chesapeake Bay is brackish, and may be sheltered sufficiently to limit wave-generated sea-salt aerosols.

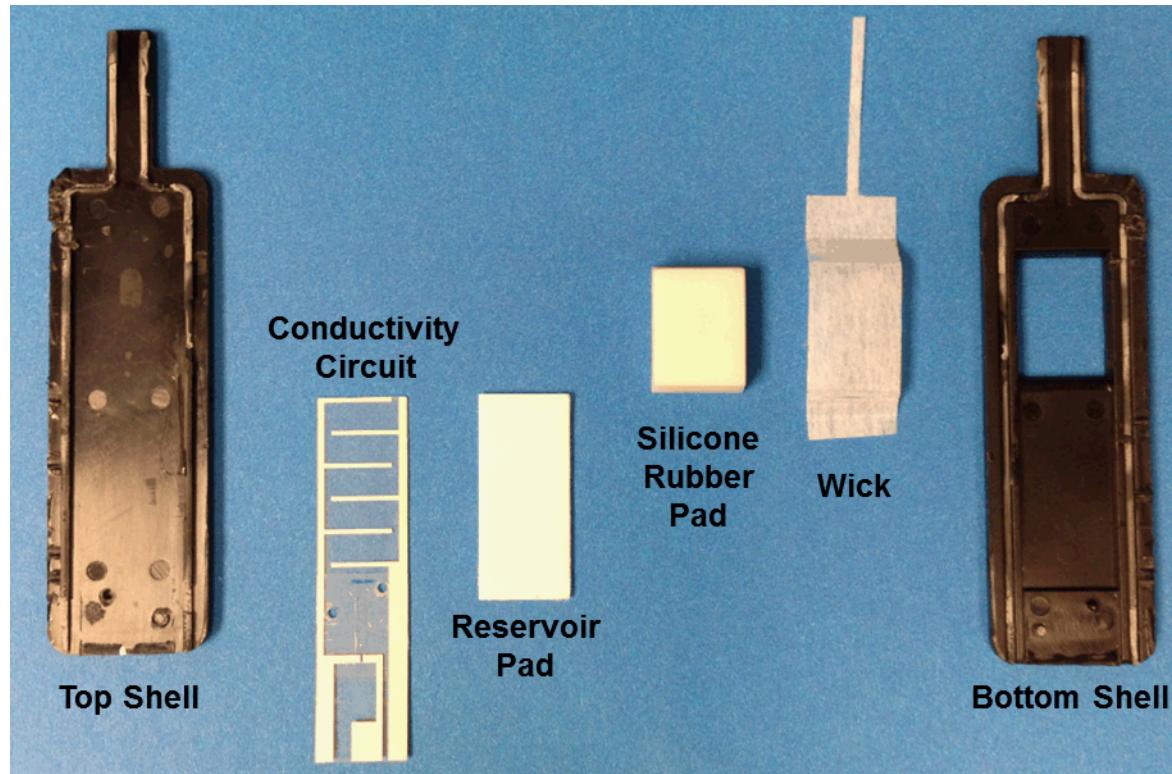
Salt-Smart Analyses

- Salt-smart® sensors developed to measure sea salt deposition on metal surfaces in shipyards, prior to painting the cleaned surface.
- Sample salts over a known surface area, providing salt amounts present per unit area.
- Fluid (DI water) is drawn by capillary action through a wick contacting the surface, and into an internal pad
- In ordinary usage, the conductivity of the wet pad is measured is measured, and chloride concentration is provided, assuming conductive salts are sea salt in composition.



Salt-Smart Analyses

- To accurately measure salt composition, we will disassemble the Salt-smart sensor, rinse the internal surfaces, and soak the wick and reservoir pad in DI water to extract salts. Extracted fluid is measured by IC (anions) and ICP (cations)



Testing Salt-Smart Efficiency

Saltsmart® sensors were applied to metal plates coated with a known amount of salts per unit area. After application, the sensors were disassembled and salts leached. The extracted salt concentrations were measured.

- Salt assemblage used: $\text{NaCl} + \text{Na}_2\text{SO}_4 + \text{MgCl}_2$
- Three salt loadings (185, 322, and 615 $\mu\text{g}/\text{cm}^2$), applied with an airbrush
- Three blanks were run to assess contaminants in the ampoule fluid or from the internal components
- Test to determine distribution of salt in wick vs. the reservoir pad

Salt-smart Efficiency

Results

- Blanks low
- Recovery good (80-90% of deposited weight) — some of the deposited salts were hydrous (e.g., Kieserite ($\text{MgSO}_4 \cdot \text{H}_2\text{O}$), Pentahydrite ($\text{MgSO}_4 \cdot 5\text{H}_2\text{O}$), Bloedite ($\text{Na}_2\text{Mg}(\text{SO}_4)_2 \cdot 4\text{H}_2\text{O}$), and Bischofite ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$), so missing mass was at least in part water
- Little remains in wick relative to reservoir pad
- Charge balances good (<5%), analytical methods appropriate

| Ion | Amount, μg | | | | | | | | | |
|---------------------------|-----------------------|--------|-------|-----------|-----------|-----------|-----------|---------|----------------|---------------|
| | BL1-10 | BL2-10 | BL3-8 | 185-8 min | 322-8 min | 322-6 min | 615-8 min | BL-Amp. | Wick-test wick | Wick-test pad |
| Na^+ | nd. | nd. | nd. | 93.1 | 288 | 283 | 529 | nd. | 3.27 | 142 |
| K^+ | nd. | nd. | nd. | nd. | nd. | nd. | nd. | nd. | nd. | nd. |
| Ca^{2+} | 1.21 | 0.521 | 0.445 | 1.24 | 1.62 | 1.69 | 1.75 | 0.074 | 0.246 | 1.67 |
| Mg^{2+} | nd. | nd. | nd. | 7.96 | 22.8 | 22.5 | 39.8 | nd. | 0.313 | 13.2 |
| F^- | n.a. | n.a. | n.a. | 0.075 | n.a. | n.a. | n.a. | 0.608 | n.a. | n.a. |
| Cl^- | 0.935 | 0.516 | 0.349 | 167 | 469 | 459 | 832 | 0.571 | 12.1 | 196 |
| NO_3^- | 2.79 | n.a. | n.a. | 0.597 | 0.851 | n.a. | 0.437 | n.a. | 0.431 | n.a. |
| SO_4^{2-} | 0.607 | n.a. | n.a. | 24.7 | 73.2 | 71.1 | 127 | n.a. | 1.84 | 30.2 |
| Sum, μg | 5.54 | 1.04 | 0.794 | 295 | 855 | 838 | 1529 | 1.25 | 18.2 | 383 |
| $\mu\text{g}/\text{cm}^2$ | 1.85 | 0.345 | 0.265 | 98.3 | 285 | 279 | 510 | 0.418 | 6.05 | 128 |
| % Dep. mass | — | — | — | 53.1 | 88.5 | 86.7 | 82.9 | — | — | — |
| Charge balance | — | — | — | -4.8 | -1.0 | -0.6 | 0.5 | — | -36 | 8.8 |

Salt-smart Efficiency

Results

No preferential leaching of chlorides versus sulfates: Molar ratios of cations and anions are nearly identical in the deposited salts and the analyses.

| Molar element ratios | Deposited salts | 185-8 min | 322-8 min | 322-6 min | 615-8 min | Wick test - wick | Wick test - pad |
|-------------------------|-----------------|-----------|-----------|-----------|-----------|------------------|-----------------|
| SO_4/Cl | 0.058 | 0.054 | 0.058 | 0.057 | 0.056 | 0.056 | 0.057 |
| Na/Cl | 0.970 | 0.859 | 0.946 | 0.951 | 0.98 | 0.418 | 1.12 |
| Na/Mg | 13.2 | 12.4 | 13.4 | 13.3 | 14 | 11.04 | 11.4 |
| Mg/Cl | 0.074 | 0.069 | 0.071 | 0.071 | 0.07 | 0.038 | 0.098 |
| Mg/SO_4 | 1.26 | 1.28 | 1.23 | 1.25 | 1.24 | 0.672 | 1.72 |

Salt-Smart Analyses: Conclusions

- For the conditions evaluated (6-8 minute contact time, horizontal surface):
 - Method works adequately for sampling salts on metal surfaces
 - Saltsmart effective for salt loadings up to at least 500 $\mu\text{g}/\text{cm}^2$
 - No preferential sampling of chloride vs. sulfate salts
- For a vertical surface:
 - Test samples from Holtec mock-up test analyzed. Results not yet available.