IWTU Process Sample Analysis Report

Nick Soelberg

April 2013

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April 2013

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SUMMARY

CH2M-WG Idaho (CWI) requested that Battelle Energy Alliance (BEA) analyze various samples collected during June – August 2012 at the Integrated Waste Treatment Facility (IWTU). Samples of IWTU process materials were collected from various locations in the process. None of these samples were radioactive. These samples were collected and analyzed to provide more understanding of the compositions of various materials in the process during the time of the process shutdown that occurred on June 16, 2012, while the IWTU was in the process of nonradioactive startup.

Proximate, Ultimate and SEM/EDS Analyses

Proximate and ultimate analyses were performed to measure the moisture, ash (inorganic), volatile matter, fixed carbon, C, H, N, and O in the samples.

Samples were analyzed for particle morphology, particle size, and elemental composition using scanning electron microscopy (SEM) and energy dispersive x-ray spectroscopy (EDS). The SEM provides views and photographs of these samples at varying magnifications from about 100x to 2,000x, so we can see individual particles and morphologies. The EDS provides elemental composition of the individual particles viewed in the SEM. However, the accuracy of the EDS for seeing lighter elements like C is less than for heavier elements like oxygen and higher; and it can't really see H at all.

These analyses show that the Process Gas Filter (PGF), Off-gas Filter (OGF), and High Efficiency Particulate Air (HEPA) filter samples have some similarities and differences that can be used to indicate different types of materials present in these locations of the IWTU.

- Calcined coal (Coal-01 sample): This calcined coal has moderately low moisture (higher than might be expected considering it has been calcined), expectedly high carbon content, and moderately low N and ash content. The results of the Coal-01 sample analysis are generally similar to the average analysis for the BESTAC coal used in the pilot tests.
- The charcoal (Charcoal-01 sample): This charcoal has moderately low moisture and compared to the calcined coal, lower carbon content, higher oxygen content, and somewhat higher ash content. The results of the Charcoal-01 sample analysis are generally similar to the average analysis for the P6 charcoal used in the pilot tests.
- PGF-01 sample from the Product Gas Filter (PGF) canister: The PGF-01 sample is nearly all inorganic alumina and bauxite material. The particles have the visible appearance typical of alumina particles with a few interspersed bauxite and coal/charcoal particles. The elemental composition is typical of alumina particles (Al and O), with fewer bauxite particles (Al, O, and lesser Si) and coal/charcoal (mostly C with lesser other elements like O and K) particles.
- The two additional PGF samples (PGF-02 and PGF-05), collected from the top of the PGF tubesheet and a PGF filter bundle, respectively, are quite different from the PGF-01 sample. These samples have higher moisture content, lower ash/inorganic content, and higher C, N, and O contents. These samples have particle compositions mostly consistent with coal or charcoal particles, with high C and lesser O, Al, Si, and K. These samples also appear to contain, as expected, some bauxite particles and are enriched in coal/charcoal ash.
- The OGF-01, OGF-05, and OGF-07 particles, collected from the Off-gas Filter (OGF) tubesheet, filter bundle No. 1, and the OGF bottom, respectively, are also mostly charcoal/coal, ash from oxidized charcoal/coal, and small amounts of bauxite. The OGF-05 and OGF-07 samples appear

similar to the PGF-05, OGF-01, and HEPA samples (from the inlet and outlet locations of the High Efficiency Particulate Air (HEPA) filters), although these two OGF samples have higher K than do the OGF-01 and HEPA samples, and some wider variations in some other elements such as C and Al. These wider ranges in C and Al may be simply due to the selection of some particles in these analyses that were either mostly pure bauxite or pure charcoal/coal.

- Most particles in the HEPA samples analyzed by SEM/EDS contain 60-80 wt% or more carbon, and may be charcoal particles. Particles from the HEPA samples that contained higher levels of Al and O, without Si, are alumina-origin, although these particles also tended to have measurable C. Particles with both elevated Al and Si, but without other elements, may be bauxite-origin. Particles that contained measurable levels of other elements (Fe, Mg, S/Mo, K, Ca, or Ti) may be enriched in coal or charcoal ash. The particles in the HEPA samples were more cohesive than were the particles in the PGF-01 sample.

TGA analyses

The thermogravimetric (TGA) analyses did not work out as planned. While the TGA instrument operation used to perform the proximate analyses was able to be modified to match the desired TGA conditions (10°C/min temperature increase, and flow of either N₂ or air to provide the desired reducing or oxidizing conditions), the actual operation did not achieve the desired oxidizing or reducing conditions.

Particle Size Analyses

Using SEM, we obtained particle size distribution (PSD) and particle composition data for three different SEM views for each sample. In each SEM view, we typically selected up to 4 particles on which we determined composition using EDS, so each sample has particle elemental analysis on at least 10 particles. We chose particles of different size and appearance in each view for composition analysis.

The particle size distributions for all but the PGF-01 (PGF canister) and the PGF-02 (top of PGF tubesheet) are all rather similar, ranging between less than 1 micron to less than 40 microns in size.

Individual particles in the PGF-01 sample ranged in size from under 10 microns to over 300 microns, and a few particles larger than 400 microns were observed. Most of these particles were alumina; relatively fewer particles were bauxite origin, and only 1-2 wt% were coal particles. Most of the coal particles were less than 100 micron in diameter. The larger particles were alumina or bauxite.

Particle and Bulk Density Analyses

The particle and bulk densities also show that the PGF-01 sample is quite different from the other samples. The particle density of the PGF-01 sample, at 3.79 g/cc, is very close to that of alumina at 3.8-3.9 g/cc. The particle density and bulk densities of the PGF-02 and PGF-05 samples are much lower, similar to the particle densities of the OGF and HEPA samples.

The particle densities of the PGF-02, PGF-05, the OGF samples, and the HEPA samples, which are similar and range between 1.86 g/cc and 2.00 g/cc, are close to a particle density from literature for black carbon (soot) of 1.8 gm/cc.

The bulk densities of the PGF-02, PGF-05, the OGF, and the HEPA samples were similar and ranged between 0.32 g/cc and 0.68 g/cc, depending on how consolidated the particles were. The bulk densities for the PGF, OGF, and the HEPA samples increased an average of about 40% when the samples were tapped to consolidate the particles.

Gasket Material Strength and Compression Tests

Tensile strength and compression tests were performed on samples of ceramic gasket material. The preliminary results and analysis procedure are summarized below.

The as-received gasket material contained practically non-measurable levels of moisture, and about 9.8 wt% binder [plus any other materials (such as water of hydration) that evaporates, oxidizes, or volatilizes at 650°C]. The as-received material retains physical integrity when carefully handled, but can be readily torn or damaged. It has low tensile and compression strength.

After heating for a day at 650°C, the material is less durable and more friable, presumably due to loss of binder. It has much lower tensile and compressive strength. Under sufficient pressure the heat-treated material disintegrates to dust, where-as the as-received material flattens under pressure but does not disintegrate.

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The author thanks the analysts who performed the analyses. Tammy Trowbridge performed the scanning electron microscopy (SEM) energy dispersive spectroscopy (EDS) and particle size analyses. Rachel Emersion performed the ultimate analyses, proximate analyses, thermogravimetric analysis (TGA), and some of the density measurements. Sharna Rossberg also performed some of the density measurements. Rachel Emerson, Randy Lloyd, and Mike Heighes performed the gasket analyses.

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ACRONYMS

ASTM	American Society for Testing and Materials
BEA	Battelle Energy Alliance
CWI	CH2M-WG Idaho
EDS	Energy dispersive x-ray spectroscopy
HMPD	harmonic mean particle diameters
HEPA	High Efficiency Particulate Air
IWTU	Integrated Waste Treatment Facility
MMPD	Mass mean particle diameter
OGF	Off-gas Filter
PGF	Product Gas Filter
PSD	Particle size distribution
SEM	Scanning electron microscopy
TGA	Thermal Gravimetric Analysis

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1. INTRODUCTION

CH2M-WG Idaho (CWI) requested that Battelle Energy Alliance (BEA) analyze various samples collected during June – August 2012 at the Integrated Waste Treatment Facility (IWTU). Samples of IWTU process materials were collected from various locations in the process. None of these samples were radioactive. These samples were collected and analyzed to provide more understanding of the compositions of various materials in the process during the time of the process shutdown that occurred on June 16, 2012, while the IWTU was in the process of nonradioactive startup.

The samples and the analyses that were performed are summarized in Table 1-1. This report documents the results of these sample analyses.

Table 1-1. IWTU sample analysis matrix.

Sample	Description	Analyses							
		Proximate/ TGA (ASTM) (Note 1)	Ulti- mate	TGA oxidizing (Notes 2, 4)	TGA reducing (Notes 3,4)	SEM/ EDS/ PSD	Particle density	Bulk density	Water solubility
Charcoal-01	As-received charcoal	Done	Done	-	-	-	-	-	-
Coal-01	As-received coal	Done	Done	-	-	-	-	-	-
Gasket-01	Sheet of ceramic fiber gasket material	Done: Mass and dimensional change, tensile strength, and compressibility before and after heating to 650°C							
PGF-01	From PGF canister	Done	Done	-	-	Done	Done	Done	Done
PGF-02	From top of PGF tubesheet (middle)	Done	Done	Done	Done	Done	Done	Done	-
PGF-05	From PGF filter element bundle	Done	Done	Done	Done	Done	Done	Done	-
OGF-01	Top of tubesheet	-	-	-	-	Done	-	-	-
OGF-05	#1 filter bundle surface	Done	Done	Done	Done	Done	-	-	-
OGF-07	OGF bottom	-	-	-	-	Done	Done	Done	-
HEPA-03	Bank B upstream of prefilter	Done	Done	Done	Done	Done	Done	Done	Done
HEPA-04	Bank B after prefilter	Done	Done	-	-	Done	Done	Done	Done
HEPA-02	Bank B after primary (Stage 1) filter	Done	Done	-	-	Done	Done	Done	Done
HEPA-01	Bank B after Stage 2 filter	Done	Done	Done	Done	Done	Done	Done	Done
<p>Notes:</p> <ol style="list-style-type: none"> 1. The American Society for Testing and Materials (ASTM) proximate/Thermal Gravimetric Analysis (TGA) analysis is done under an N₂ atmosphere to 950°C to measure moisture and volatile content; after cooling to 600°C, it converts to an O₂ atmosphere and heats to 750°C, to measure fixed carbon and ash. 2. The “TGA oxidizing” test was to be a proximate/TGA modified from the ASTM proximate analysis procedure to better match the oxidizing TGA provided for the charcoal by the charcoal vendor. In this modification, only an oxidizing mode was used; the heatup rate was 10°C/minute to a temperature of at least 640°C (it was actually heated to 950°C). 3. The “TGA reducing” test was to be a proximate/TGA modified to better match the reducing TGA provided for the charcoal by the charcoal vendor. In this modification, only a reducing mode was used; the heatup rate was 10°C/minute to a temperature of at least 640°C (it was actually heated to 950°C). 4. Neither of the modified TGA tests resulted in useable data. The mass loss in the oxidizing TGA did not keep up with the heat rate, so there was continued mass loss even after the samples were heated to 950°C and held at that temperature. The mass loss in the reducing TGA was similar (although smaller in magnitude) to the mass loss in the oxidizing test until the samples were heated to 950°C and held at that temperature until the weight stabilized. The final weights for the reducing TGAs matched the final weights for the oxidizing TGAs (and also matched the ash contents indicated by the proximate analyses) indicating that the TGA instrument was still allowing ambient air influx, which interfered with the reducing TGA. 5. SEM = Scanning electron microscopy; EDS = Energy dispersive x-ray spectroscopy; PSD = Particle size distribution. 									

2. PROXIMATE AND ULTIMATE ANALYSES

The proximate and ultimate analyses are shown in Table 2-1 and Figure 2-1. The results show that the Process Gas Filter (PGF), Off-gas Filter (OGF), and High Efficiency Particulate Air (HEPA) filter samples have some similarities and differences that can be used to indicate different types of materials present in these locations of the IWTU.

- Calcined coal (Coal-01 sample): This calcined coal has moderately low moisture (higher than might be expected considering it has been calcined, expectedly high carbon content, and moderately low N and ash content. The results of the Coal-01 sample analysis are generally similar to the average analysis for the BESTAC coal used in the pilot tests.
- The charcoal (Charcoal-01 sample): This charcoal has moderately low moisture and compared to the calcined coal, lower carbon content, higher oxygen content, and somewhat higher ash content. The results of the Charcoal-01 sample analysis are generally similar to the average analysis for the P6 charcoal used in the pilot tests.
- PGF-01 sample the Product Gas Filter (PGF) canister: the low moisture content, high “ash” (inorganic) value, and low H, C, and O values indicate that this sample is nearly all inorganic alumina and bauxite material. This indication is consistent with indications from the particle size, density, and elemental analyses.
- The two additional PGF samples (PGF-02 and PGF-05), collected from the top of the PGF tubesheet and a PGF filter bundle, are quite different from the PGF-01 sample. These samples have higher moisture content, lower ash/inorganic content, and higher C, N, and O contents. These results indicate that these samples are primarily coal/charcoal and ash from coal/charcoal that has been oxidized, with small amounts of alumina/bauxite. This indication is consistent with indications from the particle size, density, and elemental analyses.
- The OGF-01, OGF-05, and OGF-07 particles, collected from the Off-gas Filter (OGF) tubesheet, filter bundle No. 1, and the OGF bottom, respectively, are also mostly charcoal/coal, ash from oxidized charcoal/coal, and small amounts of bauxite. The OGF-05 and OGF-07 samples appear similar to the PGF-05, OGF-01, and HEPA samples (from the inlet and outlet locations of the High Efficiency Particulate Air (HEPA) filters), although these two OGF samples have higher K than do the OGF-01 and HEPA samples, and some wider variations in some other elements such as C and Al. These wider ranges in C and Al may be simply due to the selection of some particles in these analyses that were either mostly pure bauxite or pure charcoal/coal.
- The HEPA samples are 63-71 wt% C, 26-35% other [mostly inorganic particles of attrited bauxite (and possibly some alumina) and charcoal ash], and 2.4-2.7 wt% H and N. These samples are similar to the OGF and PGF samples, except for higher moisture, H and N levels (as shown in Table 2-1 and Figure 2-1), which suggests that material in the HEPA samples might not have been exposed to as high temperatures for as long and the material collected from the PGF and OGF areas.

Table 2-1. Proximate and ultimate analyses.

Sample Name	Description	Moisture, wt% (AR)	Ash, wt% (AR)	H, wt% (AR)	C, wt% (AR)	N, wt% (AR)	O+S, wt% (AR)	Total, wt% (AR)	Volatile, wt% (AR)	Fixed C, wt% (AR)
Coal-01	Calcined coal used in the DMR	3.02	4.32	2.81	81.3	0.72	7.87	100.00	12.99	79.67
BESTAC coal (EDF-10311) for comparison	Coal used in pilot plant testing	3.95	5.11	3.01	82.5	0.81	4.63	100.00	17.88	73.06
Charcoal-01	Charcoal used in the DMR and CRR during startup	2.75	5.96	2.01	73.5	0.74	15.07	100.00	19.41	71.88
Charcoal comparison (EDF-10311)	Charcoal used in pilot plant testing	3.12	7.38	2.86	75.5	0.46	10.71	100.00	24.97	64.52
PGF-01	From PGF canister	0.05	98.4	0.01	1.1	0.08	0.31	100.00	0.62	0.89
PGF-02	From top of PGF tubesheet (middle)	1.07	12.7	0.61	78.5	1.17	6.02	100.00	9.39	76.89
PGF-05	From PGF filter element bundle	1.09	20.4	0.53	68.4	1.34	8.27	100.00	11.73	66.82
OGF-05	From #1 filter bundle surface	1.56	20.2	0.38	66.1	1.11	10.70	100.00	13.78	64.51
HEPA-03	Bank B upstream of prefilter	2.12	16.8	1.29	70.9	1.51	7.42	100.00	14.92	68.29
HEPA-04	Bank B after prefilter	1.58	25.6	1.11	62.8	1.35	7.56	100.00	13.29	61.14
HEPA-02	Bank B after primary (Stage 1) filter	1.80	22.9	1.16	65.5	1.48	7.21	100.00	13.76	63.39
HEPA-01	Bank B after secondary (Stage 2) filter	1.40	23.5	1.10	65.4	1.56	7.08	100.00	13.05	65.36
Notes:										
1. AR = As-received rather than dried.										
2. Oxygen content in the organic matrix (not oxygen in the ash content) is determined by difference.										
3. The C analyses for the Coal-01, Charcoal-01, PGF-02, PGF-05, and OGF-05 samples were not valid; the ultimate analyzer was undergoing maintenance. Meanwhile, the C content of these samples is estimated based on the fixed carbon content which is provided by the proximate analysis.										
4. The volatile matter and fixed C values are not included in the moisture, ash, H, C, N, and O total.										
[ultimate proximate analyses ITWU 7nov12.xlsx]avg as received

Figure 2-1. Proximate and ultimate analyses for coal, charcoal, PGF, OGF, and HEPA samples.

3. TGA ANALYSES

The TGA analyses did not work out as planned. While the TGA instrument operation used to perform the proximate analyses was able to be modified to match the desired TGA conditions (10°C/min

temperature increase, and flow of either N₂ or air to provide the desired reducing or oxidizing conditions), the actual operation did not achieve the desired oxidizing or reducing conditions. The oxidizing condition was apparently not oxidizing enough because, as Figures 3-1 and 3-2 show, mass loss never stabilized as temperature increased, and did not stabilize until after a temperature of 950°C was reached and maintained for some time. (We could, in a future TGA test, use pure O₂ to see if that improves the oxidizing TGA).

Likewise, the reducing condition was apparently not sufficiently reducing, because after the final temperature of 950°C was reached, mass loss continued, and the final mass eventually matched the final mass of the oxidizing test (and matched the ash content of the respective samples) indicating that the samples were not isolated from a source of oxygen during the test.

Figure 3-1. TGA mass loss with increasing temperature, at a heat rate of 10°C/minute, and hold time at 950°C until constant weight was achieved. While this figure only shows the two PGF samples that were analyzed for clarity in the figure, similar TGA results were obtained for the OGF and HEPA samples that were analyzed.

Figure 3-2. The same PGF sample TGA results over the range of 940-960°C. The final weights match the ash contents determined by proximate analysis.

4. SEM/EDS AND PARTICLE SIZE ANALYSES

Samples were analyzed for particle morphology, particle size, and elemental composition using scanning electron microscopy (SEM) and energy dispersive x-ray spectroscopy (EDS). The SEM provides views and photographs of these samples at varying magnifications from about 100x to 2,000x, so we can see individual particles and morphologies. The EDS provides elemental composition of the individual particles viewed in the SEM. However, the accuracy of the EDS for seeing lighter elements like C is less than for heavier elements like oxygen and higher; and it can't really see H at all. So we have also included ultimate analyses to get better C, H, and N analyses, for characterizing the presence of coal or charcoal materials.

Using SEM, we obtained particle size distribution (PSD) and particle composition data for three different SEM views for each sample. In each SEM view, we typically selected up to 4 particles on which we determined composition using EDS, so each sample has particle elemental analysis on at least 10 particles. We chose particles of different size and appearance in each view for composition analysis.

4.1 SEM/EDS Results

Table 4-1 summarizes the SEM/EDS results. The detailed SEM/EDS analyses are shown in Appendix A. SEM photographs of the samples that correspond to the SEM/EDS and SEM/PSD analyses are shown in Appendix B. The SEM pictures and the elemental compositions from EDS support the indication from the other analyses that:

- The PGF-01 sample is nearly all inorganic alumina and bauxite material. The particles have the visible appearance typical of alumina particles with a few interspersed bauxite and coal/charcoal particles. The elemental composition is typical of alumina particles (Al and O), with fewer

bauxite particles (Al, O, and lesser Si) and coal/charcoal (mostly C with lesser other elements like O and K) particles.

- The two additional PGF samples (PGF-02 and PGF-05), collected from the top of the PGF tubesheet and a PGF filter bundle, are quite different from the PGF-01 sample. These samples have particle compositions mostly consistent with coal or charcoal particles, with high C and lesser O, Al, Si, and K. These samples also appear to contain, as expected, some bauxite particles and are enriched in coal/charcoal ash.
- The OGF-01, OGF-05, and OGF-07 particles, collected from the Off-gas Filter (OGF) tubesheet, filter bundle No. 5, and the OGF bottom are also mostly charcoal/coal, ash from oxidized charcoal/coal, and small amounts of bauxite. The OGF-05 and OGF-07 samples appear similar to the OGF-01 and HEPA samples (from the inlet and outlet locations of the High Efficiency Particulate Air (HEPA) filters), although these two OGF samples have higher K than do the OGF-01 and HEPA samples, and some wider variations in some other elements such as C and Al. These wider ranges in C and Al may be simply due to the selection of some particles in these analyses that were either mostly pure bauxite or pure charcoal/coal.
- Most particles in the HEPA samples contain 60-80 wt% or more carbon, and may be charcoal particles. Particles from the HEPA samples that contained higher levels of Al and O, without Si, are alumina-origin, although these particles also tended to have measurable C. Particles with both elevated Al and Si, but without other elements, may be bauxite-origin. Particles that contained measurable levels of other elements (Fe, Mg, S/Mo, K, Ca, or Ti) may be enriched in coal or charcoal ash.

Table 4-1. Elemental compositions of individual particles in the PGF and HEPA samples.

Sample	Description	Elemental composition						Comments
		C	O	Al	Si	K	Others (Fe, Mg, S/Mo, Ca, Ti)	
PGF-01	Fines captured in PGF, from a product canister	23-70	18-29	8-43	0.5-4	0-0.7	<10	Mix of mostly alumina particles, some bauxite particles, and 1-2 wt% coal particles
PGF-02	From top of PGF tubesheet (middle)	79-85	11-16	0.8-1.6	0.7-5	0-0.1	<2	Appears to be mostly coal particles
PGF-05	From PGF filter element bundle	41-96	3-31	0-27	0-49	0	<3	Appears to be mostly coal particles
OGF-01	Top of tubesheet	48-82	12-23	2-28	0.6-2	0-0.7	<2 (except 1 particle with 23 wt% Fe, perhaps an outlier)	Appears quite similar to the HEPA samples except for larger particle size
OGF-05	#1 filter bundle surface	12-95	4-31	0-56	0-50	0-2	<12	Appears to be mostly charcoal/coal particles
OGF-07	OGF bottom	4-97	2-36	0.1-24	0.1-58	0-6	<15	Appears to be mostly charcoal/coal particles
HEPA-03	Bank B upstream of prefilter	66-81	13-20	1-13	1-7	0	<2	Mostly charcoal particles with charcoal ash and attrited bauxite particles (possibly attrited alumina particles in small amounts)
HEPA-04	Bank B after prefilter	54-81	16-22	1-24	1-10	0	<2	
HEPA-02	Bank B after primary (Stage 1) filter	52-80	15-23	2-20	2-16	0-0.8	<5	
HEPA-01	Bank B after Stage 2 filter	62-80	15-29	1-14	1-6	0-1	<2	

Note: EDS does not readily discriminate between S and Mo.

4.2 Qualitative Cohesiveness Test

The particles in the HEPA samples, being mostly small carbon particles and not very dense, are quite cohesive. While not a quantitative test, the cohesiveness is indicated by tapping on the 4 oz-sized sample bottles and then rotating the sample bottles from vertical to determine the angle of repose in the bottles. In multiple tests, the angle of repose was found to be at least 70 degrees (almost vertical) to over 90 degrees (past vertical). This test is shown in video file “2012-06-22_PGF-01 and HEPA-01 samples.mp4.”

The particles in the PGF-01 sample, with a large fraction being relatively dense alumina and bauxite, are much less cohesive. The cohesiveness is indicated by estimating the angle of repose in the 4-oz sample bottle. This qualitative test, which is shown in the video file “2012-06-22_PGF-01 and HEPA-01 samples.mp4” indicates a ~20-30 degree angle of repose, so the particles in the PGF-01 sample are less cohesive than those in the HEPA samples.

4.3 Particle Size distributions

Figure 4-1 summarizes the cumulative particle size distributions for the PGF, OGF, and HEPA samples. Figure 4-2 shows the same results as in the prior figure, but with a shortened x-axis to better show particles in the 0-50 micron size range. The particle size distributions for all but the PGF-01 (PGF canister) and the PGF-02 (top of PGF tubesheet) are all rather similar, ranging between less than 1 micron to less than 40 microns in size. As shown by these and other PGF sample analyses (ultimate, proximate, SEM/EDS, and density measurements), the material captured in the PGF, on the top of the PGF tubesheet, and on one of the PGF filter bundles has varied in both composition and particle size.

Figure 4-1. Cumulative PSD for the PGF, OGF, and HEPA filter samples.

Figure 4-2. Cumulative PSD for the PGF, OGF, and HEPA filter samples (shortened x-axis to better show particles in the 0-50 micron size range).

The spread of PSDs for the PGF-05, OGF, and HEPA samples in Figure 4-2 is not entirely consistent with the expectation that the PSDs should tend to increasingly small particles progressing through the PGF, OGF, and HEPA samples. The PSD measured for HEPA-2, collected at the outlet of the primary (stage 1) filter, for example, is larger than for most of the other samples. This could in part be due to expected inaccuracy of the method of counting particles of different sizes, viewed by SEM, to estimate the PSDs. The inaccuracy could be the highest for the largest 5-10 particles shown in these figures, if those 5-10 particles in the SEM view are not entirely representative of the rest of the sample. So trying to differentiate between the PSDs of the grouped PGF-05, OGF, and HEPA samples as determined by this method may not be valid. While this method is valid for discriminating between the PSDs of the PGF-01 and PGF-02 samples and the other samples, it may not be valid for discriminating between the HEPA samples.

The cumulative PSDs for the individual samples are shown in Figures 4-3 through 4-12.

Individual particles in the PGF-01 sample ranged in size from under 10 microns to over 300 microns, and a few particles larger than 400 microns were observed. Most of these particles were alumina; relatively fewer particles were bauxite origin, and only 1-2 wt% were coal particles. Most of the coal particles were less than 100 micron in diameter. The larger particles were alumina or bauxite.

Figure 4-3. Cumulative PSD for the PGF-01 sample collected from a product canister.

Figure 4-4. Cumulative PSD for the PGF-02 sample collected from the PGF tubesheet (middle).

Figure 4-5. Cumulative PSD for the PGF-05 sample collected from the PGF filter element bundle.

Figure 4-6. Cumulative PSD for the OGF-01 sample collected from the top of the OGF tubesheet.

Figure 4-7. Cumulative PSD for the OGF-05 sample collected from the OGF #1 filter element bundle surface.

Figure 4-8. Cumulative PSD for the OGF-07 sample collected from the bottom.

Figure 4-9. Cumulative PSD for the HEPA-03 sample collected from Bank B upstream of the prefilter.

Figure 4-10. Cumulative PSD for the HEPA-04 sample collected from Bank B downstream of the prefilter.

Figure 4-11. Cumulative PSD for the HEPA-03 sample collected from Bank B after the primary (Stage 1) filter.

Figure 4-12. Cumulative PSD for the HEPA-01 sample collected from Bank B after the second (Stage 2) filter.

The PSDs for the HEPA samples ranged from under 1 micron to about 30 micron. Half of the numbers of particles ranged about 3 micron or smaller. While a trend in particle size where the HEPA Bank B prefilter inlet particles might be largest, and the most down-stream samples may have the smallest

PSD might be expected, this trend is not apparent in these data, perhaps because there is too much similarity in properties of the particulate captured in the areas of the HEPA filters.

If attrited alumina particles are present in the HEPA samples, they are not present in large amounts, and they are considerably attrited to particle sizes less than 10-30 microns, much smaller than the range of alumina particles found in the PGF-01 sample.

The individual OGF and HEPA samples are mostly coal/charcoal. The average PGF, OGF and HEPA harmonic and mass mean particle diameters (HMPDs and MMPDs) are shown in Table 4-2.

Table 4-2. HMPD and MMPD for the PGF, OGF, and HEPA samples.

Sample	HMPD, um	MMPD, um	50 wt% less than, um
PGF-01	152	222	207
PGF-02	245	205	185
PGF-05	17.9	11.8	11.2
OGF-01	38.1	32.7	27
OGF-05	40.8	20.5	20.5
OGF-07	23.0	15.4	15
avg OGF	34.0	22.9	20.8
HEPA-01	11.8	11.0	10.5
HEPA-02	38.9	23.3	24.3
HEPA-03	23.8	15.5	16.7
HEPA-04	11.2	9.0	8.5
avg HEPA	21.4	14.7	15.0
		[11sept2012 psd analyses.xlsx]Summary	

5. PARTICLE AND BULK DENSITY

The particle and bulk densities of the samples analyzed to date are summarized in Tables 5-1 and 5-2. The PGF samples are very different. The particle density of the PGF-01 sample, at 3.79 g/cc, is very close to that of alumina at 3.8-3.9 g/cc. The particle density and bulk densities of the PGF-02 and PGF-05 samples are much lower, similar to the particle densities of the OGF and HEPA samples.

The particle densities of the PGF-02, PGF-05, the OGF samples, and the HEPA samples, which are similar and range between 1.86 g/cc and 2.00 g/cc, are close to a particle density from literature for black carbon (soot) of 1.8 gm/cc.

The bulk densities of the PGF-05, the OGF, and the HEPA samples were similar and ranged between 0.32 g/cc and 0.68 g/cc, depending on how consolidated the particles were. The bulk densities for the PGF, OGF, and the HEPA samples increased an average of about 40% when the samples were tapped to consolidate the particles.

Table 5-1. Particle density for the PGF and HEPA samples.

Sample ID	Sample mass, g	Avg. density, g/cc	Std. dev., %
PGF-01	21.982	3.79	0.0044
PGF-02	6.014	1.96	0.0026
PGF-05	4.649	2.00	0.0180
OGF-01	5.385	1.92	0.0134
OGF-05	4.551	1.86	0.0159
OGF-07	4.562	1.89	0.0090
HEPA-03	3.907	1.89	0.0049
HEPA-04	4.029	1.97	0.0054
HEPA-02	4.483	1.93	0.0010
HEPA-01	4.100	1.94	0.0003
Notes:			
1. A Quantachrome Pentapyc was used to measure particle density. The instrument measures the density of each sample up to 7 times and takes the first 3 runs to have a % standard deviation of less than 0.3.			
[IWTU Particle Density 8nov12.xlsx]Sheet1			

Table 5-2. Bulk density of the PGF, OGF, and HEPA samples.

Sample Name	Loose Bulk Density, g/cc	Tapped Bulk Density, g/cc	% increase
PGF-01	2.06	2.26	10%
PGF-02	0.62	0.68	9%
PGF-05	0.43	0.60	40%
OGF-01	0.47	0.61	30%
OGF-05	0.40	0.54	34%
OGF-07	0.50	0.63	27%
HEPA-03	0.40	0.55	38%
HEPA-04	0.43	0.60	39%
HEPA-02	0.42	0.58	38%
HEPA-01	0.32	0.52	60%
Notes:			
1. ASTM D 7481-09 standard was used for PGF-01, PGF-05, OGF-07, and the HEPA samples. ASABE S269.4 Standard was used for PGF-02, OGF-01, and OGF-05 samples.			
2. 50 mL graduated cylinder substituted for 100 mL.			
3. For PGF-01, PGF-05, OGF-07, and the HEPA samples: A vortex mixer used in place of mechanical tapped density tester for 5:00 min per sample or until there was less than 2% difference after recording the volume each minute as specified in ASTM D 7481-09.			
4. For PGF-02, OGF-01, and OGF-05 samples: The tapped density was determined by tapping the graduated cylinder while it was being filled.			
[Bulk Density IWTU process samples 7nov12.xlsx]Sheet1			

6. CERAMIC GASKET MATERIAL ANALYSES

Tensile strength and compression tests were performed on samples of ceramic gasket material. The preliminary results and analysis procedure are summarized below.

6.1 Gasket Material Strength and Compression Test Procedure

A simplified analysis procedure was developed in collaboration with CWI representatives to obtain the needed results as soon as practical. The procedure was:

1. Cut out six 0.75 inch wide, 6 inch long strips. Trim the strips to the specifications in the modified Figure 1 of ASTM-E8/E8M. Record length and width dimensions of each.
2. Cut out six 2-inch-diameter disks.
3. Weigh the strips, dry them at 100-107°C to constant weight, and record the as-received and dry weights of the strips.
4. Take photos of the strips and circles both before and after the heat treating and the tensile strength and compression tests.
5. Heat three strips and three disks at 650°C in an air-atmosphere oven for 24 hours. Record final length and width, and weight. Take photos and note any changes in appearance.
6. Perform tensile strength test on the six strips. Provide tensile strength data. Elongation data or other data not necessary.
7. Perform compression test on the six disks to measure the thickness and compression under pressure.

6.2 Moisture Content and Weight Loss on Heating

The moisture content and weight loss on heating are shown in Table 6-1. The gasket material has almost non-measurable levels of moisture, as indicated by the low average as-received value of 0.08 wt%. The standard deviation on this triplicate moisture measurement was twice the average value.

Table 6-1. Gasket material moisture content and mass loss after heating to 650°C.

Sample Name	Moisture, wt%	Moisture standard deviation, wt%	% mass loss	Mass loss standard deviation, wt%
As received	0.08	0.17	---	---
After heating	0.07	0.01	9.79	0.0374
1. Moisture content was determined by heating to 100-107 C.				
2. The samples were heated in an air-atmosphere oven at 650 C for 24 hrs to determine mass loss.				
[moisture-ash_Gasket Ceramic Strips_20120729.xlsx]SUMMARY				

After heating for 24 hrs in an air-atmosphere furnace at 650°C, the samples had an average mass loss of about 9.8 wt%. This mass loss is attributed to volatilization/oxidation of organic binder material, although a portion of the mass loss may also be due to other factors such as any amount of water of

hydration in the inorganic ceramic material, that would tend to evaporate at temperatures of around 300-500°C.

There was no measureable change in dimensions after heating at 650°C, although the material became detectably softer and more friable, and less durable during handling, presumably due to loss of binder.

6.3 Tensile Strength and Compressibility

Tensile strength of the gasket material was tested both before and after heating using a tensile strength testing instrument. Results of the tensile strength tests are:

- As-received: 30-40 N, low compared to many other materials.
- After heating at 650°C: <<10 N; one of the triplicate samples fell apart during handling. Tensile strength significantly decreased due to the heat treatment.

The compressibility of the gasket material was tested both before and after heating using a compressive stress-strain instrument. Results of the compression tests are:

- The as-received disks compressed from a thickness of about 0.3 mm to about 0.03 mm under a pressure of 25 MPa (3,600 psig). Although considerably flattened under high pressure, the disks still retained their physical integrity.
- After heating at 650°C: No numerical data is available because under high compression, the material disintegrated into practically a powder. Under less compression, the disintegration was less.

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APPENDIX A: SEM/EDS ANALYSES

Note to reader: Each SEM/EDS analysis in Appendix A corresponds to one of the numbered particles in the photos in Appendix B, according to the sample and photo number in each of the micrographs in Appendix B, and the same sample and photo numbers in the figures below.

The wt % values in this appendix are the elemental weight percent values measured using SEM/EDS. The At % values are the elemental atom percentages, equivalent to elemental mole percentages.

Element	Wt %	At %
C K	23.44	35.54
O K	29.03	33.04
Fe L	01.31	00.43
Na K	00.24	00.19
Mg K	00.22	00.17
Al K	42.90	28.95
Si K	01.85	01.20
S K	00.21	00.12
K K	00.10	00.05

Element	Wt %	At %
C K	66.39	76.66
O K	18.83	16.32
Fe L	01.48	00.37
Mg K	00.00	00.00
Al K	09.98	05.13
Si K	01.73	00.85
S K	01.30	00.56
K K	00.05	00.02
Ca K	00.25	00.09

<i>Element</i>	<i>Wt %</i>	<i>At %</i>
<i>C K</i>	79.74	84.72
<i>O K</i>	17.70	14.12
<i>AlK</i>	01.18	00.56
<i>SiK</i>	00.90	00.41
<i>S K</i>	00.49	00.19
<i>EDAX ZAF QUANTIFICATION</i>		
<i>STANDARDLESS SEC TABLE : USER</i>		

<i>Element</i>	<i>Wt %</i>	<i>At %</i>
<i>C K</i>	64.73	75.20
<i>O K</i>	18.92	16.50
<i>MgK</i>	00.07	00.04
<i>AlK</i>	14.31	07.40
<i>SiK</i>	00.69	00.34
<i>S K</i>	00.85	00.37
<i>K K</i>	00.07	00.02
<i>CaK</i>	00.36	00.13

KV:10.00
TILT: 0.00
TAKE-OFF:35.76
AMPT:3.2
DETECTOR TYPE :SDD APOLLO X
RESOLUTION :127.51

<i>Element</i>	<i>Wt %</i>	<i>At %</i>
<i>C K</i>	57.14	68.64
<i>O K</i>	23.17	20.89
<i>MgK</i>	00.17	00.10
<i>AlK</i>	18.28	09.77
<i>SiK</i>	00.89	00.46
<i>S K</i>	00.07	00.03
<i>K K</i>	00.00	00.00
<i>CaK</i>	00.28	00.10

KV:10.00

TILT: 0.00

TAKE-OFF:35.76

AMPT:3.2 DETECTOR TYPE :SDD APOLLO X

RESOLUTION :127.51

<i>Element</i>	<i>Wt %</i>	<i>At %</i>
<i>C K</i>	53.73	66.31
<i>O K</i>	21.95	20.34
<i>Al K</i>	23.59	12.96
<i>Si K</i>	00.72	00.38
<i>EDAX ZAF QUANTIFICATION STANDARDLESS SEC TABLE : USER</i>

<i>Element</i>	<i>Wt %</i>	<i>At %</i>
<i>C K</i>	62.72	74.00
<i>O K</i>	18.80	16.65
<i>Al K</i>	01.95	01.03
<i>Si K</i>	16.30	08.22
<i>S K</i>	00.24	00.10
EDAX ZAF QUANTIFICATION STANDARDLESS SEC TABLE : USER		

KV:10.00
TILT: 0.00
TAKE-OFF:35.76
AMPT:3.2
DETECTOR TYPE :SDD APOLLO X
RESOLUTION :127.51

<i>Element</i>	<i>Wt %</i>	<i>At %</i>
<i>C K</i>	61.89	73.50
<i>O K</i>	19.47	17.36
<i>Fe L</i>	01.97	00.50
<i>Na K</i>	00.20	00.13
<i>Al K</i>	06.79	03.59
<i>Si K</i>	09.67	04.91
EDAX ZAF QUANTIFICATION STANDARDLESS SEC TABLE : USER		

KV:10.00
TILT: 0.00
TAKE-OFF:35.75
AMPT:3.2
DETECTOR TYPE :SDD APOLLO X
RESOLUTION :127.51

<i>Element</i>	<i>Wt %</i>	<i>At %</i>
<i>C K</i>	81.30	85.82
<i>O K</i>	16.77	13.29
<i>Al K</i>	01.00	00.47
<i>Si K</i>	00.92	00.42
EDAX ZAF QUANTIFICATION STANDARDLESS SEC TABLE : USER		

KV:10.00
TILT: 0.00
TAKE-OFF:35.75
AMPT:3.2
DETECTOR TYPE :SDD APOLLO X
RESOLUTION :127.51

<i>Element</i>	<i>Wt %</i>	<i>At %</i>
<i>C K</i>	66.87	76.98
<i>O K</i>	18.31	15.82
<i>Fe L</i>	01.27	00.31
<i>Al K</i>	10.97	05.62
<i>Si K</i>	02.34	01.15
<i>S K</i>	00.24	00.11
EDAX ZAF QUANTIFICATION STANDARDLESS SEC TABLE : USER		

KV:10.00
TILT: 0.00
TAKE-OFF:35.75
AMPT:3.2
DETECTOR TYPE :SDD APOLLO X
RESOLUTION :127.51

<i>Element</i>	<i>Wt %</i>	<i>At %</i>
<i>C K</i>	75.23	81.49
<i>O K</i>	19.98	16.25
<i>Al K</i>	02.50	01.20
<i>Si K</i>	02.29	01.06
<i>EDAX ZAF QUANTIFICATION STANDARDLESS SEC TABLE : USER</i>		

KV:10.00
TILT: 0.00
TAKE-OFF:35.75
AMPT:3.2
DETECTOR TYPE :SDD APOLLO X
RESOLUTION :127.51

<i>Element</i>	<i>Wt %</i>	<i>At %</i>
<i>C K</i>	67.04	76.85
<i>O K</i>	18.72	16.11
<i>Na K</i>	00.17	00.10
<i>Al K</i>	06.67	03.40
<i>Si K</i>	06.76	03.31
<i>Ca K</i>	00.65	00.22
<i>EDAX ZAF QUANTIFICATION STANDARDLESS SEC TABLE : USER</i>		

KV:10.00
TILT: 0.00
TAKE-OFF:35.75
AMPT:3.2
DETECTOR TYPE :SDD APOLLO X
RESOLUTION :127.51

<i>Element</i>	<i>Wt %</i>	<i>At %</i>
<i>C K</i>	76.66	82.20
<i>O K</i>	20.40	16.42
<i>Al K</i>	01.67	00.80
<i>Si K</i>	01.28	00.59
<i>EDAX ZAF QUANTIFICATION</i>		
<i>STANDARDLESS SEC TABLE : USER</i>		

KV:10.00

TILT: 0.00

TAKE-OFF:35.75

AMPT:3.2

DETECTOR TYPE :SDD APOLLO X

RESOLUTION :127.51

KV:10.00
TILT: 0.00
TAKE-OFF:35.73
AMPT:3.2
DETECTOR TYPE :SDD APOLLO X
RESOLUTION :127.51

Element	Wt %	At %
C K	61.58	72.75
O K	20.51	18.19
Al K	02.25	01.18
Si K	15.19	07.67
S K	00.48	00.21
EDAX ZAF QUANTIFICATION STANDARDLESS SEC TABLE : USER

KV:10.00
TILT: 0.00
TAKE-OFF:35.73
AMPT:3.2
DETECTOR TYPE :SDD APOLLO X
RESOLUTION :127.51

Element	Wt %	At %
C K	78.88	85.11
O K	14.76	11.96
Al K	02.86	01.37
Si K	02.64	01.22
S K	00.85	00.34
EDAX ZAF QUANTIFICATION STANDARDLESS SEC TABLE : USER

KV:10.00
TILT: 0.00
TAKE-OFF:35.73
AMPT:3.2
DETECTOR TYPE :SDD APOLLO X
RESOLUTION :127.51

Element	Wt %	At %
<i>C K</i>	53.31	67.61
<i>O K</i>	18.72	17.82
<i>FeL</i>	02.05	00.56
<i>AlK</i>	19.61	11.07
<i>SiK</i>	03.41	01.85
<i>S K</i>	00.41	00.19
<i>K K</i>	00.76	00.30
<i>CaK</i>	00.64	00.24
<i>TiK</i>	01.10	00.35
EDAX ZAF QUANTIFICATION STANDARDLESS SEC TABLE : USER

KV:10.00
TILT: 0.00
TAKE-OFF:35.73
AMPT:3.2
DETECTOR TYPE :SDD APOLLO X
RESOLUTION :127.51

Element	Wt %	At %
<i>C K</i>	51.57	65.27
<i>O K</i>	21.61	20.54
<i>FeL</i>	02.34	00.64
<i>NaK</i>	00.88	00.58
<i>AlK</i>	14.19	08.00
<i>SiK</i>	08.54	04.62
<i>S K</i>	00.24	00.11
<i>K K</i>	00.24	00.09
<i>CaK</i>	00.39	00.15
EDAX ZAF QUANTIFICATION STANDARDLESS SEC TABLE : USER

KV:10.00
TILT: 0.00
TAKE-OFF:35.82
AMPT:3.2
DETECTOR TYPE :SDD APOLLO X
RESOLUTION :127.51

<i>Element</i>	<i>Wt %</i>	<i>At %</i>
<i>C K</i>	80.47	86.01
<i>O K</i>	14.67	11.77
<i>Al K</i>	02.09	00.99
<i>Si K</i>	02.14	00.98
<i>S K</i>	00.63	00.25
EDAX ZAF QUANTIFICATION STANDARDLESS SEC TABLE : USER

KV:10.00 TILT: 0.00
TAKE-OFF:35.82
AMPT:3.2
DETECTOR TYPE :SDD APOLLO X
RESOLUTION :127.51

<i>Element</i>	<i>Wt %</i>	<i>At %</i>
<i>C K</i>	55.69	67.88
<i>O K</i>	22.34	20.44
<i>Al K</i>	11.15	06.05
<i>Si K</i>	10.54	05.49
<i>S K</i>	00.28	00.13
EDAX ZAF QUANTIFICATION STANDARDLESS SEC TABLE : USER

<i>Element</i>	<i>Wt %</i>	<i>At %</i>
<i>C K</i>	76.01	83.16
<i>O K</i>	15.81	12.99
<i>Al K</i>	04.01	01.95
<i>Si K</i>	03.30	01.54
<i>S K</i>	00.88	00.36
<i>EDAX ZAF QUANTIFICATION STANDARDLESS SEC TABLE : USER</i>		

KV:10.00 TILT: 0.00
TAKE-OFF:35.82
AMPT:3.2
DETECTOR TYPE :SDD APOLLO X
RESOLUTION :127.51

<i>Element</i>	<i>Wt %</i>	<i>At %</i>
<i>C K</i>	56.89	68.63
<i>O K</i>	22.89	20.73
<i>Al K</i>	09.68	05.20
<i>Si K</i>	10.54	05.44
<i>EDAX ZAF QUANTIFICATION STANDARDLESS SEC TABLE : USER</i>		

KV:10.00
TILT: 0.00
TAKE-OFF:35.79
AMPT:3.2
DETECTOR TYPE :SDD APOLLO X
RESOLUTION :127.51

<i>Element</i>	<i>Wt %</i>	<i>At %</i>
<i>C K</i>	78.93	85.02
<i>O K</i>	15.63	12.64
<i>MgK</i>	00.24	00.13
<i>AlK</i>	01.77	00.85
<i>SiK</i>	01.68	00.77
<i>S K</i>	00.33	00.13
<i>K K</i>	00.31	00.10
<i>CaK</i>	01.11	00.36

<i>Element</i>	<i>Wt %</i>	<i>At %</i>
<i>C K</i>	76.97	83.45
<i>O K</i>	17.18	13.99
<i>MgK</i>	00.13	00.07
<i>AlK</i>	02.18	01.05
<i>SiK</i>	01.66	00.77
<i>S K</i>	00.65	00.27
<i>K K</i>	01.06	00.35
<i>CaK</i>	00.17	00.06

<i>Element</i>	<i>Wt %</i>	<i>At %</i>
<i>C K</i>	79.21	84.91
<i>O K</i>	16.24	13.07
<i>MgK</i>	00.04	00.02
<i>AlK</i>	01.77	00.85
<i>SiK</i>	01.55	00.71
<i>S K</i>	00.77	00.31
<i>K K</i>	00.13	00.04
<i>CaK</i>	00.28	00.09

<i>Element</i>	<i>Wt %</i>	<i>At %</i>
<i>C K</i>	79.94	85.53
<i>O K</i>	15.26	12.26
<i>MgK</i>	00.08	00.04
<i>AlK</i>	02.36	01.12
<i>SiK</i>	01.82	00.83
<i>S K</i>	00.49	00.20
<i>K K</i>	00.00	00.00
<i>CaK</i>	00.05	00.02

<i>Element</i>	<i>Wt %</i>	<i>At %</i>
<i>C K</i>	71.00	79.76
<i>O K</i>	17.39	14.66
<i>F K</i>	00.14	00.10
<i>MgK</i>	00.09	00.05
<i>AlK</i>	05.22	02.61
<i>SiK</i>	04.91	02.36
<i>S K</i>	00.43	00.18
<i>K K</i>	00.18	00.06
<i>CaK</i>	00.64	00.21

<i>Element</i>	<i>Wt %</i>	<i>At %</i>
<i>C K</i>	79.50	84.55
<i>O K</i>	17.94	14.33
<i>MgK</i>	00.09	00.05
<i>AlK</i>	00.79	00.37
<i>SiK</i>	00.87	00.40
<i>S K</i>	00.59	00.23
<i>K K</i>	00.00	00.00
<i>CaK</i>	00.21	00.07

<i>Element</i>	<i>Wt %</i>	<i>At %</i>
<i>C K</i>	67.49	76.78
<i>O K</i>	20.22	17.27
<i>MgK</i>	00.12	00.07
<i>AlK</i>	05.42	02.74
<i>SiK</i>	05.54	02.70
<i>S K</i>	00.28	00.12
<i>K K</i>	00.23	00.08
<i>CaK</i>	00.70	00.24

<i>Element</i>	<i>Wt %</i>	<i>At %</i>
<i>C K</i>	79.38	84.97
<i>O K</i>	16.20	13.01
<i>MgK</i>	00.04	00.02
<i>AlK</i>	02.00	00.95
<i>SiK</i>	01.74	00.79
<i>S K</i>	00.51	00.20
<i>K K</i>	00.00	00.00
<i>CaK</i>	00.14	00.04

<i>Element</i>	<i>Wt %</i>	<i>At %</i>
<i>C K</i>	61.65	72.80
<i>O K</i>	19.95	17.68
<i>MgK</i>	00.10	00.06
<i>AlK</i>	14.17	07.45
<i>SiK</i>	03.50	01.77
<i>S K</i>	00.23	00.10
<i>K K</i>	00.15	00.05
<i>CaK</i>	00.25	00.09

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APPENDIX B: SEM MICROGRAPHS THAT CORRESPOND TO THE SEM/EDS ANALYSES

Note to reader: Each SEM/EDS analysis in Appendix A corresponds to one of the numbered particles in the photos below, according to the sample and photo number in each of the micrographs below, and the same sample and photo numbers in the figures in Appendix A.

PGF-01-1

PGF-01-2

PGF-01-3

PGF-02-1

PGF-02-2

PGF-02-3

PGF-05-1

PGF-05-2

PGF-05-3

OGF-01-3

OGF-01-4

OGF-01-5

OGF-05-1

OGF-05-2

OGF-05-3

OGF-07-1

OGF-07-2

OGF-07-3

HEPA-03-1

HEPA-03-2

HEPA-03-3

HEPA-04-1

HEPA-04-2

HEPA-04-3

HEPA-02-1

HEPA-02-2

HEPA-02-4

HEPA-01-1

HEPA-01-3

HEPA-01-4