PNNL-SA-62011, Rev. 1

U.S. DEPARTMENT OF ENERGY

Prepared for the U.S. Department of Energy
under Contract DE-AC05-76RL01830

Results of Self-Absorption Study on the Versapor® 3000 Filters for Radioactive Particulate Air Sampling

JM Barnett, VI Cullinan, DS Barnett, TLT Trang-Le, M Bliss, LR Greenwood
and MY Ballinger

February 2009

Pacific Northwest
NATIONAL LABORATORY

PACIFIC NORTHWEST NATIONAL LABORATORY
operated by
BATTELLE
for the
UNITED STATES DEPARTMENT OF ENERGY
under Contract DE-ACO5-76RL01830

Printed in the United States of America

Available to DOE and DOE contractors from the
Office of Scientific and Technical Information,
P.O. Box 62, Oak Ridge, TN 37831-0062;
ph: (865) 576-8401
fax: (865) 576 5728
email: reports@adonis.osti.gov

Available to the public from the National Technical Information Service,
U.S. Department of Commerce, 5285 Port Royal Rd., Springfield, VA 22161
ph: (800) 553-6847
fax: (703) 605-6900
email: orders@nits.fedworld.gov
online ordering: http://www.ntis.gov/ordering.htm

PNNL-SA-62011, Rev. 1

Results of Self-Absorption Study on the Versapor® 3000 Filters for Radioactive Particulate Air Sampling

JM Barnett, VI Cullinan, DS Barnett, TLT Trang-Le, M Bliss, LR Greenwood and MY Ballinger

February 2009

Prepared for
the U.S. Department of Energy
under Contract DE-AC05-76RL01830

Pacific Northwest National Laboratory
Richland, Washington 99352

Summary

Since the mid-1980s, Pacific Northwest National Laboratory (PNNL) has used a value of 0.85 as the correction factor for self absorption of activity for particulate radioactive air samples collected from building exhaust for environmental monitoring. This value accounts for activity that cannot be detected by direct counting of alpha and beta particles. Emissions can be degraded or blocked by filter fibers for particles buried in the filter material or by inactive dust particles collected with the radioactive particles. These filters are used for monitoring air emissions from PNNL stacks for radioactive particles. This paper describes an effort to re-evaluate self-absorption effects in particulate radioactive air sample filters (Versapor® 3000, 47 mm diameter) used at PNNL. There were two methods used to characterize the samples. Sixty samples were selected from the archive for acid digestion to compare the radioactivity measured by direct gas-flow proportional counting of filters to the results obtained after acid digestion of the filter and counting again by gas-flow proportional detection. Thirty different sample filters were selected for visible light microscopy to evaluate filter loading and particulate characteristics. Mass-loading effects were also considered.

Filter ratios were calculated by dividing the initial counts by the post-digestion counts with the expectation that post-digestion counts would be higher because digestion would expose radioactivity embedded in the filter in addition to that on top of the filter. Contrary to expectations, the post digestion readings were almost always lower than initial readings and averaged approximately half the initial readings for both alpha and beta activity. Before and after digestion readings appeared to be related to each other, but with a low coefficient of determination (R^2) value. The ratios had a wide range of values indicating that this method did not provide sufficient precision to quantify self-absorption effects.

The microscopy analysis compares different filter loadings and shows that smaller particle sizes (under 10 micron) can readily be seen on the more lightly loaded filters. At higher loadings, however, the particle size is harder to differentiate. This study provides data on actual stack emission samples showing a range of mass loading conditions and visual evidence of particle size and distribution and also presents the difficulties in quantifying self-absorption effects using actual samples.

Contents

Summary 5
1.0 Introduction 7
2.0 Methods 8
2.1 Sample Filter Count and Acid Digest Count Comparison 8
2.2 Mass Loading Evaluation 9
2.3 Reflected Light Microscopy 10
3.0 Results 10
3.1 Sample Filter Count and Acid-Digest Count Comparison 10
3.2 Mass Loading Evaluation 15
3.3 Reflected Light Microscopy 15
4.0 Conclusions 16
5.0 Acknowledgements 17
6.0 References 17
Appendix A—Raw Data From Filter Analyses 18
Appendix B—Filter and Sample Weights 22
Appendix C—Microscopy Images 26

Figures

1. Plot of the Filter Data Counted Before and After Digestion – Alpha Activity 13
2. Plot of the Filter Data Counted Before and After Digestion – Beta Activity 14
3. Plot of the Alpha to Beta Initial Filter Data Results 14

Tables

1. Results from Counting Stack Filters Before and After Digestion 12

1.0 Introduction

Since the mid-1980s, Pacific Northwest National Laboratory (PNNL) has used a value of 0.85 as the correction factor for self absorption of alpha particles emitted from the particulate material collected from building exhaust for environmental monitoring on air samples (Higby 1984); this correction factor has also been equally applied at PNNL to samples analyzed for beta particles. This value accounts for activity that cannot be detected by direct counting of alpha and beta particles. Emissions can be degraded or blocked by filter fibers for particles buried in the filter material or by inactive dust particles collected with the radioactive particles. ANSI/HPS N13.1-1999, *Sampling and Monitoring Releases of Airborne Radioactive Substances From the Stacks and Ducts of Nuclear Facilities*, recommends that for filter media, if the penetration of radioactive material into the collection media or self-absorption of radiation by the material collected would reduce the count rate by more than 5%, a correction factor should be used (ANSI 1999).

Over the past 6 years, the Effluent Management group coordinated an effort to evaluate the current particulate radioactive air sample filters (the Versapor® 3000, 47 mm diameter)[a] used at PNNL for self-absorption effects. The two methods employed were 1) a comparison between the measured gross alpha and gross beta activity deposited on sample filters as measured with a gas flow proportional detector against the measured activity after the sample filters were acid digested and the sample material dried onto a planchet and analyzed again with a similar counting geometry by a gas flow proportional detector and 2) reflected light microscopy. Mass loading was also considered as a part of the filter analyses conducted.

Versapor® membrane filters are composed of an acrylic copolymer on a nylon substrate. They are manufactured in pore sizes ranging from 0.2 to 3 μm and the filter diameters over the range of 25 to 293 mm. For uses in air filtration, the typical flow rates range from 1.8 to 52 L min^{-1} cm^{-2}. The typical thickness is 190 μm.

For this study, the Versapor® 3000 filters (3 μm pore size) with a 47-mm diameter were used. When installed on fixed-head radioactive-air-stack sampler, they have a ~41-mm active diameter (13.2 cm^2 active sampling area). Samples are collected over a 2 week period. For radioactive air emissions sampling, the exhaust air passes through a minimum of one stage of high-efficiency particulate air (HEPA) filter(s); however in some cases unfiltered building air and the filtered exhaust may be combined creating a mechanism for additional unfiltered non-radioactive particles to also be sampled. Sample flow rates generally range from 28 to 85 L min^{-1}, which is on the low end of the maximum rated flow rate of 900 L min^{-1} (52 L min^{-1} cm^{-2}). Face velocities range from 0.35 to 1.1 m s^{-1}. Barnett and Kane (1993) have previously shown that the Versapor® 3000 filters may be operated in this range in unfiltered systems without sampling volume degradation due to particulate loading. At the end of the sampling period, the sample filter is removed and sent in for gross alpha and gross beta analysis.

(a) Pall Gelman Versapor® Membranes, Krackeler Scientific, Inc., 57 Broadway, Albany, NY 12202.

The self-absorption correction factor of 0.85 was based on data obtained from the direct alpha counting and photon spectrometry of glass-fiber filter samples where the filters were exposed to 0.66 μm, 1.72 μm, and 3.07 μm monodispersed aerodynamic diameter particles over face velocities between 0.5 and 2.0 m s^{-1} incremented in 0.5 m s^{-1} steps. Three tests were conducted at each of the four face velocities used for each particle size available resulting in a total of 36 tests conducted. Monodispersed particles of $^{239}PuO_2$ were obtained from Lovelace Inhalation Toxicology Research Institute and subsequently suspended in a solution of ammonium hydroxide with a pH of 10.0. Test aerosols were generated from these solutions using a compressed air aerosol nebulizer. The study involved conditions which were designed to maximize the effects of particle size and velocity on burial depth in glass-fiber filters. The smallest particle size (0.66 μm) was chosen for use at PNNL because it was closest to the theoretical typical penetrating size for fiber filters (0.1 to 0.3 μm) and could potentially account for the burial losses at the velocities tested. There were four sets of data at the lowest particle size yielding an overall self-absorption factor of 85% ± 9% (1σ) as determined by the reported mean fraction detected results. Higby concluded that "a correction which assumes 10 to 15% losses would ensure that concentrations of airborne alpha emitting radionuclides would not be underestimated by collection and analysis on glass-fiber filters." Considering all of the reported mean fraction detected results for the 0.66 to 3.07 μm range, a self-absorption factor of 88% ± 13% (1σ) was obtained. (Higby 1984)

In addition to Higby (1984), other studies by Haung et al. (2002), Luetzelschwab et al. (2000), and Stevens and Toureau (1963) have discussed major factors affecting measurements of radioactivity on air samples collected on filters. These factors include collection face velocity, particle size, type of filter, and filter loading. Higby showed that smaller particles have higher losses due to burial in the filter matrix and that losses are less as velocity and particle size increase due to inertial impaction on the top layer of the filter; he determined the minimum burial depth for an alpha particle to be lost due to absorption is ~3.7 mg cm^{-2}. This is the calculated range for ^{239}Pu particles in glass so alpha particles would be difficult to count at the bottom of loading greater than this value. Stevens and Toureau (1963) investigated filter types, dust loading, and particle size on the energy spectrum and noted that glass fiber filters have a near surface collection with detection efficiencies of 85 to 100% of 0.2 to 1.0 μm radioactive particles under low face velocities of 0.35 m s^{-1}. Dust loading also affects measurements with higher losses in alpha radiation readings at higher dust loadings. Luetzelschwab et al. (2000) recommended assuming 28% losses for a loading of 2.3 mg cm^{-2} and 40% when the loading is increased to 3.3 mg cm^{-2}. Haung showed that loading is not important if the radioactive layer is a thin layer on top of the nonradioactive dust (e.g. detecting a sudden pulse release of radioactivity) rather than distributed throughout. These factors are considered in this study for radioactive air sampling from PNNL building stacks and results from actual stack samples presented.

2.0 Methods

2.1 Sample Filter Count and Acid Digest Count Comparison

In the summer of 2002, 60 samples were selected from the archive of sample filters that showed both a sample particulate loading and had also returned a positive result for radioactive material. These samples were sent back to the laboratory for further analysis; first by counting the filters again and then dissolving the particulate (radioactive and non-radioactive) material using acid digestion, drying the solution onto a planchet, and conducting a second count. The assumption was that digesting the sample filter would recover all radioactive material, even that embedded in the filter, and the self-absorption factor could be determined by taking the result of the first filter count and dividing by the result of the digested count. An average self-absorption factor could be determined from the results.

The selected sample filters were counted 500 min each in a 2π geometry by gas-flow proportional counting using a LB4100/W Low Background Alpha/Beta Counting System[b] as if they had just been collected. The 47 mm filter is mounted directly on a planchet (no chemical processing) with the loaded face of the filter exposed. Two-sided tape is placed on a 50.8 mm diameter by 3.2 mm deep stainless steel dish and the filter is lightly tapped down using tweezers or small glass rod. The filter may not extend beyond the lip of the planchet dish and additional tape may be used to help hold down a curled filter. Commercial grade P-10 gas (90% argon and 10% methane) is used with the detector system during counting. Alpha and beta instrument crosstalk is controlled by adjusting the instrument discriminator settings. The results were logged, and then the individual samples were run through an acid-digest process to separate the radioactive material from the filter and other non-radioactive particulate matter.

The individual digested samples were again counted 500 min each by gas-flow proportional counting. The sample material is first dissolved in a small amount of 16 M HNO_3 and then transferred to a tared sample planchet. The transfer vessel is rinsed at least three more times using 2- to 8-M HNO_3 and each time adding the rinsate to the planchet to assure quantitative transfer of the sample material. The solution is evaporated under a heat lamp to dryness and heated until there are no visible fumes. Tared sample planchets are then weighed again to determine the mass of the sample material deposited. Dried samples are then counted using the same LB4100/W Low Background Alpha/Beta Counting System and the results were logged. Sample process controls for the acid digestion include preparing sample blanks and sample spikes for analysis with the regular samples; these results are used in determining the digestion process efficiency. Results from both counting processes were compared.

The LB4100/W Low Background Alpha/Beta Counting System has the capability to count 16 samples at one time. The average alpha detector efficiency is 35 ± 3%, and the average beta detector efficiency is 55 ± 3%.

There are several sources of error associated with the sample results and are compensated for in determining the overall sample activity uncertainty. For the standard counting of a filter sample, these include net uncertainty in counts, decay factor uncertainty, detector efficiency uncertainty, and particulate abundance uncertainty. When counting digested samples that are evaporated onto a sample planchet, additional errors may be introduced including those associated with the digestion process, sample size, mass loading, moisture absorbed by the sample residue after preparation and prior to counting, and non-uniformity of the sample residue on the sample planchet. Other effects affecting the sample result include the count time, background, and counting efficiency.

2.2 Mass Loading Evaluation

An effort to weigh the sample filters in an attempt to measure the weight of the sample material analyzed was evaluated. Because the archived filters had not been weighed before use in the field, 50 new filters were weighed in an effort to determine an average filter tare weight (Mettler-Toledo AT400[c] analytical balance) that could be used as a basis to establish a probable sample mass on each filter. In addition to

---

[b] Canberra Industries, Inc., (formerly Oxford Nuclear Measurements Group, Oxford Instruments Ltd.), 800 Research Parkway, Meriden, CT 06450.

[c] Mettler-Toledo, Inc., 1900 Polaris Parkway, Columbus, OH 43240.

evaluating the 50 new filters, the sample planchets were also weighed before and after the solution was dried down to estimate an average mass loading for 20 samples.

2.3 Reflected Light Microscopy

A set of 30 random sample filters was selected from the archive for evaluation. The sample filters had a varying degree of particulate loading where the selection of light, medium, and heavy particulate loading was determined by visual appearance. These 30 sample filters were compared to a set of unused new filters. A reference image was taken of the entire area of each sample filter using a digital camera. Magnified images of the surface were collected using an Olympus BX Series[d] microscope.

The Olympus BX Series microscope system consists of: Olympus MicroSuite software, a digital Soft Imaging System ColorView II™ digital visible light camera, a 20X objective lens calibrated with a stage micrometer, and a Prior Scientific ProScan II™ motorized microscope stage. The Olympus MicroSuite software calculates and inserts the scale bar based upon a reference measurement. The MicroSuite software controls the ProScan™ stage which allows the software to create a surface reconstruction, called the Extended Focal Image, from multiple images collected in the z-dimension. The software also uses the images collected in the z-dimension to create a topographic 3D rendering of the surface. A series of images were captured for each sample over a distance of approximately 0.03 mm in the z-dimension. Both the Extended Focal Image and 3-D renderings were necessary for evaluation. The 3D rendering can be used to exaggerate the z-dimension topography.

The upper illuminator of the BX series microscope did not provide adequate image contrast, so two auxiliary lights were placed above either side of the microscope stage. Typical particle sizes were measured on select sample filters. Although a range of particle sizes was measured, distribution statistics were not acquired. Particle shapes were too irregular to assume equivalent spherical diameter. Dimensional measurements of representative captured particles were in the micrometer range for these samples. The goal was to obtain a range of the typical particle sizes of sample material and to estimate the extent of material burial within the filter.

3.0 Results

3.1 Sample Filter Count and Acid-Digest Count Comparison

Because of the low emission rate, the radioactivity on stack samples from PNNL facilities are often below detection limits. For this study, archive samples were selected so that results were likely to be above the critical level[e] (Lc) based on previous readings and thus would have detectable radioactivity. Appendix A provides detailed information on the samples and the raw data associated with them. The measurements of radioactivity in total pCi on the filter before and after digestion are shown in Table 1 for the 60 samples analyzed. Results indicate that the alpha results yielded a before/after digestion ratio (self-absorption factor) of 2.1 ± 2.9 (2σ) and a median value of 1.7. Thus, on average, more activity was measured by directly counting the filters before digestion than by counting after acid digestion. Only 5 samples had greater activity after acid digestion than before. The minimum ratio was 0.7, and the maximum ratio was

---

[d] Olympus America, Inc., 3500 Corporate Parkway, Center Valley, PA 18034.

[e] Lc is the net sample count (gross counts minus background counts) which must be exceeded before the sample is said to contain any measurable radioactive material above background. (Currie 1968)

8.7 with a wide variation encompassing the value of 1 where no difference can be detected between the two separate counts (no self-absorption), 0.85 (current self-absorption correction factor), and many other values.

Similar results were seen for beta analysis and these are also presented in Table 1. The beta results yielded a before/after digestion ratio of 1.9 ± 4.3 (2σ) and a median value of 1.4. It is again shown that more activity was measured by directly counting the filters before digestion than by counting after acid digestion. There were 8 samples where the activity after digestion was greater than the initial activity measure in the sample. The minimum ratio was 0.1, and the maximum ratio was 16.6.

The before and after digestion results were plotted against each other to further explore the data. Figures 1 and 2 show the plots and the coefficients of determination (R^2 values) for alpha results and beta results respectively. A relationship can be seen in both figures, but the R^2 values (the proportion of variance explained by the model) are weak at 0.66 for alpha results and 0.76 for beta results.

Figure 3 shows the plot of the initial alpha to initial beta results; the R^2 is 0.995. In general this shows that there are generally more betas measured than alpha by a factor of 1.37. The high degree of correlation does not follow through when comparing the digested alpha to digested beta results where the R^2 is 0.697. The results do not correlate well with routine samples where there are typically 8.7 times as many betas measured as alphas; however, routine samples are only counted for 10 min whereas the samples counted in this study were counted for 500 min each.

The method used did not provide results suitable to quantify self-absorption in actual stack samples. As mentioned earlier, errors associated with the initial sample filter counts and the additional errors from the acid digestion process may, in part, account for the low correlation in the data.

Table 1. Results from Counting Stack Filters Before and After Digestion

	Alpha			Beta		
	Initial	After Digestion		Initial	After Digestion	
Sample Id.	pCi/ sample	pCi/ sample	Ratio	pCi/ sample	pCi/ sample	Ratio
02-3047	1.45	1.26	1.15	2.04	5.75	0.35
02-3048	0.68	0.51	1.32	1.07	3.85	0.28
02-3049	0.07	0.05	1.43	0.08	1.73	0.04
02-3050	1.97	1.15	1.72	3.07	1.56	1.97
02-3051	0.96	0.68	1.42	1.59	3.98	0.40
02-3052	0.04	0.04	1.06	0.12	0.80	0.15
02-3053	0.09	0.11	0.83	0.58	0.33	1.77
02-3054	0.30	0.30	0.99	0.45	0.35	1.28
02-3055	1.35	1.02	1.32	1.85	2.37	0.78
02-3056	0.26	0.14	1.90	0.43	0.56	0.77
02-3090	0.27	0.14	1.97	0.39	0.14	2.81
02-3091	0.12	0.10	1.24	0.24	2.14	0.11
02-3092	0.15	0.21	0.70	0.25	0.23	1.09
02-3093	0.09	0.10	0.90	0.11	0.16	0.67
02-3094	1.55	1.34	1.16	2.84	2.68	1.06
02-3095	0.89	0.71	1.25	1.51	1.22	1.24
02-3096	0.91	0.90	1.02	1.39	1.34	1.04
02-3097	2.12	1.68	1.27	3.34	2.89	1.16
02-3098	0.16	0.09	1.77	0.30	0.11	2.77
02-3099	7.66	4.65	1.65	11.01	7.64	1.44
02-03180	0.29	0.09	3.01	0.43	0.36	1.22
02-03181	12.49	3.09	4.04	16.47	9.16	1.80
02-03182	0.06	0.04	1.50	2.02	0.12	16.61
02-03183	7.13	0.82	8.73	10.26	6.61	1.55
02-03184	8.44	1.04	8.13	12.60	3.87	3.26
02-03185	3.32	1.92	1.73	4.73	2.13	2.22
02-03186	3.70	1.05	3.54	5.12	1.48	3.47
02-03187	0.83	0.36	2.30	1.25	1.15	1.09
02-03188	2.05	0.67	3.04	2.90	1.95	1.49
02-03189	1.18	0.41	2.86	1.83	1.23	1.48
02-03190	0.64	0.26	2.48	0.89	0.74	1.20
02-03191	0.39	0.21	1.85	1.05	0.57	1.85
02-03192	0.17	0.11	1.60	0.84	0.26	3.27
02-03193	6.73	1.37	4.91	10.34	5.06	2.04
02-03194	20.38	5.72	3.56	27.87	9.82	2.84
02-03195	7.81	5.22	1.49	10.64	5.28	2.01
02-03196	0.86	0.27	3.21	1.33	0.41	3.26
02-03197	1.08	0.33	3.31	1.28	0.64	1.99
02-03198	0.59	0.30	1.97	0.93	0.92	1.00
02-03199	0.60	0.19	3.22	0.88	0.60	1.46
02-03358	8.44	4.57	1.85	12.10	7.31	1.65

	Alpha			Beta		
	Initial	After Digestion		Initial	After Digestion	
Sample Id.	pCi/ sample	pCi/ sample	Ratio	pCi/ sample	pCi/ sample	Ratio
02-03359	2.37	1.44	1.65	2.95	1.10	2.68
02-03360	4.61	4.78	0.97	7.08	6.75	1.05
02-03361	2.89	1.97	1.47	3.77	3.13	1.20
02-03362	0.15	0.14	1.10	0.36	0.27	1.37
02-03363	2.28	1.86	1.23	3.47	2.13	1.63
02-03364	5.90	4.13	1.43	8.02	6.07	1.32
02-03365	6.17	4.24	1.46	8.59	6.54	1.31
02-03366	1.49	0.49	3.04	1.78	0.26	6.75
02-03367	0.46	0.36	1.29	0.65	0.49	1.32
02-03348	6.53	5.53	1.18	9.68	7.96	1.22
02-03349	11.50	7.62	1.51	15.60	11.50	1.36
02-03350	3.58	1.75	2.05	5.02	2.57	1.95
02-03351	0.39	0.27	1.46	0.64	0.24	2.65
02-03352	1.38	0.63	2.19	2.11	1.00	2.11
02-03353	3.14	1.99	1.57	4.68	2.76	1.69
02-03354	1.40	0.66	2.13	1.88	0.75	2.50
02-03355	0.96	0.60	1.60	1.33	1.03	1.28
02-03356	1.74	0.96	1.81	2.46	1.95	1.26
02-03357	7.10	4.21	1.69	9.85	7.33	1.34
Average			*2.09*			*1.90*
Standard Deviation			*1.46*			*2.18*
2-Sigma			*2.86*			*4.27*
Max			*8.73*			*16.61*
Min			*0.70*			*0.04*

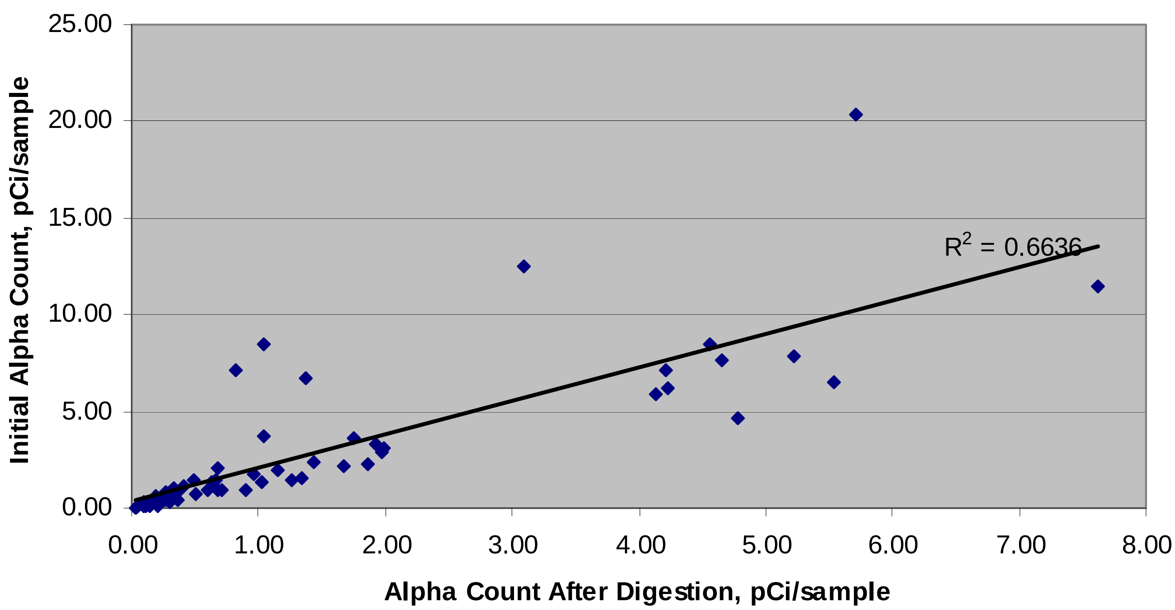


Figure 1. Plot of the Filter Data Counted Before and After Digestion – Alpha Activity

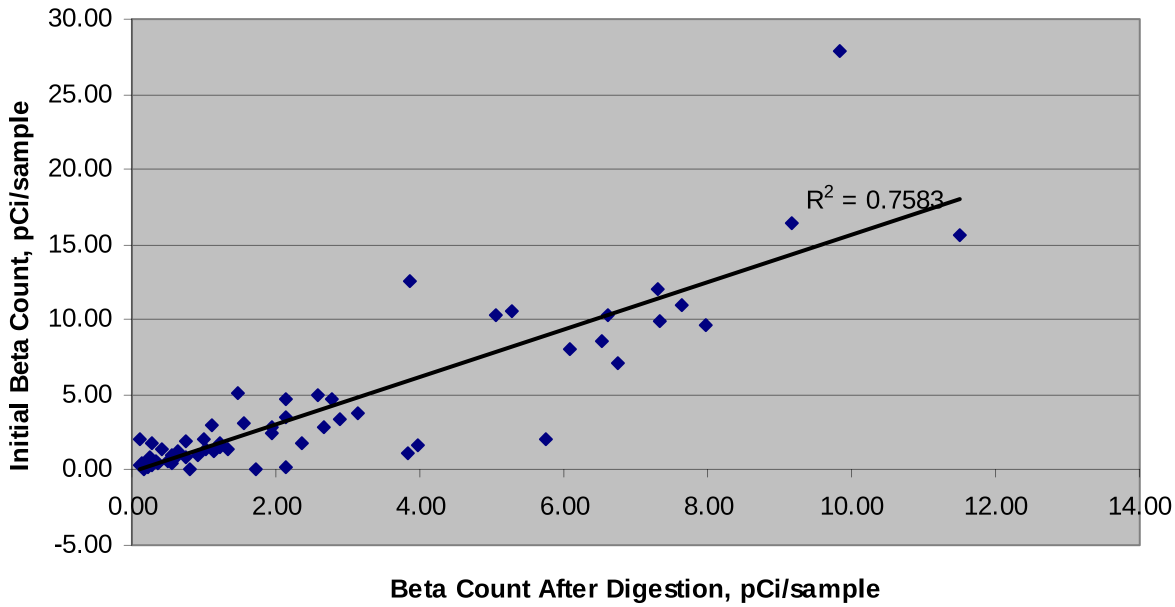


Figure 2. Plot of the Filter Data Counted Before and After Digestion – Beta Activity

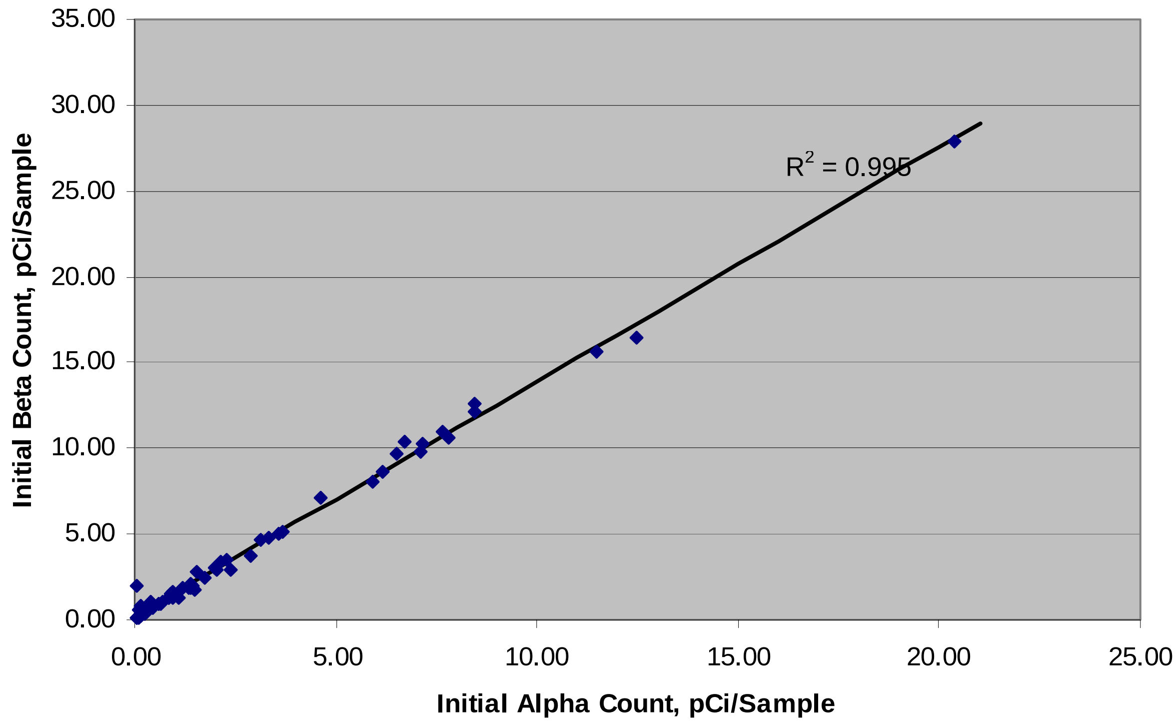


Figure 3. Plot of the Alpha to Beta Initial Filter Data Results

3.2 Mass Loading Evaluation

Fifty new glass fiber filters were weighed to establish a standard tare weight (see data in Appendix B). The average mass of the 50 new filters was 0.102 ± 0.008 (1σ) g and the range was 0.09 to 0.12 g. The ratio of the maximum to minimum weighed filter was 1.39. These effects raised concern as to whether the tare weight of the archive sample material could be precisely enough established without having pre-weighed them individually before use. Filter weight gain has been reported as 5 to 30 mg (Luetzelschwab 2000) for environmental air samples with a one-week collection period; this gain is in the noise of sample filter tare weight uncertainty.

Twenty of the archive samples were weighted prior to the acid digestion process (see data in Appendix B). For these 20 samples, the average loaded filter weight was 0.104 ± 0.006 (1σ) g and the range was 0.09 to 0.12 g which is very similar to the unused new filter data above. The ratio of the maximum to minimum archive sample filter weight was 1.25. For comparison, the results of six filter blanks that were also analyzed in this process result in an average filter weight of 0.109 ± 0.003 (1σ) g.

The weight of the sample material after digesting and drying onto the planchets was measured and was very small. For the 20 samples weighed, the average net sample weight after digestion was 1.1 ± 0.6 (1σ) mg per sample. This results in a mass loading of 0.08 ± 0.05 (1σ) mg cm^{-2} on the active area of the sample filters assuming it represents the total before digestion sample mass. For the digested filter blanks, the average net filter blank weight after digestion was 0.8 ± 1.0 (1σ) mg; two of the filter blanks had a negative net sample weight. Given that on average about half the radioactivity was measured after digestion versus before digestion, it is possible that some losses occurred in the digestion and drying process which would affect before digestion mass estimates.

Based on these results, it would be difficult to ascertain filter weight gain in the archived samples with such a wide range of tare weight possibilities as evidenced in the contrast between 1) the new and archive sample filter results, and 2) the net planchet weights of the archive samples and the filter blanks. While definitive sample mass loading cannot be determined, for the 20 samples evaluated, a mass loading of ~0.08 mg cm^{-2} per sample was obtained.

3.3 Reflected Light Microscopy

It was observed from the visible light microscopy that the vast majority of the particulate loading rests on top of the filter fiber media and it is difficult to determine how much material, if any, is imbedded in the filter. The structural fibers within the filter appear to be ~30 µm in diameter. Particle sizes on the surface were generally from 1 to 10 µm with the typical size being from 2 to 6 µm as shown in Appendix C. There is no indication of particulate overloading or particles obscuring other particles. The 3-D rendering allows the viewer to look topographically at the fibers on the sample face. The dark color on the edge of the 3D renderings represents the color of the sample surface at the edge of the image.

It is likely that radioactive material is well distributed throughout the particle loading as opposed to a thin layer on top of a particle layer as evaluated by Huang et al (2002). However, the size of radioactive particles compared to the size of nonradioactive particles is unknown. The radioactive particles may be in the smallest size range because they are emitted from HEPA-filtered locations before reaching the stack sampling system whereas other nonradioactive contributions to the stack sample may not be HEPA-filtered because they originate downstream of filtration.

The downstream side of a sample filter with heavy particulate loading was viewed under the microscope in an effort to determine if any of the particulate penetrated through the sample filter. This evaluation found the downstream side of the sample filter to be free from particulate matter. This is in contrast to the particles that were routinely found on the face of the sample filters.

The typical edge thickness of the filter was measured to be 49 μm (Appendix C) whereas the manufacturer stated filter thickness is 190 μm. It is likely that the edge thickness is compressed during the manufacturing process resulting in a smaller thickness at the edge over that of the active filter area. It is also presumed that the filter edge is compressed during use which could also contribute to an edge thickness smaller than the actual filter thickness.

4.0 Conclusions

Large error is associated with the sample filter analysis comparison and subsequently with the estimation of the absorption factor resulting in an inadequate method to estimate losses from self-absorption in the sample filter. Issues that influence the data results and the ability to make a determination of the self-absorption factor include the sensitivity of the instruments used, the filter paper variations, mass loading, and detector efficiencies. While there was very good correlation between the initial alpha results to initial beta results, the results do not appear to follow through from the initial to the after digested results obtained.

Mass-loading of filters could not be determined because the tare weights of the archive filters had not been previously measured and a generic tare weight had too much variability. The mass loading on the sample filter as determined after digestion and drying was ~0.08 mg cm^{-2}. The value may not be indicative of the total filter mass loading given that there may be undetermined losses associated with the digestion process.

While it is difficult to determine how much material is imbedded in the filter, observations from the microscopy analysis indicate that the vast majority of the particles remain on the top of the filter. Particle sizes collected at face velocities of 0.35 to 1.0 m s^{-1} were typically in the 2- to 6-μm range on sample filters evaluated. There is no indication of particulate overloading or particles obscuring other particles that would create significant self shielding. No particles were seen on the downstream side of the filter as would be expected if there were significant particulate penetration through the filter.

Sample analysis, mass loading, and reflected light microscopy were used to obtain a greater understanding of the self-absorption of activity on Versapor® 3000, 47 mm diameter filters, used at PNNL for particulate radioactive air sampling collected from building exhaust for environmental monitoring. The sample analysis comparison indicates more activity is measured by directly counting the sample filter rather than counting after the acid digestion process, however, the results were insufficient to quantitatively verify the current self-absorption factor or to recommend a different value. Comparing analytical results with the results from the mass loading and reflected light microscopy studies, and recognizing the competing factors between the mass loading on the filter and the particle penetration in the filter, the continued use of a 0.85 self-absorption factor is a conservative correction factor.

5.0 Acknowledgements

The authors would like to acknowledge the support of Betty Anderson, Jill Brandon, Wayne Cosby, Eric Damberg, Lori Darnell, Dan Edwards, Karl Pool, Chuck Sonderquist, Monte Sula, and Laurie True.

6.0 References

American National Standards Institute. 1999. *Sampling and Monitoring Releases of Airborne Radioactive Substances From the Stacks and Ducts of Nuclear Facilities*. Health Physics Society, ANSI/HPS N13.1-1999, McLean, Virginia.

Barnett JM, and JE Kane II. 1993. "Flow Rate Through a Filter with a 25-mm Diameter Aperture for Hanford Site Alpha Continuous Air Monitors." *Radiation Protection Management* 10(4):41-46.

Currie L. 1968. "Limits for Qualitative Detection and Quantitative Determination." *Journal of Analytical Chemistry* 40:586-593.

Higby DP. 1984. *Effects of Particle Size and Velocity on Burial Depth of Airborne Particles in Glass Fiber Filters*. Pacific Northwest Laboratory, PNL-5278, Richland, Washington.

Huang S, SD Schery, RE Alcantara, JC Rodgers, and PT Wasiolek. 2002. "Influence of Dust Loading on the Alpha-Particle Energy Resolution of Continuous Air Monitors for Thin Deposits of Radioactive Aerosols." *Health Physics* 83(6):884–891.

Luetzelschwab JW, C Storey, K Zraly, and D Dussinger. 2000. "Self Absorption of Alpha and Beta Particles in a Fiberglass Filter." *Health Physics* 79(4):425-430.

Stevens DC, and AER Toureau. 1963. *The Effect of Particle Size and Dust Loading on the Shape of Alpha Pulse Height Spectra of Air Sample Filters*. Atomic Energy Research Establishment, AERE-R 4249, Harwell, Berkshire, England, United Kingdom.

Appendix A—Raw Data From Filter Analyses

The two tables (Tables A.1 and A.2) in this appendix show 1) the initial filter sample results and 2) the after digestion sample results. The information below provides the detail for the individual columns of data:

Sample ID	The unique identification number associated with the sample for tracking purposes.
Alpha Efficiency	The detector efficiency for alpha radiation measurements where 'Eff.' is the efficiency in decimal and '+/-' is the error associated with the detector alpha efficiency also in decimal format.
Beta Efficiency	The detector efficiency for beta radiation measurements where 'Eff.' is the efficiency in decimal and '+/-' is the error associated with the detector beta efficiency also in decimal format.
Eff.	The efficiency of the alpha or beta detector in decimal format.
+/-	The error associated with the detector efficiency in decimal format.
Background (cpm)	The alpha or beta detector count rate, without a source or sample, expressed in counts per minute (cpm).
Alpha	Results for alpha particle measurements.
Beta	Results for beta particle measurements.
Variance	The variance is the square of the standard deviation (the detector efficiency error divided by the detector efficiency) of the indicated alpha or beta detector efficiency and associated error shown.
MDA (pCi)	The Minimum Detectable Activity (MDA) given in picocuries (pCi).
Background (pCi)	The alpha or beta activity as determined by the background detector count rate and the detector efficiency without a source or sample present expressed in picoCuries.
Alpha Results	The results of the alpha sample measurement showing the activity of the sample, the two-sigma error associated with the result, the value of the critical level (Lc) in picoCuries and whether or not the sample is above or below the critical level value.
Beta Results	The results of the beta sample measurement showing the activity of the sample, the two-sigma error associated with the result, the value of the critical level in picoCuries and whether or not the sample is above or below the critical level value.
pCi/sample	The resultant picoCuries in the sample.
+/-, 2σ (pCi)	The two-sigma sample error associated with the sample result in picoCuries.
Lc (pCi)	The critical level for the individual sample results in picoCuries.
>,< Lc	The sample indicator as to whether or not the result is greater than or less than the critical level.

Table A.1. Initial Results from Filter Analysis

	Initial Filter Sample Results																			
	Alpha Efficiency		Beta Efficiency		Background (cpm)		Variance		MDA (pCi)		Background (pCi)		Alpha Results				Beta Results			
Sample ID	Eff.	+/-	Eff.	+/-	Alpha	Beta	Alpha	Beta	Alpha	Beta	Alpha	Beta	pCi/ sample	+/- 2σ (pCi)	Lc (pCi)	>,< Lc	pCi/ sample	+/- 2σ (pCi)	Lc (pCi)	>,< Lc
02-3047	0.37	0.005	0.56	0.016	0.12	1.31	0.0002	0.0008	0.09	0.20	0.15	1.06	1.45	0.14	0.04	>Lc	2.04	0.20	0.10	>Lc
02-3048	0.37	0.005	0.56	0.016	0.19	1.09	0.0002	0.0008	0.12	0.18	0.24	0.88	0.68	0.11	0.06	>Lc	1.07	0.15	0.09	>Lc
02-3049	0.37	0.005	0.56	0.016	0.02	1.19	0.0002	0.0008	0.04	0.19	0.02	0.95	0.07	0.03	0.02	>Lc	0.08	0.11	0.09	<Lc
02-3050	0.37	0.005	0.56	0.016	0.11	1.29	0.0002	0.0008	0.09	0.19	0.13	1.03	1.97	0.15	0.02	>Lc	3.07	0.26	0.10	>Lc
02-3051	0.38	0.005	0.57	0.016	0.08	1.61	0.0002	0.0008	0.08	0.21	0.09	1.27	0.96	0.11	0.04	>Lc	1.59	0.18	0.09	>Lc
02-3052	0.37	0.005	0.55	0.016	0.04	1.35	0.0002	0.0008	0.06	0.20	0.05	1.10	0.04	0.05	0.03	>Lc	0.12	0.13	0.10	>Lc
02-3053	0.37	0.005	0.58	0.017	0.08	2.09	0.0002	0.0009	0.08	0.24	0.09	1.62	0.09	0.05	0.04	>Lc	0.58	0.16	0.12	>Lc
02-3054	0.37	0.005	0.56	0.006	0.03	1.36	0.0002	0.0001	0.05	0.20	0.04	1.10	0.30	0.06	0.02	>Lc	0.45	0.13	0.10	>Lc
02-3055	0.38	0.005	0.58	0.016	0.02	1.23	0.0002	0.0008	0.04	0.19	0.02	0.96	1.35	0.12	0.02	>Lc	1.85	0.19	0.09	>Lc
02-3056	0.38	0.005	0.58	0.017	0.07	1.27	0.0002	0.0008	0.07	0.19	0.08	0.98	0.26	0.06	0.03	>Lc	0.43	0.12	0.09	>Lc
02-3090	0.37	0.005	0.56	0.016	0.14	1.23	0.0002	0.0008	0.10	0.19	0.17	0.99	0.27	0.08	0.05	>Lc	0.39	0.13	0.09	>Lc
02-3091	0.37	0.005	0.56	0.016	0.21	1.13	0.0002	0.0008	0.12	0.18	0.26	0.92	0.12	0.08	0.06	>Lc	0.24	0.12	0.09	>Lc
02-3092	0.37	0.005	0.56	0.016	0.02	1.13	0.0002	0.0008	0.04	0.18	0.02	0.90	0.15	0.04	0.02	>Lc	0.25	0.12	0.09	>Lc
02-3093	0.37	0.005	0.56	0.016	0.10	1.35	0.0002	0.0008	0.09	0.20	0.13	1.08	0.09	0.06	0.04	>Lc	0.11	0.12	0.10	>Lc
02-3094	0.38	0.005	0.57	0.016	0.08	1.55	0.0002	0.0008	0.08	0.21	0.10	1.22	1.55	0.14	0.04	>Lc	2.84	0.24	0.10	>Lc
02-3095	0.37	0.005	0.55	0.016	0.06	1.32	0.0002	0.0008	0.07	0.20	0.08	1.07	0.89	0.10	0.03	>Lc	1.51	0.18	0.10	>Lc
02-3096	0.37	0.005	0.58	0.017	0.09	2.00	0.0002	0.0009	0.08	0.23	0.11	1.55	0.91	0.11	0.04	>Lc	1.39	0.19	0.11	>Lc
02-3097	0.37	0.005	0.56	0.006	0.05	1.34	0.0002	0.0001	0.06	0.20	0.06	1.09	2.12	0.16	0.03	>Lc	3.34	0.20	0.10	>Lc
02-3098	0.38	0.005	0.58	0.016	0.02	1.16	0.0002	0.0008	0.04	0.18	0.02	0.90	0.16	0.04	0.02	>Lc	0.30	0.12	0.09	>Lc
02-3099	0.38	0.005	0.58	0.017	0.03	1.18	0.0002	0.0008	0.05	0.18	0.03	0.91	7.66	0.34	0.02	>Lc	11.01	0.70	0.09	>Lc
02-3180	0.37	0.005	0.56	0.016	0.12	1.24	0.0002	0.0008	0.09	0.18	0.15	1.00	0.29	0.08	0.04	>Lc	0.43	0.13	0.09	>Lc
02-3181	0.37	0.005	0.56	0.016	0.03	1.09	0.0002	0.0008	0.05	0.17	0.03	0.87	12.49	0.49	0.02	>Lc	16.47	1.00	0.08	>Lc
02-3182	0.37	0.005	0.56	0.016	0.11	1.41	0.0002	0.0008	0.09	0.19	0.14	1.13	0.06	0.06	0.04	>Lc	2.02	0.20	0.09	>Lc
02-3183	0.38	0.005	0.57	0.016	0.13	1.60	0.0002	0.0008	0.09	0.20	0.15	1.26	7.13	0.33	0.04	>Lc	10.26	0.65	0.10	>Lc
02-3184	0.37	0.005	0.55	0.016	0.06	1.34	0.0002	0.0008	0.06	0.19	0.07	1.09	8.44	0.37	0.03	>Lc	12.60	0.79	0.09	>Lc
02-3185	0.37	0.005	0.58	0.017	0.08	2.03	0.0002	0.0008	0.07	0.22	0.10	1.57	3.32	0.21	0.03	>Lc	4.73	0.35	0.11	>Lc
02-3186	0.37	0.005	0.56	0.006	0.05	1.38	0.0002	0.0001	0.06	0.19	0.06	1.12	3.70	0.22	0.03	>Lc	5.12	0.24	0.10	>Lc
02-3187	0.38	0.005	0.58	0.016	0.02	1.07	0.0002	0.0008	0.04	0.17	0.03	0.84	0.83	0.09	0.02	>Lc	1.25	0.15	0.08	>Lc
02-3188	0.38	0.005	0.58	0.017	0.03	1.12	0.0002	0.0008	0.05	0.17	0.03	0.86	2.05	0.15	0.02	>Lc	2.90	0.24	0.08	>Lc
02-3189	0.38	0.005	0.59	0.017	0.06	2.20	0.0002	0.0008	0.06	0.23	0.07	1.68	1.18	0.12	0.03	>Lc	1.83	0.21	0.11	>Lc
02-3190	0.37	0.005	0.56	0.016	0.12	1.24	0.0002	0.0008	0.09	0.18	0.15	1.00	0.64	0.10	0.04	>Lc	0.89	0.15	0.09	>Lc
02-3191	0.37	0.005	0.56	0.016	0.03	1.09	0.0002	0.0008	0.05	0.17	0.03	0.87	0.39	0.07	0.02	>Lc	1.05	0.15	0.08	>Lc
02-3192	0.37	0.005	0.56	0.016	0.11	1.41	0.0002	0.0008	0.09	0.19	0.14	1.13	0.17	0.07	0.04	>Lc	0.84	0.15	0.09	>Lc
02-3193	0.38	0.005	0.57	0.016	0.13	1.60	0.0002	0.0008	0.09	0.20	0.15	1.26	6.73	0.32	0.04	>Lc	10.34	0.66	0.10	>Lc
02-3194	0.37	0.005	0.55	0.016	0.06	1.34	0.0002	0.0008	0.06	0.19	0.07	1.09	20.38	0.71	0.03	>Lc	27.87	1.66	0.09	>Lc
02-3195	0.37	0.005	0.58	0.017	0.08	2.03	0.0002	0.0008	0.07	0.22	0.10	1.57	7.81	0.35	0.03	>Lc	10.64	0.68	0.11	>Lc
02-3196	0.37	0.005	0.56	0.006	0.05	1.38	0.0002	0.0001	0.06	0.19	0.06	1.12	0.86	0.10	0.03	>Lc	1.33	0.15	0.10	>Lc
02-3197	0.38	0.005	0.58	0.016	0.02	1.07	0.0002	0.0008	0.04	0.17	0.03	0.84	1.08	0.11	0.02	>Lc	1.28	0.15	0.08	>Lc
02-3198	0.38	0.005	0.58	0.017	0.03	1.12	0.0002	0.0008	0.05	0.17	0.03	0.86	0.59	0.08	0.02	>Lc	0.93	0.14	0.08	>Lc
02-3199	0.38	0.005	0.59	0.017	0.06	2.20	0.0002	0.0008	0.06	0.23	0.07	1.68	0.60	0.09	0.03	>Lc	0.88	0.17	0.11	>Lc
02-3358	0.37	0.005	0.56	0.016	0.12	1.23	0.0002	0.0008	0.09	0.18	0.14	0.99	8.44	0.37	0.04	>Lc	12.10	0.76	0.09	>Lc
02-3359	0.37	0.005	0.56	0.016	0.19	1.13	0.0002	0.0008	0.11	0.18	0.23	0.91	2.37	0.18	0.05	>Lc	2.95	0.24	0.09	>Lc
02-3360	0.37	0.005	0.56	0.016	0.02	1.05	0.0002	0.0008	0.04	0.17	0.02	0.84	4.61	0.25	0.02	>Lc	7.08	0.47	0.08	>Lc
02-3361	0.37	0.005	0.56	0.016	0.11	1.35	0.0002	0.0008	0.09	0.19	0.14	1.07	2.89	0.19	0.04	>Lc	3.77	0.29	0.09	>Lc
02-3362	0.37	0.005	0.55	0.016	0.07	1.33	0.0002	0.0008	0.07	0.19	0.08	1.08	0.15	0.06	0.03	>Lc	0.36	0.13	0.09	>Lc
02-3363	0.37	0.005	0.56	0.006	0.05	1.27	0.0002	0.0001	0.06	0.19	0.06	1.03	2.28	0.17	0.03	>Lc	3.47	0.20	0.09	>Lc
02-3364	0.38	0.005	0.58	0.016	0.02	1.06	0.0002	0.0008	0.03	0.16	0.02	0.83	5.90	0.29	0.01	>Lc	8.02	0.52	0.08	>Lc
02-3365	0.38	0.005	0.58	0.017	0.02	1.14	0.0002	0.0008	0.04	0.17	0.03	0.88	6.17	0.30	0.02	>Lc	8.59	0.55	0.08	>Lc
02-3366	0.38	0.005	0.59	0.017	0.07	2.14	0.0002	0.0008	0.07	0.23	0.08	1.64	1.49	0.13	0.03	>Lc	1.78	0.20	0.11	>Lc
02-3367	0.34	0.005	0.55	0.016	0.03	0.92	0.0002	0.0008	0.05	0.16	0.04	0.76	0.46	0.08	0.02	>Lc	0.65	0.13	0.08	>Lc
02-3348	0.37	0.005	0.56	0.016	0.17	1.17	0.0002	0.0008	0.10	0.18	0.20	0.95	6.53	0.31	0.05	>Lc	9.68	0.62	0.09	>Lc

	Initial Filter Sample Results																			
	Alpha Efficiency		Beta Efficiency		Background (cpm)		Variance		MDA (pCi)		Background (pCi)		Alpha Results				Beta Results			
Sample ID	Eff.	+/-	Eff.	+/-	Alpha	Beta	Alpha	Beta	Alpha	Beta	Alpha	Beta	pCi/ sample	+/- 2σ (pCi)	Lc (pCi)	>,< Lc	pCi/ sample	+/- 2σ (pCi)	Lc (pCi)	>,< Lc
02-3349	0.37	0.005	0.56	0.016	0.21	1.08	0.0002	0.0008	0.12	0.17	0.26	0.88	11.50	0.46	0.06	>Lc	15.60	0.95	0.08	>Lc
02-3350	0.37	0.005	0.56	0.016	0.02	1.03	0.0002	0.0008	0.04	0.17	0.02	0.83	3.58	0.21	0.02	>Lc	5.02	0.35	0.08	>Lc
02-3351	0.37	0.005	0.56	0.016	0.11	1.21	0.0002	0.0008	0.09	0.18	0.14	0.96	0.39	0.08	0.04	>Lc	0.64	0.13	0.09	>Lc
02-3352	0.38	0.005	0.57	0.016	0.11	1.53	0.0002	0.0008	0.08	0.20	0.13	1.21	1.38	0.13	0.04	>Lc	2.11	0.21	0.10	>Lc
02-3353	0.37	0.005	0.55	0.016	0.05	1.28	0.0002	0.0008	0.06	0.19	0.05	1.04	3.14	0.20	0.03	>Lc	4.68	0.34	0.09	>Lc
02-3354	0.37	0.005	0.58	0.017	0.05	1.98	0.0002	0.0008	0.06	0.22	0.06	1.54	1.40	0.13	0.03	>Lc	1.88	0.21	0.11	>Lc
02-3355	0.37	0.005	0.56	0.006	0.04	1.30	0.0002	0.0001	0.05	0.19	0.05	1.06	0.96	0.10	0.02	>Lc	1.33	0.15	0.09	>Lc
02-3356	0.38	0.005	0.58	0.016	0.02	1.05	0.0002	0.0008	0.04	0.16	0.03	0.82	1.74	0.14	0.02	>Lc	2.46	0.21	0.08	>Lc
02-3357	0.38	0.005	0.58	0.017	0.02	1.16	0.0002	0.0008	0.04	0.17	0.02	0.89	7.10	0.32	0.02	>Lc	9.85	0.62	0.08	>Lc

Table A.2 After Digestion Results from Filter Analysis

	After Digestion Sample Results																			
	Alpha Efficiency		Beta Efficiency		Background (cpm)		Variance		MDA (pCi)		Background (pCi)		Alpha Results				Beta Results			
Sample ID	Eff.	+/-	Eff.	+/-	Alpha	Beta	Alpha	Beta	Alpha	Beta	Alpha	Beta	pCi/ sample	+/- 2σ (pCi)	Lc (pCi)	>,< Lc	pCi/ sample	+/- 2σ (pCi)	Lc (pCi)	>,< Lc
02-3047	0.33	0.004	0.52	0.007	0.15	1.21	0.0001	0.0002	0.12	0.20	0.20	1.05	1.26	0.14	0.05	>Lc	5.75	0.28	0.10	>Lc
02-3048	0.33	0.004	0.51	0.007	0.20	1.18	0.0002	0.0002	0.14	0.20	0.27	1.04	0.51	0.11	0.06	>Lc	3.85	0.23	0.10	>Lc
02-3049	0.33	0.004	0.52	0.007	0.03	1.11	0.0001	0.0002	0.05	0.20	0.03	0.97	0.05	0.04	0.02	>Lc	1.73	0.17	0.10	>Lc
02-3050	0.32	0.004	0.52	0.007	0.10	1.22	0.0002	0.0002	0.10	0.21	0.14	1.07	1.15	0.13	0.05	>Lc	1.56	0.17	0.10	>Lc
02-3051	0.33	0.004	0.53	0.007	0.10	1.57	0.0001	0.0002	0.10	0.23	0.13	1.35	0.68	0.10	0.05	>Lc	3.98	0.24	0.11	>Lc
02-3052	0.33	0.004	0.52	0.007	0.05	1.44	0.0001	0.0002	0.07	0.22	0.07	1.25	0.04	0.04	0.03	>Lc	0.80	0.15	0.11	>Lc
02-3053	0.33	0.004	0.53	0.007	0.06	2.14	0.0001	0.0002	0.08	0.26	0.08	1.81	0.11	0.05	0.03	>Lc	0.33	0.16	0.13	>Lc
02-3054	0.33	0.004	0.51	0.007	0.03	1.32	0.0002	0.0002	0.05	0.21	0.03	1.16	0.30	0.06	0.02	>Lc	0.35	0.14	0.11	>Lc
02-3055	0.34	0.004	0.54	0.007	0.03	1.36	0.0001	0.0002	0.05	0.21	0.04	1.13	1.02	0.11	0.02	>Lc	2.37	0.19	0.10	>Lc
02-3056	0.34	0.004	0.53	0.007	0.01	1.25	0.0001	0.0002	0.04	0.20	0.02	1.06	0.14	0.04	0.02	>Lc	0.56	0.14	0.10	>Lc
02-3090	0.33	0.004	0.52	0.007	0.15	1.21	0.0001	0.0002	0.12	0.20	0.20	1.05	0.14	0.08	0.05	>Lc	0.14	0.12	0.10	>Lc
02-3091	0.33	0.004	0.51	0.007	0.20	1.18	0.0002	0.0002	0.14	0.20	0.27	1.04	0.10	0.08	0.06	>Lc	2.14	0.18	0.10	>Lc
02-3092	0.33	0.004	0.52	0.007	0.03	1.11	0.0001	0.0002	0.05	0.20	0.03	0.97	0.21	0.05	0.02	>Lc	0.23	0.12	0.10	>Lc
02-3093	0.32	0.004	0.52	0.007	0.10	1.22	0.0002	0.0002	0.10	0.21	0.14	1.07	0.10	0.06	0.05	>Lc	0.16	0.13	0.10	>Lc
02-3094	0.33	0.004	0.53	0.007	0.10	1.57	0.0001	0.0002	0.10	0.23	0.13	1.35	1.34	0.14	0.05	>Lc	2.68	0.20	0.11	>Lc
02-3095	0.33	0.004	0.52	0.007	0.05	1.44	0.0001	0.0002	0.07	0.22	0.07	1.25	0.71	0.10	0.03	>Lc	1.22	0.16	0.11	>Lc
02-3096	0.33	0.004	0.53	0.007	0.06	2.14	0.0001	0.0002	0.08	0.26	0.08	1.81	0.90	0.11	0.03	>Lc	1.34	0.19	0.13	>Lc
02-3097	0.33	0.004	0.51	0.007	0.03	1.32	0.0002	0.0002	0.05	0.21	0.03	1.16	1.68	0.14	0.02	>Lc	2.89	0.21	0.11	>Lc
02-3098	0.34	0.004	0.54	0.007	0.03	1.36	0.0001	0.0002	0.05	0.21	0.04	1.13	0.09	0.04	0.02	>Lc	0.11	0.13	0.10	>Lc
02-3099	0.34	0.004	0.53	0.007	0.01	1.25	0.0001	0.0002	0.04	0.20	0.02	1.06	4.65	0.25	0.02	>Lc	7.64	0.33	0.10	>Lc
02-3180	0.33	0.004	0.52	0.007	0.13	1.20	0.0001	0.0002	0.09	0.17	0.17	1.04	0.09	0.07	0.04	>Lc	0.36	0.13	0.09	>Lc
02-3181	0.33	0.004	0.51	0.007	0.20	1.17	0.0002	0.0002	0.11	0.17	0.27	1.03	3.09	0.21	0.06	>Lc	9.16	0.38	0.09	>Lc
02-3182	0.33	0.004	0.52	0.007	0.02	1.04	0.0001	0.0002	0.04	0.16	0.02	0.91	0.04	0.03	0.02	>Lc	0.12	0.12	0.08	>Lc
02-3183	0.32	0.004	0.52	0.007	0.09	1.15	0.0002	0.0002	0.08	0.17	0.13	1.00	0.82	0.11	0.04	>Lc	6.61	0.30	0.08	>Lc
02-3184	0.33	0.004	0.53	0.007	0.12	1.72	0.0001	0.0002	0.09	0.21	0.17	1.48	1.04	0.13	0.04	>Lc	3.87	0.24	0.10	>Lc
02-3185	0.33	0.004	0.52	0.007	0.07	1.25	0.0001	0.0002	0.07	0.18	0.10	1.08	1.92	0.16	0.03	>Lc	2.13	0.18	0.09	>Lc
02-3186	0.33	0.004	0.51	0.007	0.05	1.27	0.0002	0.0002	0.06	0.18	0.07	1.11	1.05	0.12	0.03	>Lc	1.48	0.17	0.09	>Lc
02-3187	0.34	0.004	0.53	0.007	0.02	1.14	0.0001	0.0002	0.04	0.17	0.03	0.97	0.36	0.07	0.02	>Lc	1.15	0.15	0.08	>Lc
02-3188	0.34	0.004	0.54	0.007	0.03	1.30	0.0001	0.0002	0.04	0.17	0.03	1.08	0.67	0.09	0.02	>Lc	1.95	0.17	0.09	>Lc

	After Digestion Sample Results																			
	Alpha Efficiency		Beta Efficiency		Background (cpm)		Variance		MDA (pCi)		Background (pCi)		Alpha Results				Beta Results			
Sample ID	Eff.	+/-	Eff.	+/-	Alpha	Beta	Alpha	Beta	Alpha	Beta	Alpha	Beta	pCi/ sample	+/- 2σ (pCi)	Lc (pCi)	>,< Lc	pCi/ sample	+/- 2σ (pCi)	Lc (pCi)	>,< Lc
02-3189	0.34	0.004	0.54	0.007	0.06	1.57	0.0001	0.0002	0.06	0.19	0.08	1.31	0.41	0.08	0.03	>Lc	1.23	0.16	0.09	>Lc
02-3190	0.33	0.004	0.52	0.007	0.13	1.20	0.0001	0.0002	0.09	0.17	0.17	1.04	0.26	0.06	0.02	>Lc	0.74	0.13	0.08	>Lc
02-3191	0.33	0.004	0.51	0.007	0.20	1.17	0.0002	0.0002	0.11	0.17	0.27	1.03	0.21	0.06	0.02	>Lc	0.57	0.12	0.07	>Lc
02-3192	0.33	0.004	0.52	0.007	0.02	1.04	0.0001	0.0002	0.04	0.16	0.02	0.91	0.11	0.04	0.02	>Lc	0.26	0.12	0.08	>Lc
02-3193	0.32	0.004	0.52	0.007	0.09	1.15	0.0002	0.0002	0.08	0.17	0.13	1.00	1.37	0.14	0.04	>Lc	5.06	0.26	0.09	>Lc
02-3194	0.33	0.004	0.53	0.007	0.12	1.72	0.0001	0.0002	0.09	0.21	0.17	1.48	5.72	0.30	0.06	>Lc	9.82	0.39	0.09	>Lc
02-3195	0.33	0.004	0.52	0.007	0.07	1.25	0.0001	0.0002	0.07	0.18	0.10	1.08	5.22	0.27	0.02	>Lc	5.28	0.26	0.08	>Lc
02-3196	0.33	0.004	0.51	0.007	0.05	1.27	0.0002	0.0002	0.06	0.18	0.07	1.11	0.27	0.08	0.04	>Lc	0.41	0.13	0.08	>Lc
02-3197	0.34	0.004	0.53	0.007	0.02	1.14	0.0001	0.0002	0.04	0.17	0.03	0.97	0.33	0.09	0.04	>Lc	0.64	0.16	0.10	>Lc
02-3198	0.34	0.004	0.54	0.007	0.03	1.30	0.0001	0.0002	0.04	0.17	0.03	1.08	0.30	0.07	0.03	>Lc	0.92	0.15	0.09	>Lc
02-3199	0.34	0.004	0.54	0.007	0.06	1.57	0.0001	0.0002	0.06	0.19	0.08	1.31	0.19	0.06	0.03	>Lc	0.60	0.14	0.09	>Lc
02-3358	0.33	0.004	0.52	0.007	0.13	1.18	0.0001	0.0002	0.09	0.17	0.18	1.02	4.57	0.25	0.02	>Lc	7.31	0.31	0.08	>Lc
02-3359	0.33	0.004	0.51	0.007	0.18	1.14	0.0002	0.0002	0.11	0.17	0.25	1.00	1.44	0.13	0.03	>Lc	1.10	0.16	0.09	>Lc
02-3360	0.33	0.004	0.52	0.007	0.03	1.07	0.0001	0.0002	0.05	0.17	0.04	0.93	4.78	0.26	0.02	>Lc	6.75	0.30	0.08	>Lc
02-3361	0.32	0.004	0.52	0.007	0.09	1.10	0.0002	0.0002	0.08	0.17	0.13	0.96	1.97	0.16	0.03	>Lc	3.13	0.20	0.08	>Lc
02-3362	0.33	0.004	0.53	0.007	0.11	1.54	0.0001	0.0002	0.09	0.19	0.16	1.32	0.14	0.05	0.02	>Lc	0.27	0.11	0.08	>Lc
02-3363	0.33	0.004	0.52	0.007	0.07	1.23	0.0001	0.0002	0.07	0.18	0.10	1.06	1.86	0.16	0.04	>Lc	2.13	0.18	0.08	>Lc
02-3364	0.33	0.004	0.53	0.007	0.07	2.74	0.0001	0.0002	0.07	0.26	0.09	2.32	4.13	0.25	0.05	>Lc	6.07	0.29	0.08	>Lc
02-3365	0.33	0.004	0.51	0.007	0.05	1.34	0.0002	0.0002	0.06	0.19	0.06	1.18	4.24	0.24	0.02	>Lc	6.54	0.30	0.08	>Lc
02-3366	0.34	0.004	0.53	0.007	0.02	1.13	0.0001	0.0002	0.04	0.17	0.03	0.96	0.49	0.09	0.04	>Lc	0.26	0.12	0.08	>Lc
02-3367	0.34	0.004	0.54	0.007	0.02	1.22	0.0001	0.0002	0.04	0.17	0.03	1.01	0.36	0.09	0.04	>Lc	0.49	0.15	0.10	>Lc
02-3348	0.33	0.004	0.52	0.007	0.13	1.18	0.0001	0.0002	0.09	0.17	0.18	1.02	5.53	0.29	0.04	>Lc	7.96	0.34	0.08	>Lc
02-3349	0.33	0.004	0.51	0.007	0.18	1.14	0.0002	0.0002	0.11	0.17	0.25	1.00	7.62	0.35	0.05	>Lc	11.50	0.44	0.08	>Lc
02-3350	0.33	0.004	0.52	0.007	0.03	1.07	0.0001	0.0002	0.05	0.17	0.04	0.93	1.75	0.15	0.02	>Lc	2.57	0.19	0.08	>Lc
02-3351	0.32	0.004	0.52	0.007	0.09	1.10	0.0002	0.0002	0.08	0.17	0.13	0.96	0.27	0.08	0.04	>Lc	0.24	0.12	0.08	>Lc
02-3352	0.33	0.004	0.53	0.007	0.11	1.54	0.0001	0.0002	0.09	0.19	0.16	1.32	0.63	0.10	0.04	>Lc	1.00	0.16	0.10	>Lc
02-3353	0.33	0.004	0.52	0.007	0.07	1.23	0.0001	0.0002	0.07	0.18	0.10	1.06	1.99	0.16	0.03	>Lc	2.76	0.20	0.09	>Lc
02-3354	0.33	0.004	0.53	0.007	0.07	2.74	0.0001	0.0002	0.07	0.26	0.09	2.32	0.66	0.10	0.03	>Lc	0.75	0.19	0.13	>Lc
02-3355	0.33	0.004	0.51	0.007	0.05	1.34	0.0002	0.0002	0.06	0.19	0.06	1.18	0.60	0.09	0.03	>Lc	1.03	0.16	0.09	>Lc
02-3356	0.34	0.004	0.53	0.007	0.02	1.13	0.0001	0.0002	0.04	0.17	0.03	0.96	0.96	0.12	0.03	>Lc	1.95	0.18	0.09	>Lc
02-3357	0.34	0.004	0.54	0.007	0.02	1.22	0.0001	0.0002	0.04	0.17	0.03	1.01	4.21	0.24	0.02	>Lc	7.33	0.32	0.08	>Lc

Appendix B—Filter and Sample Weights

Table B.1 shows the individual tare weights of 50 new Versapor® 3000 filters from lot number 00721, product number P/N 60129 (629-66387). The maximum weight was 0.1212 g and the minimum weight was 0.0870 g; the maximum to minimum weight ratio is 1.39. The average weight is 0.102 ± 0.008 g, and the 2-σ error is 15.2%.

Tables B.2 and B.3 show the results for digested sample weights (Table B.2) and the digested filter blank weights (Table B.3). The average net sample mass is 1.1 ± 0.6 mg and the average net digested filter blank mass is 0.8 ± 1.0 mg.

Weight values shown in Tables B.1, B.2 and B.3 are all in grams (g). Filter numbers (Filter #) and sample identification numbers (Sample ID) are provided for tracking purposes.

Table B.1 Tare Weights of New Filters

Filter #	Weight (g)
1	0.1059
2	0.1069
3	0.1100
4	0.1143
5	0.1090
6	0.1054
7	0.1212
8	0.0989
9	0.0989
10	0.1024
11	0.0986
12	0.1077
13	0.1037
14	0.1046
15	0.0976
16	0.1007
17	0.1037
18	0.0936
19	0.1109
20	0.1029
21	0.1085
22	0.1085
23	0.0978
24	0.1065
25	0.1075
26	0.0895
27	0.0921

Filter #	Weight (g)
28	0.1004
29	0.1067
30	0.1160
31	0.1029
32	0.0945
33	0.1097
34	0.1126
35	0.1160
36	0.0975
37	0.0903
38	0.0901
39	0.0923
40	0.0870
41	0.1021
42	0.0904
43	0.0923
44	0.0960
45	0.1002
46	0.1014
47	0.0970
48	0.1024
49	0.0880
50	0.1033
Average	*0.1019*
StDev	*0.0079*
Max	*0.1212*
Min	*0.0870*

Table B.2 Mass Results of Digested Sample Filters

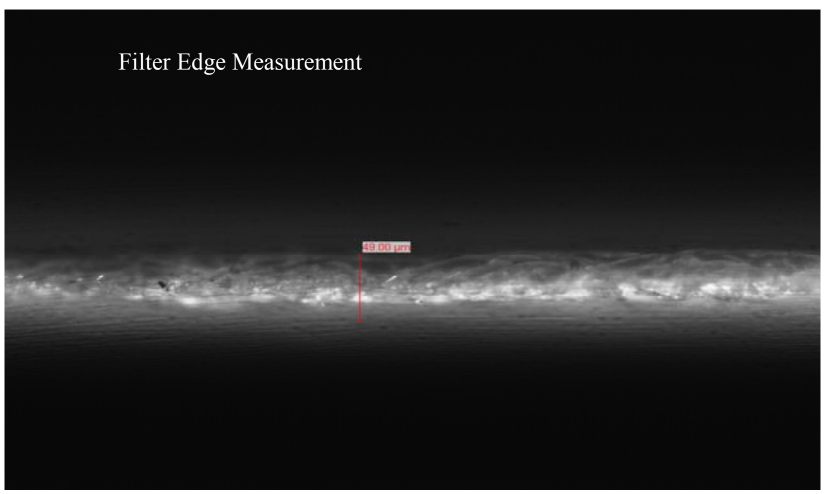
Sample ID	Filter Weight (g)	Planchet Gross Weight (g)	Planchet Tare Weight (g)	Net Sample Weight (g)
02-3047	0.1112	7.7116	7.7102	0.0014
02-3048	0.1092	7.6891	7.6884	0.0007
02-3049	0.1016	7.7208	7.7197	0.0011
02-3050	0.1046	7.6825	7.6821	0.0004
02-3051	0.1066	7.6917	7.6911	0.0006
02-3052	0.1005	7.6641	7.6631	0.0010
02-3053	0.1003	7.7008	7.6989	0.0019
02-3054	0.1067	7.645	7.6442	0.0008
02-3055	0.1024	7.7026	7.7006	0.0020
02-3056	0.0997	7.7184	7.7178	0.0006
02-3090	0.1064	7.6615	7.6607	0.0008
02-3091	0.0945	7.6835	7.6826	0.0009
02-3092	0.1032	7.7379	7.7349	0.0030
02-3093	0.1084	7.6925	7.6912	0.0013
02-3094	0.1166	7.6561	7.6553	0.0008
02-3095	0.0979	7.704	7.7033	0.0007
02-3096	0.1000	7.7050	7.7040	0.0010
02-3097	0.0935	7.6708	7.67	0.0008
02-3098	0.1047	7.7167	7.7158	0.0009
02-3099	0.1117	7.6676	7.6672	0.0004
Average	*0.1040*	*7.6911*	*7.6901*	*0.0011*
StDev	*0.0058*	*0.0246*	*0.0243*	*0.0006*
Max	*0.1166*	*7.7379*	*7.7349*	*0.0030*
Min	*0.0935*	*7.6450*	*7.6442*	*0.0004*

Table B.3 Mass Results of Digested Filter Blanks

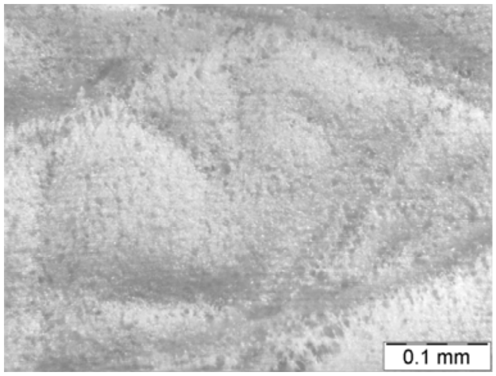
Sample ID	Filter Weight (g)	Planchet Gross Weight (g)	Planchet Tare Weight (g)	Net Sample Weight (g)
Blank 1	0.1083	7.6735	7.6728	0.0007
Blank 2	0.1082	7.7156	7.7144	0.0012
Blank 3	0.1054	7.6897	7.6872	0.0025
Blank 4	0.1067	7.6618	7.6619	-0.0001
Blank 5	0.1100	7.6902	7.6906	-0.0004
Blank 6	0.1143	7.7128	7.7121	0.0007
Average	*0.1088*	*7.6906*	*7.6898*	*0.0008*
StDev	*0.0031*	*0.0212*	*0.0209*	*0.0010*
Max	*0.1143*	*7.7156*	*7.7144*	*0.0025*
Min	*0.1054*	*7.6618*	*7.6619*	*-0.0004*

Appendix C—Microscopy Images

The reflected light microscopy images shown here include an image of a new unused filter edge with a typical thickness of 49 µm. Also included are images of 1) a clean unused filter, 2) light loading, 3) medium loading, and 4) heavy loading on sample filters. These images include 1) a trio of filter images including an extended focal image, a digital photo, and 3D rendering; and 2) particle size measurements from extended focal images. The images between the trio image sets and the particle size measurements are from different sample filters. Particle sizes on the surface were generally from 1 to 10 µm with the typical size ranging between 2 and 6 µm. The dark color on the 3D renderings represents the color of the sample surface at the edge of the image.

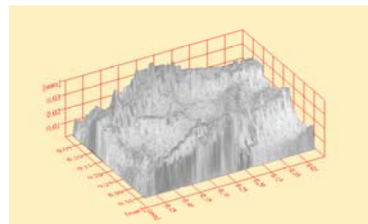


Clean Filter
Unused Filter 1



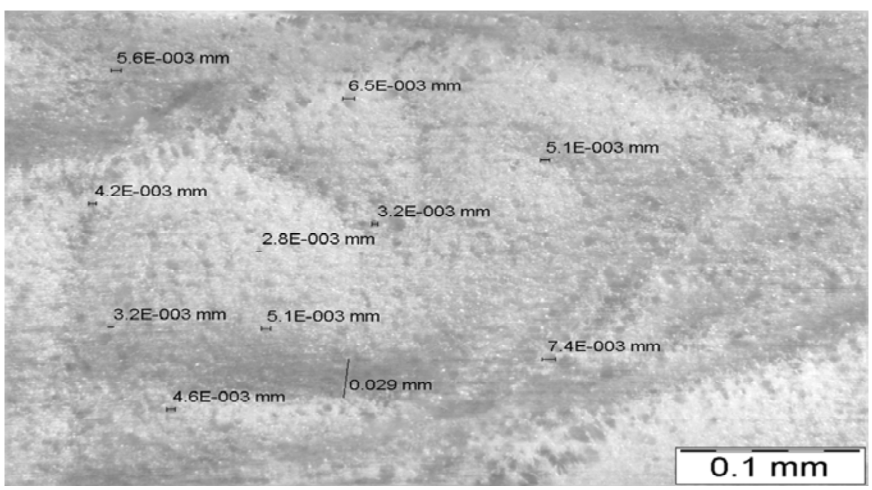
Extended Focal Image

Photograph

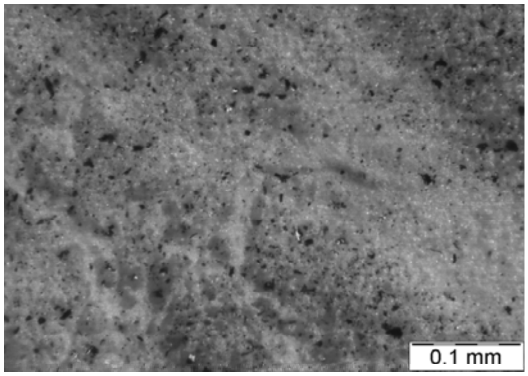


3D Rendering

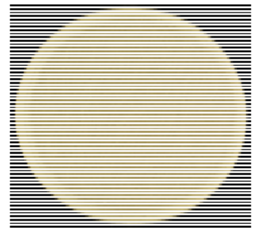
Clean Filter
Extended Focal Image - Particle Size
Unused Filter 1



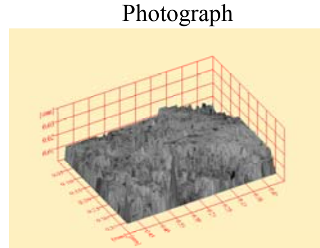
Light Particulate Loading
Sample 03-446 Batch #32 1



Extended Focal Image



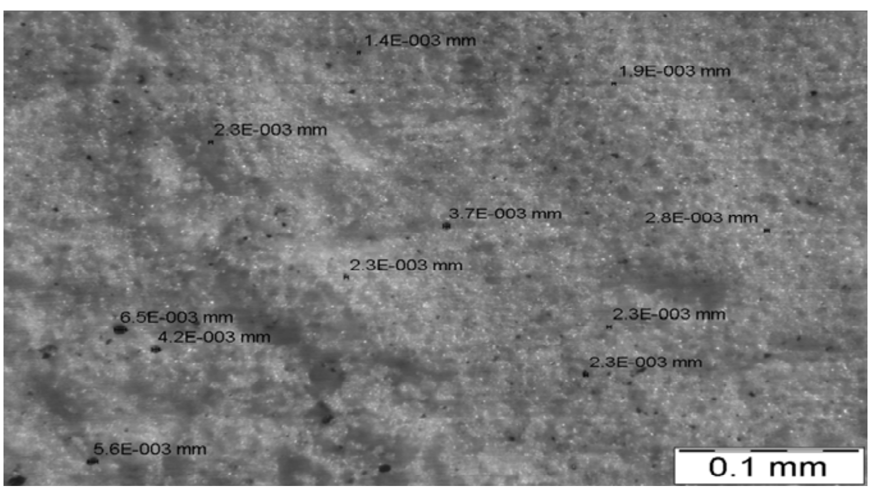
Photograph



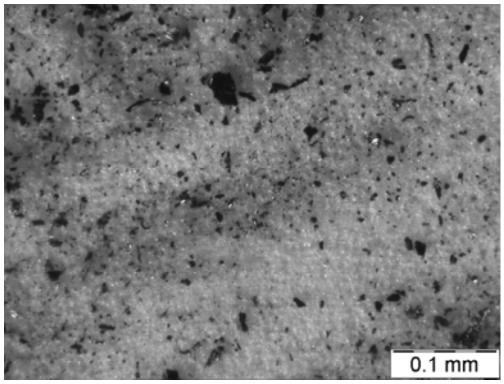
3D Rendering

Light Particulate Loading
Extended Focal Image - Particle Size

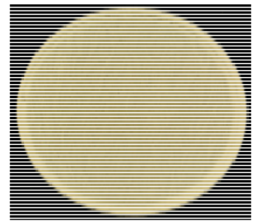
Filter 03-801 Batch 6 1



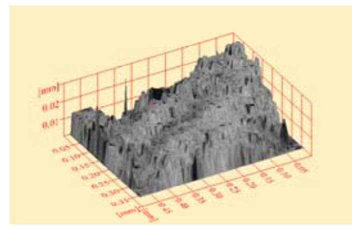
Medium Particulate Loading
Sample 03-1149 Batch #19 1



Extended Focal Image



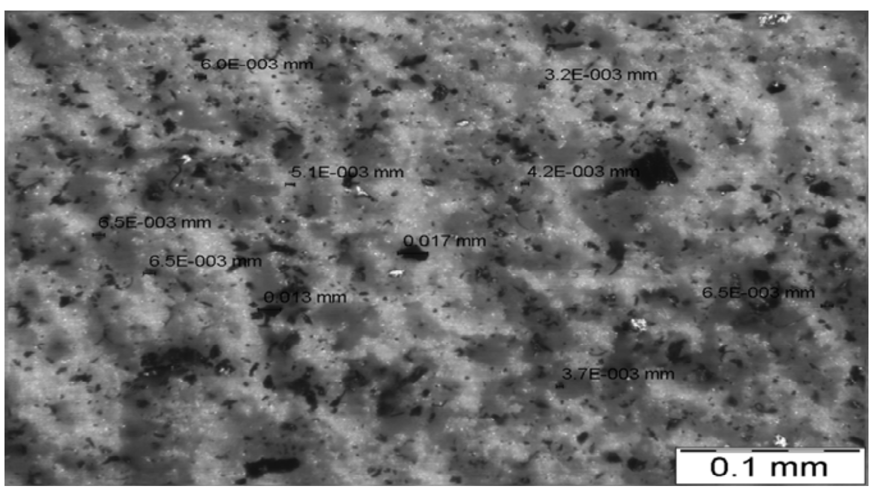
Photograph



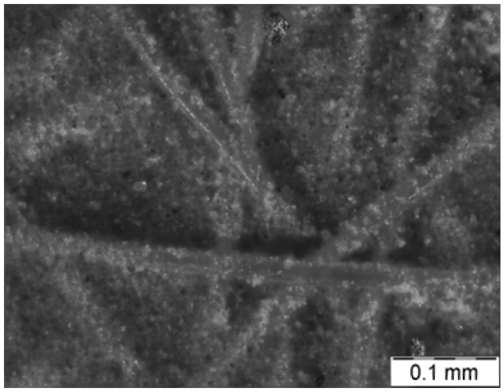
3D Rendering

Medium Particulate Loading
Extended Focal Image – Particle Size

Filter 03-891 Batch 10 1

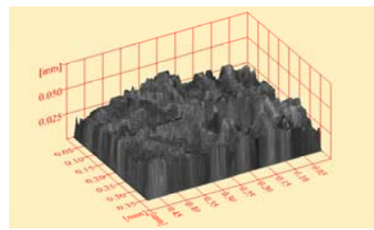


Heavy Particulate Loading
Sample 04-00224 Batch #30 3



Extended Focal Image

Photograph



3D Rendering

Heavy Particulate Loading
Extended Focal Image- Particle Size
Filter 04-203 Batch 32 4