



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

August 2008



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

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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 a correction factor for the self absorption of activity of particulate radioactive air samples. More recently, an effort was made to evaluate the current particulate radioactive air sample filters (Versapor® 3000) used at PNNL for self-absorption effects. There were two methods used in the study: 1) to compare the radioactivity concentration by direct gas-flow proportional counting of the filter to the results obtained after acid digestion of the filter and counting again by gas-flow proportional detection and 2) to evaluate sample filters by high resolution visual/infrared microscopy to determine the depth of material loading on or in the filter fiber material. Sixty samples were selected from the archive for acid digestion in the first method, and about 30 samples were selected for high resolution visual/infrared microscopy. Mass-loading effects were also considered. From the sample filter analysis, a large error is associated with the average self absorption factor; however, when the data are compared directly one-to-one, statistically, there appears to be good correlation between the two analytical methods. The mass loading of filters evaluated was  $<0.2 \text{ mg cm}^{-2}$  and was also compared against other published results. The microscopy analysis shows that the sample material remains on the top of the filter paper and does not imbed into the filter media. Results of the microscopy evaluation led to the conclusion that there is not a mechanism for significant self absorption. The overall conclusion is that self-absorption is not a significant factor in the analysis of filters used at PNNL for radioactive air stack sampling of radionuclide particulates and that an applied correction factor is conservative in determining overall sample activity. A new self-absorption factor of 1.0 is recommended.



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## 1.0 Introduction

Since the mid-1980s, Pacific Northwest National Laboratory (PNNL) has used a value of 0.85 as a correction factor to account for the self absorption of activity on particulate radioactive air samples (Higby 1984). Over the past 4 years, the Effluent Management group coordinated an effort to evaluate the current particulate radioactive air sample filters (the Versapor® 3000)<sup>(a)</sup> used at PNNL for self-absorption effects. The two methods employed were 1) a comparison of analyzed filters that were then acid digested and analyzed again and 2) high-resolution microscopy. Mass loading was also considered as a part of the filter analyses conducted. Results are discussed, and a new correction factor is reported.

The Versapor® 3000 filters are composed of an acrylic copolymer on a nylon substrate with a pore size of 3 µm and a typical thickness of 190 µm. Versapor® 3000 filters have a 47-mm diameter and when installed have a 41-mm active diameter (13.2 cm<sup>2</sup> active sampling area). They are installed on fixed-head radioactive-air-stack samplers for 2 weeks. Sample flow rates generally range from 28 to 85 L min<sup>-1</sup>, which is on the low end of the rated flow rate of 900 L min<sup>-1</sup> (52 L min<sup>-1</sup> cm<sup>-2</sup>). At the end of the sampling period, the sample filter is removed and sent in for gross alpha and gross beta analysis.

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 particles over face velocities between 0.5 and 2.0 m s<sup>-1</sup>. This particle size was chosen because it was closest to the 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 results of interest yielding a self-absorption factor of 85% ± 9%. Under general conditions, this result would verify that concentrations of airborne radionuclides would not be underestimated by collection and analysis. Considering all of the data tested in the 0.66- to 3.07-µm range, a self-absorption factor of 88% ± 6% is obtained (Higby 1984).

Other studies by Haung et al. (2002), Luetzelschwab et al. (2000), and Stevens and Toureau (1963) have shown that, depending on the type of filter used, dust loading on the filter may not impair the sample results provided the deposited layer remains thin ( $\leq 0.1 \text{ mg cm}^{-2}$  and potentially up to  $10 \text{ mg cm}^{-2}$ ). However, degradation in sample results has been shown for sample loadings as little as  $\sim 0.4 \text{ mg cm}^{-2}$  and upwards of 40% self absorption when the particulate matter is  $3.3 \text{ mg cm}^{-2}$ .

## 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 extracting the radioactive material using acid digestion and conducting a second count. The assumption was that digesting the sample filter would recover all radioactivity, even that embedded in the filter, and the self-

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

absorption factor could be determined by taking the result of the filter count and dividing by the result of the digested count. An average self-absorption factor would be determined from the results.

The selected sample filters were counted by gas-flow proportional counting as if they had just been collected. 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 by gas-flow proportional counting. The results from both counting processes were compared.

## 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. New filters were weighed in an effort to determine an average filter weight that could be used to establish a probable sample mass on each filter.

## 2.3 High-Resolution Visual/Infrared Microscopy

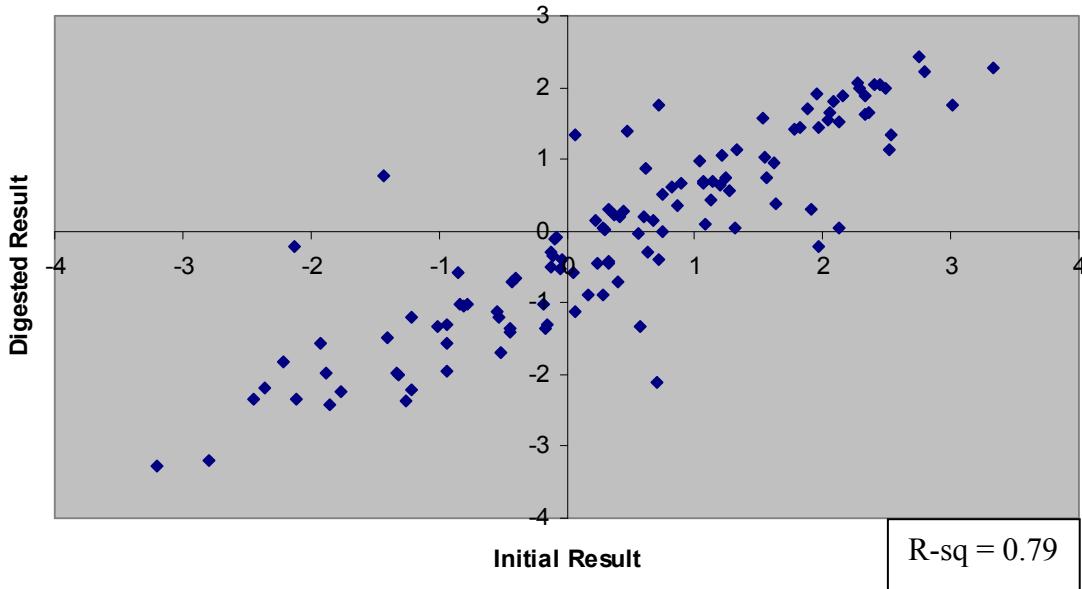
In 2005, 30 samples were selected from the archive for evaluation using high-resolution visual/infrared microscopy. The filters had a varying degree of sample material loading and were compared to a set of unused new filters. Measurements were made in the micrometer range for these filters. The filters were photographed, and images of the particulate loading were made in both 2-D and 3-D renderings for evaluation.

# 3.0 Results

## 3.1 Sample Filter Count and Acid-Digest Count Comparison

Appendix A shows the results of the 60 samples analyzed; only one sample set failed to have all data results exceed the critical level ( $L_c$ ) limiting condition and was therefore excluded. Results indicate that the combination of alpha and beta results yielded a self-absorption factor of  $2.01 \pm 3.66$  ( $2-\sigma$ ) and a median value of 1.53. Thus, on average, more activity was measured by directly counting the filters than by acid digestion. The minimum self-absorption factor was 0.04, and the maximum self-absorption factor was 16.61. The results showed a wide variation encompassing the value of 1 where no difference can be detected between the two analysis methods. Therefore, the data were subjected to an additional statistical review. When the natural logs of all of the results are plotted against each other (Figure 1), the relationship appears to be linear with an R-squared value of 0.79, indicating a high level of agreement between the initial set of data (filter analysis) and the final set of data (digested analysis).

Comparing the analytical results of the two methods led to a conclusion that there is no self-absorption effect if the digested samples provided an unbiased result of true activity. This lack of self-absorption was further investigated by physically viewing other archived filters to see if the particles rested on top of the filters or if the particles could be buried within the fibers.



**Figure 1.** Natural Log Plot of the Filter Data

### 3.2 Mass Loading Evaluation

The average weight after weighing 50 new filters was 0.1 g. However, the  $2\sigma$  error was greater than 15% and the ratio of the maximum to minimum weighed filter was 1.39. These effects raised concern as to whether the weight of the sample material could be reliably estimated without having pre-weighed each filter before use. Therefore, this factor is not included in the results.

However, the sample weights on the planchets were measured and are very small—less than 2.5 mg per sample. This results in a mass loading of  $<0.2 \text{ mg cm}^{-2}$  on the active area of the sample filters.

### 3.3 High Resolution Visual/Infrared Microscopy

Results from the high-resolution microscopy measurements show that the particulate loading rests on the top of the filter fiber media and does not imbed deeply into the filter. The filter fiber is on the order of 30  $\mu\text{m}$  in diameter. Particulate sizes were generally from 1 to 10  $\mu\text{m}$  with the typical size being from 2 to 6  $\mu\text{m}$ . A series of plots are shown in Appendix B. The 3-D rendering allows the viewer to look within the filter and indicates little particulate penetration.

## 4.0 Conclusions

From the sample filter analysis, a large error is associated with the average self absorption factor; however, when the data are compared directly one to one, statistically, there appears to be good correlation between the two analytical methods. Issues that influence the data results include the sensitivity of the instruments used, the filter paper variations, and detector efficiencies. The overall conclusion is that self absorption is insignificant for this filter media.

The Higby (1984) study concluded that a self-absorption factor of 10 to 15% for sample filters would not underestimate the concentration of radioactivity on the filter for particulates sizes from 0.66 to 3.07  $\mu\text{m}$  and a face velocity from 0.5 to 2.0  $\text{m s}^{-1}$ . However, Higby (1984) also found that glass fiber filters tend to be surface collectors for larger particle sizes, which is similar to results obtained by Stevens and Toureau (1963). The data in this study show particle sizes typically in the 2- to 6- $\mu\text{m}$  range and confirm these previous studies that the particles tend to collect on the surface of the filter.

Mass-loading effects are in general agreement with those previously reported by Haung et al. (2002). The low mass loading of  $<0.2 \text{ mg cm}^{-2}$  for this study is sufficiently small enough to conclude that self absorption is of little concern for this filter type and associated use.

The high resolution visual/infrared microscopy analysis shows that the sample material remains on the top of the filter paper and does not imbed into the filter media. Thus, the conclusion from physically viewing the filters is that there is not a mechanism for significant self absorption.

Taking the two methods together in addition to the mass-loading information, the overall conclusion is that self-absorption is not a significant factor in the analysis of filters used by PNNL for stack sampling of radioactive particles. Therefore, a new self-absorption correction factor of 1.0 (i.e., there is no self-absorption) is recommended for PNNL as well as the continued use of the Versapor® 3000 filter and analysis by gas-flow proportional counting.

## 5.0 References

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

**Table A1.** Results from Filter Analysis

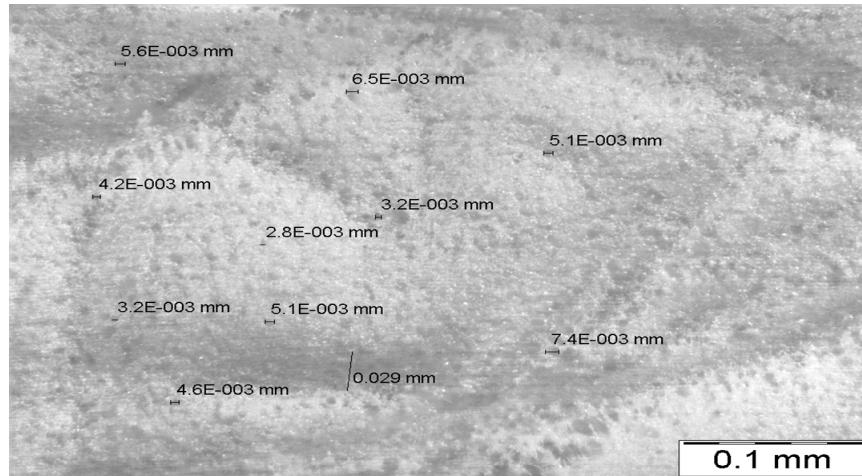
Sample Id.	Alpha Efficiency		Beta Efficiency		Background (cpm)		Variance		MDA (pCi)		Bckgd (pCi)		Alpha		Beta		Alpha Efficiency		Beta Efficiency		Background (cpm)		Variance		MDA (pCi)		Bckgd (pCi)		Alpha		Beta								
	Eff.	+/-	Eff.	+/-	Alpha	Beta	Alpha	Beta	Alpha	Beta	Alpha	Beta	pCi/sample	+/- s	Lc,pCi	>,< Lc	Lc,pCi	>,< Lc	Eff.	+/-	Alpha	Beta	Alpha	Beta	Alpha	Beta	pCi/sample	+/- s	Lc,pCi	>,< Lc	Lc,pCi	>,< Lc	Alpha	Beta					
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	>Lc	2.04	+/- 0.20	0.10	0.33	+/- 0.004	0.52	+/- 0.007	0.15	0.21	0.20	0.15	1.26	+/- 0.14	0.05	>Lc	5.75	+/- 0.28	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	>Lc	1.07	+/- 0.15	0.09	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	0.37	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	>Lc	0.08	+/- 0.11	0.09	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	0.37	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	>Lc	3.07	+/- 0.26	0.10	>Lc	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	0.38	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	>Lc	1.59	+/- 0.18	0.09	>Lc	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	0.37	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	>Lc	0.12	+/- 0.13	0.10	>Lc	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	0.37	0.005	0.58	0.017	0.08	2.05	0.0002	0.0009	0.08	0.24	0.09	1.62	0.09	+/- 0.05	>Lc	0.58	+/- 0.16	0.12	>Lc	0.33	+/- 0.004	0.53	+/- 0.007	0.08	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	0.37	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	>Lc	0.45	+/- 0.13	0.10	>Lc	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	0.38	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	>Lc	1.85	+/- 0.19	0.09	>Lc	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	0.38	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	>Lc	0.43	+/- 0.12	0.09	>Lc	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	0.37	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	>Lc	0.39	+/- 0.13	0.09	>Lc	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	0.37	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	>Lc	0.24	+/- 0.12	0.09	>Lc	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	0.37	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	>Lc	0.25	+/- 0.12	0.09	>Lc	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	0.37	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	>Lc	0.11	+/- 0.12	0.10	>Lc	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	0.38	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	>Lc	2.84	+/- 0.24	0.10	>Lc	0.33	+/- 0.004	0.53	+/- 0.007	0.10	1.57	0.0001	0.0002	0.010	0.23	0.13	1.35	1.34	+/- 0.14	+/- 0.05	>Lc	2.68	+/- 0.20	0.11	>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	>Lc	1.51	+/- 0.18	0.10	>Lc	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	0.37	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	>Lc	1.39	+/- 0.19	0.11	>Lc	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	0.37	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	>Lc	3.34	+/- 0.20	0.10	>Lc	0.33	+/- 0.004	0.51	+/- 0.007	0.03	1.32	0.0002	0.0002	0.05	0.21	0.03	1.68	1.68	+/- 0.04	+/- 0.02	>Lc	2.89	+/- 0.21	0.11	>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	>Lc	0.30	+/- 0.12	0.09	>Lc	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	0.38	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	>Lc	11.01	+/- 0.70	0.09	>Lc	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	0.37	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	>Lc	0.43	+/- 0.13	0.09	>Lc	0.33	+/- 0.004	0.52	+/- 0.007	0.13	1.20	0.0001	0.0002	0.09	0.17	0.07	1.07	0.17	+/- 0.07	+/- 0.02	>Lc	0.36	+/- 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	+/- 0.17	>Lc	16.47	+/- 1.00	0.08	>Lc	0.33	+/- 0.004	0.51	+/- 0.007	0.20	1.17	0.0002	0.0002	0.11	0.17	0.07	1.03	3.09	+/- 0.21	+/- 0.06	>Lc	9.16	+/- 0.38	0.09	>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	0.66	+/- 0.10	>L																								



## Appendix B—High Resolution VIS/IR Shots

### Unused Filter 1

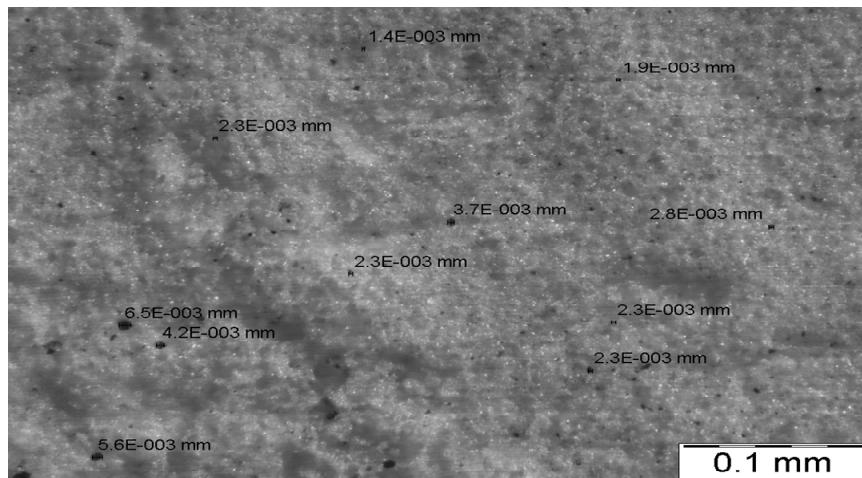
Clean filter – 20 X Extended Focal Image



12/20/2006

### Filter 03-801 Batch 6 1 Particle Size

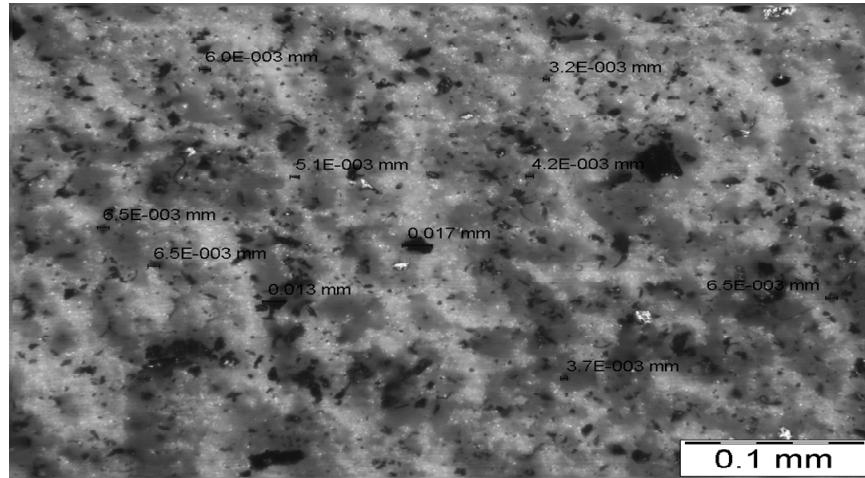
Low Dirty Level (Dusty Filter) – 20 X Extended Focal Image



12/20/2006

## Filter 03-891 Batch 10 1 Particle Size

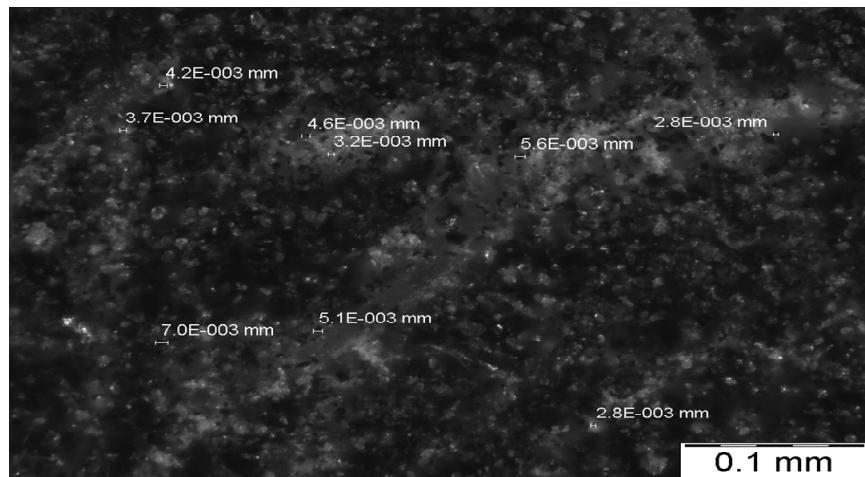
Medium Dirty Level (Dirty Filter) – 20 X Extended Focal Image



12/20/2006

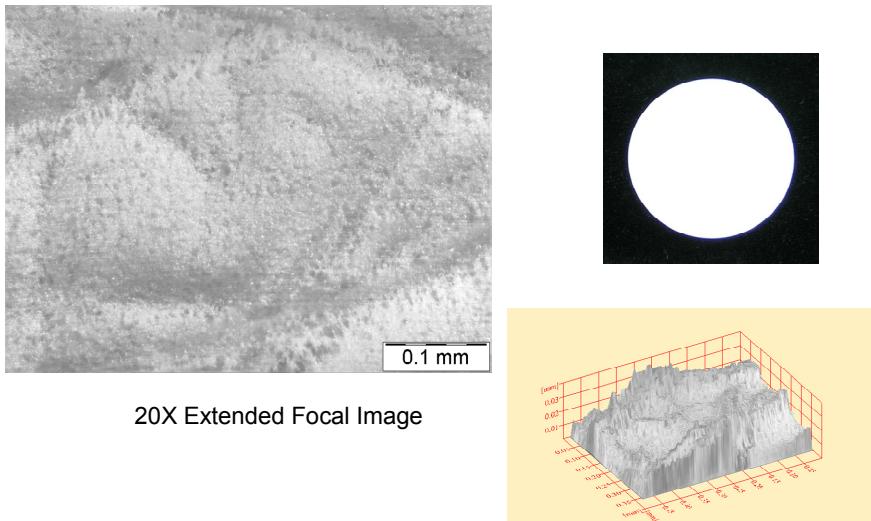
## Filter 04-203 Batch 32 4 Particle Size

High Dirt Level (Very Dirty) – 20 X Extended Focal Image



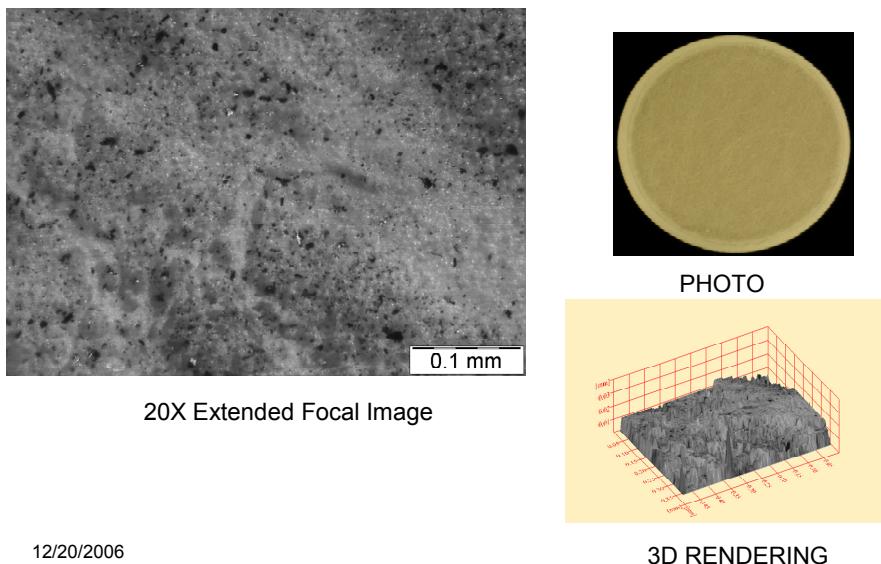
12/20/2006

## *Unused Filter 1 - Clean Filter*



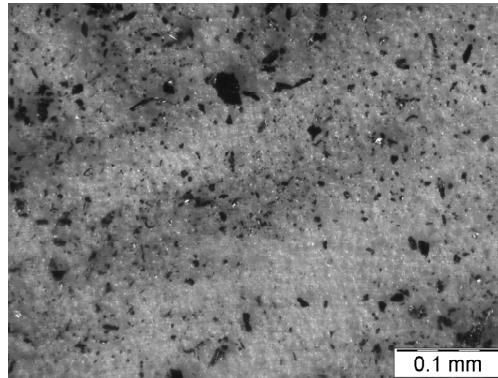
12/20/2006

## *Dusty Filter-Sample 03-446 Batch #32 1*

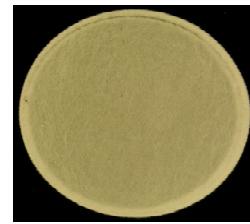


12/20/2006

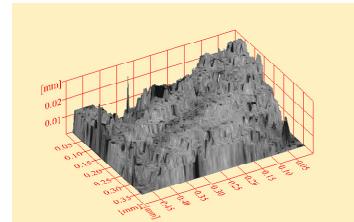
*Dirty Filter-Sample 03-1149 Batch #19 1*



20X Extended Focal Image



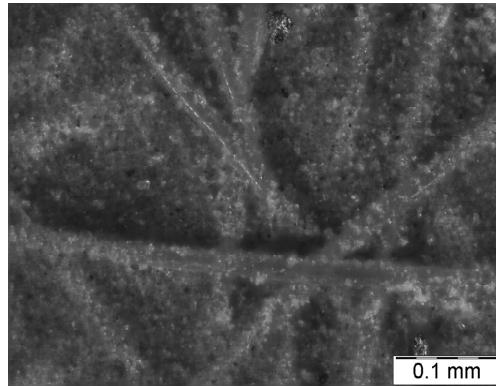
Photograph



3D Rendering

12/20/2006

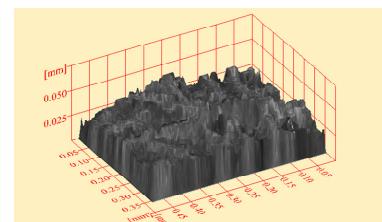
*Very Dirty -Sample 04-00224 Batch #30 3*



20X Extended Focal Image



Photograph



3D Rendering

12/20/2006