Final Report on the World Trade Center (WTC) Dust Screening Method Study

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ACRONYMS:

ATSDR	Agency for Toxic Substances and Disease Registry
AVG	Average
COPC	Contaminant of Potential Concern
EPA	U.S Environmental Protection Agency
EPIC	Environmental Photographic Interpretation Center
ERT	U S EPA's Emergency Response Team
HEPA	High Efficiency Particulate Air
LI	Long Island
MMVF	Man Made Vitreous Fibers
MQO	Measurement Quality Objective
ND	Non-Detect
NEIC	U S EPA's National Enforcement Investigations Center
NERL	U S EPA's National Exposure Research Laboratory
NJ	New Jersey
NYCDOMH	New York City Department of Health and Mental Hygiene
ORD	U S. EPA's Office of Research and Development
PM	Particulate Matter
$PM_{2.5}$	Particulate Matter Smaller than 2 5 microns
QAPP	Quality Assurance Project Plan
SD	Standard Deviation
SEM	Scanning Electron Microscopy
USGS	U S Geological Survey
WTC	World Trade Center

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EXECUTIVE SUMMARY

The September 11, 2001 attack on the World Trade Center (WTC) covered a large area with dust and debris To assist in determining if residual contamination exists in the indoor environment, the U S Environmental Protection Agency (EPA) initiated a study to sample indoor environments that may have been impacted by the WTC collapse A critical component of this study is determining whether sampled dust originated from the collapse of the WTC or instead is urban dust originating from other sources This report describes work performed to develop and validate a screening method for indoor dust that can be used to determine whether dust sampled is from the collapse of the World Trade Center towers

Dispersion models, monitoring, photos, interviews, and satellite data were reviewed to discern areas that were likely impacted by WTC emissions and those that were not (US EPA 2002, 2004) A total of 117 samples were collected from both impacted and non-impacted areas A subset of these samples were analyzed by EPA's National Exposure Research Laboratory (NERL) and National Enforcement Investigations Center (NEIC), and United States Geological Survey (USGS) to evaluate the slag wool levels in the dust and develop an analytical method The analytical method that was developed screens for three materials that are believed to be present in large quantities in WTC dusts slag wool, elements of concrete, and gypsum This method involves the use of Scanning Electron Microscopy (SEM) to determine the quantity of each of the materials present

Five commercial laboratories, along with the three above listed government labs, were recruited to test the screening method Thirty-two dust samples, consisting of both confirmed background samples and a confirmed background dust spiked with varying amounts of confirmed WTC dust, were sent out to the eight labs The labs were provided the samples "blind" They did not know which samples were background dust and which were non-impacted dust spiked with WTC dust In addition to the thirty-two samples, one of the five commercial laboratories also received twenty-eight background samples to increase the available data characterizing background locations

The data reported by these laboratories indicated the following

1) Five of the eight laboratories were able to reasonably measure the slag wool concentrations in non-impacted dust spiked with confirmed WTC dust

2) A substantial amount of variability in slag wool measurements was found within labs and between labs Despite this variability, slag wool measurements appear to be sensitive enough to distinguish WTC dust (defined as 4 Albany) spiked at the 10% level from background dust.

3) The levels of gypsum and elements of concrete in the spiked samples were indistinguishable from the levels in the background samples This suggests that, while these components may have been elevated in dust samples collected near the WTC site in September 2001 (as found by USGS in their studies on WTC dust), they are also commonly found in the indoor environment and would not be useful as WTC signature

components

4) Analysis of samples during method development showed elevated levels of slag wool in samples from several impacted locations compared to slag wool levels measured at background locations

I. INTRODUCTION AND BACKGROUND

The objective of this effort was to develop and validate a means of determining whether dust sampled as part of EPA's planned sampling program contains residual contamination attributable to the collapse of the WTC towers The tested screening method is a critical component of the sampling program as it will be used for two primary purposes: 1) to determine the geographic extent of the dust remaining from the collapse impact, and 2) along with the results from contaminants of potential concern (COPC) testing, to determine the need for a clean-up of the sampled areas

The USGS has published two reports that provided the basis for the initial hypothesis that a WTC collapse signature is comprised of three marker components slag wool, gypsum and elements of concrete The first report discusses the analysis and interpretation of indoor and outdoor WTC dust samples collected near Ground Zero, days and weeks after September 11, 2001 (Meeker et al , 2005). From this work, we see that the WTC dust samples are dominated by gypsum, concrete, and man-made vitreous fibers (MMVF), mainly slag wool It is on the basis of these key results that gypsum, elements of concrete, and slag wool were identified as candidates for a WTC signature The second report discusses the analysis of EPA supplied samples taken from several indoor locations well outside of the WTC impacted area (background) These samples were taken between September of 2004 and April of 2005 Slag wool was absent from many of these background samples, but Lowers et al (2005a) state that the samples do have gypsum present, which they speculate might be due to the presence of wall board in the sampled apartments Because of the lack of slag wool in these samples, USGS concluded that these samples did not contain WTC dust USGS also concluded that perhaps slag wool is the single most critical of the three WTC dust constituents when distinguishing WTC dust from other common dusts

Other studies also identified MMVF and gypsum as predominant components of WTC dust In a study of air and settled dust quality in apartments in Lower Manhattan, the Agency for Toxic Substances and Disease Registry (ATSDR) and the New York City Department of Health and Mental Hygiene (NYCDOMH) found significantly more MMVF and gypsum in samples taken from Lower Manhattan apartments as compared to samples taken from apartments in areas above 59th Street (NYCDOMH/ATSDR, 2002) They also concluded that gypsum was seen at a higher percentage level in the Lower Manhattan dust samples as compared to the comparison area samples In a comprehensive study of the composition of settled dust in the Deutsche Bank building at 130 Liberty Street, R J Lee identified numerous hazardous contaminants that were present in the dust at levels much higher than in background office buildings, and among those substances identified in their "WTC signature" were mineral wool and gypsum (R J Lee, 2004)

If the WTC building collapse signature components of slag wool, gypsum, and elements of concrete are not present, then one could conclude that WTC building collapse dust is not present However, since these components might be present in typical New York City dust, and as slag wool is a component of insulating materials in currently constructed buildings, it is possible that a test might show them to be present even though WTC dust never impacted the sampled area A 'screening test' will, by its design, result in some fraction of such false positives (i e a location without residual WTC dust that tests positive for the above components) However, an appropriate 'screening test' would result in very few, if any, false negatives (i e a location with residual WTC dust that tests negative for the above components)

II. METHOD DEVELOPMENT

Sample Collection

EPA acquired 117 dust samples during the time period of September 2004 to April 2005 Twenty-one 'impacted' samples were taken by the EPA at two buildings that were part of the Deutsche Bank complex located at 130 Liberty Street and 4 Albany Street Both affected buildings were uninhabited and slated for demolition Fifty samples were taken from locations well beyond the impacted zone (based on modeling, monitoring and photo analysis, these samples are considered to be 'background' dust) Forty-six samples were taken from locations that were possibly impacted, but were a bit farther from the WTC site than the known 'impacted' samples None of these forty-six samples were used in the method validation study, but several were evaluated during both the method/protocol development phase and post-study In addition, one impacted sample was obtained from the USGS This sample was a composite sample of outdoor and indoor WTC dust collected in September of 2001

A standard method utilizing a High Efficiency Particulate Air (HEPA) vacuum collector was used by EPA to collect most bulk dust samples Information on this method is provided in the Quality Assurance Plan (QAPP) for this study (Appendix A) Some bulk dust samples were collected from residential and commercial vacuum cleaner bags

Modeling and satellite photography were used to determine sampling locations for the collection of the 117 samples Figures 1a and 1b (EPA 2002, EPIC 2004) are examples of modeling and photographic analysis used to distinguish non-impacted or background locations. Figure 1a shows ORD-modeled WTC Plume Dispersion on September 11, 2001 at 12 noon The values indicated by red are hourly $PM_{2.5}$ concentrations (in $\mu g/m^3$) measured at pre-existing NJ and NY State-operated PM monitoring stations in northern New Jersey and New York City Red, orange, and yellow shading represent most likely areas of plume dispersion (red = estimated dilution to 100th to 500th and dark blue = dilution to < one millionth of pollutant concentration at WTC source). As seen in this figure, the plume very rapidly diluted to concentrations less than 1/1000 (which is the yellow area) of the initial source strength at Ground Zero. Figure 1b shows the boundaries of collapse deposition debris as determined by aerial photographs This photograph was taken on September 13, and shows the four areas of "confirmed", "probable", "possible", and "no dust" from the collapse. These areas were used in the determination of strata used in the design for the overall sampling program.

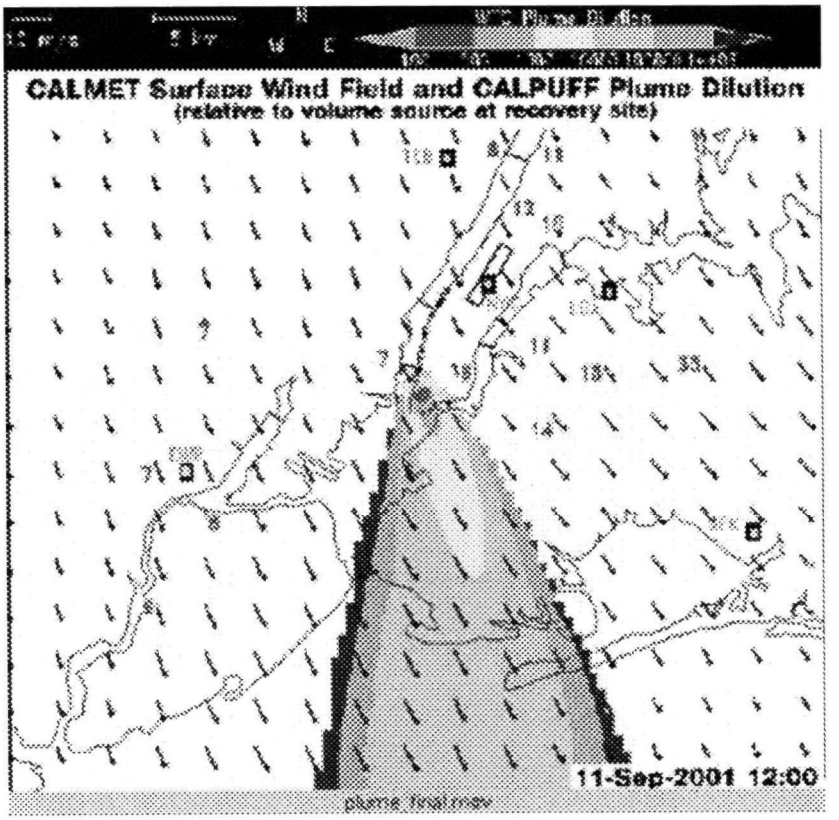


Figure 1a: ORD-modeled WTC Plume Dispersion on September 11, 2001 at 12 noon. (Source: Exposure and Human Health Evaluation of Airborne Pollution from the World Trade Center Disaster (External Review Draft). U.S. Environmental Protection Agency, Washington, D.C., 2002.)

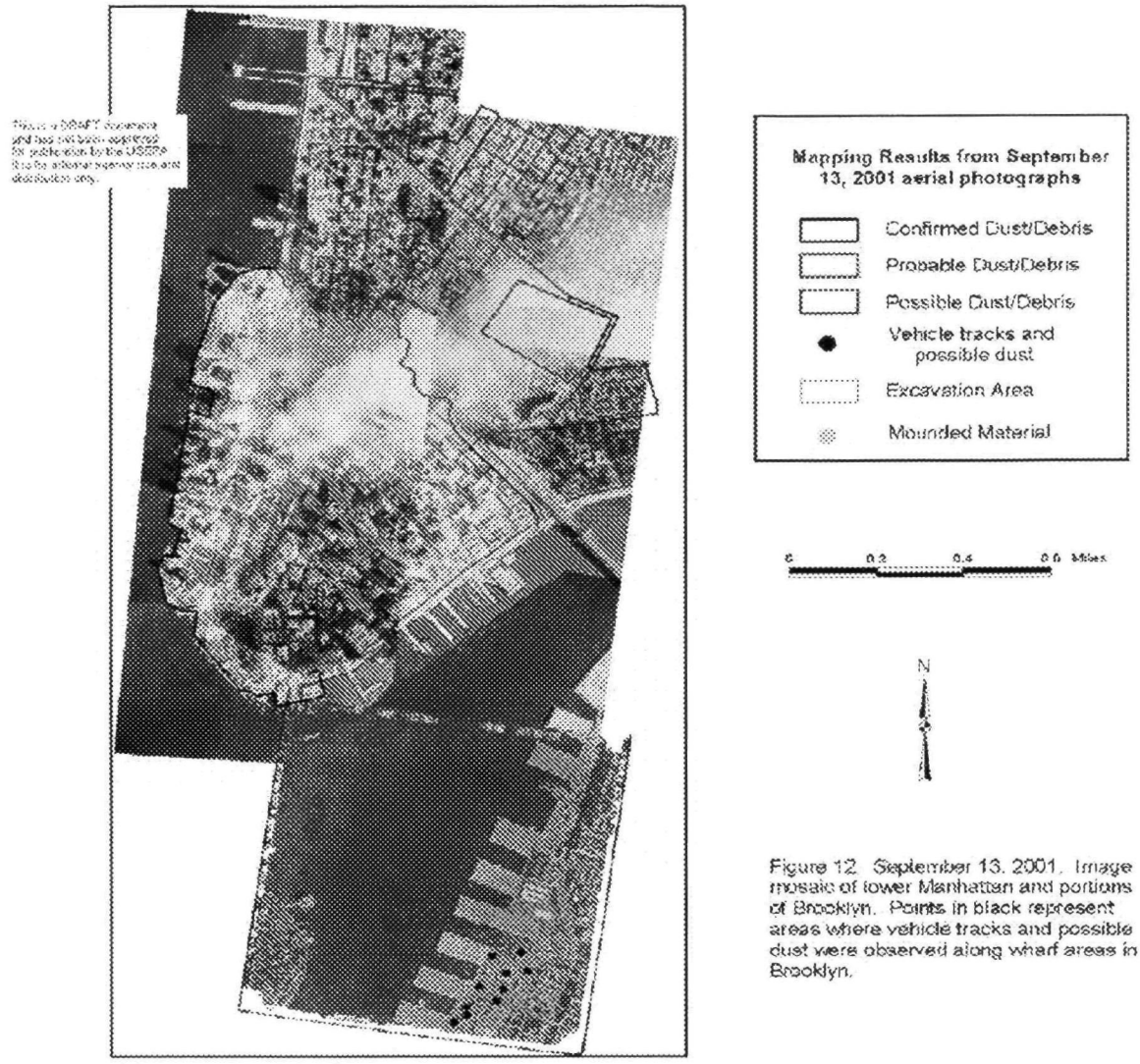


Figure 12. September 13, 2001. Image mosaic of lower Manhattan and portions of Brooklyn. Points in black represent areas where vehicle tracks and possible dust were observed along wharf areas in Brooklyn.

Figure 1b: Display of boundaries of expected deposition based on analysis conducted by EPA's Environmental Photographic Interpretation Center (Updated by EPIC from the figure which appears in EPIC, 2004).

Preliminary Analysis of Collected Samples for Slag Wool
Most of the collected samples were analyzed for slag wool content by the EPA's National Exposure Research Laboratory (NERL) Scanning Electron Microscopy (SEM) Laboratory. This analysis was performed as part of the EPA's development of a protocol for sample preparation and analysis and for preliminary sample characterization. These samples were not analyzed for elements of concrete or gypsum as an analytical method for these components had not yet been developed The data acquired during this method/protocol development effort are presented in Appendix B Caution should be used with these data as it was obtained while the method was being developed Post-study data acquired by NERL are also presented in Appendix C.

In evaluating the method development data acquired by NERL (Appendix B), there appears to be a distinction between samples taken in impacted areas versus background samples Eighteen of the 21 samples from impacted areas had slag wool at concentrations of greater than 100,000 slag wool fibers per gram of dust, with a range of 69,000 to 13,400,000, while all of the samples from background areas had concentrations less than 100,000 fibers/gram, ranging from no slag wool detected (in 12 of 47 samples) to 92,800 fibers/gram of dust

Based on this preliminary work, the USGS, the EPA's Office of Research and Development (ORD), the EPA's National Enforcement Investigations Center (NEIC), and experts five commercial testing laboratories (denoted labs A-H in Appendix E), worked together to develop an analytical method to identify the presence and concentration of the screening constituents (i e. slag wool, gypsum and elements of concrete) in indoor dust This method was reviewed by the WTC Expert Technical Panel's signature subcommittee and is presented in Appendix D The composition of this technical panel can be found at http //www epa.gov/wtc/panel

III. METHOD VALIDATION STUDY

Study Design
The basis for the WTC dust screening method discussed above is as follows if a unit has been impacted, those materials that are found in WTC dust will be found in the dust collected from the unit The materials under consideration are: 1) slag wool, 2) elements consistent with concrete and 3) gypsum. The study described herein was intended to validate the WTC dust screening method by demonstrating the following things

1) that the above described materials are reasonable markers for WTC dust (by showing that these markers distinguish WTC-laden dust from background dust),
2) that WTC dust at a diluted concentration can be distinguished from background, and
3) that the analytical method works well enough and is able to be carried out by enough analytical laboratories to 1) evaluate the above materials as markers and 2) distinguish WTC dust from background dust

The first of these three objectives was partially addressed in method development work, which focused on slag wool As described in the previous section, slag wool was found to be elevated in locations deemed "impacted", while slag wool was not detected or detected at low concentrations in "background" areas

Five independent laboratories and three government laboratories participated in this method validation phase One government laboratory analyzed only a small portion of the samples, but this lab was critical in the method development Each laboratory attended a two day session during which the method was further developed and discussed, and the protocol was adapted to suit each laboratory's equipment.

Following this session, the laboratories received dust samples consisting of both confirmed background samples (10 samples plus duplicates for a total of 20) and confirmed non-impacted dust spiked with varying amounts of confirmed WTC dust (6 spiked samples plus duplicates for a total of 12). Specifically, a sample that was characterized and confirmed as non-impacted (designated in Appendix B as NE Queens maid service) was split, and the splits were spiked at levels of 1, 5, and 10% total mass with two different characterized and confirmed WTC dusts These spiked samples were then homogenized as documented in the QAPP for this study (Appendix A). The two spiking dusts were 1) a composite sample of predominantly outdoor dust collected in September of 2001 by USGS, and 2) dust collected by the U S EPA from the Deutsche Bank building at 4 Albany Street in September of 2004 The 4 Albany Street building borders the south side of the WTC complex. Six spiked samples were prepared for each laboratory, these were split so that each laboratory received 12 spiked samples. Each laboratory also received 10 non-impacted background samples that were also split, resulting in a total of 20 background samples. Thirty-two samples in all were sent for analysis to the eight labs

In addition to the 32 samples, one of the five commercial laboratories also received 28 background samples to increase the available data characterizing background locations.

The labs were provided the 32 samples "blind", they did not know which samples were pure background dust, and which were the spiked dust To ensure sufficient results for spiked samples, the government laboratory that was only able to analyze a small portion of the samples was asked to analyze only the 12 spiked samples Again, they were not told the identity of these samples (Lab C) The labs had five weeks to analyze all samples The final data from all laboratories, including the data for the additional 28 background samples, were reviewed, evaluated and analyzed by the EPA and the EPA's prime contractor This prime contractor's from this analysis is presented in Appendix E

Composition of Spiked Samples

The USGS performed an analysis of the spiked, homogenized samples prior to the samples being sent to the labs The measured levels were in the approximate range for the spiking percent (1, 5, and 10%) based on the undiluted concentration level of each WTC dust and, in all but one case, each percent level was fully distinguishable from the others (Figures 2 and 3) The variability in the measured levels was expected due to the difficulty in homogenizing dusts that have large particle size distributions, and the fact that components of WTC dust will vary within a sample because of the nature of the source Given these difficulties and the measurement results, these dusts were determined to be reasonably homogeneous

As seen in Figures 2 and 3, the level of slag wool differs between the two WTC dusts, with the pure dust that was collected from 4 Albany Street in 2004 more than an order of magnitude lower than the dust collected by the USGS in September of 2001. The pure dust from 4 Albany

Street had slag wool levels at 500,000 fibers/gram of dust versus approximately 11,000,000 fibers/gram of dust for the USGS collected sample. There are likely explanations for this large difference in slag wool levels. The USGS sample was a composite of multiple outdoor samples and one indoor sample taken during September of 2001. The 4 Albany was an indoor sample was taken three years post 9/11 in September of 2004. As this 4 Albany sample was taken exclusively inside of a building, it was not only diluted by three years accumulation of urban background dust, but was also characteristic of dust that had penetrated the shell of a building as opposed to that deposited on the ground outside.

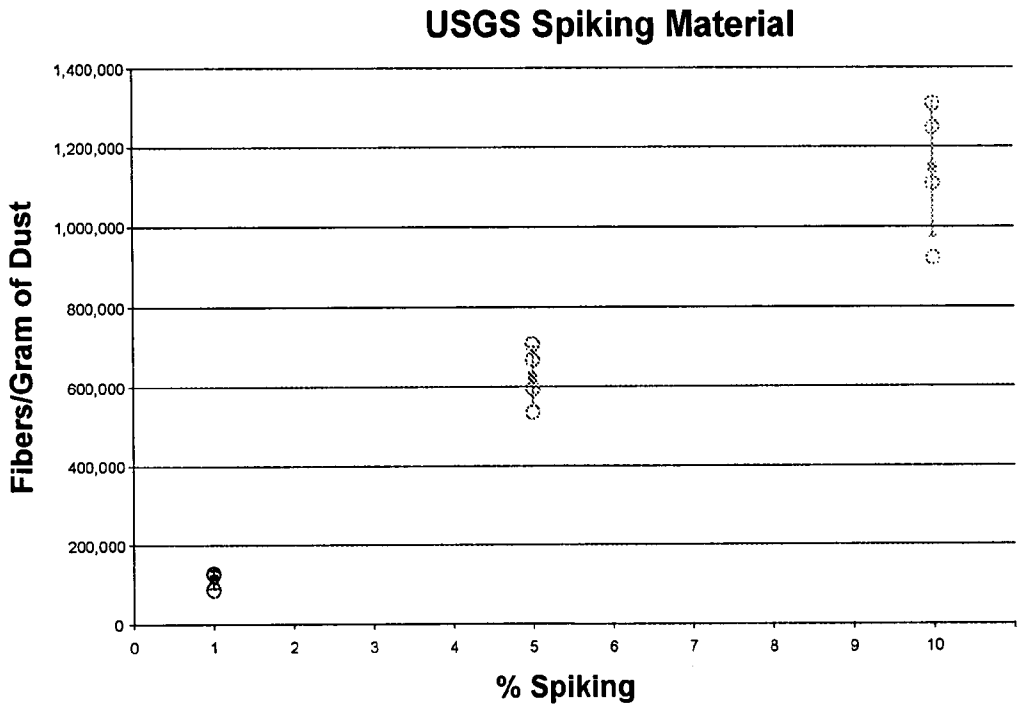


Figure 2: USGS Spiking Material Results. Analysis was conducted by USGS prior to being sent to labs for study. Pure dust averaged approx. 11,000,000 fibers/gram. (Figure provided by USGS)

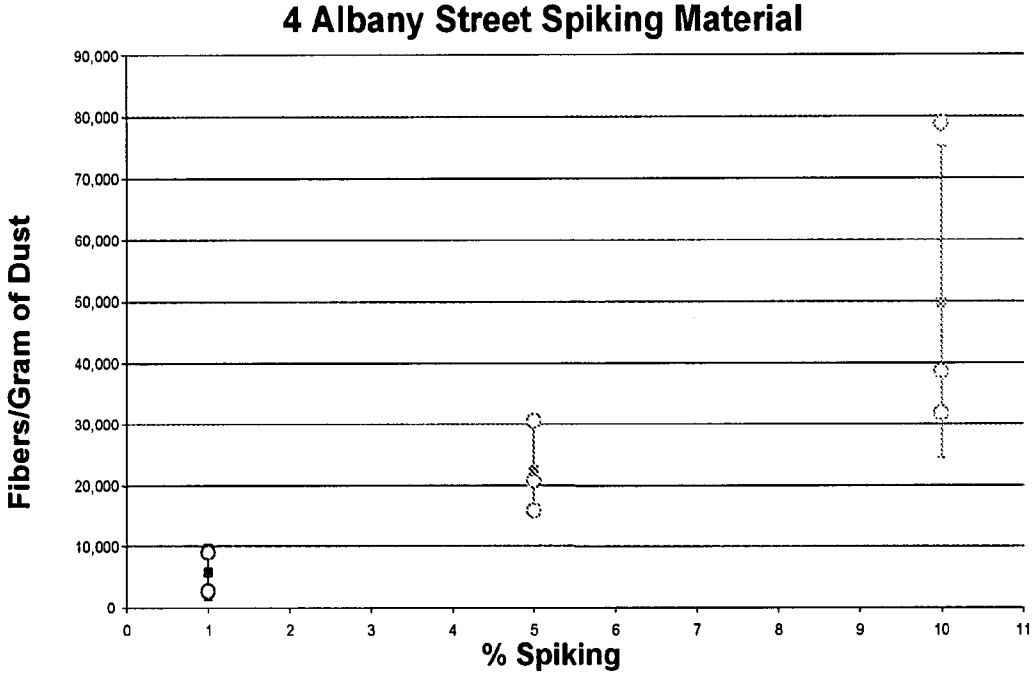


Figure 3: 4 Albany Street Spiking Material Results. Analysis was conducted by USGS prior to being sent to labs. Pure dust averaged approx. 500,000 fibers/gram. (Figure provided by USGS)

IV. RESULTS AND DISCUSSION

Development of Study Results

The final report from the prime contractor with all raw analytical and calibration data can be found in Appendix E. A summary of the study results that includes the data from the 28 additional background samples analyzed by a single commercial laboratory is provided in Table I, as well as Figures 4-7. A map of the origin of the samples analyzed during this study is shown in Figure 8.

All background sample data used in Table I and Figures 4-7 are from the Greater NY City area. Background samples taken in Research Triangle Park, North Carolina are not included as they are not representative of NY City background dust. Data for all background sample results may be found in Tables 3 and 4 of the Versar report in Appendix E. It should be noted that the Research Triangle Park samples show higher slag wool levels than NY City area background samples. This is due to the presence of slag wool containing ceiling tiles in the building sampled. Note also that Table I indicates two average values for background slag wool. These values reflect the inclusion and exclusion of two samples collected in New Jersey (NJ) and Long Island (LI) that were extremely high in slag wool fibers, likely due to their insulation, fireproofing or ceiling tiles. Based on these results it is likely that some false positive results will occur in buildings with slag wool-based ceiling tiles, fireproofing or insulation. .

Three of the commercial laboratories, designated as labs E, F and G, reported analytical data that are not consistent with other five labs Generally, these labs were not able to distinguish differences between the three spiking levels In addition, these labs did not meet the measurement quality objectives (MQOs) for the spiked samples put forth in the QAPP for this study (Appendix A Section A.7.1) Thus, the data from these three labs are not considered in the results presented in Table I and Figures 4-7 The statistical analysis performed to make this determination is presented in Appendix F In addition, Lab H was not considered when determining concrete and gypsum levels as their data were at least two times higher than the sample average without these data (Table I and Figures 6 and 7)

In discussions with the commercial laboratories, it was determined that some labs did not have the personnel or the equipment to perform the required analysis in the given timeframe, thus, data quality became an issue Additionally, labs that had less experience with slag wool analysis felt that a clearer definition, in addition to that provided in the catalog developed by USGS in Lowers et al., 2005b, of slag wool was needed to distinguish it from other mineral wools Finally, labs that were unable to automate the gypsum and concrete analysis expressed their belief that the method was too long and complicated for accurate quantitative dust analysis All laboratory comments will be taken into consideration in when finalizing the protocol

	Background (Greater NY Area)	USGS Spiked (Collected 9/01)	4 Albany Spiked (Collected 9/04)
Slag Wool Average (fibers/g dust)	*AVG ± SD* 35,950 ± 74,300 17,740 ± 15,835* *Range of Samples* ND* - 369,230 ND* - 60,000**	*1%* 94,000 ± 25,740 *5%* 452,510 ± 100,640 *10%* 870,280 ± 310,420	*1%* 17,270 ± 7,880 *5%* 52,510 ± 26,140 *10%* 88,540 ± 18,300
Elements of Concrete (% Area)	*AVG ± SD* 15 6 ± 5 7 *Range of Samples* 6 – 30.5	*1%* 20 ± 6 *5%* 19 ± 7 *10%* 16 ± 2	*1%* 15 ± 1 *5%* 18 ± 4 *10%* 16 ± 3
Gypsum (% Area)	*AVG ± SD* 9 5 ± 3 4 *Range of Samples* 4 – 16 5	*1%* 9 ± 6 *5%* 7 ± 3 *10%* 6 ± 0 5	*1%* 9 ± 4 *5%* 5 ± 2 *10%* 7 ± 2

- **ND=Non Detect (Zero slag wool fibers)
- *Two extremely high values from NJ and LI removed

Table 1: Avg, Standard Dev., and Range of Results for Background and Spiked Samples (Data Summarized from Tables 1, 2, 3 and 4 of Appendix E).

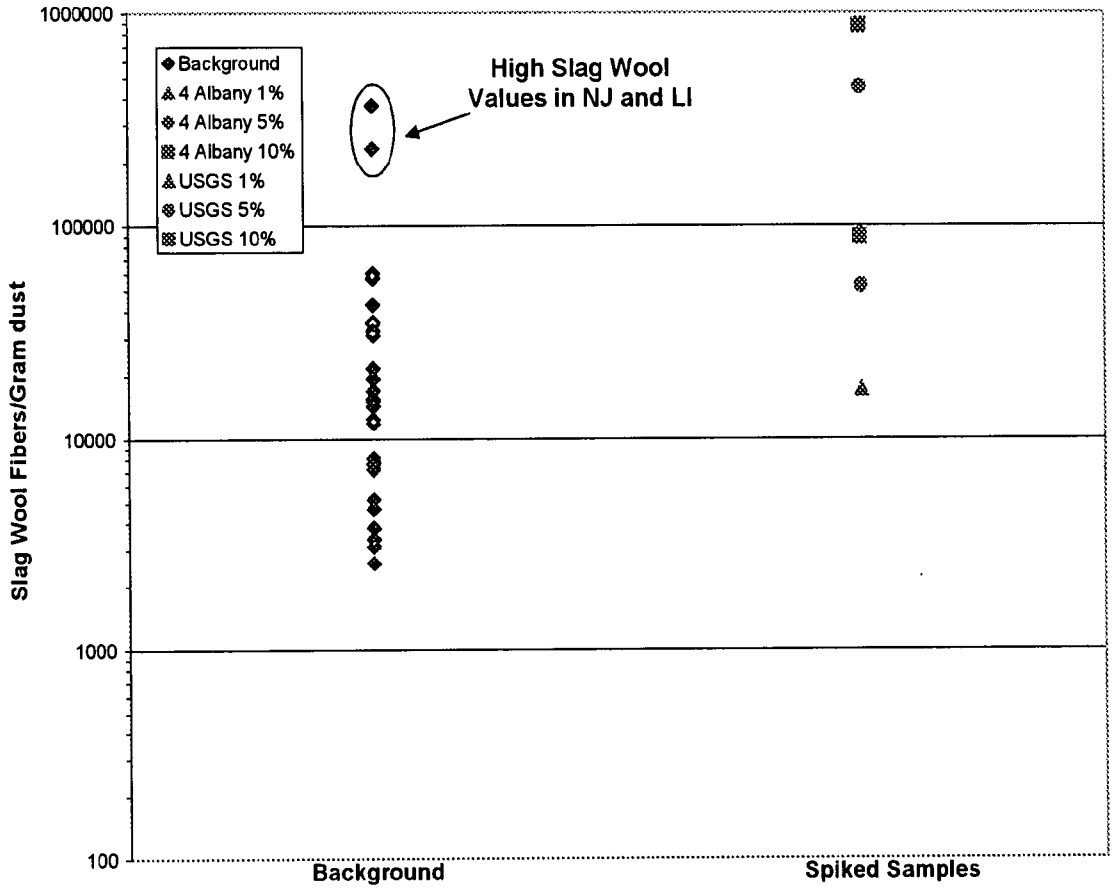


Figure 4: Average Slag Wool (Fibers/Gram of Dust) in background and spiked samples. (Data from Tables 3 and 4 Appendix E)

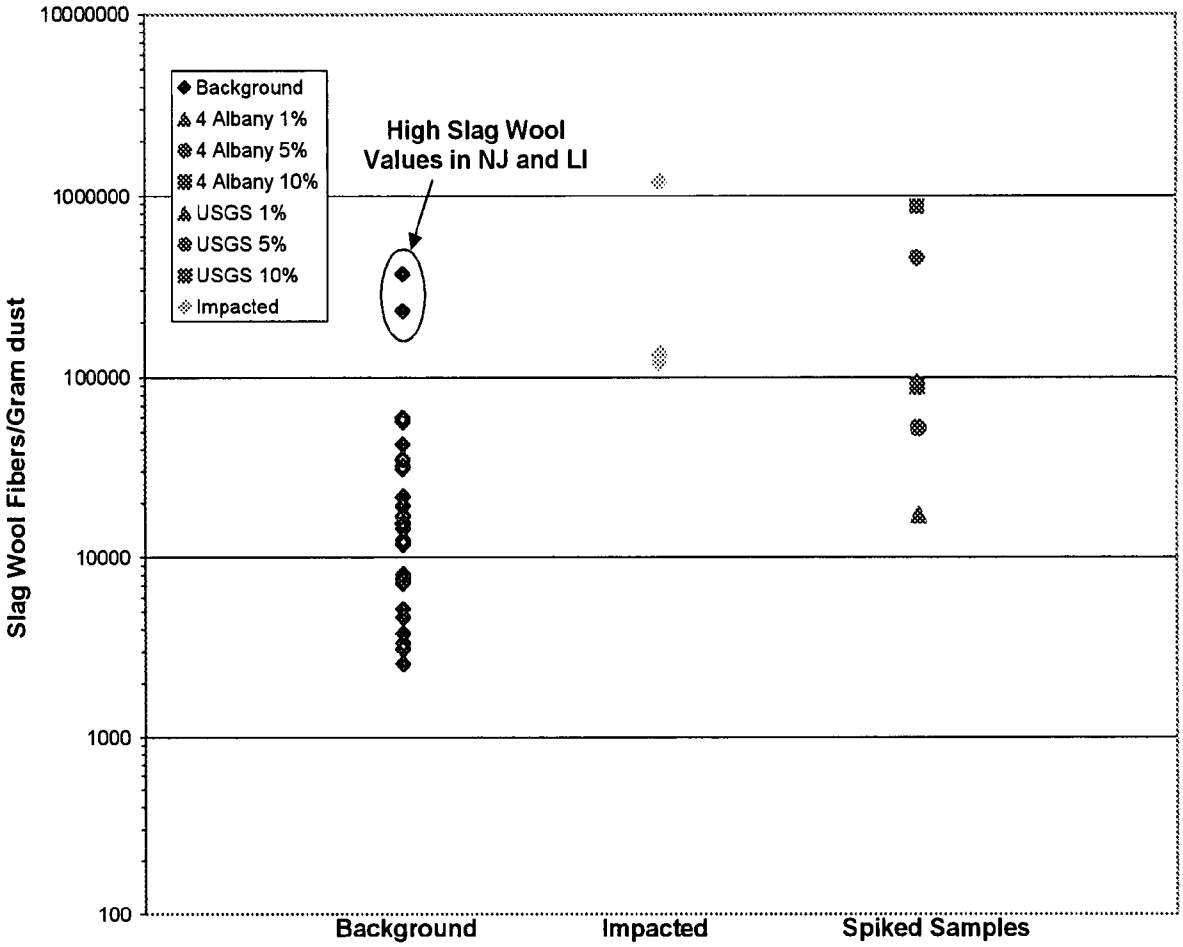


Figure 5: Average Slag Wool (Fibers/Gram of Dust) in background, spiked and impacted samples. Impacted samples are locations that are shown in satellite pictures to have been affected by WTC Collapse Dust. Slag wool results for impacted samples were derived during method development and were not part of this method validation; they are provided for comparative purposes. These impacted samples range from 0.1 to 1.6 miles from the WTC site (see Figure 8 for sample origin location). Data from Appendix B (Impacted) and Tables 3 and 4 of Appendix E (Background and Spiked).

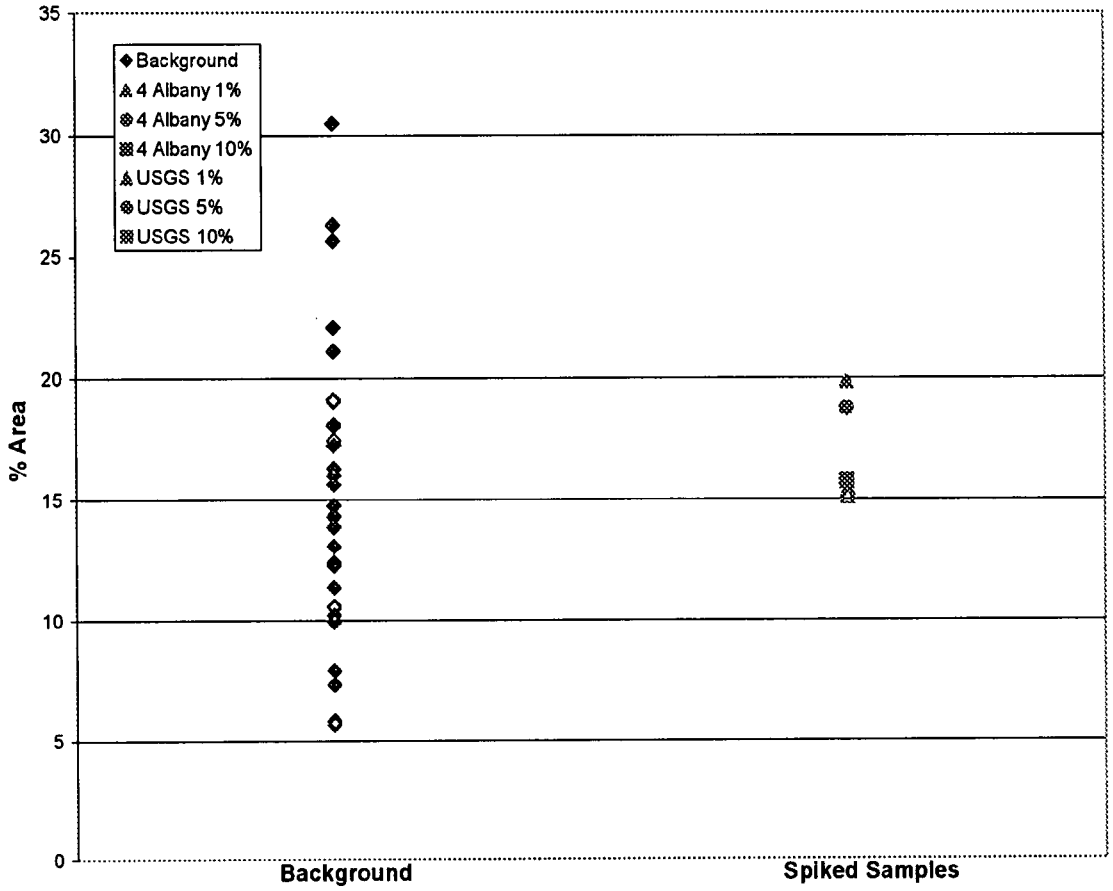


Figure 6: Average of Elements of Concrete (% Area) in background and spiked samples. (Data from Tables 1 of Appendix E)

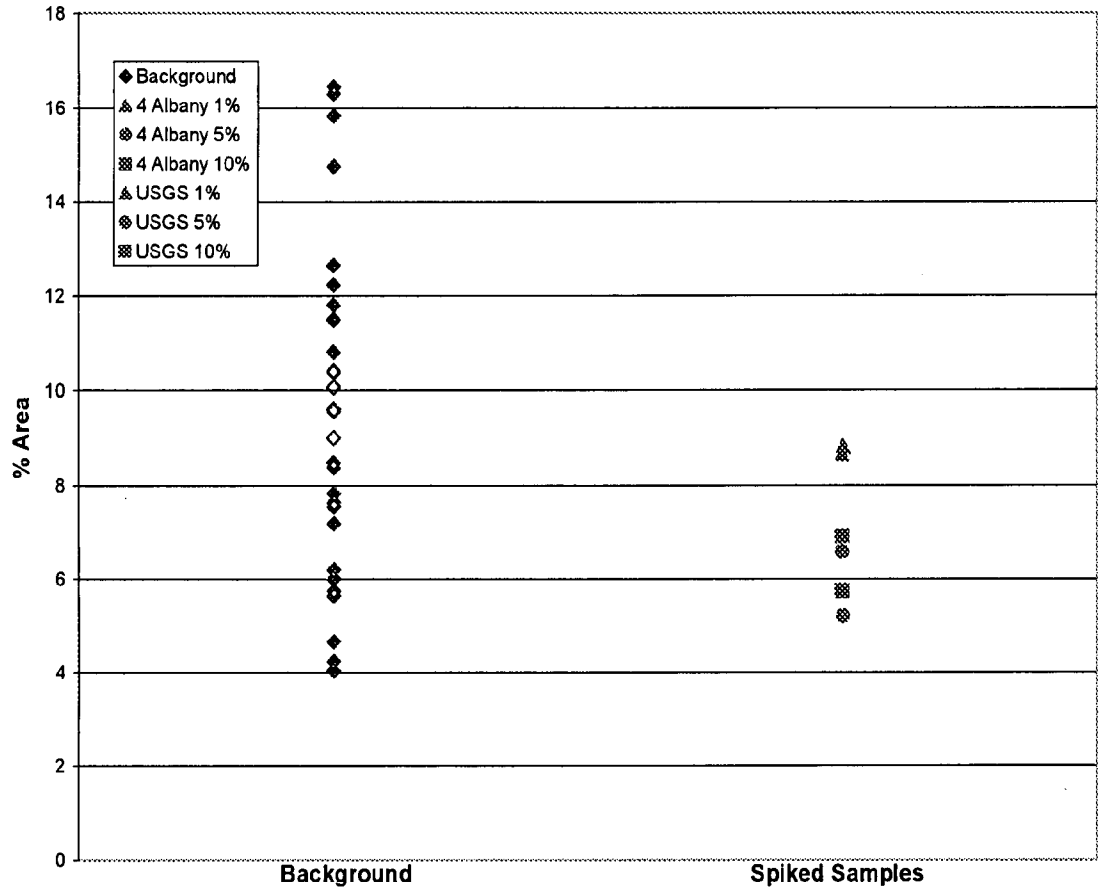
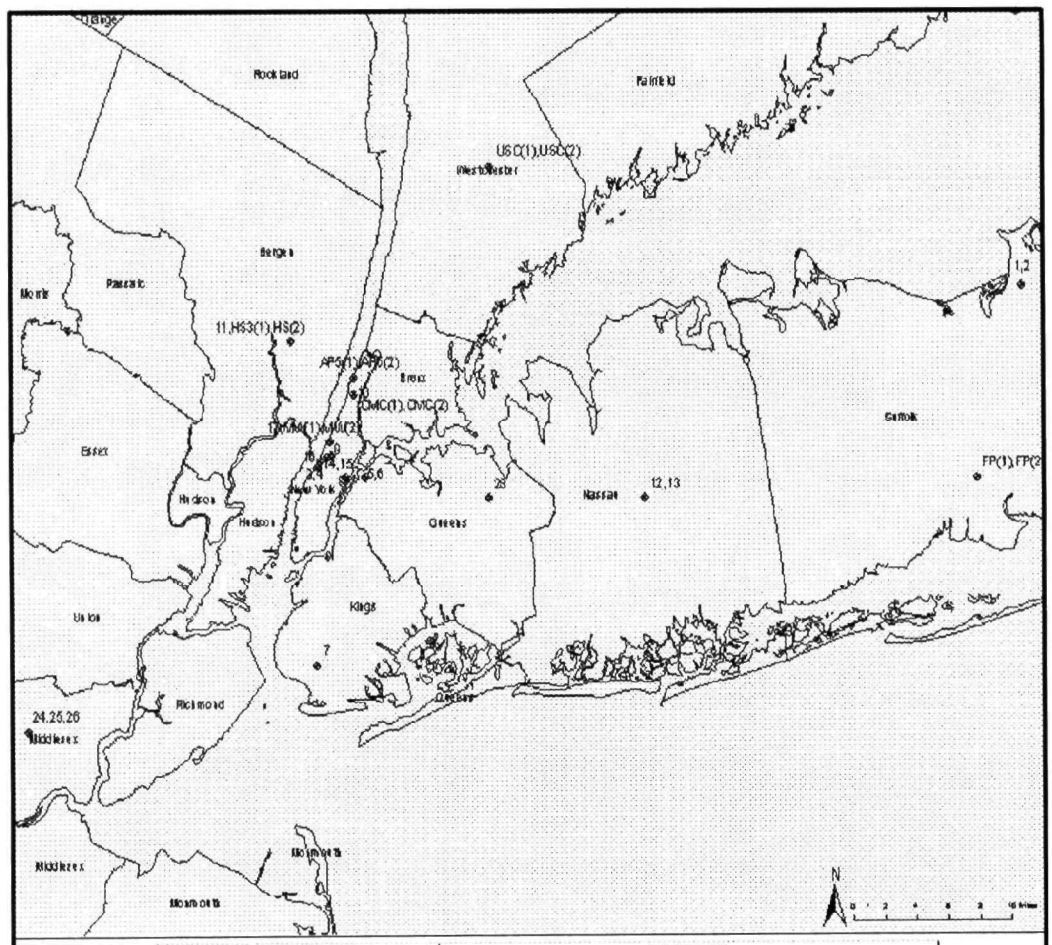


Figure 7: Average of Gypsum (% Area) in background and spiked samples. (Data from Tables 2 of Appendix E)



DESIGNATION	LOCATION
1,2	Stony Brook, LI
3,4	West End Ave between 72nd and 73rd Streets
5,6	30th Ave between 21st and 23rd, Queens
7	70th Street between 20th and 21st Ave, Brooklyn
8	79th St between York and East End Ave, Manhattan
9	92nd Street between Columbus and CPW, Manhattan
10	Columbia Medical Center, W. 168th St, Manhattan
11,HS3(1),HS(2)	Teaneck, NJ
14,15	88th Street between Amsterdam and Columbia, Manhat
16	80th Street between Riverside and East End Ave, Ma
17,MW(1),MW(2)	West End Ave between 105th and 106th Streets
18,19,20,21,22,23,C1-RTP1+2	Research Triangle Park, NC
24,25,26	Edison, NJ
AP5(1),AP5(2)	Chittenden Avenue, Manhattan
CMC(1),CMC(2)	Columbia Medical Center, W. 168th St, Manhattan
WGS(1),WGS(2)	Nassau County, LI
USC(1),USC(2)	Federal Courthouse, White Plains, NY
MUNYC1(1)+(2),MUNYC2(1)+(2)	Northern Manhattan, Above 70th Street
12,13	Nassau County
28	NE Queens
27	Long Beach Island, NJ
FP(1),FP(2)	Federal Courthouse, Central Islip, NY

Figure 8: Map of the origin of the samples analyzed during this study (Reference Appendix D for sampling data).

Discussion

Slag wool appears to be an indicator for WTC dust and can be distinguished from background dust at all three spiking levels for the USGS dust and at the 10% level of the 4 Albany Street dust The 4 Albany Street dust is considered to be WTC impacted dust but as noted earlier, the 4 Albany dust likely had lower levels of slag wool due to the fact that it was an indoor dust that was not sampled until three years after the WTC collapse

Levels of gypsum and elements of concrete have no discernable relationship to the level of WTC dust There does not appear to be a distinguishable difference between levels of concrete and gypsum in background dust and the samples spiked with WTC dust, despite USGS analysis of WTC dust from 2001 (Meeker, 2005) showing elevated levels of these components. This is likely due to the fact that while these components may seem high in WTC dust, they are also high in general background dust as they are common building materials

While method development (Appendix B and summarized in Section II above) work showed that dusts from known impacted locations generally had slag wool levels above 100,000 fibers/gram, several samples taken within this impacted zone and analyzed during method development showed lower levels of slag wool Two likely explanations can be offered for these results First, as the data in Appendix B was acquired during method development, it must be viewed as such, and second, multiple cleanings of the inhabited areas since September 11, 2001 may have removed residual WTC collapse contamination. The majority of these samples were taken in fully inhabited buildings, from locations within the buildings that can be characterized as either 'accessible' or 'infrequently accessed' areas. These terms are described in the final draft EPA sampling program, and they denote areas that are accessed by people over the course of time, such as counter tops or rugs (accessible) or underneath furniture (infrequently accessed) For this reason alone, it is encouraging that a substantial amount of the dust sampled in late 2004 and beyond had high levels of slag wool

While there was ample evidence of higher levels of slag wool associated with the WTC dust and lower levels associated with background, there is high variability in slag wool measurements within and between labs Estimates of within lab relative standard deviations based on analysis of duplicate samples of the 4 Albany Street data are 55%, 24% and 14% for the 1%, 5% and 10% dilution levels, respectively Estimates of between lab relative standard deviations based on the 4 Albany Street data are 64%, 70% and 29% for the 1%, 5% and 10% dilution levels, respectively (looking at results from analysis of the same spike level samples by multiple labs) Causes of the high levels of variability may include:

- Procedures to homogenize the spiked samples did not result in complete mixing and distribution of fibers; they instead resulted in a 'reasonably' homogeneous sample given the large size variation of the dust components
- Components of both non-impacted/background and WTC dusts will vary within a sample because of the inherent nature of the dust samples Thus, the samples received by the labs may vary in content
- Operator experience with the target components appeared to be an issue – post-study discussion indicated that labs representatives with less familiarity with slag wool expressed a belief that further guidance as to its definition was needed

- The variability in the mass of dust used for the analysis, as the protocol allows for a range, not a specific mass, to be used This range is essential due to the extreme differences in slag wool levels possible between background and spiked samples

Finally, it is noted that Table I indicates two average values for background slag wool. These values reflect the inclusion and exclusion of two samples (and their duplicates) collected in New Jersey (NJ) and Long Island (LI) that were extremely high in slag wool fibers, likely due to their insulation, fireproofing or ceiling tiles. Similarly, it was earlier noted that samples taken from a North Carolina building due also to slag wool used in ceiling tiles were not included in the interpretative analyses Based on these results, it is likely that some false positive results will occur in buildings with slag wool-based ceiling tiles, fireproofing or insulation

V. CONCLUSIONS

The interlaboratory results indicate that the better performing labs are capable of distinguishing the difference between 1, 5 and 10% 4 Albany Street dust Also, despite the high levels of within sample and within lab variability, the method using slag wool appears to be sensitive enough to distinguish 10% 4 Albany Street dust from background dust Additional evaluation of the data will be performed to further understand the variability Measures will be taken (i e standards will be sent regularly to each lab) during EPA's planned sampling program to evaluate the accuracy and precision of the laboratories

In summary, the data developed in this study support the following findings

1) Five of the eight laboratories were able to reasonably measure the slag wool concentrations in background dust spiked with confirmed WTC dust

2) High levels of variability in slag wool measurements, both within labs and between labs, were observed in the data Despite this variability, the slag wool method appears to be sensitive enough to distinguish WTC dust from background dust at the 10% level (defined as, 4 Albany Street)

3) The levels of gypsum and elements of concrete in the spiked samples were indistinguishable from the levels in the background samples This observation suggests that, while these components may have been elevated in dust samples collected near September 2001, as found by USGS in their studies on WTC dust, they are also commonly found in the indoor environment and would not be useful as WTC signature components

4) Analysis of samples during method development generally showed slag wool levels in samples from impacted locations to be greater than slag wool levels in samples from background locations

VI. REFERENCES:

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IX. APPENDICES

APPENDIX A: QUALITY ASSURANCE PROJECT PLAN FOR THE WORLD TRADE CENTER (WTC) SCREENING METHOD STUDY

(Due to formatting - this document will be provided under separate cover)

APPENDIX B: DATA ACQUIRED BY EPA NERL DURING METHOD DEVELOPMENT

Samples Collected at Background Locations

	slag wool fibers/ gram of dust	Average of Duplicates (slag wool fibers/gram dust)
Residential		
West End Ave between 72nd and 73rd Streets, Manhattan	2 53E+04	
	5 47E+04	
30th Avenue between 21st and 23rd St, Queens	2 80E+04	
	2 26E+04	
E 79th Street between York and East End Ave, Manhattan	4 93E+04	
Chittenden Avenue, Manhattan	1 53E+04	
	2 87E+04	2 20E+04
92nd Street between Columbus and CPW, Manhattan	2 42E+03	
80th Street between Riverside and West End Ave, Manhattan	1 46E+04	
Edison, NJ	0 00E+00	
Stony Brook, LI	1 79E+04	
	2 90E+04	
70th Street between 20th and 21st Ave, Brooklyn	4 09E+04	
	4 77E+04	4 43E+04
Teaneck NJ	0 00E+00	
Long Beach Island, NJ	0 00E+00	
West End Avenue between 105th and 106th Streets, Manhattan	1 77E+04	
Edison, NJ	4 12E+03	
88th Street between Amsterdam and Columbia, Manhattan	8 35E+03	
	0 00E+00	
	5 74E+03	
North East Queens (Maid Service)	0 00E+00	
	0 00E+00	
	0 00E+00	
	5 37E+03	
	1 02E+04	
	1 27E+04	
	0 00E+00	
	1 63E+04	
	6 43E+03	
	0 00E+00	
	1 65E+04	
	0 00E+00	
Nassau County, Long Island (Maid Service)	0 00E+00	
	1 95E+04	
Business		
Port Authority Bldg, Port of Newark, NJ	3 86E+04	
	3 45E+04	
	7 32E+04	
	5 09E+04	
	1 85E+04	
	6 60E+04	

	8 58E+04	
Columbia Medical Center, W 168th St , Manhattan	0 00E+00	
	1 33E+04	
Edison, NJ	9 09E+04	
Federal Courthouse, Quarropas St, White Plains	9 28E+04	
Federal Courthouse, Islip, Long Island	9 00E+04	
Samples Collected at Known Impacted Locations		
Business		
290 Broadway, Manhattan	6.92E+04	
	8 81E+04	
	1 64E+05	
	1 95E+05	1 20E+05
	8.35E+04	
Broadway between Maiden Lane and John Street, Manhattan	1 33E+05	
Deutsche Bank Bldg, 130 Liberty Street, Manhattan	2 79E+05	
	4 71E+06	
	5.77E+06	
	6 60E+06	6 19E+06
	1 18E+07	
	1 22E+07	
	1 13E+05	
	2 06E+05	
	2 14E+05	
	2 25E+05	2 30E+05
	2 28E+05	
	2 78E+05	
Deutsche Bank Bldg, 4 Albany Street, Manhattan	6 36E+05	
	1 67E+06	
USGS Composite Sample Collected Sept 2001	1 34E+07	
Samples Collected at Locations with Unknown Impact		
Residential		
John Street between Gold and Pearl, Manhattan	1 26E+04	
South End Avenue between Albany and Liberty, Manhattan	9 17E+03	
River Terrace, Manhattan	0 00E+00	
40th Street between Tunnel Exit St and 2nd Ave, Manhattan	2 91E+03	
Orange Street between Henry and Hicks, Brooklyn	1 11E+04	
24th Street between 8th and 9th Ave, Manhattan	3 32E+03	
Montague between Montague Terrace and Hicks Street, Manhattan	5 03E+03	
Houston and Mulberry Streets, Manhattan	6 30E+03	
Business		
Port Authority Bldg, Columbia St, Brooklyn	2 06E+05	
	9 89E+04	
	1 30E+05	
	1 94E+05	
	1 12E+04	
	3 06E+05	

Governor's Island	5 07E+04
	5 75E+05
	8 79E+04
Varick Street, Manhattan	9 57E+04
Samples Collected Outside of NY City	
Business	
Research Triangle Park, NC	5 00E+04
	8 96E+04

APPENDIX C: DATA ACQUIRED BY EPA NERL POST-STUDY

Samples Collected at Background Locations		
Residential		
Composite –North East Queens (Maid Service)	1 06E+04	
	1 49E+04	1 28E+04
Business		
Port Authority – Port of Newark, NJ	9 77E+03	
Samples Collected at Impacted Locations		
Business		
Governor's Island	1 93E+04	
	6 39E+05	
	1 21E+06	
Port Authority Bldg, Columbia St, Brooklyn	1 22E+05	

APPENDIX D: PROTOCOL USED FOR THE SCREENING METHOD STUDY

Protocol for Preparation and Analysis of Residential and Office Space Dust by Polarized Light Microscopy and Scanning Electron Microscopy with Energy Dispersive X-Ray Spectroscopy

June 27, 2005

Prepared by:
U.S. Environmental Protection Agency
National Enforcement Investigations Center/ National Exposure Research Laboratory/National Homeland Security Research Center
Denver, CO and Research Triangle Park, NC

The use of trade names does not imply endorsement and are used for illustrative purposes only

Contents

1.0 Purpose

This document describes sample preparation and analytical screening procedures for bulk samples of dust collected from residential and commercial office environments These methods are collectively referred to as the protocol

2.0 Scope/Application

The protocol describes polarized light microscopy (PLM) and scanning electron microscopy (SEM) with energy dispersive spectrometry (EDS) to screen bulk dust samples for mineral slag wool, particles consistent with concrete compositions, and gypsum The analysis methods include operating parameters and particle identification criteria.

2.1 Limitations of the Method and Future Considerations

This protocol provides a means of analyzing for particles consistent with those found in dust present after the collapse of the World Trade Center (WTC) in New York City Components of WTC Dust have been documented and catalogued by the U S Geological Survey Denver Microbeam Facility and the images and characteristics shall be used in identification of particles (1)

The x-ray mapping procedure in sections 12 2 3 and 12 2 4 and the calculations presented in section 13 0 only determine the maximum percentage of non-gypsum, calcium-rich particles, which may include non-concrete materials The particle analysis procedure presented in section 12 2 5 is the preferred procedure for determining the percentages of gypsum and concrete particles in the sample

The x-ray mapping and image analysis procedure relies heavily on the thresholds for backscattered electron images Binary (particles white and background black) backscattered electron images (BEI) should be used to reduce errors in setting thresholds in Photoshop

3.0 Definitions

1 PLM – Polarized Light Microscopy
2 SEM – Scanning Electron Microscope
3 EDS – Energy Dispersive Spectrometry
4 SEI – Secondary Electron Image
5 BEI – Backscattered Electron Image
6 Mineral Wool – lightweight vitreous fibrous material composed of rock wool and slag wool and used especially for heat and sound insulation
7 Rock Wool – a man-made vitreous fiber (MMVF) component of mineral wool containing magnesium, aluminum, silicon, and calcium Sodium and potassium may also be present Iron oxide is typically 3-12% by weight
8 Slag Wool – a man-made vitreous fiber (MMVF) component of mineral wool containing magnesium, aluminum, silicon, and calcium Sodium and potassium may also be present Iron oxide is typically less than 2% by weight
9 HEPA – High-Efficiency-Particulate-Air Filter

4.0 Summary of Method

1. Weigh sample to nearest 0.0005 g

2 Split the sample, archive half and keep half for analysis
3 Ash half of the sample for analysis
4 Sieve the ashed sample to 150 µm
5. Split the <150 um ashed portion Archive three quarters of the sample Keep one quarter for PLM and SEM/EDS analysis
6 Weigh the quarter and place it in enough isopropanol to get a 10-20 mg per mL dilution Apply an aliquot to a glass slide, let dry, and add 1 55 (or 1 605) refractive index oil. Analyze by PLM for mineral wool.
7 Prepare a sample for SEM/EDS analysis using the same dilution prepared for PLM.
8 Apply an aliquot of the sample to an aluminum sample stub with a carbon adhesive tab covered by a piece of polycarbonate filter (13-mm diameter or punched out of a larger filter to fit the size of the stub)
9 Identify fibers by EDS and record the occurrence of fibers > 25 µm in length at 100 x magnification to get a statistical representation of fiber compositions
10. Prepare 10-fold dilution of the suspension from step 7 and apply an aliquot to a polycarbonate/adhesive tab substrate affixed to an aluminum sample stub Alternatively, a lighter loading can be prepared by filtering the diluted suspension through a 25-mm diameter, 0.4-µm pore size, polycarbonate filter and affix this to a carbon adhesive tab affixed to an aluminum sample stub
11 Collect x-ray maps of 10 fields at 500 x magnification for major elements, especially Ca, S, and Fe and use Adobe Photoshop or similar software to determine the area percent of gypsum and Ca-rich particles Fe-rich particles may also be identified in this step
12 Perform particle analysis via computer-controlled SEM/EDX analysis

5.0 Interferences

Interferences include possible contamination of samples by airborne dust or through improperly cleaned glassware and sieves Interferences are minimized by performing all procedures involving dry dust in a clean room, cleaning countertops and glassware thoroughly before proceeding and placing particle-free wipes on all working surfaces To avoid cross-contamination, properly clean all glassware, sieves, and tools between samples

6.0 Safety

Respirable particles which may present a health hazard may exist in the sample Bulk samples may release respirable particles during handling All procedures involving dry dust samples will be performed under a negative flow High-Efficiency-Particulate-Air Filter (HEPA) hood Samples handled outside of the HEPA hood will be covered with aluminum foil or placed in sealed glass jars

7.0 Apparatus and Materials

1 HEPA negative flow hood
2 Forceps
3 Kimwipes
4 Stainless steel spatula
5 Weighing paper
6 Programmable furnace [not required for validation study]
7 Ceramic crucibles with lids [not required for validation study]

8 Analytical balance (accuracy to 0 0005 g)
9 Retsch ultrasonic sieve shaker (AS200 Basic), or similar [not required for validation study]
10 Sample sieves, 3-inch diameter (recommended), 150-µm (100-mesh) opening, with lid and bottom pan similar [not required for validation study]
11 SEM aluminum sample stubs
12 Conductive carbon adhesive tabs
13 Eppendorf pipette, 10-µL capacity
14 Disposable pipette tips
15 1 – 10 mL pipette
16 Glass vials for sonicating dust in isopropanol suspension (holds 10-mL volume)
17 Razor blade
18 Ultrasonic bath
19 50 mL glass beaker
20 Polycarbonate filters (25-mm diameter, 0 4-µm pore size)
21 Polycarbonate filters (13-mm diameter, 0 4-µm pore size), or borer to cut larger filters to SEM stub size
22 11-mm diameter cork borer
23 Millipore filter apparatus for use with 25 mm filters
24 125 mL Nalgene bottles
25 Hand-held vacuum pump
26 High-vacuum carbon evaporator with rotating stage
27 Glass etri dishes with lids
28 Adobe Photoshop Software, or similar
29 Glass petrographic slides
30 Glass cover slips
31 Polarized light microscope for mineral identifications
32 Scanning Electron Microscope with the following attributes
    a Resolution 5 nm (at 25 kV, WD=10 mm – system dependent) or better
    b Accelerating Voltage 10 to 20 kV
    c Minimum magnification range 50x to 200,000x
    d SEI (secondary electron image)
    e BEI (backscattered electron image)
    f Energy dispersive x-ray detector and analyzer for EDS analysis
    g Ability to collect x-ray maps or particle analysis software (preferably both)

8.0 Reagents

1 Isopropanol, reagent grade [CAS No 67-63-0]
2 1 55 or 1 605 Refractive Index Oil

9.0 Sample Storage

Dust samples will be stored in an air-tight container, such as a sealed glass jar Samples placed in reagents will be labeled appropriately and stored according to laboratory safety standards Samples prepared for analyses will be stored in a protective container, such as a plastic case or covered etri dish, to prevent contamination

10.0 Quality Control

Quality control is implemented by thoroughly cleaning glassware and spatulas, keeping working surfaces clean, and preventing cross contamination. During ashing, particles may be suspended if slow heating is not achieved Following the ashing program as outlined will minimize flashing, which can cause particles to become airborne Covered crucibles will be used to prevent contamination caused by flashing Used Eppendorf pipette tips and weighing papers will be discarded and new tips and papers will be used for each

sample

Duplicate samples shall be prepared to determine the precision of the analysis In addition, sample blanks shall be prepared These blanks are checks for cross contamination during handling of the samples Blanks shall be prepared at the same time and in the same manner as samples

10.1 Calibration

Calibration of the EDS system must be completed at least once at the beginning and again at the end of each analytical session Backscattered electron image (BEI) calibration should be performed at the beginning of the session and anytime the backscattered image brightness and/or contrast is adjusted

EDS calibration for both qualitative and quantitative (not required by this method but could be useful for identification of particle type) analysis is accomplished by the analysis of a polished carbon-coated reference standard The recommended material is USGS BIR1-G basalt glass mounted in epoxy in a brass tube, polished, and carbon coated using a carbon evaporator (2, 3)

The calibration reference material should be analyzed at the same operating conditions to be used for the analysis including beam current, accelerating voltage, working distance, detector dead time, and sample tilt (= 0°) For BIR1-G the analysis should be performed with a beam size of 10-20 µm or equivalent area raster All calibration spectra will be saved with the corresponding data set The calibration data will be used for inter- as well as intra-laboratory comparisons This calibration is in addition to, and not a substitute for the normal EDS calibration recommended by the EDS manufacturer which will be performed at regular intervals as specified by the EDS manufacturer

Backscattered electron detector calibration can be performed on the same BIR1-G material by adjusting the detector brightness and contrast to achieve the following conditions The epoxy on the BIR1-G reference material will be at 0 in a 256 grayscale image and the brass mounting tube will be at 256 The BIR1-G basalt glass should fall at approximately 130-140 gray scale units

11.0 Procedure

11.1 Weighing and Splitting

Weighing and splitting should be performed under a negative flow HEPA hood If the fan speed is set too high, loss of particles may occur. The fan speed may need to be adjusted to prevent the loss of fine particles

Obtain an analytical balance with an accuracy of 0 0005 g and preweigh a clean piece of weighing paper Transfer the dust from the sample vial to the weighing paper and determine the weight of the dust Split the sample with a clean razor blade using the cone-and-quarter method If there are large clumps of organic fibers, such as hair or lint, temporarily remove the hair with a pair of forceps and tap the forceps lightly with another tool over a piece of weighing paper to remove fine particles. Center the fine fraction on the paper and split the sample into four equal parts using a razor blade Collect opposite corners (½ of the sample) for analysis and archive the other half Quarter the larger organic fiber bundles the same way, keeping half to proceed to the ashing step and half for archival purposes

Place the two quarters for ashing into a preweighed crucible Weigh the split and record the results

11.2 Ashing

Place the ceramic crucibles containing the samples into a furnace

The furnace program should proceed as follows

1 Increase temperature by 1 °C/minute until sample reaches 250 °C
2 Hold temperature at 250 °C for 4 hours
3 Increase temperature by 1 °C/minute until sample reaches 480 °C
4 Hold temperature at 480 °C (sufficient for decomposing organics) for 8 hours Do not exceed 500 °C
5 Shut off furnace
6 Allow sample to cool before removing from furnace
7 Weigh the ashed sample to the nearest 0 0005 g and record the result

11.3 Sieving

Sieve the sample through a 150-µm sieve using a Retsch ultrasonic sieve shaker, or similar Three-inch diameter sieves are recommended to minimize sample loss from particles being trapped in the sieve The ultrasonic shaker will be operated at 20-minute intervals at the following settings. 20, 40, 60, 70, 80, then back down to 50 and 20. This will provide amplitudes ranging from 0 to 1 5 mm

Transfer the large and small fractions to clean pieces of weighing paper and weigh to the nearest 0 0005 g Archive the fraction greater than 150-µm

11.4 Preparation of Sample for Polarized Light Microscopy

Split the less than 150-µm sample fraction using the cone and quarter method Collect one corner for analysis and archive the other three quarters Weigh the quarter split to the nearest 0 0005 g and place it into a glass vial Make a suspension of 10-20 mg dust per mL of isopropanol The amount of isopropanol needed will vary depending on the amount of dust, the target dilution is 10-20 mg per mL

Cut an Eppendorf pipette tip with a razor blade to increase the opening to approximately 1 mm

Place the suspension in an ultrasonic bath for one minute, then remove the suspension from the ultrasonic bath and shake it gently to suspend all particles Collect a 10-µL aliquot of the mixture using an Eppendorf pipette with the modified tip and transfer to a glass slide Prepare 4 such slides Allow them to dry, then add a drop of 1 55 (or 1 605) refractive index oil

11.5 Preparation of Sample for SEM Analysis

Prepare the SEM substrate on aluminum stubs using 0 4-µm pore size polycarbonate filters, carbon adhesive tabs. Using an 11 mm filter punch and placing the filter between two filter separators, punch a circle the size of the

carbon tab into the filter Place carbon adhesive tab affixed to an aluminum stub on the dull side of the 11-mm polycarbonate filter such that the shiny side of the filter exposed If available, a 13-mm diameter polycarbonate filter may be used in place of the punched out 11-mm filter

Collect a 10-µL aliquot of the mixture from the PLM sample preparation using the Eppendorf pipette with the modified tip and transfer to a prepared polycarbonate/adhesive tab substrate This will yield a loading on a 12-mm SEM stub of about 100-200 µg, which is a moderately heavy loading. Adjust the number of aliquots as needed to obtain the target loading

Prepare a 10-fold dilution of the above suspension to get a suspension of 1-2 mg dust per mL of isopropanol Sonicate the suspension in an ultrasonic bath for one minutes. Remove the suspension and gently shake it to suspend all particles. Wait one minute to allow the coarse particles to settle Collect a 10-µL aliquot of the suspended mixture using an Eppendorf pipette with the modified tip and transfer to a prepared polycarbonate/adhesive tab substrate This will yield a loading on a 12-mm SEM stub of about 10-20 µg, which is a light loading Adjust the number of aliquots as needed to obtain the target loading.

Alternatively, prepare a lightly loaded sample using the filtration method as follows Use a Millipore filter apparatus for use with 25-mm filters for filtration. Place a few drops of isopropanol on the fritted glass surface and place the 25-mm polycarbonate filter (0 4-um pore size) on the isopropanol. Attach the top of the apparatus and add a few milliliters of isopropanol to the filter so that no part of it is exposed to air Sonicate the suspension (diluted as described in previous paragraph) in an ultrasonic bath for one minute. Remove the suspension and gently shake it to suspend all particles Wait one minute to allow the coarse particles to settle. Collect 1 mL of the suspended mixture using a pipette and filter it through the polycarbonate filter Actual amounts for filtration will vary based on sample loading. The goal is to have a loading on a 12-mm SEM stub of about 10-20 µg, or about 5-10 percent area coverage, which is a light loading Adjust the volume of the aliquot to filter as needed to obtain the target loading

Place the filter on a carbon adhesive tab on a standard SEM aluminum mount The filter needs to be completely flat on the SEM stub. This can be achieved by forming the wet filter into a gentle U-shape using forceps and the side of the forefinger, then placing the bottom curve of the filter onto the center of the carbon adhesive tab and slowly releasing the sides so they lay flat. Trim the edges of the filter using a razor blade

After drying, coat the samples on the polycarbonate or polycarbonate/adhesive tab substrates with carbon using a carbon evaporator with a rotating stage Transfer the stubs to the SEM in a clean, covered container

12.0 Analysis

12.1 Analysis by Polarized Light Microscopy

Polarized light microscopy will be conducted using the general techniques outlined in EPA 600/R93/116 (4) For this procedure, four slides (prepared as described in section 11 4) will be analyzed The fraction of fibers with refractive index greater than 1 55 (or 1 605) will contain mineral wool, which includes both slag wool and rock wool, and possibly some E-type glass and ceramic fibers The fraction of fibers with refractive index less than 1 55 (or 1 605) will contain primarily soda-lime glass fibers For the validation study, numbers of fibers greater than **and** less than 1 55 (1 605) refractive index will be counted Dispersion staining and becke line techniques may be used Fiber point counting will be performed at 100 x magnification

If more than 20 mineral wool fibers are found, continue counting and recording all of the fibers above and below the index oil refractive index Report both raw fiber counts per refractive index category and number of fibers from each category per gram of sample Continue on to step 12 2 1 to determine the ratio of slag wool to other fibers with refractive index greater than 1 55 (or 1 605) using EDS as described below

If less than 20 mineral wool fibers are found on each slide, count the number of slag wool fibers using SEM/EDS and report as number of fibers per gram of sample

12.2 Analysis by SEM/EDS

12.2.1 Screening for Slag Wool

Operating conditions for the JEOL 6460-LV SEM are 15 kV, 0 5-5-nA beam current, 10-mm working distance (system dependent), and zero degree tilt

Place the more concentrated sample deposited directly on the polycarbonate/adhesive tab substrate into the SEM Use the backscattered electron mode at 100x magnification to quickly distinguish carbon fibers from inorganic fibers (carbon fibers may be visible, but not as bright in a BEI) Identify all inorganic fibers over 25 µm in length (smaller fibers cannot be reliably detected at the 100x operating magnification) When an inorganic fiber is found, identify the composition of the particle by EDS Slag wool is the primary fiber of interest Record all inorganic fiber results as number of fibers for each fiber type

For the samples with high fiber loading, as determined by PLM as described in section 12 1, count fibers per type until a statistical representation of the ratios of fiber compositions in the sample is achieved Report the ratio (by fiber number) of slag wool fibers to total MMVF fibers corresponding to the high RI Use this ratio to correct the total number for high RI fibers counted by PLM to number of slag wool fibers present

For the samples with low fiber loading, as determined by PLM as described in section 12 1, scan the entire stub to determine the number of fibers per type Report the slag wool fiber results as the number of slag wool fibers/gram of sample

12.2.2 EDS Screening for Gypsum/Anhydrite

Place the more concentrated sample deposited directly on the polycarbonate/adhesive tab substrate in the SEM Choose a random field at 100x magnification and perform an EDS analysis on the entire field Look for the presence of sulfur in this field If sulfur is present, continue to Section 12 2 3 or 12 2 5 for analysis of gypsum and concrete by mapping or particle analysis If it is not present, repeat the analysis on another random field If sulfur is still not present, mark the sample as non-detect (ND) for sulfur

12.2.3 X-Ray Mapping for Gypsum

Place a more dilute sample, deposited directly on the polycarbonate/adhesive tab substrate or prepared by filtration, in the SEM Collect binary backscattered electron images (particles white and background black, shadow off) and secondary electron images for 10 non-overlapping, random fields at 500 x magnification Collect x-ray maps for Na, Mg, Al, Si, S, Ca, and Fe at each of these fields Fields containing MMVF will not be used for this analysis Operating parameters for the SEM are the same as those for analyzing slag wool Acquisition parameters for x-ray mapping using the NORAN System Six Software are time constant 14 (mapping mode, 11333 cps), 10-20 % deadtime, 256 x 256 image resolution, 20 second frame time, and 100 frames collected (about 40 minutes total acquisition time) Secondary electron images will be used for reference only Save all of the maps and electron images in TIFF format

Open the backscattered electron image and the Ca and S x-ray maps in Adobe Photoshop Make sure that all of the element maps are the same size and resolution by choosing Image Size from the Image Menu and changing the pixel size or the resolution as needed The presence of gypsum can be determined by overlapping the Ca and S maps

Perform the following functions in Adobe PhotoShop (A macro is in development to perform the following functions to decrease user time and human errors in adjusting the threshold)

1 Convert each of the three images to grayscale (Image → Mode → Grayscale)
2 Perform an auto contrast and brightness on each image and map to increase the scale of colors (Image → Adjustments → Auto Levels)
3 Threshold each element map, Ca and S (do not analyze the backscattered electron image at this time), by going to the Image Menu and choosing Adjustments → Threshold Adjust the threshold to 128 The background will be black and the particles white
4 Invert the image (Image→ Adjustments →Invert) to make the background white and the particles black
5 Copy the S map and paste it over the Ca map in a separate layer in the file and change the opacity (located in the Layers window) to 50 % for the S map layer The black areas are gypsum/anhydrite
6 Display a histogram of the image in expanded mode by selecting the Histogram tab on the Navigator Window (or under the Image Menu in some versions of Photoshop) Place the cursor over the line for the black area and record the percentile for the black area This is the percentage of particles containing Ca and S in the entire field

NOTE If a binary backscattered electron image is obtained during data collection, then steps 7-11 may be deleted The Invert function will, however, need to be applied to make the particles black and the background white before continuing to step 12

7 Begin analysis of the backscattered electron image Select the particles by going to the Select Menu and choosing Color Range Go to the selection pulldown menu and choose Highlights
8 Fill the selection with black by going to the Edit Menu → Fill and choosing black from the color pulldown menu
9 Select the inverse areas by going to the Select Menu and selecting Inverse
10 Fill the selection with white by going to the Edit Menu → Fill and choosing white from the color pull down menu
11 Deselect the area by clicking on the image
12 Perform the Threshold and Histogram functions for the backscattered electron image

as outlined in 3 and 6 Record the histogram result for the backscattered electron image

Determine the area percent of gypsum by performing the calculations in Section 13 0

12.2.4 X-Ray Mapping for Ca-Rich Particles

Analysis of components of concrete will be performed on the same fields as the gypsum/anhydrite analysis At this time, only a method for the determination of the area percent of Ca-rich particles is presented See Section 2 1 for discussion

Perform the following steps on the Ca x-ray map Tiff file in Adobe Photoshop

1 Convert the Ca x-ray map to grayscale (Image → Mode → Grayscale)
2 Perform an auto contrast and brightness on the map to increase the scale of colors (Image → Adjustments → Auto Levels)
3 Threshold the Ca map by going to the Image Menu and choosing Adjustments → Threshold Adjust the threshold to 128 The background will be black and the particles white
4. Invert the image (Image→ Adjustments →Invert) to make the background white and the particles black
5 Display a histogram of the image Place the cursor over the line for the black area and record the percentile for the black area This is the area percent coverage of particles containing Ca in the entire field

Determine the maximum area percent coverage of non-gypsum, Ca-rich particles by performing the calculation in Section 13 0

12.2.5 Particle Analysis for Identification of Gypsum and Concrete.

Place the more dilute sample, deposited directly on the polycarbonate/adhesive tab substrate or prepared by filtration, in the SEM Particle analysis will be used to identify gypsum and concrete particles

Perform particle analysis at 500 x magnification All other operating parameters for the SEM are the same as those used to analyze for slag wool (Section 12 2 1) A binary backscattered electron image should be used in particle analysis mode Particle analysis parameters should be set to analyze all particles in the field greater than 0 5 µm and to separate touching particles For particles greater than 5 µm, scan the entire particle, spot analysis is adequate for smaller particles The x-ray spectrum and counts for all particles, and an image of particles > 20 µm long, will be recorded and saved Other particle parameters to be reported will include the maximum, minimum, and average diameters, the aspect ratio, and area of each particle

It will be necessary to review data collected by automated software to ensure data integrity An Excel spreadsheet, in conjunction with images and x-ray data, may be used for this purpose Particles should be sorted into one of three categories Ca-S (gypsum), Ca-rich, and Other Aid in identification of particles may by facilitated by referencing the U S Geological Survey's WTC Dust Particle Atlas (1) A particle classification protocol will be developed based on the data from the validation study

The number of particles analyzed will be determined using the results of the validation study For the study, the area percent of each component should be within 10% relative error or better Typically, data for 1000 – 1200 particles should be acquired

Results for particle analysis will be recorded as area percent gypsum and area percent concrete particles for each field and average area percent for the each component in the sample

13.0 Data Analysis and Calculations

Table 60 To determine the concentration of slag wool in fibers/gram, perform the following calculations

Determine the number of fibers with RI > 1 55 (or 1 605)

fibers identified – mg of sample on slide × 1000 = fibers/gram on slide

Determine the percentage of fibers with the composition of slag wool with RI > 1 55 (or 1 605)

Fibers/gram on slide × # fibers identified as slag wool / Total number of fibers identified by EDS with RI > 1 55 (or 1 605) = fibers slag wool/gram on slide

Back calculate to the number of fibers per gram of the original sample

Fibers slag wool/g on slide × g after sieving × g sample after ashing / g before sieving × g sample before ashing = Total f/g of sample

Table 61 To determine the area percent of gypsum/anhydrite from the x-ray mapping procedure, perform the following calculations

Determine the area percent of gypsum/anhydrite in each field of view

% of black area in Ca-S map overlay / % of black area in BSE image × 100 = area % gypsum

Calculate the average percentage of gypsum/anhydrite for the sample

$\frac{(\text{area \% gypsum})_{f1} + (\text{area \% gypsum})_{f2} + \ldots}{\text{number of fields}}$ = Avg area % gypsum

Table 62 To determine the maximum area percentage of Ca-rich particles, which includes concrete particles, from the x-ray mapping procedure, perform the following calculations

Determine the area percent of non-gypsum Ca-rich particles in each field of view

(% black area Ca map) – (% black area Ca-S map) / % black area on BSE image = % non-gypsum Ca-rich particles

Calculate the average percentage of non-gypsum Ca-rich particles for the sample

$\frac{(\text{area \% Ca-rich particles})_{f1} + (\text{area \% Ca-rich particles})_{f2} + \ldots}{\text{number of fields}}$ = Avg area % Ca-rich particles

Table 63 Calculate the area percent for gypsum and concrete by summing the areas of each particle in for each particle type and dividing by the total area analyzed

$\frac{\text{area gypsum 1} + \text{area gypsum 2} + \ldots}{\text{total area analyzed}} \times 100$ = area percent gypsum (do likewise for concrete)

Rules for concrete and gypsum classification are currently being developed

14.0 References

1 Lowers, Heather A , Meeker, Gregory P , Brownfield, Isabelle K , 2005 World Trade Center Dust Particle Atlas U S Geological Survey Open-File Report 2005-1165 On the web at http //pubs usgs gov/of/2005/1165/

2 Meeker, G P , Taggart, J E , and Wilson, S A , 1998 A Basalt Glass Standard for Multiple Microanalytical Techniques Proceedings Microscopy and Microanalysis 1998 Microscopy Society of America

3 A polished and carbon coated calibration reference sample of BIR1-G may be obtained by contacting Stephen Wilson, U S Geological Survery, MS 973, Denver Federal Center, Denver, CO, 80225, swilson@usgs gov

4 Perkins, R L and Harvey, B W , 1993, TEST METHOD Method for the Determination of Asbestos in Bulk Building Materials, EPA/600/R-93/116

15.0 Appendix: DATA SHEETS

Determination of Slag Wool Fibers in Dust- PLM with Dispersion Staining

Sample ID Project
Analyst
Circle One Original Duplicate Triplicate Date

General Sample Appearance _____
Homogeneous? Y

Structure #	RI Fluid		Dispersion Staining		Becke Line		Fiber			Comments
	1.55	1.605	>RI	<RI	>RI	<RI	MW	non-MW	chrysotile	

SEM Sheet

Reference ASTM - D5755-03

Report Number: _____
Sample Number: _____
File Name. _____
Sample Description: _____
_____
_____
_____

Preparation Date: _____ By: _____
Analysis Date: _____ By: _____
Computer Entry Date: _____ By: _____
Sample weight: _____ grams
Dilution Volume: _____ mL
Volume Aliquot: _____ uL
Magnification: _____ X

Structure #	Field #	Fiber Type	Length (Microns)	Width (Microns)	Image	EDS

Gypsum and Concrete Report Sheet: X-Ray Mapping Procedure

Sample Number:_____ Analyst:_____ Date:_____

Field #	File Name	% Particles in EEI	% Ca Sarea	% Ca-rich area	% Gypsum	% Ca-rich Particles

Averages

APPENDIX E: REPORT FROM THE U.S. EPA CONTRACTOR ON THE SCREENING METHOD STUDY

Versar
6850 Versar Center
Springfield, VA 22151

Ms Jacky Rosati
US Environmental Protection Agency
E-305-03 109 T W Alexander Drive
Research Triangle Park, NC 27711

July 21, 2005

Dear Ms Rosati

Attached is a *preliminary* report based on analytical data *thus far received*, for dust samples collected primarily in the New York City area Most of the samples were taken in areas that, it is believed, were not affected by particulate matter generated during the World Trade Center (WTC) collapse (i e , background samples) Some of the samples were spiked with one or the other of two dusts that are believed to have originated from the WTC collapse. The analytical protocol was developed by the government, specifically for this project, and was modified as the project developed. The purpose of the testing was to determine if the spiked background dusts could be distinguished from those samples that were not spiked

Three parameters were measured to make this determination· (1) slag wool fiber content, (2) calcium-rich particle content, and (3) gypsum particle content

The analytical data indicate that

- With respect to calcium-rich particles and gypsum particles, spiked samples cannot readily be distinguished from background samples
- With respect to slag wool content in the samples spiked with the first of the two WTC dusts, spikes at the 10% level may be statistically identifiable as WTC-contamination, although spikes at or below the 5% level are probably not identifiable
- With respect to slag wool content, samples spiked with 5% and 10% of the second of the two WTC dusts are easily identifiable as WTC-contaminated Even at the 1% spike level, samples may be statistically identifiable

The attached *preliminary* report will explain the above conclusions in more detail However, it must be noted that all of the analytical data from the eight laboratories that performed the analysis has not yet been received Nevertheless, it is believed that the above conclusions will not likely change once those additional data are incorporated

Sincerely,

Stephen M Schwartz, P E , Q E P
Project Manager

Versar

Preliminary Report
of
Analysis of New York City Area Dust Samples

Purpose:

The objective of this study is to determine if New York City area dusts that are contaminated with varying levels of dusts known to originate from the collapse of the World Trade Center (WTC) can be distinguished from background dusts that are believed not to be contaminated with WTC dusts

Project Summary:

In the initial portion of the testing, 10 dust samples from New York City areas that are believed not to be contaminated with dusts originating from the collapse of the WTC were used These are referred to as the first set of *background* samples An additional background dust sample was spiked at 1, 5, and 10 percent levels (by weight) with dust believed to have originated from the WTC collapse An additional background sample was spiked at 1, 5, and 10 percent levels with a second dust sample that is believed to have originated from the WTC collapse Therefore, a set of 16 samples was generated

- 10 different background dusts
- 3 samples, each consisting of one background dust sample spiked with one source of WTC dust at 1, 5, and 10% levels
- 3 samples, each consisting of one background dust sample spiked with a second source of WTC dust at 1, 5, and 10% levels

Initially, 32 samples were sent to each of eight analytical laboratories (three U S government, and five private) The 32 samples consisted of two identical sets (i e , duplicates) of the 16 samples discussed above The private laboratories did not know that there were duplicate samples Further, they did not know which, if any, of the samples contained WTC spikes

Subsequently, a second set of 28 *different* background samples was analyzed to obtain a better understanding of the variability of background dusts These 28 samples were sent to only one of the five private laboratories

It was ultimately agreed that each of the laboratories would perform the following three Scanning Electron Microscopy-based (SEM) analyses on each of the samples they received (see Methodology and Data Analysis section)

- Slag wool fiber content (in number of fibers per gram of dust) Slag wool was a significant component of the WTC insulation material
- Calcium-rich particle content (in area percent concentration in the SEM field) Such particles are assumed to be indicative of cement/concrete-like particles
- Gypsum particle content (in area percent concentration in the SEM field) Such particles are assumed to be indicative of "dry wall" (i e , gypsum-containing wall board)

Conclusions:

A number of conclusions can be drawn from the analytical results thus far obtained. It is not expected that data that are subsequently received will substantially change these conclusions. It must be noted that there are several caveats that affect the quality of the data Those are discussed later in this report

2 With respect to calcium-rich particles and gypsum particles, spiked samples cannot readily be distinguished from background samples

Tables 1 and 2 present the analytical data *thus far available* for calcium-rich and gypsum content respectively Analysis was performed using SEM and x-ray mapping (XRM) techniques The shaded areas represent the samples spiked with 1, 5, and 10% WTC dust. The others areas are background samples Sample designations followed by "(1)" and "(2)" are duplicate samples (Samples received by the laboratories had random identification numbers, so that the laboratories did not know if any samples were duplicates, nor did they know if any samples contained WTC dust) In addition, Table 3 is the analysis of a subsequent 28 background samples, analyzed by only laboratory "B" Analysis of calcium-rich and gypsum particles for this sample set is shown on Table 3

The average of all background samples (including the second set of 28 samples) for calcium-rich particles is 22 3 area percent, with a high value of 66 5% and a low value of 4 2% The average for the spiked samples is 20 7%, with the highest value being 25 9% The 1, 5, and 10% spiked samples do not show any trend with respect to calcium-rich particle content (i e., they do not show any increase as the spike level increases).

The average of all background samples (including the second set of 28 samples) for gypsum particles is 11 7 area percent, with a high value of 56 5% and a low value of 0 1% The average for the spiked samples is 9 3%, with the highest value being 32 8% The 1,5, and 10% spiked samples do not show any trend with respect to gypsum particle content

3. With respect to slag wool content in the samples spiked with the first of the two WTC dusts, spikes at the 10% level may be statistically identifiable as WTC-contamination, although spikes at or below the 5% level are probably not identifiable

Table 4 presents all the analytical data thus far available for SEM slag wool fiber analysis (as the number of slag wool fibers per gram of dust). The shaded areas represent samples that are spiked at the 1, 5, and 10% levels with WTC dust Table 3 also presents additional slag wool fiber background-only sample data (next to last column) It can be seen from Figure 1 that for those spiked samples designated as "DB" that at the 5% spike level, the slag wool concentrations probably do not exceed one standard deviation above the average slag wool background concentration (including the Table 3

background data) However, at the 10% spike level, the slag wool concentration typically exceeds one standard deviation (see Figure 2), but never exceeds two standard deviations above the average background sample concentration The average background concentration is about 27,400 fibers per gram. The standard deviation is about 40,100 fibers per gram [1]

It should be noted that there is a trend showing a clear increase in slag wool fiber concentration from the 1% to the 10% spike level (see "DB" sample shaded area on Table 4) However, the numerical values of those concentrations, as noted above, are still less than two standard deviations above the average concentration.

4. With respect to slag wool content, samples spiked with 5% and 10% of the second of the two WTC dusts are easily identifiable as WTC-contaminated Even at the 1% spike level, samples may be statistically identifiable.

The slag wool content data for the samples spiked with the WTC dust shown in Table 4 as "USGS" are easily identifiable As can be seen in Figures 4 and 5, samples spiked with the USGS WTC dust at the 5 and 10% levels are essentially all more than two standard deviations above the average background sample concentration. (Average plus two standard deviations would be about 108,000 fibers per gram.[2]) At the 1% spike level though, WTC dust is more difficult to identify because the slag wool concentrations are mostly between one and two standard deviations above the average background sample (see Figure 3)

5 With respect to slag wool content, clearly, there is a large difference between the two WTC dust spikes used In the "DB"-spiked samples, as noted above, it is expected to be more difficult to determine a significant slag wool fiber concentration difference from background The "USGS"-spiked samples clearly had significantly more slag wool fiber content than the "DB" samples

6 Examining Tables 1, 2, and 4 and the Figures, it can be seen that the analyses for the duplicate samples rarely replicate one another However, the variation between duplicate sample values (i e , intralab) is about half of the variation between individual laboratory values (interlab).[3]

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[1] Background concentration data for this analysis excluded several samples that were known to have high slag wool content, specifically the C1-RTP samples (see Table 4), and samples C2,3,4,5,6 (see Table 3)

[2] Ibid

[3] For slag wool fiber analysis, the average difference between the analyses of duplicates (i e , intralab differences) is about 50% of one standard deviation of the between-laboratories analyses (i e , interlab differences) For both calcium-rich and gypsum particle analysis the average intralab difference is 20% of the interlab difference

Methodology and Data Analysis:

The analytical protocol was developed specifically for this project by one of the government laboratories, and modified by all laboratory participants at a meeting held for that purpose All laboratory participants held weekly conference calls as the analytical program was proceeding to discuss general issues with the protocol Additional modifications were made to the protocol based on those conference calls

The original protocol included analysis by Polarized Light Microscopy (PLM), so data are also available for PLM analysis The PLM analyses were curtailed because it became obvious that PLM could not adequately differentiate between fiber types Further, total fiber concentrations were also determined, both by PLM and SEM methods, but those data are not presented in this report

Caveats:

There are a few factors that may contribute to data uncertainty Nevertheless, it is unlikely that these factors will alter the above major conclusions Some of these factors are as follows:

1 As noted earlier, not all of the analytical data have been received
2 Dust samples were collected by several methods Evaluation of the sampling methodology was not part of the study
3 To determine fiber concentration, fibers were counted using an SEM Different laboratories diluted samples to different levels before counting, introducing some variability of results
4 Laboratory equipment capabilities and personnel skills varied

TABLE 1: SEM X-Ray Mapping – Calcium-Rich Area Percent

Sample Designations	Laboratory Letter Codes								Location Key:
	A	B	C	D	E	F	G	H	
AP5(1)		23 4		20 4	14 4	11 6	30 7	45 8	Chittenden Avenue, Manhattan
AP5(2)		22 4		22 1	16 8	9 8	39 6	48 9	
CMC(1)		27 7		21 8	6 7	7 9	55 1	60 4	Columbia Medical Center, W 68th Street, Manhattan
CMC(2)		34 1		21 5	20 4	10 1	38 9	55 2	
HS3(1)		10 3		14 1	6 4	15 3	29 1	63 1	Teaneck, NJ
HS3(2)		17 8		13 3	14 8	6 5	44 0	49 7	
WGS(1)		22 8		13 2	13 9	7 7	58 4	53 4	Nassau County, LI
WGS(2)		19 9		16 3	5 7	7 4		52 3	
MW(1)		12 2		14 2	12 6	7 6	49 3	46 3	West End Ave Between 105th and 106th Streets, Manhattan
MW(2)		12 0		10 9	8 3	5 7		49 8	
DB1%(1)		18.2	15 6	14.0	18 3	8.9	55.9	50 2	4 Albany Street Spiked into NE Queens background dust
DB1%(2)		13 1		14 1	15.4	9.4		52.2	4 Albany Street Spiked into NE Queens background dust
DB5%(1)		13.4	23 0	16.1	10 8	9.0	40 0	49 0	4 Albany Street Spiked into NE Queens background dust
DB5%(2)		20.5		12 9	4.1	7 5		39.6	4 Albany Street Spiked into NE Queens background dust
DB10%(1)		14.4		15.2	8.6	8.0	50.7	40 6	4 Albany Street Spiked into NE Queens background dust
DB10%(2)		12 6	18.9	14 9	10.8	8 1		48.8	4 Albany Street Spiked into NE Queens background dust
C1-RTP(1)		13 2		11 3	7 5	6 1	57 1	66 5	Research Triangle Park, NC
C1-RTP(2)		16 2		11 9	5 6	4 2		61 0	Research Triangle Park, NC
USGS1%(1)		16.5		17 4	12 4	7.6		43 2	USGS Dust Spiked into NE Queens background dust
USGS1%(2)		21 0	26.6	11 1	5 7	7 4		41.9	USGS Dust Spiked into NE Queens background dust
USGS5%(1)		14 8		14.2	11 9	6.7		53 6	USGS Dust Spiked into NE Queens background dust
USGS5%(2)		14 6	26.4	16 2	10.7	8 3		51.8	USGS Dust Spiked into NE Queens background dust
USGS10%(1)		17.0		15.8	10 9	8.9		40 0	USGS Dust Spiked into NE Queens background dust
USGS10%(2)		17 9		12 1	9 8	8 3		45.1	USGS Dust Spiked into NE Queens background dust
USC(1)		12 3		11 1	19 5	5 4	43 5	46 1	Federal Courthouse, White Plains, NY
USC(2)		9 4		9 5	6 6	7 1		40 3	
FP(1)		13 0		11 6	5 9	6 2	42 4	70 9	Federal Courthouse, Central Islip, LI

FP(2)		10 5		10 3	10 3	8 2		61 5	
MUNYC1(1)		25 4		17 1	15 2	6 3	55 6	39 6	Northern Manhattan, Above 70th Street
MUNYC1(2)		19 7		14.0	31 9	8 0		36 6	
MUNYC2(1)		20 4		19 9	27 7	8 9	45 4	56 8	Northern Manhattan, Above 70th Street
MUNYC2(2)		17 6		14 4	13 6	7 3		57 8	

Samples spiked with WTC dust, at 1, 5, and 10% levels are shaded. All others are background samples

TABLE 2: SEM X-Ray Mapping - Gypsum Area Percent

Sample Designations	Laboratory Letter Codes							
	A	B	C	D	E	F	G	H
AP5(1)		8 0		14 4	0 9	2 5	34 1	26 1
AP5(2)		20 3		11 3	1 8	1 6	31 3	33 7
CMC(1)		4 3		4 8	0 2	1 1	26 1	22 4
CMC(2)		6 9		3 0	1 0	1 0	30 8	17 6
HS3(1)		5 9		9 2	0 3	5 7	44 0	42 9
HS3(2)		14 9		11 0	2 3	1 5	29 0	40 5
WGS(1)		2 9		5 4	0 2	0 4	19 0	42 2
WGS(2)		6 1		4 7	0 2	0 3		39 1
MW(1)		3 8		7 0	0 2	0 7	23 2	37 6
MW(2)		5 4		5 3	0 1	1 1		41 6
DB1%(1)		7 2	13.8	5.7	3.0	0.6	22.0	28.0
DB1%(2)		7 1		5.2	1.1	1.3		30.0
DB5%(1)		7.3	3.4	5 5	0.7	1.2	29 1	24.3
DB5%(2)		6 1		5.5	0.1	1.6		28.9
DB10%(1)		6 5		7.8	0.5	1.0	25.7	27.0
DB10%(2)		5 0	8.7	4.8	0.6	1 9		28.5
C1-RTP(1)		8 5		9 7	0 2	1 3	24 5	53 4
C1-RTP(2)		8 7		8 2	0 3	0 8		50 4
USGS1%(1)		6 3		5.8	1.0	0.9		29.4
USGS1%(2)		5 4	15 2	4.1	0.2	0 9		29.2
USGS5%(1)		7 7		5.7	0 9	1.1		29.3
USGS5%(2)		2 5	9.8	4.1	0.5	2.4		21.7
USGS10%(1)		6 3		7.1	1.2	1 1		30.9
USGS10%(2)		4.8		4.8	0.7	1.4		32.8
USC(1)		4 8		5 2	1 2	0 7	24 9	27 1
USC(2)		6 2		4 2	0 2	2.4		32 4

Location Key:

Chittenden Avenue, Manhattan

Columbia Medical Center, W 68th Street, Manhattan

Teaneck, NJ

Nassau County, LI

West End Ave Between 105th and 106th Streets, Manhattan

4 Albany Street Spiked into NE Queens background dust
4 Albany Street Spiked into NE Queens background dust
4 Albany Street Spiked into NE Queens background dust
4 Albany Street Spiked into NE Queens background dust
4 Albany Street Spiked into NE Queens background dust
4 Albany Street Spiked into NE Queens background dust
Research Triangle Park, NC
Research Triangle Park, NC
USGS Dust Spiked into NE Queens background dust
USGS Dust Spiked into NE Queens background dust
USGS Dust Spiked into NE Queens background dust
USGS Dust Spiked into NE Queens background dust
USGS Dust Spiked into NE Queens background dust
USGS Dust Spiked into NE Queens background dust
Federal Courthouse, White Plains, NY

FP(1)		11 6		5 4	0 3	1 2	24 5	56 5	Federal Courthouse, Central Islip, LI
FP(2)		4 4		6 1	0 6	1 5		40 0	
MUNYC1(1)		10 5		9 2	1 2	0 9	26 8	24 1	Northern Manhattan, Above 70th Street
MUNYC1(2)		3 0		5 5	1 4	1 0		26 3	
MUNYC2(1)		5 5		6 1	9 2	2 5	31 0	30 8	Northern Manhattan, Above 70th Street
MUNYC2(2)		4 2		6 0	0 7	1 8		29 5	

Samples spiked with WTC dust, at 1, 5, and 10% levels are shaded. All others are background samples

Table 3: New York City Background Dust Samples

Sample No.	EPA Sample ID	SEM XRM		SEM (Heavy Loading)		Particle Count		
		Calcium-Rich (area %)	Gypsum (area %)	Slag Wool (fibers/g)*	Total Fibers (fibers/g)	Slag Wool	Total Fibers	
1	HS1-06-01**	5 8	5 8	35,565	104,603	9	25	Stony Brook, LI
2	HS1-06-02	16 3	4 0	230,769	523,077	15	34	
3	AP2-07-01	12 4	8 5	32,432	113,514	6	21	West End Ave between 72nd and 73rd Streets
4	AP2-07-02	9 9	7 2	7,692	130,769	2	34	
5	AP3-08-01	10 5	4 7	12,500	212,500	2	34	30th Ave between 21st and 23rd, Queens
6	AP3-08-02	17 3	7 6	<3,636	21,818	0	6	
7	HS2-09-01	25 6	9 6	7,605	22,814	2	6	70th Street between 20th and 21st Ave, Brooklyn
8	AP4-10-01	13 1	11 5	42,857	485,714	3	34	79th St between York and East End Ave, Manhattan
9	AP7-14-01	17 5	6 0	3,333	23,333	1	7	92nd Street between Columbus and CPW, Manhattan
10	CMC-17-01	30 5	10 4	4,651	23,256	1	5	Columbia Medical Center, W 168th St, Manhattan
11	HS3-18-01	14 3	9 0	11,858	71,146	3	18	Teaneck, NJ
12	WGS6557	10 2	4 2	34,826	44,776	7	9	Nassau County, LI
13	WGS5826-1	18 1	6 2	15,564	54,475	4	14	Nassau County LI
14	PT152W88	17 5	8 4	17,021	46,809	4	11	88th Street between Amsterdam and Columbia, Manhattan
15	PT152W88-2ndFl	15 8	5 6	19,305	42,471	5	11	88th Street between Amsterdam and Columbia, Manhattan
16	CY321W80	14 7	12 2	30,888	34,749	8	9	80th Street between Riverside and East End Ave, Manhattan
17	MW924WEAve	21 1	10 1	8,097	28,340	2	7	West End Ave between 105th and 106th Streets
18	C2**	7 9	6 1	46,703	102,890	11	24	Research Triangle Park, NC
19	C3	16 8	3 4	170,309	321,696	18	34	Research Triangle Park, NC
20	C4	13 7	3 8	160,772	227,760	24	34	Research Triangle Park, NC
21	C4 (no date)	17 5	8 8	488,372	790,698	21	34	Research Triangle Park, NC
22	C5	16 7	7 2	74,236	148,472	17	34	Research Triangle Park, NC
23	C6	10 0	10 9	280,762	415,039	23	34	Research Triangle Park, NC
24	N-01S	12 3	7 8	369,231	523,077	24	34	Edison, NJ

25	Nevins Ct	16 0	9 6	<4,367	91,703	0	21	Edison, NJ
26	E Curtis Ave**	7 9	9 0	5,173	24,138	2	7	Edison, NJ
27	LBI	7 3	16 3	<3,636	61,818	0	17	Long Beach Island, NJ
28	Mixture	19 1	11 8	7,194	35,971	2	10	NE Queens
Average		**14.9**	**8.1**	**84,709**	**168,837**			
Standard Deviation		**5.5**	**3.0**	**128,759**	**200,808**			
Coeff. Of Variance		**0.4**	**0.4**	**1.5**	**1.2**			

* A fiber count of one fiber was used to calculate the analytical sensitivity for non-detects

** Internal laboratory duplicates were run on these samples The result shown is the average of the two duplicates ("<" samples were assumed to be 0)

TABLE 4: SEM - Slag Wool Fiber Count/Gram of Sample

Sample Designations	Laboratory Letter Codes								Location Key:
	A	B	C	D	E	F	G	H	
AP5(1)	non-det	3,663		non-det	<249	<500	2,470	<7,386	Chittenden Avenue, Manhattan
AP5(2)		<3636		6,980	<667	500	13,910	<7,698	
CMC(1)	non-det	3,448		11,800	<282	<4,500	5,780	<7,241	Columbia Medical Center, W 68th Street, Manhattan
CMC(2)		<3875		9,620	309	667	6,100	<6,289	
HS3(1)	16,393	7,299		19,000	<286	2,750	<6,320	<7,576	Teaneck, NJ
HS3(2)		7,692		18,600	<667	5,060	7,370	34,813	
WGS(1)	5,900	34,221		26,400	<256	1,630	9,480	16,077	Nassau County, LI
WGS(2)		10,753		18,100	6,990	<30,500	3,520	18,399	
MW(1)	12,232	18,939		18,700	1,320	1,000	13,630	17,301	West End Ave Between 105th and 106th Streets, Manhattan
MW(2)		3,717		31,800	893	<45,500	18,080	<9,497	
DB1%(1)	5,747	10,909	5,451	29,900	<2,000	1,920	7,650	15,924	4 Albany Street Spiked into NE Queens background dust
DB1%(2)	34,826	17,422	9,133	27,300	3,770	12,500	1,320	16,038	4 Albany Street Spiked into NE Queens background dust
DB5%(1)	72,562	29,197	32,385	50,800	31,000	1,700	6,230	107,143	4 Albany Street Spiked into NE Queens background dust
DB5%(2)	67,797	25,271	33,646	35,800	6,900	14,700	13,040	70,472	4 Albany Street Spiked into NE Queens background dust
DB10%(1)	104,575	66,421	74,837	113,000	108,000	7,000	12,900	114,638	4 Albany Street Spiked into NE Queens background dust
DB10%(2)	84,746	77,778	57,644	95,100	20,400	34,100	25,210	96,696	4 Albany Street Spiked into NE Queens background dust
C1-RTP(1)	246,914	159,011		269,000	168,000	38,000	84,650	188,088	Research Triangle Park, NC
C1-RTP(2)		173,585		165,000	21,900	160,000	39,930	318,143	Research Triangle Park, NC
USGS1%(1)	98,039	109,091	50,293	119,000	366,000	79,800	9,200	90,992	USGS Dust Spiked into NE Queens background dust
USGS1%(2)		83,032	50,160	104,000	18,700	79,500	25,370	137,363	USGS Dust Spiked into NE Queens background dust
USGS5%(1)	600,000	404,332		681,000	227,900	433,000	66,450	672,926	USGS Dust Spiked into NE Queens background dust
USGS5%(2)		343,284	364,813	146,000	191,000	197,000	73,330	347,904	USGS Dust Spiked into NE Queens background dust
USGS10%(1)	1,218,855	840,231	531,277	1,620,000	1,410,000	629,000	144,120	734,767	USGS Dust Spiked into NE Queens background dust
USGS10%(2)		1,366,470	521,212	238,000	271,000	372,000	33,040	413,153	USGS Dust Spiked into NE Queens background dust
USC(1)	73,394	56,025		91,800	33,700	15,600	<3,230	29,268	Federal Courthouse, White Plains, NY
USC(2)		41,199		40,700	7,890	48,400	3,540	74,212	
FP(1)	18,519	18,051		16,300	1,100	12,400	11,920	28,249	Federal Courthouse, Central Islip, LI
FP(2)		16,470		31,800	3,920	30,500	<1,181	25,489	

MUNYC1(1)	10,840	7,220		14,400	14,900	13,100	<2,545	6,803	
MUNYC1(2)		3,745		20,200	1,960	<22,300	<1,228	41,118	Northern Manhattan, Above 70th Street
MUNYC2(1)	41,298	28,777		66,500	1,390	17,800	<12,453	123,106	
MUNYC2(2)		48,507		45,500	24,200	30,500	2,330	59,473	Northern Manhattan, Above 70th Street

Samples spiked with WTC dust, at 1, 5, and 10% levels are shaded. All others are background samples.

For data analysis purposes

- Non-det = Non-detect – zero slag wool fibers were noted in the sample
- <# indicates that the value was less than the detection limit of the respective laboratory When this result was reached, the value was divided by the square root of 2.

Table 5: SEM - Slag Wool Fiber Count

Sample Designations	Laboratory Letter Codes								Location Key:
	A	B	C	D	E	F	G	H	
AP5(1)	0	1		0	0	0	2	0	Chittenden Avenue, Manhattan
AP5(2)		0		3	0	1	6	0	
CMC(1)	0	1		5	0	0	4	0	
CMC(2)		0		4	1	0	5	0	
HS3(1)	3	2		8	0	1	0	0	Teaneck, NJ
HS3(2)		2		8	0	3	4	4	
WGS(1)	1	9		11	1	1	6	2	Nassau County, LI
WGS(2)		3		8	7	0	3	2	
MW(1)	2	5		8	6	1	6	2	
MW(2)		6		14	2	0	22	0	
DB1%(1)	1	3	1	13	0	1	7	2	4 Albany Street Spiked into NE Queens background dust
DB1%(2)	7	5	2	12	4	2	1	2	4 Albany Street Spiked into NE Queens background dust
DB5%(1)	8	8	7	22	7	1	6	6	4 Albany Street Spiked into NE Queens background dust
DB5%(2)	12	7	7	16	8	2	11	10	4 Albany Street Spiked into NE Queens background dust
DB10%(1)	16	18	12	48	13	2	10	13	4 Albany Street Spiked into NE Queens background dust
DB10%(2)	15	21	12	42	9	3	25	12	4 Albany Street Spiked into NE Queens background dust
C1-RTP(1)	20	45		116	16	4	22	24	Research Triangle Park, NC
C1-RTP(2)		46		72	30	4	17	37	Research Triangle Park, NC
USGS1%(1)	15	30	9	54	27	11	11	10	USGS Dust Spiked into NE Queens background dust
USGS1%(2)		23	11	47	23	4	22	15	USGS Dust Spiked into NE Queens background dust
USGS5%(1)	99	112		194	25	>20	64	43	USGS Dust Spiked into NE Queens background dust
USGS5%(2)		92	62	65	21	19	27	39	USGS Dust Spiked into NE Queens background dust

USGS10%(1)	181	45	124	450	38	19	18	41	USGS Dust Spiked into NE Queens background dust
USGS10%(2)		45	129	105	19	16	9	49	USGS Dust Spiked into NE Queens background dust
USC(1)	6	13		39	6	13	0	3	
USC(2)		11		18	6	4	2	8	
FP(1)	3	5		7	3	2	2	3	
FP(2)		5		14	4	1	0	3	
MUNYC1(1)	1	2		6	4	3	0	1	
MUNYC1(2)		1		9	2	0	0	5	
MUNYC2(1)	7	8		28	3	3	0	13	
MUNYC2(2)		13		20	24	3	1	7	

Samples spiked with WTC dust, at 1, 5, and 10% levels are highlighted in yellow.

ADDENDUM TO VERSAR REPORT: SEM CALIBRATION DATA

MEMORANDUM

TO. Jacky Rosati

CC. David Friedman

FROM Stephen Schwartz

DATE August 5, 2005

SUBJECT BIR-1G Sample Analyses

Identical mounted and polished reference samples, each designated BIR-1G, were sent to each of the five private laboratories participating in the analyses of dust samples from New York City and elsewhere The samples were analyzed by Scanning Electron Microscopy/Energy Dispersive X-ray Spectrometry (SEM/EDX) to determine their elemental content The purpose of the study was to determine the variation within and between each of the laboratories, and to assess their ability to identify elements using this technology

Each of the five laboratories analyzed their BIR-1G sample between 4 and 11 times, as convenient (there was no requirement for a specific number of analyses). The average elemental concentration data for each laboratory is presented in the attached table For calcium (Ca), magnesium (Mg), silicon (Si), and oxygen (O)[4], which constitute over 80% by weight of the elemental composition, the standard deviation *within* each laboratory, for each element, was typically much less than 10% (i.e, the coefficient of variation) Likewise, the coefficient of variation *between* laboratories for Ca, Mg, Si, and O, as shown on the attached table, was also much less than a 10% (The graphic presentations of the EDX spectra within and between laboratories also appear to be extremely similar.

Therefore, it can be concluded that each of the laboratories was easily able to achieve excellent precision, by SEM/EDX, in quantifying the elements that were present in larger concentrations

[4] Some of the laboratories reported results as the weight percent of the elemental oxides, specifically Na_2O, MgO, Al_2O_3, SiO_2, CaO, TiO_2, FeO, K_2O, and MnO_2 Oxide values were converted to individual elemental values (e g , Al_2O_3 is about 53% Aluminum, and 47% oxygen by weight)

AVERAGE ELEMENTAL CONCENTRATION REPORTED FOR BIR-1G SAMPLES (Weight Percent of Sample)

Lab	Sodium	Magnesium	Aluminum	Silicon	Calcium	Titanium	Iron	Potassium	Manganese	Oxygen
A										
B										
C										
D										
E	0 44	5 02	8 11	21 60	9 66	0 68	9 68	NR	NR	44 84
F										
G	1 14	5 82	8 75	24 76	7 68	0 52	5 56	0 00	0 00	45 78
H	1 84	5 10	7 88	22 20	10 30	0 64	8 37	0 03	0 21	43 43
AVERAGES	1 14	5 31	8 25	22 85	9 21	0 61	7 87	0 02	0 11	44 68
Standard Dev. (% of Average)	61 40	8 33	5 48	7 35	14 83	13 58	26 73	141 42	141 42	2 65

NR - Not Reported

APPENDIX F: STATISTICAL ANALYSIS AND INTERPRETATION OF TEST RESULTS – LABORATORY QUALIFICATION

Slag wool fiber content as a discriminator for residual WTC contamination in indoor dust sample: Interpretation of multi-laboratory test results

Introduction

Eight laboratories were each challenged with a number of blinded dust sample aliquots to determine number of slag wool fibers per gram These samples included background dusts from various locations in NYC and a series of samples of common household dust spiked with different levels of WTC collapse dust collected in 2001 or in 2004 The purpose of this endeavor was to assess whether or not the method developed can be used by qualified laboratories to discriminate between WTC and non-WTC impacted dust samples.

In the following discussion, individual laboratories are evaluated and ranked for validity and precision, and then the top performers are further evaluated as groups to determine the expected confidence level for the slag wool content of any individual and randomly assigned)sample.

Laboratory Qualification

Validity: Assessment of validity was conducted by analysis of a series of spiked samples where the expected response ratios are known. The challenge samples consisted of a large volume of non-impacted background dust collected in 2004 from locations in Northeast Queens over ten miles from the WTC site This dust was subsequently spiked with 1, 5, and 10% WTC dusts by weight using either bulk collapse dusts collected in September 2001 immediately following the disaster (designated as USGS dust), or nominally undisturbed dusts collected in 2004 in the abandoned Deutsche Bank (DB) complex that borders the south side of the WTC complex (designated as 4 Albany dust).

Using units of (# slagwool fibers)/(gram of dust), preliminary analyses showed a mean value of 12,200,000 (s d 1,697,056) for USGS dust, 579,667 (s d 173,782) for 4 ALBANY dust, and a nominal background level of 7,190 Based on these data, the expected values of slope expressed as [(# slagwool fibers)/(gram dust)]/[% spike level] are 121,928 and 5,725, respectively for USGS and 4 Albany spikes Specifically, each lab was furnished two samples each of 1, 5, 10% spikes from both 4 Albany and USGS series for a total of 12 spiked samples plus a series of 20 additional background samples collected from random locations all over the greater NYC area

Scatterplots and linear least squares regressions were constructed for each lab and for each of the two spike series Preliminary inspection showed no apparent violations of underlying assumptions required for regression analysis (primarily homogeneity of variance), as such no lognormal transformation was performed. Using a forward selection strategy, it was found that a higher order polynomial model does not statistically improve the linear fit, this is expected as the sample set is designed as a linear progression Also, a simpler but more general "runs" test for each linear regression confirmed these results Data handling and manipulation was performed with Microsoft Excel SP-2, statistical analyses were performed with SAS 9.1.3 XP-Pro (proc rsreg/lackfit, proc reg, proc

mixed, and proc univariate); graphing, ANOVA, and various other statistical results were performed or verified with GraphPad Prism 3 03 The linear regression results are given in Table 1, a summary of SAS proc rsreg/lackfit results are given in Table 2

Table 1· Summary of linear least squares regression results

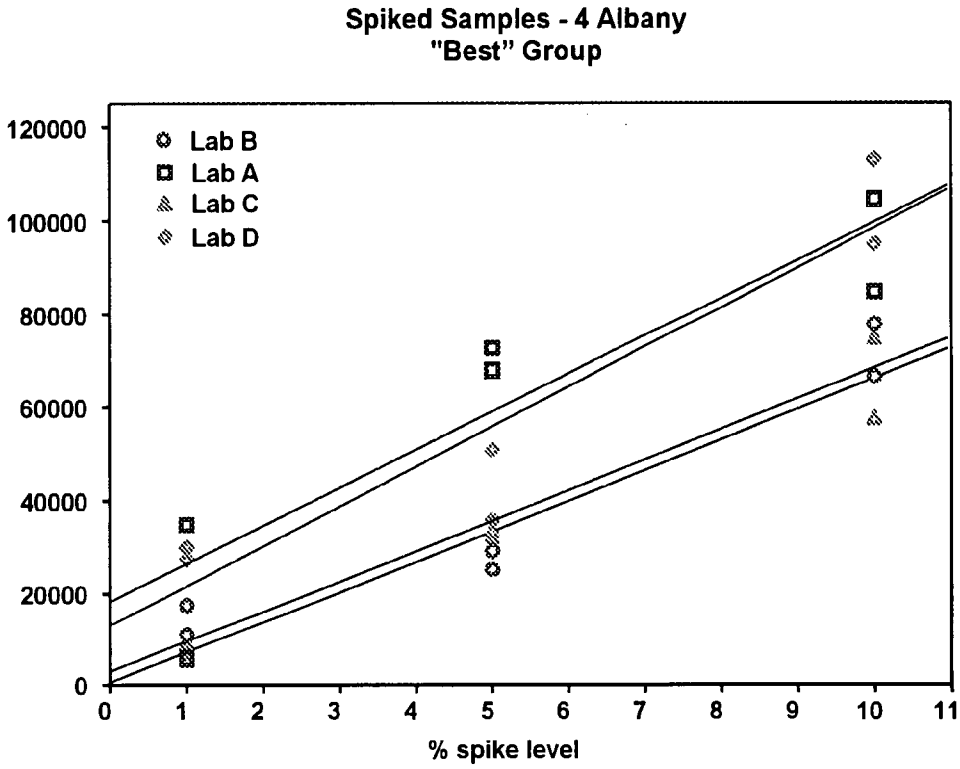
Lab	DB spikes (4 Albany)				USGS spikes (2001 dusts)				
	slope	95% CI (+/-)	sig slope p-value	r2	slope	95% CI (+/-)	sig slope p-value	r2	
A	8126	1760	0.0099	0.8421	124500	488	0.0025	1 0000	note: n =3
B	6541	971	0.0025	0 9190	113300	23460	0.0085	0 8537	
C	6554	691	0.0007	0 9573	52380	5914	0 0030	0.9632	
D	8538	1515	0 0049	0 8881	91340	58230	0.1918	0.3608	
E	6936	3632	0 1288	0 4769	74240	49810	0.2104	0.3571	
F	1523	1288	0.3027	0 2588	45370	14020	0 0298	0 7321	
G	1630	610	0.0559	0 6404	7750	4607	0.1678	0.4144	
H	9695	2645	0.0215	0.7705	49510	21790	0.0855	0 5634	

Table 2 Summary of "lack of fit" tests

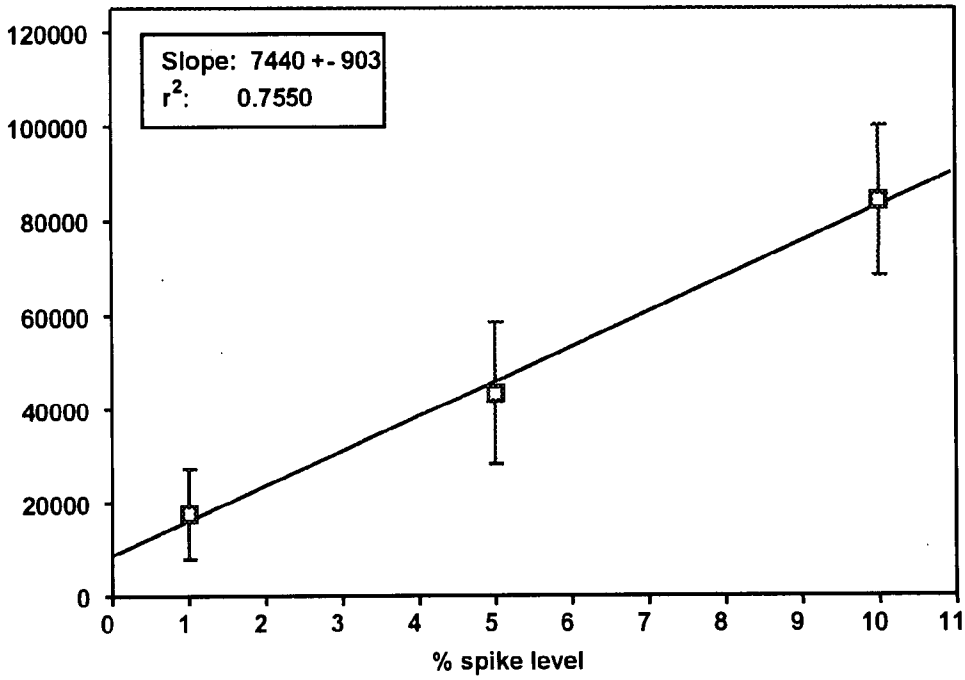
Lab	r2 linear	r2 quad	p-value linear	pvalue quad
A	0 8421	0 0590	0 0149	0 2732
B	0.9190	0 0564	0.0018	0 0791
C	0.9573	0 0001	0 0036	0 9441
D	0.8881	0.0706	0.0040	0 1087
E	0 4769	0.0197	0.1904	0 7547
F	0 2588	0 0445	0 3386	0 6913
G	0.6404	0 0069	0.1018	0.8240
H	0.7705	0 1455	0.0135	0 1070
A B,C,and D	0 7550	0.0032	0 0001	0 6038
A,B,C,D and H	0 7027	0.0023	0.0001	0 6473

Based on these summaries the linear model is appropriate Laboratories A and B demonstrate excellent performance across the board each has r^2 values > 0 80, significant positive slopes with $p < 0\ 05$, and slopes with the expected magnitude We caution that Lab A only has three points for the USGS spike results (yellow highlights, Table 1) Fields highlighted in blue indicate potential problem areas If only the 4 Albany spike series are considered, then Labs C and D can be added to the preferred performer group This is reasonable because the range covered here is more likely to reflect the range of concern for unknown samples Although the Lab H results demonstrate a lower r^2 value and a larger 95% CI for slope, this is caused by a single outlying point. As such, there is no reason to exclude Lab H from the analysis Because Labs E, F, and G fail in more than one category in both spiked data sets, they are not included in the remaining study analysis

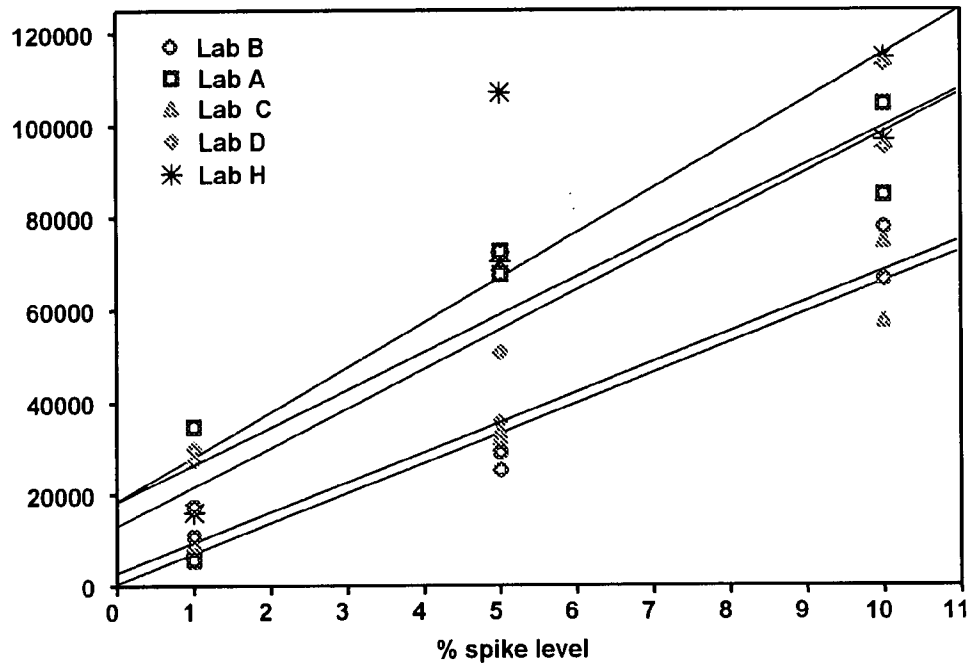
From the above discussion, we can construct two groups of laboratories based on the estimated validity of their results: "Best", consisting of Labs A, B, C, and D and "very good" consisting of the best group plus Lab H. In the following series of figures, the individual and composite linear regression results for the groups are demonstrated graphically.



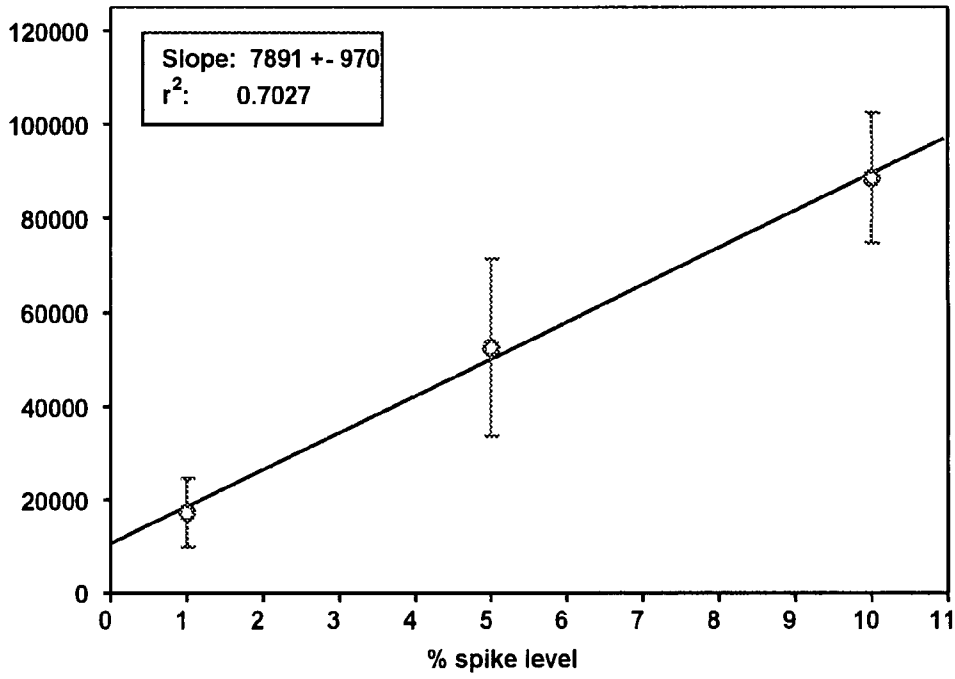
Spiked Samples - 4 Albany
"Best" Group Combined
(Labs A, B, C, and D)



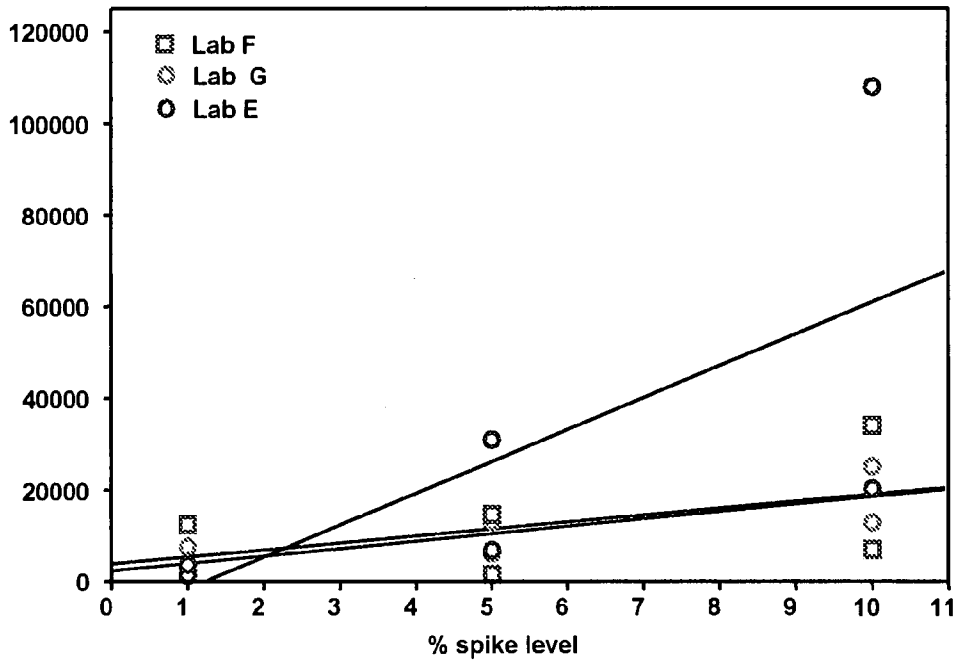
Spiked samples - 4 Albany
"Very Good" Group
(Labs A, B, C, D and H)

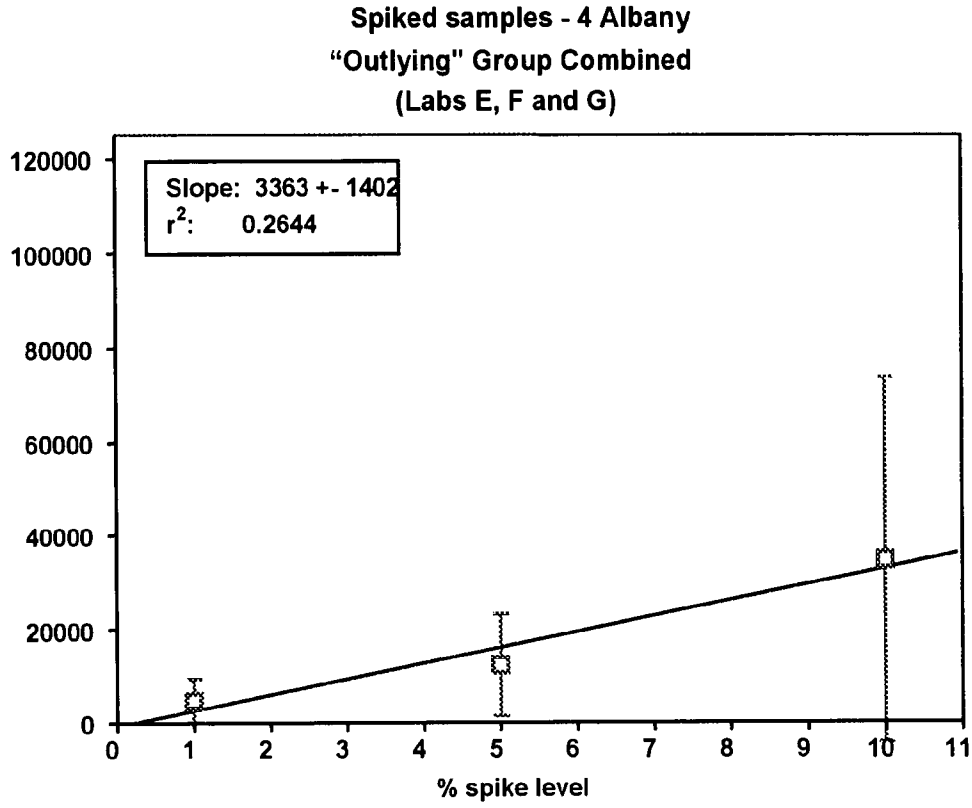


Spiked samples - 4 Albany
"Very Good" Group Combined
(Labs A, B, C, D and H)



Spiked samples - 4 Albany
"Outlying" Group
(Labs E, F and G)





Precision: Up until this point, validity has been assessed only with those samples for which there is some prior knowledge of content. For assessing precision, however, one can use all of the samples (including unknowns) because each laboratory received aliquots of the same set of 32 samples. Furthermore, the sample structure is such that these 32 samples are comprised of 16 paired samples allowing within laboratory precision estimates as well. Although there are a number of statistical options for proceeding, an analysis of variance (ANOVA) and intra-class correlation coefficients (ICC) are pragmatic for these circumstances as samples and laboratories are used in groups. Preliminary analyses of "within" and "among" laboratory results indicate that the underlying distributions (considering all 32 sample results) are not normal based on the Shapiro-Wilk (S-W) test, and that natural log transformation of the data should used to perform analysis of variance. The only exception is the USGS data set where only three pairs of samples are reported and thus the natural space numbers did not require transformation. Table 3 shows the results for the ICC analyses within laboratories, and also for the groups (Labs A, B, C, D) and (Labs A, B, C, D, H) aggregated. The variance components and p-values for the S-W normality test are also given. The lower part of Table 3 gives the aggregated results for the background samples only; Laboratories A and C did not contribute to these statistics but it is expected that they would perform similarly.

Table 3 Summary statistics for intra-class correlation coefficients

All available Pairs

IntraClass Correlation Calculations: from SAS proc mixed
Log Space data

Lab	n obs	Int	Res	ICC	S-W p-value
A	6	7.6570E-01	5.4910E-01	0.5824	0.2786
A^	6	1.3310E+09	2.1025E+08	0.8636	0.7679
B	32	2.7493E+00	1.7470E-01	0.9403	0.1059
C	10	1.9870E+00	7.3050E-01	0.7312	0.0342
D	31	1.3966E+00	2.7860E-01	0.8337	0.8847
E	32	5.5988E+00	1.7150E+00	0.7655	0.6012
F	32	2.8559E+00	1.3172E+00	0.6844	0.6057
G	32	1.0962E+00	6.7860E-01	0.6176	0.8571
H	32	2.0647E+00	3.2810E-01	0.8629	0.1571
A,B,C,and D	80	2.0491E+00	3.0250E-01	0.8714	0.3296
A,B,C,D and H	112	2.0820E+00	2.8740E-01	0.8787	0.1540

^ natural space

All NYC Background Pairs

IntraClass Correlation Calculations: from SAS proc mixed
Log Space data

Lab *	n obs	Int	Res	ICC	S-W p value
A,B,C,and D	36	6.4570E-01	3.5070E-01	0.6480	0.2657
A,B,C,D and H	54	6.9800E-01	3.4290E-01	0.6706	0.2571

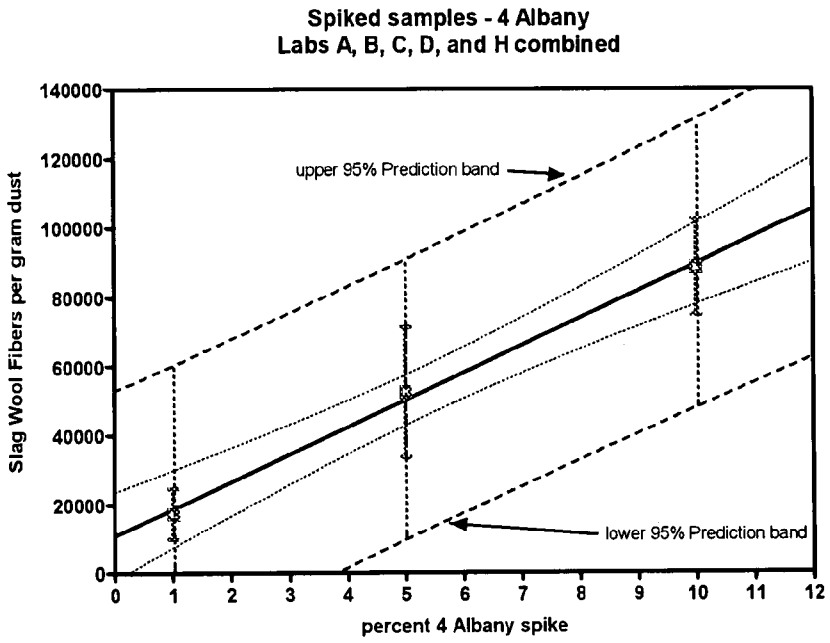
*Laboratories A and C did not report paired New York City background data

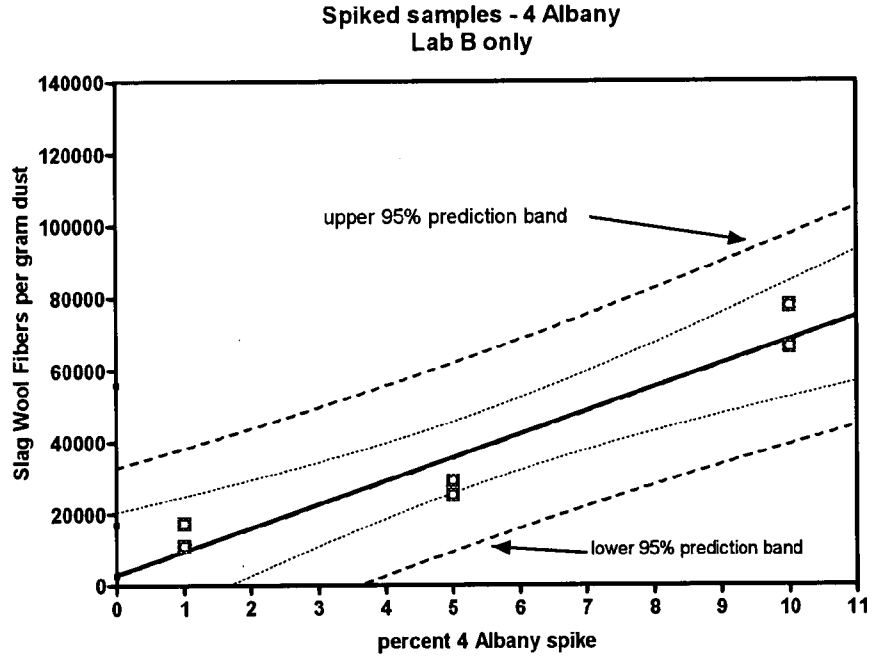
From this exercise, we see that all of the individual laboratories demonstrate reasonable ICCs (generally above 0.6) Furthermore, the laboratory groups chosen to demonstrate good validity show ICCs greater than 0.87 when all data are considered. When only the New York City background samples are analyzed, the ICCs are somewhat lower. These results can be interpreted to mean that about 35% of the variance is attributable to variability in the pooled laboratory analyses, and the remainder to true differences among the background samples

As a further assessment of inter-laboratory precision, the between laboratory ANOVA shows no reason to reject the null hypothesis (Ho = no difference, in natural log space) among Laboratories A, B, D, and H Laboratory C was left out of this analysis because they reported no background data at all

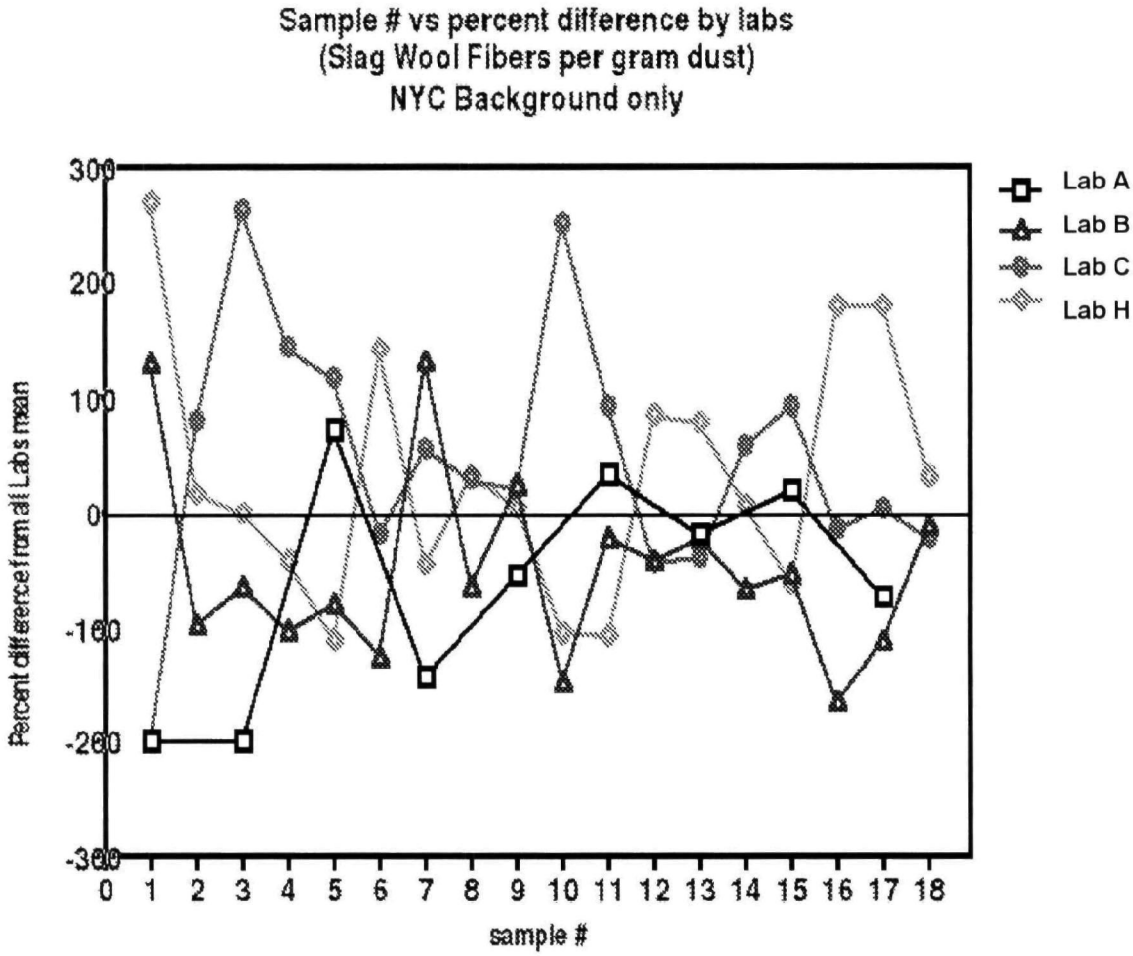
Evaluation of Unknown Samples

In the previous section we qualified a group of laboratories for measurement of unknowns based on spiked samples (validity), and comparative precision measures based on ICC and ANOVA. We now assume that these laboratories are statistically similar and combine their spike results into a single response graph. Based on these results, we calculate 95% confidence intervals and 95% prediction bands as illustrated in the figures below.





The major effort here is to estimate the performance of the aggregate laboratory group (A, B, C, D, and H) with respect to the group of samples from the greater New York City area designated as "background" or "non-WTC impacted". The composite behavior of these samples is illustrated below with respect to the analytical laboratories. The graph indicates no consistent (high or low) percent bias from the cross laboratory means. This confirms the conjecture made earlier that these laboratories are statistically similar. We caution that Laboratory C did not provide any background data at all and could not be directly included here, however, it is assumed that it would behave like the others.



The next step is to assess how an individual (presumably unknown) dust sample assay relates to the amount of spiked 4 Albany dust percentage. Given the graph and underlying statistics of the above figure entitled "Spiked Samples – 4 Albany, Labs A, B, C, D and H Combined), one can calculate the x-value in % spiked 4 Albany equivalent and the 95% confidence interval for the prediction for any unknown sample measurement from any laboratory. This is essentially the use of the prediction band graph above in reverse. As such the prediction of "x" and the CI take the following form:

$$X_{predicted} = (Y_{bar} - a)/(b)$$

$$CI = X_{predicted} \pm [t(RSE)/b] * \{1/m + 1/n + [(Y_{bar} - y_{bar})^2/(b^2(n-1)s_x^2)\}^{1/2}$$

where Y_{bar} is the mean laboratory measurement, a and b are the intercept and slope of the regression, t is the critical t-value for n-2 degrees of freedom, RSE is the residual standard error, m is the # of replicate measurements, n is the number of calibration points, y_{bar} is the mean of the regression y data, and s_x is the standard deviation of the x values of the regression data.

The reported slag wool results for the background samples can now be interpreted Table 5 shows the results for each background sample measurement across all participating laboratories as a prediction of the percent equivalent 4 Albany spike level and half of the 95% confidence interval associated with the measurement There are a total of 63 measurement results in the table

Table 5· Results for each background sample across all participating laboratories as a prediction of the percent equivalent 4 Albany spike level and ± 95% confidence interval.

Lab*	A		B		D		H	
Sample #	Percent	CI +-	Percent	CI +-	Percent	CI +-	Percent	CI +-
1	-1 35	5 43	-0 89	5 40	-1.35	5 43	-0 69	5 38
2			-1 03	5 41	-0.47	5 37	-0 66	5 38
3	-1 35	5 43	-0 92	5.40	0.14	5 33	-0 71	5 39
4			-1 01	5 41	-0 14	5 35	-0 79	5 39
5	0 72	5 29	-0.43	5 37	1 05	5 28	-0 68	5 38
6			-0 38	5 36	1.00	5 28	3 06	5 20
7	-0 61	5.38	2 98	5 20	1 99	5 23	0 68	5 30
8			0 01	5 34	0 94	5 28	0 98	5 28
9	0 20	5 33	1 05	5 28	1 02	5 28	0.84	5 29
10			-0 88	5 40	2 68	5 21	-0 50	5 37
25	7 95	5 21	5 75	5 17	10.28	5 31	2 35	5 22
26			3 87	5 18	3.80	5 18	8 05	5.21
27	0 99	5 28	0.93	5 28	0 71	5 30	2 23	5 22
28			0 73	5.29	2.68	5 21	1.88	5.24
29	0 02	5 34	-0 44	5.37	0 47	5 31	-0.49	5 37
30			-0.88	5 40	1 21	5 27	3 86	5 18
31	3 88	5 18	2.29	5 22	7.07	5 18	14 25	5.63
32			4.79	5 17	4 41	5 17	6 18	5 17

*Laboratory C was left out of this analysis because they reported no background data

We note that negative entries above are only statistical constructs Of the 63 background measurements in this table, 7 (or about 11%) exceed the 4 Albany 5% spike level, 2 of the 63 measurements exceed the 10% 4 Albany spike level If the upper confidence limits are considered, 42 out of 63 (67%) exceed the 5% spike level and 7 of 63 (11%)

exceed the 10% spike level For instance for sample 25 at lab A the percent equivalent of the fiber measurement is 7 95% and the upper confidence limit is 7 95% + 5 21% = 13 16%

As a further exercise, we calculated the same statistics for data within only one laboratory (choosing Laboratory B as the example), these results do not include scatter in the regression from the other qualified laboratories Here we find some improvement we see 3 of 18 values (16 7%) exceed the 5% 4 Albany dust level and 0 of 18 values exceed the 10% 4 Albany dust level For the upper confidence levels, 6 of 18 values exceed the 5% 4 Albany dust level and 1 of 18 values exceed the 10% 4 Albany dust level.

Conclusions

The conclusions are based solely on the analytical data provided from the laboratory test and a few analyses of the 100% WTC spike samples From validity estimates based on expected slopes and data scatter of WTC spiked samples, five of eight laboratories (A, B, C, D, and H) were used for further analysis Intra-class correlation coefficients (with natural log transformation) for individual labs and for the group of five demonstrate similar and reasonable values (>0 7) when all available data are considered One-way ANOVA analysis of Laboratories A, B, D, and H results provides no evidence to reject the null hypothesis (that the results are from the same distribution) Laboratory C was not included here because of insufficient reported data but, based on spike sample statistics, it is likely that that they too would fall into this category

Under the practical constraints that the five laboratories are used at random with one analysis per unknown sample, we cannot expect statistical discrimination at the 1% or 5% 4 Albany spike equivalent level because the upper 95% prediction bounds exceeds the 5% spike equivalent level across the board Reasonable discrimination is possible at the 10% 4 Albany spike equivalent level because the lower bound on10% equivalent measurements is approximately equal to the mean at 5% Albany spike equivalent level

A1. Quality Assurance Project Plan for World Trade Center (WTC) Screening Method Study

Project # WTC-1
Revision # 3
August 8, 2005

Prepared by:
U.S. Environmental Protection Agency
Office of Research and Development
Research Triangle Park, NC 27711

Approvals given below indicate that technical and administrative reviews have been conducted, and reviewer comments for the document preceding the signature date have been resolved

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Dr Jacky A Rosati, EPA Principal Investigator, ORD, NHSRC Date

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David Friedman, EPA Principal Investigator & Project Officer, ORD, IOAA Date

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Shirley Wasson, EPA QA Representative, ORD, NRMRL Date

ACRONYMS

COPCs	Contaminants of Potential Concern
DQI	Data Quality Indicator
DQO	Data Quality Objective
EPA	U S Environmental Protection Agency
ERT	Emergency Response Team (EPA)
HEPA	High Efficiency Particulate Air
HVAC	Heating, Ventilation and Air Conditioning
IOAA	Immediate Office of the Assistant Administrator
MQI	Measurement Quality Indicator
NERL	National Exposure Research Laboratory (EPA)
MQO	Measurement Quality Objective
NHSRC	National Homeland Security Research Center (EPA)
NRMRL	National Risk Management Research Laboratory (EPA)
ORD	Office of Research and Development (EPA)
PLM	Polarized Light Microscopy
QA	Quality Assurance
QC	Quality Control
QAPP	Quality Assurance Project Plan
SEM	Scanning Electron Microscopy
SOP	Standard Operating Procedure
TEM	Transmission Electron Microscopy
USGS	US Geological Survey
WTC	World Trade Center

A2. TABLE OF CONTENTS

FIGURES

TABLES

A. PROJECT MANAGEMENT

Presented herein is the site Quality Assurance Project Plan (QAPP) for the World Trade Center Screening Method Validation Study The QAPP has been developed in accordance with the United States Environmental Protection Agency (EPA) guidance for Quality Assurance Project Plans (EPA QA/G-5, EPA/240/R-02/009) December 2002 This plan is based on information currently available and may be modified on site in light of field observations/results and other acquired information Any modifications or deviations from this QAPP shall be approved by EPA and documented

A3. Distribution List

The Distribution List documents who shall receive copies of the approved QAPP and any subsequent revisions or amendments to the QAPP The U.S EPA shall distribute the QAPP to all project team members and shall ensure that the project team members are familiar with any and all QA issues A complete copy of the QAPP and any subsequent revisions shall be maintained on file at the U S EPA RTP office and shall be available upon request. The following personnel will receive copies of the approve QAPP for the WTC Screening Method Study (contact information for these people can be found in A4)·

EPA

1. Jacky Rosati, EPA, ORD, NHSRC, Principal Investigator
2 David Friedman, EPA, ORD, OAA, Principal Investigator
3 Rajeshmal Singhvi, EPA/ERT, WAM – Sampling Contract
4 Teri Conner, EPA, ORD, NERL ,WAM – SEM contract
5. Shirley Wasson, EPA, ORD, NRMRL, APPCD – Quality Assurance

Other Government Agencies

6 Greg Meeker, USGS – IAG Manager

Contractors

7 Cindy Kleinman, Lockheed Martin –Sampling Contractor
8 Bob Willis, Alion Sciences – SEM Contractor
9 Steve Schwartz, Versar – Analysis Prime Contractor
10 Keith Rickabaugh, RJ Lee Group, Inc – Analysis Subcontractor
11 Rich Brown, MVA Scientific Consultants – Analysis Subcontractor
12. Garth Freeman, MAS, Inc – Analysis Subcontractor
13 John Newton, EMSL Analytical Inc – Analysis Subcontractor
14 Jeannie Orr, Reservoir Environmental, Inc – Analysis Subcontractor

A4. Project Organization

A4.1 Responsibilities and Roles

Dr Jacky Rosati of the EPA, ORD, NHSRC and David Friedman, ORD, OAA shall have the oversight authority for all work conducted for this project and shall act as backup and work assignment manager on the Versar analytical contract, respectively Dr Rosati has prepared the Validation Study QAPP, and Shirley Wasson, EPA, ORD, NRMRL, APPCD will perform the QA review of this QAPP Dr Rosati and Mr Friedman shall provide technical assistance to ensure that sample collection and analysis work is completed efficiently, and in compliance with the applicable Scope of Work and all applicable rules.

A4.1.1 Analytical Responsibilities

Steve Schwartz, Versar, shall arrange and oversee the analysis work by analytical laboratories Mr Schwartz and his subcontractors shall adapt, adopt and follow the Quality Assurance Project Plan prepared by EPA for all sample analysis activities performed for this project All appropriate data, original field forms/data sheets, shall be collected and completed in accordance with the instructions contained in the contract and provided to EPA

The analytical laboratories retained by Versar include RJ Lee Group, MVA Scientific, MAS, EMSL Analytical, and Reservoir Environmental Dr Rosati will arrange and oversee the analysis work by the U S government analytical laboratories (Greg Meeker, USGS, Teri Conner, EPA, NERL) All analytical and government laboratories shall conduct the required analysis within the requested turnaround time, input the relevant analytical data into the appropriate spreadsheets, and other similar duties as described in its contract Scope of Work, IAG or as requested by Dr. Rosati All data for this project are considered confidential and only the EPA is authorized to allow for their release

The primary contractor for the environmental sampling shall follow the QAPP that they have prepared entitled "Generic Quality Assurance Project Plan for WTC Residue Sampling New York City, NY, March 2005 (Appendix A). All appropriate data, original field forms/data sheets, and chain-of-custody forms shall be collected and completed in accordance with the instructions contained in the contract and provided to EPA All samples shall be handled as instructed by EPA, to include sample sieving, ashing, splitting, archiving and distributing

A4.1.2 Sampling Responsibilities

Raj Singhvi, EPA, ERT Work Assignment Manager shall oversee the sample collection performed by Lockheed Martin (Cindy Kleinman), as directed by Dr Jacky Rosati. All sampling appointments shall be arranged by Raj Singhvi, EPA/ERT for Lockheed Martin (Cindy Kleinman) All samples are to be archived and stored in a safe location as described in Section 3 1

Lockheed Martin, the primary contractor for the environmental sampling shall follow the QAPP that they have prepared entitled "Generic Quality Assurance Project Plan for WTC Residue Sampling New York City, NY, March 2005 (Appendix A) All appropriate data, original field forms/data sheets, and chain-of-custody forms shall be collected and completed in accordance with the instructions contained in the contract and provided to EPA. All samples shall be handled as instructed by Dr Rosati, to include sample sieving, ashing, splitting, archiving and distributing All data shall be considered confidential Only the EPA is authorized to release any data collected for this project

A4.2 Reporting Relationships

The Project Organizational Chart (Figure 1) shows the reporting relationships between all of organizations involved in this project, including the lead organization (i.e., EPA) and all contractors and subcontractors.

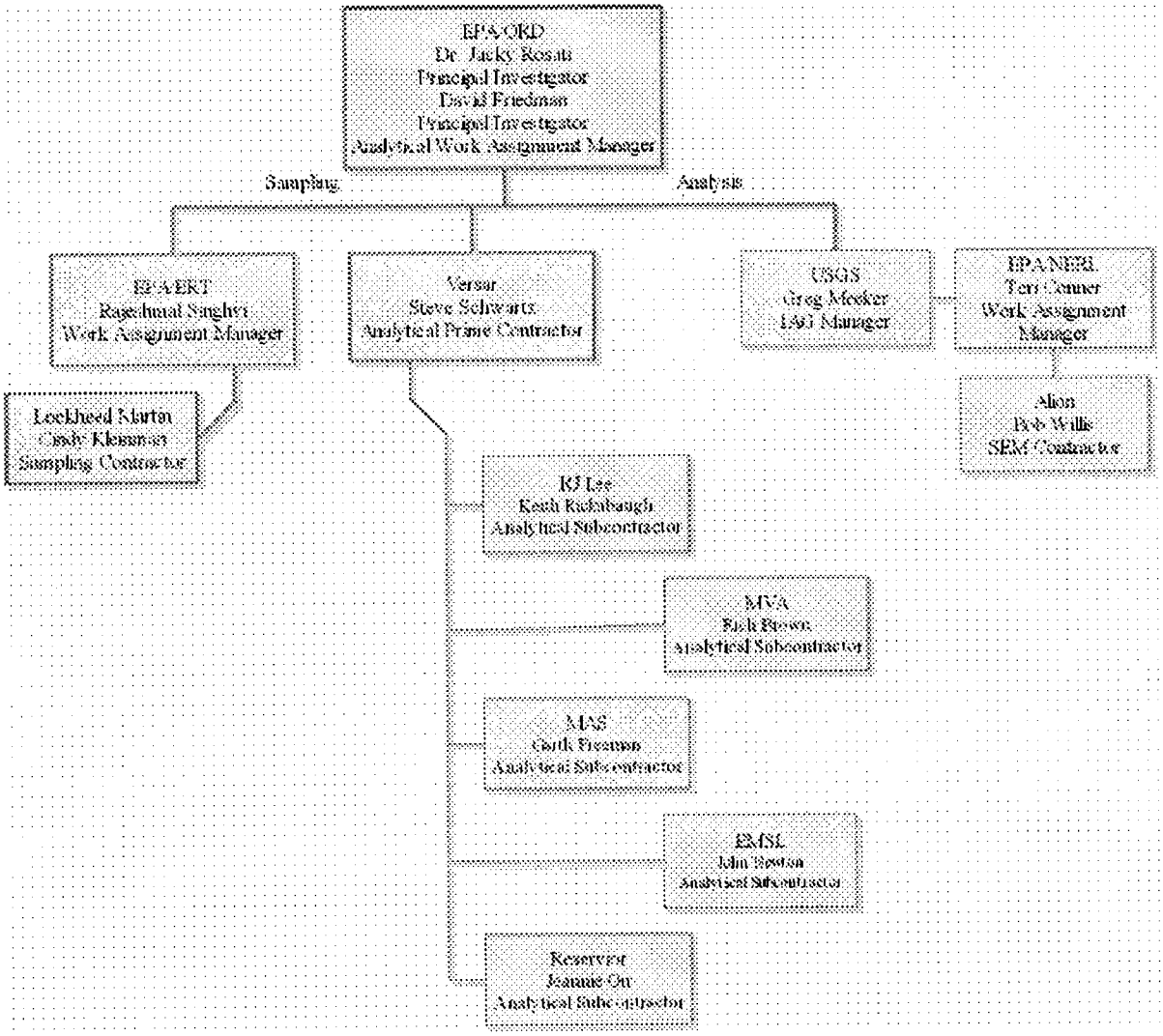

Figure 1: Project Organizational Chart

Contact Information for the above listed personnel (alphabetical):

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A5. PROBLEM DEFINITION AND BACKGROUND

A5.1 Introduction

The objective of this effort is to develop and evaluate a means of determining whether dust sampled as part of EPA's future sampling program contains residual contamination attributable to the collapse of the WTC towers The tested screening method is a critical component of the sampling program as it will be used, along with the results from contaminants of potential concern (COPC) testing, to determine the need for cleanup

The USGS has published two reports which provide the basis for the WTC dust signature adopted in this sampling program The first report discusses the analysis and interpretation of indoor and

outdoor WTC dust samples collected near Ground Zero, days and weeks after September 11, 2001 (Meeker, et al, 2005) From this work, we see that the WTC dust samples are dominated by gypsum, concrete, and man-made vitreous fibers (MMVF), mainly slag wool It is on the basis of these key results that gypsum, elements of concrete, and slag wool were identified as candidates for a WTC signature The second report discusses the analysis of EPA supplied samples taken from several indoor locations well outside of the WTC impacted area (background) These samples were taken between September of 2004 and April of 2005 Slag wool is absent from many of these background samples, but Lowers et al (2005a) state that the samples do have gypsum present, which they speculate might be due to the presence of wall board in the sampled apartments Because of the lack of slag wool in these samples, it was concluded that these samples did not contain WTC dust It was also concluded that perhaps slag wool is the single most critical of the three WTC dust constituents when distinguishing WTC dust from other common dusts.

Other studies also identified MMVF and gypsum as predominant components of WTC dust In a study of air and settled dust quality in apartments in Lower Manhattan, the Agency for Toxic Substances and Disease Registry (ATSDR) and the New York City Department of Health and Mental Hygiene (NYCDOMH) found significantly more MMVF and gypsum in Lower Manhattan apartments as compared to comparison areas above 59th St (NYCDOMH/ATSDR, 2002). Meanwhile, no MMVF was found in comparison locations. They also concluded that gypsum was seen at a higher percentage in dust in Lower Manhattan samples as compared to the comparison area samples In a comprehensive study of the composition of settled dust in the Deutsche Bank building at 130 Liberty St, R J Lee identified numerous hazardous contaminants that were present in the dust at levels much higher than in background office buildings, and among those substances identified in their "WTC signature" were mineral wool and gypsum (R J Lee, 2004).

If the WTC building collapse signature components of slag wool, gypsum, and elements of concrete are not present, then one could conclude that WTC building collapse dust is not present However, since these components might be present in typical New York City dust, as slag wool is a component of insulating materials in currently constructed buildings, it is possible that a test might show them to be present even though WTC dust never impacted the sampled area. A 'screening test' will, by its design, result in some fraction of such false positives (a location without residual WTC dust that tests positive for the above components) However, an appropriate 'screening test' would result in very few, if any, false negatives (a location with residual WTC dust that tests negative for the above components).

A5.2. Method Development

EPA acquired 117 dust samples during the time period of September 2004 to April 2005. Twenty-one 'impacted' samples were taken by the EPA at two buildings that were part of the Deutsche Bank complex located at 130 Liberty Street and 4 Albany Street Both buildings were uninhabited and slated for demolition Fifty samples were taken from locations well beyond the impacted zone, these samples are considered to be 'background' dust Forty-six samples were taken from locations that were possibly impacted but were a bit farther from the WTC site than the known 'impacted' samples None of these forty-six samples are used in the study, but several were evaluated during the development of the analytical method. In addition, one impacted sample was obtained from the USGS. This sample was a composite sample of outdoor and indoor WTC dust collected in September of 2001

While a standard method using a HEPA vacuum collector was used by EPA to collect most bulk dust samples (Appendix C), some bulk dust samples were collected from residential and commercial vacuum cleaner bags. Whether collected by HEPA vac or by acquiring vacuum cleaner bag, all samples are handled (sieved, split and stored) as described in the sampling QAPP in Appendix C Many of the above samples were analyzed for slag wool content by the EPA's National Exposure Research Laboratory (NERL) Scanning Electron Microscopy (SEM) Laboratory This analysis was performed as part of the EPA's development of a standard protocol for sample preparation and analysis (Appendix B), to determine the sample status (background or impacted) and content.

There appears to be a clear distinction between samples taken in impacted areas versus background samples All of the impacted samples had slag wool at concentrations of greater than 100,000 fibers per gram of dust, with a range of 113,000 to 13,400,000, while all of the background samples had concentrations less than 100,000 ppm, ranging from no slag wool identified in10 samples to 92,800 fibers of slag wool per gram of dust Based on this preliminary work, the USGS, the EPA's Office of Research and Development, the EPA's National Enforcement Investigations Center (NEIC), and a number of experts from the commercial testing laboratory community, worked together to develop an analytical method to identify the presence and concentration of the screening constituents (slag wool, gypsum and elements of concrete) in indoor dust This method was reviewed by the WTC Expert Technical Panel's signature subcommittee and is presented in Appendix B The composition of this technical panel can be found at http //www.epa gov/wtc/panel

A.5.3 Screening Method Study

The hypothesis that is the foundation for the WTC dust screening method is as follows If a unit has been impacted, those materials that are found in WTC dust (markers) will be found in the dust collected from the unit. The materials under consideration are. 1) slag wool, 2) elements consistent with concrete, and 3) gypsum Since slag wool is a major component of WTC collapse dust, if a sample does not contain 'significant' levels of this marker, the unit would not be considered to contain WTC residuals The other markers will be used to distinguish samples containing non-WTC slag wool from those containing WTC slag wool. It is expected that data from this study will define the term 'significant level'

Five independent laboratories and three government laboratories will participate in this final method validation phase One government laboratory will analyze only a small portion of the samples, but this lab was critical in the method development. Each laboratory attended a two day session during which the method was further developed and discussed, and procedures to adapt the method to suit each laboratory's equipment were determined Following this session, the laboratories received dust samples consisting of both confirmed background samples (10 samples plus duplicates) and a confirmed non-impacted dust spiked with varying amounts of confirmed WTC dust (6 spiked samples plus duplicates) The spiked dust contains known quantities (concentrations) of the screening materials The labs were provided the samples "blind", thus, they did not know which samples were pure background dust, and which were the spiked dust

While the goal was to validate a method of differentiating between samples of dust that contain residues from the WTC collapse from those that do not, since the three primary materials (slag

wool, and elements of concrete and gypsum) identified above are all normally found in dusts present in the New York area, it is possible that the proposed screen may yield some percentage of false positive identifications of WTC dust

A6. Project/Task Description

A.6.1 Sampling
Dispersion models, photos, interviews, and satellite data were reviewed to discern areas that were likely impacted by WTC emissions and those that were not. Samples to be used to study the above discussed protocol were collected from within both of these areas (background and impacted) Samples were analyzed by EPA's NERL and USGS for content verification, and confirmation of background or impacted status by evaluating levels of slag wool in the dust collected by SEM.

A.6.2 Sample Preparation
WTC dust was spiked into confirmed non-impacted dust at three levels (1, 5, and 10% of total mass) and homogenized The dusts were all characterized by the USGS and U S EPA NERL prior to spiking The two spiking dusts were 1) a composite sample from USGS of predominantly outdoor dust collected in September of 2001, and 2) dust collected by the U S EPA from the Deutsche Bank building at 4 Albany Street in September of 2004 The 4 Albany Street building borders the south side of the WTC complex The USGS performed an analysis of the spiked samples prior to the samples being sent to labs. The spiked samples showed varied levels of slag wool, this was expected due to the difficulty in homogenizing dust containing large fibers, and the fact that components of WTC dust will vary within a sample because of the nature of the source Despite this variability, the measured levels were in the approximate range
expected for the spiking percent (1, 5, and 10%) and, in all but one case, each percent level was fully distinguishable from the other in all but one case

Analysis by USGS, NEIC and NERL determined that the levels of slag wool differs between the two WTC dusts, with the pure dust from 4 Albany Street more than an order of magnitude lower in slag wool than that provided by USGS (approximately 500,000 fibers/gram of dust vs approximately 11,000,000 fibers/gram of dust, respectively) There are likely two explanations for this significant difference in slag wool levels. The USGS sample was a composite of multiple outdoor samples and one indoor sample taken during September of 2001 The 4 Albany sample was taken three years post 9/11 in September of 2004 This sample was taken exclusively inside of a building, thus, the dust was not only diluted by three years of urban background dust, but was also characteristic of dust that had penetrated the shell of an unopened building as opposed to that dropping on the ground outside

A.6.3 Analytical Study
Five independent laboratories were recruited for a final test of the screening method Each laboratory attended a two day session during which the method was further developed and discussed, and procedures to adapt the method to suit each laboratory's equipment was determined These laboratories have received 32 of the collected dust samples consisting of both confirmed background samples and confirmed background samples spiked with varying amounts of confirmed WTC dust (Sample Distribution Table shown in Section B 5) The spiked dust contained known quantities (concentrations) of the screening materials and reasonable homogeneity was confirmed by USGS The labs were provided the samples "blind", thus, they did

not know which samples were pure background dust, and which were background dust samples spiked with WTC dust The labs have several weeks to analyze all dust samples. They have been asked to provide data as to the quantity of screening materials present in the dust in a standardized format (Appendix B). The final data from all laboratories will be evaluated to determine if they were able to distinguish background samples from WTC spiked samples In addition, criteria such as time for analysis, and intra- and interlaboratory variability will be considered when determining validity of the method

A.6.4 Tasks and Timeframes

Sample Collection	September 2004-May 2005
Method/Protocol Development	February 2005-June 2005
Screening Study	June 2005-August 2005
Completion of Reports	August 2005
Peer Review	August 2005-September 2005

A7. Quality Objectives and Criteria

A7.1 Data Quality Objectives (DQOs)

The data quality objectives for this project are based on data acquired in the methods development stage of this study. An inter-lab data quality objective was determined using the variability within each dust sample for slag wool fibers, one of the markers for WTC dust It was determined that data could be acquired with a relative certainty of $\pm$ 35%. An intra-lab data quality objective was determined using the variability within each dust sample for slag wool as well It was determined that data could be acquired with a relative certainty of $\pm$ 30%

A7.2 Measurement Quality Objectives (MQOs)

The accompanying tables (Tables 1 and 2) list Measurement Quality Objectives (MQOs) for this intralaboratory (within lab) and interlaboratory (within sample) variability. Accuracies and precision were taken from preliminary data and manufacturer's specifications.

Measurement Parameter	Analysis Method	MQO for Accuracy	MQO for Precision	MQO for Completeness
Individual dust sample mass	Microbalance	+/- 5%	+/- 5%	85%
Fibers/Concrete Particles/Gypsum Particles	SEM	+/- 30%	+/- 30%	85%
Fibers	PLM	+/- 30%	+/- 30%	85%

Table 1 Measurement Quality Objectives (MQOs) for Intralaboratory Variability (within lab)

Measurement Parameter	Analysis Method	MQO for Accuracy	MQO for Precision	MQO for Completeness
Individual dust sample mass	Microbalance	+/- 5%	+/- 5%	85%
Fibers/Concrete Particles/Gypsum Particles	SEM	+/- 30%	+/- 30%	85%
Fibers	PLM	+/- 30%	+/- 30%	85%

Table 2 Measurement Quality Objectives (MQOs) for Interlaboratory Variability (within sample)

Intralaboratory MQOs will be calculated based on results within each lab for the 32 samples Interlaboratory MQOs will be calculated based on the composite result for each lab and compared with other labs for the 32 samples Both sets of MQOs will be compared with the target MQOs listed above Accuracy will be based on how close the labs are to the calculated overall laboratory mean for each sample or set of samples (i e each spiking percentage or background set of samples) and precision will be based on the duplicate results within each lab (relative % difference)

A8 Special Training/Certifications

All laboratories and analysts chosen for this work will have training in both Polarized Light Microscopy (PLM) and Scanning Electron Microscopy (SEM)

A9 Documents and Records

Documents generated (or to be generated) during this project and responsible party

1) Sampling Access Agreement – EPA
2) Sampling Information Sheet - EPA
3) Sampling and Sample Handling QAPP – Lockheed Martin
4) Analytical Method/Protocol to be used in study - EPA
5) Screening Study QAPP - EPA
6) Prime Contractor Report on Screening Study - Versar
7) EPA Report (separate from Prime Contractor Report) on Screening Study - EPA

The Screening Study QAPP will be distributed as indicated in Section A3 of this document. Dr Rosati will distribute the QAPP to Versar and the government labs, USGS and EPA NERL as well as EPA, ERT Versar will be responsible for distributing the QAPP to all analytical subcontractors (RJ Lee, MVA, MAS, EMSL and Reservoir) and EPA, ERT will be responsible for distributing the QAPP to its sampling contractor, Lockheed Martin.

Sampling and analytical data will be reported to Dr Rosati by EPA/ERT and Versar, respectively, on a weekly basis These data will be presented in a spreadsheet format The final data shall be presented to Dr Rosati in a report format, both electronically (including a final data spreadsheet) and hard copy

B. DATA GENERATION AND ACQUISITION

B1. Sampling Process Design

Dispersion models, photos, interviews, and satellite data were reviewed to discern areas that were likely impacted by WTC dust and those that were not Impacted samples were collected very close to the WTC site, and background samples were collected from areas distinctly outside of those that were 'likely' impacted Samples were collected from federal buildings, office buildings and private residences on a volunteer basis A pre-sampling survey of building and sampling areas, including photos of sampling areas (if permitted by building owners) and notes on building usage, to identify conditions that might compromise samples (e g , smoking or cooking areas) was developed Additionally, an access agreement was signed by the unit occupant/owner and an information sheet regarding the sampling was provided by the sampling contractor to the occupant/owner (Appendix D and E, respectively)

B2. Sampling Methods

Sampling followed the approved "Generic Quality Assurance Project Plan for WTC Residue Sampling New York City, NY, March 2005 (Appendix A) In each building identified for sampling, dust samples were collected from at least three areas 1) one sample from a track-in area near a building entrance, preferably in a carpeted area, 2) two samples from relatively undisturbed areas (e g , on top of bookcases, under furniture), and 3) other areas showing visible accumulation of settled dust, including HVAC ducts A standard method (REAC SOP 2040 - Collection of Indoor Dust Samples from Carpeted Surfaces for Chemical Analysis using a Nilfisk GS-80 Vacuum Cleaner) using a HEPA vacuum was used by EPA to collect bulk dust samples (Appendix C)

B3. Sample Handling and Custody

Once samples were collected, they were sieved to 150 microns Sieving was performed by the method in REAC SOP 2040 - Collection of Indoor Dust Samples from Carpeted Surfaces for Chemical Analysis using a Nilfisk GS-80 Vacuum Cleaner (Appendix C).

Once samples were sieved, they were ashed as described in Protocol for Preparation and Analysis of Residential and Office Space Dust by Polarized Light Microscopy and Scanning Electron Microscopy with Energy Dispersive X-Ray Spectroscopy, May 18, 2005 (Appendix B) 0 25 mg of each ashed sample was archived Ashed samples were disseminated as instructed by EPA (Dr. Rosati) As not all collected samples were used in the study, remaining samples will be sealed and stored in a limited access area All samples are accompanied by chain-of-custody forms To ensure that these important samples are properly collected, tracked, stored, and distributed, quality assurance (QA) procedures were in place prior to any sample collection (Appendix C)

B3.1 Homogenization

Homogenization of spiked samples was performed by the USGS Standards Laboratory after ashing occurred First, the background material was transferred to a 16 ounce glass container and an expanded metal mixing card inserted The container was sealed, and placed on a roller mixer where the contents were mixed for a total of eight hours Each spiking material was then

transferred to individual four ounce glass containers. A laminated customized paper mixing card was inserted and the container was resealed and placed on a horizontal roller mixer Once pre-mixing of the two dust types was complete, three concentrations of spiking material were prepared according to Table 3 as follows

Mass Target Conc Wt, %	background material, g	spiking material, g
1	29.7	0.3
5	28 5	1 5
10	27 0	3 0

Table 3: Mass concentrations of spiked samples

The appropriate amount of background and spiking material were weighed into plastic weighing boats and then transferred to pre-labeled four ounce glass bottles. A laminated customized paper mixing card was inserted and the container sealed and placed on a horizontal roller mixer The samples were blended for a total of ten hours After blending a total of six aliquots, ~0 5g was removed from each container using a spatula and transferred to individual one ounce vials The six samples from each concentration underwent SEM analysis at the USGS to assure reasonable sample homogeneity has been accomplished. The samples were then shipped to EPA, ERT for archiving and distribution

B4. Analytical Methods

The USGS, the EPA's National Exposure Research Laboratory (NERL) and EPA's National Enforcement Investigations Center (NEIC) developed a method to screen for the three key materials slag wool, elements of concrete, and gypsum This method involves the use of polarized light microscopy (PLM) or scanning electron microscopy (SEM) to determine the quantity of each of the materials present (Appendix B)

Data will be reported on the standardized sheets in the appendix of this protocol Data to be reported includes

- Slag wool (fibers/gram of dust) and length/width
- Elements of concrete (area %)
- Gypsum (area %)

B5. Quality Control

Quality control is addressed in Section 10 0 of the standard protocol in Appendix B entitled "Protocol for Preparation and Analysis of Residential and Office Space Dust by Polarized Light Microscopy and Scanning Electron Microscopy with Energy Dispersive X-Ray Spectroscopy" In addition to the items referred to in this section, several measures have been taken to ensure the quality of this study

- Standard protocol – a standardized protocol will be used for all sample preparation and analysis. This was determined to be a necessity during the scoping part of this work. This standardized protocol should help to minimize interlaboratory variability

- Duplicates– duplicates of all samples were provided for analysis Analysis of these duplicates will allow us to determine intralaboratory variability when using a standardized protocol

- Blind samples – all labs received the same 32 samples, and all samples were coded so that this was a 'blind' study, no lab knows what they are analyzing nor will they be able to compare their results with other laboratories

Sample Distribution Table (note. each of the eight laboratories has been given a letter A-H)

Sample Designations	Laboratory Letter Codes							
	A	B	C	D	E	F	G	H
AP5(1)								
AP5(2)								
CMC(1)								
CMC(2)								
HS3(1)								
HS3(2)								
WGS(1)								
WGS(2)								
MW(1)								
MW(2)								
DB1%(1)								
DB1%(2)								
DB5%(1)								
DB5%(2)								
DB10%(1)								
DB10%(2)								
C1-RTP(1)								
C1-RTP(2)								
USGS1%(1)								
USGS1%(2)								
USGS5%(1)								
USGS5%(2)								
USGS10%(1)								
USGS10%(2)								
USC(1)								
USC(2)								
FP(1)								
FP(2)								
MUNYC1(1)								
MUNYC1(2)								

MUNYC2(1)								
MUNYC2(2)								

Samples spiked with WTC dust, at 1, 5, and 10% levels are shaded. All others are background samples. Total of 20 background samples (10 samples + 10 duplicates) and twelve spiked samples (6 samples + 6 duplicates)

B6. Instrument/Equipment Testing, Inspection and Maintenance

Vacuum cleaners used for sampling were maintained as described in Appendix A Generic Quality Assurance Project Plan for WTC Residue Sampling New York City, NY, March 2005.

B7. Instrument/Equipment Calibration and Frequency

All microbalances used in this study shall calibrated annually Scanning Electron Microscopes (EDS system) shall be calibrated on a daily basis as discussed in Appendix B entitled "Protocol for Preparation and Analysis of Residential and Office Space Dust by Polarized Light Microscopy and Scanning Electron Microscopy with Energy Dispersive X-Ray Spectroscopy, Section 10 1

B8. Inspection/Acceptance of Supplies and Consumables

Inspection and acceptance of all consumables used during sampling will be performed as described in Appendix A Generic Quality Assurance Project Plan for WTC Residue Sampling New York City, NY, March 2005

Inspection and acceptance of all consumables used during sample analysis will be performed by the analytical and government laboratories Consumables are listed under apparatus and materials in Appendix B entitled "Protocol for Preparation and Analysis of Residential and Office Space Dust by Polarized Light Microscopy and Scanning Electron Microscopy with Energy Dispersive X-Ray Spectroscopy.

B9. Non-Direct Measurements

Not applicable

B10. Data Management

Data from this study will be reported by the subcontractors and government labs to the prime contractor on standardized data sheets found in Appendix B "Protocol for Preparation and Analysis of Residential and Office Space Dust by Polarized Light Microscopy and Scanning Electron Microscopy with Energy Dispersive X-Ray Spectroscopy, May 18, 2005." Data will be compiled and analyzed by the prime contractor in a electronic spreadsheet, and a report of this data will be written

C. ASSESSMENT AND OVERSIGHT

- All work will be overseen by the principal investigators of this study. Weekly conference calls will be held with EPA, the prime contractor and all subcontractors to assess progress and discuss any issues or problems that may have arisen Data is to be submitted to the prime contractor by the subcontractors on a weekly basis as this data is obtained Due to the rapid nature of this study, no interim reports are required.

- All stakeholders (EPA, USGS and contractors) will be provided a copy of the QAPP and will review it for correctness

- All sampling and sample preparation performed by Lockheed Martin will be under direct oversight of quality assurance personnel and audits will be conducted as noted in Appendix A (Sampling QAPP)

- All contracting laboratories are required to employ standard QA practices and all work should be performed under the oversight of in-house quality assurance personnel

- All data will undergo evaluation and review by the prime contractor prior to being assembled into a report to the EPA The EPA will perform its own assessment and evaluation of the study data and issues, and will assemble this information into an overall EPA report

- The study will undergo a formal EPA peer review once it has been completed Peer reviewers will be provided all data and reports, and will be given 6 weeks to perform a full evaluation of the study In addition, all data and reports will also be provided to the WTC Technical Panel for their review and comments

D. DATA VALIDATION AND USABILITY

- All data shall be provided by the subcontractors to the prime contractor on the spreadsheets provided in Appendix B "Protocol for Preparation and Analysis of Residential and Office Space Dust by Polarized Light Microscopy and Scanning Electron Microscopy with Energy Dispersive X-Ray Spectroscopy, May 18, 2005.", Section 15 0
- The prime contractor will review and verify the data before compiling it into a report
- EPA will evaluate the prime contractor report, along with the compiled data to determine:
    - whether the MQO's presented in Table I of this QAPP were met
    - whether the study described herein demonstrated the following
        - that slag wool, gypsum and elements of concrete are reasonable markers for WTC dust (by showing that these markers distinguish WTC-laden dust from background dust),
        - that WTC dust at a diluted concentration can be distinguished from background, and
        - that the analytical method works well enough, and is able to be carried out by enough analytical laboratories to 1) evaluate the above materials as markers and 2) distinguish WTC dust from background dust

- EPA will prepare a final report documenting all data, analysis and conclusions based on the above evaluation

APPENDICES

A Generic Quality Assurance Project Plan for WTC Residue Sampling New York City, NY, March 2005
B Protocol for Preparation and Analysis of Residential and Office Space Dust by Polarized Light Microscopy and Scanning Electron Microscopy with Energy Dispersive X-Ray Spectroscopy, May 18, 2005
C REAC SOP 2040 - Collection of Indoor Dust Samples from Carpeted Surfaces for Chemical Analysis using a Nilfisk GS-80 Vacuum Cleaner
D Access Agreement
E Information Sheet

REFERENCES

Lowers, H A , G.P Meeker, and I K Brownfield (2005a) Analysis of Background Residential Dust for World Trade Center Signature Components Using Scanning Electron Microscopy and X-ray Microanalysis U S Geological Survey Open File Report 2005-1073

Lowers, H A , Meeker, G P , I K Brownfield (2005b) World Trade Center Dust Particle Atlas. U S Geological Survey Open-File Report 2005-1165. http //pubs usgs gov/of/2005/1165/

Meeker, G P , A.M Bern, H A Lowers, and I K. Brownfield (2005) Determination of a Diagnostic Signature for World Trade Center Dust using Scanning Electron Microscopy Point Counting Techniques U S Geological Survey Open File Report 2005-1031.

NYCDOHMH/ATSDR (2002) New York Department of Health and Mental Hygiene and Agency for Toxic Substances and Disease Registry. Final Technical Report of the Public Health Investigation To Assess Potential Exposures to Airborne and Settled Surface Dust in Residential Areas of Lower Manhattan Agency for Toxic Substances and Disease Registry, US Department of Health and Human Services, Atlanta, GA.

R J Lee (2004) Signature Assessment 130 Liberty Street Property Expert Report WTC Dust Signature Prepared for Deutsche Bank May, 2004 R J Lee Group, Inc 350 Hochberg Road, Monroeville, PA 15146

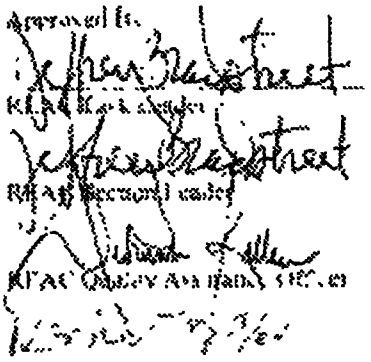
APPENDIX A: SAMPLING QAPP

A1. GENERIC QUALITY ASSURANCE PROJECT PLAN FOR WORLD TRADE CENTER (WTC) RESIDUE SAMPLING NEW YORK CITY. NEW YORK

U.S. EPA Work Assignment No 0-089
Lockheed Martin Work Order No · EACOO089
U S EPA Contract No · EP-C-04-032

Prepared For
United States Environmental Protection Agency/Environmental Response Team
Edison, NJ

March 2005



A2. TABLE OF CONTENTS

A PROJECT MANAGEMENT

This project generic Quality Assurance Project Plan (QAPP) was prepared in accordance with *EPA Requirements for Quality Assurance Project Plans (QAPPs),* EPA QA/R-5 and the *Response Engineering and Analytical Contract (REAC) Program QAPP.*

A3 DISTRIBUTION LIST

The following personnel will receive copies of the approved QAPP for the World Trade Center (WTC) Residue Sampling , Work Assignment (WA) No 0-089

1. Rajeshmal Singhvi, Environmental Protection Agency/Environmental Response Team (EPA/ERT)
   Work Assignment Manager (WAM)
2. Jacky Rosati, EPA, Research Triangle Park North Carolina (NC)
3 Eletha Brady-Roberts, National Homeland Security Research Center (NHSRC)
4 Jeffrey Bradstreet, REAC Air Response Section Leader/Task Leader (TL)/Quality Control (QC)
   Coordinator
5 Deborah Killeen, REAC Quality Assurance Officer (QAO)
6 Dennis Miller, REAC Program Manager

A4 PROJECT ORGANIZATION

The following individuals will participate in the project

EPA/ERT
Rajeshmal Singhvi -WAM
Jacky Rosati - Project Coordinator
Jeff Catanzarita - Technical Auditor
Eletha Brady-Roberts - NHRSC Quality Assurance (QA)

REAC
Jeffrey Bradstreet -TL/QC Coordinator
Miguel Trespalacios - Senior Air Sampling Scientist
Michael Hoppe - Environmental Scientist/Sampler
TBD - Environmental Scientists/Samplers
Richard Magan - Technician/Sampler
Deborah Killeen - QAO

Laboratories that will receive residue samples for chemical marker/signature identification on this project include·

Department of Environmental Sciences and Engineering, University of North Carolina, United States Geological Survey (USGS) Denver Microbeam Laboratory, and Other commercial laboratories to be determined

The REAC TL/QC Coordinator for the project is the primary point of contact with the EPA/ERT WAM. The XL is responsible for the completion of the Work Plan (WP) and QAPP, project team organization, and supervision of all project tasks, including reporting and deliverables The EPA NHSRC will provide oversight and guidance in the field through the WAM

A5, PROBLEM DEFINITION

The World Trade Center (WTC) attacks on 11 September 2001caused the airborne release of two types of dusts those related to the building collapse and fine participate matter from the subsequent fires There are concerns among residents of New York City (NYC)about the potential health effects of WTC dusts that might remain in buildings in NYC. The goal of this study is to collect dust samples from areas near the WTC and distant from the WTC (background NYC dusts) These dust samples will be used to validate chemical markers or signatures for WTC dust - as compared to background dust - for a larger sampling effort to identify indoor areas still contaminated with WTC dust. The markers or signatures for WTC dust are being developed by laboratories at EPA, USGS, and several universities By sampling a number of contaminated and uncontaminated sites and by utilizing recently collected samples, the WTC signatures can be validated or improved for the larger sampling study to delineate contaminated areas

The residue from the collapse of the WTC Towers may contain heavy metals (primarily lead, arsenic, and mercury), polynuclear aromatic hydrocarbons (PAHs), and other contaminants that EPA designated laboratories are investigating to associate specific analytes with the WTC.

A6. PROJECT DESCRIPTION AND SCHEDULE

The purpose of this project is to collect dust samples from designated buildings that may be from the collapse of the WTC towers or from background sites for comparison EPA will identify contaminated and uncontaminated buildings in NYC and obtain access for REAC personnel to conduct sampling To the extent possible, contaminated buildings that are within both the dust and the fire plumes will be selected, so that current dust samples for these two types of emissions can be collected EPA may identify several groups of buildings over time, as permission for access is obtained EPA will provide REAC personnel with the street address and the name and phone number for a contact person in each building identified for surveying

This activity will involve conducting scoping surveys of buildings identified by the EPA in the NYC Area, preparing sampling plans, collecting samples, splitting samples for multiple laboratories, shipping samples, and archiving samples for up to two years for future analysis and report preparation

The schedule of activities and reports is as follows

WP	6 October 2004
Draft Generic QAPP	6 October 2004
Final Generic QAPP	18 March 2005
Draft Building Survey Form	15 October 2004
• Collect Residue Samples	As scheduled by EPA
• Prepare and Send Sample Aliquots	4 Days after sampling
• final Report	10 Days after sampling

A7 DATA QUAIJTY OBJECTIVES AND CRITERIA FOR MEASUREMENT OF DATA

The focus of this project is to collect dust samples that may be contaminated with materials from the destruction of the WTC towers or are potentially uncontaminated This QAPP covers the collection, storage and shipment of the samples to EPA designated laboratories The specific chemical markers or signatures associated with WTC dust samples are investigatory and will be used to further define the project Once defined, the specific chemical markers or signatures will be used to further define the project

A8 TRAINING AND CERTIFICATION

The training of all field personnel involved with sampling activities is intrinsic to their position and required responsibilities They will have the following documented training

- Occupational Safety and Health Administration (OSHA) 40-hour and 8-hour refresher in Hazardous
  Waste Operations (20 CFR1910 120)

- Department of Transportation (DOT) hazardous materials shipping
- First Aid and Cardiopulmonary Resuscitation (CPR) training

A9 DOCUMENTS AND RECORDS

The RE AC Program QAPP serves as the basis for this generic QAPP The most current approved version is available to all REAC technical personnel as an uncontrolled copy on the REAC Local Area Network (LAN) Documents and records that will be generated during this project include.

WP
Draft Generic QAPP
- Final Generic QAPP
- Field logbooks
- Site maps
- Photos of Sampling Locations
- Chain of Custody forms
- Final Reports

The Final Report will provide a description of the project, field procedures, sample preparation procedures, difficulties encountered and will include validated final copies of chain of custody forms. All documentation will be recorded in accordance with REAC standard operating procedure (SOP) #2002, *Sample Documentation* and REAC SOP #4001, *Logbook Documentation* The final report will be prepared using REAC SOP #4021, *Preparation of Final Reports*

B, DATA GENERATION AND ACQUISITION

B1 SAMPLING PLAN DESIGN

Judgmental sampling will be used to select sample locations that are most likely to represent WTC residue or background dust This will be based on historical information, visual inspection and best professional judgment of the WAM and sampling team. This type of sampling is used to identify contaminants present in areas potentially having the highest concentration of contaminants. Additional samples may be collected when requested by the WAM and EPA NHSRC personnel

During the sampling of EPA specified buildings, dust samples will be collected from each of the buildings up to 20 in accordance with the EPA approved generic QAPP. Sampling will likely be performed at two types of areas in each building a high traffic area (to characterize tracked-in dust) and a lowtraffic area (to represent settled indoor dust) Two low traffic areas will be specified for a total of three areas that will be sampled. The desired high traffic area is to be an area near a main entrance, preferably carpeted The desired low-traffic areas include areas infrequently cleaned, such as the top of elevator housing, under refrigerators, behind file cabinets, above ceiling tiles, on high shelves, or in other areas that show visible dust accumulation and are infrequently disturbed Sampling will not be restricted to carpeted areas as the intent of the sampling is to obtain the desired residue. If vacuum sampling is not possible or preferred, sweep sampling will be used to collect the residue Sampling will not be conducted in areas that would likely contain chemicals in dusts that would interfere with the analysis for the WTC markers The following areas will be avoided in the sampling effort

- Areas with significant cigarette or cigar smoke, incense, or
  burning candles
  Areas near major outdoor combustion sources (e g , power plants)

Due to the inability to obtain triplicate samples (once an area is sampled, little residual remains), three samples will be collected in the same general area, for a total of nine samples from a building (i e , three sample areas times three samples in each general area). The proximity of the samples in

each general area will be determined as a result of visual inspection in the field and discussions with the WAM

B2 SAMPLING METHODS

Vacuum sampling will be performed in accordance with modified REAC SOP #2040, *Collection of Indoor Dust Samples From Carpeted Surfaces for Chemical Analysis Using a Nilfisk GS-80 Vacuum Cleaner* This method may afford collection of samples large enough for analysis of both purported organic and inorganic signatures Although the method specifies the size and shape of the areas to be sampled and the mass to be collected, the sample collection procedure will vary to accommodate the site-specific conditions and ensure that an adequate sample is obtained If it is not feasible to use the vacuum method of sampling, samples will be collected in bulk by sweeping the residue into a pan or sample bag in accordance with modified ERT/REAC SOP #2011 *Chip, Wipe and Sweep Sampling* The sample handling and data collection requirements specified in modified REAC SOP #2040 will be followed

The area to be sampled is not measured before sampling, but after the sample is collected This is a modification of both REAC SOP #2040 and ERT/REAC #2011. REAC SOP #2040 is further modified in that samples will also be collected from non-carpeted surfaces, the amount of sample collected will be visibly checked and dust weight calculations will not be performed Sweep sampling utilizes a dedicated, hand held sweeper brush to acquire the sample from an area. The area sampled is measured after sampling

Sample Volume, Container, Preservation and Holding Time. The collected samples are placed into appropriately sized glass jars or zip-lock plastic bags Storage of the samples collected by sweep or vacuum are maintained in a refrigerated unit at 4 ± 2 degrees Celsius (°C) after sieving

Sampling Equipment Decontamination. The nozzles, wands and hoses are decontaminated after use with a bottle brush, to remove any accumulated dust in the hose and nozzle When the nozzle is clean, it is removed and sprayed with reagent grade methanol and allowed to air dry on a clean surface The wand and hose are then cleaned with the bottle brush To continue a new polyliner and collection bag for the collection of another sample is installed

B3 SAMPLE HANDLING AND CUSTODY

In the field, sampling data are recorded on a Vacuum Sampling Work Sheet or in a dedicated project logbook Cham of custody (COC) records will be used to document the collection of dust samples by vacuum or bulk. All COC records will receive a peer review in the field prior to shipment of the samples in accordance with REAC SOP #4005, *Chain of Custody Procedures*

All samples will be delivered to the REAC facility and sieved in accordance with modified REAC SOP 2040, *Collection of Indoor Dust Samples From Carpeted Surfaces for Chemical Analysis Using a Nilfisk GS-80 Vacuum Cleaner* and modified ERT/REAC SOP #2011 *Chip, Wipe and Sweep Sampling* The samples will be sieved through a No 100 sieve (150 microns [fim]) After sieving, the samples will either be transferred to jars, which will be placed into Ziplock™ storage bags, or directly into Ziplock™ storage bags, and then placed into a holding refrigerator with the corresponding COC record.

Scribe* spreadsheet formats will be used for sample management REAC is required by contract to use Scribe" to track and log the samples In addition a unique sample numbering system has been established to each sample, which identifies the site identification, event number and the sample number Additional information is provided with the sample number to identify whether it is a sieved (S) or coarse (C) fraction and the weight of the fraction in grams, e g , SO. 1

The samples collected by REAC personnel will be shipped to the designated laboratory for analysis in accordance with REAC SOP #2004, *Sample Packaging and Shipment* One of the four aliquots of each sample will be retained and stored by REAC staff for up to two years in a secure refrigerator,

B4 ANALYTICAL METHODS

Once specific chemical markers or signatures have been defined, EPA personnel in consultation with NHSRC will be able to determine which analyses will be appropriate The laboratories specified under Section A4 are conducting the investigatory work

B5 QUALITY CONTROL

This QAPP covers the collection, storage and shipment of the samples to EPA designated laboratories for analysis Quality control for the field and storage procedures are as follows·

- Field documentation on Field Sampling Worksheets or in logbooks
  Documentation of temperature for the dedicated secure refrigerator

Duplicate samples will not be taken due to the nature of the sampling method Once an area is vacuumed, little residual sample remains Quality control for the laboratory procedures will be specified by the EPA/NHSRC

B6. INSTRUMENT/EQUIPMENT TESTING, INSPECTION AND MAINTENANCE

The Nilfisk vacuums used in the collection of the residue samples will be maintained in accordance with established specifications On a quarterly basis, the parts of the Nilfisk vacuum cleaners are inspected for cracks and breaks An inventory of available supplies is conducted every three months.

B7. INSTRUMENT/EQUIPMENT CALIBRATION AND

FREQUENCY The instrument/equipment calibration frequency is

not applicable to this QAPP

B8 INSPECTION/ACCEPTANCE OF SUPPLIES AND

CONSUMABLES

REAC personnel are responsible for the procurement, inspection, and acceptance of supplies and consumables for this WA. The vacuum cleaner filters purchased by REAC personnel must meet the requirements specified by the manufacturer The REAC TL and Group Leaders are responsible for ensuring that the correct filters and sampling bags are specified in the purchase orders and verifying upon receipt that the correct parts have been shipped It is the responsibility of the EPAVERT to provide adequate facilities, equipment and supplies for REAC to perform all field related tasks for this WA

B9 NON-DIRECT

MEASUREMENTS This section is not

applicable to this QAPP BIO

DATA MANAGEMENT

The QAPP is identified by the footer located on the bottom left hand corner of the page The file identification represents the structure and the filename The filename starts with the 3-digit WA number preceded by a "zero", then the deliverable type (D or N) to identify the document as a deliverable or non-deliverable followed by the document type For amended or revised documents, the letters "A" and "R" for amended and revised, respectively, and the appropriate amendment or

revision number (e g 1,2,3) are added after the document type After the document type and revision/amendment code (if any), a six-digit code based on the month, day and year (mmddyy) is added to indicate the date the document was delivered to the client.

Field sampling data will initially be recorded on field data sheets and in field notebooks Samples will be identified by the field assigned sample number Paper versions of all deliverables (Work Plan, Generic QAPP and Final Reports) will be provided to the ERT WAM and stored in the REAC Central Files Electronic versions of all deliverables will be saved on the REAC archive drive in accordance with Administrative Procedures (AP) #34, *Archiving Electronic Files* All data deliverables for this WA will be posted to the ERT-Information Management System (IMS) web site as either a Scribe* electronic data deliverable (EDD) or in portable document format (pfd) Submission of the deliverable to the appropriate ERT-IMS website will be considered delivery to the WAM as of the date and time such deliverables are received on the website

Field log books will also be archived once the project is completed and the Work Assignment 0-089 is closed All SOPs referenced in this QAPP are available on the REAC LAN.

C. ASSESSMENT/OVERSIGHT

C1. ASSESSMENT AND RESPONSE ACTIONS

The REAC TL, Air Response Section Leader, QAO and QC Coordinator are responsible for QC assessments and corrective action for this WA These personnel have the authority to issue stop work orders The tasks associated with this QAPP are assessed through the use of peer reviews, technical reviews and/or technical system audits, and management system reviews Peer review enables the reviewers to identify and correct reporting errors before reports are submitted Technical reviews are conducted by those immediately responsible for overseeing or performing the work (self-assessments) An independent assessment or technical audit will be performed by Jeff Catanzarita Management system reviews establish compliance with prevailing management structure, policies and procedures, and ensures that the required data are obtained

Peer reviews are conducted on project deliverables to ensure a technical review with respect to content, completeness and the overall quality of the deliverable prior to submittal to the EPA/ERT The responsibilities of the review team and the sequence in which the deliverable is reviewed, is outlined in REAC AP #22, *Peer Review of REAC Deliverable** The REAC QAO will audit data deliverables on a biannual basis to determine compliance with the peer review procedures

The EPA/ERT WAM for this task will be present and will have the responsibility for verifying that the proper SOPs and sampling procedures are followed If any technical issues or deficiencies are identified, they will be reported to the REAC TL for immediate resolution or corrective action Any changes in scope of work will be documented on a Field Change Form and approved by the WAM

C2 REPORTS TO MANAGEMENT

Monthly technical reports will be prepared for this WA when hours have been charged on a monthly basis. These reports will detail the accomplishments for the past month, any problems encountered, solutions to rectify the problem, contacts and meetings, goals for the next month, and an estimate of the of the total labor hours and costs for the next reporting period The monthly technical reports are submitted to the EPA/ERT Project Officer and WAM

On a quarterly basis, the REAC QAO provides a report to the REAC Program manager and the ERT QA Manager that summarizes the quality assurance (QA) activities on a quarterly period These reports include results of performance evaluation samples, system audits (internal and external), summary of non-conformance and corrective actions, preparation of SOPs for analytical and operational activities, training, contacts/meetings and other QA activities

REAC Report	Recipients
Monthly Progress	EPA/ERT Project Officer and WAM
Quarterly QA Reports	EPA/ERT Project Officer and WAM

D DATA VALIDATION AND USABILITY

D1 DATA REVIEW, VERIFICATION AND VALIDATION

For field activities, it is necessary to determine whether the samples were collected using the sampling design specified in element B1, whether the samples were collected according to a specific method or SOP as specified in element B2, and whether the collected samples have been recorded and handled properly as in element B3 Field sampling worksheets and field notes will be reviewed by the RE AC TL for completeness The COC records will be reviewed to ensure that the field information has been accurately reflected on the COC records

D2 VERIFICATION AND VALIDATION METHODS

Verification occurs at each level in the field to ensure that appropriate outputs are being generated routinely Records produced electronically or maintained as hard copies are subject to data verification During field activities, records associated with sample collection such as field data sheets, COC records, logbook documentation, or electronic devices to log samples are verified Naming conventions for the initial samples and samples fractions produced during sieving are verified by the RE AC TL Chain of custody records are verified along with refrigerator and freezer logs to ensure the integrity of the samples

There is no analytical data being generated under this WA, therefore, procedures for verifying and validating data, including the chain of custody for data throughout the life cycle is not applicable

D3 RECONCILIATION WITH USER REQUIREMENTS

Responsibility lies with the EPA, thus, this element is not applicable to this QAPP

REFERENCES

Response Engineering and Analytical Contract 2003 *Quality Assurance Project Plan for the Response, Engineering, and Analytical Contract,* Revision 0 0

U S Environmental Protection Agency 1990, *Quality Assurance/Quality Control Guidance for Removal Activities,* EPA/540/G-9/004, Office of Emergency and Remedial Response

U S. Environmental Protection Agency 2001 *EPA Requirements for Quality Assurance Project Plans (QAPPs),* EPA/240/B -01/003, Office of Environmental Information.

TABLE I Field Sampling Summary
World Trade Center (WTC) Residue Sampling
March 2005

Analytica l Parameter	Sampling Method	Preservation	Total Samples	Maximum Number Samples
Dust/Settled Particulate	Nilfisk GS-80 Vacuum Cleaner	Up to 2 years at 4 degrees C +/-2 degrees C	Up to 9 per Building	9
Dust/Settled Particulate	Sweep	Up to 2 years at 4 degrees C +/-7 degrees C	Up to 9 per Building	9

APPENDIX B: PROTOCOL

Protocol for Preparation and Analysis of Residential and Office Space Dust by Polarized Light Microscopy and Scanning Electron Microscopy with Energy Dispersive X-Ray Spectroscopy

June 27, 2005

Prepared by:
U.S. Environmental Protection Agency
National Enforcement Investigations Center/ National Exposure Research Laboratory/National Homeland Security Research Center
Denver, CO and Research Triangle Park, NC

The use of trade names does not imply endorsement and are used for illustrative purposes only.

Contents

1.0 Purpose

This document describes sample preparation and analytical screening procedures for bulk samples of dust collected from residential and commercial office environments. These methods are collectively referred to as the protocol

2.0 Scope/Application

The protocol describes polarized light microscopy (PLM) and scanning electron microscopy (SEM) with energy dispersive spectrometry (EDS) to screen bulk dust samples for mineral slag wool, particles consistent with concrete compositions, and gypsum. The analysis methods include operating parameters and particle identification criteria.

2.1 Limitations of the Method and Future Considerations

This protocol provides a means of analyzing for particles consistent with those found in dust present after the collapse of the World Trade Center (WTC) in New York City Components of WTC Dust have been documented and catalogued by the U S Geological Survey Denver Microbeam Facility and the images and characteristics shall be used in identification of particles (1)

The x-ray mapping procedure in sections 12 2 3 and 12 2 4 and the calculations presented in section 13 0 only determine the maximum percentage of non-gypsum, calcium-rich particles, which may include non-concrete materials The particle analysis procedure presented in section 12 2 5 is the preferred procedure for determining the percentages of gypsum and concrete particles in the sample

The x-ray mapping and image analysis procedure relies heavily on the thresholds for backscattered electron images Binary (particles white and background black) backscattered electron images (BEI) should be used to reduce errors in setting thresholds in Photoshop

3.0 Definitions

1 PLM - Polarized Light Microscopy
2 SEM - Scanning Electron Microscope
3 EDS - Energy Dispersive Spectrometry
4 SEI - Secondary Electron Image
5 BEI - Backscattered Electron Image
6 Mineral Wool – lightweight vitreous fibrous material composed of rock wool and slag wool and used especially for heat and sound insulation
7 Rock Wool - a man-made vitreous fiber (MMVF) component of mineral wool containing magnesium, aluminum, silicon, and calcium Sodium and potassium may also be present Iron oxide is typically 3-12% by weight
8 Slag Wool - a man-made vitreous fiber (MMVF) component of mineral wool

containing magnesium, aluminum, silicon, and calcium. Sodium and potassium may also be present. Iron oxide is typically less than 2% by weight

9. HEPA - High-Efficiency-Particulate-Air Filter

4.0 Summary of Method

1 Weigh sample to nearest 0.0005 g
2 Split the sample, archive half and keep half for analysis
3 Ash half of the sample for analysis
4. Sieve the ashed sample to 150 µm.
5 Split the <150 um ashed portion Archive three quarters of the sample Keep one quarter for PLM and SEM/EDS analysis
6 Weigh the quarter and place it in enough isopropanol to get a 10-20 mg per mL dilution. Apply an aliquot to a glass slide, let dry, and add 1 55 (or 1 605) refractive index oil Analyze by PLM for mineral wool.
7 Prepare a sample for SEM/EDS analysis using the same dilution prepared for PLM
8 Apply an aliquot of the sample to an aluminum sample stub with a carbon adhesive tab covered by a piece of polycarbonate filter (13-mm diameter or punched out of a larger filter to fit the size of the stub)
9 Identify fibers by EDS and record the occurrence of fibers > 25 µm in length at 100 x magnification to get a statistical representation of fiber compositions
10 Prepare 10-fold dilution of the suspension from step 7 and apply an aliquot to a polycarbonate/adhesive tab substrate affixed to an aluminum sample stub Alternatively, a lighter loading can be prepared by filtering the diluted suspension through a 25-mm diameter, 0 4-µm pore size, polycarbonate filter and affix this to a carbon adhesive tab affixed to an aluminum sample stub
11. Collect x-ray maps of 10 fields at 500 x magnification for major elements, especially Ca, S, and Fe and use Adobe Photoshop or similar software to determine the area percent of gypsum and Ca-rich particles Fe-rich particles may also be identified in this step
12 Perform particle analysis via computer-controlled SEM/EDX analysis

5.0 Interferences

Interferences include possible contamination of samples by airborne dust or through improperly cleaned glassware and sieves. Interferences are minimized by performing all procedures involving dry dust in a clean room, cleaning countertops and glassware thoroughly before proceeding and placing particle-free wipes on all working surfaces To avoid cross-contamination, properly clean all glassware, sieves, and tools between samples

6.0 Safety

Respirable particles which may present a health hazard may exist in the sample Bulk samples may release respirable particles during handling All procedures involving dry dust samples will be performed under a negative flow High-Efficiency-Particulate-Air

Filter (HEPA) hood Samples handled outside of the HEPA hood will be covered with aluminum foil or placed in sealed glass jars

7.0 Apparatus and Materials

1 HEPA negative flow hood
2 Forceps
3 Kimwipes
4 Stainless steel spatula
5 Weighing paper
6 Programmable furnace [not required for validation study]
7 Ceramic crucibles with lids [not required for validation study]
8. Analytical balance (accuracy to 0 0005 g)
9 Retsch ultrasonic sieve shaker (AS200 Basic), or similar [not required for validation study]
10 Sample sieves, 3-inch diameter (recommended), 150-µm (100-mesh) opening, with lid and bottom pan similar [not required for validation study]
11 SEM aluminum sample stubs
12 Conductive carbon adhesive tabs
13 Eppendorf pipette, 10-µL capacity
14 Disposable pipette tips
15. 1 – 10 mL pipette
16 Glass vials for sonicating dust in isopropanol suspension (holds 10-mL volume)
17 Razor blade
18 Ultrasonic bath
19 50 mL glass beaker
20 Polycarbonate filters (25-mm diameter, 0 4-µm pore size)
21 Polycarbonate filters (13-mm diameter, 0 4-µm pore size), or borer to cut larger filters to SEM stub size
22 11-mm diameter cork borer
23 Millipore filter apparatus for use with 25 mm filters
24 125 mL Nalgene bottles
25 Hand-held vacuum pump
26 High-vacuum carbon evaporator with rotating stage
27 Glass petri dishes with lids
28 Adobe Photoshop Software, or similar
29 Glass petrographic slides
30 Glass cover slips
31 Polarized light microscope for mineral identifications
32 Scanning Electron Microscope with the following attributes
    a. Resolution 5 nm (at 25 kV, WD=10 mm – system dependent) or better
    b Accelerating Voltage. 10 to 20 kV
    c Minimum magnification range 50x to 200,000x
    d SEI (secondary electron image)
    e BEI (backscattered electron image)
    f Energy dispersive x-ray detector and analyzer for EDS analysis

g. Ability to collect x-ray maps or particle analysis software (preferably both)

8.0 Reagents

1 Isopropanol, reagent grade [CAS No. 67-63-0]
2 1 55 or 1.605 Refractive Index Oil

9.0 Sample Storage

Dust samples will be stored in an air-tight container, such as a sealed glass jar Samples placed in reagents will be labeled appropriately and stored according to laboratory safety standards Samples prepared for analyses will be stored in a protective container, such as a plastic case or covered petri dish, to prevent contamination

10.0 Quality Control

Quality control is implemented by thoroughly cleaning glassware and spatulas, keeping working surfaces clean, and preventing cross contamination During ashing, particles may be suspended if slow heating is not achieved Following the ashing program as outlined will minimize flashing, which can cause particles to become airborne Covered crucibles will be used to prevent contamination caused by flashing Used Eppendorf pipette tips and weighing papers will be discarded and new tips and papers will be used for each sample

Duplicate samples shall be prepared to determine the precision of the analysis In addition, sample blanks shall be prepared These blanks are checks for cross contamination during handling of the samples. Blanks shall be prepared at the same time and in the same manner as samples

10.1 Calibration

Calibration of the EDS system must be completed at least once at the beginning and again at the end of each analytical session Backscattered electron image (BEI) calibration should be performed at the beginning of the session and anytime the backscattered image brightness and/or contrast is adjusted

EDS calibration for both qualitative and quantitative (not required by this method but could be useful for identification of particle type) analysis is accomplished by the analysis of a polished carbon-coated reference standard The recommended material is USGS BIR1-G basalt glass mounted in epoxy in a brass tube, polished, and carbon coated using a carbon evaporator (2, 3)

The calibration reference material should be analyzed at the same operating conditions to be used for the analysis including beam current, accelerating voltage, working distance, detector dead time, and sample tilt (= 0°) For BIR1-G

the analysis should be performed with a beam size of 10-20 μm or equivalent area raster All calibration spectra will be saved with the corresponding data set. The calibration data will be used for inter- as well as intra-laboratory comparisons This calibration is in addition to, and not a substitute for the normal EDS calibration recommended by the EDS manufacturer which will be performed at regular intervals as specified by the EDS manufacturer

Backscattered electron detector calibration can be performed on the same BIR1-G material by adjusting the detector brightness and contrast to achieve the following conditions The epoxy on the BIR1-G reference material will be at 0 in a 256 grayscale image and the brass mounting tube will be at 256 The BIR1-G basalt glass should fall at approximately 130-140 gray scale units

11.0 Procedure

11.1 Weighing and Splitting

Weighing and splitting should be performed under a negative flow HEPA hood If the fan speed is set too high, loss of particles may occur The fan speed may need to be adjusted to prevent the loss of fine particles

Obtain an analytical balance with an accuracy of 0 0005 g and preweigh a clean piece of weighing paper Transfer the dust from the sample vial to the weighing paper and determine the weight of the dust Split the sample with a clean razor blade using the cone-and-quarter method If there are large clumps of organic fibers, such as hair or lint, temporarily remove the hair with a pair of forceps and tap the forceps lightly with another tool over a piece of weighing paper to remove fine particles. Center the fine fraction on the paper and split the sample into four equal parts using a razor blade Collect opposite corners (½ of the sample) for analysis and archive the other half Quarter the larger organic fiber bundles the same way, keeping half to proceed to the ashing step and half for archival purposes

Place the two quarters for ashing into a pre-weighed crucible Weigh the split and record the results

11.2 Ashing

Place the ceramic crucibles containing the samples into a furnace.

The furnace program should proceed as follows

1 Increase temperature by 1 °C/minute until sample reaches 250 °C
2 Hold temperature at 250 °C for 4 hours
3 Increase temperature by 1 °C/minute until sample reaches 480 °C
4. Hold temperature at 480 °C (sufficient for decomposing organics) for 8 hours Do not exceed 500 °C

5 Shut off furnace
6 Allow sample to cool before removing from furnace.
7 Weigh the ashed sample to the nearest 0 0005 g and record the result

11.3 Sieving

Sieve the sample through a 150-μm sieve using a Retsch ultrasonic sieve shaker, or similar Three-inch diameter sieves are recommended to minimize sample loss from particles being trapped in the sieve. The ultrasonic shaker will be operated at 20-minute intervals at the following settings· 20, 40, 60, 70, 80, then back down to 50 and 20. This will provide amplitudes ranging from 0 to 1 5 mm

Transfer the large and small fractions to clean pieces of weighing paper and weigh to the nearest 0 0005 g Archive the fraction greater than 150-μm

11.4 Preparation of Sample for Polarized Light Microscopy

Split the less than 150-μm sample fraction using the cone and quarter method Collect one corner for analysis and archive the other three quarters Weigh the quarter split to the nearest 0 0005 g and place it into a glass vial Make a suspension of 10-20 mg dust per mL of isopropanol. The amount of isopropanol needed will vary depending on the amount of dust; the target dilution is 10-20 mg per mL.

Cut an Eppendorf pipette tip with a razor blade to increase the opening to approximately 1 mm

Place the suspension in an ultrasonic bath for one minute, then remove the suspension from the ultrasonic bath and shake it gently to suspend all particles Collect a 10-μL aliquot of the mixture using an Eppendorf pipette with the modified tip and transfer to a glass slide. Prepare 4 such slides Allow them to dry, then add a drop of 1 55 (or 1 605) refractive index oil

11.5 Preparation of Sample for SEM Analysis

Prepare the SEM substrate on aluminum stubs using 0 4-μm pore size polycarbonate filters, carbon adhesive tabs Using an 11 mm filter punch and placing the filter between two filter separators, punch a circle the size of the carbon tab into the filter Place carbon adhesive tab affixed to an aluminum stub on the dull side of the 11-mm polycarbonate filter such that the shiny side of the filter exposed If available, a 13-mm diameter polycarbonate filter may be used in place of the punched out 11-mm filter

Collect a 10-μL aliquot of the mixture from the PLM sample preparation using the Eppendorf pipette with the modified tip and transfer to a prepared polycarbonate/adhesive tab substrate This will yield a loading on a 12-mm SEM

stub of about 100-200 µg, which is a moderately heavy loading Adjust the number of aliquots as needed to obtain the target loading

Prepare a 10-fold dilution of the above suspension to get a suspension of 1-2 mg dust per mL of isopropanol Sonicate the suspension in an ultrasonic bath for one minutes Remove the suspension and gently shake it to suspend all particles Wait one minute to allow the coarse particles to settle Collect a 10-µL aliquot of the suspended mixture using an Eppendorf pipette with the modified tip and transfer to a prepared polycarbonate/adhesive tab substrate. This will yield a loading on a 12-mm SEM stub of about 10-20 µg, which is a light loading Adjust the number of aliquots as needed to obtain the target loading.

Alternatively, prepare a lightly loaded sample using the filtration method as follows Use a Millipore filter apparatus for use with 25-mm filters for filtration Place a few drops of isopropanol on the fritted glass surface and place the 25-mm polycarbonate filter (0 4-um pore size) on the isopropanol Attach the top of the apparatus and add a few milliliters of isopropanol to the filter so that no part of it is exposed to air. Sonicate the suspension (diluted as described in previous paragraph) in an ultrasonic bath for one minute. Remove the suspension and gently shake it to suspend all particles Wait one minute to allow the coarse particles to settle Collect 1 mL of the suspended mixture using a pipette and filter it through the polycarbonate filter Actual amounts for filtration will vary based on sample loading The goal is to have a loading on a 12-mm SEM stub of about 10-20 µg, or about 5-10 percent area coverage, which is a light loading Adjust the volume of the aliquot to filter as needed to obtain the target loading

Place the filter on a carbon adhesive tab on a standard SEM aluminum mount The filter needs to be completely flat on the SEM stub This can be achieved by forming the wet filter into a gentle U-shape using forceps and the side of the forefinger, then placing the bottom curve of the filter onto the center of the carbon adhesive tab and slowly releasing the sides so they lay flat Trim the edges of the filter using a razor blade.

After drying, coat the samples on the polycarbonate or polycarbonate/adhesive tab substrates with carbon using a carbon evaporator with a rotating stage Transfer the stubs to the SEM in a clean, covered container

12.0 Analysis

12.1 Analysis by Polarized Light Microscopy

Polarized light microscopy will be conducted using the general techniques outlined in EPA 600/R93/116 (4) For this procedure, four slides (prepared as described in section 11 4) will be analyzed The fraction of fibers with refractive index greater than 1 55 (or 1 605) will contain mineral wool, which includes both slag wool and rock wool, and possibly some E-type glass and ceramic fibers The

fraction of fibers with refractive index less than 1.55 (or 1.605) will contain primarily soda-lime glass fibers. For the validation study, numbers of fibers greater than **and** less than 1 55 (1.605) refractive index will be counted Dispersion staining and becke line techniques may be used. Fiber point counting will be performed at 100 x magnification

If more than 20 mineral wool fibers are found, continue counting and recording all of the fibers above and below the index oil refractive index Report both raw fiber counts per refractive index category and number of fibers from each category per gram of sample. Continue on to step 12 2 1 to determine the ratio of slag wool to other fibers with refractive index greater than 1.55 (or 1 605) using EDS as described below

If less than 20 mineral wool fibers are found on each slide, count the number of slag wool fibers using SEM/EDS and report as number of fibers per gram of sample

12.2 Analysis by SEM/EDS

12.2.1 Screening for Slag Wool

Operating conditions for the JEOL 6460-LV SEM are 15 kV, 0 5-5-nA beam current, 10-mm working distance (system dependent), and zero degree tilt

Place the more concentrated sample deposited directly on the polycarbonate/adhesive tab substrate into the SEM. Use the backscattered electron mode at 100x magnification to quickly distinguish carbon fibers from inorganic fibers (carbon fibers may be visible, but not as bright in a BEI). Identify all inorganic fibers over 25 µm in length (smaller fibers cannot be reliably detected at the 100x operating magnification) When an inorganic fiber is found, identify the composition of the particle by EDS. Slag wool is the primary fiber of interest. Record all inorganic fiber results as number of fibers for each fiber type

For the samples with high fiber loading, as determined by PLM as described in section 12 1, count fibers per type until a statistical representation of the ratios of fiber compositions in the sample is achieved Report the ratio (by fiber number) of slag wool fibers to total MMVF fibers corresponding to the high RI Use this ratio to correct the total number for high RI fibers counted by PLM to number of slag wool fibers present

For the samples with low fiber loading, as determined by PLM as described in section 12.1, scan the entire stub to determine the number of

fibers per type Report the slag wool fiber results as the number of slag wool fibers/gram of sample

12.2.2 EDS Screening for Gypsum/Anhydrite

Place the more concentrated sample deposited directly on the polycarbonate/adhesive tab substrate in the SEM Choose a random field at 100x magnification and perform an EDS analysis on the entire field Look for the presence of sulfur in this field If sulfur is present, continue to Section 12 2 3 or 12 2 5 for analysis of gypsum and concrete by mapping or particle analysis If it is not present, repeat the analysis on another random field If sulfur is still not present, mark the sample as non-detect (ND) for sulfur

12.2.3 X-Ray Mapping for Gypsum

Place a more dilute sample, deposited directly on the polycarbonate/adhesive tab substrate or prepared by filtration, in the SEM Collect binary backscattered electron images (particles white and background black, shadow off) and secondary electron images for 10 non-overlapping, random fields at 500 x magnification Collect x-ray maps for Na, Mg, Al, Si, S, Ca, and Fe at each of these fields Fields containing MMVF will not be used for this analysis Operating parameters for the SEM are the same as those for analyzing slag wool. Acquisition parameters for x-ray mapping using the NORAN System Six Software are time constant 14 (mapping mode, 11333 cps), 10-20 % deadtime, 256 x 256 image resolution, 20 second frame time, and 100 frames collected (about 40 minutes total acquisition time) Secondary electron images will be used for reference only Save all of the maps and electron images in TIFF format

Open the backscattered electron image and the Ca and S x-ray maps in Adobe Photoshop Make sure that all of the element maps are the same size and resolution by choosing Image Size from the Image Menu and changing the pixel size or the resolution as needed The presence of gypsum can be determined by overlapping the Ca and S maps

Perform the following functions in Adobe PhotoShop (A macro is in development to perform the following functions to decrease user time and human errors in adjusting the threshold)

1 Convert each of the three images to grayscale (Image → Mode → Grayscale)
2 Perform an auto contrast and brightness on each image and map to increase the scale of colors (Image → Adjustments → Auto Levels)
3 Threshold each element map, Ca and S (do not analyze the

backscattered electron image at this time), by going to the Image Menu and choosing Adjustments → Threshold. Adjust the threshold to 128 The background will be black and the particles white

4 Invert the image (Image→ Adjustments →Invert) to make the background white and the particles black

5 Copy the S map and paste it over the Ca map in a separate layer in the file and change the opacity (located in the Layers window) to 50 % for the S map layer The black areas are gypsum/anhydrite

6 Display a histogram of the image in expanded mode by selecting the Histogram tab on the Navigator Window (or under the Image Menu in some versions of Photoshop) Place the cursor over the line for the black area and record the percentile for the black area. This is the percentage of particles containing Ca and S in the entire field.

NOTE. If a binary backscattered electron image is obtained during data collection, then steps 7-11 may be deleted The Invert function will, however, need to be applied to make the particles black and the background white before continuing to step 12.

7 Begin analysis of the backscattered electron image Select the particles by going to the Select Menu and choosing Color Range Go to the selection pulldown menu and choose Highlights

8 Fill the selection with black by going to the Edit Menu → Fill and choosing black from the color pulldown menu.

9. Select the inverse areas by going to the Select Menu and selecting Inverse

10 Fill the selection with white by going to the Edit Menu → Fill and choosing white from the color pulldown menu

11 Deselect the area by clicking on the image

12 Perform the Threshold and Histogram functions for the backscattered electron image as outlined in 3 and 6. Record the histogram result for the backscattered electron image

Determine the area percent of gypsum by performing the calculations in Section 13.0.

12.2.4 X-Ray Mapping for Ca-Rich Particles

Analysis of components of concrete will be performed on the same fields as the gypsum/anhydrite analysis At this time, only a method for the determination of the area percent of Ca-rich particles is presented See Section 2 1 for discussion.

Perform the following steps on the Ca x-ray map Tiff file in Adobe Photoshop

1 Convert the Ca x-ray map to grayscale (Image → Mode → Grayscale)
2 Perform an auto contrast and brightness on the map to increase the scale of colors (Image → Adjustments → Auto Levels)
3 Threshold the Ca map by going to the Image Menu and choosing Adjustments → Threshold Adjust the threshold to 128. The background will be black and the particles white
4 Invert the image (Image→ Adjustments →Invert) to make the background white and the particles black
5. Display a histogram of the image Place the cursor over the line for the black area and record the percentile for the black area This is the area percent coverage of particles containing Ca in the entire field

Determine the maximum area percent coverage of non-gypsum, Ca-rich particles by performing the calculation in Section 13 0

12.2.5 Particle Analysis for Identification of Gypsum and Concrete.

Place the more dilute sample, deposited directly on the polycarbonate/adhesive tab substrate or prepared by filtration, in the SEM Particle analysis will be used to identify gypsum and concrete particles

Perform particle analysis at 500 x magnification. All other operating parameters for the SEM are the same as those used to analyze for slag wool (Section 12 2 1) A binary backscattered electron image should be used in particle analysis mode. Particle analysis parameters should be set to analyze all particles in the field greater than 0 5 μm and to separate touching particles For particles greater than 5 μm, scan the entire particle, spot analysis is adequate for smaller particles The x-ray spectrum and counts for all particles, and an image of particles > 20 μm long, will be recorded and saved Other particle parameters to be reported will include the maximum, minimum, and average diameters, the aspect ratio, and area of each particle

It will be necessary to review data collected by automated software to ensure data integrity An Excel spreadsheet, in conjunction with images and x-ray data, may be used for this purpose Particles should be sorted into one of three categories. Ca-S (gypsum), Ca-rich, and Other. Aid in identification of particles may by facilitated by referencing the U.S Geological Survey's WTC Dust Particle Atlas (1) A particle classification protocol will be developed based on the data from the validation study

The number of particles analyzed will be determined using the results of the validation study For the study, the area percent of each component should be within 10% relative error or better. Typically, data for 1000 - 1200 particles should be acquired.

Results for particle analysis will be recorded as area percent gypsum and area percent concrete particles for each field and average area percent for the each component in the sample

13.0 Data Analysis and Calculations

1. To determine the concentration of slag wool in fibers/gram, perform the following calculations:

Determine the number of fibers with RI > 1 55 (or 1 605)

fibers identified – mg of sample on slide × 1000 = fibers/gram on slide

Determine the percentage of fibers with the composition of slag wool with RI > 1 55 (or 1 605)

$$\frac{\text{Fibers/gram on slide} \times \text{\# fibers identified as slag wool}}{\text{Total number of fibers identified by EDS with RI} > 1\ 55\ (\text{or } 1\ 605)} = \text{fibers slag wool/gram on slide}$$

Back calculate to the number of fibers per gram of the original sample

$$\frac{\text{Fibers slag wool/g on slide} \times \text{g after sieving} \times \text{g sample after ashing}}{\text{g before sieving} \times \text{g sample before ashing}} = \text{Total f/g of sample}$$

2 To determine the area percent of gypsum/anhydrite from the x-ray mapping procedure, perform the following calculations:

Determine the area percent of gypsum/anhydrite in each field of view

$$\frac{\text{\% of black area in Ca-S map overlay}}{\text{\% of black area in BSE image}} \times 100 = \text{area \% gypsum}$$

Calculate the average percentage of gypsum/anhydrite for the sample

$$\frac{(\text{area \% gypsum})_{f1} + (\text{area \% gypsum})_{f2} + \_\_\_}{\text{number of fields}} = \text{Avg area \% gypsum}$$

3 To determine the maximum area percentage of Ca-rich particles, which includes concrete particles, from the x-ray mapping procedure, perform the following calculations

Determine the area percent of non-gypsum Ca-rich particles in each field of view:

$$\frac{\text{(\% black area Ca map)} - \text{(\% black area Ca-S map)}}{\text{\% black area on BSE image}} = \text{\% non-gypsum Ca-rich particles}$$

Calculate the average percentage of non-gypsum Ca-rich particles for the sample

$$(\text{area \% Ca-rich particles})_{f1} + (\text{area \% Ca-rich particles})_{f2} + \_\_\_ = \text{Avg area \% Ca-rich particles}$$

number of fields

4. Calculate the area percent for gypsum and concrete by summing the areas of each particle in for each particle type and dividing by the total area analyzed.

$$\frac{\text{area gypsum 1 + area gypsum 2 + }}{\text{total area analyzed}} \times 100 = \text{area percent gypsum (do likewise for concrete)}$$

Rules for concrete and gypsum classification are currently being developed

14.0 References

1 Lowers, Heather A , Meeker, Gregory P , Brownfield, Isabelle K , 2005 World Trade Center Dust Particle Atlas U S Geological Survey Open-File Report 2005-1165 On the web at http //pubs usgs gov/of/2005/1165/

2 Meeker, G P , Taggart, J E , and Wilson, S A , 1998 A Basalt Glass Standard for Multiple Microanalytical Techniques. Proceedings Microscopy and Microanalysis 1998 Microscopy Society of America.

3 A polished and carbon coated calibration reference sample of BIR1-G may be obtained by contacting Stephen Wilson, U.S Geological Survery, MS 973, Denver Federal Center, Denver, CO, 80225, swilson@usgs gov

4 Perkins, R.L and Harvey, B.W., 1993, TEST METHOD: Method for the Determination of Asbestos in Bulk Building Materials, EPA/600/R-93/116

15.0 Appendix: DATA SHEETS

Determination of Slag Wool Fibers in Dust- PLM with Dispersion Staining

Sample ID

Project
Analyst

Circle One Original Duplicate Triplicate

Date

General Sample Appearance _____

Homogeneous? Y

Structure #	RI Fluid		Dispersion Staining		Becke Line		Fiber			Comments
	1.55	1.605	>RI	<RI	>RI	<RI	MW	non-MW	chrysotile	

SEM Sheet

Reference ASTM - D5755-03

Report Number: _____
Sample Number: _____
File Name: _____
Sample Description. _____
_____
_____
_____

Preparation Date: _____ By: _____
Analysis Date: _____ By: _____
Computer Entry Date: _____ By: _____
Sample weight: _____ grams
Dilution Volume: _____ mL
Volume Aliquot: _____ uL
Magnification: _____ X

Structure #	Field #	Fiber Type	Length (Microns)	Width (Microns)	Image	EDS

Gypsum and Concrete Report Sheet: X-Ray Mapping Procedure

Sample Number:_____ Analyst:_____ Date:_____

Field #	File Name	% Particles In EB	% Ca-S area	% Ca-rich area	% Gypsum	% Ca-rich Particles

Averages

APPENDIX C: REAC SOP 2040

STANDARD OPERATING PROCEDURES

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COLLECTION OF INDOOR DUST SAMPLES FROM CARPETED SURFACES FOR CHEMICAL ANALYSIS USING A NILFISK GS-80 VACUUM CLEANER

CONTENTS

11 0 HEALTH AND SAFETY

12 0 REFERENCES*

13 0 APPENDICES*

These sections affected by Revision 0 0

SUPERCEDES SOP #2040, Revision 0 0, 11/18/98, USEPA Contract 68-C4-0022

COLLECTION OF INDOOR DUST SAMPLES FROM CARPETED SURFACES FOR CHEMICAL ANALYSIS USING A NILFISK GS-80 VACUUM CLEANER

1 0 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to define the procedures for the collection of carpet-embedded dust samples that can be analyzed for lead, pesticides, or any other chemicals or elements This procedure is applicable for the collection of samples on a variety of carpeted surfaces This SOP may be modified to include the collection of dust adhering to floor surfaces but is not intended for the collection of dust containing asbestos fibers

These are standard (i e , typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure In all instances, the ultimate procedures employed should be documented and associated with the final report

Mention of trade names or commercial products does not constitute United States Environmental Protection Agency (U S EPA) endorsement or recommendation for use

2 0METHOD SUMMARY

Sample collection is performed utilizing the Nilfisk GS-80 vacuum cleaner equipped with a high efficiency particulate air (HEPA) filter A diagram of the Nilfisk GS-80 dust sampling apparatus is presented in Figure 1, Appendix A Soil and other particulate matter with aerodynamic diameters of approximately 5 microns (μm) and larger that are embedded within the carpet are collected, sieved and submitted to the laboratory for analysis

3 0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

Following collection of a sample into a dedicated collection bag, the bag is removed from the vacuum cleaner and placed into a 32-ounce(oz) glass jar or a zip-lock plastic bag Storage of the samples at ambient temperature is appropriate for samples that will be analyzed only for metals Samples for organic analysis should be maintained at approximately 4 ± 2 degrees Celsius (^{0}C)

4 0INTERFERENCES AND POTENTIAL PROBLEMS

There are no known interferences with this method

5 0EQUIPMENT/APPARATUS

5 1 Sampling Equipment

- Nilfisk Model GS-80 vacuum cleaner
- Two-meter folding ruler or similar device
- Masking tape

- Deionizer or distilled water
- Methanol, ACS grade
- Kimwipes ™ or equivalent
- Vacuum collection bags
- Bottle brush
- Scrub brush
- Plotlines
- 32-ounce glass jars or Ziploc® plastic bags
- Disposable gloves

5 2 Sieving Equipment

- 100-mesh sieve, 150-□m mean diameter, as specified in ASTM D 422, consisting of the cover, sieve and receiver pan
- Sieve shaker for mechanical sieving (CSC Scientific, Catalog Number 18480, Thomas Scientific, Catalog Number 8324-A10) or equivalent
- Analytical balance, capable of weighing 0 1milligrams (mg) and a range of 0 1 mg to 1000 grams (g)
- Disposable gloves
- Disposable dust mask
- Clean aluminum foil
- Kimwipes ™ or equivalent
- Camel hair brush (Fisher Scientific, Catalog Number 03-655) or equivalent

6 0 REAGENTS

Methanol and deionizer/distilled water are required for sampling train cleaning and decontamination

7 0 PROCEDURES

7 1 Preparation

The overall sampling strategy should be designed to address the goals of the study Users should consider factors such as foot traffic volume, types of activities, and proximity to potential sources The sampling strategy should be described in the Work Plan (WP), Quality Assurance Project Plan (QAPP), or Sampling and Analysis Plan (SAP) prepared prior to the sampling event The ideal sampling locations are those areas that conform to the overall sampling strategy For example, protocol may require the selection of a carpeted area for sampling where small children play or are likely to play

1 Determine the extent of the sampling effort, the sampling methods to be employed, the amount of dust needed to reach the desired detection limit and the types and amounts of equipment and supplies needed

2 Obtain and organize the necessary sampling and monitoring equipment

3 Decontaminate or pre-clean equipment, as specified in Section 7 5, and ensure that it is in working order

4 Prepare schedule and coordinate with staff, client, regulatory agency, as appropriate

5 Perform a general site survey prior to site entry in accordance with the site-specific Health and Safety Plan

6 Measure the area to be sampled and outline it using masking tape or other appropriate methods Draw a diagram of the room(s) where the sample(s) were taken, locating the sampled area(s)

7 2Calibration Procedures

The Nilfisk GS-80 vacuum cleaner has no flow devices that require calibration prior to sampling The sampling train shall be thoroughly inspected to ensure that it has been cleaned, properly assembled, and complete

7 3Field Operations

1 Prior to collecting a sample at a specific location, complete a Vacuum Sampling Work Sheet (Figure 2, Appendix A) recording all required information and sketch the area to be sampled

2 Select a sampling area according to the data collection design outlined in the WP, QAPP or SAP Typically, three rooms per floor are selected for sampling in each building Each sample is collected with a dedicated sampling train that has been properly assembled, cleaned, and decontaminated to ensure sample integrity The size/weight of each sample is dependent on the goals and objectives of the sampling event, the analyses requested, and the desired method detection levels (MDLs) A 100-g sample is highly desirable if multiple analyses (metals, pesticides, etc) are requested A minimum 5- to 10-g sample is required for metal analysis only

3 Using the 2-meter folding ruler or any other measuring device, outline and mark the recommended 1-square meter (m^2) portion of the carpet to be sampled

4 Begin collecting sample at one corner of the delineated sample area, moving the sampler back and forth four times over a strip running in a straight line between the defined

sampling area edges The width of the strip is defined by the width of the sampling nozzle After completing the first strip, angle over to the second strip gradually on the next pass, again completing four double passes

5 Continue sampling the delineated area until an adequate sample is collected Visual observation is used to determine if enough sample has been collected from the recommended 1-m^2 area or if a larger area is required If sampling a larger area, measure the area accurately and document accordingly

6 Wearing surgical gloves, be sure to tap with your hand on the nozzle inlet to dislodge any dust remaining in the nozzle or the hose This procedure will ensure complete sample recovery Turn off the vacuum cleaner and allow to sit undisturbed for at least 30 seconds Unsnap the two vacuum container clips to access the inside of the container Remove the polyliner and the vacuum collection bag within it Seal off the polyliner with the vacuum collection bag inside, and transfer to a properly labeled 32-oz glass jar or plastic bag depending on the analysis(es) to be performed Document the sample information on the Vacuum Sampling Work Sheet and pack properly for shipment to the laboratory

7 Remove the hose and the nozzle, and install a new polyliner and collection bag for the collection of additional samples

8 Decontaminate the vacuum components using the steps outlined in Section 7 5

7 4 Sieving Procedures

Prior to submitting dust samples to the laboratory for analysis, the samples are sieved through a 100-mesh sieve using the following procedure

1 Select a clean working area in a facility equipped with a fume hood (a 4-foot by 4-foot area is sufficient) Weigh the receiver pan on an analytical balance and record the weight

2 Wearing clean surgical gloves and a dust mask, retrieve the vacuum collection bags from the 32-ounce glass jars used to transport the bags from the field to the laboratory

3 Empty the entire contents of the bag into the 100-mesh sieve with the receiver pan attached Remove the plastic adaptor (blue ring) from the collection bag inlet and shake the bag as necessary to ensure all the contents have been transferred into the sieve

4 Place the cover on the sieve and manually or mechanically shake the sieve for a minimum of 5 minutes and a maximum of 10 minutes until all the fine dust particles are collected in the bottom receiver pan If manual shaking is performed, follow the instructions given in American Society for Testing and Materials (ASTM) D-422 "Conduct the sieving operation by means of a lateral and vertical

motion of the sieve, accompanied by a jarring action in order to keep the sample moving continuously over the surface of the sieve Continue sieving until not more than 1 mass percent of the residue on a sieve passes that sieve during 1 minute of sieving"

If mechanical shaking is performed, set up the recommended sieve shaker on an even and stable surface Proceed with the sieving operation following directions in the manufacturer's manual

5 Re-weigh the receiver pan using an analytical balance The difference in weight is the weight of the sieved sample If total weight of material is desired, the coarse material remaining on top of the sieve must be collected on a pre-weighed sheet of aluminum foil, re-weighed and the weight added to the weight of the sieved sample

6 Transfer the sieved sample from the receiver pan to an 8-oz wide-mouth glass jar Use a camel hair brush to ensure complete transfer of the sample Cap the glass jar securely

7 Document each sample Each sample must be provided with the following information identification number, date of sampling, location, analysis requested Each sample must be recorded onto a Chain of Custody form before delivery to the analytical laboratory

8 Before processing the next sample, thoroughly wipe clean the cover, sieve and receiver pan using a Kimwipe™ and deionized/distilled water Let dry prior to sieving additional samples

7 5 Sampling Train Decontamination

To decontaminate the sampling trains, move them to a well-ventilated area and perform the following

3 Assemble one of the sampling trains to be used as the decontamination unit for decontaminating the nozzles, hoses, and wands This unit must be equipped with a clean polyliner and dust bag

2 With the vacuum cleaner turned on, decontaminate the nozzles, wands, and hoses using a bottle brush to remove any accumulated dust in the hose and nozzle Be sure to tap the nozzle with your hand to remove any visible dirt that has accumulated, and use the scrub brush to remove any hair or fibers entangled on the nozzle's brush When the nozzle is considered to be clean, remove and spray with reagent grade methanol and allow to air dry on a clean surface The wand and hose are then cleaned with the bottle brush Tap your hand on the wand inlet while cleaning with the bottle brush to remove any visible dirt Repeat this procedure to decontaminate any remaining nozzles, wands, and hoses

2 Remove the used dust bag from the decontamination unit and wipe clean the inside of the

container with deionized/distilled water Spray the inside of the containers with methanol and allow to air dry When decontaminating in between residential homes, cleaning the inside of the containers with deionized/distilled water is sufficient

8 0 CALCULATIONS

The dust weight calculations for the final sieved dust fraction is performed in accordance with ASTM Method D-422 Dividing the final dust weight by the area sampled (expressed in m^2) provides dust loading in grams per squared meter (g/m^2) When the analysis results are received, the loading of analyte in micrograms per square meter of carpet area (ug/m^2) can be calculated in the same way The analysis provides concentrations in milligrams/kilogram (mg/kg) or micrograms/kilogram (□g/kg) If total (gross) dust loading of the sampled area needs to be calculated, the total dust weight before sieving must be obtained The total dust weight is divided by the area sampled to obtain total dust loading in g/m^2

9 0 QUALITY ASSURANCE/QUALITY CONTROL

There are no specific quality assurance activities which apply to the implementation of these procedures However, the following general QA procedures apply

1 All data must be documented on field data sheets or within site logbooks

2 All instruments must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan Equipment checkout and calibration activities must occur prior to sampling/operation and they must be documented

10 0 DATA VALIDATION

The information recorded during sampling will be used in conjunction with the analytical data during validation

11 0 HEALTH AND SAFETY

When working with potential hazardous materials, follow U S EPA, Occupational Safety and Health (OSHA) and corporate health and safety procedures

12 0 REFERENCES

American Society For Testing And Materials 2000 *Standard Practice for Collection of Dust from Carpeted Floor for Chemical Analysis,* Designation D 5438-00, Reprinted from the Annual Book of ASTM Standards, Philadelphia, PA

American Society For Testing And Materials 1998 *Standard Test Method for Particle Size Analysis of Soils*, Designation D 422-63, Reprinted from the Annual Book of ASTM Standards, Philadelphia, PA

Instructions for Use-Nilfisk Model GS 80, Nilfisk of America, Inc ,Malvern, PA (1987)

13 0 APPENDICES

A - Figures

APPENDIX A
Figures
SOP #2040
May 2002

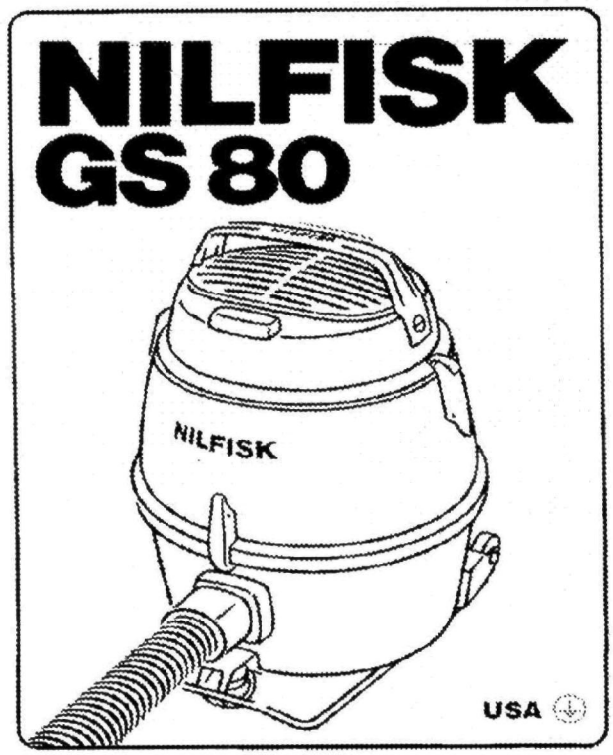
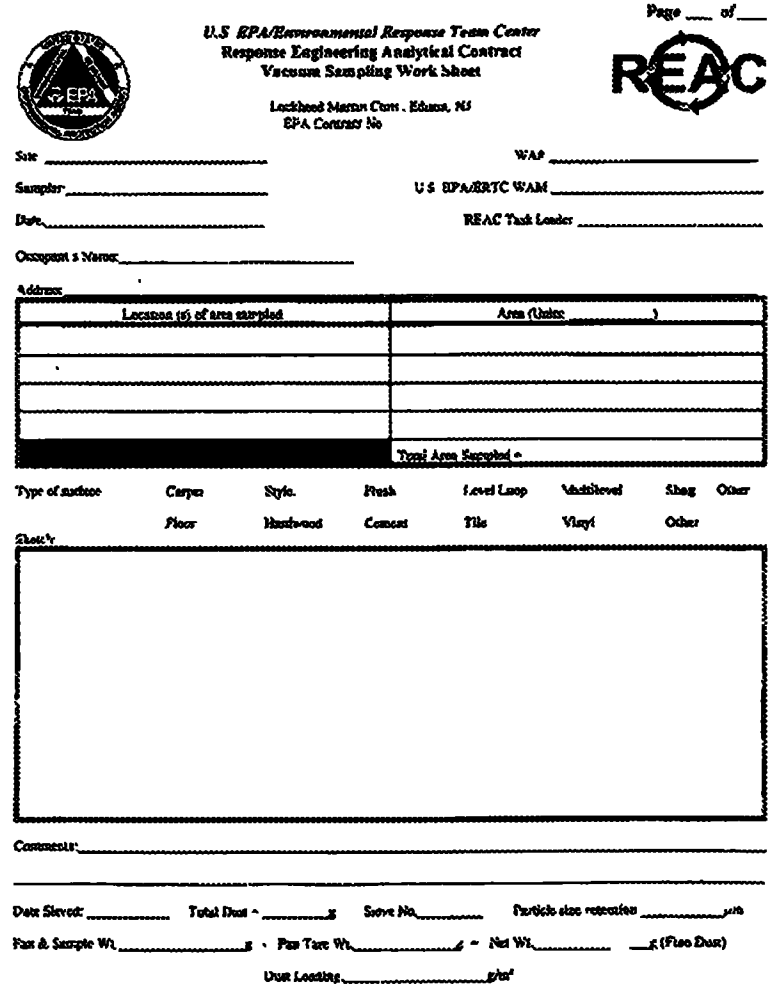


FIGURE 1. GS-80

Dust Sampling Apparatus



U.S EPA/Environmental Response Team Center
Response Engineering Analytical Contract
Vacuum Sampling Work Sheet

Lockheed Martin Corp., Edison, NJ
EPA Contract No

Page ___ of ___

Site _____ WA# _____

Sampler _____ U.S EPA/ERTC WAM _____

Date _____ REAC Task Leader _____

Occupant's Name: _____

Address: _____

Location (s) of area sampled	Area (Units: _____)
	Total Area Sampled =

Type of surface: Carpet, Style, Plush, Level Loop, Multilevel, Shag, Other
Floor, Hardwood, Cement, Tile, Vinyl, Other

Sketch:

Comments: _____

Date Sieved: _____ Total Dust = _____g Sieve No._____ Particle size retention _____µm

Pan & Sample Wt. _____g - Pan Tare Wt._____g = Net Wt._____ ___g (Fine Dust)

Dust Loading _____g/m²

FIGURE 2 Vacuum Sampling Work Sheet

SIEVE CLEANING

SCOPE AND APPLICATION

Sieves must be cleaned before each dust sample is separated into fractions Most of the "near-mesh size" particles can usually be removed from the apertures by inverting the sieve and gently tapping the frame of the sieve For sieves with apertures less than 1 millimeter (mm) (e g , 100-mesh, 150 micron [□m] sieve), the most effective method for cleaning the apertures is the use of an ultrasonic bath

EQUIPMENT/APPARATUS

- Ultrasonic bath, capable of holding a standard sieve
- Magnifying glass
- Source of air, standard hair dryer or compressed air
- Spray bottle

REAGENTS

- Ultrasonic cleaner or laboratory-grade detergent that leaves no interfering residues
- Deionized (DI) water, Type II water or equivalent
- Methanol, American Chemical Society (ACS) grade or equivalent

PROCEDURE

The following cleaning procedure will be used to clean sieves prior to use and after each sample

1 Place the sieve into an ultrasonic bath containing detergent and DI water and sonicate for approximately 10 minutes

2 Remove the sieve from the ultrasonic bath and rinse well with DI water

3 Spray the sieve with methanol

4 Dry the sieve using a standard hair dryer or a compressed air source

5 Visually inspect the sieve to ensure that there are no remaining particles present in the apertures A magnifying glass may be used to aid in this process

6 Repeat steps 1 through 5 prior to sieving subsequent samples

APPENDIX D: ACCESS AGREEMENT

REQUEST FORM
for the U.S. Environmental Protection Agency's Effort to Develop and Validate a WTC Dust Signature and to Characterize Background Dust in New York City

Name of Occupant:

Address:

Apartment Number (if applicable)

Contact Phone Numbers

REQUEST

I have read the fact sheet on the U S Environmental Protection Agency's Program to develop and validate a WTC dust signature and to characterize background dust in New York City and considered the information provided to me by the U S Environmental Protection Agency (EPA) Having considered the information regarding the sampling program, I would like to participate

AGREEMENT

On behalf of myself and any other occupants, I agree to the following

I consent to employees, authorized representatives and contractors of the EPA having access to the above referenced space for as long as necessary to conduct dust sampling activities

I agree to obtain any *required* permission for sampling activities from my building management I also agree to inform the EPA Project Monitor at least **one business day** prior to the scheduled work of any building rules that are applicable to the program, including time restrictions, appropriate entrances to the building, and elevator usage

I understand that the sampling will be performed by contractors retained by EPA I also understand that the contractors performing sampling activities are required to maintain insurance coverage for commercial general liability, workers compensation, dishonest acts of their employees and environmental impairment liability related to this work The contractors are required to maintain such insurance at all times that they are conducting sampling activities The contractors are responsible for damage or loss of property

I understand that the activities will require access to interior spaces and the use of electricity Sampling activities will be performed throughout the entire space

I understand that the program will employ various methods of dust removal from surfaces, including, but not limited to, vacuuming and wet wiping

COMMUNICATION OF RESULTS

I understand that I will receive a copy of the sampling results for the residence once an analysis has been completed and the data are quality assured Depending upon analysis and review time, these results may not be available until up to six months after sampling.

I understand that results provided for locations sampled under the signature study will only indicate whether WTC dust signature components are present, absent or inconclusive Results provided for locations sampled under the background study will indicate the presence of WTC signature dust, as well as the presence and levels of the contaminants of potential concern (COPC)

I understand that an explanation of the findings will be included in these results along with the name and contact information for a U S. EPA toxicologists/risk assessor This person will be able to answer questions regarding data interpretation and health-related issues

I understand that monitoring data in EPA's database for this effort will be made available to the public, but the identity of the participants and the specific location of the sampling will be kept confidential

AUTHORIZED SIGNATURE

I certify that I am authorized to grant this request on behalf of all the occupants of the above specified space, and I grant this request and agree to its terms

_____ _____

Signature Date

_____

Name and Title (PRINT)

_____ _____

Signature of U S EPA Representative Date

APPENDIX E: INFORMATION SHEET

The U.S. Environmental Protection Agency's Program to Develop and Validate a WTC Dust Signature and to Characterize Background Dust in New York City

The September 11, 2001 attack on the WTC covered a large area with dust, debris, and combustion by-products In order to determine if residual contamination exists, and to identify areas that may be in need of clean up, the U S EPA has undertaken studies both to identify a unique WTC dust signature, and to characterize typical indoor dust from NY City. In order to complete these studies, the EPA is seeking to acquire samples of urban dust from buildings both inside and outside of the area of lower Manhattan that was impacted by the WTC collapse You are being asked to participate in the study checked below

WTC DUST SIGNATURE STUDY

The U.S Environmental Protection Agency has initiated a study to define signatures for WTC dusts The purpose of this study is to develop and validate one or more "signatures" in indoor dust that can be used to determine whether dust sampled is from the collapse of the World Trade Center towers or not. A "signature" is a chemical or physical characteristic of a material that can be used to identify that specific material and discriminate between the material sought (WTC dust, in this case) and other similar materials (NYC urban dusts). The signature materials are not necessarily related to health concerns The signature could be something harmless but unique to the WTC source, measured only to identify the origin of other chemicals of concern that occur in the same sample The WTC signatures, if they can be developed, will support analysis to discriminate between normal indoor dusts and WTC-generated dusts

Samples from approximately 20 buildings are needed for validation of the proposed signatures Samples will be collected from approximately 10 buildings in the area that is suspected to be affected by WTC emissions, and samples will be obtained from 10 buildings that are not suspected of being affected

SAMPLING METHODS

Dispersion models, photos, interviews, and satellite data will be reviewed to discern areas that were likely impacted by WTC emissions In each building identified for sampling, dust samples will be collected from at least three areas 1) one sample from a track-in area near a building entrance, preferably in a carpeted area, 2) two samples from relatively undisturbed areas (e g , on top of bookcases, under furniture), and 3) other areas showing visible accumulation of settled dust, including HVAC ducts A standard method using a HEPA vacuum collector will be used by EPA to collect bulk dust samples Samples will be sealed and stored under refrigeration in a limited access area

To ensure that these important samples are properly collected, tracked, stored, and distributed, comprehensive quality assurance (QA) procedures will be in place prior to any sample collection There will be a pre-sampling survey of building and sampling areas, to include photos of sampling areas (if permitted by building owners) and notes on building usage, to identify conditions that might compromise samples (e g , smoking or cooking areas)

Dust samples from background and affected locations will be made available to researchers involved in developing and evaluating WTC signatures, as well as researchers characterizing typical NY City dust When the results of this work are complete, EPA will develop and release reports on these studies

COMMUNICATING RESULTS

Publicly released results will not be provided by name or specific location, thus a resident's privacy will always be preserved. The occupant will receive a copy of the sampling results for their residence once an analysis has been completed and the data are quality assured. Depending upon analysis and review time, this may take up to six months An explanation of the findings will be included in these results along with the name and contact information for a U S EPA toxicologists/risk assessor This person will be able to answer questions regarding data interpretation and health-related issues Finally, results provided for residences sampled under the signature study will only indicate whether WTC dust signature components are present, absent or inconclusive