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

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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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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

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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

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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.

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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.

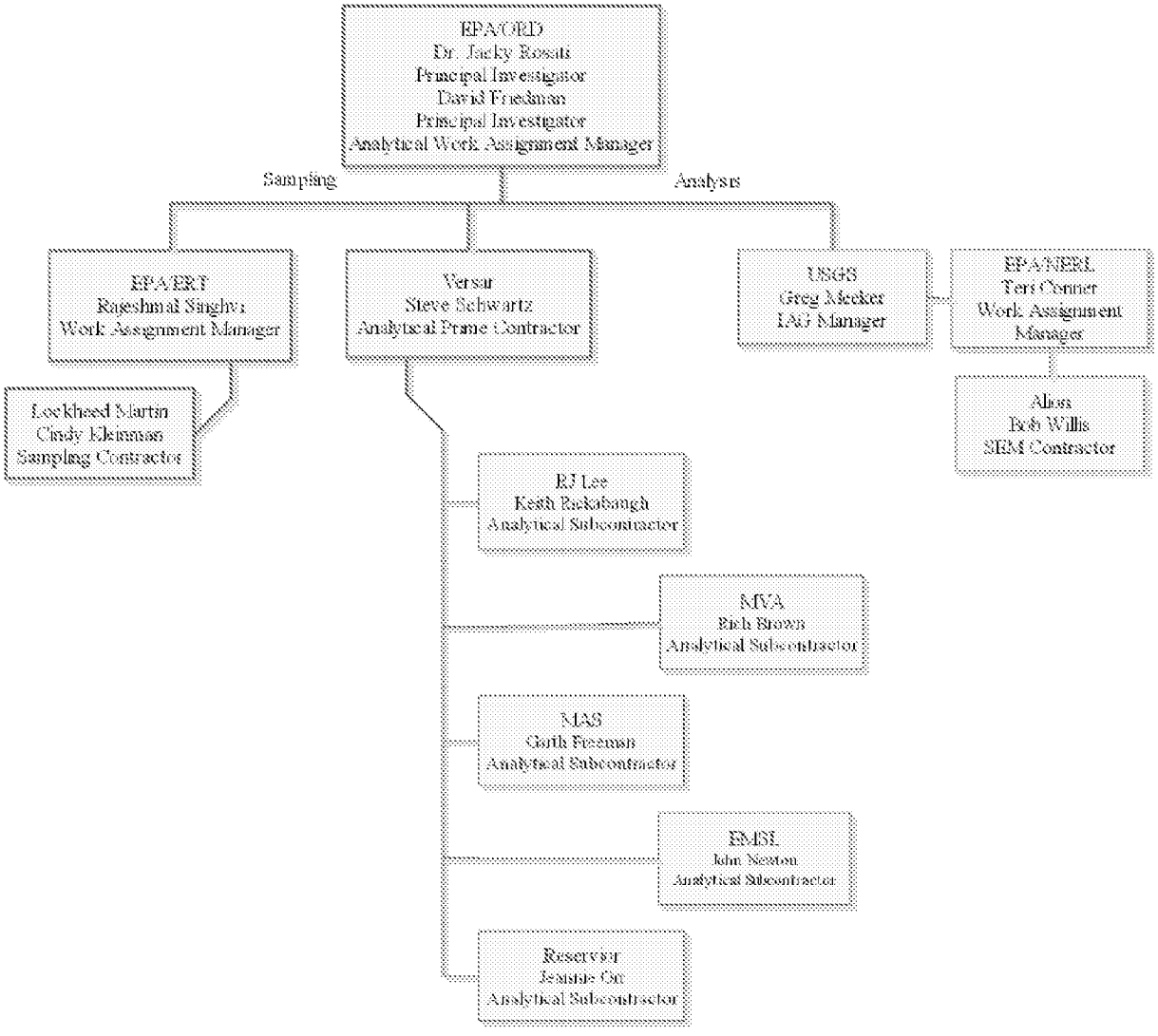


Figure 1: Project Organizational Chart

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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

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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.

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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

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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

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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)

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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.

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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

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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:

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- 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)								

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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.

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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.

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- 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.

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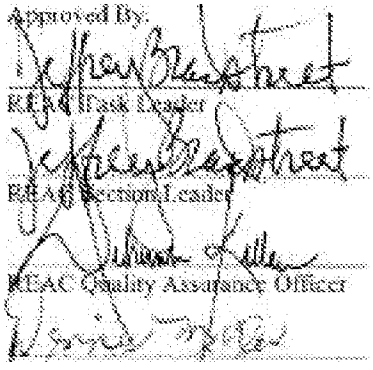
APPENDIX A: SAMPLING QAPP

Al. 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



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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

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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

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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)

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- 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

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

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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,

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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

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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 ail 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.

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

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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 eacii 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.

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

Analytical 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 +1-2 degrees C	Up to 9 per Building	9
Dust/Settled Particulate	Sweep	Up to 2 years at 4 degrees C +1-7. degrees C	Up to 9 per Building	9

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

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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

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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

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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

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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

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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.

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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

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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

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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

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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

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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:

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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.

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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.

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

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:

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

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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:

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

Rules for concrete and gypsum classification are currently being developed.

14.0 References

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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	

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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

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Gypsum and Concrete Report Sheet: X-Ray Mapping Procedure

Sample Number:_____ Analyst:_____ Date:_____

Field#	File Name	% Particles in EDS	% Ca-S area	% Ca-rich area	% Gypsum	% Ca-rich Particles

Averages

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APPENDIX C: REAC SOP 2040

REAC
Response Engineering and Analytical Contract

STANDARD OPERATING PROCEDURES

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05/17/02

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

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2.0 METHOD SUMMARY*

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE*

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

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- 5.1 Sampling Equipment*
- 5.2 Sieving Equipment*

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7.0 PROCEDURES*

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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

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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

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- 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-µm 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.

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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).

7.2 Calibration 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.

7.3 Field 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

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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

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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

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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 (µg/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.

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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

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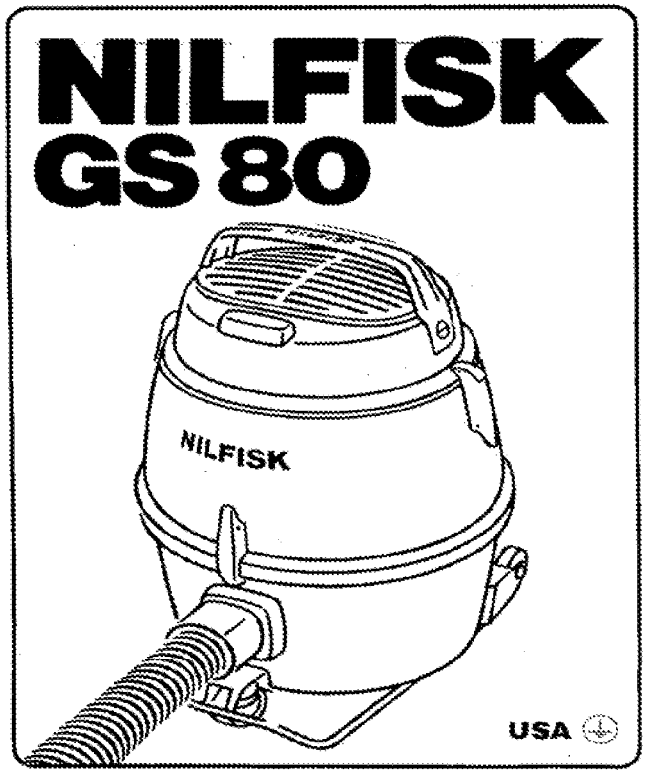


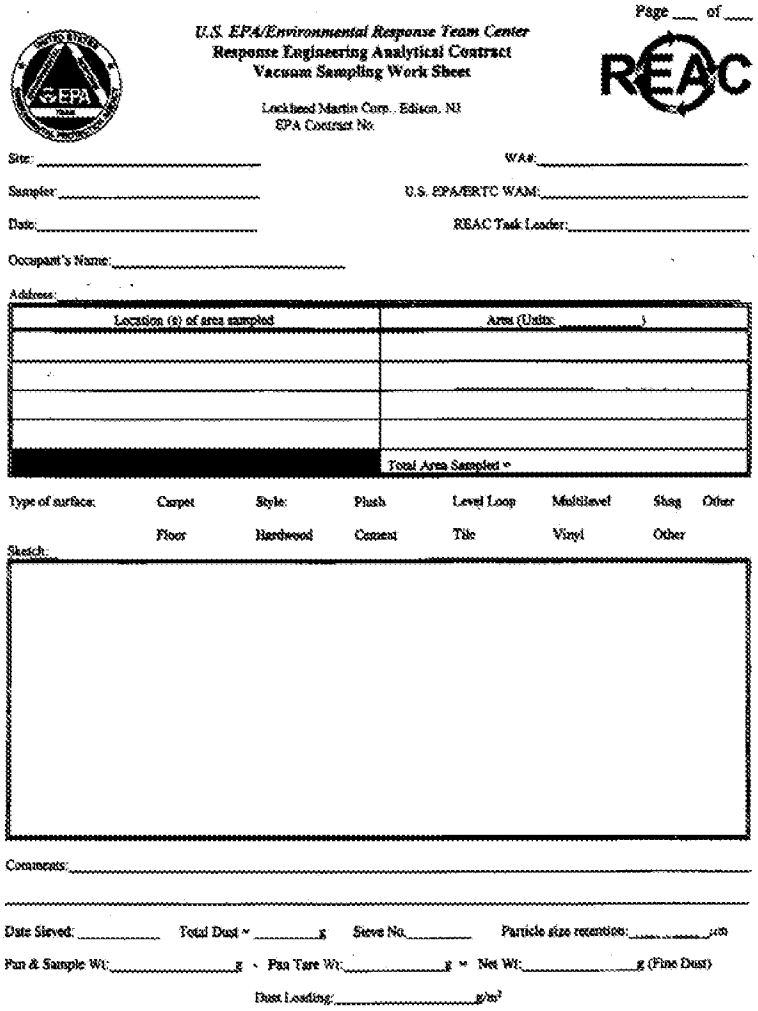
FIGURE 1. GS-80

Dust Sampling Apparatus

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

Lockheed Martin Corp., Edison, NJ
EPA Contract No.

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: _____cm

Pan & Sample Wt: _____g - Pan Tare Wt: _____g = Net Wt: _____g (Fine Dust)

Dust Loading: _____g/m²

FIGURE 2. Vacuum Sampling Work Sheet

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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 [μm] 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.

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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.

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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.

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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

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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).

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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.

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