

Peer Review of the U.S. Environmental Protection Agency's "Final Report on the World Trade Center (WTC) Dust Screening Study"

—Final Report—

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Note

This report was prepared by six peer reviewers and Eastern Research Group, Inc. (ERG), under contract to the U.S. Environmental Protection Agency (EPA) (Contract No. 68-C-02-060, Task Order 107). This report summarizes discussions from a conference call during which the six peer reviewers responded to nine charge questions regarding EPA's "Final Report on the World Trade Center (WTC) Dust Screening Study." This report summarizes key points raised during the conference call. This report does not contain a verbatim transcript of all issues discussed during the conference call, nor does it embellish, interpret or enlarge upon matters that were incomplete or unclear. Except as specifically noted, no statements in this report represent analyses by or positions of EPA or ERG.

Table of Contents

Note.....	i
List of Abbreviations	iv
Executive Summary	1
1.0 Introduction.....	3
1.1 Background.....	3
1.2 Scope of the Peer Review	4
1.3 Report Organization.....	6
2.0 Selection of a Signature	7
2.1 Charge Question 1: Selection Criteria	7
2.2 Charge Question 2: Slag Wool as a Signature	8
3.0 The Proposed Analytical Method	10
3.1 Charge Question 3: Detail of Proposed Analytical Method	10
3.2 Charge Question 4: Suggested Improvements to the Proposed Analytical Method.....	11
4.0 Data Analysis and Interpretation	12
4.1 Charge Question 5: Study Design.....	12
4.2 Charge Question 6: Statistical Analysis of Data.....	13
4.3 Charge Question 7: Data Interpretation	15
5.0 General Considerations and Alternate Approaches	16
5.1 Charge Question 8: Suggested Improvements.....	16
5.2 Charge Question 9: Other Comments	18
6.0 References.....	21

Appendix A. List of Peer Reviewers

Appendix B. Charge to Reviewers and Preliminary Written Comments

Appendix C. Responses to Questions of Clarification

List of Abbreviations

COPC	contaminant of potential concern
EDS	energy dispersive spectrometry
EPA	U.S. Environmental Protection Agency
ERG	Eastern Research Group, Inc.
NAIMA	North American Insulation Manufacturers Association
PLM	polarized light microscopy
PM	particulate matter
SEM	scanning electron microscopy
TEM	transmission electron microscopy
TIMA	Thermal Insulation Management Association
USGS	U.S. Geological Survey
WTC	World Trade Center

Executive Summary

This report summarizes an independent peer review of the U.S. Environmental Protection Agency's (EPA's) "Final Report on the World Trade Center (WTC) Dust Screening Study" (EPA 2005a). In this study, EPA developed an analytical method to screen bulk dust samples for mineral slag wool, particles consistent with concrete compositions, and gypsum (EPA 2005b). The study included a method validation component, in which spiked background dust samples with varying levels of WTC dust were prepared and then eight laboratories measured concentrations of candidate WTC signatures in spiked and non-spiked samples. Based on the data collected during this study, EPA proposed slag wool as a signature constituent of WTC dust.

Six expert peer reviewers with various affiliations and from relevant scientific disciplines were asked to provide an independent peer review of the WTC Dust Screening Study. The peer reviewers prepared preliminary written comments on the study and further discussed these comments during a 4-hour conference call on October 4, 2005. The peer review of the WTC Dust Screening Study focused on nine charge questions, which asked the peer reviewers to comment on EPA's proposed analytical method, data analysis and interpretation, and selection of slag wool as a signature for WTC dusts.

Following are the peer reviewers' main findings, organized by topic. The remainder of this report documents the discussions among the reviewers that led up to these findings.

- *Proposed Analytical Method.* The peer reviewers had concerns about laboratories' abilities to implement the proposed analytical method consistently or correctly, given that three out of the eight laboratories selected to participate in the method validation study failed to produce data of acceptable quality. Even after representatives from the eight laboratories "attended a 2-day session during which the method was further developed and discussed" and "all laboratory participants held weekly conference calls as the analytical program was proceeding to discuss general issues with the protocol" (EPA 2005a), data had to be massaged to differentiate WTC dust from background dust. The peer reviewers recommended several improvements to the proposed analytical method and recommended that EPA establish strict criteria for identifying and enabling "qualifying laboratories," should EPA decide to move forward with slag wool as a WTC signature. Refer to the summary statements in Sections 3 and 5 of this report for the peer reviewers' specific recommendations for improving and implementing the proposed analytical method.
- *Data Analysis and Interpretation.* The peer reviewers were skeptical that EPA's evaluation and interpretation of the study data were performed fairly. Peer reviewers pointed to several non-standard steps taken to enhance the study's ability to distinguish WTC dust from background dust and noted that these steps could be interpreted as attempts to prove the method's success rather than to objectively evaluate its real-world potential for fingerprinting WTC dust. The peer reviewers' two most notable concerns were:
 - The reported difference in slag wool levels for impacted and non-impacted locations was based on statistical analyses that excluded certain results from

background samples but did not exclude results from spiked samples. The background results and spiked sample results were statistically indistinguishable when the entire data set was considered.

- The study authors disqualified three out of the eight original selected laboratories during the screening study. There was no follow-up investigation to determine why these selected laboratories, presumably following the proposed method, failed to differentiate background dusts from WTC dusts. [After the peer review conference call, one peer reviewer noted that a possibility exists that the three laboratories were wrongly disqualified due to errors in an equation used to calculate the numbers of slag wool fibers per gram of dust. Section 4.2 describes this further.]

Refer to Section 4 of this report for more detailed information on the peer reviewers' comments regarding data analysis and interpretation.

- *Selection of a WTC Signature.* The peer reviewers supported EPA's conclusion that gypsum and elements consistent with concrete do not meet the WTC signature selection criteria. Regarding slag wool, the peer reviewers agreed that, from the data provided, EPA has not made the case that its proposed analytical method can reliably discriminate background dust from dust contaminated with WTC residue. Thus, the proposed method has not demonstrated the utility of slag wool as a successful signature constituent. This finding was based on critical reservations, stated above, regarding the proposed analytical method and the data analysis and interpretation. Section 2 of this report further summarizes the peer reviewers' discussions on this topic.
- *General Considerations and Alternate Approaches.* At the end of their conference call, the peer reviewers offered several recommendations to EPA for identifying a WTC signature. The reviewers classified some of these recommendations as modifications to the proposed analytical method, such as reconsidering the utility of polarized light microscopy (PLM), using fiber dimensions (in addition to fiber counts) to "fingerprint" dusts, and implementing a tiered sampling approach that would use slag wool as a screening marker for the potential presence of WTC dust followed by transmission electron microscopy (TEM) analysis of chrysotile asbestos as a confirmatory marker. The reviewers acknowledged that further evaluation would be necessary to assess the utility of these and other suggested modifications. The peer reviewers classified their remaining recommendations as entirely new approaches that could only be investigated through new research projects. Section 5 of this report lists all of the general considerations and alternate approaches the peer reviewers discussed during the conference call.

The remainder of this report presents additional information on the independent peer review of the WTC Dust Screening Study: Section 1 presents a detailed account of the peer review process; Sections 2 to 5 summarize how peer reviewers responded to the nine charge questions during the peer review conference call; and Appendix B includes copies of the peer reviewers' preliminary written comments.

1.0 Introduction

This report summarizes six experts' independent peer review of the U.S. Environmental Protection Agency's (EPA's) "Final Report on the World Trade Center (WTC) Dust Screening Study" (EPA 2005a). This report refers to the review document as the WTC Dust Screening Study.

Eastern Research Group, Inc. (ERG), under contract to EPA, organized and implemented this independent peer review according to procedures outlined in EPA's "Peer Review Handbook" (EPA 2000). Between August 19, 2005 and September 23, 2005, the six peer reviewers prepared preliminary written comments on the WTC Dust Screening Study. On October 4, 2005, the peer reviewers participated in a 4-hour conference call to discuss their preliminary written comments and to develop summary statements on several key issues.

The six peer reviewers and a technical writer from ERG wrote all of the information that appears in this summary report, except for Section 1.1, which comes almost entirely from EPA's charge to the peer reviewers. This introductory section provides background information on the WTC Dust Screening Study (Section 1.1), describes the scope of the peer review (Section 1.2), and outlines the organization of this report (Section 1.3).

1.1 Background

In the days following the terrorist attack on New York City's WTC towers, EPA, other federal agencies, and New York City and New York State public health and environmental authorities initiated numerous air monitoring activities to better understand the ongoing impact of emissions to the outside environment from that disaster. In 2002, EPA Region 2 turned its attention to the indoor environment, providing a volunteer "clean and test" or "test only" program for residents and homeowners who wished to have their apartments cleaned or tested. Asbestos was selected as a surrogate for the presence of WTC-related contamination for this program.

In March 2004, EPA convened the WTC Expert Technical Review Panel to interact with EPA and the public on plans to monitor for the presence of any remaining WTC dust in indoor environments. For more information on the WTC Expert Technical Review Panel, refer to the website: www.epa.gov/wtc/panel. Approximately 750 units in an area extending north to Houston Street in lower Manhattan and across the East River into a portion of Brooklyn were proposed to be sampled for contaminants of potential concern (COPC), as well as for specific constituents that can be used as markers to identify residual contamination by dust from the WTC collapse. These "WTC dust signature" constituents are critical to the sampling program, as they will provide the basis for estimating the geographic extent of the remaining residue in dust of the WTC collapse. Using the results of dust sampling for the WTC collapse, EPA will decide whether indoor cleanup or other activities are warranted.

Based on previous work of the United States Geological Survey (USGS) and others, EPA identified three components of the dust generated by the WTC collapse that could be used to screen sampled dust for the presence of WTC dust. These markers, or signature components, were mineral slag wool, gypsum, and elements of concrete. EPA developed the following working hypothesis for the signature: "A dust sample that contains WTC dust will have slag

wool and elements of concrete and gypsum present in ‘significant quantities’ when compared to typical indoor urban dust.” Experts from the USGS, EPA’s Office of Research and Development, EPA’s National Enforcement Investigations Center, and the commercial testing laboratory community worked together to develop an analytical method to quantify the concentration of these three markers in indoor dust.

Between September 2004 and April 2005, numerous samples were taken in impacted buildings near Ground Zero and at background locations. These samples have been analyzed for these three markers to determine whether they validly constitute a WTC signature, as suggested by earlier USGS efforts. Also, the samples were used in a method validation study whose primary purposes were twofold: (1) to evaluate the analytical method with regard to method variability (both inter- and intra-laboratory variability) and cost and expediency and (2) to assist in the determination of the lower limits of concentrations of the markers that could be reliably measured and that could be reliably distinguished from background concentrations. This method validation study involved five contract laboratories and three federal government laboratories. This study entailed spiking known background dust with varying concentrations of known WTC collapse dust, and then having the laboratories perform a blinded analysis on both spiked samples and background samples.

EPA’s study is documented more fully in the WTC Dust Screening Study, which was the subject of this peer review. EPA contracted with ERG to implement an independent peer review of the study. The remainder of this report documents this peer review. More information on EPA’s response to environmental issues associated with the WTC collapse can be found on the following website: www.epa.gov/wtc.

1.2 Scope of the Peer Review

ERG managed every aspect of this peer review, including selecting reviewers and coordinating activities before, during, and after the peer review conference call. The following sections describe what each of these tasks entailed.

1.2.1 Selecting the Peer Reviewers

ERG selected peer reviewers that met the selection criteria EPA specified in its task order for this project. Those criteria noted that: “The expertise that is desired include, but is not limited to, these fields of study: polarized light microscopy (PLM), scanning electron microscopy (SEM), transmission electron microscopy (TEM), particulate matter (PM) sampling methods, quality assurance procedures, analytical method development, and validation procedures.”

Based on these selection criteria, ERG developed a list of candidates for this peer review. Many candidates were identified by an ERG search for subject matter experts. Additional candidates were identified through a peer reviewer nomination process. In July, 2005, EPA announced the procedure by which any interested party could nominate candidates for this peer review project. ERG contacted every nominee that was received. Through these two search efforts, ERG identified more than 50 highly qualified candidate peer reviewers. ERG asked all candidates to submit information on their expertise, availability, and potential conflicts of interest. For this peer review, a conflict of interest was defined as a situation in which an individual’s activities,

interests, or relationships create a situation where the candidate may benefit from the outcome of the review.

After carefully reviewing the candidates' expertise and credentials, ERG selected six peer reviewers who, as a group, met the selection criteria and were able to provide a fair, independent, and scientifically rigorous peer review of the WTC Dust Screening Study. Appendix A lists the peer reviewers' names and affiliations. Recognizing that few individuals specialize in every scientific discipline listed in the selection criteria, ERG ensured that the collective expertise of the selected peer reviewers adequately covers the criteria (e.g., at least one reviewer has expertise in PLM, at least one reviewer has expertise in method validation studies, and so on). ERG selected peer reviewers of varying affiliations (e.g., academia, consulting companies, analytical laboratories, government agencies) in hope that the peer reviewers would offer a balanced perspective on the WTC Dust Screening Study.

1.2.2 Activities Prior to the Peer Review Conference Call

Major activities that ERG and the peer reviewers conducted prior to the peer review conference call follow:

- *Distribute peer review documents and background information.* On August 19, 2005, ERG sent the six peer reviewers packages that contained three primary review documents and four background documents. The primary review documents were the WTC Dust Screening Study (EPA 2005a), the quality assurance project plan for the study (EPA 2005c), and written guidelines for the peer review. These guidelines — commonly called a “charge” — were nine questions that addressed various aspects of the WTC Dust Screening Study, including an open-ended question that invited the peer reviewers to comment on any topics that the other questions did not explicitly address. A copy of the charge is included in this report as part of Appendix B.

The packages ERG sent to the peer reviewers also included four documents with background information. These documents were distributed to give peer reviewers additional insights on the previous characterization of WTC dusts. These background documents included three publications by USGS scientists (Meeker et al. 2001; Lowers et al. 2005; Clark et al. 2005) and an external review draft of EPA's proposed sampling program to determine the extent of WTC impacts to indoor environments (EPA 2004). The peer reviewers were also directed to the website for the WTC Expert Technical Review Panel for online access to many additional documents that contain relevant background information.

- *Facilitate questions of clarification.* The peer reviewers worked independently for 5 weeks to review the WTC Dust Screening Study and to prepare preliminary written responses to the nine charge questions. During this time, ERG asked the peer reviewers to refrain from discussing the scientific merit of the WTC Dust Screening Study with EPA or any other party. However, ERG recognized that peer reviewers might have questions of clarification about the study during the review process. ERG asked the peer reviewers to forward all such questions in writing to ERG. ERG then sent these questions to EPA for written responses. By this approach, all of the reviewers' questions of clarification

and all of EPA's written responses have been documented. Copies of the questions and answers are included in this report as Appendix C.

- *Obtain and compile the peer reviewers' preliminary written comments.* The peer reviewers' preliminary written comments were due to ERG on September 23, 2005. After receiving the complete set of comments, ERG then compiled, bound, and distributed these comments to the six peer reviewers. The peer reviewers' preliminary written comments are included in this report, without modification, as Appendix B. It should be noted that these written comments should be considered preliminary and some reviewers' technical findings might have changed based on discussions during the conference call. The preliminary written comments do not necessarily reflect the reviewers' final opinions.

1.2.3 Activities During the Peer Review Conference Call

After the peer reviewers had ample time to read through the complete set of preliminary written comments, the peer reviewers participated in a 4-hour conference call to discuss the comments and to prepare summary statements of their findings. The conference call occurred on October 4, 2005, and was attended by the six peer reviewers and a technical writer from ERG. The technical discussions were exclusively among the six peer reviewers. One of the peer reviewers (Dr. James Webber) served as the technical chair of the call. During the conference call, the peer reviewers discussed their answers to the nine charge questions.

1.2.4 Activities Following the Peer Review Conference Call

After the peer review conference call, the ERG technical writer who attended the conference call prepared a draft of this peer review summary report, which draws largely from summary statements that the peer reviewers agreed upon during the conference call. ERG distributed this draft to the six peer reviewers and asked them to verify that it accurately reflects the tone and content of the discussions during the conference call. After every peer reviewer confirmed that the draft summary report was a faithful account of the reviewers' discussions, ERG submitted the final peer review summary report (i.e., this report) to EPA.

1.3 Report Organization

The structure of this report reflects the main topic areas covered in the charge to the reviewers: selection of a signature (Section 2, charge questions 1 and 2); the proposed analytical method (Section 3, charge questions 3 and 4); data analysis and interpretation (Section 4, charge questions 5, 6, and 7); and general considerations and alternate approaches (Section 5, charge questions 8 and 9). All references cited in the text are presented in Section 6.

2.0 Selection of a Signature

The first two charge questions addressed the selection of signature constituents of WTC dusts. This section presents the peer reviewers' general summary statements in response to these questions, along with a record of discussion of associated issues raised during the peer review conference call.

2.1 Charge Question 1: Selection Criteria

The first charge question to the peer reviewers was: "The following criteria were established to assist EPA in the selection of appropriate constituents in dust to be characterized as WTC signature constituents:

- a) They are present at levels unique to WTC dust (distinct from urban dust)
- b) They are persistent for many months (not volatile)
- c) They are sufficiently homogeneous in WTC dust
- d) Available analytical methods are able to detect these screening materials with a small sample size, low minimum detection limit, and low interference from other dust components.

Based on information in the supplied documents, and any other knowledge you may bring to the table, are these criteria adequate for establishing a WTC signature marker? If the answer is no, please elaborate?"

Peer Reviewers' Summary Statement:

The peer reviewers found the four criteria that EPA used to select signature constituents to be reasonable. The peer reviewers noted that the fourth criterion is vague (e.g., what precisely is meant by a "small" sample and a "low" detection limit?) and listed two additional criteria that EPA should have considered, as documented below.

Record of Discussion:

Following is a summary of specific issues that the peer reviewers discussed during the peer review conference call when responding to charge question 1:

- *Comments on criterion that signature constituents be "sufficiently homogeneous in WTC dust."* Two peer reviewers noted that existing data show that WTC dust samples are not homogeneous, especially when comparing dust samples collected indoors to those collected outdoors, dust samples collected at street level to those collected at elevation, and dust samples collected near Ground Zero to those collected further downwind. These reviewers acknowledged that this non-homogeneity complicates efforts to select signature constituents. A peer reviewer added that many candidate constituents (e.g., slag wool) might not be homogeneous in terms of their chemical and physical properties — an observation that led to the following two recommendations.

- *Recommended additional criterion that constituents have clearly defined chemical and physical properties.* A peer reviewer questioned whether the proposed analytical method could reliably distinguish slag wool from rock wool based solely on the iron content quantified using SEM (Section 5 of this report discusses this issue further). Based on this concern, the reviewer recommended that an additional criterion for selecting WTC signatures should have been considered: Variations in signature constituents' chemical and physical properties must fall within boundaries that can be clearly defined and reliably differentiated from those of potential interferences at a pre-determined confidence level.
- *Recommended additional criterion that standards be available for gauging measurement accuracy.* Noting that the WTC Dust Screening Study had no provision for assessing whether laboratories are reliably applying analytical methods to measure target constituents at a pre-determined confidence level, a peer reviewer recommended that an additional criterion for selecting WTC signatures should have been: Standards of various concentrations can be created using the pure signature constituents for assessing if analytical results can meet a pre-determined measurement quality objective (e.g., +/- 30% accuracy for SEM analyses).

When discussing the use of standards, one peer reviewer noted that standard samples for slag wool are likely available from the North American Insulation Manufacturers Association (NAIMA). Other peer reviewers supported acquiring standard samples from NAIMA, but provided that the standards have chemical and physical properties that are representative of the slag wool found in WTC dust. One peer reviewer noted a potential challenge associated with acquiring a single representative slag wool standard: because the WTC towers were constructed over a relatively long time frame, slag wool found in the insulation, ceiling tiles, and other materials likely have varying chemical and physical properties. Nonetheless, the peer reviewers unanimously agreed that standards are needed to assess the accuracy of laboratories' measurements.

2.2 Charge Question 2: Slag Wool as a Signature

The second charge question asked the peer reviewers: "Based on information in the supplied documents, and any other knowledge you may bring to the table, do you agree or disagree with the conclusion that slag wool alone meets the criteria as a WTC marker? Please explain your answer."

Peer Reviewers' Summary Statement:

The peer reviewers supported EPA's conclusion that gypsum and elements consistent with concrete do not meet the WTC signature selection criteria. Regarding slag wool, the peer reviewers agreed that, from the data provided, EPA has not made the case that its proposed analytical method can reliably discriminate background dust from dust contaminated with WTC residue. Thus, the proposed method has not demonstrated the utility of slag wool as a successful signature constituent. Critical reservations about selecting slag wool as a signature include the following:

- Based on the distributions of background sampling results and spiked sampling results, several peer reviewers concluded that “false positives” will likely occur, especially when samples are collected near known or unknown sources of slag wool, a common building material.
- Three of the eight original laboratories invited to participate in this study were eliminated from this study. This removal of almost 40% of the selected laboratories brings into question the robustness and reproducibility of the method.
- Some reviewers were concerned about possible degradation (or even disappearance) of slag wool’s characteristics in damp environments.

Record of Discussion:

When answering this charge question during the conference call, the peer reviewers addressed several relevant issues. Some of these issues are described below and others were discussed in the peer reviewers’ responses to other charge questions:

- *Challenges associated with selecting a signature.* When responding to this and other charge questions, the peer reviewers identified several significant challenges associated with selecting a WTC signature. For instance, because some materials used to construct the WTC apparently were not considerably different from those used to construct many other buildings in Lower Manhattan, many candidate constituents are ubiquitous in background dust and therefore not unique to the WTC. Further, a peer reviewer noted efforts to find constituents that are “sufficiently homogeneous” are confounded by the fact that composition of indoor dusts associated with the WTC collapse is expected to vary with numerous factors, including distance from Ground Zero, elevation, and building configuration. Finally, as years continue to pass since the terrorist attack, WTC dust that previously entered indoor environments will continue to be diluted, thus becoming more difficult to identify.
- *Concerns regarding false positives.* Because slag wool is a common building material (i.e., not unique to the WTC), multiple peer reviewers’ preliminary written comments note that slag wool in dusts will likely be found in buildings with ceiling tiles or exposed insulation and at locations near significant building construction, renovation, or demolition activities. Accordingly, this peer reviewer questioned how well slag wool meets the first WTC signature selection criterion for uniqueness.
- *Questions regarding the persistence of slag wool.* Most peer reviewers noted in their preliminary written comments that slag wool meets the second signature selection criterion of being “persistent for many months (not volatile).” Citing studies that have shown slag wool to be somewhat soluble in saline and biological environments (e.g., Bernstein et al. 1996, IARC 2002), one reviewer questioned whether slag wool met this second criterion. This reviewer noted that EPA could conduct solubility tests to verify whether slag wool is indeed sufficiently persistent in damp environments.

After discussing the issues listed above, as well as those listed in the other charge questions, the peer reviewers debated how to phrase their summary statement on the utility of slag wool as a

WTC signature. The peer reviewers eventually agreed that the information they were provided in the WTC Dust Screening Study did not convincingly establish that slag wool met EPA's four criteria for reliable signatures; however, they also concluded that the information provided was insufficient to reject slag wool as a signature. After this discussion, the peer reviewers agreed that the method validation study and the proposed analytical method, in its current form, have not yet demonstrated the utility of slag wool as a successful marker. Refer to Sections 3 and 5 for the peer reviewers' specific recommendations for improving the proposed method.

3.0 The Proposed Analytical Method

Charge questions 3 and 4 asked the peer reviewers to comment on the level of detail in the proposed analytical method and to identify necessary improvements. This section presents the peer reviewers' general summary statements in response to these questions, along with a record of discussion of associated issues raised during the peer review conference call.

3.1 Charge Question 3: Detail of Proposed Analytical Method

The third charge question asked the peer reviewers: "Are the analytical methods written in sufficient detail such that a qualifying laboratory could follow the methodology and obtain valid results without supplemental assistance from EPA or other sources?"

Peer Reviewers' Summary Statement:

The peer reviewers had concerns about laboratories' abilities to implement the proposed analytical method consistently or correctly, given that three out of the eight laboratories selected to participate in the method validation study failed to produce data of acceptable quality. Even after representatives from the eight laboratories "attended a 2-day session during which the method was further developed and discussed" and "all laboratory participants held weekly conference calls as the analytical program was proceeding to discuss general issues with the protocol" (EPA 2005a), data had to be massaged to differentiate WTC spiked samples from background dust. Accordingly, the peer reviewers agreed that a critical element to the success of future sampling is the criteria EPA eventually establishes for identifying and enabling "qualifying laboratories." The peer reviewers recommended that these criteria might include:

- Intensive hands-on training of analysts
- Close monitoring of performance by extensive use of duplicates, spikes, and standards
- Creation of "standards" of slag wool from indoor WTC dust and other sources

Record of Discussion:

The peer reviewers briefly discussed charge question 3 during the conference call. They pointed to the fact that three out of the eight laboratories failed to generate data of acceptable quality as ample evidence that the analytical method is not sufficiently detailed or robust to enable qualifying laboratories "to obtain valid results without supplemental assistance from EPA or

other sources.” One reviewer added that failure of these laboratories to generate quality data calls into question whether EPA met one of its initial study objectives: “...that the analytical method works well enough and is able to be carried out by enough analytical laboratories to...distinguish WTC dust from background dust” (see page 9 of the WTC Dust Screening Study).

3.2 Charge Question 4: Suggested Improvements to the Proposed Analytical Method

The fourth charge question asked the peer reviewers: “If the answer to Question 3 is no, what items or information could be added so that a qualified laboratory could follow this protocol and obtain valid results without the assistance of EPA or other sources?”

Peer Reviewers’ Summary Statement:

The peer reviewers concluded that the method, as written, could not be used to differentiate WTC dusts from other dusts and recommended several modifications to the proposed method that might make it more robust. The most important improvements identified (and agreed to by all six peer reviewers) were:

- Eliminate the drop mount
- The iron content criteria used to differentiate the signature constituent slag wool from the primary interfering constituent rock wool should be based on detailed and comprehensive analysis of respective components in WTC dust and background dust samples, and not on TIMA’s general product information.
- Eliminate sieving and settling from sample preparation because these steps cause the loss of large portions of particles
- Measure fiber dimensions (see Section 5 for further discussion)
- Provide more exacting detail on aliquot withdrawal protocols
- Prepare all samples at a central laboratory for distribution to other laboratories
- Generate spikes and standards using indoor, rather than outdoor, WTC dust
- Require appropriate use of significant digits
- Describe how gravimetric-reduction data will be interpreted

Record of Discussion:

The peer reviewers referred to their preliminary written comments on charge question 4 for the reasons why they recommended the aforementioned improvements. Additional items discussed during the conference call were the need for explicit sample rejection criteria. For instance, a reviewer noted that the analytical method could specify minimum sample sizes, maximum moisture contents, or other criteria that, if met, would require laboratories to reject samples.

Another issue raised during this discussion was whether electron microscopic analyses were even necessary, as several peer reviewers noted that PLM alone might be sufficient in this screening study without the added expense of SEM analyses. The peer reviewers revisited this issue when discussing alternate approaches to the WTC Dust Screening Study (see Section 5).

4.0 Data Analysis and Interpretation

The charge to the reviewers included three questions that ask about the statistical analyses and interpretations of data collected during the method validation study. This section presents the peer reviewers' responses to these charge questions, organized into five topics.

4.1 Charge Question 5: Study Design

The fifth charge question asked the peer reviewers: "The method validation study design entailed spiking characterized background dust with characterized WTC dust at various levels, and then sending 32 blind samples (16 originals and 16 duplicates) of background and spiked dust to each of eight laboratories (five commercial laboratories and three government laboratories). These laboratories characterized the dust using the protocol described above and then sent the results back to EPA. Was this an appropriate design to achieve the goals of this method validation study?"

Peer Reviewers' Summary Statement:

The peer reviewers agreed that the general study design was generally appropriate for method validation. Two shortcomings identified were that laboratories did not analyze standard samples to gauge method accuracy and that the method's equations include unverified assumptions regarding particle losses due to settling and sieving.

Record of Discussion:

During the peer review conference call, additional issues were raised regarding EPA's study design. First, one peer reviewer noted that inconsistent use of sample preparation techniques might have accounted for the considerable inter- and intra-laboratory variability observed during the study. Accordingly, this peer reviewer noted that an improved study design would have involved having a single central laboratory prepare all samples (i.e., generate SEM stubs) and distribute these prepared samples, rather than having the eight laboratories prepare samples on their own. Second, this peer reviewer questioned why a mixture of indoor and outdoor dusts collected following the WTC disaster was used to spike samples, given that results from the WTC Dust Screening Study are to be used to evaluate dusts in indoor settings.

Another concern about the method study design was the assumption that iron content, as measured by SEM, can be used to differentiate slag wool from rock wool. One reviewer noted that this assumption was apparently based entirely on data provided the Thermal Insulation

Management Association (TIMA).¹ However, this peer reviewer presented data from other sources (see page 10 of Shu-Chun Su's preliminary written comments in Appendix B) suggesting that differences in iron content might be insufficient for distinguishing these materials. This peer reviewer noted that an improved study design would have thoroughly characterized the compositional variation of slag wool and rock wool in WTC dust during the initial stages of method development, rather than relying on published values that might not be adequately representative of the material found in WTC dust.

When discussing the unverified assumptions in the method's equations, a peer reviewer explained that, when the participating laboratories sieved the original 32 samples through a 100-mesh screen, the partitioning ratio between coarse and fine slag wool was not shown to be constant. For instance, the sieving step could have removed 10% of the slag wool in some samples, while this same step could have removed 20% of the slag wool in other samples. Similarly, this peer reviewer questioned the impact of the "one-minute waiting period" before aliquots were extracted from the diluted suspension, given that inconsistent application of this procedure could have resulted in variable fractions of coarser particles precipitating to the bottom of the beaker. The peer reviewers' recommendations (see Section 3.2) to eliminate sieving and settling from sample preparation would address these concerns.

4.2 Charge Question 6: Statistical Analysis of Data

The sixth charge question asked the peer reviewers: "Did EPA and Versar adequately evaluate and interpret the results from the eight laboratories, as documented by the supplied documents on NYC dust analyses?"

Peer Reviewers' Summary Statement:

The peer reviewers were skeptical that EPA's evaluation and interpretation of the study data were performed fairly. Peer reviewers pointed to several non-standard steps taken to enhance the study's ability to distinguish WTC dust from background dust. These steps could be interpreted as attempts to prove the method's success rather than to objectively evaluate its real-world potential for fingerprinting WTC dust. The peer reviewers' two most notable concerns were:

- The reported difference in slag wool for impacted and non-impacted locations was based on statistical analyses that excluded certain sampling results from the background locations, but not from the spiked samples. The background results and spiked sample results were statistically indistinguishable when the entire data set was considered.
- The study authors disqualified three out of the eight originally selected laboratories that participated in the WTC Dust Screening Study. There was no follow-up investigation to determine why these three selected laboratories, presumably following the proposed method, failed to differentiate background dusts from WTC dusts.

¹ When reviewing a draft of this report, another peer reviewer noted that he thought USGS researchers noted differences in iron content between slag wool and rock wool based on analyses of WTC dust samples, not based on the TIMA data.

- After the peer review conference call, one peer reviewer submitted this additional summary statement: Because the equation used to calculate the final analytical results (the number of slag wool fibers per gram of dust) does not account for non-uniform depletion of slag wool fibers due to sieving and due to coarse particle precipitation during sample preparation, the equation underestimates the number of slag wool fibers per gram of dust for all spiking and background samples analyzed by all eight participating laboratories. Had the underestimations been corrected for in the analytical results, the spiking sample results of the three disqualified laboratories could have been closer to the expected values than the other five laboratories. Therefore, their results might have been wrongly disqualified. [Note: This comment was submitted shortly before the final report was drafted and reflects the opinion of one peer reviewer. The other peer reviewers did not comment on this matter.]

Record of Discussion:

The peer reviewers discussed many issues raised in their preliminary written comments when responding to this charge question. Issues raised included, but were not limited to, the following:

- *Exclusion of data from three laboratories.* Nearly every peer reviewer was troubled by the fact that EPA's conclusion regarding the use of slag wool as a signature constituent was only reached after data from three laboratories were rejected. Most peer reviewers found the decision to exclude these laboratories' data to be unjustified and genuine cause for concern. Given that the stated purpose of the WTC Dust Screening Study was to validate an analytical method, peer reviewers noted that all analytical data should have been considered unless EPA had evidence of laboratories gross failure to perform.
- *Significance of low fiber counts and large data extrapolations.* When applying the proposed analytical method, laboratories first counted the number of slag wool fibers on an SEM stub. These raw fiber counts were relatively low (i.e., generally less than 20 fibers per SEM stub). From these raw data, laboratories computed the numbers of fibers per gram of dust using large extrapolation factors. Thus, even though data throughout the main body of the report for the WTC Dust Screening Study are presented in thousands of fibers per gram of dust (using many significant figures), the actual observations made in the analytical laboratory are based on relatively small fiber counts. This was of particular concern to reviewers because measurement error is known to increase as fiber count decreases. To illustrate this concern, one peer reviewer stepped through back-of-the-envelope calculations that, given the range of fibers counted in individual samples, the measurement quality objective for accuracy (+/-30%) stated in the quality assurance project plan likely could not be met (see Dan Crane's preliminary written comments in Appendix B).
- *Comparison of data across laboratories.* After examining the raw fiber counts generated by the eight analytical laboratories (see pages 58 and 59 in the WTC Dust Screening Study), one peer reviewer noted that the summary report failed to examine or even acknowledge one of the more notable trends among the data: Laboratory D consistently reported considerably higher fiber counts than did any other laboratory. This observation

raised further questions about laboratories' ability to implement the proposed method consistently.

- *Interpretation of background samples.* In addition to their concerns regarding excluding data from selected laboratories, the peer reviewers were troubled by the fact that EPA also excluded several background samples from its statistical analyses. One peer reviewer noted that, when all background samples are considered, levels of slag wool in background dust are statistically indistinguishable from levels in the WTC dust from 4 Albany Street. Peer reviewers were further concerned that no detailed explanations were provided to adequately justify the decision to exclude background samples collected in three locations (New Jersey, Long Island, and Research Triangle Park).
- *Validity of underlying data.* One peer reviewer noted that an inherent assumption in the statistical analyses is that the underlying data are valid. Given concerns raised elsewhere about the analytical method (e.g., the potential inability to distinguish slag wool from rock wool, the unverified assumptions regarding particle losses during sieving and settling), this peer reviewer was not convinced that the original data were of sufficient quality to warrant such detailed statistical analyses.

4.3 Charge Question 7: Data Interpretation

The seventh charge question asked the peer reviewers: "EPA has observed differences in slag wool concentration which discriminate between background dust and dust contaminated with WTC residue. Do you agree that the data and analysis presented in the documents support this observation? Please explain your answer."

Peer Reviewers' Summary Statement:

As indicated in the previous summary statements, the peer reviewers generally do not believe that EPA made the case from the study data that the proposed method reliably discriminate background dust from dust contaminated with WTC residue.

Record of Discussion:

Issues discussed during the peer review conference call related to charge question 7 included the following:

- *Concerns about inadequate statistical power.* One of the main conclusions of the WTC Dust Screening Study is that "...slag wool measurements appear to be sensitive enough to distinguish WTC dust (defined as 4 Albany) spiked at the 10% level from background dust" (EPA 2005a). However, when examining the analytical data (see page 58 and 59 of the WTC Dust Screening Study), peer reviewers noted that this finding was reached by comparing two samples spiked at the 10% level using dusts from 4 Albany Street and the background samples that EPA included in its evaluation. With this limited number of samples and the broad error ranges associated with low fiber counts, a peer reviewer questioned whether the number of samples considered in the method validation study was sufficient to reliably discriminate the background dusts from WTC dusts.

- *Comments on required sensitivity.* When discussing whether the proposed method allows for discrimination between background dusts and WTC dusts, one peer reviewer referred to the following quote in the WTC Dust Screening Study: "...at the 10% spike level, the slag wool concentration typically exceeds one standard deviation, but never exceeds two standard deviations, above the background level" (EPA 2005a). This peer reviewer disagreed with EPA's conclusion that this difference between 10% WTC dust and background dust was "sensitive enough" to qualify slag wool as a signature, though it was noted that the signature selection criteria did not establish a minimum concentration difference (or sensitivity) between background dust and WTC dust that must be achieved to designate a signature constituent.
- *Interpretation of data in Figure 2 of the review document.* When responding to this charge question, one peer reviewer noted that the USGS data plotted in Figure 2 of the WTC Dust Screening Study appear to show great promise for slag wool as a signature constituent, and this peer reviewer questioned why data generated during the method validation study were not similarly convincing. Other peer reviewers provided several explanations. First, these reviewers advised against focusing too much on trends in Figure 2, because these trends are based largely on outdoor dust samples collected shortly after the WTC collapse; they noted that the data shown in Figure 3 are more relevant, given that they represent indoor dusts collected much more recently (i.e., September 2004). Focusing on the data in Figure 3, these reviewers said the concentration differences between the three spiking levels are not nearly as pronounced. Further, noting that the concentrations in Figure 3 are based on only three samples and on large extrapolations from the raw fiber count data, a peer reviewer cautioned against drawing strong conclusions from the trend line.

5.0 General Considerations and Alternate Approaches

The final two charge questions were open-ended questions asking peer reviewers to discuss general considerations and alternate approaches for identifying signature constituents in WTC dusts. This section summarizes how the peer reviewers responded to these questions during the peer review conference call.

5.1 Charge Question 8: Suggested Improvements

The eighth charge question asked the peer reviewers: "Is there any other way in which the proposed signature marker(s) can be used to determine the extent to which WTC dust may have penetrated and remains in indoor environments?"

Peer Reviewers' Summary Statement:

Fiber dimensions and chemical composition of slag wool should be further investigated. Fibers from the WTC collapse would likely include a greater proportion of smaller fibers given the impact energy of the building collapse, and indoor fibers from the WTC collapse are expected to be smaller due to the removal of large fibers by passage from outdoor to indoor environments.

The chemical composition of slag wool in WTC dusts has apparently not been adequately characterized.

Record of Discussion:

Specific issues discussed during the peer review conference call related to charge question 8 include the following:

- *Relevance of fiber dimension.* Peer reviewers acknowledged throughout the conference call that dust samples from non-impacted locations would undoubtedly contain slag wool, given the widespread use of this material in building construction. Accordingly, relying entirely on fiber *counts* for identifying a WTC signature would ultimately result in “false positives,” especially for indoor environments with ceiling tiles and locations near recent building construction, renovation, or demolition activities.

However, some peer reviewers suspected that *dimensions* of slag wool in WTC dust might be unique when compared to *dimensions* of slag wool in background dust. Two observations were noted in support of this statement: (1) the tremendous impact energy associated with the WTC collapse presumably led to smaller fiber dimensions in the WTC dust cloud as compared to fiber dimensions observed in the original building materials; and (2) larger fibers (as compared to smaller fibers) were more likely to be removed from the WTC dust cloud due to gravitational settling and “filtering” as these dusts passed from outdoor to indoor environments. Given these observations, the peer reviewers recommended that EPA further investigate whether considering fiber dimensions of slag wool might enhance the proposed signature. They noted further that EPA could begin to address this recommendation by comparing fiber dimensions observed in two sets of existing samples: the USGS WTC slag wool (which is largely composed of outdoor dusts) and dusts from 4 Albany Street (which is entirely composed of indoor dusts).

- *Chemical composition of slag wool in WTC dust.* A peer reviewer reiterated an earlier comment that EPA’s method development work relied entirely upon composition data (most notably, iron content) reported by TIMA to distinguish slag wool from rock wool, even though these published data might not be representative of the chemical composition of slag wool found in WTC dusts. This peer reviewer advocated a more thorough characterization of the chemical, physical, and optical properties of all candidate signature constituents prior to selecting a final signature.
- *Further evaluation of gypsum and anhydrite.* While the peer reviewers agreed that gypsum and elements consistent with concrete alone did not meet EPA’s signature selection criteria (see Section 2.2), one peer reviewer noted that anhydrite might hold promise as a potential WTC signature. This suggestion was based on the fact that the heat of combustion when jet fuel burned in the WTC towers and during subsequent fires in the debris pile might have created anomalous amounts of anhydrite. Accordingly, this peer reviewer wondered if anhydrite, or the relative amounts of gypsum and anhydrite, could be used as a WTC signature. The other peer reviewers did not support or reject this suggestion, but one reviewer clarified that the overwhelming majority of dusts in indoor

environments apparently originated from the dust cloud released during the physical collapse of the WTC towers (and not from the combustion by-products from jet fuel or the debris pile). Another issue discussed, but not resolved, was whether gypsum would be sufficiently persistent for use as a signature, particularly in damp environments.

The peer reviewers raised additional issues when responding to this charge question, such as recommendations that EPA evaluate the utility of multi-component signatures and that EPA reconsider the utility of PLM in its analytical method. However, the reviewers discussed these topics in greater detail when responding to the final charge question (see Section 5.2).

5.2 Charge Question 9: Other Comments

The final charge question asked the peer reviewers: “Are there any additional comments or concerns about this study that have not been addressed by the other questions?”

Peer Reviewers’ Summary Statement:

Most of the peer reviewers’ comments on this charge question fell into two general categories: modifying the proposed analytical method or investigating the utility of new approaches. The specific comments are documented in the record of discussion, below.

Record of Discussion:

During the conference call, the peer reviewers discussed their responses to the final charge question. A summary of those responses follows. References to individual peer reviewers’ preliminary written comments are included for further information on the specific recommendations.

(1) Recommendations for modifying or enhancing the proposed approach

- Given the shortcomings of slag wool alone as a signature (e.g., the likely false positives), some reviewers recommended that EPA consider implementing a tiered sampling approach that would use slag wool as a screening marker for the potential presence of WTC dust followed by TEM analysis of chrysotile asbestos as a confirmatory marker. Most reports indicate chrysotile’s ubiquity in WTC dust, that the WTC collapse and combustion produced unique chemical and morphological properties in individual fibers, that standardized and robust collection and TEM analytical methods exist, and that there are dozens of certified TEM laboratories with extensive chrysotile analytical capabilities.

This suggestion generated considerable discussion. On the one hand, several peer reviewers supported this recommendation after reviewing the supporting arguments presented in the preliminary written comments (see Jim Webber’s response to charge question 9 and Frank Ehrenfeld’s response to charge question 8, both in Appendix B). On the other hand, some peer reviewers noted that the utility of asbestos as a signature (based on indoor air samples) has already been investigated, but found to be of limited utility especially when compared to the utility of various manmade vitreous fibers. In response, one peer reviewer noted that a 2002 study (Chatfield and Kominsky 2002) documented relatively high levels of chrysotile asbestos in indoor environments near Ground Zero

using various bulk and surface dust sampling methods. Based on these and other data, this reviewer said that the potential utility of chrysotile asbestos as a confirmatory signature warrants further consideration.

- Throughout the conference call, multiple peer reviewers recommended that EPA reconsider the utility of PLM as a supplementary analysis to, or possibly in replacement of, SEM. The rationale behind this recommendation is stated earlier in this report and also in reviewers' preliminary written comments in Appendix B (see Dan Crane's response to charge question 8 and Mickey Gunter's and Shu-Chun Su's responses to charge question 9).
- If EPA proceeds with slag wool as its signature, a peer reviewer recommended that positive detections for slag wool should trigger physical site surveys for evidence of other slag wool sources. These other sources include presence of ceiling tiles or exposed insulation and proximity to large construction, renovation, or demolition sites.
- As Section 5.1 of this report notes, several peer reviewers recommended that EPA investigate whether fiber dimension along with fiber count would allow a more definitive differentiation between WTC dusts and background dusts.
- Building upon comments raised earlier in the conference call, some peer reviewers recommended that EPA improve methods to identify critical features of slag wool specific to WTC dusts. Noting that iron content and its resolution by SEM/EDS is currently used to differentiate slag wool from rock wool, the reviewers recommended EPA use field studies of WTC dusts to thoroughly characterize the chemical and physical properties of slag wool and other manmade vitreous fibers found in this matrix, rather than relying on data published in the TIMA atlas. Another suggestion was for EPA to investigate the utility of the Emmons Double Variation Method when characterizing slag wool in the dust samples (in Appendix B, see the first issue raised by Frank Ehrenfeld in response to charge question 9).
- One peer reviewer noted that, while the WTC Dust Screening Study acknowledges that use of slag wool as a signature will likely result in some "false positives," the report fails to evaluate the likelihood of observing "false negatives." Because the sampling protocol was not developed to evaluate minimum detection limits, the reviewer concluded that the study did not establish a lower bound of contamination that the method can reliably find. Should EPA adopt the proposed method, further discussion and evaluation of the detection limit was recommended.
- A peer reviewer recommended that EPA consider revising its method to quantify the *mass* of slag wool in samples (which would account for fiber dimension), instead of quantifying the *counts* of slag wool in these samples (see Ernest McConnell's response to charge question 9 in Appendix B).
- A peer reviewer agreed with the WTC Dust Screening Study's finding that vacuuming dust from a square-meter area renders the space unusable for replicate samples. However, this reviewer added that the study did not quantify sampling efficiency is expected to

vary with different surface types. Questioning the assumption made during method development that canister vacuums are 100% efficient in collecting dusts, this reviewer recommended further evaluation to judge and verify the proposed field collection techniques.

(2) Recommendations for investigating new approaches

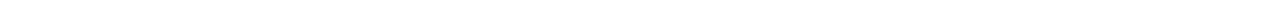
The peer reviewers' responses to charge question 9 also included recommendations that essentially involved considering new approaches to identifying WTC signatures, as opposed to mere modifications to EPA's proposed approach. The following recommendations are discussed in greater detail primarily in one peer reviewer's preliminary written comments (see Mickey Gunter's responses to charge question 9 in Appendix B):

- Add academic scientists to the study design team to take advantage of their in-depth expertise, flexibility to pursue research, and immunity from political fallout.
- Explore use of multiple analytical methods (especially "bulk" methods) with the potential for creating a multi-component signature. Specific analytical methods mentioned included PLM, SEM, inductively coupled plasma (ICP) spectroscopy, X-ray fluorescence (XRF) spectroscopy, and X-ray diffraction (XRD) techniques. One peer reviewer wondered if very simple markers, such as pH, might hold promise when searching for multi-component signatures.
- Compare the chemical composition of particulate filters (both PM₁₀ and PM_{2.5}) collected at ambient air monitoring stations nearest Ground Zero before the WTC collapse to the chemical composition of corresponding filters collected after the WTC collapse.
- Recognizing that improved characterization of WTC building materials would likely help EPA identify the most appropriate signatures, one peer reviewer recommended that EPA consider researching WTC building construction documentation or analyzing samples of debris that has been disposed of at Fresh Kills Landfill. Other reviewers, however, were not convinced of the utility of testing materials in the landfill, given the level of effort required to obtain a representative sample and the fact that even a statistically-based sample of debris in the landfill might not be representative of the material in the WTC dust cloud that infiltrated into indoor environments.
- Throughout their discussions, peer reviewers wondered if all available sampling results were considered in the selection of WTC signatures. Noting that a large number of samples have been collected since 2001, both by public and private parties, one peer reviewer suggested that EPA have a contractor attempt to acquire as much existing data as possible and then conduct further statistical analyses of the combined data sets. These analyses, he noted, would have to account for numerous factors, such as the sampling method used, the analytical method used, whether samples were collected before or after cleaning, and so on. This peer reviewer noted that multivariate statistical analyses of a broader set of data might reveal viable multi-component signatures that are not readily discernable from the smaller set of samples that EPA considered in the WTC Dust Screening Study.

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Appendix A. List of Peer Reviewers





United States
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Appendix B. Charge to Reviewers and Preliminary Written Comments

Note: Each peer reviewer prepared the preliminary written comments in this appendix prior to the peer review conference call. These written comments are preliminary and were used to help identify key issues for discussion. After reviewing the entire set of preliminary written comments, some reviewers' opinions on specific matters might have changed. Accordingly, these comments should be viewed as preliminary, while the content of the main body of this report reflects the peer reviewers' final findings.

CHARGE TO REVIEWERS

CHARGE QUESTIONS (August 17, 2005)
World Trade Center Signature Study Peer Review

BACKGROUND

In the days following the terrorist attack on New York City's World Trade Center (WTC) towers, EPA, other federal agencies, and New York City and New York State public health and environmental authorities initiated numerous air monitoring activities to better understand the ongoing impact of emissions to the outside environment from that disaster. In 2002, EPA's Region 2 turned its attention to the indoor environment, providing a volunteer "clean and test" or "test only" program for residents and homeowners who wished to have their apartments cleaned and/or tested. Asbestos was selected as a surrogate for the presence of WTC-related contamination for this program.

In March 2004, EPA convened the WTC Expert Technical Review Panel to interact with the Agency and the public on plans to monitor for the presence of any remaining WTC dust in indoor environments near Ground Zero. For more information on the WTC Expert Panel, see <http://www.epa.gov/wtc/panel>. Approximately 750 units in an area extending north to Houston Street in lower Manhattan and across the East River into a portion of Brooklyn were proposed to be sampled for contaminants of potential concern (COPC), as well as for specific constituents that can be used as markers to identify residual contamination by dust from the collapse of the WTC. These "WTC dust signature" constituents are the cornerstone of the sampling program as they will provide the basis for estimating the geographic extent of the remaining residue in dust of the WTC towers collapse. Using the results of dust sampling for the WTC building collapse signature, EPA will decide whether indoor cleanup or other activities are warranted at this time.

Based on the previous work of the United States Geological Survey (USGS) and others, EPA identified three components of the dust generated by the WTC towers collapse that could be used to screen sampled dust for the presence of WTC dust. These markers, or signature components, were mineral slag wool, gypsum and elements of concrete. EPA developed the following working hypothesis for the signature: "A dust sample that contains WTC dust will have slag wool and elements of concrete and gypsum present in 'significant quantities' when compared to typical indoor urban dust." Experts from the USGS, EPA's Office of Research and Development, EPA's National Enforcement Investigations Center, and the commercial testing laboratory community worked together to develop an analytical method to quantify the concentration of these three markers in indoor dust.

Between September 2004 and April 2005, numerous samples were taken in impacted buildings near Ground Zero and at background locations. These samples have been analyzed for these three markers to determine whether or not they validly constitute a WTC signature, as suggested by earlier USGS efforts. Also, they were used in a method validation study whose primary purposes were twofold: 1) to evaluate the analytical method with regard to method variability (as measured by both inter- and intra-laboratory variability), and cost and expediency issues, and 2) to assist in the determination of the lower limits of concentrations of the markers that could be reliably measured, and that could be reliably distinguished from background concentrations. This method validation study involved five contract laboratories and three

federal government laboratories. This study entailed spiking known background dust with varying concentrations of known WTC collapse dust, and then having the laboratories perform a blind analysis on both the spiked samples and background samples.

The charge questions focus on the following: the basis for identifying signature components; the validity of the hypothesis; whether the existence of a signature has been adequately demonstrated; and the work done to develop, validate and apply an analytical method for the three signature components.

The following documents are the primary ones being reviewed: 1) the analytical protocol used by the eight laboratories and 2) EPA and Versar (EPA contractor) reports documenting the validation study including the background and hypothesis, analyses of samples taken to verify the hypothesis, and the results and interpretation of the method validation study. Several background documents will also be available to the reviewers. These include the draft final sampling plan to be used to evaluate the presence and levels of contaminants of potential concern of any remaining WTC dust in indoor environments near Ground Zero (this plan includes an overview of the Signature Study); two USGS reports on the signature development; and other pertinent reports that provide additional background on the characterization of WTC dust. The reviewers are not being asked to provide comments on these background documents. If the reviewers believe they need any additional documents or clarification of information provided in the documentation provided, they will be supplied through the peer review contractor.

CHARGE QUESTIONS

Basis for Development of a Signature:

Q1) The following criteria were established to assist EPA in the selection of appropriate constituents in dust to be characterized as WTC signature constituents:

- a) They are present at levels unique to WTC dust (distinct from urban dust);
- b) They are persistent for many months (not volatile);
- c) They are sufficiently homogeneous in WTC dust; and
- d) Available analytical methods are able to detect these screening materials with a small sample size, low minimum detection limit, and low interference from other dust components.

Based on information in the supplied documents, and any other knowledge you may bring to the table, are these criteria adequate for establishing a WTC signature marker? If the answer is no, please elaborate.

Documentation of the Existence of a Signature:

Q2) Based on information in the supplied documents, and any other knowledge you may bring to the table, do you agree or disagree with the conclusion that slag wool alone meets the criteria as a WTC marker? Please explain your answer.

Analytical Method Development:

Q3) Are the analytical methods written in sufficient detail such that a qualifying laboratory could follow the methodology and obtain valid results without supplemental assistance from EPA or other sources?

Q4) If the answer to Question 3 is no, what items or information could be added so that a qualified laboratory could follow this protocol and obtain valid results without the assistance of the EPA or other sources?

Method Validation Study:

Q5) The method validation study design entailed spiking characterized background dust with characterized WTC dust at various levels, and then sending 32 blind samples (16 originals and 16 duplicates) of background and spiked dust to each of eight laboratories (five commercial laboratories and three government laboratories). These laboratories characterized the dust using the protocol described above and then sent the results back to EPA. Was this an appropriate design to achieve the goals of this method validation study?

Q6) Did EPA and Versar adequately evaluate and interpret the results from the eight laboratories, as documented by the supplied documents on NYC dust analyses?

Dust collected from currently occupied buildings is expected to have lower levels of the key WTC constituents as compared to dust sampled near September 11, 2001 in time or sampled more recently but in uninhabited heavily impacted buildings. EPA will use the results of this method validation study to determine the final distinguishing concentrations for the WTC marker(s). If currently sampled dust has this marker(s) at or above such a distinguishing concentration, EPA would consider the sampled dust to "contain residues of WTC dust" for purposes of estimating the geographic extent of WTC impacts and making cleanup decisions. The key requirement for a distinguishing concentration is that it be adequate to distinguish between dusts that do not contain WTC residues from those that do with a reasonably low false positive error rate.

Q7) EPA has observed differences in slag wool concentration which discriminate between background dust and dust contaminated with WTC residue. Do you agree that the data and analysis presented in the documents support this observation? Please explain your answer.

Q8) Is there any other way in which the proposed signature marker(s) can be used to determine the extent to which WTC dust may have penetrated and remains in indoor environments?

Q9) Are there any additional comments or concerns about this study that have not been addressed by these questions?

**PRELIMINARY COMMENTS
FROM
DANIEL T. CRANE, M.E.**

CHARGE QUESTIONS (August 17, 2005)
World Trade Center Signature Study Peer Review

Preface to Response to Charge Questions:

In the days following the terrorist attack on New York City's World Trade Center (WTC) towers, EPA, other federal agencies, and New York City and New York State public health and environmental authorities initiated numerous air monitoring activities to better understand the ongoing impact of emissions to the outside environment from that disaster. In 2002, EPA's Region 2 turned its attention to the indoor environment, providing a volunteer "clean and test" or "test only" program for residents and homeowners who wished to have their apartments cleaned and/or tested. Asbestos was selected as a surrogate for the presence of WTC-related contamination for this program.

In March 2004, EPA convened the WTC Expert Technical Review Panel to interact with the Agency and the public on plans to monitor for the presence of any remaining WTC dust in indoor environments near Ground Zero. For more information on the WTC Expert Panel, see <http://www.epa.gov/wtc/panel>. Approximately 750 units in an area extending north to Houston Street in lower Manhattan and across the East River into a portion of Brooklyn were proposed to be sampled for contaminants of potential concern (COPC), as well as for specific constituents that can be used as markers to identify residual contamination by dust from the collapse of the WTC. These "WTC dust signature" constituents are the cornerstone of the sampling program as they will provide the basis for estimating the geographic extent of the remaining residue in dust of the WTC towers collapse. Using the results of dust sampling for the WTC building collapse signature, EPA will decide whether indoor cleanup or other activities are warranted at this time.

Based on the previous work of the United States Geological Survey (USGS) and others, EPA identified three components of the dust generated by the WTC towers collapse that could be used to screen sampled dust for the presence of WTC dust. These markers, or signature components, were mineral slag wool, gypsum and elements of concrete. EPA developed the following working hypothesis for the signature: "A dust sample that contains WTC dust will have slag wool and elements of concrete and gypsum present in 'significant quantities' when compared to typical indoor urban dust." Experts from the USGS, EPA's Office of Research and Development, EPA's National Enforcement Investigations Center, and the commercial testing laboratory community worked together to develop an analytical method to quantify the concentration of these three markers in indoor dust.

Between September 2004 and April 2005, numerous samples were taken in impacted buildings near Ground Zero and at background locations. These samples have been analyzed for these three markers to determine whether or not they validly constitute a WTC signature, as suggested by earlier USGS efforts. Also, they were used in a method validation study whose primary purposes were twofold: 1) to evaluate the analytical method with regard to method variability (as measured by both inter- and intra-laboratory variability), and cost and expediency issues, and 2) to assist in the determination of the

lower limits of concentrations of the markers that could be reliably measured, and that could be reliably distinguished from background concentrations. This method validation study involved five contract laboratories and three federal government laboratories. This study entailed spiking known background dust with varying concentrations of known WTC collapse dust, and then having the laboratories perform a blind analysis on both the spiked samples and background samples.

The charge questions focus on the following: the basis for identifying signature components; the validity of the hypothesis; whether the existence of a signature has been adequately demonstrated; and the work done to develop, validate and apply an analytical method for the three signature components.

The following documents are the primary ones being reviewed: 1) the analytical protocol used by the eight laboratories and 2) EPA and Versar (EPA contractor) reports documenting the validation study including the background and hypothesis, analyses of samples taken to verify the hypothesis, and the results and interpretation of the method validation study.

The answers to the charge questions below reflect my experience with industrial hygiene laboratories and the way in which they analyze particulates by microscopy. They further reflect my knowledge and experience with the issues surrounding the measurement of the COPCs.

The EPA requested that the Light Microscopy portion of the study not be reviewed as it was applied in this protocol.

CHARGE QUESTIONS

Basis for Development of a Signature:

Q1) The following criteria were established to assist EPA in the selection of appropriate constituents in dust to be characterized as WTC signature constituents:

- a) They are present at levels unique to WTC dust (distinct from urban dust);

Answer to Q1 a):

With respect to constituents of concrete dust, and gypsum/anhydrite as markers, I agree with the study that while comprising some of the most common constituents of the dust, these components are not unique in any way in their physical properties as components of the WTC debris. In addition, these components are ubiquitous in urban settings. While there must be an elevated level of these components in the shadow of the dust plume, their inspecificity of composition necessarily excludes them from the marker suite because it is expected that they will lead to an unacceptably high number of false positive results.

Considering slag wool as a marker, the evaluation study provides an answer, albeit not in a straightforward manner. The collected background samples

showed that slag wool was not broadly prevalent away from ground zero, or the drift zone of the debris cloud. The exceptions to this were the samples excluded on the basis of the presence of ceiling tiles or other building material containing slag wool in the sampled location. The data from this study indicates that slag wool could be used as a marker, in the impacted area. There is no special attribute of the slag wool itself to tie it to the World Trade Centers. It was and is a common component in the insulation and other building materials used in the WTC as well as many other buildings in the area. However, the number of fibers in the collected dust reflect that in the considered region, with the exception of buildings with an internal source, the slag wool fibers seen reflect mostly fibers generated in the collapse of the World Trade Center buildings.

Because of the known positive interference, the use of this marker demands a site survey for slag wool containing materials in any sampled building where a positive survey result is found. Any positive results in the presence of an internal source must render as suspect the decision that WTC signature dust is present.

b) They are persistent for many months (not volatile);

Answer to Q1 b):

This report demonstrated that the slag wool component has been persistent in the sampled locations since the collapse of the World Trade Centers. I believe that the slag wool component, due to its physical nature is sufficiently persistent and pervasive in the dust to serve as a marker.

With respect to the persistence of concrete and gypsum/anhydrite, it appears that these components were indeed found. This demonstrates that they are persistent. In the case of concrete dust, it is to be expected that it is persistent over the relatively short time since the event. In the case of gypsum/anhydrite, it is known that these materials react with water and this may alter the persistence of the component dependent upon the exposure history of the dust.

Note however, that any problems referent to concrete and gypsum/anhydrite are moot if they are discounted as potential marker components.

c) They are sufficiently homogeneous in WTC dust.

Answer to Q1 c):

Virtually all samples in the dispersion zone contained the three marker components. However, presence does not imply homogeneity at any particular sampling size. At the individual analytical sample size, the heterogeneity of the collected material virtually guarantees that the samples are inhomogeneous. This does not disqualify the material as a marker material. It does limit the precision with which it can be quantified, and subsequently re-sampled after cleanup to certify the cleanup.

d) Available analytical methods are able to detect these screening materials with a small sample size, low minimum detection limit, and low interference from other dust components.

Answer to Q1 d):

The analytical technique has a putative detection limit of 1 target particle counted in the SEM analyzed area. This has not been reduced to a protocol detection limit in terms of fibers or particles per gram of collected material. The recovery efficiency of fibers and other particles from the sample surfaces is suspected to be variable and unknown, but assumed by the framers of the protocol to be sufficiently high to make a clean-up decision. This is based on their statement in several places that a second sample would not be expected to provide an equivalent sample if taken from the same area. This seems to be a reasonable assumption. However, no data is given in the study that documents the rate of collection in resampling the same areas.

The particle size detection limit is set to 0.5 μm . At 500 X magnification this is approximately 0.25 mm on the SEM screen. That approximates the viewing pitch of a color screen, or near single pixel resolution. This seems too small to be of practical use, although it will certainly pick up the very smallest particles visible larger than that.

With reference to interferences, using the USGS catalog of WTC particles, the analyst should be able to sort out most, if not all of the particles of interest. Other MMVF present in the samples are sufficiently different in chemistry such as to be distinguishable by competent microscopists.

Based on information in the supplied documents, and any other knowledge you may bring to the table, are these criteria adequate for establishing a WTC signature marker? If the answer is no, please elaborate.

General answer to Q1:

Having investigated a variety of materials for the purpose of establishing the original COPCs and knowing that the three suggested materials are the most commonly found materials in the WTC debris, it is apparent that they provide the highest potential to be found in locations impacted by WTC dust. They may provide a surrogate for measurement for the COPCs which may be present at levels too low to be reliably measured.

Concrete dust and gypsum/anhydrite dust are too widespread in areas unaffected by WTC and are at levels too unpredictable within the impacted area to be of any real value as a tracer or surrogate for dispersion of WTC dust.

In the study, dispersion of slag wool seemed to be confined to the impacted area. Where it can be shown that the slag wool did not originate from internal sources

in a sampled building, It would appear that slag wool represents the best candidate as a surrogate for the COPCs.

Slag wool alone of the three has the potential to be used as a marker. However, it is apparent that any protocol which hopes to adequately determine WTC impact on a particular building must be accompanied by a building survey as an adjunct for any positive result to determine whether or not there is an internal source which can account for the positive sample. In addition, if a history of open-air demolition is available, it should be added to the record as a potential source for the target materials. This is especially true if the demolition was by implosion or other very dusty mode.

It is also apparent that the actual detection limit for the protocol is unknown. If it is assumed that the vacuum sampling technique has an adequate sampling efficiency such that a decision can be made, then slag wool represents the best surrogate for WTC dust sampling. However, the high uncertainty associated with the analysis makes resampling problematic in that a second sample after clean-up may not adequately represent the level of remaining contaminant.

Documentation of the Existence of a Signature:

Q2) Based on information in the supplied documents, and any other knowledge you may bring to the table, do you agree or disagree with the conclusion that slag wool alone meets the criteria as a WTC marker? Please explain your answer.

Answer to Q2:

Slag wool is not specific to the WTC collapse. It is essential to remember that while it is a significant source in the impacted area, there may be other significant sources which may confound the proposed surveys. A history of open-air demolitions, if any should be sought. Also, it is well to remember that slag wool is being used as a surrogate for the detection of other COPCs which may be at levels below current technology to measure. This is because of the ubiquity of slag wool in WTC dust. That same ubiquity must be respected as a confounder in the interpretation of any results.

Slag wool alone of the three has the potential to be used as a marker. However, it is apparent that any protocol which hopes to adequately determine WTC impact on a particular building must be accompanied by a building survey as an adjunct for any positive result to determine whether or not there is an internal source which can account for the positive sample.

It is also apparent that the actual detection limit for the protocol is unknown. If it is assumed that the vacuum sampling technique has an adequate sampling efficiency such that a decision can be made, then slag wool represents the best surrogate for WTC dust sampling. However, the high uncertainty associated with

the analysis makes resampling problematic in that a second sample after clean-up may not adequately represent the level of remaining contaminant.

Analytical Method Development:

Q3) Are the analytical methods written in sufficient detail such that a qualifying laboratory could follow the methodology and obtain valid results without supplemental assistance from EPA or other sources?

Answer to Q3: No

The study was begun with a joint meeting to correlate the analyses of the laboratories. Even in the face of this training session, three of the laboratories were adjudged unable to adequately perform the method as written. It is unclear whether the attendees to the sessions were the actual analysts or other laboratory representatives. With the level of technique required to properly apply the protocol, it is essential that more intensive procedural correlation take place.

Q4) If the answer to Question 3 is no, what items or information could be added so that a qualified laboratory could follow this protocol and obtain valid results without the assistance of the EPA or other sources?

Answer to Q4:

This analytical protocol is heavily reliant on technique. It is essential that correlation of effort be accomplished with the actual analysts who will be performing the tests. This collaboration should consist of hands-on mentored training with sufficient trials such that the analysts are demonstrably proficient.

In addition, the protocol provides for two different mounting techniques for SEM analysis: liquid drop and filtration. These two mounting methods should not be expected to provide equivalent mounts. For a random field-choice counting method, the particulate must be randomly distributed on the filter. In my experience, the drop method tends to deposit the particulate in a non-random fashion due to surface tension effects, or deposition techniques, often leaving areas with visibly higher concentrations of material. For low concentration samples, the non-random deposition may not be readily apparent. The drop method is perfect for quick work requiring identification of particles. The liquid drop technique has the advantage that it is relatively quick, while the filtration technique may take a very long time, due to pore blockage (polycarbonate filters do not have a very high porosity.) Also, if the hydrophilic treatment is removed from the polycarbonate filters by allowing them to dry after wetting with the isopropanol and before the filtration supernatant is added to the filtration chimney, the filters will then be hydrophobic and it will be very difficult to pull the charge through the filter.

However, unless the test laboratories agree to and use the same technique (preferably the filtration technique,) there is a significant window for disagreement and precision broadening. If the method used is the liquid drop, sample preparation technique correlation between laboratories is critical and may be uncontrollable even with a higher degree on inter-laboratory coordination.

Method Validation Study:

Q5) The method validation study design entailed spiking characterized background dust with characterized WTC dust at various levels, and then sending 32 blind samples (16 originals and 16 duplicates) of background and spiked dust to each of eight laboratories (five commercial laboratories and three government laboratories). These laboratories characterized the dust using the protocol described above and then sent the results back to EPA. Was this an appropriate design to achieve the goals of this method validation study?

Answer to Q5:

The goals of this study can be simply reduced to validation of the sampling and analytical protocol for the identification of three common components of WTC dust. The study aimed to determine whether or not the method could discriminate between various spiked concentration levels. In addition, it was established that areas inside the impacted area show significantly increased levels of the target substances.

Given that this was designed as a screening method, it still appears to be time intensive, especially with respect to the post-processing required for the concrete and gypsum/anhydrite analysis. The study did not comprise a large number of samples. As a result, the precision of the method cannot be tightly specified.

This, however, does not preclude the study from meeting its basic goals. The study had sufficient power to demonstrate that two of the target substances could not be used as viable markers for WTC dust. It further showed that the test laboratories could detect and measure the concentration of slag wool fibers in spiked and background samples.

It is less clear that the method, as tried, has the power to provide quantitative results. This will be addressed more in-depth in the answer to question 6. It is appropriate to remember that it was conceived as a screening method, and as such, should not be expected to provide highly quantitative results.

As a screening method, the protocol is adequate to establish that slag wool fibers are present in the samples to some undetermined level of detection. This leaves the question unanswered as to how little contamination this method can detect. The upshot is that when the method detects fibers, it is certain that slag wool is

present (and similarly assumed that the COPCs, for which it is surrogate, are present.) When the result is negative, it is unclear whether or not slag wool is present. This is of some concern as certain surfaces with greasy or otherwise “sticky” properties may not be adequately sampled.

Q6) Did EPA and Versar adequately evaluate and interpret the results from the eight laboratories, as documented by the supplied documents on NYC dust analyses? Dust collected from currently occupied buildings is expected to have lower levels of the key WTC constituents as compared to dust sampled near September 11, 2001 in time or sampled more recently but in uninhabited heavily impacted buildings. EPA will use the results of this method validation study to determine the final distinguishing concentrations for the WTC marker(s). If currently sampled dust has this marker(s) at or above such a distinguishing concentration, EPA would consider the sampled dust to "contain residues of WTC dust" for purposes of estimating the geographic extent of WTC impacts and making cleanup decisions. The key requirement for a distinguishing concentration is that it be adequate to distinguish between dusts that do not contain WTC residues from those that do with a reasonably low false positive error rate.

Answer to Q6:

The method was adjudged adequate by Versar to determine whether or not a property is contaminated with WTC dust. To the extent that the protocol is considered a screening technique, it shows that areas expected to contain the target contaminants do indeed show them, and the areas expected to be free of the contaminants, apparently do not. This begs the question as to why some of the urban samples outside the impacted area (background) do not show any fiber slag wool. Slag wool was not unique to the WTC, and it is expected that some samples of dust from these background areas should show some slag wool fibers. The stark stratification of results serves to highlight the lack of power for the study to investigate the detection level of the procedure.

Simply, in the absence of a site source of fiber, a positive sample is likely to represent a property contaminated with WTC dust. The opposite cannot be inferred from the data in this study.

The exclusion of the results from three of the laboratories in establishing acceptable confidence limits, *for fiber counting*, is unjustified. Given the known expertise of the particular laboratories recruited for the study, exclusion of any of the three is genuine cause for concern. In Appendix F, upper and lower 95% confidence limits are established using data from laboratories designated A, B, C, D, and H. Laboratories E, F, and G were designated as commercial laboratories which had unacceptable performance. This left only two of the original five commercial laboratories. The analysis in Appendix F highlights some important differences between the laboratories, or the techniques they used. Reasons for the differences were not discussed at length in the report. However, it was not the stated aim of this project to identify or capacitate any lab or group of labs. It was to validate the protocol. Except in the case of gross failure of a laboratory to

perform, the data from all laboratories should be included in any evaluation of the data. Further, because of the known breadth of error in particle counting, it is questionable whether the number of samples in the study provided sufficient power to the analysis to legitimately make an exclusion decision. Finally, contract commercial laboratories selected by the bidding process are unlikely to perform differently than the aggregate of the five selected laboratories. Indeed, one or more of the excluded laboratories may win the bid. The entire suite of data should be used to characterize the performance of the protocol.

When examining the data from the perspective of fibers per gram as in the Versar preliminary report Table 4 (page 56), the data is represented as relatively large numbers with significant precision (many significant figures.) However, when the data is examined from the perspective of the raw number of fibers examined, Table 5, (page 58,) a different picture is seen. With the exception of laboratory D, the actual number of fibers counted is seen to be quite low. Indeed, laboratory D systematically shows significantly higher counts than any of the other laboratories. No mention is made in the report of this quite striking result.

The counting of particles is only approximated by normal statistics when there are large numbers of particles. When the numbers or counted particles are low, the statistics may be log-normal, as suggested in the text, or may sometimes be described by Poisson statistics. It may be that the data need be analyzed using distributionless analysis if the data does not suggest a particular distribution. Also, if the counts are sufficiently low, a bias correction may be necessary to correctly model the data. Such bias corrections are empirically determined. In addition, it is usually the case that there is a subjective component to the variance in addition to that expected directly from the distribution.

It is sufficient to note that a Data Quality Objective of ± 30 or $\pm 35\%$ is inappropriate for the small numbers of fibers seen. An unsophisticated look at the data assumes a Poisson approximation is for ease of calculation. The variance goes as $1/n$, and the standard deviation goes as $1/\sqrt{n}$. If the background results from table 3, p 54 of the Versar report are used to calculate a pooled average for $1/\sqrt{n}$, then a relative standard deviation can be seen to be 41%. (A value of 1 was used in place of 0 counts to represent a detection limit.) Using a normal approximation for 95% confidence limits, they are seen to be $\pm 81\%$. A distributionless estimation would be higher. This provides broader acceptance limits than those outlined in the review documents. Again, this does not include any estimate of subjective uncertainty, always present in particle counts.

The impact of this is that clean-up decisions will be made on the basis of very few fiber counts, even though the reported number in terms of fibers per gram will be very high. Any measurements made subsequent to the cleanup will be expected to be lower than the initial survey. The decision band may be sufficiently broad as to make such comparisons impossible.

Q7) EPA has observed differences in slag wool concentration which discriminate between background dust and dust contaminated with WTC residue. Do you agree that the data and analysis presented in the documents support this observation? Please explain your answer.

Answer to Q7:

Limited to the data contained in this report, it appears that slag wool alone can be used to discriminate against background concentrations where there are no discoverable slag wool sources which could account for a positive result. These may include, but are not limited to slag wool containing ceiling tile, insulation, or known demolition sites which could have contaminated the site. It has been noted elsewhere in this evaluation that the converse may not necessarily be true. This study did not investigate the detection limit in sufficient detail to determine the minimum concentrations that could be found in potentially impacted buildings.

Q8) Is there any other way in which the proposed signature marker(s) can be used to determine the extent to which WTC dust may have penetrated and remains in indoor environments?

Answer to Q8:

Given the length of time since the collapse, surface sampling in the way followed in this protocol appears to have the best chance to collect consistent samples (for the purpose of the study.) Other surface sampling techniques, such as tape sampling, might provide a more efficient sampling of target surfaces, and thereby provide a measure of lower contamination amounts (speculative.) But, tape sampling would have the disadvantage of being from a much smaller surface and consequently being less precise, providing less opportunity for replicate analysis and would not integrate well with the data from the current study. Aggressive air sampling, such as previously used for the original measurement of COPCs, has the known deficiency of being very close to the detection limits for the methodology involved and is not practical in re-occupied spaces.

With respect to Polarized Light Microscopy (PLM), it should be noted that so long as the study was centered on all three of the marker substances, the PLM was, perhaps, extraneous. However, when the study excluded concrete and Gypsum/anhydrite, PLM should have re-emerged as a viable alternative. It can be used quickly as a go/no-go test to determine the presence of mineral wool. As such, it would not necessarily be used quantitatively, although many industrial hygiene analysts are already trained in semi-quantitatively determining percentages using this technique. An analytical infrastructure already exists nationwide for accreditation of the techniques involved, along with the training availability. For the semi-quantitative purposes of the study, I believe that PLM still offers a less-expensive and quicker alternative.

Q9) Are there any additional comments or concerns about this study that have not been addressed by these questions?

Answer to Q9:

The study properly chose to step away from the concrete dust and gypsum/anhydrite markers due to their widespread presence in the target environment. Although slag wool was not specific to the World Trade Center construction, this limited study seems to show that it is possible that it can be used as a marker for the intrusion of WTC dust to spaces in the impacted area. The bulk of the comments in this section refer to the protocol as it is used in the establishment of slag wool as a marker.

As a small study, it necessarily has limitations, and some of these should be noted. The report correctly recognizes that vacuuming up sample from a 1 square meter area (or more) renders that space unusable for a replicate sample. However, it does not quantify the sampling efficiency on any surface; even a few representative carpet, fabric or hard surfaces. This could have been done on soft surfaces by replicate vacuuming; on hard surfaces by the same or by pre/post tape samples. Canister vacuums are not anywhere near 100% efficient. This is demonstrated daily by door-to-door salesmen using collection techniques very similar to those contained in the sampling protocol.

Related to this is the fact that this overall protocol is likely to have false positives. No false negatives were noted. There were indeed negatives. The sampling protocol was not constructed in such a way as to evaluate the detection limit for contamination. It is not known how small a contamination this method can reliably find. Presumably, the analytical protocol has a relatively low detection limit, but this was not addressed in the report. The spiked samples at 1% were all positive definite, indicating that a lower detection limit is probable.

The analytical method itself is workable as it relates to slag wool. The gypsum/anhydrite and concrete analysis would be quite cumbersome and time consuming for any laboratory unequipped with automated chemical and spatial analysis. That must be part of the bid if it is included in the final protocol.

In applying the analytical procedures, there are some physical laboratory correlation issues related to lab technique that must be addressed in a group setting with all of the actual analysts involved in the work. Laboratory technique is learned by doing rather than reading. It is an art. It is essential that the technicians actually performing the work be jointly trained, or uniformly trained in some way; either by coming together as in the study or by a key person training *all* of the technicians. Representative training is questionable in quality and ability to provide the necessary precision and technical expertise.

Along with this, within the laboratory protocol there were two distinct methods of sample preparation that, in my experience, will lead to different results. Only the filtration technique should be used. Liquid drop techniques do not generally provide a sufficiently random particle distribution on the sample.

Either or both of the issues above may have resulted in the perceived deficiencies of the excluded laboratories, and in the notably higher results overall for laboratory D.

In the SEM analysis, the laboratories used the USGS BIR1-G for calibration of the chemical analysis. Although not critical to the study at hand, the magnification calibration should be documented somewhere for reference, especially because some of the labs did document fiber size. As noted above, the lower size selection criterion was 0.5 μ m. Because the analytical magnification of choice was 500x, this is 0.25mm on the viewing screen. (Unless the magnification is calibrated to the hard-copy device, and the screen is uncalibrated.) If this is the case, the viewing screen should be calibrated in order that the analyst knows how big the analytical targets are.

With respect to the aim of the project, the protocol design must be sufficiently robust as to be used to establish that the space is clean after any cleanup or other remediation. Such validation should be accomplished before any chance of re-entrainment can take place either from missed sources in the space, from common ventilation plenums, or from sources generally exterior to the target space.

**PRELIMINARY COMMENTS
FROM
FRANK E. EHRENFELD III**

General Outline for Response:

Acknowledgements:

- H. Sonny Robb, Craig Liska, and Thom Snyder for conversations and analytical work on WTC dust samples. Specifically, work on MMVF characterization by PLM, SEM, and TEM using archived samples from various clients and locations in and around the Lower Manhattan site. Thanks also to John Napolitan for literature and extended database searches. Special thanks to Lou Solebello for his vast knowledge of fiber characterization techniques.

Charge Questions:

Basic Development of a Signature:

Q1 – Are the listed criteria adequate for establishing a WTC signature marker?

Documentation of the Existence of a Signature:

Q2 – Does the slag wool marker alone meet the criteria as a WTC marker?

Analytical Method Development:

Q3 – Are the analytical methods written in sufficient detail such that a qualifying laboratory could follow the methodology and obtain valid results without supplemental assistance from EPA or other sources?

Q4 – What information would I add so that a qualified laboratory could follow this protocol and obtain valid results without the assistance of EPA or other sources?

Method Validation Study:

Q5 – Was this method validation study appropriate to achieve the goals outlined?

Q6 – Did EPA/Versar adequately evaluate and interpret the results correctly?

Q7 – Does this study's data support the observations made by Lowers in OFR 2005-1073?

Q8 – What underlying issues relating to these marker(s) and regarding the penetration and persistence of the WTC dust on indoor environments can be further measured and explored?

Q9 – What additional comments or concerns about this study have not been addressed by these charge questions?

Basic Development of a Signature:

Q1 – Are the listed criteria adequate for establishing a WTC signature marker?

The listed criteria to establish an appropriate marker include:

- a) It must be *present at levels unique* to WTC dust.
- b) It must be *persistent*.
- c) It must be sufficiently *homogeneous*.
- d) Available methods are able to detect these screening materials with a small sample size, low minimum detected limit, and low interference from other dust components.

Based upon the review of previous work outlined by EPA, yes it is my opinion that these are adequate for establishing a WTC signature marker. It is obvious that any marker would have to be present at levels that are unique. Can the marker be successfully differentiated from background or ambient levels? Yes, this should be included as part of the criteria. Should the marker be persistent enough to be sampled and measured years after the catastrophic event? Absolutely. Should the marker be sufficiently homogeneous? Of course.

Should available (and established) analytical methods be able to detect the marker even when there is little sample size, even when there would be a terrible detection limit, and even when other materials might interfere with these measurements? Ideally yes. Is this charge question asking about the specifics of slag wool as a marker? No, it only concerns a general list of criteria that should be met for WTC dust. As such, I agree that any marker should meet these criteria.

Documentation of the Existence of a Signature:

Q2 – Does the slag wool marker alone meet the criteria as a WTC marker?

If this question is ‘does slag wool’ meet the criteria for a WTC marker? Then yes there has been evidence presented to draw that conclusion. Is the evidence overwhelming or compelling? No. Does this translate into ‘the *only* marker that meets the criteria is slag wool’, or ‘slag wool is the *only* marker that meets the criteria exclusive of other components established in the dust’? Then no, there is strong evidence that other components meet the criteria.

Regardless of its catastrophic source, WTC dust may be the most studied mixture of building dust and debris in the history of forensic and environmental science. Though only a limited series of samples, certainly the evidence presented by Lowers¹ and Meeker² is enough to conclude that slag wool meets the above criteria. Their findings are clear, beyond reproach, and influential in this determination. That said, there is some concern over the homogeneity of slag wool at geographically extreme locations and at elevated sites in the impacted area.

It would be helpful to learn more about the background samples collected at RTI since the slag wool component of these dust samples far exceeded those studied by Lowers. Were these collected using the same field sampling protocols³? In fact the question still remains that though they “are not included as they are not representative of NY City background dust”(page 12, paragraph 2, section IV)⁴, they might still represent background dust in many building throughout NYC that have slag wool containing building materials. See also Q9.

But what about other well defined and studied markers? Do these also meet the criteria? Some do and some do not. For the purposes of this being a brief response, here are a few questions to consider...

1. Will ‘qualified’ labs be able to differentiate these markers? See Q3
2. What are the expectations for sampling and analysis costs? See Q9
3. Will sample analysis be cost effective? See Q8 and Q9.
4. Though asbestos is present at known⁵ levels in NYC background dust, why was asbestos abandoned as a marker? Clearly the asbestos in the published ambient levels could be differentiated from the asbestos in the WTC dust. Newman’s⁶ opinion is clear on the subject, though he does not list *adequate*⁸ reasons for his conclusions. There are not

World Trade Center Signature Study Peer Review

- persuasive reasons for this oversight? [*Recently I have been made aware that Newman was referring to the asbestos as a surrogate issue and not necessarily asbestos as a marker*]. See Q8 and Q9.
5. Please also consider the distribution of slag wool (and heavier dust components) in a diminished gradient from ground zero to limited geographic locations (and higher elevations). This is a real weakness in the study that may need to be further explored. See Q8 and Q9.
 6. Could a primary and secondary marker system be employed to verify WTC dust origins? This was postulated by Meeker² in his conclusion's third paragraph. See also Q8 and Q9.
 7. Is there significant separation between background and WTC slag wool levels to conclude that it alone meets the marker criteria? Please note (page 48 of the Final Report) that Versar points out that for the DB source samples "at the 10% spike level, the slag wool concentration typically exceeds one standard deviation, but never exceeds two standard deviations above the average background sample concentration." Again even for the USGS source samples Versar states that "the 5 and 10% levels are essentially all more than two standard deviations above the average background." EPA says (page 21 Conclusions) that this is "sensitive enough". I'm not sure that I can agree.

Regardless of the case for additional markers, a very compelling argument is repeatedly made in the final report⁴ (p. 6 section I) and in ancillary documents⁷ (Appendix A1, p. 10, section A5.1.) that the basis for an "appropriate screening test" would result in some false positives but very few, if any, false negative. Slag wool meets the criteria.

Analytical Method Development:

Q3 – Are the analytical methods written in sufficient detail such that a qualifying laboratory could follow the methodology and obtain valid results without supplemental assistance from EPA or other sources?

Overall, yes the protocols and step by step analytical procedures should be sufficient for a ‘qualified’ lab to employ successfully without assistance. There are comments/suggestions in Q4 that may clarify certain steps found in the proposed method.

There is some question about what defines a ‘qualified’ laboratory. Considering the abandonment of data from three of the eight laboratories retained by Versar for this study, it is questionable if enough independent laboratories could be qualified. Indeed at the bottom of page 19 of Appendix A mention is made that the method evaluation will include evidence “that the analytical method works well enough, and is able to be carried out by enough analytical laboratories...” What are the expectations of the panel concerning ‘works well enough’ and ‘enough analytical laboratories...’? Will the panel develop criteria concerning laboratory qualifications? Would they, should they consider allowing a government laboratory to handle the analytical work with a contracted QC lab? See also Q9.

The DQOs and MQOs that are outlined in A7 seem to allow for a wide enough range of variability. Please consider the impact of a sentence of Appendix A again (page 13 section A.6.3.) that briefly addresses the subject of method validity by saying that “criteria such as time for analysis, and intra- and inter- laboratory variability will be considered”. Now consider that almost 40% of the data did not meet muster. Does this mean that the method’s procedures were unable to be followed or that the laboratory staff was not competent enough to follow through? I think the latter.

What are the criteria for determining if a lab is qualified? Appendix A1 (p 14 section A8) lists only training by PLM and SEM as qualifiers. On page 19 of the Appendix (section C) it notes that all contract labs are required to employ standard QA practices and be under in-house QA personnel.

World Trade Center Signature Study Peer Review

Versar's preliminary report Addendum dated August 5, 2005 (page 60 of the Final Report) indicates that each of the five laboratories analyzed a glass standard reference material to determine statistical acceptance limits. The accompanying table seems to display data for three laboratories, not five. See also Q4.

Versar's statistical analysis of laboratory performance notes on page 63 of the Final Report that "Because Labs E, F, and G fail in more than one category in both spiked data sets, they are not included in the remaining study." That's it?! The one paragraph coverage on page 13 of the Final Report touches upon this issue and does conclude that "all laboratory comments will be taken into consideration when finalizing the protocol." This deserves an investigation that can, in a one or two page memo, list possible reasons why these labs failed to meet qualifying status. This memo would go beyond the reasons listed on page 20 of the Final Report for high variability. These include operator/analyst experience. This is understandable in many analytical investigations, but surely technicians must be engaged who have basic knowledge of the target analytes. This begs the question: how were the labs in this study selected?

Q4 – What information would I add so that a qualified laboratory could follow this protocol and obtain valid results without the assistance of EPA or other sources?

This answer assumes that a ‘qualified’ laboratory meets some sort of criteria for staff experience and background as well as minimum facilities and instrumentation requirements.

1. The initial mass of samples and sub-samples seems to be an issue noted at the top of page 21 of the Final Report. Please amend the method to include a tighter range.
2. Please also list sample rejection criteria. Please consider: Too little sample (ex. If less than 200mg?), too many interferences (ex. Loaded with ash or combustion product), too much moisture, cross contamination with other submitted samples, etc.
3. Standardize or list the acceptable particle analysis software so that there is no variation. (Appendix p. 39 Section 7).
4. Please standardize or provide acceptable parameters for sonication energy and calibration information. (Appendix page 42 Section 11.5).
5. Define “the more concentrated sample” with an empirical range. (Appendix A page 43 Section 12.2.1).
6. Please expand on step 12.2.1 on page 43 of Appendix A referring to “counting fibers per type until a statistical representation of the ratios of fiber compositions in the sample is achieved.”
7. Please specify the duration and sensitivity of the scan used to determine Gypsum/Anhydrite in Section 12.2.2 on page 44.
8. Section 12.2.3 notes 40 minute total acquisition time per map for each of 10 fields. Is this overkill? Won’t 400 plus minutes be terribly expensive for each sample? Is this really a ‘screening’ method? Could this be trimmed down without affecting sensitivity?
9. Section 12.2.5 implies that 1000-1200 particles will have to be individually characterized to achieve the required sensitivity of 10% relative error. Is this accurate? Again, is this really a ‘screening’ method? Please amend.
10. This same section (Appendix A page 46 section 12.2.5) indicates that particles over 20 μm long will have to scanned and images recorded and saved. If you digest the information displayed in Meeker² Figure 9 is this a burden of documentation that is unnecessary? Please amend.

11. It would be helpful to see a mock data report or Certificate of Analysis listing all of the expected reporting limits, variables, concentrations, densities, LOQs etc. This might be supplied along with the examples of Data Sheets supplied in the Final Report's Appendix section 15.
12. Page 60 of the report copies a Versar Memo concerning a glass standard reference material. The interpretation of this data should be limited to the inter-laboratory variation of instrumental detection. Is this a commentary about the method's ability to successfully measure these elements in real-world particles? It only partially achieves this. Should a section of the method include expanded detail on expected calibrations of software, EDS sensitivity, etc.?
13. What could be implemented to further qualify a laboratory? Any on-site inspections or demonstration of proficiency using prepared standards and spiked samples?

Method Validation Study:

Q5 – Was this method validation study's design appropriate to achieve the goals outlined?

Was the design appropriate? Yes. The MQOs, DQOs, quality assurance, and other controls were selected and placed in the study with sensible foresight. The design seems to be sound. There is some question as to the abandonment of PLM and exclusion of TEM as a screening test. See Q8. There is also some concern about the evolution and final course that the method followed. See Q9.

Was this method validation study able to achieve the goals outlined on page 9 Section III of the Final Report? Or put another way, did the study validate the method? Not quite. These are the three goals as outlined in the method:

- 1) To demonstrate that slag wool is a 'reasonable' marker for WTC dust. *This has been achieved and is further discussed in Q2 and Q7.*
- 2) That WTC dust at a diluted concentration can be distinguished from background. *The method has limited utility and too many loose ends to pronounce it valid.*
- 3) That the analytical method works well enough and is able to be carried out by enough analytical laboratories to a) evaluate the markers and b) to distinguish WTC dust from background dust. *There is persuasive evidence that this study has demonstrated that the lab community has not achieved a level of competency to meet this last objective.*

Reasoning for the last item concerns the ever increasing complex and convoluted analytical procedures and the production of data beyond what I would consider reasonable for a 'screening' method. Instead, this proposed 'screening' method seems to lean to a full inorganic particle investigation, complete with quantitative and qualitative analytical procedures and the subtle nuances that define both characterization and forensic science. See Q9.

Please see my comments concerning 'qualifying' laboratories in Q3 above.

Q6 – Did EPA/Versar adequately evaluate and interpret the results correctly?

Versar's analysis starts on page 45 of the Final Report. Their evaluation of the analytical data summarized in three bullet points on that page seems to be adequate. Indeed their conclusion on page 72 clearly states that "the conclusions are based solely on the analytical data provided from the laboratory test and a few analyses of the 100% WTC spike samples." If their task was to provide analysis of the data alone, then they have indeed fulfilled their mission. EPA's responsibility, on the other hand, is probably broader. Certainly the WTC panel will expect a thorough review of all the attributes of the study in addition to the analysis of the data. Taking this into consideration, then no, EPA did not adequately evaluate and interpret the results from the eight labs etc.

It was not *eight* labs it was five. The EPA's Executive Summary (page 4 of the Final Report and page 21 of the Conclusion) made no mention of the significance of the failure of almost half of the contract labs to produce acceptable data. This is an omission that though may not be serious, certainly raises speculation that other factors may not have been properly addressed (ex. RTI, NJ, and LI samples). Page 13 of the Final Report briefly mentions this issue but without its proper weight. Instead of (page 4 of the Executive Summary and page 21 of the Conclusions) the weak pronouncement that 60% of the labs 'reasonably' were able to measure the marker, it would have been refreshing to have read that either "the method has clearly validated the hypothesis that slag wool meets all of the qualifications of a marker and that the method works well enough that enough labs can successfully employ the method" or that "the method, in its current form has not yet demonstrated its utility".

Is there significant separation between background and WTC slag wool levels to conclude that it alone meets the marker criteria? Please note (page 48 of the Final Report) that Versar points out that for the DB source samples "at the 10% spike level, the slag wool concentration typically exceeds one standard deviation, but never exceeds two standard deviations above the average background sample concentration." Again even for the USGS source samples Versar states that "the 5 and 10% levels are essentially all more than two standard deviations above the average background." EPA says (page 21 Conclusions) that this is "sensitive enough". Considering the strengths and weaknesses of most commercial testing laboratories with these capabilities, I'm not sure that I can agree.

This shortcoming in interpretation seems even more relevant when accounting for the interjection in this charge question of additional caveats and disclaimers about the employment of this method on currently occupied spaces and their expected lower levels of COPC. The charge question states that “the key requirement for a distinguishing concentration is that it be adequate to distinguish between dusts that do not contain WTC residues from those that do with a reasonably false positive error rate”. There is a question of confidence regarding this study’s limited data set and the 10% spiked level that it claims is ‘reasonably’ measured. Perhaps the study might be expanded to increase confidence in its efficacy. See Q9.

Versar’s data reduction is well reasoned and appropriate. I agree with their logic and their statement (page 63 of the Final Report) that C and D labs should be added to the preferred performer group of labs. Lab H’s inclusion is probably warranted for the reasons listed. Versar’s model for precision is satisfactory. I have trouble interpreting Table 5 on page 71!

Both Versar’s data analysis and EPA’s conclusions regarding gypsum and concrete components seems to be validated.

Q7 – Does this study’s data support the observations made by Lowers in OFR 2005-1073 and Meeker in OFP 2005-1031? Does the study’s data support the observations by NERL and its preliminary analysis?

Evaluating the three studies recently presented by EPA and comparing them to this method’s conclusions yields mixed results.

In her discussion Lowers states that “the maximum amount of COPC, such as Chrysotile asbestos derived from WTC dust could be calculated by using the relative proportions of phases found in WTC dust by Meeker”. Maybe, but there has been other studies^{9, 10} offering data on various COPC (often expressed in different units of measurement – see also Q9) that also could be used to estimate dust components (even those that might meet the definition of marker). Further work on slag wool containing background samples (ex. RTI samples discussed earlier) would have to be done to connect-the-dots towards this conclusion. Lowers did not analyze (or at least report) any Chrysotile detected in the 1073 study. Furthermore, the technology employed (SEM/EDS) may not have been sufficient to accurately detect asbestos as a COPC. Her data supports the hypothesis that this study promulgates... that slag wool is a sufficient marker for WTC dust.

The most significant statement in Lowers is the last paragraph... “This study has examined only six background samples. In order to arrive at a statistically significant representation of New York City residential background dust compositions more analyses are needed. In addition, sampling background dust from office buildings and other interior environments is needed to determine background ranges of signature components in these types of interior spaces.” Therefore the ‘strength’ of the Lowers report is its data suggesting slag wool at insignificant to none detected levels in NYC background dust. The limited sample set (six) underscores its weakness. Indeed, EPA should be cautioned about proceeding with implementing any method based on so few samples. A more robust set of data would certainly have to be established.

Meeker’s conclusions are worthy of a second look. I agree with the conclusion that “if slag wool fibers are not found in settled dust samples above a predetermined critical level, it is unlikely that COPC derived from the WTC could be present at significant levels in the samples.” His last paragraph is critical. Here he states that the ability of slag wool to be a marker depends on its levels in other background samples. This continues with the statement that there is a real

World Trade Center Signature Study Peer Review

possibility of slag wool being present in background samples (ex ceiling tiles etc.). Finally, Meeker adds that the size distribution might prove useful in distinguishing WTC and non-WTC dust samples. I agree with his conclusions in that slag wool has the potential to be a marker and that the particle size distributions may be useful, especially taking into account the physical forces inflicted upon much of the dust (pulverized much finer than virgin components found in-situ). Like Lowers, the 'strength' of the Meeker report is its data suggesting slag wool at significant levels in known WTC impacted samples. The strength of Meeker's 1031 observations that support this study's premise continues in his brief mention of the correlation between the proposed marker and other COPCs. Conversely, the limited sample set again underscores its weakness. Once more, EPA should be cautioned about proceeding with implementing any method based on so few samples.

The NERL preliminary analysis data in Appendix B has more samples. The duplicate QC results are admirable. Was there an order of magnitude difference in slag wool background versus impacted sites? Was this evidence of 'distinguishing' concentrations between the two types of samples?

This screening method study did have a more vigorous set of samples (32 total background and spiked samples) than the previous preliminary EPA studies^{1,2}. Did the screening method study show conclusive evidence that slag wool levels between background dust and impacted dust can be routinely discriminated? No. Did the screening method study conclude that slag wool levels between background dust and 'spiked dust' can be discriminated at low levels (10%) by a select few labs? Yes.

Please consider that only the NERL samples used the field sampling protocol²⁴ that the study's method outlines. Was there a model for the efficacy of the vacuum collection protocol using settled dust (preferably dust that had been settled for a few years) spiked with the various WTC reference materials? Preparing the spiked standards in a laboratory setting and, from the limited data, extrapolating potentially far-reaching conclusions, may not be wise. See Q9.

Overall, the background and spiked samples examined in this study by the five qualified laboratories, support the observations previously presented by Lowers, Meeker, and NERL.

Q8 – Is there any other way in which the proposed signature marker(s) can be used to determine the extent to which WTC dust may have penetrated and remains in indoor environments?

For slag wool as the proposed signature marker: limited. For other potential markers: perhaps. First, by process of elimination, let's consider all of the COPC and eliminate those that will not meet the proposed criteria as markers.

There is compelling evidence that both gypsum and concrete in the WTC dust should not be considered as markers (page 21 of the Final Report). Indeed, four years after the catastrophic event, these components of the dust may be significantly removed in occupied spaces and greatly reduced in any space where a cursory clean-up was attempted. The data suggests that any exposure to moisture (outside samples, rain exposed, carpet cleaning, etc.) would alter (by partial dissolution and disbursement) these constituents. Finally, buildings renovated in the last four years (and any pre-existing construction debris) may preclude any significant discrimination between these markers and WTC dust.

The heavy metals and organic compounds listed as COPC may have some utility as secondary confirmation markers though their persistence and ability to significantly discriminate from typical NYC background levels may pose a problem^{20, 21, 22}.

Please consider the distribution of slag wool (and heavier dust components) in a diminished gradient from ground zero to both limited geographic locations and higher elevations. What COPC has been well studied, will easily penetrate higher elevations, is persistent, will remain unaltered (ex. gypsum and concrete are water soluble), and remain in measurable quantities even after cursory clean-ups? What COPC has established analytical methods? What COPC has guidelines in place to protect workers and establish re-occupancy thresholds? What COPC has experienced field engineering and project design in place? What COPC meets the criteria for a marker set forth by the WTC panel and listed in the first charge question? What COPC also meets the study design criteria on page 9 of the Final Report? What COPC has an experienced, well qualified, and economically competitive laboratory structure in place? The answer is obvious and the issue has been debated previously... Chrysotile asbestos in WTC dust!

The previous debate¹¹ may have been off-center as to the utility of Chrysotile as a marker. That is, the previous review concerned asbestos *in air* as a surrogate (travels with and is linked to other components) and not as a marker. It concluded that Chrysotile in air would serve as a suitable – but not stand-alone – surrogate. Why not Chrysotile asbestos in the surface dust as a marker? At least why not as a secondary marker? That peer review panel also concluded that a multi-tiered approach is feasible... “asbestos sampling results would be strengthened with the addition of lead wipe samples.”

It has already been proposed that Chrysotile and lead on surfaces be considered as a paired marker-like team²². Meeker also proposed a multi-tiered approach in his conclusions². “An analysis strategy for routine samples could evolve using rapid scans of settled dust by SEM to look for the presence of the MMVF. If found, these fibers could then be analyzed using EDS to determine fiber compositions. If the majority of fibers (>85%) detected were of slag wool composition, or if slag wool was found at a predetermined critical concentration, the sample would then be searched for gypsum and concrete particles along with other MMVF components. Further confirmation of the presence of WTC dust could then be reached by looking for secondary components in the approximate abundances found in this study. Alternatively, if slag wool, gypsum, and concrete were present, the sample could then be analyzed for COPC such as asbestos, lead, and organic compounds.”

Though asbestos is present⁵ in NYC background dust, why was asbestos abandoned as a marker? Clearly the asbestos in the published ambient levels could be differentiated from the asbestos in the WTC dust. Newman’s⁶ opinion is clear on the subject, though he does not list *adequate*⁸ reasons for his conclusions. There are not persuasive reasons for this oversight? [*Recently I have been made aware that Newman was referring to the asbestos as a surrogate issue and not necessarily asbestos as a marker*].

The public health (not to mention the risk assessment and public relations) implications of sampling for and utilizing a dual asbestos and lead approach is huge²⁵.

Therefore, slag wool (and all MMVF components?) and Chrysotile examined in the surface dust (not the air) in potentially impacted areas along with lead analysis might be re-visited. There are several advantages and limitations that would have to be considered using this approach:

World Trade Center Signature Study Peer Review

1. What are the ambient levels⁵ of Chrysotile asbestos in NYC? How much information exists in both public and private databases? Certainly this will vary greatly especially in older buildings that have poor housekeeping issues.
2. What are the levels of Chrysotile in WTC dust? This has been studied⁹. Perhaps there exists enough statistically sound data in the private sector (building owner and insurance industry?) to establish these levels with confidence. The concentrations might be studied not in percent of the WTC dust (well studied at 0.02–2.0%) but in structures per cm² of the surface area tested?
3. Porous surface sampling and the ‘carpet as a reservoir’ issues for these COPCs must be addressed⁶. It may be considered an analytical bonus that these micron-sized COPCs remain in carpet etc. even after substantial vacuuming and cleaning. Indeed, methods have been used to fully investigate some of these concerns¹².
4. The same physical forces that were mentioned by Meeker concerning MMVF and its ability to be entrained in carpet (electrostatic interactions) and to not be easily homogenized on a sample are also a concern of asbestos (strong surface charges).
5. Re-entrainment issues are well studied^{13, 14, 15}, and could be used as a final re-occupancy test along with lead on surfaces.
6. There are in-place engineering and design companies that could implement off-the-shelf protocols for clean-up and testing. This would be more cost effective than having to go through the typical learning curve with new guidelines and protocols.
7. There are in-place laboratory methods for sampling and analysis of asbestos in surface dust^{17, 18, 19}. There are in-place models for asbestos in surface dust, that though not risk based, may provide utility for this project²³.
8. The asbestos and lead laboratory community is extremely competitive and reasonably cost effective.
9. Commercial asbestos laboratories have trained staff, adequate facilities for the preparation and analysis of this target analyte, and are accredited by local, state, and national organizations that require strict QAQC programs, proficiency testing, and on-site assessments.
10. Though no asbestos in dust proficiencies exist, EPA might promote this to establish inter-laboratory precision. There have been samples generated and studies conducted among several well qualified laboratories by ASTM¹⁶ towards its objective of establishing accuracy and precision data for their methods.

11. Slag wool and other MMVF are also identifiable by TEM/EDS. In fact, at the micro level of analysis of TEM, perhaps this is an easier analysis than the study's proposed method.

Q9 – What additional comments or concerns about this study have not been addressed by these charge questions?

There are several issues that have been addressed within the context of the charge question responses. Issues dealing with standards preparation, size of the data set for the various preliminary investigations, concerns over why selected laboratories failed to produce data within statistically allowable bounds, field sampling efficacy, and the choice of slag wool as a primary marker have been discussed.

Here are additional comments and concerns in no real order of importance.

1. How would the vast amount of borosilicate present interfere with proper discrimination of the other MMVFs? The Na sensitivity must be at a maximum to differentiate by SEM/EDS. Has there been any thought to employing the Emmons Double Variation Method²⁶? This is a relatively accurate and inexpensive screening tool to compare known versus unknown glass fragments.
2. The failure of those few labs to produce qualified data is still disturbing. Could this peer review panel see information as to each lab's reasons why there were shortcomings? This is especially a concern since each lab knew the high profile importance of their task.
3. The lack of uniformity²⁷ between studies done on the WTC dust is probably very frustrating for the WTC Panel and those working on making sense of the data pool. Could EPA task a contractor to pool all of the public and private data from the last few years worth of efforts to clean impacted areas? Specifically, to add even more depth as to the efficacy of sampling procedures, cleaning procedures, engineering controls, measurable markers and/or surrogates, reasoning for re-cleaning, and baseline/MCL threshold guidelines.
4. IATL and others²⁸ have noted that a significant population of Chrysotile asbestos from WTC dust has been 'damaged'. That is, at high magnification, it is clear that there is a surface pitting on the Chrysotile surface. Most times the Chrysotile appears as single fibrils. In extreme cases these structures have a measurable sulfur peak. Might this be used as a confirmation tool to differentiate WTC Chrysotile from ambient Chrysotile in buildings that contain ACM?
5. Most times if you go to a surgeon for a medical opinion they will recommend surgery. Perhaps a poor example, but if the panel tasked an SEM-based laboratory to develop a

World Trade Center Signature Study Peer Review

screening method, then the expected was realized. SEM has limited utility²⁹ for analyzing Chrysotile asbestos fibrils. SEM also has to slog through time-consuming EDS analysis of hundreds (even thousands³⁰) of particles. This is by no means a slight against the excellent USGS lab and those who have employed their skills and expertise to assist the panel. In fact, my bias tends towards materials investigations using PLM, SEM, and TEM techniques. This is evidenced in my response in Q8.

6. What are the panel's expectation for finding 'enough qualified laboratories'?
7. What are the panel's expectations regarding costs per sample? Including collection, planning, and analysis? Appendix A section 6.3, p. 13 indicates that time/cost will be a factor in assessing the method.
8. Could a multi-tier approach be considered?
 - a. Perhaps a simple pH test might be a good starting point. All WTC dust samples measured in the first month or two after the collapse had high (>9) pH.
 - b. This might be followed by PLM screening for the characteristic minerals and their fingerprint percentages. By screening: a 100mg portion would be examined under the stereoscope, a few 5-10mg sub-samples would be further analyzed to qualify the minerals (isotropic versus anisotropic) and one sub-sample more closely characterized using multiple RI oils and comparing to known WTC dusts.
 - c. Similar sub-samples might be sent for lead analysis.
 - d. Perhaps 100cm² carpet swatches (or microvac samples) might then be analyzed for Chrysotile asbestos and MMVF phases.

Finally, what constitutes a 'screening' method? This is perhaps the 'elephant in the room' that many panelists may ignore. Is this WTC Signature Screening Method really a screening method? The concept that may have started as a simple screening protocol has become a complex and convoluted analytical procedures with the required production of data beyond what I would consider reasonable for a 'screening' method. Instead, this proposed 'screening' method seems to lean to a full inorganic particle investigation, complete with quantitative and qualitative analytical procedures and the subtle nuances that define both characterization and forensic science. Some examples:

- Meeker² sought to utilize "routine analytical techniques". The proposed method may not have evolved in that direction.

World Trade Center Signature Study Peer Review

- Qualitative and quantitative approach for slag wool (40minutes x 10 fields/scans each = 400 minutes per sample) as proposed is beyond the concept of screening.
- Qualitative and quantitative approach for gypsum and associated minerals (1000-1200 particles analyzed) as proposed is beyond the concept of screening.
- Many real samples will have limited matrix material to measure. This low level of residual dust may produce horrendous limits of detection by SEM. The conclusion that SEM can 'quickly and easily find' slag wool may be debatable.

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World Trade Center Signature Study Peer Review

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**PRELIMINARY COMMENTS
FROM
MICKEY E. GUNTER, Ph.D.**

**Mickey Gunter's comments to "Charge Questions" for WTC signature
September 11, 2005**

Q1) The following criteria were established to assist EPA in the selection of appropriate constituents in dust to be characterized as WTC signature constituents:

- a) They are present at levels unique to WTC dust (distinct from urban dust)
- b) They are persistent for many months (not volatile)
- c) They are sufficiently homogeneous in WTC dust; and
- d) Available analytical methods are able to detect these screening materials with small sample size, low minimum detection limit, and low interference from other dust components.

Based on information in the supplied documents, and any other knowledge you may bring to the table, are these criteria adequate for establishing a WTC signature marker? If the answer is no, please elaborate.

Yes the above criteria are adequate. However, the word "they" needs some discussion and there are some rather loaded words in "d." To me "they" implies that what is sought is some combination of constituents that could characterize WTC dust from other forms of New York City dust, yet only slag wool has been selected. As I will discuss more later in my comments, I think that some unique combination of materials could have been found.

In regard to "loaded words" in "d," who might these "available analytical methods" be available to? Commercial labs only, or more research-based labs? Also, many of the commercial labs, at least the ones used for this study, have their roots in asbestos work; it seems that the WTC dust is being treated more like asbestos type material (i.e., particle-based analyses are being performed) than a bulk material. "Small sample size" should have been defined; does this mean 1 mg, 1 gram, or 100 grams? Again, for typical "asbestos" methods often only sub-mg sample sizes are available; these small sample sizes in turn dictate what analytical methods can be used. But for WTC dust, multi-gram samples are available and different analytical methods might be chosen based on these larger sample sizes. The same question should be applied to what is meant by "low detection limit." The issue in "detection limit" as used herein really is one of the sensitivity of a method to correctly determine if a dust sample contains a WTC signature. This might mean being able to measure one material at the ppm level, or use several different materials measured at the percent level.

Q2) Based on information in the supplied documents, and any other knowledge you may bring to the table, do you agree or disagree with the conclusion that slag wool alone meets the criteria as a WTC marker? Please explain your answer.

Based on what has been provided, I agree that slag wool can be used to detect 10% and higher WTC components of indoor dust. I am not sure how well slag wool meets the criteria in Q1, however. For instance, the data on background levels of slag wool (in fibers/gram) are given in Table 1 (p 14) with an average and standard deviation of 35,950 and 74,300, respectively. Thus, the standard deviation appears to be larger than the

mean, which most certainly does not assure usefulness of these background data. After removing a couple of high values (and this troubles me to remove observations so the results “fit” better for the proposed use), these values drop to 17,740 and 15,835, respectively. Regardless of which numbers are used, the variability of slag wool is very high in the background samples. These high variations, in turn, decrease the sensitivity of using slag wool to determine if WTC dust is a constituent of indoor dust to 10%. Thus, while slag wool is a WTC dust marker, its variation in the background samples limits its use.

Q3) Are the analytical methods written in sufficient details such that a qualifying laboratory could follow the methodology and obtain valid results without supplemental assistance from EPA or other sources?

Yes.

Q4) If the answer to Question 3 is no, what items or information could be added so that a qualified laboratory could follow this protocol and obtain valid results without the assistance of the EPA or other sources?

NA

Q5) The method validation study design entailed spiking characterized background dust with characterized WTC dust at various levels, and then sending 32 blind samples (16 originals and 16 duplicates) of background and spiked dust to each of eight laboratories (five commercial laboratories and three government laboratories). These laboratories characterized the dust using the protocol described above and then sent the results back to EPA. Was this an appropriate design to achieve the goals of this method validation study?

In theory, this method was the correct approach, but there were problems in implementation.

Spiked sample homogeneity (i.e., standard preparation): The main intent of this portion of the study was for the labs to characterize the WTC dust for the WTC signature(s). As seen in all the reported data, there was a large amount of inter- and intra-laboratory variability. On page 49, four caveats are given why this variability may have occurred, but I think a fifth (and probably the most significant) caveat needs to be added: the labs prepared their own samples.

There is no doubt it is difficult to homogenize the spiked samples, and a major portion of the variability in the lab measurements might have been a combination of differences in the samples they received and/or their methods of preparing the samples. A much better test of this method would have been if each of the labs made measurements on the exact same sample set (i.e., one set of SEM mounts could have been made by USGS and sent to each of the participating labs). If this would have been done, I believe a much better idea of precision and accuracy would have been obtained on the slag wool contents.

Choice of spiking samples (outdoor vs. indoor): One of the intents of this project was to determine the WTC signature for indoor air, so it was a little strange why a mixed outdoor/indoor WTC dust sample was used for spiking. I think the size distributions (and probably compositions) of indoor and outdoor dust would differ. Thus, I do not think the indoor/outdoor WTC spiking sample should have been used, and results from it are not really helpful to address the issue of indoor air.

Fibers vs. areas for different constituents: It seemed strange that slag wool was measured based on fiber counts and the other two components were based on area. I think the fiber counting is a holdover from measuring asbestos, and it makes no sense to use it. In reality, it would have made much more sense to determine the percent mass of the components, which would be more directly related to the area than to the number of fibers.

Q6) Did EPA and Verser adequately evaluate and interpret the results from the eight laboratories, as documented by the supplied documents on the NYC dust analyses?

I think the results are basically over-interpreted. I really found little use for the statistical analysis presented on pages 62-72. The same interpretations can be gained from just looking at Figures 4-7 (i.e., that only slag wool is a reliable maker, and then only for greater than 10% WTC dust in an indoor sample).

In the fourth paragraph on page 62, values are given for the mean and standard deviation for the two spiking materials followed by a value for “nominal background level of 7,190” which I interpret to be the background. However, I have no idea where this number came from. Table 1 gives values of 35,950 or 17,740 for the background values. Following this is a discussion on theoretical slopes for regression lines for the two spiked sets of samples, but yet no mention is made of theoretical intercept. It appears the sample the USGS spiked (a non-impacted sample from NE Queens Maid service, Appendix B) contained no slag wool, so the theoretical intercept would be zero. This theoretical intercept is confirmed by the experimental data produced by the USGS in Figures 2 and 3. Applying this zero intercept most certainly would effect the results shown in the statistical analysis section.

I was also troubled by removing labs from the data analysis (i.e., just throwing anyway this data). There is a reason the labs obtained these numbers. And, even though individual labs are given by number, the labs participating in this study all have experience in doing these sorts of analyses. Also, the three labs that were thrown out were all commercial labs, while lab H, another commercial lab, did not perform as well as the three government labs and the one commercial lab deemed to be of higher quality. So if these labs have trouble, won't all commercial labs? Possibly, as I stated above, the problem was in the sample preparation and not in the analytical work. It also seemed strange to me to exclude data in other parts of the report, such as the two high slag wool values that are excluded from the data in Table 1 and Figure 4. Rationalizing throwing away data so the results “fit” is troublesome at best.

Q7) EPA has observed differences in slag wool concentration which discriminate between background dust and dust contaminated with WTC residue. Do you agree that the data and analysis presented in the documents support this observation? Please explain your answer.

Yes. I think it is fairly clear from the data in Table 1 (p 14) that the proposed method herein can be used. However, as pointed out above, this method will only work to a detection limit of 10%, or so, for WTC indoor dust in NYC indoor dust. I am not sure if that sensitivity is sufficiently low.

Q8) Is there any other way in which the proposed signature marker(s) can be used to determine the extent to which WTC dust may have penetrated and remains in indoor environments?

Yes, but I think it would require considerable more research. I can only speculate on some of the uses because I have not studied the WTC dust in detail, but I can offer some ideas.

As pointed out in the documents, gypsum will dehydrate to anhydrite with sufficient heating. Possibly anhydrite might occur in some of the combusted products from the WTC and could be used as a marker for that portion of the debris. In fact, both gypsum and anhydrite show up in an X-ray diffraction pattern shown in the USGS (2001) open file report (ofr-01-0429) on the World Trade Center. (Of course a study would need to be done to determine the background levels of anhydrite in New York residences, but I assume there would be very little.)

Even though outdoor dust is less of a concern than indoor dust, I think it would have been useful to obtain PM10 and PM2.5 filters and analyze them to gain a better idea of the composition of New York dust before and after September 11, 2001. I assume the state of New York would have these filters; I have done similar work in Idaho and we obtained the filters from the Idaho Department of Environmental Quality.

It seems logical to me to better characterize the materials that comprised the World Trade Center, especially the slag wool, concrete, and gypsum. This could be accomplished in two ways: 1) obtain construction records and 2) obtain samples of the building which are now in a landfill. There might have been some unique property of these constituents that would earmark them to WTC dust. For instance, a special quarry may have been opened solely to provide aggregate for the concrete, or there may be some special chemical characterizers (possibly the trace element composition) of the slag wool or a similar unique composition to the gypsum or the window glass.

Q9) Are there any additional comments or concerns about this study that have not been addressed by these questions?

I have several comments that did not seem to fit in the above. I appreciate having this section for those comments. Hopefully these comments will be helpful in this work and possibly in future complicated analytical problems. My comments are intended to be constructive criticisms, but some are rather blunt and should not be taken personally by

those who have done the work. Many of these people have been thrown into these issues, forced to work under political and time pressures, and may not have in-depth backgrounds in the issues in which they are trying to find solutions. I think working under these conditions is problematic in itself and will not yield the type of results that our society deserves.

Polarized light microscopy (PLM): I was taken aback, and do not believe the statement on p 49, second paragraph that states “The PLM analyses were curtailed because it became obvious that PLM could not adequately differentiate between fiber types.” The only *obvious* aspect of this statement is that whoever concluded this has little to no knowledge of how to use a PLM or how to interpret the optical properties of materials! Refractive indices of materials are very sensitive to chemical composition and because the chemistry of the fiber types differ; so too will their refractive indices. Observation of the spectra of the slag wool given in the USGS open file report (2005-1165) showed similar compositions, so one would assume the refractive indices of the slag wool might not vary that much. Possibly someone has looked into these relationships, but without being told this was considered it is hard to make that assumption. In fact, the PLM might be a much more useful method for differentiating among the different fibers and obtaining slag wool content than SEM. I think it would be worth pursuing this area of research with a skilled microscopist who understands the relationships between the optical properties of materials and their compositions.

Likewise, the optical properties of gypsum and anhydrite could be used to differentiate these two minerals. Similar methods as those with the SEM and percent area detection could be developed. Gypsum and anhydrite can also be easily distinguished from each other optically, while this would be almost impossible with chemical data from the SEM. And if anhydrite could be related to combustion products of the WTC dust, it could also be used as a marker.

Use of multiple analytical methods and different markers: At the outset of this section, I want to state that Greg Meeker and the USGS staff have done a wonderful job over the past 3-4 years in developing and employing micro-analytical methods to help understand societal issues surrounding Libby, Montana amphiboles and now analysis of the WTC dust. However, I think other methods could also be used to help characterize and fingerprint WTC dust. As I stated above, I have done no research on WTC dust, but I often work in similar areas. For instance, I just finished a project where I used PLM (for particle identification and morphology), SEM-EDX (for particle morphology, chemistry, and identification), ICP (for bulk chemical analysis), XRF (for bulk chemical analysis), and powder X-ray diffraction (XRD, for bulk mineral identification and phase quantification) all integrated into one project. It seems like other analytical methods could have been used in conjunction with SEM work. Clearly, PLM could have been used, but it appears those attempting to use it were not knowledgeable on how it might have been used.

In geological research, we typically ask questions like “what is the source of a mineral or rock?” To answer this we often look for the rare and uncommon constituents in the

materials. For instance, trace elements and isotopes are often used. For WTC dust, chemical analysis by XRF or ICP might reveal a trace-element signature. Possibly someone performed these experiments and nothing could be found; if so, this should be noted. While isotope analyses are hard to perform and expensive, they too might provide a definite answer with less subjectivity than counting fibers. Since it appears that millions of dollars are going to be spent dealing with WTC dust, it might be worth the expense to do this type of analytical work up front.

As I have stated in this section, as a practicing mineralogist I use multiple analytical methods, and these methods change depending on the questions at hand. Another advantage of multiple methods is they can tie a research project together. In other words, one can obtain, for example, phases percentages by two separate methods. When this is done, the two values had better be in agreement. When only one method is used, there is no way to cross-check the results. Although this may seem like a silly analogy to use in such a serious matter, it was told to me years ago when I was a mineralogy student “When the only tool you have is a hammer, every problem is a nail.” It seems to me that this could be the case in the particle analysis business, where the only way to analyze something is to use an electron beam instrument because this is the only “tool” the commercial labs have.

Bulk vs. particle analysis: The concluding statement from the last section serves as the introduction to this section. I attended a series of talks about WTC dust in 2002. Many of the speakers talked about their sample filters being “overloaded” and hard to analyze. I had no idea what they meant and was intrigued to discover they had too much sample to analyze by electron beam methods. As a mineralogist, too much sample is a good thing; it opens the door for other types of analytical methods such as powder X-ray diffraction or “whole-rock” chemical analysis by XRF. Both of these methods require gram-scale amounts of sample. However, I think the problem is that most of the commercial labs working on these “environmental” issues have cut their teeth on mineralogical analysis by doing particle analysis with TEMs and SEMs. These are wonderful instruments and mineralogical knowledge has greatly advanced since their development, but they should be integrated into a project and not the sole instrument utilized.

Another major advantage of these bulk methods is they are much less subjective. For instance, in particle analysis particles are counted in a small amount of material and these values are extrapolated to larger volumes. In the case of slag wool fibers, only 0.01 to 0.05 grams of samples were analyzed, from samples sizes that ranged from 10 grams and higher. The fiber counts ranged from 0 to 450 (Table 5, p 58). These values are then extrapolated to fibers/gram and such values as non-detect to 1,6200,000 fibers/gram appear in Table 4 (p 56). Thus, the whole is described in terms of only a small portion of the sample. We in the geological community do not really use these point-counting methods any longer if we have sufficiently large samples to perform other types of analysis. There is no doubt in my mind that bulk methods would be very useful to fingerprint the WTC dust. Once done, the bulk methods could complement and be integrated into particle methods and make a more convincing argument. It appears to me

