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

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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 sat 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 PM_{10} 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

6.0 References

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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 by 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 ±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 ±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 0.5µm. 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 Lowers[1] and Meeker[2] 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 protocols[3]? 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 known[5] 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's[6] 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*] 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 Meeker[2] 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 report[4] (p 6 section I) and in ancillary documents[7] (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.

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 then 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 Meeker[2] Figure 9 is this a burden of documentation that is un-necessary? 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 occupies 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

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) then 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 be can 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 protocol[24] 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 debate[11] 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 team[22] Meeker also proposed a multi-tiered approach in his conclusions[2] "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 conformation 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 present[5] 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's[6] 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 huge[25].

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:

1 What are the ambient levels[5] 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 studied[9] 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 addressed[6] 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 concerns[12]

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 project[23]

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 ASTM[16] 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 then 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 Method[26]? 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 uniformity[27] 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 others[28] 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

screening method, then the expected was realized SEM has limited utility[29] for analyzing Chrysotile asbestos fibrils SEM also has to slog through time-consuming EDS analysis of hundreds (even thousands[30]) 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

- Meeker[2] sought to utilize "routine analytical techniques". The proposed method may not have evolved in that direction

- 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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22- US EPA June 30, 2005. Draft Final Plan Proposed Sampling Program to Determine Extent of WTC Impacts to the Indoor Environment p9-12

23- US EPA June 30, 2005. Draft Final Plan Proposed Sampling Program to Determine Extent of WTC Impacts to the Indoor Environment p10-11

24- US EPA June 30, 2005 Draft Final Plan Proposed Sampling Program to Determine Extent of WTC Impacts to the Indoor Environment P24

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26- Emmons, R C American Mineralogist, 13, 504-514 (1929? And 14, 415-426 (1930?)

27- US EPA June 30, 2005 Draft Final Plan. Proposed Sampling Program to Determine Extent of WTC Impacts to the Indoor Environment. P22

28- Conversations with Dr James Millette in 2002 and 2003

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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 you 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 it 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 form 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 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

that the only reason to use these particle analysis methods *only* is that they are what is routinely used by commercial environmental labs

Approaching multi-faceted analytical issues Lastly, I think an issue like WTC dust is a national concern and should be treated as such, and bring to bear the scientific resources of our entire country. If another such incident occurs, and one might be occurring currently in New Orleans, the federal government should attempt to tap the intellectual capital that exits in the USA, especially among university-based researchers This is not to belittle the government labs, there are many fine people in these organizations (e g , Meeker and company at USGS) who have risen to the call to help with these disasters My statement is also not intended to be self-serving, but to point out that academicians are much more flexible to pursue research projects and do not fear any political fallout from their results. Also, academicians are often experts in certain fields based on decades of education and self-selecting research, while scientists at government labs are often thrown into projects of which they have no in-depth background For instance, in issues surrounding Libby, Montana, I worked with some individuals whose backgrounds were in organic chemistry While they were competent scientists, they had no background in mineralogy I could not imagine if I were thrown into some issue that would require me to have an in-depth knowledge of the nomenclature and analytical methods used in organic chemistry! I propose that EPA, USGS, and other government agencies work to set up a program with the academic community to bring to bear their expertise to work in concert with the government labs on these important issues I am sure many faculty, and their students, would be very interested in working on such issues of societal importance

PRELIMINARY COMMENTS
FROM

ERNEST E. McCONNELL, Ph.D.

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7 September 2005

To Eastern Research Group, Inc

Subj Determination of a Diagnostic Signature for World Trade Center Dust using Scanning Electron Microscopy Point Counting Techniques

I have reviewed the subject document and offer the following comments per the charge

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), wet for long periods,
c) They are sufficiently homogeneous in WRC 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 samples

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

Answer. The criteria appear reasonable and adequate to identify a signature WTC component that will differentiate it from background dust

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 WRC marker? Please explain your answer

Answer From the material provided, the selection of slag wool should be suitable for most cases. None of the other possibilities are as good and using them would be redundant in most cases However, I would like to point out that slag wool is somewhat soluble in physiological saline and biological environments While most of this solubility data has been derived to

answer biological questions, e.g how long slag wool fibers persist in the lung after inhalation, the data probably have some relevance to the WTC analysis For example, in in vitro systems for solubility, slag wool dissolves at a rate of 119± 41 ng/cm2/hr (IARC, 2002). In the lung slag wool also disappears over a relatively short period of time, albeit this is also a function of breakage and physiological removal in addition to dissolution For example, the reported half-life of fibers >20 um L in the lung is only 8 1 days (Bernstein et al , 1996) Therefore, it is imperative that the EPA (or others) establish how stable slag wool is in damp and wet environments and factor this into its sampling protocol and evaluation of the findings While this does not dissuade me from agreeing to the use of slag wool as a signature mineral, it needs to be factored into the evaluation of a given dust sample, i e was it from a wet environment? At a minimum, the EPA, etc should do a solubility study of pure slag wool and WTC dust containing slag wool to see the impact of a water environment, i e does it dissolve and if so, how rapidly? If such studies showed that slag wool dissolves, then one might want to use a different endpoint, e g cement, as the signature material in cases where the dust was subjected to water for some period of time The problem of relative solubility should be answered prior to establishing a sample analysis protocol

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 The methods are written in enough detail and should easily be adopted by a testing laboratory This is not a difficult analysis to conduct However, I question the need for scanning electron microscopy (SEM) to identify the slag wool All of the man-made vitreous fibers (MVF) of concern (slag wool, rock wool and fiberglass) are visible in the light microscopy (LM) range, but just as importantly, there is a minimal amount of these fibers in the submicroscopic range (<0 25 um) Finally, as noted in 12 1 (pg 37) of the Final Report, the refractive index using polarized LM can differentiate mineral wool fibers (slag and rock wool) from other MVFs. An option that might be worth pursuing is to first examine a given dust sample with polarized LM using a refractive index cutoff of 1 55 If the number of these fibers doesn't meet the "cut-off" criterion for a WTC dust sample, one could stop there. If it exceeds the level of concern, then it could be further analyzed with SEM to determine whether it is rock wool or slag wool or some other MVF But, considering the objectives of the dust analysis, SEM may still be "overkill" even in this case. The use of LM alone would certainly cut down on the amount of time required and significantly reduce the cost

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: See above comment I would add, however, that evaluations of this import should only be conducted by a highly qualified laboratory and should always be validated by the use of positive and negative control samples (blind).

Ernest McConnell

Method Validation Study:

Q5) The method validation study design entailed spiking characterized background dust with characterized WRC 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 (5 commercial laboratories and 3 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 This is a standard and acceptable approach However, I was surprised by how much difference there was in the results among the various labs One explanation that was provided was that the mixing may not have been adequate Since this was such a critical part of the protocol, it is perplexing why this simple procedure would have been a problem

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

Answer I think the results were properly interpreted However, before determining the "final cut-off" for the number of fibers to be used as the level to identify a WTC dust sample, the Agency may want to revisit this data and censor the data from the labs that are obvious outliers as was done by Versar, rather than using an average of all of the labs

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 markers (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

Answer. It is apparent that there are differences in the WTC dust and background dust It also seems reasonable to use slag wool as a signature component However, I think the "cut-off" level needs to be better substantiated, especially in light of the findings of the interlaboratory pilot study This can be addressed to some degree by using a conservative number In addition, one might select one number to identify environments that require radical clean-up but a different number for minor amelioration Finally, the issue of slag wool solubility as noted above, and its effect on potential fiber numbers needs to be considered

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 It is imperative that the "sampling person" clearly identify any preexisting source of slag wool This was clearly identified as a possible problem by analysis data from the EPA Research Triangle Park samples which showed high levels of slag wool due to natural contamination from their ceiling tiles

Has any effort been made to see if there is a simple chemistry method that would identify a signature marker? For example, if cement is the major component of WTC dust, there would be a significant level of calcium in such a sample. Slag wool is also calcium rich (38%) Would calcium be in, or as high in background dust? If calcium levels qualify as a signature marker, this methodology would certainly be a much more efficient way of answering the two primary purposes of the survey, i.e 1) determine the geographic extent of the remaining WTC dust, and 2) determine the need for clean-up. A cut-off level of calcium could be established as was done for slag wool.

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

Answer I have one additional comment The Final Report (Tbl 1, pg 14) suggests that fiber number will be used to establish the level of concern In my experience the "total" fiber number of MVFs can be misleading unless one knows the length distribution For example, the number of fibers in a given mass of dust would be much higher if the fibers are short than one where they are short This may be important in a scenario such as the WTC because the large fibers would tend to settle out sooner than the short ones, even though the total numbers may not be that much different For this reason I think it would be better to us the mass of slag wool rather than fiber numbers per gm of dust This could be expressed as a percent of the dust or ppm as has been done in the USGS document (Tbl 1).

References:

Bernstein et al (1996) Evaluation of soluble fibers using the inhalation biopersistence model, a nine-fiber comparison Inhal. Toxicol. 8.345-385

IARC (2002) Man-made Vitreous Fibres IARC Monographs on the Evaluation of Carcinogenic Risks to Humans (Vol 81). International Agency for Research on Cancer (World Health Organization), Lyon, France

PRELIMINARY COMMENTS
FROM

SHU-CHUN SU, Ph.D.

COMMENTS ON FINAL REPORT ON THE WORLD TRADE CENTER (WTC) DUST SCREENING STUDY (CONTRACT NO. 68-C-02-060 TASK ORDER NO. 107)

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September 23, 2005

Summary

The validity of the key analytical results (number of slag wool fiber per gram of dust) is highly questionable because of

- the lack of any standard for evaluating the accuracy of results.
- the incorrect equation used for the calculation of final results as a result of the ignoring of slag fiber depletion through sieving and the required one-minute coarse particle precipitation during sample preparation;
- the unproven reliability of differentiating the signature constituent slag wool from the interfering constituent rock wool basing on the detection of low concentration of Fe by energy dispersive spectroscopy analysis with scanning electron microscopy, and
- the huge extrapolation factors of 5,000 to 10,000 for calculating the final results for 1 gram of samples from the slag wool fiber count obtained from the measurements of 0 0001 to 0 0002 gram of samples

Therefore, the conclusions in the Final Report need to be re-evaluated when valid analytical data are available

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

a) Yes

b) Yes, if not significantly diluted by urban dusts

c) Yes However, the homogeneity of signature constituents needs to be clarified and better defined If it means only the concentration, it should be expanded to include also the chemistry and

physical properties used by the analytical protocol to identify and differentiate them from interference constituents. Otherwise, if those diagnostic properties vary too much reliable identification will be rather difficult, resulting in unacceptable rates of false negatives and/or false positives The reason that I emphasized the importance of the homogeneity of chemical and physical properties was because I noticed the limited effort to characterize the chemistry of SW (slag wool) and other interfering MMVF constituents and the complete lack of effort to characterize their optical properties, such as refractive indices, during the initial phase of the method development. It was not clear that besides the 3 SW EDS (energy dispersive X-ray spectroscopy) spectra presented in the ATLAS[1] how many more SW and RW (rock wool) fibers have been analyzed to determine their respective variation ranges of Fe, which is the key element used to distinguish SW from RW by the analytical protocol, using a more accurate and reliable technique than EDS. I will discuss this issue in more details later

d) This is not a well-defined question There is no way one can answer this question without knowing

- how small a sample size is considered to be small? x micrograms, x milligrams, or x grams?
- how low a minimum detection limit is considered to be low? 100 ppm, 1%, or 10,000 fibers per gram?
- what does it mean by "low interference"? The number of interference constituents? The concentration of interference constituents? The degree of difficulty to differentiate interference constituents from the signature component(s)?

I believed that two more criteria for signature constituents should be added

e) The variations of their chemical and physical properties are within boundaries that can be clearly defined and reliably differentiated from those of potential interference constituents at a predetermined confidence level (95%, 99%, 99.9%, etc.)

f) Standards of various concentrations can be created using the pure signature constituents for assessing if the analytical results can meet a predetermined MQO (Measurment Quality Objective), such as ±30%.

I consider that the flaws I found in the WTC dust screening method development and validation studies, which will de discussed in greater details, are partially due to the failure in the recognition of the above two criteria

[1] Lowers, H A , Meeker, G P , I K Brownfield (2005) World Trade Center Dust Particle Atlas U S Geological Survey Open-File Report 2005-1165

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?

The current study and results only show that SW (slag wool) MIGHT BE a good candidate to meet the first 3 criteria

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?

The FR[2] seems to have already provided a clear answer to this question Among the five commercial laboratories contracted for this study. RJ Lee. MVA, EMSL, MAS, and Reservoir, EPA determined that three of them (Labs E, F. and G) had failed to produce data of acceptable quality

It can be assumed that there must have been a rigorous process in selecting contract laboratories for this important study All five laboratories must have been considered to be highly qualifying ones among many environmental and analytical laboratories in the U.S , which have not only PLM (polarized light microscopy) but also SEM (scanning electron microscopy) and EDS capabilities

It is an alarming fact that 60% of highly qualifying commercial laboratories selected by EPA for this important study have failed to produce valid results

It is equally alarming that Q3 seems to correlate the inability to follow the analytical protocol and produce valid results ONLY with the insufficient detail of the documented analytical methodology as if had the analytical protocol been documented with more details the high rate of failure would not have happened

There could be other reasons for a laboratory to fail to produce valid results besides the insufficient detail of the documented analytical methodology.

- the lack of competence of the designated analyst(s) in performing the written analytical methods.
- some inherited flaws in the analytical protocol itself, or
- everything above

But I certainly could not agree that "*some labs did not have the personnel or the equipment to perform the required analysis in the given timeframe*" (p 13 of FR) was one of the reasons It is a well-known fact that EPA would not have selected a laboratory without the required equipment (PLM. SEM. and EDS) and the EXCEL data files provided by EPA for all 8 laboratories, including Labs E F. and G, showed that every laboratory has the required equipment. The lack of personnel should not have been an issue either. Any laboratory that had been awarded this important EPA contract would have to fulfill its obligations by committing adequate personnel to meet any deadline set by EPA.

[2] USEPA et al (2005) Final Report on the World Trade Center (WTC) Dust Screening Method Study 72pp

Since Q3 also emphasizes "*without supplemental assistance from EPA or other sources,*" let's not forget the fact that "*Each laboratory has attended a two day session during which the method was further developed and discussed, and procedures to adapt the method to suit each laboratory's equipment were determined*" (p 26. PSP[3]) and "*weekly conference calls will be held with EPA, the prime contractor and all subcontractors to assess progress and discuss any issues or problems that may have arisen*" (p 18. QAPP[4]). Again, if any laboratory reported a personnel issue during a weekly conference call it would have been immediately and properly addressed Therefore, it can be concluded that all five commercial laboratories had been assisted and supported by EPA all along the sample analysis and the lack of personnel or equipment had never been an issue

All available facts documented in the FR seem to strongly indicate that ***there is at least a greater than 50% chance that a qualifying laboratory will not be able to 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?

While the detail of the documented methodology is important, it is probably more important that the analytical procedures should not contain steps that can't be easily followed in a consistent manner either by the same analyst or different analysts This issue is of special importance to this project because the analytical protocol extrapolates the SW fiber counts obtained from 0 0001 to 0 0002 g sample on a single SEM stub to SW fiber counts in 1 g of sample. The preparation of SEM stubs with accurate sample loading directly affects the accuracy of the final analytical results

For example, Section 11 5 (Preparation of Sample for SEM Analysis) of Appendix D[5] reads "*Prepare a 10-fold dilution of the above suspension to get a suspension of 1-2 mg dust per mL of isopropanol Sonicate the suspension in an ultrasonic bath for one minute Remove the suspension and gently shake it to suspend all particles Wait one minute to allow the coarse particles to settle Collect a 10-µL aliquot of the suspended mixture using an Eppendorf pipette with the modified tip and transfer to a prepared polycarbonate/adhesive tab substrate* "

Any method developer with experience dealing with particle suspensions would avoid steps like "*wait one minute*" because practical experiences have shown that it can't be carried out with consistency

[3] USEPA (2005) Proposed Sampling Program to Determine Extent of World Trade Center Impacts to the Indoor Environment (Provided to the Review Panel as a background document)
[4] USEPA (2005) Final Report on the World Trade Center (WTC) Dust Screening Method Study Appendix A Quality Assurance Project Plan For The World Trade Center (WTC) Screening Method Study
[5] USEPA (2005) Final Report on the World Trade Center (WTC) Dust Screening Method Study Appendix D Protocol Used for the Screening Method Study

to ensure a reproducible particle loading whether by the same analyst or different analysts Even a few seconds difference, e g , 2 seconds vs 10 seconds after the one-minute waiting period, in collecting the aliquot with a pipette may result in different particle loadings that are significantly enough to affect accurate calculation of the sample weight on the SEM stub Another inherited uncertainty of this procedure is that the exact position of the pipette tip in the liquid column will also affect the amount of particles in the 10-μL aliquot because there is a particle concentration gradient from the top to the bottom

What makes the execution of the analytical procedures more complicated is that there is another step of aliquot extraction from the initial suspension to prepare the said 10-fold diluted suspension The method developers assumed that the sample loading in each aliquot could be accurately calculated from the ratio of the aliquot volume to the volume of the sampled suspension. In ideal world such assumption is undeniably correct In real world, however, such assumption should not be taken for granted and must be rigorously tested and verified so that the variability of sample weight on sample stub can be determined and used to refine the final result calculation

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?

This would have been an appropriate approach if appropriated standards with known concentrations of SW are also analyzed by each participating laboratory to determine whether the ±30% accuracy objective has been achieved. One of the critical flaws of this study was the lack of any standard Therefore, the MQO for accuracy (±30%) stated in A7 2 of QAPP have been completely ignored by the study The Project Management should have caught this nonconformance with QAPP and taken appropriate corrective action to address this issue of utmost importance

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.

I disagreed

1) The evaluation and interpretation as well as conclusions presented in the FR were all based on two assumptions.

- the SWG (SW Fiber count per gram of dust) data of the "Very Good" Group (Labs A, B. C D. and H) were valid and could be used to reliably validate the analytical protocol.

– the SWG data of the "Outlying" Group (Labs E, F. and G) were invalid and should be discarded because they didn't fit with the results of the "Very Good" Group

Are we sure that the SWG data of the "Very Good" Group are valid?

Even though QAPP has detailed procedures for quality control (B5. p.16), data validation (D, p 19), etc , it seems that there have been no serious effort to check whether the equation used to calculate SWG was correct and all the assumptions adopted by the analytical protocol were valid

The following equation is from Section 13 0 (Data Analysis and Calculations) of Appendix D

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

Because the FR has not presented an equation for calculating the fiber counts based on SEM/EDS analysis, I guessed that Fibers slag wool/g on slide in the equation can be replaced with Fibers slag wool/g on SEM stub

This equation implies at least two assumptions

a) all SW fibers would be in the fraction < 150 μm after sieving Since it is a given fact that there are SW and other MMVF fibers longer than 150 μm and sieving has never been a very effective way to separate fibrous material by their length, there must be SW and other MMVF fibers not only longer but also shorter than 150 μm in the coarse fraction Therefore, this implied assumption and the above equation can't be correct Because all calculations of the SW fiber count results were based on this incorrect equation. the validity of all SW fiber results presented in the FR is highly questionable even though the SW fiber count ratios among the 1%, 5%, and 10% spiking samples showed a marginally reasonable linear relationship for the five "Very Good" laboratories

Had this factor. i.e , *the ratio of SWG in >150 μm fraction to SWG in <150 μm fraction*, been a constant for every single one of the 20 background samples and 12 spiked samples prepared by EPA. there would be no need to make any correction to the existing SWG data Such a possibility is most likely not true

b) all SW fibers would remain in the diluted suspension after letting coarse particle precipitate for one minute during SEM sample preparation because there is no correction factor in the above equation to address the depletion of SW fibers in the suspension after the one minute precipitation period It can be safely concluded that the coarse fraction at the bottom of glass vial contains also SW fibers If the

depletion of SW fibers in the suspension due to the precipitation during the one-minute waiting period is not factored in the calculation of SWG, the results could not be valid

Had this factor, i e , *the ratio of SWG in precipitated fraction to SWG in suspension fraction*, been a constant not only for different analysts but also for different laboratories, there would be no need to make any correction to the existing SWG data Again, such a possibility is most likely not true

Now, let's take a look at the actual data presented in the FR

Table 1. Comparison of the observed and theoretical SWG data of the "Very Good" Group

Spiking Level	Theoretical SWG*	Observed SWG**	Difference%	Ratio to $1\%_{OLs}$
1%	5797	17265	198	-
5%	28983	52507	81	3 0
10%	57967	88543	53	5 1

* 579667 for 4 Albany pure dust from Appendix F[6].
** Table 4 of Appendix E[7]

The above comparison indicates that there are huge differences between the observed and the predicted results and that the ratios of $(5\%/1\%)_{Observed}$ and $(10\%/1\%)_{Observed}$ are also far from the predicted 5 and 10 Were the above discrepancies considered to be acceptable and in conformance with the interlaboratory MQOs (Table 2, QAPP)? If not, why were there no corrective actions?

2) BIR-1G SEM/EDS data precision analysis

Versar concluded that "*each of the laboratories was easily able to achieve excellent precision, by SEM/EDX, in quantifying the elements that were present in larger concentrations*"[8] based on the average elemental concentration data reported by five commercial laboratories. It indicated that each laboratory had analyzed the standard between 4 and 11 times and the average values reported by them were based on 4 to 11 individual analyses, respectively The proper way of assessing the SEM/EDS precision for BIR-1G is to calculate the estimated standard deviation or coefficient of variation from all individual measurement results instead of the 3 mean values because analysts did not analyze each MMFV 4 to 11 times.

On the other hand, the above conclusion about "*elements that were present in larger concentration*" is irrelevant to the current analytical protocol because the identification of SW and its differentiation of from RW are based on determining Fe at LOW concentration levels (Fig 1 on the next page) As indicated in the FR, the FeO%wt range is 0 - 2 for SW and 3 - 12 for RW What is important is not the precision of the high concentration elements (Si, Al, Mg, Ca, etc) but the precision of Fe at

[6] USEPA (2005) Final Report on the World Trade Center (WTC) Dust Screening Method Study Appendix F Statistical Analysis and Interpretation of Test Results – Laboratory Qualification
[7] USEPA (2005) Final Report on the World Trade Center (WTC) Dust Screening Method Study Appendix E Report from U S EPA Contractor on the Screening Method Study
[8] Addendum to Appendix E SEM Calibration Data

very low concentration level. The selection of BIR-1G as the standard for EDS precision assessment is therefore not adequate if SW is the only signature constituent to be used for screening WTC dust from background dust because of BIR-1G's high concentration levels of FeO (8.34%) and Fe_2O_3 (2.06%)[9]. A proper standard should have FeO+ Fe_2O_3 (EDS can not differentiate Fe^{2+} from Fe^{3+}) concentration no more than 5%wt. An additional standard with FeO+ Fe_2O_3 around 1%wt will also be helpful.

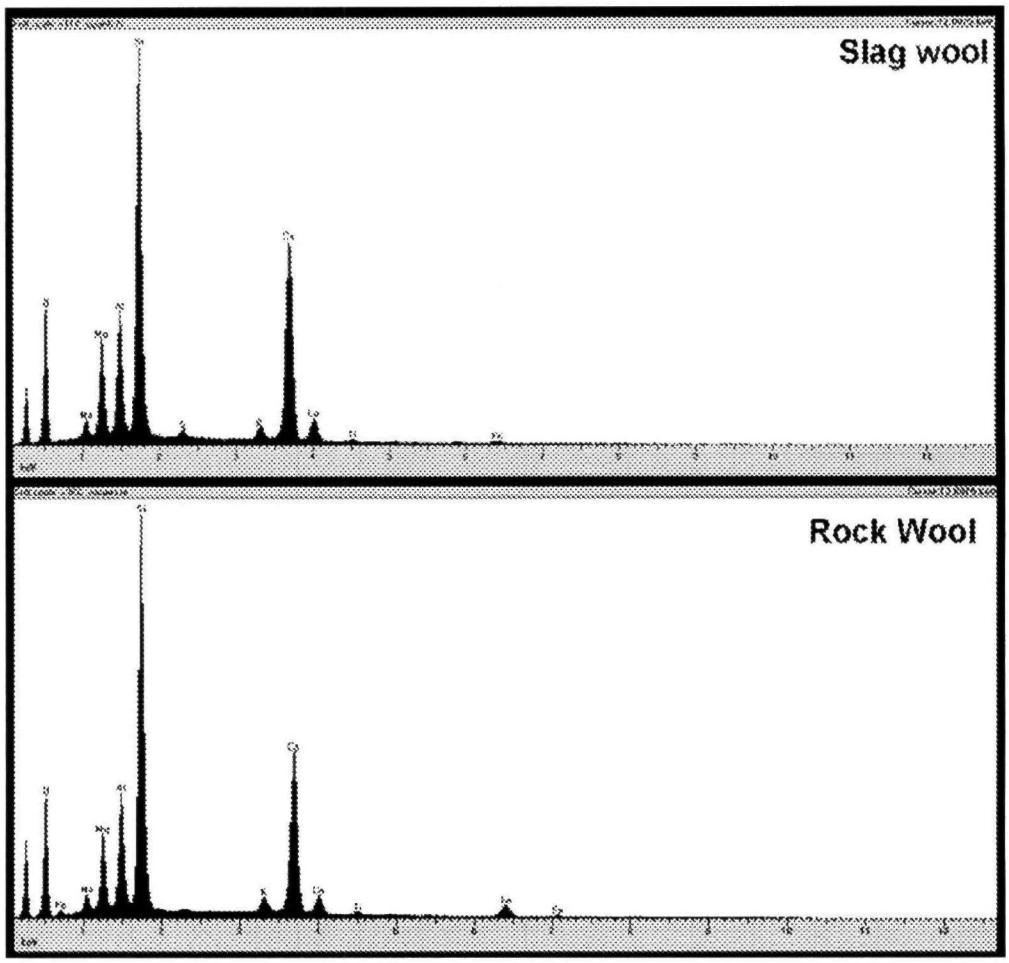

Fig. 1. The comparison of EDS spectra of SW and RW (ATLAS: SLAGWOOL-02.TIF and ROCKWOOL-02.TIF)

[9] Smith, DB (1998) USGS Certificate of Analysis: Icelandic Basalt, BIR-1. http://minerals.cr.usgs.gov/geochem/icelandic.pdf

Q7) EPA has observed differences in slag wool concentration that 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.

No, because such an observation would be valid only if the SWG results were valid As shown by my comments for Q6, I am not convinced that the SWG results used to form this observation are valid

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?

I don't know

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

1) The reliability of differentiating the SW from the interfering RW basing on the detection of low concentration of Fe by SEM/EDS

EPA relied on the generalized data provided by TIMA[10] for the chemical and physical properties of MMVF fibers in WTC dusts and believed that SEM/EDS could be used to reliably differentiate SW whose FeO varies from 0 to 2%wt. from RW whose FeO varies between 3 to 12%wt Instead of conducting a comprehensive study of the compositional variations of MMVFs. especially SW and RW. in WTC and background dusts to determine their actual elemental compositions. EPA based its method development work on the above FeO concentration data provided in a single TIMA document I believed that this was a critical flaw of the method development

Unlike those strictly formulated products. neither SW fibers nor RW fibers are known for the stability of their chemical and physical properties because the diversity of the raw materials and the not-so-stringent specifications of the final products The data in the table[11] on the next page indicates that RW's Fe_2O_3 content could be as low as 0.30% If RW's Fe_2O_3 content could be this low, Fe is no longer a reliable diagnostic element for SW-RW differentiation This example shows that there might be a possibility of overlapping FeO%wt ranges for SW and RW. resulting either false negatives or false positives in SW counts

There should be a very rigorous and comprehensive study of the compositional variation of MMVFs in WTC and background dusts, especially SW and RW fibers. to establish the variation range of Fe content at the very beginning of method development Many polished sections of dust samples should be prepared and analyzed by WDS (wavelength dispersive spectroscopy) using a microprobe so that the

[10] Thermal Insulation Management Association, 1991, Nomenclature of Man-Made Vitreous Fibers TIMA Inc 72p
[11] Commission on Life Sciences (2000)Review of the U S Navy's Exposure Standard for Manufactured Vitreous Fibers http //www nap edu/books/0309070937/html/15 html

ACTUAL chemical compositions of various MMVF fibers in WTC dusts can be accurately determined and the variation ranges of Fe, the key element used to differentiate SW fibers from RW fibers, can be accurately established These data are indispensable to determine whether Fe can be used to reliably differentiate SW from RW at a predetermined confidence level

TABLE 2-1 Typical Chemical Composition of Some Commercial MVF[a]

	Composition (%)									
Name[b]	11	A	C	21	F	G	22	RCF-1	X-607	Insofrax
Class	Glass	Glass	Glass	Rock	Rock	Rock	Slag	RCF	RCF sub [c]	RCF sub [c]
Components										
SiO_2	63 40	65 00	61 70	46 30	56 30	60 10	38 40	47 70	58 30	76 20
Fe_2O_3	0 30	0 10	0 10	13 20	**0.30**	6 10	0 00	1 00	0 10	0 30
TiO_2	0 06	0 02	0 02	2 60	0 10	0 05	0 50	2 10	0 05	0 08
Al_2O_3	3 90	1 90	1 00	13 50	3 20	0 40	10 60	48 00	1 30	1 40
CaO	7 40	7 40	7 20	10 00	26 10	18 80	38 00	0 07	38 70	0 20
MgO	2 80	2 60	2 90	9 10	6 40	8 30	9 90	0 08	0 40	21 50
Na_2O	15 40	16 10	16 10	3 10	3 20	5 50	0 40	0 00	0 30	0 07
K_2O	1 30	0 70	0 60	1 40	0 70	0 20	0 50	0 20	0 10	0 10
B_2O_2	4 50	4 70	9 20	0 00	0 00	0 00	0 00	0 01	0 00	0 00
P_2O_5	0 00	1 10	1 10	0 40	2 90	0 08	0 00	0 10	0 40	0 03
SO_3	0 30	0 03	0 20	0 00	0 00	0 05	1 80	0 00	0 00	0 00
Cr_2O_3	0 00	0 00	0 00	0 04	0 00	0 00	0 00	0 03	0 00	0 00
MnO	0 01	0 00	0 01	0 20	0 00	0 00	0 70	0 00	0 00	0 01
ZrO_2	0 00	0 00	0 00	0 00	0 00	0 00	0 00	0 10	0 00	0 00
Total	99 40	99 60	100 00	99 80	99 10	99 50	100 80	99 40	99 30	99 90

[a]Data derived from Bernstein et al (1996), Maxim et al (1999a), and McConnell et al (1994, 1995, and 1999), Material Safety Data sheets for Isofrax fibers from the Unifrax Corporation, Niagara Falls, NY
[b]Name 11, Certain Teed B glass wool fiber, A, new glass wool, C, new glass wool, 21, rock wool, F, rock wool, G, rock wool, 22, slag wool, RCF-1 - kaolin-based refractory ceramic fiber, X-607, rock wool produced by Unifrax, Isofrax, refractory ceramic fiber
[c]Substituted RCF

Another practical concern is the criteria used by SEM analyst to differentiate SW EDS spectrum from that of RW during routine analysis As shown in Fig 1 which is entirely based on EPA's data, how could an analyst make a reliable identification simply from the visual inspection of the Fe peak height?

2) Problematic sampling procedures

- the result of a single 12 mm SEM stub with 10 to 20 μg of fine dust samples was used to determine the total SW fiber counts in one gram of dust samples The extrapolation factor was too high (5,000 to 10,000) to ensure the accuracy of the final SW fiber counts The data in Table 1 clearly show the effect of this excessive extrapolation the error propagation is most pronounced for low concentration samples

- the sample preparation procedure contains two steps of aliquot sampling the first one was from the initial dust suspension and the second one was from the diluted dust suspension As discussed previously, the accuracy of dust weight calculation based on the volumes of aliquots is highly questionable

– the "*wait one minute*" problem as discussed in the comments for Q4

3) The PLM procedure has been ruled out as a feasible method for SW fiber quantification. I consider that this conclusion is premature and should be re-examined because if the fundamental flaws[12] in the method development of the PLM procedure can be corrected there might be a possibility that PLM could be a more accurate, efficient, and cost-effective technique than SEM/EDS. However, this could not be accomplished if the method development team has no experts with strong background in optical crystallography, optical mineralogy, stereology, and automated image processing and analysis

[12] The discussion of this topic could be another lengthy review

PRELIMINARY COMMENTS
FROM

JAMES S. WEBBER, Ph.D.

Peer Review of

Final Report on the World Trade Center (WTC) Dust Screening Method (August 17, 2005)

Prepared by

James S. Webber, PhD

September 23, 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 all 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

While rather broad, I agree that these criteria should be adequate for establishing a WTC signature marker

However, the use of *low minimum detection limit* invites controversy In a strict analytical sense, *minimum detection limit* is predicated on a level of acceptable uncertainty, or precision To many microscopists, however, *detection limit* refers to a microscope's capability to detect a single particle of interest in a field of other particles The difference between the two interpretations is usually several orders of magnitude because, despite the extreme sensitivity of the microscope, the reproducibility is often poor because of non-uniform particle distribution and analyst subjectivity, among other factors The poor precision of the proposed method is discussed later

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.

While I concur with the Final Report's conclusion that gypsum and concrete dust are too ubiquitous to be useful fingerprints, I disagree with the conclusion that slag wool alone meets the criteria as a WTC marker Specifically, **Q1)a)** requires a marker to be *unique to WTC dust* Slag wool is a common building material which can be expected near any building demolition site, near major renovations, or even where a workman's ladder has brushed ceiling tiles or insulation The second paragraph on page 9 points to an overlap of slag wool (around the 100,000 fibers/gram concentration) in samples from both the impacted and background areas The elimination of NJ and LI "outliers" from Table 1 (page 14) is another indicator of slag wool's inadequacy as a solo marker Simply put, false positives are guaranteed because of slag wool's broad application in past and present building products, as acknowledged twice (*likely that false positive results*) in the report, on the last sentence on page 12 and again in the final sentence before the Final Report's Conclusions on page 21

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?*

On the basis of the poor interlaboratory results (evidenced by the disqualification of three of eight (almost half!) of the laboratories), I'm skeptical that the analytical methods applied as-written by a typical laboratory would produce valid results The five surviving laboratories are laboratories with stellar national and international reputations resulting from substantial experience in non-routine research projects Yet the high inter-laboratory relative standard deviations (RSD) from these elite laboratories on page 20 indicate how difficult it will be to derive meaningful results if there is a need for newly initiated laboratories Despite carefully controlled conditions during the study, poor reproducibility was ascribed (bottom of page 20) to possible non-homogeneity of prepared samples and operator experience This poor precision would indicate violation of **Q1)d)**'s requirement of *low minimum detection limit* The acknowledgment that *the protocol was adapted to suit each laboratory's equipment* (top of page 10) and the need for *weekly conference calls* (top of page 49) by laboratory participants adds to my doubt that this protocol will be uniformly adoptable.

Of course, the term *qualifying laboratory* in **Q3** could be the caveat by which analyses are limited to a select cadre of laboratories If this term is applied to laboratories that are winnowed out in a process similar to that used to eliminate three of the eight original laboratories, results from real-world samples might approach the quality of this inter-laboratory study

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?

Two SEM preparation methods are allowed in §11.5. 1) the drop method in the 3rd paragraph of page 36 and 2) the filtration method in the 4th paragraph I would strongly recommend that the drop method be eliminated because of the likelihood of uneven particle distribution caused by surface tension and other physical anomalies of the drop. Filtration invariably produces a more uniform particle distribution than produced by a drop mount. That being said, I would further recommend that the filtration set-up in that section be revised to prevent bubble (void) formations on the filter The filtration apparatus should be assembled dry and vacuum applied *before* introducing the isopropanol suspension See precautions in Section 6 3 2 of EPA Method 100 1 (Chatfield, 1984)

The method requires that the sample be ashed, with gravimetric tracking, to remove organic materials The use of this data is not explained WTC dust was primarily inorganic in nature, which would imply that a high concentration of organic material in a field sample represents substantial dilution by local dusts Unfortunately, this was not addressed in the 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?*

The method validation study design was appropriate for determining the suitability of a WTC dust marker. The results, however, as detailed in my response to **Q6** below, did not convincingly demonstrate that suitability for slag wool

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 sample 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.

The fact that a linear regression fit the spiked samples' concentrations versus results reported by the commercial laboratories was promising in that a linear relationship was expected between these spike levels. Furthermore, using a Standard-Additions approach, extrapolation of the *Spiked Samples - Albany "Best" Group Combined* sample regression (top figure on page 65) to the *% spike level* baseline yields approximately (–)1.2%, which is equivalent to 7000 fibers per gram in the background dust This is equivalent to the 7194 fibers per gram of the NE Queens background material (from Table 3, page 55)

However, the need to eliminate various laboratories to achieve this fit is troubling Eliminating three of eight (37%) commercial laboratories for poor performance raises questions about the universal applicability of the method - I would assume that the eight laboratories chosen initially were neither bargain-basement laboratories nor chosen at random from the yellow pages. The additional temporary elimination of another of the surviving laboratories (H in several figures) increases concern about the rigor of the method

Only laboratories A & B performed well with the USGS spikes, raising further doubts about the effectiveness of the method to distinguish a WTC signature Convincing demonstration of the method's suitability for widespread use would have been inter-laboratory regressions that matched the USGS results presented in Figure 3, where the best-fit regression line intercepted near zero.

As to *reasonably low false positive error rate*, I reiterate my conclusions under **Q2)** they are a given

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.

The elimination of so many background samples from statistical consideration is vexing Appendix E, Table 3 (pages 54-55) lists 28 background samples Six of these, or 21%, contain greater than the landmark 100,000 fibers per gram With these samples included, RSD for slag wool exceeds 150%, which effectively runs confidence limits from (less than) zero to well over the average for 4 Albany. In other words, background samples would be statistically indistinguishable from WTC samples The dismissal of the high-fiber background samples from the study portends problems in the real world how will an analyst or decision maker know *a priori* which high-fiber samples are simply from high background?

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

Method development should have evaluated measurements of fiber lengths and widths – WTC fibers are probably smaller than background fibers because the impact energy of the WTC collapse shattered many Additionally, any indoor WTC dust will probably contain smaller slag wool fibers than outdoor WTC dust because of the removal of larger fibers by settling during dust's passage from outdoor to indoor environments, as hinted in the last sentence on page 11 It would be worthwhile to compare fiber dimensions of USGS (outside) WTC slag wool to fiber dimensions of 4 Albany (inside) slag wool

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

I am obviously not persuaded that the proposed method's limitation of a WTC marker to slag wool will be effective in determining WTC dust boundaries The convoluted disqualification of laboratories and elimination of various background samples demonstrates the tenuous nature of this simplistic approach. Below I make several cases for possible improvement of a WTC dust signature

The Case for More Conditions on Slag Wool Analysis

A slag-wool-only marker would be useful in determining extent of WTC plume only with several additional conditions

1) Investigate the particle-size distribution of slag wool in WTC dust If differences are measured between these fibers and those in background fibers, better discrimination could be made
2) When positive results are reported, investigate the surroundings of the sample's origin for potential contamination, e g., slag wool in building materials or demolition of nearby structures
3) Participating laboratories would have to be screened with excruciating care Qualifying criteria would be similar to those that culled out almost half of the laboratories in the original study
4) Design the survey with an overload (>25%) of QC Mix large numbers of samples with spikes and known concentrations with the field samples to track the laboratory's (and method's) performance in real time

The Case for Chrysotile as a WTC Dust Fingerprint

After encountering the multiple difficulties with the slag-wool-only design, I am truly puzzled by the absence of consideration of one of the most promising WTC markers. *Chrysotile* is never mentioned in the report and *asbestos* occurs only once, in a reference This despite an earlier WTC report by R J. Lee (2003) in which chrysotile was detailed as the most important constituent of a multi-component *WTC Dust Signature* Chrysotile almost certainly exceeds slag wool in meeting the marker criteria of **Q1**

Q1 a) Chrysotile is more unique to WTC dust than is slag wool Because of the ban on most uses of asbestos thirty years ago, chrysotile is no longer a ubiquitous component of dust Certainly not at the levels found in WTC dust. According to the R J Lee report,

chrysotile is at least 4 *orders of magnitude* greater in WTC dust than in background dust. Furthermore, the report continues, the chrysotile in WTC dust is often distinguishable from other chrysotile:

1) Mg/Si ratio is significantly reduced in WTC fibers
2) WTC fiber size differs from chrysotile fibers typically found in ambient dust
   a) WTC fibers are highly fibrilized (sheared apart) by the energy of the collapse, with mean width <0.1 μm.
   b) Fibers tend to be longer than in ambient dust
3) WTC fibers are often coated with residual metals (Al, Fe, Zn) released during combustion of the WTC materials

Adding to their advantage over slag wool, the generally smaller size of chrysotile fibers makes them more likely to infiltrate the indoor areas of concern

Q1 b) Chrysotile is certainly durable – it's been around for millions of years

Q1 c) Chrysotile has been detected at levels between 0 5% and 2% in original analyses of WTC dust, probably the most homogeneous distribution of chrysotile in any large-scale environmental sample

Q1 d) Chrysotile at a ~1% level would be easily detected by SEM (and certainly by TEM, as discussed below) Our TEM analysis of WTC materials in storm-sewer run-off during the first post-collapse rainfall (September 14) revealed a substantial population of fibers wide enough (>0 25 μm) for easy detection by SEM at a magnification of 4000X (At that magnification, widths on the screen would be > 1 mm) Concentrations of fibers longer than 10 μm and wider than 0 25 μm were 100 million fibers per gram debris, most of which was the fine plaster and cement particles common in WTC dust. This is about an order of magnitude greater than the slag wool concentrations

The Case for Transmission Electron Microscope Analysis of Chrysotile

I would further recommend that TEM be considered for analysis of chrysotile in dust TEM holds many advantages over SEM for analysis of asbestos

1) Detection of the narrowest chrysotile fibers is possible by TEM's superior resolution To the point, on the basis of our analysis of the run-off sample, if all chrysotile fiber sizes were included, chrysotile concentrations would be almost 20 *billion* fibers per gram This would allow use of greater dilution, which would substantially reduce the amount of obscuring material in a field of view Using Chatfield's (1984) recommended upper limit of loading, 20 $\mu g/cm^2$, a 0.001 mm^2 TEM grid opening would contain 35 chrysotile fibers

- an easy counting job with much less obscuring background than SEM analysis of the same dust

2) Standardized methods exist for preparation and analysis by TEM that would require minimum modification
3) TEM is a more standardized instrument for chrysotile analysis Differences between TEMs are less than between SEMs, which have problems in standardization of contrast, scan rates, etc There will be no need to have the protocol *adapted to suit each laboratory's equipment* (top of page 10)
4) There exists an army of several dozen certified/accredited TEM laboratories with extensive experience analyzing asbestos under conditions similar to those of the proposed protocol
5) Analysis of a fiber in a TEM preparation will probably have less interference from x-rays from other particles because of the more dispersed sample
6) If there is any question about a fiber's identity, TEM can utilize electron diffraction to unequivocally identify chrysotile SEM lacks this capability

Finally, the Case for a Stratified Approach

Because of slag wool's many shortcomings as a fingerprint, application of the suggested slag-wool-only protocol should be limited to screening. If a dust sample is negative at the 10% level as outlined in the proposed protocol, consider the dust to have originated outside the WTC dust plume On the other hand, a positive result would trigger analysis for chrysotile using TEM Quantitation of chrysotile concentrations along with qualitative characteristics of WTC chrysotile would settle the question of WTC source

This stratified approach would be only moderately more expensive because methods already exist and TEM laboratories are competitive in their pricing To paraphrase retired EPA guru, Mike Beard, "It is folly to base a million-dollar abatement project on a five dollar analysis "

References

Chatfield, E J , and M J. Dillon. 1984 Analytical Method for Determination of Asbestos Fibers in Water *EPA-600/4-84-043*

R. J Lee, 2003 *130 Liberty Street Property, WTC Dust Signature Report, Asbestos, December 2003* 21 pages

Appendix C. Responses to Questions of Clarification

Question 1:

Final Report, page 13, top "Three of the commercial laboratories, designated as labs E, F and G, reported analytical data that are not consistent with other five labs Generally, these labs were not able to distinguish differences between the three spiking levels In addition, these labs did not meet the measurement quality objectives (MQOs) for the spiked samples put forth in the QAPP for this study (Appendix A Section A 7 1) Thus, the data from these three labs are not considered in the results presented in Table I and Figures 4-7 " Please provide details of MQO deficiencies for all eight of the original commercial laboratories.

EPA Response:

MQO comparisons were only made for the spiked sample data The following is a synopsis of the analysis.

Intralaboratory

Once it was determined that Labs E, F and G were not able to distinguish differences between the three levels for the spiked WTC samples, we compared the data to the MQOs for intralaboratory precision For labs E, F and G, data for five of the six sets of spiking duplicates fell outside of the ± 30% intralaboratory precision level All other labs did reasonably well with regard to precision Thus, based on both of these results (not being able to distinguish between the spiking levels and not meeting intralaboratory precision MQOs), the data from Labs E, F and G were not used

Data for intralaboratory accuracy showed a great deal of variability This is, in part, due to the fact that we do not have a 'standard' test dust against which we can compare our data – instead, MQO qualification was calculated in two ways: 1) each labs average was compared to an assumed amount of 'pure dust' This assumed amount was based on the percentage level and the original dust that was used for spiking (i.e pure USGS dust contained 11 E+06 fibers per gram of dust, thus, the 10% spiking level should contain 1.1E+06 fibers per gram of dust, etc.) and 2) each labs average was compared to the overall average for all labs for each sample In both instances, the results showed high levels of variability, thus, the MQO for accuracy (± 30%) was not helpful in choosing lab data to consider in our analysis

Two labs (A and C) failed to meet the 95% MQO for intralaboratory completeness Note that it only took one missing value to fail meeting this MQO

Interlaboratory

Interlaboratory precision was determined by averaging the duplicate samples for each lab, and then determining an average, standard deviation and relative standard deviation for the set of eight labs When the three labs (E, F, and G) are left out, the estimated overall precision of the study improves substantially

Interlaboratory accuracy was evaluated by averaging the duplicate samples for each lab, and then determining an average for the set of eight labs. This showed a great deal of variability. As stated previously, some of this variability is due to the fact that we do not have a 'standard' test dust against which we can compare our data MQO qualification was calculated by assuming the 'amount of pure dust' per sample based on the percentage level and the original dust that was used for spiking (i.e pure USGS dust contained 11 E+06 fibers per gram of dust, thus, the 10% spiking level should contain 1 1E+06 fibers per gram of dust) When the three labs (E, F, and G) are left out, the accuracy of the study improves for one set of spiked samples (USGS, all %'s), and meets the MQO for those samples When the three labs are left in the analysis, the overall accuracy of the study does not meet the MQO for either set of spiked samples

The study meets the interlaboratory 95% MQO for completeness when only considering spiked samples

Question 2:

Final Report, Appendix D §12.1 Analysis by Polarized Light Microscopy Is this the full bench protocol that the laboratories used or was further detail provided during interlaboratory discussions or by other means?

EPA Response:

The full bench protocol was provided in the report It was realized part way through the study that PLM was not a time-saving measure and thus, will not be part of a final protocol for the proposed sampling

Question 3:

QAPP, §A5 2 Method Development (penultimate sentence) This method was reviewed by the WTC Expert Panel's signature subcommittee and is presented in Appendix B The Appendices list that appears on page 20 identifies Appendix B as the PLM/SEM protocol Please provide a copy of the review referenced in A5 2

EPA Response:

A formal written review was not provided by the subcommittee members Drs Paul Lioy and Mort Lippmann provided comments by email A PDF of these emailed comments is provided under separate cover. Mr Meeker was involved in the development of the protocol and did not provide written comments

Question 4:

Draft Final Plan, page 1, paragraph 5· Using a peer review contract, EPA solicited comment from non-panel experts on the use of asbestos as a surrogate for determining risk from other contaminants and provided a report on those comments back to the panel The non-panel experts generally supported the use of asbestos as a surrogate, but encouraged the concurrent testing for lead Many individual members of the panel, however, did not support the position that

asbestos was an appropriate surrogate in determining risk for other contaminants What was the panel's rationale for elimination of chrysotile as a surrogate?

EPA Response:

First, by "panel. in the question just above, EPA assumes this refers to the WTC Expert Technical Review Panel (WTC Panel), and not the asbestos-as-surrogate peer review sub-panel In any case, it would be appropriate to simply comment that the WTC Panel did not concur with the primary finding of the asbestos-as-surrogate sub-panel While some on the sub-panel did not support the position that asbestos was an appropriate surrogate, it is fair to conclude that the "majority" opinion, although the sub-panel was unambiguously not a consensus panel, was that asbestos would serve as an appropriate surrogate.

The report from that panel is posted on the WTC Panel web site under the heading, "Background Documents " A good summary of the individual sub-panel comments on asbestos as a surrogate can be found on pages 7-9 of this report

The WTC Panel concluded that a "WTC signature" would be a more powerful tool for the purpose of determining the extent of remaining contamination from the WTC collapse The contaminants of potential concern (COPC) EPA tested for during its cleanup program (asbestos, PAHs, dioxins, lead and mercury) are all common urban contaminants With the exception of lead, the COPC were found at low levels and low frequency during EPA testing A distinct "WTC signature" would allow attribution of COPC found in sampled dust to the WTC rather than other sources

Question 5:

It's clear the five commercial labs were provided blind samples, but were the three government labs? I think they were, I just wanted to be sure

EPA Response:

All labs were provided with blinded samples

Question 6:

Nowhere (that I kind find) do the documents we have discuss sample size There is mention of a 32 oz jar for the vacuum cleaner and a 1,000 gram maximum reading for a scale. I get the idea the collected samples are on the order of 500 grams Is this correct?

EPA Response:

As much sample as possible was collected Sample sizes ranged from 10 grams or so up to many hundreds of grams

Question 7:

Would EPA provide a copy of the Occupational Safety and Health Administration Field Operations Manual, "Instruction CPL 2-2.0A, March 1984, Chapter VII. Sampling for Surface Contamination 1.h"?

EPA Response:

The reference requested can be viewed online at
http //www osha gov/dts/osta/otm/otm_ii/otm_ii_2 html

Question 8:

Has anyone measured the refractive indices of the MMVF in the WTC dust? I'm specifically interested in the slag wool and mineral wool discussed in our review documents

EPA Response:

Message forwarded to ERG by EPA reads. "I don't think the specific refractive index or the RI range was measured for the slag wool present in the WTC dust I do know that we did not measure it here at NEIC, but instead used the values from TIMA, 1991 I also know that the slag/mineral/rock wool fibers were easily identified using both 1 550 and 1 605 RI oils "

Question 9:

Please provide copies of all of the data sheets generated by the laboratories for the PLM and SEM analyses

EPA Response:

EPA provided ERG with 16 data files that were generated by 8 laboratories (some laboratories' data are documented in more than one file) All of these files were forwarded to the peer reviewers

Question 10:

Section 11 4 (Preparation of Sample for Polarized Light Microscopy) includes the following statement· "Allow them to dry, then add a drop of 1 55 (or 1.605) refractive index oil " Since no guidelines are provided to determine which oil, 1 550 or 1 605, is to be used, does this mean it was at the discretion of analysts to decide which oil should be used?

EPA Response:

Yes

Question 11:

Section 11 4 (Preparation of Sample for Polarized Light Microscopy) includes the following statement· "Allow them to dry, then add a drop of 1 55 (or 1 605) refractive index oil." For the 10% spiked sample, which of the 8 laboratories (A,B,C,D,E,F,G,H) used 1.550?

EPA Response:

Laboratories F and H.

Question 12:

Section 12 1 (Analysis by Polarized Light Microscopy) includes the following statement· "The fraction of fibers with refractive index greater than 1 55 (or 1 605) will contain mineral wool, which includes both slag wool and rock wool, and possibly some E-type glass and ceramic fibers " Please explain in details in which occasions all fibers > 1 550 are slag wool (SW), rock wool (RW), and E-type glass (EG) and ceramic fibers (CF) and in which occasions only those > 1 605 are SW, RW, EG, and CF

EPA Response:

According to TIMA, 1991, slag wool and rock wool have a refractive index (RI) of 1 6-1 8. E-type glass and ceramic fibers have an RI of 1 55-1 57 So, depending on which RI oil was used, 1.55 or 1 605, EG and CF may or may not be identified These four fiber types are the types that have been identified in WTC Dust samples and background samples which have RI>1 55 Clarification of fiber type, by chemistry, is to be performed in the SEM/EDS analysis This secondary clarification is part of the reason that PLM was not a time-saving step as expected

Question 13:

Section 12 2 1 (Screening for Slag Wool) includes the following statement "When an inorganic fiber is found, identify the composition of the particle by EDS Slag wool is the primary fiber of interest " Please explain in details the criteria used to differentiate slag wool fibers from other MMVF fibers by EDX.

EPA Response:

The criteria used to differentiate slag wool from other MMVF is from the USGS Open File Report 2005-1165 "Particle Atlas of World Trade Center Dust" by Heather Lowers and Gregory Meeker This atlas is available at http://pubs.usgs.gov/of/2005/1165/508OF05-1165.html This was the definition used for identification of MMVF for this purpose and was available for all laboratories to use The pertinent text is as follows

> **MMVF and glass fragments**
>
> Man-made vitreous fibers (MMVF) are abundant in WTC dust Glass fibers range in diameter from < 1 μm to > 50 μm with lengths up to several hundred micrometers The best compositional match for the majority (>85%) of WTC glass fibers is slag wool, a by-product of pig iron production (TIMA, 1991) Pieces of yellow thermal insulation found in bulk samples are composed of soda-lime glass fibers (Meeker and others, 2005b) Very few fibers of this composition exist in the

fine (<150 μm) microscopic portion of the dust Rock wool is also present as a trace constituent of the fine dust portion

Rock wool and slag wool can have similar EDS spectra. The two can be distinguished based on the presence of iron Slag wool will generally have less than 2 weight percent FeO whereas rock wool contains from 3 to 12 weight percent FeO (TIMA, 1991) Soda-lime glass has a distinct EDS spectrum from both slag wool and rock wool. The Na peak is higher and Ca, Mg, and Al peaks are smaller in the soda-lime glass spectrum than the slag wool and rock wool spectra.

Glass shards, fragments, and spheres are also present in the dust samples The microscopic glass shards and fragments are less abundant than the ubiquitous slag wool fibers in the fine dust (<150 μm) Most of the glass fragments fall within the compositional range for soda lime glass, a common type used as window glass (TIMA, 1991) Other glass fragments are present which contain mostly Si with trace amounts of Na, K, and/or Al The majority (> 90%) of glass spheres, generally less than 500 μm in diameter, are of slag wool composition.

Sample EDS spectra from the atlas can be found following links from the text

Question 14:

Section A7 2 of the QAPP (Measurement Quality Objectives) includes the following statement. "The accompanying tables (Tables 1 and 2) list Measurement Quality Objectives (MQOs) for this intralaboratory (within lab) and interlaboratory (within sample) variability Accuracies and precision were taken from preliminary data and manufacturer's specifications " Please explain in detail how accuracy was evaluated for the slag wool analysis by PLM

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

EPA Response:

It wasn't. It was decided part way through the study not to use PLM as it was determined not to be a time saving measure (as intended)

Question 15:

Section A5 2 of the QAPP (Method Development) includes the following statement. "This method was reviewed by the WTC Expert Panel's signature subcommittee and is presented in Appendix B " The Appendices list that appears on page 20 identifies Appendix B as the PLM/SEM protocol Please provide a copy of the review referenced above

EPA Response:

A formal written review was not provided by the subcommittee members Drs Paul Lioy and Mort Lippmann provided comments by email A PDF of these emailed comments is provided under separate cover. Mr Meeker was involved in the development of the protocol and did not provide written comments

EPA asked that the peer reviewers be aware of the web site for the World Trade Center Expert Technical Review Panel (http //www epa.gov/wtc/panel/) Presentations and summary reports from panel meetings are posted on the web site, along with a number of background documents related to development of the draft WTC sampling plan and the WTC dust screening study On the "Background Documents" page, EPA has also posted comments from individual members of the WTC panel

One panelist, David Newman of the New York Committee for Occupational Safety and Health, has specifically asked the reviewers be made aware of some of his comments that EPA has posted links to below

March 1 (page 13)
http //www epa gov/wtc/panel/pdfs/comments/02-23-05_PanelMembersComments pdf

May 27 (page 3)
http //www epa gov/wtc/panel/pdfs/comments/combo_comments_052405 pdf

June 6 (page 10)
http //www epa gov/wtc/panel/pdfs/comments/combo_comments_052405 pdf