Analysis of Background Residential Dust for World Trade Center Signature Components Using Scanning Electron Microscopy and X-Ray Microanalysis

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Introduction

This Open File Report (OFR) describes the analysis of six background samples of urban residential dust collected in Manhattan and Long Island by the U.S. Environmental Protection Agency (USEPA). This report is a supplement to U.S. Geological Survey OFR 2005-1031 (Meeker and others, 2005) that defines signature components of dust dispersed by the collapse of the World Trade Center (WTC). These signature components can be used as a fingerprint for identifying WTC dust contamination in building units. The data presented in Meeker and others (2005) suggest that the presence and relative abundance of slag wool, mineral wool, and soda-lime glass along with the presence of concrete particles and Ca sulfates (primarily gypsum and anhydrite) could be used as a fingerprint for WTC dust.

Bulk background dust samples analyzed in this study were collected between November 2004 and January 2005 from residential units at the approximate locations shown in Figure 1. The samples represent vacuumed surfaces from windowsills, carpets, bathroom vents, and tops of storage units. These samples were analyzed to determine if and at what levels components identified as WTC signature components, primarily slag wool, exist in background dust. It was assumed that the samples analyzed for this report were not affected by the dust cloud generated by the collapse of the World Trade Center buildings.

Sample Preparation

The bulk samples, as provided by USEPA, were split using the cone and quarter technique. Half of each sample was ashed using a high temperature muffle furnace to remove the organic fraction. The furnace was programmed so the temperature increased 1 ºC/minute to 250 ºC and held for 4 hours. After 4 hours at 250 ºC, the temperature increased 1 ºC/minute to 480 ºC and was held for 8 hours, after which time the program was reversed to step down to room temperature. The samples were kept in covered crucibles throughout the ashing process to prevent cross contamination. The progressive heating is intended to minimize flash combustion of the samples, which could cause sample loss and/or cross contamination. The upper temperature limit of 480 ºC was

chosen to prevent the decomposition of chrysotile. Melting temperatures of slag wool and rock wool range from 1149-1260 ºC (TIMA, 1991) so it is unlikely these phases were affected by the ashing process. Gypsum converts to anhydrite at these temperatures; however, for purposes of identification by energy dispersive x-ray spectrometry (EDS), gypsum and anhydrite appear identical. Most concrete phases are not significantly affected by heating to 480 ºC.

The ashed portion of each sample was split using the cone and quarter technique. Half the sample was placed in a 4 mL glass vial with 1 mL isopropanol alcohol. An ultrasonic bath was used to disperse the particles. After sonification, the sample was shaken to suspend the particles. A micropipette with the tip cut to produce a ~1 mm opening was used to remove six 5 µL aliquots from the top, middle, and bottom of the solution. The aliquots were placed onto an adhesive carbon tab attached to an aluminum sample mount. After drying, the mounted sample was coated for analysis using a carbon evaporator.

Methods

Analyses were performed using a JEOL 5800LV electron microscope equipped with an Oxford ISIS (EDS) and analysis system. Typical analytical conditions were 15 KeV accelerating voltage, 0.1 – 3 nA beam current and zero degree tilt. Data were processed using standardless quantitative analysis as described in Meeker and others (2005).

The entire sample mount was examined at 100X magnification. Each field of view was inspected for the presence of man-made vitreous fibers (MMVF). If MMVF were present, area percent coverage of the field was determined using binary images derived from backscattered electron images (Figure 2). In addition, the category of MMVF (slag wool, rock wool, soda-lime glass, other) and dimensions were determined. EDS spectra of typical slag wool, rock wool, and soda-lime glass are shown in Figures 3, 4, and 5, respectively.

Results

The results of the analyses are summarized in Table 1. Soda-lime, Ca-Al-Si, and inorganic carbon fibers were the most common fibers observed. Slag wool fibers were observed at low levels in four of the six samples examined. All samples contained SiO_2 (probably quartz), talc, $CaSO_4$ (gypsum or anhydrite), calcium-magnesium carbonate, calcium carbonate, and Fe-rich phases.

Table 1. Summary results of MMVF in background samples.

Sample	1	2	3	4	5	6
# of Soda-Lime Fibers Observed	2	n.d.	3	58	2	3
# of Slag Wool Fibers Observed	n.d.	1	1	6	n.d.	1
# of Rock Wool Fibers Observed	n.d.	n.d.	n.d.	1	n.d.	2
# of other MMVF Fibers Observed	n.d.	n.d.	39	2	n.d.	0
Weight of material before ashing (g)	0.060	0.582	0.283	0.070	0.092	0.107
Weight after ashing (g)	0.027	0.021	0.009	0.013	0.063	0.030
Weight for prep (g)	0.008	0.013	0.004	0.008	0.036	0.018
Weight of sample on stub (g)	2.34E-04	3.99E-04	1.23E-04	2.37E-04	5.34E-04	5.40E-04
Weight of slag wool fibers on stub (g)	---	3.17E-09	9.67E-09	7.18E-08	---	7.64E-09
% Slag Wool in sample	---	2.85E-05	2.39E-04	5.56E-03	---	4.02E-04
ppm (wt.)	---	0.3	2.4	56	---	4.0

n.d. = not detected, --- = not calculated because of absence of slag wool
The analysis time to determine these concentrations was approximately 30-60 minutes.

The weight fraction of slag wool was determined for each sample in which it was observed. The weight of the slag wool fibers was calculated by multiplying the density, 2.8 g/cm^3 (TIMA, 1991), by the volume, assuming the width was equivalent to the diameter of the fiber. It was assumed that all of the slag wool in the analyzed aliquot was concentrated in the ashed residue. The weight percentage of slag wool in the bulk sample was calculated as follows:

$$\frac{((\text{Volume of slag wool} \times \text{Density}) \div \text{Weight of sample on stub}) \times \text{Weight of ashed sample}}{\text{Weight of unashed sample}}$$

Discussion

The ability to determine the amount of WTC dust in a sample using signature components depends on the abundance of background signature components from sources other than the WTC (Meeker and others, 2005). These signature components (slag wool, gypsum, and particles compatible with concrete) are common building materials and therefore could be present from sources not related to the collapse of the WTC buildings.

In the six background samples analyzed in this study, MMVF were present as minor to trace components. Soda-lime glass (a type identified in WTC dust) and Ca-Al-Si glass fibers (probably E-type glass (TIMA, 1991), a type not identified in WTC dust) dominated the MMVF types (Table 1). Rock wool was the least common MMVF present. The relative proportions of MMVF types present in these background samples are not consistent with the proportions found in WTC dust (Meeker and others, 2005). Furthermore, the mass concentrations of slag wool, the primary fiber type in WTC dust was very low, ranging from not detected to 56 ppm (Table 1).

For the purposes of defining a detection limit for WTC contamination in collected dust, one must consider the case where any slag wool, no matter how low the concentration, might be derived from WTC dust contamination. As seen in Table 1, the abundances of slag wool are extremely low and the detection limits will depend on the weight of the unashed sample and the weight of the sample analyzed in the SEM. In Table 1, the smallest amount of slag wool detected was approximately 0.3 ppm.

The highest abundance of slag wool found in the background samples was 56 ppm by weight (sample 4). A conservative approach for an unknown sample (i.e. not background) containing a similar amount of slag wool would be to assume that all of this material was derived from WTC dust. Therefore, the maximum amount of contaminants of potential concern (COPC), such as chrysotile asbestos or crystalline silica, derived from WTC dust could be calculated by using the relative proportions of phases found in WTC dust by Meeker and others (2005). The highest ratio of chrysotile to slag wool was 1.8:30

(sample USGS 12) and the highest level of silica to slag wool was 3.4:30 (sample USGS 12) (Meeker and others, 2005). If one were to assume that all of the slag wool present in sample 4 was due to WTC contamination, then the amount of chrysotile and silica contributed from WTC dust in sample 4 would be ~3 ppm and ~6 ppm, respectively, based these ratios. Maximum likely levels of other WTC dust-derived COPCs could be approximated in a similar manner. The approach outlined above would only be necessary if a general background level for slag wool cannot be established by collection and analysis of additional samples.

The use of relative proportions of other WTC signature components such as concrete phases and $CaSO_4$ (gypsum) as identifiers of WTC contamination is difficult at such trace levels. The presence of gypsum in all of the background samples may limit its use as a signature component where the slag wool content is in the low ppm range. At these levels it may be sufficient to define a maximum possible WTC contribution based on slag wool only.

Conclusions

Six residential background samples were analyzed for slag wool and other WTC signature components in this study. Slag wool was not detected at 100X magnification in two of the six samples. The other four samples contained low concentrations of slag wool at levels of 56, 4.0, 2.4, and 0.3 ppm by weight. Gypsum, another signature component, was identified in all of the samples probably because it is the main component of wall board. It is therefore likely that some amount of gypsum will be present in any sample from a unit, residential or otherwise, where gypsum wall board is used. It is also likely that the gypsum background levels will prove to be higher than slag wool. This study suggests that when slag wool is present at levels in the low ppm range it may not be possible to use the presence of gypsum as a signature identifier of WTC dust. In such cases it may be adequate to assume a maximum concentration of WTC COPCs based on the slag wool content.

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 of background dust from office buildings and other interior environments is needed to determine background ranges of signature components in these types of interior spaces.

Acknowledgements

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TIMA (Thermal Insulation Manufacturers Association), 1991. Nomenclature of Man-Made Vitreous Fibers. TIMA Inc. 72p.

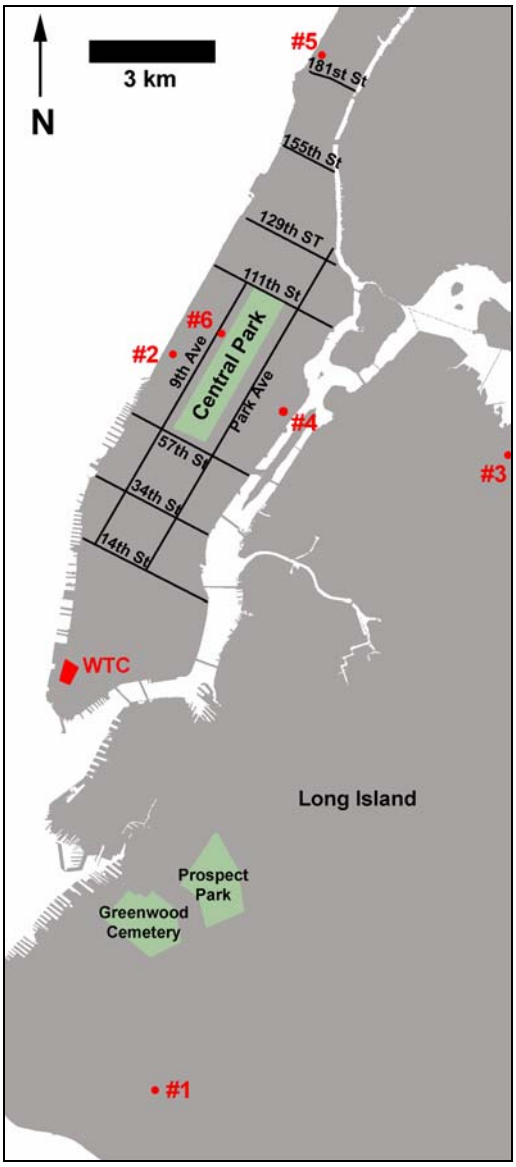

Figure 1. Map of New York City showing approximate locations of residential units where samples were collected (#1 to #6).

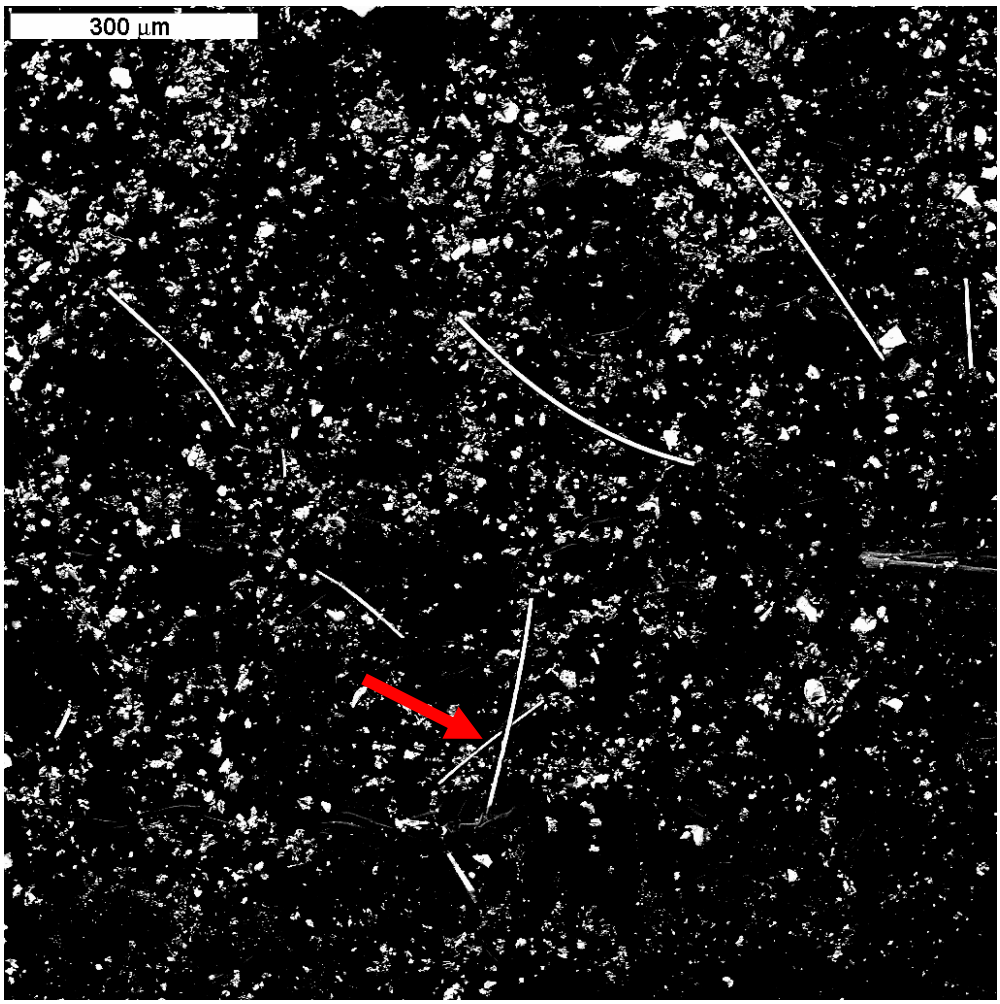

Figure 2. Binary backscattered image of field of view at 100X (sample 3). The majority of the fibers are soda-lime glass. The one slag wool fiber in this image is identified with a red arrow.

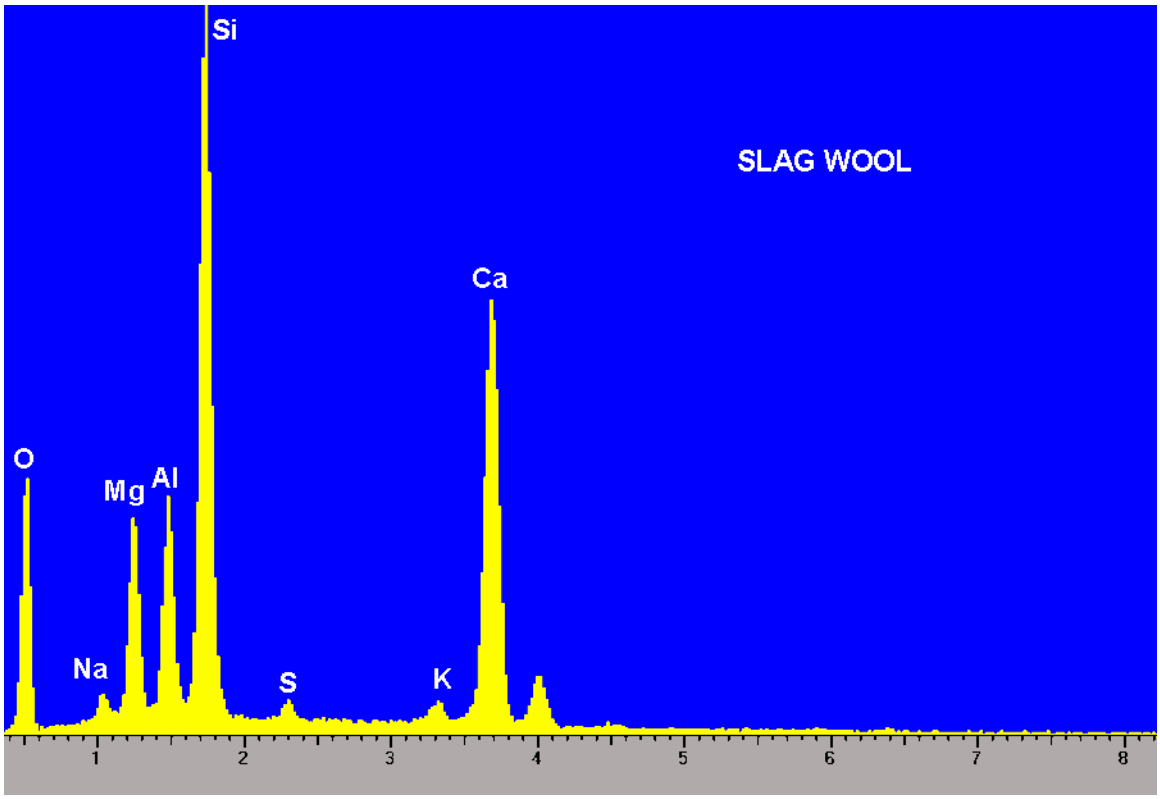

Figure 3. EDS spectrum of slag wool.

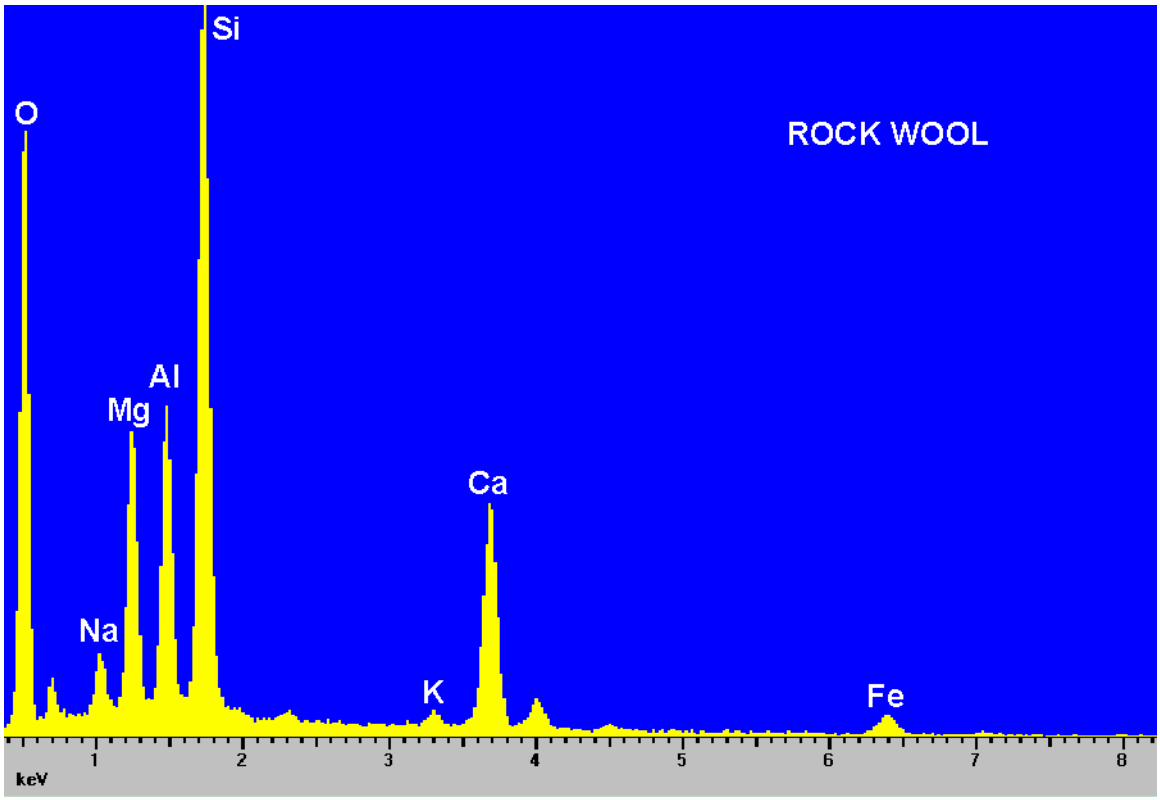

Figure 4. EDS spectrum of rock wool.

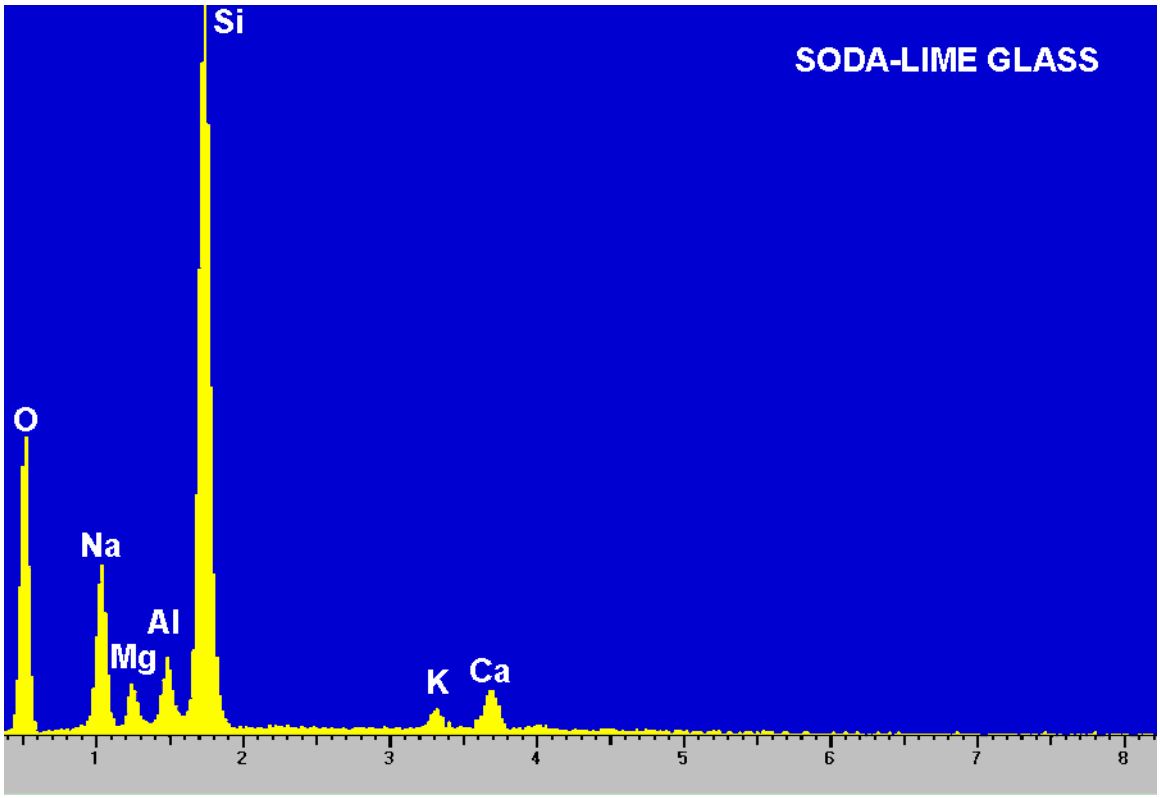

Figure 5. EDS spectrum of soda-lime glass.