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Morphological and Optical Characterization of Amphiboles from Libby, Montana U.S.A. by Spindle Stage Assisted - Polarized Light Microscopy

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KEYWORDS

Asbestos, polarized light microscopy, PLM, spindle stage, Libby, Montana, tremolite, amphibole, extinction angle, aspect ratio

INTRODUCTION

Asbestos has been a major health concern in the United States since the 1960s (1). Since then, much has been learned about common asbestos minerals and presented in several works (2-4). For instance, we know that the most commonly used asbestos variety, chrysotile - a serpentine mineral, appears to be less harmful than the more rarely used amphibole asbestos varieties (5-7). Also, several studies have shown that the fibrous variety of tremolite, i.e., tremolite-asbestos may be the most harmful of the amphibole minerals (8-12). The creation of regulatory agencies, like the Occupational Safety and Health Administration (OSHA) in 1970, and the regulations they have developed since 1972, have greatly reduced the risk of asbestos-related diseases to the point where, over the past decade, asbestos has fallen off the front page of the newspapers and out of the minds of the general public. This changed on November 18, 1999, when the Seattle Post-Intelligencer published an article about asbestos-related diseases of former miners in Libby, Montana (13). The miners worked in the world's largest vermiculite mine owned by W.R. Grace from 1963 to its closure in 1990. It had previously been owned by Zonolite Corporation with operations since 1923. The vermiculite ore was reported to contain approximately 5% tremolite-asbestos and exposure to this impurity in the ore caused an increase of asbestos-related disease in the miners. This article caught the attention of the United States Environmental Protection Agency (EPA), which arrived on the scene in a few days. Since then, millions of dollars have been spent on remediation in the area and health studies have begun.

Originally, the only amphibole believed to be in the mine in Libby was tremolite, however recent work (14) showed that two samples from the mine are winchite, which is not one of the six regulated asbestos minerals. Gunter et al. (15) confirmed these results using the same set of Libby amphibole samples in this morphological study.

ASBESTOS NOMENCLATURE – DISTINGUISHING AMPHIBOLE FRAGMENTS FROM FIBERS

Although not commonly viewed this way, there are two basic definitions of asbestos; one is physical and the other chemical. As with any definition, problems arise with natural samples based on our limitation to formally describe nature.

The physical definition of asbestos deals with its morphology or shape. Regulatory agencies consider a particle to be asbestos, for counting purposes, if its aspect ratio is 3:1 or greater and the particle is over 5 μm in length (5, 7, 16). This is, of course, very different from the physical characteristics a mineralogist would use – that the particle must have a fibrous form (see reference 19 for an overview of asbestos terms).

The chemical definition of asbestos used by regulatory agencies for identification includes six mineral species. These minerals are chrysotile, crocidolite, amosite, tremolite, actinolite, and anthophyllite (5, 7, 16). Chrysotile is the asbestos form of serpentine, a sheet silicate. The others in this group are all amphiboles. Crocidolite and amosite are asbestiform varieties of the amphibole minerals riebeckite and grunerite, respectively. Thus the names chrysotile, crocidolite, and amosite always denote asbestos minerals, while tremolite, actinolite, and anthophyllite can occur in

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either a non-asbestos (non-fibrous) or asbestos (fibrous) form, with the non-asbestos form being much more common in the geological environment.

There has been considerable controversy, for over 20 years, on distinguishing cleavage fragments, or single crystals of amphiboles, from fibers of amphiboles (10, 20-22). The underlying reason is that cleavage fragments, when inhaled, appear to be less harmful than fibers (12, 19, 23). Based on a review of all the existing literature, cleavage fragments of the amphibole minerals were deregulated in 1992 (23). Regulatory agencies simply use the aspect ratio to make the distinction between fragments and fibers, however, as we show in this paper (and has been shown by other researchers: 5, 16, 19, 21), this definition simply does not work. Fibers and fragments possess different physical properties and, as always, the physical properties of a mineral are directly related to its structure.

The structural difference between a fragment and a fiber is that fibers of asbestos are made up of many crystals, i.e. they are polycrystalline. They occur as fiber bundles comprised of individual fibrils, much like a rope is made of many small strands; giving asbestos its incredibly high tensile strength and flexibility (24). And, as Wylie (16) points out, common asbestos fibril sizes range from 500 Å in chrysotile to 6,000 Å in some amphibole-asbestos samples. Fragments, in turn, are single crystals. Thus, any analytical method that could distinguish polycrystalline materials (e.g., intergrown fibers) from a single crystal (e.g., growth or cleavage fragments) would work to distinguish fibers from fragments. This can be determined with polarized light microscopy on particles as small as 1 μm; however, when the particles reach a width and thickness of a few microns certain useful optical properties (i.e., extinction characteristics) become difficult to observe and measure due to lower retardation. In addition, Wylie (21) noted that monoclinic amphiboles (e.g., tremolite and actinolite) yield parallel extinction when they occur as fibers, instead of the expected inclined extinction. While this method works most of the time, it has limitations as discussed herein.

Diffraction methods (X-rays or electrons) can also be used to determine crystallinity i.e., single versus polycrystallinity. Wylie (21) showed that amphibole fibers display a polycrystalline diffraction pattern in the ab-plane. TEM methods have also been used on very small samples. When an amphibole particle is rotated about its c-axis, the electron diffraction patterns remain the same if it is a fiber, but changes if it is a single crystal (19).

Typically, cleavage fragments of amphiboles expose the (110) plane. However, it has been shown by past researchers (25) that single small crystals of amphiboles are flattened on (100); our study confirms this observation. In fact, this study shows that there is a possible relationship between crystal size and (110) or (100) surface development. It has also been shown that amphibole fibers are flattened on (100) (24, 26). Thus, we speculate that it might not be the fibrous form of the amphibole alone that poses the health risk, but the exposed surface, i.e., (110) surfaces may be less harmful than (100) surfaces and perhaps these surfaces, by exposing different planes of atoms in the mineral, may react differently in the human lung. Also, the surface area would be greater for a given volume of material as particle size decreases.

With the recent concerns at Libby, the definition of asbestos by the regulatory agencies comes into question; this should result in changes in regulations. For instance, as outlined in (15), the health risks associated with whatever amphiboles occur at Libby are significant. It appears that, regardless of species type, all amphibole-asbestos should be regulated. This might also extend to all fibrous silicates in general. For instance, erionite, a fibrous zeolite, has been shown to induce mesothelioma in very high amounts in lab animals and been linked to outbreaks of mesothelioma in Turkey (27). The common denominator in most of these health-related mineral problems is fibrous silicates, and perhaps they should all be regulated. However, quartz, which was recently upgraded to a Group 1 human carcinogen, is not fibrous (29). Again, silicates seem to be the common thread (27-32). Clearly this needs to be revised in light of Libby to include, at the least, all amphibole-asbestos. At present, we are left with only the six "asbestos minerals" being regulated.

GOALS OF THE STUDY

In this study we attempted to characterize the shape of particles and classify them as either single crystals, which we termed as fragments, or multiple crystals, which we termed as fibers. As such, photomicrographs of the samples provide a qualitative description. We made thousands of optical measurements on the samples in this study, and quantified these data in a series of descriptive tables. The "Results" and "Discussion" are divided into two distinct but complementary sections: analyses done on grain mounts, which is the common method of characteriz-

ing asbestos particles, and analyses of single particles with the aid of the spindle stage.

One of our goals for examining single particles was to aid in understanding our observations on grain mounts i.e., we could determine the precise extinction angles when the particles were mounted on the spindle stage, and to observe the morphological characteristics of the particles in 3D as compared to 2D in the grain mounts. Other researchers have measured aspect ratios for amphibole particles in grain mounts (e.g., 16-17), but none have done this with the spindle stage. With the spindle stage, the thickness, length and width can be measured so that the volume of a particle can be calculated. Wylie et al. (18) made a similar set of measurements on the thickness of smaller amphibole particles using both an SEM and TEM.

MATERIALS

Three separate samples were chosen for this study: a non-asbestos tremolite from our teaching collection (called UI tremolite herein), a NIST tremolite asbestos standard (NIST asbestos standard #1867), and amphiboles collected from the former vermiculite mine near Libby, Montana by the author (MEG) in October 1999. The UI tremolite sample was selected to represent a non-fibrous amphibole and to obtain data on cleavage fragments. The NIST tremolite was selected for a comparison to the Libby amphiboles. In general, tremolite samples were selected because the amphiboles from Libby had been reported to be tremolite. Since this project started, Wylie and Verkouteren (14) showed this not to be the case; they determined that two samples of Libby amphibole were winchite. Our ongoing research (15) also found the samples to be winchite and richterite. Nevertheless, the tremolite samples chosen for this study were used to compare differences in morphology and optical characteristics to the Libby amphiboles, because no winchite and/or richterite standards exist at this time. However, winchite-asbestos has been shown to occur in nature (33).

The Libby samples were further divided based on occurrence at the mine. Three samples were chosen. One was collected, in place, from one of the mined-out benches (15), called "outcrop" in this work. A second sample was taken from a 2 cm vein of amphibole in the biotite pyroxenite, the rock mined for vermiculite, called "vein" herein. The third was taken from an approximately 20 cm boulder consisting entirely of amphibole, which was resting on the ground in the middle of the abandoned mine, labeled "float."

EXPERIMENTAL METHODS

Two separate optical procedures were used to characterize the three different amphibole samples. One procedure employed a PLM to measure particle dimensions (i.e., length and width by use of a calibrated eyepiece), morphology, and extinction angles to determine if a particle was either a fiber or fragment in grain mounts. The second procedure used the PLM equipped with a spindle stage to measure particle dimensions (i.e., length, width, and thickness with the aid of a Vicker's image splitting eyepiece), morphology, and extinction angles as a function of orientation to determine if a particle was either a fiber or fragment.

Grain mounts were made for each of the samples by placing a small of amount of each on a standard petrographic slide with 1.55 refractive index liquid. This refractive index value was chosen so the particles could be easily seen in plane polarized light. Each sample was prepared as follows. The UI tremolite was crushed and sieved to –60 mesh (250 μm). The NIST tremolite, which was provided from NIST already comminuted, was sieved to –60 mesh (250 μm). The Libby samples were crushed, pulled apart, and sieved to –60 mesh (250 μm). An extra step was added for both the NIST and Libby samples; they were placed in acetone and ultrasonicated to further break the particles apart.

For the spindle stage study, single particles were selected from the same samples as prepared for the grain mounts. These single crystals were attached to a glass fiber with fingernail polish with their long dimension approximately parallel to the fiber and placed on the spindle stage with the aid of a goniometer head (34). By angular adjustments on the goniometer head, each particle was made parallel with the rotation axis of the spindle stage. In this manner, the width and thickness were observed and measured. Additionally, extinction angles were measured on the (hk0) planes, i.e., (100), (010), and (110) or on the planes corresponding to the widest and thinnest portions of the crystals.

RESULTS - GRAIN MOUNTS

Eleven (11) total grain mounts were prepared. One slide for each of the UI tremolite and NIST tremolite was prepared and three slides were prepared for each of the three Libby samples (outcrop, vein, float). On each slide, 100 particles were chosen at random and their width and length were measured. They were classified as either fragment or fiber based on mor-

phological and optical properties (i.e., extinction characteristics) and their extinction angles were measured. Also, each particle was briefly described. It would be impractical to list all of the data, so select photomicrographs (Figures 1-3) and a series of tables (all tables are located in the Appendix, pp. 132-138) are used to summarize it.

Figure 1 shows grain mount photomicrographs of the UI tremolite (Figs. 1A and 1B), the NIST tremolite (Figs. 1C and D), and the Libby amphibole (Figs. 1E and 1F). The photomicrographs in the left column were taken in plane-polarized light, and in the right column the same sample is photographed again but this time in crossed polars. There is a distinct increase in the aspect ratio when comparing the UI tremolite, to the NIST tremolite asbestos, to the Libby amphibole. The circled particles in Figures 1A, 1C, and 1E would be classified as asbestos if based on aspect ratio alone (12:1, 16:1, 30:1, respectively), however, the circled particle in Figure 1A is a cleavage fragment and not asbestos, as is the circled particle in Figure 1C. This distinction is made based on morphology and extinction conditions as shown in the corresponding Figures 1B and 1D.

All of the important characteristics of the particle circled in Figure 1E are difficult to show in two photomicrographs. However, morphologically, the blunt ends would indicate it is a fragment but its curvature would indicate it is a fiber. The particle shows inclined extinction in Figure 1F and it shows complete, sharp extinction as the stage is rotated. For these reasons, this particle is classified as a fragment. If the extinction had not been complete, we would not have classified it as either a fragment or a fiber because it would have showed characteristics of both fibers and fragments.

It is also noteworthy to point out that, for the UI tremolite, most of the particles are visible in both plane polarized and crossed polarized light, while this is not the case for the other two samples. The particles in the UI tremolite sample have a higher retardation because they are lying on (110) while particles in the other two samples more commonly are resting on (100). This phenomenon will be elaborated on in the "Discussion" section.

Table 1 gives the particle count based on width and length. Notice there are 100 particles for the UI tremolite and only 99 particles for the NIST tremolite asbestos; one of the particles in the NIST sample was calcite. For the Libby samples, data from the three slides were combined, yielding a total of 300 particles for each. The Libby outcrop sample had two calcite particles and the Libby vein had one.

Given the length and width data, aspect ratios were calculated for all of the samples. Table 2 lists the percentage of particles with different aspect ratio ranges for the five samples. Also given in Table 2 are the divisions of the particles into three groups: fibers, fragments, and not classified based on morphology. Table 3 merely combines the three Libby samples into one and is similar to Table 2. Table 4 is a summary of the five samples classified based on aspect ratio (Table 4A) and by morphology (Table 4B). Table 5 again lists the five samples, but this time they are broken down on a particle count based on four extinction conditions: 1) "parallel," when the particle exhibited parallel extinction, 2) "inclined," when the particle exhibited inclined extinction, (also included in this column is the average extinction angle and its standard deviation), 3) "isotropic," when the particle exhibited near-zero retardation, and 4) "cannot measure," for particles that never went extinct or had wavy extinction.

RESULTS - SINGLE PARTICLES

In order to characterize the size (i.e., length, width, and thickness), extinction characteristics, and morphology of the three samples in this study; ten (10) particles of the UI non-asbestos tremolite, twenty-five (25) particles of the NIST tremolite, and fifty (50) particles of the Libby vein samples were mounted on glass fibers and observations and measurements were made with the aid of a spindle stage equipped PLM. Tables 6, 7, and 8 list the results for length, width, thickness, aspect ratio (l/w), aspect ratio (l/t), aspect ratio (w/t), the extinction angles (measured on two different planes), and the morphological characterization of these 85 particles. Table 6 lists these results for the UI tremolite sample in two different manners. Table 6A lists measurements for the widest and thinnest directions of the particle. These were obtained by rotating the sample about the spindle axis to find the largest and smallest dimensions. For all of the particles except #4 and #10, these directions do not correspond to the (100) or (010) directions, which is to be expected for an amphibole exhibiting (110) cleavage. Particles #4 and #10 are flattened on (100), which is obvious by the fact that they exhibit parallel extinction. In Table 6B, each particle was rotated so the (100) direction was brought parallel to the stage of the microscope; this is determined by the condition of parallel extinction. Its width and extinction condition were measured on

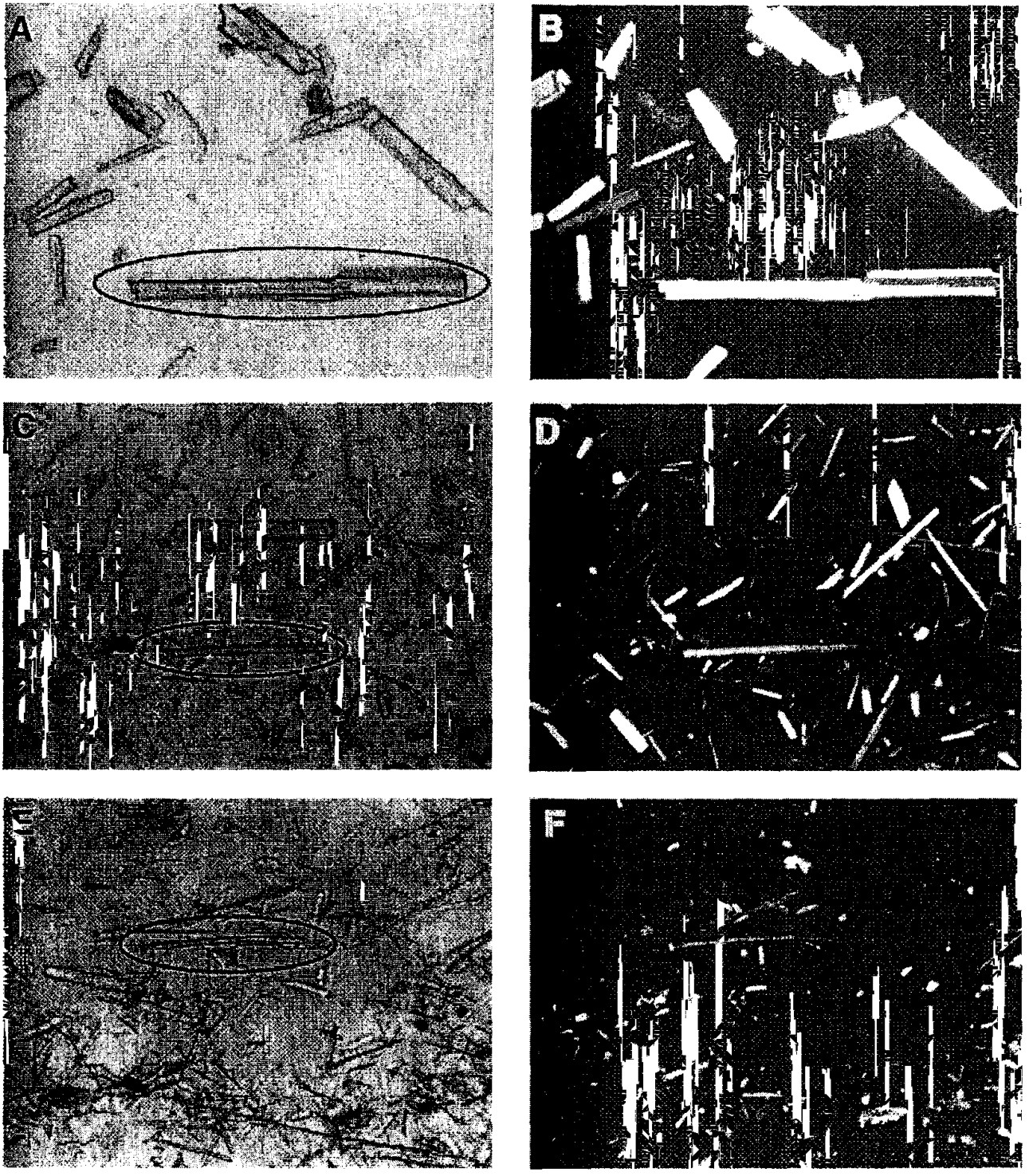

Figure 1: Photomicrographs of UI non-asbestos tremolite (A and B), NIST tremolite asbestos (C and D), and Libby amphibole (E and F). Photographs in left column correspond to those in the right column, with those in the left column taken in plane-polarized light and those in the right column taken in cross-polarized light. Circled minerals are discussed in the text. (Field of view is approximately 500 μm wide; samples are immersed in a 1.55 refractive index liquid.)

(100). The particle was then rotated and its thickness and extinction condition were measured on (010).

Figures 2 and 3 show photomicrographs of differing morphologies of the three samples immersed in a 1.55 refractive index liquid using the spindle stage. The images are of the same particles in the left and right columns, except the crystals have been rotated 90° about the spindle axis. Each particle was attached with fingernail polish (fluid-looking material) onto a glass fiber (the fibers are approximately 100 to 200 μm in diameter). Figure 2A is a photomicrograph of a single UI tremolite particle (particle #9, Table 6) viewed perpendicular to its widest direction; Figure 2B is the same particle as in Figure 2A, except the crystal has been rotated 90° to view it normal to its thinnest direction. Figures 2C to 2H are photomicrographs of the NIST tremolite sample. Figures 2C and 2D are of particle #5, Table 7 and Figures 2E and 2F are of particle #7, Table 7; both of these particles are considered fiber bundles based on their morphology. Figures 2G and 2H are NIST tremolite #21, Table 7 which is considered a fragment based on its morphology.

In Figure 3 are four samples depicting the three differing morphologies encountered in the samples from Libby. Figures 3A and 3B are of particle #7, Table 8, considered a fiber bundle, as is particle #22, Table 8 (Figures 3C and 3D). Figures 3E and 3F are of a particle considered to be a fiber mass (particle #18, Table 8). Lastly, Figures 3G and 3H show a fragment of the Libby amphibole (particle #21, Table 8). It is worth noting the orientations of the three fragments shown in this series of photomicrographs. In Figure 2A, we are looking down on the (110) surface; this is typical of cleavage fragments. In Figures 2G and 3G, we are looking at the (100) surface; this is typical of smaller amphibole crystals, i.e., they are flattened on (100).

DISCUSSION - GRAIN MOUNTS

Based solely on observation of Figure 1, there is an increase in the aspect ratio going from the UI tremolite (Figure 1A) to the NIST tremolite (Figure 1C) to the Libby amphibole (Figure 1E). The data in Tables 1 and 2 quantify this increase in aspect ratios observed in the Figures. Table 2 shows the percent non-asbestos, based on aspect ratio, to be 52% for the UI non-asbestos tremolite and 8% for the NIST tremolite asbestos. For the three Libby samples, these values are 0%, 5.4%, and 8.7% for the outcrop, vein, and float, respectively. Combining the three Libby samples, they would have 5% non-asbestos particles based on aspect ratio. Very different results are obtained basing the asbestos and non-asbestos proportions on morphology. Table 4 summarizes the data for all five samples and classifies each based on both aspect ratio (Table 4A) and morphology (Table 4B). Based on morphology, and mineralogical considerations, the entire UI tremolite sample is non-asbestos, as compared to 52% non-asbestos based on aspect ratios. For the NIST tremolite sample, 52% is non-asbestos based on morphology, while only 8% was non-asbestos based on aspect ratio. Lastly, the combined Libby sample shows the smallest amount of non-asbestos particles based on morphology, 33%, and aspect ratio, 5%. Also, note in Table 4 that we were unable to classify, as either fiber or fragment, approximately 30% of the NIST and Libby samples. Thus, the results based on aspect ratio differ significantly from those based on morphology, especially for the non-asbestos UI tremolite sample.

Our aspect ratio data yield similar results to two other studies. Wylie (35) found that a non-asbestos tremolite had 47% of the particles with an aspect ratio greater than 3 and 3% with an aspect ratio greater than 10, as compared to 48% and 4%, respectively, for the UI tremolite sample.

Basically, there are three types of particles in this study: fibers, cleavage fragments (which exhibit (110) cleavage), and single crystals, which are usually flattened on (100). Observation of extinction conditions has helped past researchers distinguish monoclinic amphibole fibers from cleavage fragments (21); in fact, OSHA mentions this method. The premise for this is that a fiber will show parallel extinction whereas a fragment will show inclined extinction.

Figure 4 shows sketches of monoclinic amphiboles with optical orientations similar to tremolite, winchite, and richterite. The lower illustration in Figure 4A represents an amphibole resting on its (110) cleavage surface. In this orientation, the sample would show inclined extinction; however, this orientation does not represent the true extinction angle (the angle between c and Z) which would be observed when a sample rested, or was viewed, on its (010) surface (lower illustration, Fig. 4B). Parallel extinction can occur because fiber bundles are elongated parallel to the c axis and the individual fiber's a- and b-axes are at random directions to this elongation; thus, the Z direction would average out over many particles to be parallel to the long direction of the fiber. This again means that an asbestos particle is really a polycrystalline material, while a fragment is a single crystal. This difference in crystallinity can be observed optically. However, if a single crystal of a monoclinic amphib-

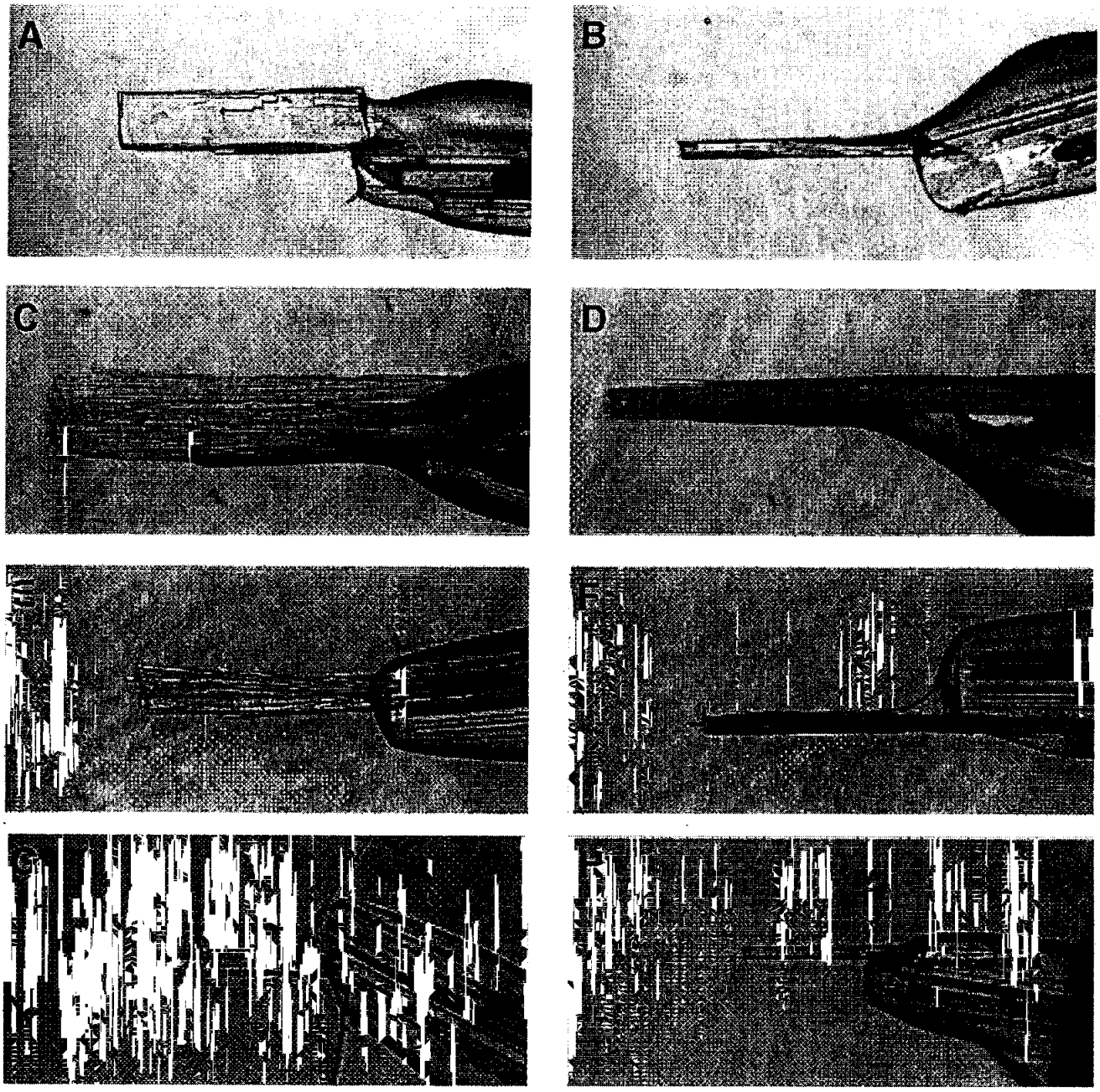

Figure 2. A) Image of UI tremolite #9 fragment (Table 6) viewed perpendicular to its thinnest direction; length is 562 μm; B) Sample in A rotated 90°; C) Image of NIST tremolite #5 fiber bundle (Table 7) viewed perpendicular to its thinnest direction; length is 728 μm; D) Sample in C rotated 90°; E) Image of NIST tremolite #7 fiber bundle (Table 7) viewed perpendicular to its thinnest direction; length is 594 μm; F) Sample in E rotated 90°; G) Image of NIST tremolite #21 fragment (Table 7) viewed perpendicular to its thinnest direction; length is 302 μm; H) Sample in G rotated 90°.

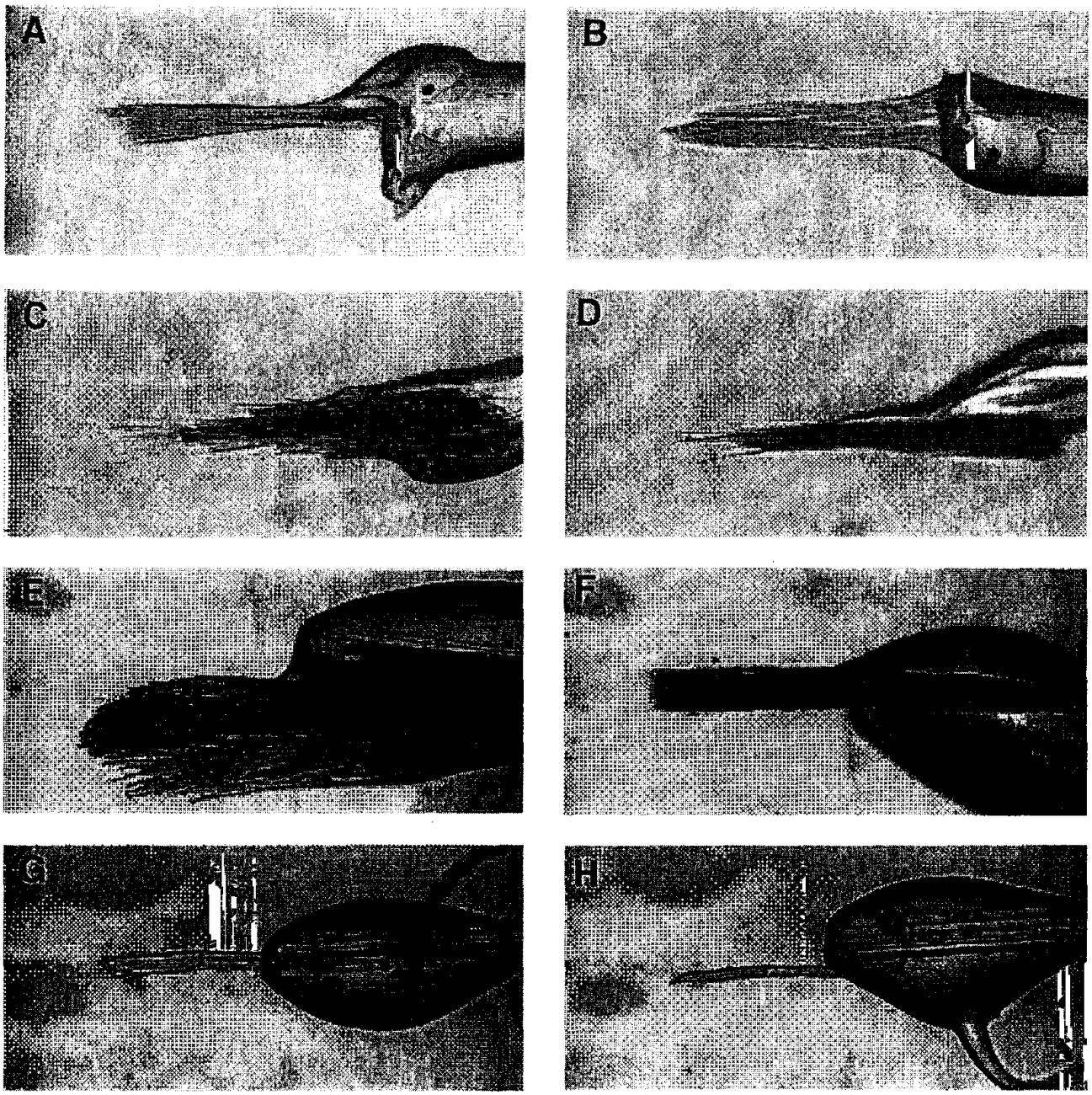

Figure 3. A) Image of Libby #7 fiber bundle (Table 8) viewed perpendicular to its thinnest direction; length is 537 µm; B) Sample in A rotated 90°; C) Image of Libby #22 fiber bundle (Table 8) viewed perpendicular to its thinnest direction; length is 512 µm; D) Sample in C rotated 90°; E) Image of Libby #18 fiber mass (Table 8) viewed perpendicular to its thinnest direction; length is 438µm; F) Sample in E rotated 90°; G) Image of Libby #47 fragment (Table 8) viewed perpendicular to its thinnest direction; length is 375 µm; H) Sample in G rotated 90°.

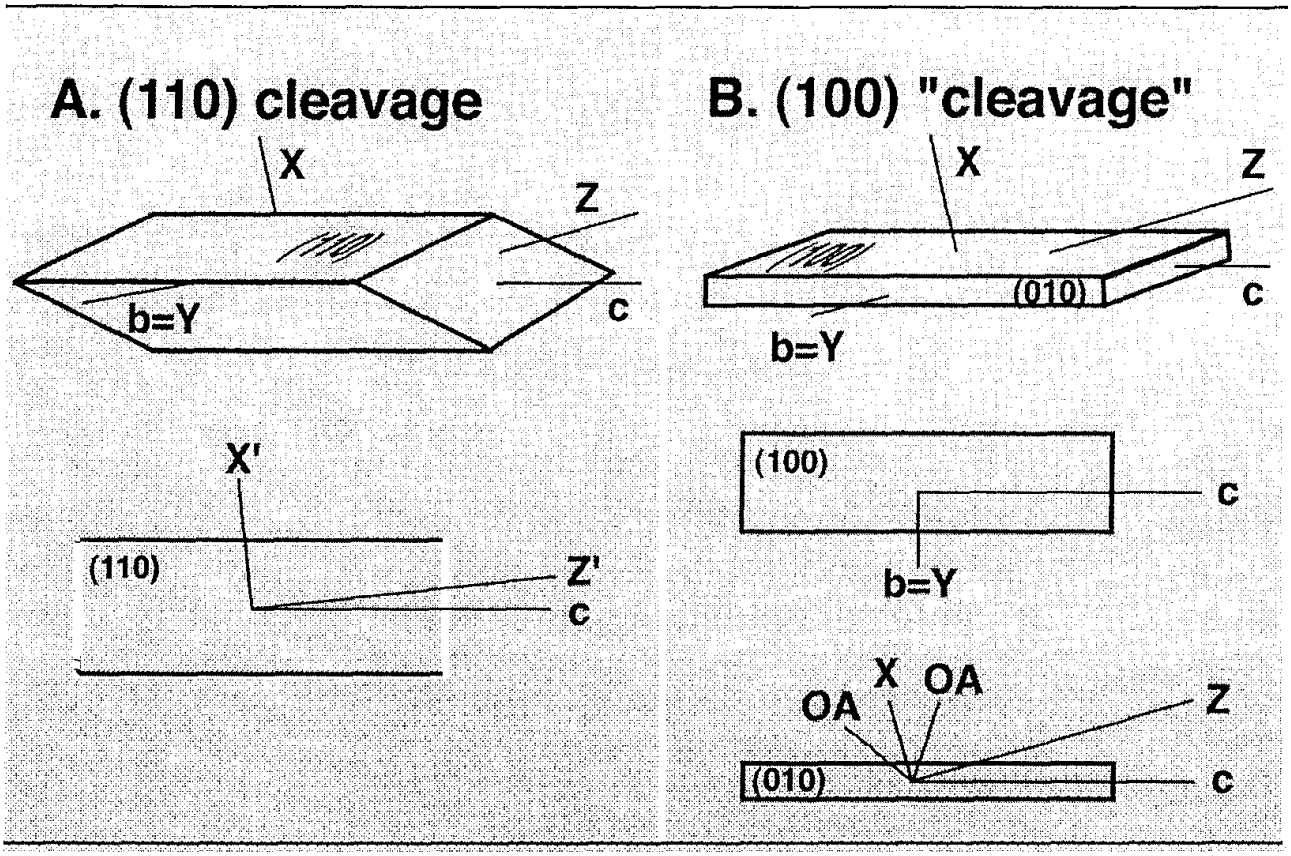

Figure 4. A) Typical cleavage fragment of a monoclinic amphibole (top) showing the (110) cleavage faces, crystallographic axes, and optical vibration directions (indicated by X' and Z'), and a similar crystal (bottom) resting on a (110) cleavage surface. B) A monoclinic amphibole (top) flattened on (100) and elongated along c, a crystal (middle) resting on (100) that would show parallel extinction (middle), and the view (bottom) looking down b on the (010) plane. The optic axes are indicated by OAs.

ole is flattened on (100), it will also show parallel extinction (Fig. 4B). Lastly, extinction positions become increasingly more difficult to observe as the particles become thinner because the retardation decreases.

Compounding this problem, especially for particles (e.g. tremolite and winchite) resting on the (100) surface, is a decrease in the birefringence of that plane based on the optical orientation of the mineral, because a circular section (isotropic view) of the indicatrix is near parallel to the microscope stage (Fig. 4B). Thus, precautions need to be taken when using extinction data for determining fibers vs. fragments. In this study we have measured the extinction angles for the differing directions for all three of our samples, in order to use these data to help interpret which form the samples have.

Su and Bloss (37) give equations for calculating extinction angles for any (hk0) plane in a monoclinic amphibole based on its optical orientation and 2V, and they warn how extinction angles are often misinterpreted. For instance, it is often assumed that the extinction angle increases from zero for a sample resting on (100) to a maximum when the sample rests on (010). This assumption is not always true (i.e., the maximum "extinction" angle may occur on some (hk0) plane other than (010)). Bandli and Gunter (13) have shown that the Libby samples exhibit (100) and (110) faces. Thus, we expect different extinction angles depending on the face the sample rested on.

The circled crystal in Figure 1A, the UI tremolite sample, is resting on (110) and exhibits inclined extinction in Figure 1B. This sample is in the orientation as shown in the bottom sketch in Figure 4A. In this orientation, the sample has an extinction angle of 13°, which is not the true extinction angle (as measured on (010)) of 16°. Table 5 summarizes the extinction data for all the samples in this study. For the UI tremolite, 99 of the particles rested on (110) and yielded

an extinction angle of 13°, while one fragment rested on (100) and gave parallel extinction. For the NIST tremolite sample in Figure 1D (the circled crystal in 1C), the crystal shows inclined extinction indicating that the sample is resting on its (110) surface. Table 5 shows that 15 of the 99 NIST tremolite fragments were in this orientation, while 22 of them showed parallel extinction. Thus, 59% of the NIST fragments with observable extinction rested on (100), while 1% of the UI tremolite fragments were flattened on (100). These particles were fragments even though they exhibited parallel extinction; they are single crystals based on morphology. Also, note that 12 of the fragment's retardations were too low to observe extinction conditions.

The major difference between the Libby samples and the NIST tremolite is the larger number of "isotropic" particles in the former. For the Libby sample, the optical orientation, and thus extinction angle, differs from the tremolite samples. The extinction angle for the Libby samples is 20°, based on the single particle data in Table 8. Also, these samples have a lower retardation; thus, more "isotropic" particles occur. At first glance, it appears that more of the Libby fragments exhibit inclined extinction than the NIST samples. This would imply that more of the Libby particles rest on (110) than (100). However, this is probably not the case. Assuming that all the "isotropic" particles result from samples resting on (100), then for the NIST sample 29% of the particles rest on (110) and 67% on (100), and for the Libby samples 26% rest on (110) and 70% on (100).

DISCUSSION - SINGLE CRYSTALS

Observations from the photographs in Figures 2 and 3 reveal a trend in the size and shape of the three samples used in the study and the morphological characteristics of the fibers vs. fragments. Figures 2A and 2B show a UI tremolite sample viewed perpendicular to its widest dimension (Fig. 2A) and its thinnest direction (Fig. 2B). Clearly this is a single crystal (parallel sides, blunt ends), and its width to thickness ratio would be high when compared to the single crystal fragments of the NIST tremolite (Figs. 2G and 2H) and the Libby amphibole (Figs. 3G and 3H) viewed in similar orientations. The samples appear similar morphologically, the aspect ratios (l/w) are higher for the NIST and Libby samples, but the width to thickness aspect ratios appear lower. The remaining five sets of photographs are of fibers bundles and masses from the NIST tremolite (Figs. 2C to 2F) and the Libby amphibole (Figs. 3A to 3F). Differences in the morphology can be observed between these fiber bundles and single crystals. It is worth noting these particles were admixed in the deposits, i.e. they occurred together in the rock.

As seen in the photos of the fiber bundles in Figures 2 and 3, some of the samples appear more fibrous when viewed perpendicular to their widest direction (left column in Figures 2, 3). When the samples are rotated 90°, some of them appear much less fibrous (right column in Figures 2, 3). This is especially true in Figures 3D and 3F. A somewhat reverse observation for the NIST tremolite samples occurred. In Table 7, 11 of 25 samples had parallel extinction on the widest section, as would be the case if they were flattened on (100), as shown in Figure 4B. However, when rotated 90° the samples never went extinct, and although they appeared morphologically to be fragments (blunt end, parallel sides), they were fibers. Some of the NIST tremolite particles in grain mounts, that we classified as fragments, are probably fibers. This observation was only possible by rotating the samples and observing them in an orientation that would rarely be seen in a grain mount.

After these initial observations, our goal was to quantify the morphology so that we could calculate aspect ratios and measure extinction conditions for different orientations. The UI tremolite was used as a non-asbestos standard. We mounted 10 samples on a spindle stage in order to measure the thickest direction, corresponding to the width of the particle, and the thinnest direction, corresponding to the thickness of the particle (Table 6). The single crystals were rotated about the spindle axis until these directions were located. Data obtained in this manner are shown in Table 6A. These data show extinction angles that would be measured when the samples were viewed perpendicular, or near so, to (110) for all the samples except #4 and #10, which were viewed perpendicular to their (100) surfaces. The average value for extinction angles measured on the width is 14° which is nearly the same as was found in the grain mounts, 13°. Next, to measure the true extinction angle we repeated the measurements made in Table 6A, except each sample was rotated to place the (010) plane in the microscope stage, yielding an extinction angle of 16° (Table 6B). As was expected, in all cases these samples exhibited parallel extinction when (100) was in the plane of the microscope stage. Regardless of which table one uses, the aspect ratios increase significantly for l/t when compared to l/w.

Table 7 lists data for the 25 particles measured for the NIST tremolite. For the NIST tremolite, the 10 single crystals yielded an extinction of 16°, which differs from the value of 12° in Table 5 for the NIST samples in the grain mount. This is because all of the single crystal particles measured on the spindle stages were flattened on (100), and some of the grain mount samples were on (110). Eleven of the 15 fiber bundles in the NIST sample showed parallel extinction on their widest direction (i.e., how they would rest in a grain mount); this confirms the observations of Wylie (21). However, based on their morphology, we would classify these particles as fragments and explain the parallel extinction by the fact that they rested on (100). As stated above, we only classified these particles as fibers when we rotated them 90° and noted they never went extinct in that orientation. We could also observe a fibrous nature in this orientation that did not exist in the other orientation but only in crossed polars (particle #7, Table 7). The remaining 4 particles never went extinct in any orientation (for example, particle #5, Table 7).

Table 8 gives the individual measurements and observations for the 50 particles of the Libby amphibole vein sample. As was the case for the NIST samples, we classified the Libby samples as either fragments or fibers based on their morphology, but there were two types of fibers in this sample: fiber bundles (e.g., particle #7, Table 8, Figs. 3A and 3B) similar to those in the NIST sample and fiber masses (e.g., particle #18, Table 8, Figs. 3E and 3F). The fiber bundles tended to have parallel extinction regardless of the orientation (i.e., the setting of the spindle stage rotation), while the fiber masses had measurable extinction angles in both the widest and narrowest directions, but the angles do not correspond to any extinction angles. There possibly was a different mode of occurrence for the masses and the bundles; however, all of these particles came from the same sample and should have undergone similar conditions of formation. The fragments yielded an average extinction angle of 20°, which is similar to that obtained from the grain mounts, although there was considerable scatter in the grain mount data.

CONCLUSION

Five amphibole samples were characterized with polarized light microscopy and the spindle stage. They include three amphibole samples from the former vermiculite mine located in Libby, Montana that were collected by the author (MEG) in October, 1999 (Libby amphibole) together with a NIST tremolite-asbestos standard (NIST tremolite) and a non-asbestos tremolite from the University of Idaho teaching collection (UI tremolite). Amphiboles from all of the samples were characterized as standard grain mounts and as single particles using the polarized light microscope and the spindle stage.

The size and morphology were determined for approximately 1000 particles in the grain mounts. Also, the length, width and thickness for 85 single particles were measured with the assistance of the spindle stage. This includes fifty (50) single particles of the Libby amphibole, twenty-five (25) of the NIST tremolite, and ten (10) of the UI tremolite. In addition, extinction angles for different (hk0) planes were measured by adjusting the particles so their crystallographic c-axes were parallel to the rotation axis of the spindle and related to the observations in the grain mounts.

Based on the regulatory counting criteria of asbestos (i.e., an aspect ratio of 3:1 or higher), 95% of the Libby amphibole, 92% of the NIST tremolite, and 48% of the UI tremolite were asbestos. Based on morphology, 36% of the Libby amphibole, 19% of the NIST tremolite, and 0% of the UI tremolite were asbestos.

One of the main goals of this study was to better characterize the Libby samples; no doubt over the next several years many similar studies will be performed. However, to date, there is only one study of the samples at Libby, and it is not in the open literature but rather in an EPA report (36). The study found that 100% of the particles had an aspect ratio greater than 3:1, 88% greater than 10:1, and 52% greater than 20:1. Again, this compares well to our study in which we found 95% greater than 3:1, 73% greater than 10:1, and 49% greater than 20:1.

The application of the spindle stage also made it easier to distinguish between fibers and non-fibrous cleavage fragments. It was found that many of the NIST tremolite particles appearing as fragments in grain mounts appear as fibers upon rotation. Extinction angles were also determined for different (hk0) planes and these data were used to help interpret the observations made on the grain mounts. These observations showed that the non-asbestos samples mainly rested on their (110) surfaces, although the smaller of these were flattened on (100); the small fragments in the NIST tremolite and Libby amphibole were predominantly flattened on (100).

APPENDIX

Table 1. Size Distribution (By Particle) for UI Tremolite, NIST Tremolite, and Libby Amphibole as Determined from Grain Mounts with a PLM

Sample	Width(μm)	Length (μm)				
		0-10	11-20	21-50	51-100	>100
UI tremolite	0-1	0	0	0	0	0
(n=100)	1.1-2	0	0	0	0	0
	2.1-5	0	0	0	0	0
	5.1-10	0	0	1	0	0
	>10	0	0	0	0	99
NIST	0-1	3	0	0	0	0
(n=99)	1.1-2	4	2	6	2	1
	2.1-5	2	7	11	6	1
	5.1-10	1	4	4	9	12
	>10	0	1	3	8	12
Libby outcrop	0-1	0	1	2	2	1
(n=298)	1.1-2	2	5	29	34	12
	2.1-5	1	3	24	45	51
	5.1-10	0	0	7	20	51
	>10	0	0	0	1	7
Libby vein	0-1	21	33	29	12	4
(n=299)	1.1-2	14	19	15	22	16
	2.1-5	6	8	14	13	27
	5.1-10	1	0	9	3	17
	>10	0	1	5	6	4
Libby float	0-1	26	34	48	14	14
(n=300)	1.1-2	18	20	33	10	14
	2.1-5	10	7	9	2	5
	5.1-10	3	6	1	5	2
	>10	0	1	8	2	8

Table 2. Percent of Fibers, Fragments, and Not Classified in the UI Tremolite, NIST Tremolite, and Libby Amphibole Determined Morphologically and Grouped by Aspect Ratio (l/w)

Sample	Aspect Ratio	Fibers(%)	Fragments (%)	Not Classified (%)	Total (%)
UI tremolite	<3	0	52	0	52
	3-5	0	29	0	29
	6-10	0	15	0	15
	11-20	0	4	0	4
	21-50	0	0	0	0
	51-100	0	0	0	0
	>100	0	0	0	0
NIST tremolite	<3	0	7	1	8
	3-5	1	18	7	26
	6-10	3	7	9	19
	11-20	9	14	10	33
	21-50	4	5	1	10
	51-100	2	1	1	4
	>100	0	0	0	0
Libby outcrop	<3	0	0	0	0
	3-5	0	3	0	3
	6-10	2	7	2	11
	11-20	8	8	7	23
	21-50	17	14	12	43
	51-100	7	3	3	13
	>100	3	2	2	7
Libby vein	<3	0	5	0.4	5.4
	3-5	0.4	8	3	11.4
	6-10	1	8	7	16
	11-20	5.5	5	11	21.5
	21-50	12	6	8	26
	51-100	6	2	1	9
	>100	10	0.7	0	10.7
Libby float	<3	0	8	0.7	8.7
	3-5	0	6.5	4	10.5
	6-10	3	4	10	17
	11-20	6	7	11	24
	21-50	12	2	10	24
	51-100	7	0.4	0.7	8.1
	>100	7	0.7	0	7.7

Table 3. Percent of Fibers, Fragments, and Not Classified in the Three Libby Amphibole Samples Combined from Table 2, and Grouped by Aspect Ratio (l/w)

Aspect Ratio	Fibers (%)	Fragments (%)	Not Classified (%)
<3	0	4.3	0.3
3-5	0.1	5.8	2.3
6-10	2	6.3	6.3
11-20	6.5	7	10
21-50	13	7	10
51-100	7	1.8	1.6
>100	7	1.1	0.6

Table 4. Summary of Classification of Fibers, Fragments, and Not Classified for the UI Tremolite, NIST Tremolite, and Libby Amphibole Based on Aspect Ratio and Morphology

	Sample	Fibers (%)	Fragments (%)	Not Classified (%)
A. Aspect Ratio	UI tremolite	48	52	-
	NIST	92	8	-
	outcrop	100	0	-
	vein	95	5	-
	float	91	9	-
	total (Libby)	95	5	-
B. Morphology	UI tremolite	0	100	0
	NIST	19	52	29
	outcrop	37	37	26
	vein	35	35	30
	float	35	29	36
	total (Libby)	36	33	31

Table 5. Summary of Extinction Measurements for UI Tremolite, NIST Tremolite, and Libby Amphibole in Grain Mounts[1]

Sample	Parallel	Inclined	"Isotropic"	Cannot Measure	Total
UI tremolite					
fragments	1	99 / 13°(4)	0	0	100
NIST					
fibers	13	0	6	0	19
fragments	22	15 / 12°(5)	12	2	51
not classified	7	1	21	0	29
total	42	16	39	2	99
Libby					
fibers					
outcrop	45	0	61	1	107
vein	18	0	83	5	106
float	18	0	83	1	102
total	81	0	227	7	315
fragments					
outcrop	16	31 / 27°(13)	73	1	121
vein	2	30 / 21°(8)	67	2	101
float	5	21 / 20°(8)	55	8	89
total	23	82	195	11	311
not classified					
outcrop	11	2	45	12	70
vein	1	0	90	1	92
float	3	0	105	1	109
total	15	2	240	14	271

[1]Entries in the table represent the number of particles in each sample that have the characteristics listed in the column heading. "Isotropic" means the particle's retardation was too low to observe extinctions. "Cannot measure" means the particle never went extinct or had wavy extinction. Also in the inclined column is the average extinction angle with its standard deviation in parentheses.

Table 6. Morphological Measurements Obtained with the Aid of a Spindle for Ten Particles of the UI Tremolite Sample[1]

A. Width (w) and thickness (t) obtained from the widest and thinnest part of the sample; extinction angles (e.a. on w and e.a. on t) were obtained in these same orientations.

Particle	l (μm)	w (μm)	t (μm)	e.a. on w	e.a. on t	l/w	l/t	w/t
1	297	114	34	12°	15°	2.6	8.7	3.4
2	381	149	82	15°	16°	2.6	4.6	1.8
3	437	133	28	12°	17°	3.3	15.6	4.8
4	403	55	27	parallel	15°	7.3	14.9	2.0
5	667	127	98	14°	16°	5.3	6.8	1.3
6	134	96	73	16°	13°	1.4	1.8	1.3
7	442	59	32	16°	11°	7.5	13.8	1.8
8	567	146	106	11°	16°	3.9	5.3	1.4
9	562	120	38	13°	18°	4.7	14.8	3.2
10	852	76	50	parallel	15°	11.2	17.0	1.5

B. Width (w100) and thickness (t010) obtained on (100) and (010) planes; extinction angles (100 e.a. and 010 e.a.) were obtained in these same orientations.

Particle	l (μm)	w100 (μm)	t010 (μm)	100 e.a.	010 e.a.	l/w100	l/t010	w100/t010
1	297	104	42	parallel	17°	2.9	7.1	2.5
2	381	140	85	parallel	17°	2.7	4.5	1.6
3	437	123	71	parallel	17°	3.6	6.2	1.7
4	403	55	27	parallel	15°	7.3	14.9	2.0
5	667	103	93	parallel	14°	6.5	7.2	1.1
6	134	74	74	parallel	17°	1.8	1.8	1.0
7	442	33	44	parallel	16°	13.4	10.0	0.8
8	567	143	81	parallel	17°	4.0	7.0	1.8
9	562	113	32	parallel	16°	5.0	17.6	3.5
10	852	76	50	parallel	15°	11.2	17.0	1.5

[1]All ten particles were fragments based on morphology, while 7 of 10 would be classified as asbestos based on aspect ratio.

Table 7. Morphological Measurements Obtained with the Aid of a Spindle for Twenty-five Particles of the NIST Tremolite Sample[1]

Particle	l (μm)	w (μm)	t (μm)	l/w	l/t	w/t	e.a. on w	e.a. on t	type
1	493	83	54	6	9	1.5	parallel	never	fiber bundle
2	169	8	6	21	28	1.3	parallel	16°	fragment
3	744	88	40	8	19	2.2	parallel	never	fiber bundle
4	709	57	22	12	32	2.6	parallel	never	fiber bundle
5	728	175	78	4	9	2.2	never	never	fiber bundle
6	815	116	84	7	10	1.4	never	never	fiber bundle
7	594	78	39	8	15	2.0	parallel	never	fiber bundle
8	226	16	12	14	19	1.3	parallel	never	fiber bundle
9	435	29	15	15	29	1.9	parallel	17°	fragment
10	756	33	19	23	40	1.7	parallel	13°	fragment
11	1023	71	16	14	64	4.4	parallel	never	fiber bundle
12	644	40	29	16	22	1.4	parallel	never	fiber bundle
13	561	9	5	62	112	1.8	never	never	fiber bundle
14	630	95	67	7	9	1.4	never	never	fiber bundle
15	445	107	52	4	9	2.1	parallel	never	fiber bundle
16	146	32	21	5	7	1.5	parallel	never	fiber bundle
17	536	18	7	30	77	2.6	parallel	16°	fragment
18	875	27	20	32	44	1.4	parallel	16°	fragment
19	521	58	36	9	14	1.6	parallel	18°	fragment
20	473	42	28	11	17	1.5	parallel	17°	fragment
21	302	49	25	6	12	2.0	parallel	15°	fragment
22	602	39	14	15	43	2.8	parallel	never	fiber bundle
23	920	28	20	33	46	1.4	parallel	15°	fragment
24	718	48	18	15	40	2.7	parallel	17°	fragment
25	579	86	35	7	17	2.5	parallel	never	fiber bundle

[1]Width (w) and thickness (t) obtained from the widest and thinnest part of the sample; extinction angles (e.a. on w and e.a. on t) were obtained in these same orientations. Particle "type" determined based on morphological characteristics.

Table 8. Morphological Measurements Obtained with the Aid of a Spindle Stage for Fifty Particles of the Libby Vein Sample[1]

Particle	l (μm)	w (μm)	t (μm)	l/w	l/t	w/t	e.a. on w	e.a. on t	type
1	333	47	21	7	16	2.2	never	22°	fiber bundle
2	530	62	47	9	11	1.3	never	never	fiber mass
3	660	68	42	10	16	1.6	17°	22°	fiber bundle
4	577	122	67	5	9	1.8	parallel	parallel	fiber bundle
5	438	116	64	4	7	1.8	parallel	parallel	fiber bundle
6	654	60	32	11	20	1.9	parallel	parallel	fiber bundle
7	537	99	54	5	10	1.8	parallel	parallel	fiber bundle
8	362	83	63	4	6	1.3	10°	parallel	fiber mass
9	387	53	52	7	7	1.0	15°	10°	fiber bundle
10	321	46	28	7	11	1.6	parallel	19°	fragment
11	428	105	47	4	9	2.2	13°	parallel	fragment
12	492	78	58	6	8	1.3	parallel	parallel	fiber bundle
13	519	77	31	7	17	2.5	9°	parallel	fiber bundle
14	940	157	101	6	9	1.6	parallel	17°	fragment
15	1341	52	31	26	43	1.6	never	never	fiber bundle
16	354	180	162	2	2	1.1	39°	7°	fiber mass
17	541	105	61	5	9	1.7	parallel	parallel	fiber bundle
18	438	141	87	3	5	1.6	14°	13°	fiber mass
19	328	168	85	2	4	2.0	20°	parallel	fiber mass
20	700	73	69	10	10	1.1	parallel	parallel	fragment
21	392	142	66	3	6	2.2	10°	parallel	fragment
22	512	73	55	7	9	1.3	parallel	parallel	fiber bundle
23	316	52	38	6	8	1.4	parallel	parallel	fiber bundle
24	467	28	13	17	36	2.2	7°	parallel	fragment
25	714	73	29	10	25	2.5	parallel	19°	fragment
26	432	91	44	5	10	2.1	parallel	22°	fragment
27	423	70	56	6	8	1.3	22°	18°	fragment
28	591	74	38	8	16	1.9	15°	10°	fiber bundle
29	1460	71	36	21	41	2.0	never	never	fiber bundle
30	481	37	13	13	37	2.8	parallel	23°	fragment
31	764	142	111	5	7	1.3	never	never	fiber mass
32	661	45	28	15	24	1.6	parallel	21°	fragment
33	772	30	24	26	32	1.3	parallel	parallel	fiber bundle
34	542	53	39	10	14	1.4	parallel	parallel	fiber bundle
35	481	35	25	14	19	1.4	parallel	15°	fragment
36	627	57	48	11	13	1.2	20°	parallel	fiber bundle
37	483	26	12	19	40	2.2	parallel	23°	fragment
38	456	36	32	13	14	1.1	parallel	22°	fragment
39	587	29	23	20	26	1.3	parallel	parallel	fiber bundle
40	728	26	12	28	61	2.2	parallel	22°	fragment
41	738	140	103	5	7	1.4	12°	parallel	fiber bundle
42	363	89	81	4	4	1.1	parallel	parallel	fiber bundle
43	309	22	21	14	15	1.0	parallel	parallel	fiber bundle
44	546	74	40	7	14	1.9	parallel	23°	fragment
45	321	10	8	32	40	1.3	parallel	parallel	fiber bundle
46	327	50	44	7	7	1.1	parallel	21°	fragment
47	375	40	24	9	16	1.7	parallel	23°	fragment
48	710	50	34	14	21	1.5	parallel	parallel	fiber bundle
49	497	20	7	25	71	2.9	parallel	16°	fragment
50	703	17	17	41	41	1.0	27°	20°	fiber bundle

[1]Width (w) and thickness (t) obtained from the widest and thinnest part of the sample; extinction angles (e.a. on w and e.a on t) were obtained in these same orientations. Particle "type" determined based on morphological characteristics.

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(26) Veblen, D.R. and Wylie, A.G. "Mineralogy of amphiboles and 1:1 layer silicates"; In *Reviews in Mineralogy: Health effects of mineral dusts*; G.D. Guthrie, Jr., B.T. Mossman Eds.; 1993; 28, 61-138.

(27) Ross, M., Nolan, R.P., Langer, A.M., and Cooper, W.C. "Health effects of mineral dusts other than asbestos"; *In Reviews in Mineralogy: Health effects of mineral dusts*; G.D. Guthrie, Jr. and B.T. Mossman Eds.; 1993; 28, 361-409.

(28) Van Oss, C.J., Naim, J.O., Costanzo, P.M., Giese, R.F., Jr, Wu, W., and Sorling, A.F. "Impact of different asbestos species and other mineral particles on pulmonary pathogenesis"; *Clays and Clay Minerals* **1999**, 47, 697-707.

(29) Gunter, M.E. "Quartz – the most abundant mineral species in the earth's crust and a human carcinogen"; *Journal of Geoscience Education* **1999**, 47, 341-349.

(30) Guthrie, G.D., Jr. "Mineral properties and their contributions to particle toxicity"; *Environmental Health Perspectives* **1997**, 105, 1003-1011.

(31) Guthrie, G.D., Jr. "Mineralogical factors affect the biological activity of crystalline silica"; *Applications to Occupational Environmental Hygiene* **1995**, 10, 1126-1131.

(32) Guthrie, G.D., Jr. "Biological effects of inhaled minerals"; *American Mineralogist* **1992**, 77, 225-243.

(33) Wylie, A.G. and Huggins, C.W. "Characteristics of a potassian winchite-asbestos from the Allamoore Talc District, Texas"; *Canadian Mineralogist* **1980**, 18, 101-107.

(34) Gunter, M.E. and Twamley, B. "A new method to determine the optical orientation of biaxial minerals: A mathematical approach"; *Canadian Mineralogist* **2001**, 39, 1701-1711.

(35) Wylie, A.G. and Schweitzer, P. "The effects of sample preparation and measuring methods on the shape and shape characterization of mineral particles: The case of wollastonite"; *Environmental Research* **1982**, 27, 52-73.

(36) Atkinson, G.R., Rose, D., Thomas, K., Jones, D., Chatfield, E.J., and Going, J.E. "Collection, analysis and characterization of vermiculite samples for fiber content and asbestos contamination"; Reports to EPA 1982, Project 4901-A32, Contract No. 68-01-5915.

(37) Su, S.C. and Bloss, F.D. "Extinction angles for monoclinic amphiboles and pyroxenes: a cautionary note"; *American Mineralogist* **1984**, 69, 399-403.

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June 6, 2006

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Response, Planning and Assessment Branch
Superfund Division
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United States Environmental Protection Agency
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RE: Evaluation of EPA's Analytical Data from the El Dorado Hills Asbestos Evaluation Project

This letter serves as RJ Lee Group's response to your letter of March 9, 2006 requesting information related to RJ Lee Group's report on the "Evaluation of EPA's Analytical Data from the El Dorado Hills Asbestos Evaluation Project". RJLG is pleased to be provided the opportunity to clarify any ambiguity in its earlier report and to respond to the comments and concerns expressed in the EPA Region 9 response. We have attempted to provide complete, clear, and concise descriptions of the technical basis of our approach to the issues and the corresponding underlying methodologies.

RJLG has prepared responses to your requests in the same order that they were presented in your March 9th letter. Detailed supporting documentation is provided in separate exhibits. If you have any questions regarding our response please feel free to contact me directly at (724) 387-1810.

Sincerely,

Richard J. Lee, Ph.D.
President & CEO

Table of Contents

Executive Overview

In a letter[1] (hereafter referred to as Meers) dated March 9, 2006, EPA Region 9 requested that RJ Lee Group (RJLG) supplement its commentary on the El Dorado study. Subsequent to its March 9, 2006 letter, EPA Region 9 published a response to many of the issues raised in the RJLG report. Additionally, Mr. Greg Meeker[2] of USGS (and on behalf of EPA) provided Ms. Vickie Barber with comments on portions of the RJLG analysis.

RJLG's responses to the major issues and questions raised in the documents mentioned above are summarized here. Detailed responses to individual questions are provided in the following sections, along with backup documentation.

The most substantive issue raised in EPA Region 9's response is a proposed modification to the analytical methods for defining and measuring asbestos in a naturally occurring environment. Region 9 proposes that: *"amphibole or serpentine minerals that are asbestiform and meet the size definition of PCM fibers, should be counted as asbestos – regardless of the manner by which they were formed."*[3]

EPA Region 9's definition implies that any amphibole or serpentine particle longer than 5 μm, which has a 3:1 aspect ratio, is treated as asbestos. Region 9 further suggests that the risk assessments based on commercial asbestos exposures are suitable for assessing the significance of airborne exposures to amphibole and serpentine particles meeting this definition in a naturally occurring environment, regardless of how the particles were formed.[4]

EPA Region 9's definition of asbestos would have significant economic impact and analytical implications. The Region 9 definition of asbestos

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[1] D. Meers (2006). Letter to W. Ford and to R. Lee, dated March 9, 2006.

[2] G. Meeker (undated). Response to questions submitted by Dr. Vicky Barber, Superintendent of Schools, El Dorado County, California regarding asbestiform amphiboles. Hereafter referred to as Meeker 2006.

[3] EPA Region 9, April 20, 2006. Response to the November 2005 NSSGA Report. Page11.

[4] Reference needed.

would represent a significant shift in the policy of EPA and would likely put the policy of the Agency in conflict with OSHA, other Federal Agencies and international organizations.[5,6,7,8] Given the widespread occurrence of nonasbestos amphiboles and serpentines throughout the US (Figure 1), the adoption of the Region 9 definition on a national basis could have significant economic consequences for both public and private organizations, including school districts in newly developing areas such as El Dorado.[9] Commercial entities including real estate developers, the crushed stone industry and the mining industry would have to commit additional resources to monitoring product quality and potential exposures using sophisticated methods. By classifying nonasbestos particles as asbestos, exported products may be considered "asbestos-containing" in other countries[10] while not assuring public health.

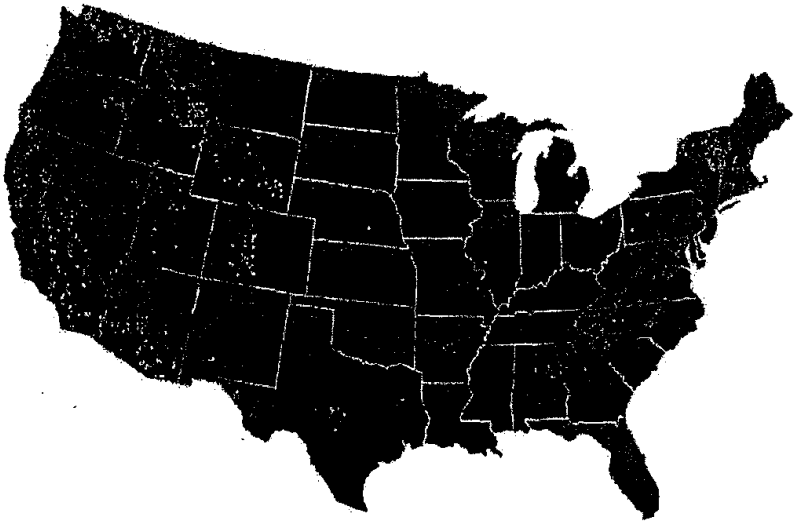

Figure 1. Green shaded areas demonstrate regions where amphibole and pyroxene minerals may likely occur. Yellow dots represent possible locations of where asbestiform minerals may occur according to the USGS database.

The ability of methods to identify exposures to asbestiform fibers would be diminished since analytical[11] and monitoring resources could be

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[5] 1992 OSHA Ruling, preamble

[6] OSHA ID-191

[7] Beard to Sally Stasnet letter

[8] CPSC Crayon Report

[9] V. L. Barber letter to Stephen L. Johnson, March 10, 2006.

[10] H. Benjelloun (2000). "The European Union's Ban on Asbestos", *The Synergist*, August 2000, p. 20 – 25.

[11] As of June 1, 2006, there are 82 NVLAP accredited TEM laboratories. http://ts.nist.gov/ts/htdocs/210/214/scopes/temtm.htm.

consumed measuring exposures to the nonasbestos particles instead of asbestos fibers. Such nonasbestos particles are far more prevalent in the environment than those meeting the geological/mineralogical definition of asbestos. This could result in undercounting and increased uncertainties in the estimate of exposures to 'long, thin' asbestos particles[12], and the consumption of the limited available resources remediating widespread occurrences of nonasbestos minerals, for which RJLG is unaware of any studies documenting a public health benefit.

A second argument in Region 9's response is that the geological/mineralogical definition of asbestiform (*"bundles of flexible, readily separable fibers, of nearly constant diameter, having parallel sides and a high aspect ratio"*) is applicable to environments with commercial asbestos exposures, but not applicable to environments with exposures to unprocessed naturally occurring asbestos.

The geological/mineralogical definition of asbestos is an essential element of the process by which potential asbestos exposure is detected and assessed. Naturally occurring asbestos is identified through the observation of localized veins of asbestos fibers with the geological/mineralogical characteristics of asbestos and can be found in El Dorado County and other parts of California, Figure 2. Broadening the definition of asbestos would result in increased confusion about the location and extent of asbestos minerals since laboratories may report the presence of asbestos in samples of ordinary rock which contain nonasbestos amphibole minerals.

[12] EPA Region 9, April 20, 2006. Response to the November 2005 NSSGA Report. Page 13: "By concentrating on PCME structures, other fiber size classifications may not have been counted to statistical significance. This may have resulted in under counts of other fiber sizes (e.g., the "Berman Crump" protocol fibers referred to in the R. J. Lee Report)."

Figure 2. Asbestiform amphibole collected from Harvard Way as observed under low magnification optical microscopy. Sample collected within several hundred yards of test site.

Numerous epidemiology studies, animal experiments and risk assessments have documented the link between asbestos fibers and disease (asbestosis, lung cancer, and mesothelioma). ***All of these studies have documented this link on the basis of an exposure to commercially-produced asbestos, fibers that were mined, processed, and sold as a pure mineral product with only nominal contamination by non-asbestos fragments.***

EPA has conducted a review to establish an integrated risk assessment model (IRIS[13]). The epidemiology studies referenced in IRIS documents (with the exception of chrysotile miners and millers) are based on exposures to commercial asbestos fibers, not to mixed atmospheres of asbestos and nonasbestos particles as claimed by EPA Region 9.[14] The only epidemiology studies that may have included a substantial population of nonasbestos mineral fibers were those of the chrysotile miners and millers, a study that was explicitly excluded from the IRIS model. Berman has noted that extension of a risk model to new environments is dependent on the ability to measure the same asbestos fiber population assessed in establishing the risk.[15,16]

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[13] US Environmental Protection Agency, Integrated Risk Information System (IRIS), http://www.epa.gov/iris/subst/0371.htm.

[14] EPA (2006), page 2.

[15] EPA Tech Support Document

[16] D. W. Berman (2006). "Evaluation of the Approach Recently Proposed for Assessing Asbestos-Related Risk in El Dorado County, California", page 14.

Inspection of mines and quarries for the occurrence of asbestos is performed as a means of ensuring that products will be asbestos-free. Field geologists and mining engineers rely on the geological and mineralogical definition of asbestos in performing inspections for the presence of asbestos and potential for exposure to asbestos. Current regulations and analytical methods measure the concentration of the asbestos minerals. Analytical methods, including OHSA 191 and EPA 600/R-93/116, define asbestos in terms of its geological/mineralogical characteristics (aspect ratio, flexibility, bundles). Yamate and the EPA asbestos atlas use microscopic characteristics of asbestiform fibers to define the attributes of asbestos. (Photo from Atlas showing images and SAED pattern of asbestos fibers with characteristics of 0.53 nm spacing defined by Yamate as characteristic of amphibole asbestos)

Non-asbestos amphiboles and serpentines have not been associated with Asbestos Disease. The physical characteristics of geologically asbestiform fibers are significantly different than those of nonasbestos fragments. Over the last 40 years a significant body of literature has accumulated demonstrating that potency is most associated with populations of "long-thin" fibers.[17] Populations of nonasbestos fragments have different characteristics than asbestiform populations

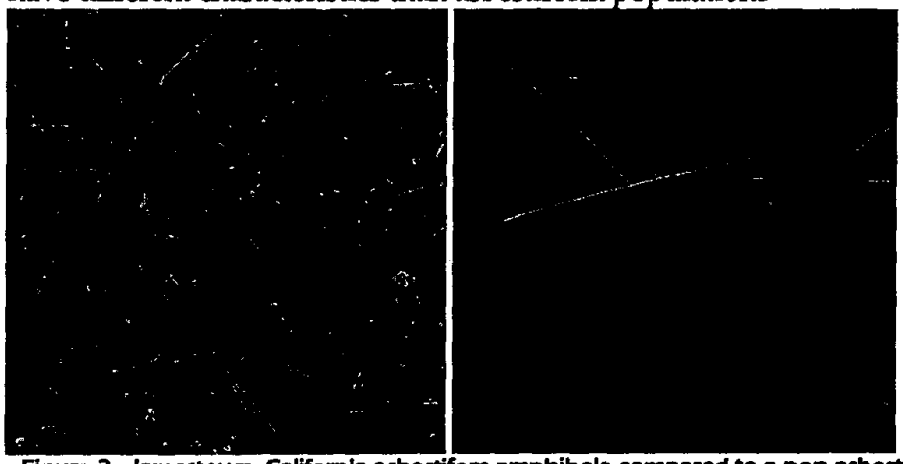

Figure 3. Jamestown, California asbestifom amphibole compared to a non-asbestiform amphibole population showing morphological differences.
Legend: Red-massive Blue-prismatic Yellow-bundles Green-acicular Purple-fiber

[17] Stanton, Davis, Crump, Berman, Wylie, Kleumphal

Nonasbestos populations have few thin particles when compared to asbestos populations, including those found in the El Dorado Study, Figure 4.

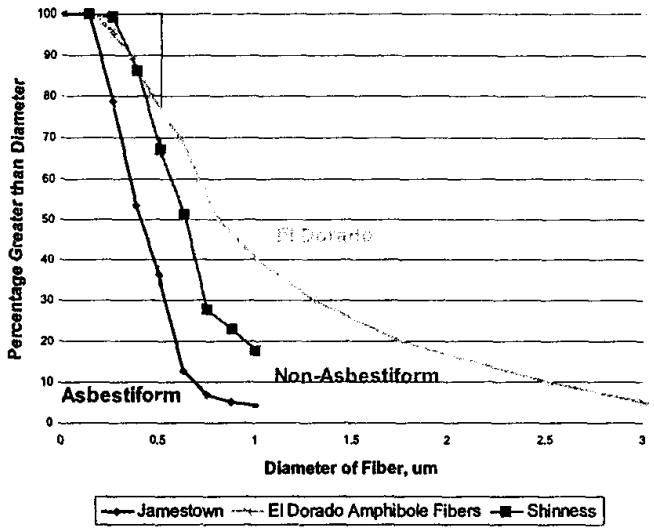

Figure 4. Comparison of asbestiform amphibole from Jamestown, non-asbestiform amphibole from Shinness, and El Dorado amphibole particle size populations.

Inspection of mines and quarries for the occurrence of asbestos is performed as a means of ensuring that products will be asbestos free. Field geologists and mining engineers rely on the geological definition of asbestiform in performing inspections for the presence of asbestos and potential for exposure to asbestos. Current regulations and analytical methods measure the concentration of the asbestiform varieties of minerals. Analytical methods, including OSHA ID-191 and EPA 600/R-93/116, define asbestos in terms of its geological characteristics (aspect ratio, flexibility, bundles). Yamate and the EPA asbestos atlas use microscopic characteristics of asbestiform fibers to define the attributes of asbestos.

Nonasbestos amphiboles and serpentines have not been associated with asbestos disease. OSHA examined the cleavage fragment issue in 1992[18] and concluded that cleavage fragments do not present an asbestos risk. Hard-rock mines and quarries often contain nonasbestos amphiboles including hornblende, actinolite and tremolite, such as that found in the El

[18] OSHA 1992 preamble, found at http://www.osha.gov/pls/oshaweb/owasrch.search_form?p_doc_type=PREAMBLES&p_toc_level=1&p_keyvalue=Asbestos~(1992~-~Original)

Dorado study, but there has been no asbestos disease in the mine workers.[19] The NSSGA has provided the El Dorado County School District and the EPA a number of studies[20] conducted since 1992 that support this conclusion.

Figure 5. Amphibole Particles recently counted as asbestos fibers from a Taconite mine where the laboratory failed to follow proper counting rules.

Current analytical procedures use the geological definition of asbestos fibers to distinguish asbestos fibers from nonasbestos amphiboles and serpentines. Asbestos counting methods published over the past 30 years define asbestos as the mineral fibers in the asbestiform habit of six specified mineral species. Asbestiform fibers, regardless of their regulatory status, have unique shapes, crystallinity, and surface texture as well as optical properties that differentiate them from nonasbestos particles.[21] Asbestos fibers are characterized by parallel sides, curvature with sufficient length, and proper termination of the ends. When an analyst utilizes these characteristics, as defined in the analytical procedures, to conduct the analysis, the vast majority of nonasbestos particles are excluded from the count of asbestos fibers.

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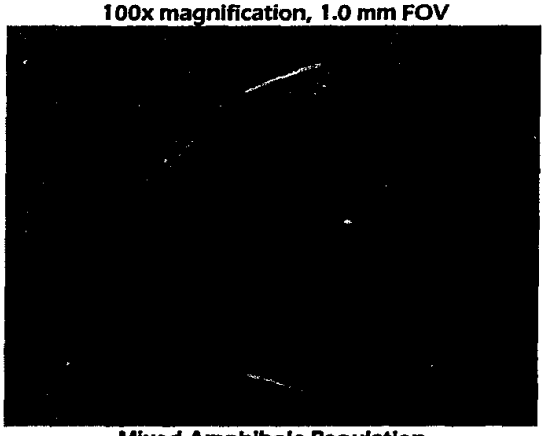
[19] Homestake, Taconite, Vanderbilt

[20] NSSGA CD provided to V. Barber.

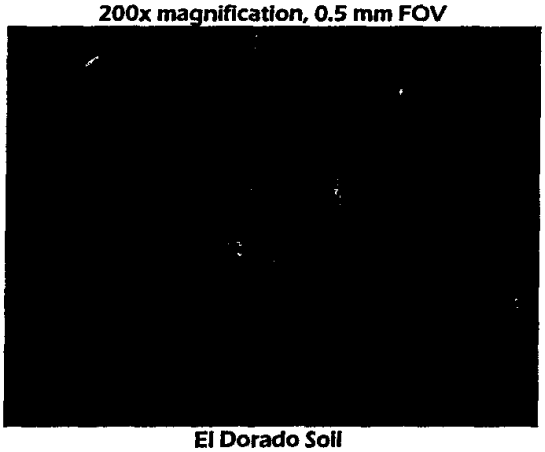
[21] Campbell, Wylie, Beard, Lee/Fisher

Asbestiform San Andreas Tremolite
100x magnification, 1.0 mm FOV

Asbestiform San Andreas Tremolite Particles
200x magnification, 0.5 mm FOV



Mixed Amphibole Population
200x magnification, 0.5 mm FOV



El Dorado Soil
40x magnification, 2.5 mm FOV

Figure 6. Comparison of Asbestiform and Non-Asbestiform Amphiboles from California compared to the Amphiboles observed in El Dorado Soils.

The combination of these defining characteristics can be used in the PLM, SEM or TEM to distinguish asbestiform from nonasbestos amphiboles with a high degree of reliability.

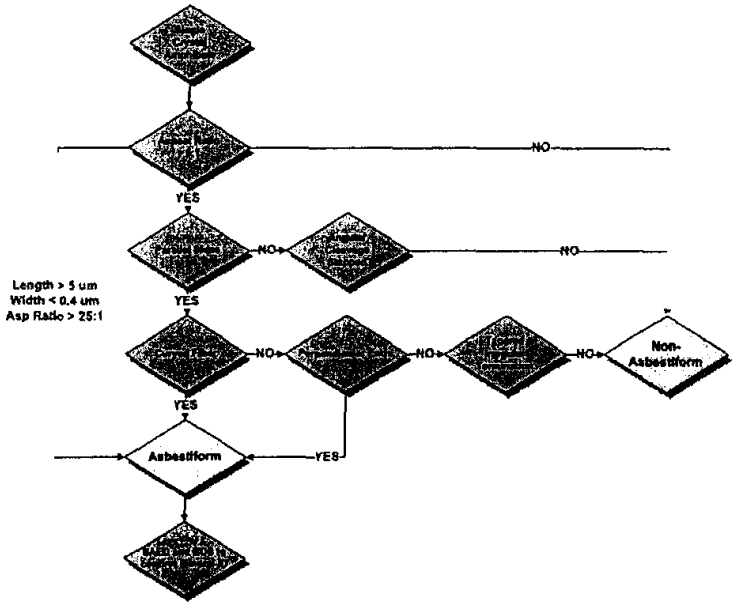


Figure 7. Proposed Methodology for determination of mineral habit. Methodology of left side of figure would include most asbestiform fibers

Many of the methods, however, have been validated and used primarily for assessment of commercial asbestos fibers. Laboratories whose principal business is analyzing samples taken after asbestos abatement projects may need supplemental guidance and training to effectively analyze samples from mixed mineral environments.

The amphibole particles in the El Dorado report do not have the characteristics of geologically asbestiform particles. The length of fibers in the El Dorado data set is directly proportional to the width of the fibers. The average aspect ratio is about 6.3 to 1 for fibers longer than 5 μm. Only a portion of the particles that are three times longer than they are wide have parallel sides. The amphibole particles in the soil have a high index of refraction characteristic of hornblende, not actinolite or tremolite, and extinction angles uniformly above 10 degrees. These particles exhibit the characteristics of a population of cleavage fragments not asbestiform particles.

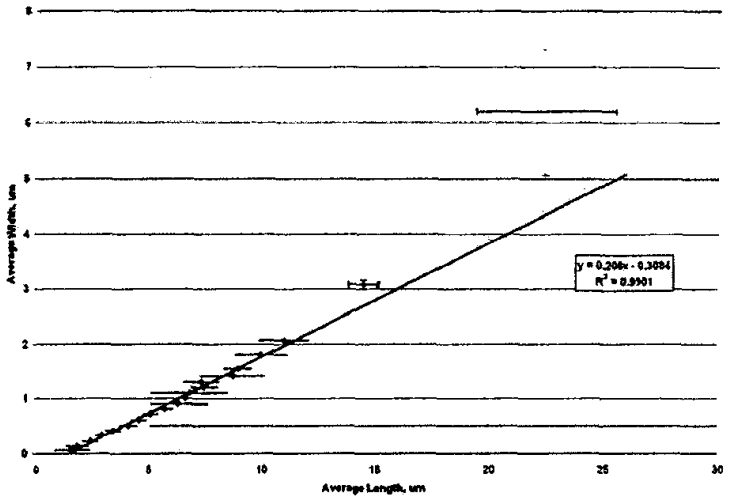


Figure 8. Relationship of average lengths and widths of amphibole fibers observed in El Dorado indicating that the population is not asbestiform. The Berman-Crump Risk Fiber region is highlighted in yellow.

Response to EPA Data Production Request

Sections 1 – 13 respond to the specific contained in the Meers letter. For clarity, the request is placed at the beginning of the section in italics.

1.0 EPA Air Samples

Please list all the analytical techniques (including full method name and reference number) that the R. J. Lee Group [sic] used to evaluate the EPA air samples. Please provide all documents generated as a result of that analysis including: laboratory count sheets, laboratory notes and logbook pages, sketches, images, spectra, diffraction patterns, chain-of-custody forms and other sample tracking sheets. Provide the same information for any and all quality control ("QC") samples analyzed along with the investigative samples and all required calibrations and other technical notes generated during the review of the EPA air samples. Please include a general description of the instruments (including make and model) used in the analyses and provide the analyst's names and qualifications to perform analysis for each of the analytical/preparation methods employed. If no actual laboratory analysis was performed, or if, in addition to laboratory analyses, other reviews were performed, identify such reviews, explain the steps taken for conducting the review, and provide all available documents of such reviews.

1.1 Evaluation of EPA Analyses

RJLG has not yet received air filters analyzed by LabCor, and agrees with EPA Region 9, that a more comprehensive review could be performed if the actual grids or filters analyzed by LabCor were made available by EPA. Thus, RJLG has no relevant records to provide vis a vis any independent RJLG analysis of EPA air samples.

1.2 Evaluation of EPA Data

Due to an incomplete data production, RJLG's review of EPA's air sample data was conducted over a six-month period of time, and included an informal, preliminary presentation of the data evaluation at the 2005 ASTM Johnson conference (see attachment).

1.2.1 Additional Reviews Performed

RJLG received PDF copies of Lab/Cor's TEM count sheets which included particle length and width data, and reference to x-ray spectra and quantification data, and SAED patterns and identifications.

RJLG evaluated the replicate and duplicate counts performed by Lab/Cor and compared them to accepted performance criteria for such counts.

RJLG evaluated the particle size data to determine if the population of particles had the known characteristics of asbestos populations.

RJLG evaluated Lab/Cor's conversion of the EDS chemical analysis to atoms per formula unit, and independently performed such a calculation to evaluate the mineral assignments made as a result of the conversion. RJLG compared the resulting aluminum concentrations with values found in the literature for asbestiform and nonasbestos calcium amphiboles.

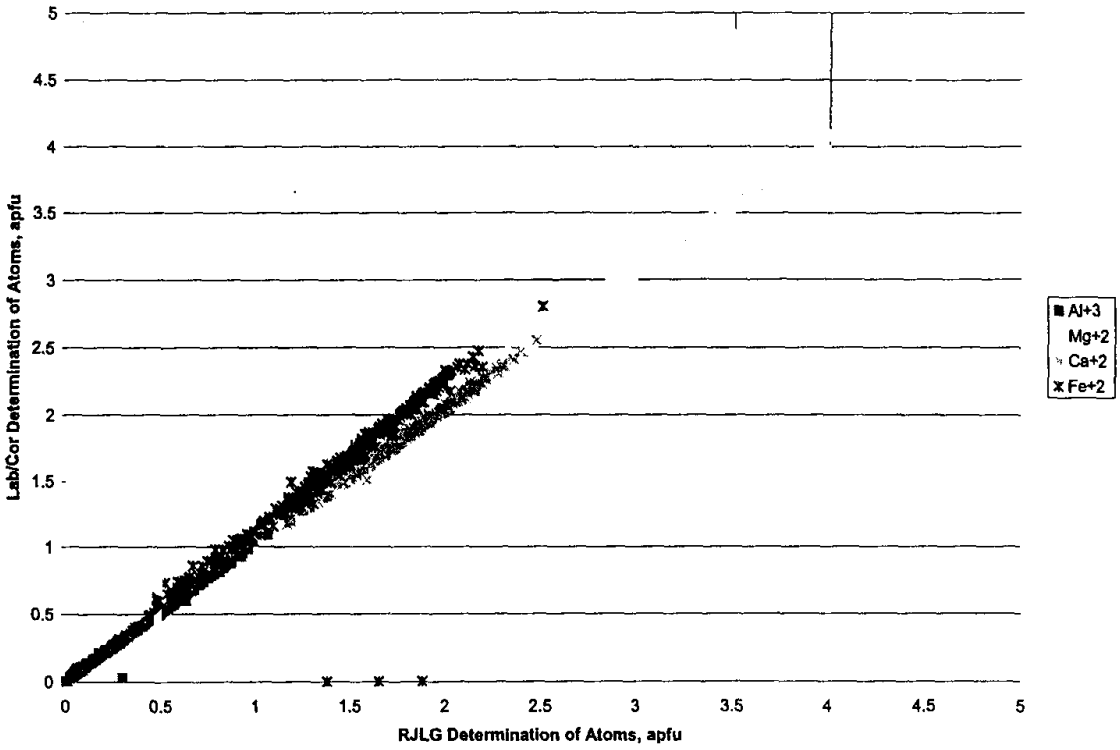
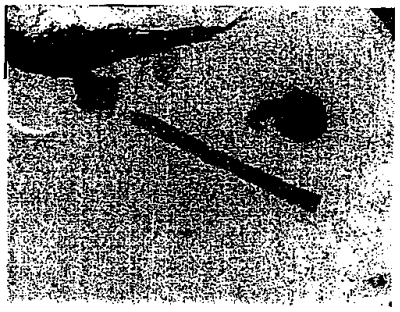
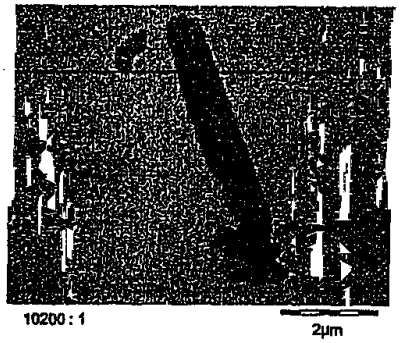


Figure 2. A comparison of the number of atoms for four elements calculated by RJ Lee Group and Lab/Cor. The data are based on the quantitative EDS data reported by Lab/Cor. The data show excellent agreement in the number of atoms, with minor variations in iron content due to the assumed valence state of iron.

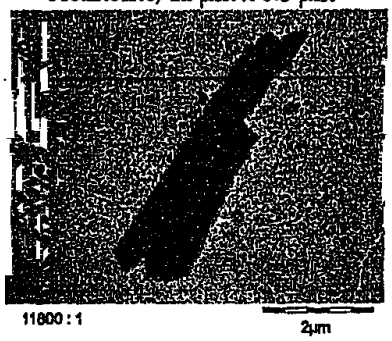
RJLG reviewed the photographs of particles counted as asbestos by Lab/Cor in light of the counting rules specified in the ISO 10312 method.



Sample AAMS-D09-100504, #5,
Actinolite, 12 μm x 0.8 μm



Sample SRA-R03-100704, #4,
Actinolite, 4.8 μm x 0.65 μm



Sample NRA-R05-101004, #1,
Actinolite, 5.2 μm x 1.2 μm

Figure X.

RJLG evaluated the SAED patterns provided by Lab/Cor, re-measured the patterns when possible, and compared the results of RJLG's SAED pattern identification with Lab/Cor's results.

RJLG evaluated the reliability of Lab/Cor's EDS chemical data of individual particles by considering the possibility that the silicon content was overestimated.

RJLG personnel traveled to El Dorado Hills, and with the assistance of local officials there, collected specimens of probable asbestos and nonasbestos minerals, then analyzed those samples to compare the appearance and chemistry of the particles with photographs produced by Lab/Cor and with particles found in splits of EPA soil samples.

1.2.2 Steps Taken for Conducting Additional Reviews

RJLG graphed the particle size data in various formats and compared those expected for asbestos and nonasbestos particle populations.

RJLG plotted the reported concentrations for aluminum on a ternary plot and compared those concentrations with the upper limit on the concentration of aluminum in asbestiform amphiboles reported in Verkouteren and Wylie[34].

RJLG reviewed the quantitative output of Lab/Cor's conversion of the observed chemistry (weight percent of oxides) to the number of atoms in an amphibole mineral used to determine the IMA classification[30] for the particle.

RJLG reviewed Lab/Cor's SAED pattern analysis, noting that numerous patterns had measurements inconsistent with results that would be expected from amphiboles indicating potential issues with the measurement process and/or misidentification of the mineral. RJLG found that Lab/Cor systematically chose actinolite as the best mineral match for an SAED pattern even if hornblende or other minerals were a better fit of the data.

1.2.3 Documentation of Additional Reviews

Naturally Occurring Asbestos from El Dorado has the characteristics of asbestos described by EPA as the target analyte in the QAPP. Naturally occurring amphibole asbestos (NOA) specimens (tremolite) collected from an outcrop near one of the El Dorado Hills test sites, as well two other locations in California, were analyzed by RJLG and the results are described section 14.

The NOA specimens had the characteristics of asbestos as described in the EPA QAPP, and as recognized in all regulations and methods of analysis as diagnostic of asbestos, namely bundles of fine, flexible, readily separable fibers. Particles photographed by Lab/Cor showed none of these characteristics. (Attachment xx) Each of the NOA samples analyzed by RJLG had both a population of asbestos and nonasbestos particles as defined by Campbell[22], Wylie[23], and others.

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[22] Campbell, W. J., R. L. Blake, L. L. Brown, E. E. Cather, J. J. Sjoberg (1977), 'Selected Silicate Minerals and Their Asbestiform Varieties - Mineralogical Definitions and

NOA from El Dorado is tremolite asbestos, not actinolite or hornblende. The NOA specimens from California, including El Dorado Hills, had the composition of tremolite. The fibers had no significant aluminum or iron concentration. These particles have different shapes and chemistry than those reported as amphiboles by Lab/Cor, 80 percent of which contained a significant amount of aluminum or iron. (Attachment xx)

Natural amphibole asbestos occurrences have been the subject of debate in El Dorado County for nearly a decade. Veins of asbestos that outcropped locally have been identified, excavated and covered. It is good laboratory and field practice to obtain and analyze reference materials, particularly in complex mineral environments.[24] Had EPA obtained, and Lab/Cor analyzed, material specimens from the known asbestos veins that outcropped locally in El Dorado County, EPA would have observed that the particles Lab/Cor reported in the samples from the EPA test site were not consistent with those present in the region.

1.2.4 Data Production

RJLG's initial review was performed on data submitted by the EPA to the El Dorado Hills (EDH) School District and community members. Although RJLG requested a complete set of data and documents from the EPA; only a partial set was sent on August 8, 2005 by Karen Ladd (Ecology and Environment, Inc.). The data were received on two compact discs: "Raw Data Summaries Grouped by Activity" and "Raw Data Summaries Plus Detail Grouped by Activity". The information contained on the "Detail" CD was the same information that was previously provided to RJLG by community members. The information contained on the CDs was limited to a summary page for each sample analysis and the associated count sheets for the sample.

On September 6, 2005, RJLG received a more complete set of data on two CDs. The data on each CD were organized by Lab/Cor job number. Within each job number was the previously provided information along

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Identification-Characterization', Bureau of Mines, United States Department of Interior, Information Circular 8751, pp. 1-55.

[23] A.G. Wylie (19XX). "Discriminating Amphibole Cleavage Fragments from Asbestos: Rationale and Methodology ", Exposure Assessment and Control Asbestos/Other Fibrous Material, p. 1065 – 1069.

[24] Steel, Yamate

with a cover letter summarizing the job, diffraction patterns and EDS spectra for selected particles, an evaluation sheet for each chemistry, and some limited photographs of the particles observed in the microscope.

All data were produced as scanned images of count sheets, diffraction pattern computer screen dumps, chemistries and the evaluations of the chemistries. All of the pages were stored in a pdf format.

1.2.5 Data Entry

Each file on the September 6 CDs were printed and stored for use as reference materials.

The count sheet data on the CDs were converted to spreadsheets using a program called Able2Extract. After conversion, the data were manually verified for each count sheet by comparing the converted data with the pdf sheet. All of the columns on the count sheet (except those listed as "Count Categories") were reviewed and verified. (The "Count Categories" column received a cursory review.) Any revision to the converted data was made on-line to the appropriate spreadsheet. Only the Lab/Cor project files listed as "direct preparation" were converted – the indirect preparation files were not evaluated because the indirect procedure is known to modify the size and number of mineral particles. A final data validation step included the examination of the data for all particles that fit into one of five categories and the correction of observed errors: 1) missing values; 2) data entries inconsistent with ISO 10312 counting rules; 3) particles with aspect ratios less than 3:1; 4) amphibole particles thinner than 0.1 μm or wider than 3 μm; and 5) particles with lengths shorter than 0.5 μm or longer than 20 μm.

1.2.6 Diffraction Patterns

The diffraction (SAED) pattern data were manually entered into spreadsheets, listing the fiber identification, fiber dimensions, reported zone and highlighted matching zone for each diffraction pattern. All data entries were verified by a second person with any corrections made on-line to the spreadsheet.

1.2.7 Spectra

The reported chemistry data (EDS) were manually entered into a spreadsheet. The information for each EDS included a particle identification, the reported chemistry (oxides percentages), assigned

atoms, and mineral identification. All data entries were verified by a second person with any corrections made on-line to the spreadsheet.

2.0 Soil Preparation Methodology

Please list the soil preparation methods the R.J. Lee Group [sic] used to prepare splits of the EPA soil samples for analysis. A complete response to this inquiry will include information on whether a microscopic/stereoscopic analysis of the soil samples was conducted prior to any sample handling or preparation, drying times, moisture content, grinding (types and brands of grinders), sieving (sizes), and any other information required to provide a complete description of the preparation procedure used by the R.J. Lee Group [sic] for splits of the EPA soil samples.

RJLG received 23 soil samples from Youngdahl Consulting Group on August 26, 2005 (Fed Ex shipment 791183164750). These samples were identified as split samples that had been provided to Youngdahl by Ecology & Environment, Inc. RJLG did not split these samples or perform an examination of the samples prior to any sample handling or preparation. Upon receipt, the samples were assigned RJLG sample numbers and forwarded to our optical laboratory for analyses (see section 3).

RJLG prepared the soil samples for analysis and performed PLM and XRD analysis for asbestos in accordance with the published methods, but noted the presence of hornblende, a nonasbestos amphibole mineral. RJLG performed elutriation tests on selected soil samples in accordance with the Berman-Kolk method. RJLG prepared and analyzed TEM samples from four of the soil samples in accordance with published methods.

RJLG identified and photographed particles having a length to width ratio greater than 3:1 in the SEM and TEM.

RJLG prepared samples for and performed Computer-controlled Scanning Electron Microscopy on soil samples.

2.1 Drying Times

The soil samples received were visibly dry and were not further dried prior to analyses. All results are reported on an "as-received" basis.

2.2 Grinding

The samples were not ground prior to analysis by RJLG in order to homogenize or otherwise produce a uniform particle size.

2.3 Sieving

On a follow-up analysis of soil sample NYT-SJ3-100804-FG2, the sample was dry screened using a 120 mesh (125 μm) sieve (from Gilson Company, Inc.). The screening was performed in general accordance with ASTM D4749.[25]

2.4 Other

Selected samples were also analyzed by scanning electron microscopy (SEM) or by X-ray Powder Diffraction (XRD) in accordance with published methods (see section 4). Prior to submitting the samples for these analyses, the samples were subdivided in general accordance with ASTM C702 (Standard Practice for Reducing Samples of Aggregate to Testing Size).

3.0 Analytical Methodology

Please list the analytical techniques (including full method name and reference number) that the R.J. Lee Group used to evaluate splits of the EPA soil samples. Please provide all documents generated as a result of that analysis including: laboratory count sheets, laboratory notes and logbook pages, sketches, images, chain-of-custody forms and other sample tracking sheets. Also include the same information for any and all QC samples analyzed along with the investigative samples, all required calibrations, and any other technical notes generated during the analyses of splits of the EPA soil samples. Please include a general description of the instruments (including make and model) used in the analyses and provide the analysts' names and qualifications to perform an analyses for each of the analytical/preparation techniques employed. Regardless of method, please provide laboratory count sheets and a full description of all exceptions or modifications to the analytic techniques.

The soil samples were evaluated using the procedures described in the EPA method listed at 40 CFR Part 763, Appendix E to Subpart E (Interim

[25] ASTM (2002). Standard Test Method for Performing the Sieve Analysis of Coal and Designating Coal Size, D4749.

Method for the Determination of Asbestos in Bulk Insulation Samples). The scanning electron microscopy analyses of the samples were performed in general accordance with the analytical portion of ISO 14966 (Ambient air — Determination of numerical concentration of inorganic fibrous particles — Scanning electron microscopy method). The weight percent of the fine soil particulate was determined in general accordance with the analytical portion of ASTM D5756 (Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Mass Concentration). The soil samples were also evaluated using the Berman-Kolk elutriator method for releasable particulate (Modified Elutriator Method for the Determination of Asbestos in Soils and Bulk Materials, Berman and Kolk, 2000).

The analytical reports for the soil samples are included in the Attachment. The supporting documentation is shown in the following sections.

3.1 Documents generated as a Result of Analysis

PLM count sheets are found in the Attachment. Only hornblende was identified in the soil samples. RJLG compared the chemistry and appearance of the particles found in the soil samples with asbestos fibers from two samples of amphibole asbestos collected in California, including one from El Dorado County.

3.2 Laboratory Count Sheets

The laboratory count sheets for the PLM and TEM analyses are included in the Attachment. The tracings from the XRD analyses are in the Attachment.

3.3 Laboratory Notes and Logbooks

Other than the notes recorded on the count sheets, there is no laboratory notes related to the subject samples. Logbooks are not used in the PLM and TEM laboratories. Appropriate logbook pages from the XRD and SEM laboratories are in the Attachment.

3.4 Images

Photographs of various particles are in the Attachment. The photographs include those recorded using a PLM, an SEM, and a TEM.

3.5 Chains-of-Custody

The chain-of-custody form transferring the split soil samples to RJLG is in the Attachment. The split samples remain in RJLG's possession. In addition, photographs of the sample containers showing the original sample label are in the Attachment.

3.6 Sample Tracking

Sample tracking is accomplished using electronic forms.

3.7 Personal Qualifications

Personnel qualifications are attached.

4.0 QA/QC Procedures

Please submit all documents regarding the R.J. Lee Group's [sic] quality assurance/quality control ("QA/QC") procedures for asbestos analyses. Please include in this response information regarding the processes for and results of laboratory monitoring, sample preparation, laboratory analysis, data management, laboratory certifications, internal and external report review processes, and internal and external peer review processes. Please also include all Standard Operating Procedures ("SOPs"), Laboratory Quality Assurance Plans or other information relevant to or generated during the R.J. Lee Group's [sic] analyses of the EPA soil and air samples.

RJLG operates under an extensive Quality Assurance/Quality Control plan that has been thoroughly reviewed by NVLAP, AIHA, PA DEP, NY ELAP, CA ELAP and the US EPA. The appropriate QA/QC documents for this project are included in the Attachment to this response.

4.1 Certifications

RJLG maintains laboratory certifications/accreditations in a number of venues. Copies of relevant certifications are in the Attachment.

4.2 Laboratory Standard Operating Procedures

Copies of the pertinent SOPs are in the Attachment to this response.

4.3 Laboratory Monitoring

As part of its QA/QC program, RJLG performs routine monitoring of its laboratory spaces for possible asbestos contamination. Copies of the monitoring most relevant to the EDH project are in the Attachement.

5.0 Review Documents

Please identify the supporting documents or information that were made available to the Association or the three outside reviewers of the R.J. Lee Report for their respective review of the quality of the R.J. Lee Report or the quality of the data supporting the R.J. Lee Report. In addition to this statement, please also provide the documents or information in the statement that are not otherwise provided in response to this Information Request. Documents responsive to this request may include: SOPs; QA/QC procedures; performance evaluation samples; third party audits; notes; analytical techniques; literature cited in the R.J. Lee Report; other scientific literature; R.J. Lee Group [sic] procedures and documentation; written communications, phone logs; and electronic mail.

A draft version of the report was circulated among three outside reviewers (Wylie, Langer, Ross) and among the NSSGA. No supporting documentation or other publications were requested by the various reviewers. A copy of the draft report that was sent to the reviewers is in the Attachment, as well as their responses. Procedures governing project review are described in RJLG QA/QC procedures.

5.1 Third Party Audits

RJLG is routinely audited by outside personnel, usually associated with renewal of a laboratory certification. Among the groups that have audited RJLG are NVLAP and AIHA. Appropriate to this response is an audit performed of RJLG's laboratory by EPA in 2004, noting: "...auditors were hard pressed to find any deficiencies at this corporate asbestos testing laboratory. This facility could easily be considered among the most capable of those laboratories audited and approved by ASB for Superfund asbestos testing needs. All staff, from sample log-in personnel to microscopists, are extremely knowledgeable and experienced. Raw data

is managed by instrument dedicated data entry PDAs coupled to a LIMS server which makes reporting of incomplete results almost impossible. The facility also has a very good Quality Assurance Program designed to encompass all operations at the corporate, as well as, regional facility locations."

5.2 Cited Literature

Copies of the relevant literature cited in RJLG's report are in the Attachment. Copies of the relevant literature were not provided to the reviewers as they are very knowledgeable about the subject in question and already had access to the relevant literature.

5.3 Communications

A search of e-mails and records of telephone conversations was made; no such records relevant to this question were found. No letters were written as part of this project. There were phone calls between the reviewers and RJLG, but no records of these conversations were made. RJLG does not require the retention of telephone logs.

6.0 RJLG QA/QC Procedures

Please provide a statement describing the R.J. Lee Group's [sic] QA/QC procedures for each analytic technique to ensure consistency in measurements of asbestos with particulate loading or asbestos in soil (e.g., structure identification, mineral identification, diffraction patterns, reported concentrations, etc.) within the laboratory and among two or more analysts examining any respective sample. The statement should include, for each analytical technique relevant to the R.J. Lee Report, the R.J. Lee Group's variability rate among its analysts for samples of asbestos with particulate loading or asbestos in soil. Please also provide all documents that support the determination or assessment of the variability rates.

The requested procedures are described in RJLG's QA/QC documents produced in section 5 and in the procedures referenced in the analytical methods. The variability of our analysts is within the accepted variability rate of analysts as determined by the accrediting agencies. The various external audits document review of RJLG's analytical and QA/QC procedures as noted in the recent EPA audit of our Monroeville laboratory.

RJLG is an accredited laboratory that performs analyses for a wide variety of clients, including environmental engineering firms, industrial hygienists, federal, state, and local governmental agencies, and individual building owners. As such, RJLG's QA/QC procedures have been rigorously evaluated by outside agencies, including the US EPA. RJLG's analytical capabilities meet and exceed the requirements of the various accrediting agencies.

The testing performed by RJLG on the soil samples from EDH was a multi-phase test program that encompassed a variety of analytical procedures. The analytical results from each technique support the results of the other techniques performed on the samples – no amphibole asbestos was observed in the split soil samples examined by RJLG. Varying amounts of hornblende were observed in these samples.

7.0 Issues Related to Asbestos Identification

Please provide all documents that support or explain the following issues raised by the R.J. Lee [sic] Report:

7.1 Characteristics of Asbestos Fibers

The character of fibers counted as asbestos fibers in the R.J. Lee Report, specifically those relevant to establishing limitations on width or the exclusion of "cleavage fragments," considering the May 30, 2003 REPORT ON THE PEER CONSULTATION WORKSHOP TO DISCUSS A PROPOSED PROTOCOL TO ASSESS ASBESTOS-RELATED RISK, which addresses protocols on assessing asbestos related risks under the Berman-Crump method, and which recommended counting "cleavage fragments" that have equal durability and dimension as asbestos fibers, and recommended, to account for inhalation through the mouth, counting fibers up to 1.5 microns in width.

RJLG did not consider the individual fibers reported by Lab/Cor as asbestos or nonasbestos in the review of the El Dorado Report. Rather, RJLG examined the group of reported fibers to evaluate issues related to aluminum content and particle size. RJLG found that the aluminum content of the amphibole particles analyzed by Lab/Cor was higher than that reported in the scientific literature for calcic-amphibole asbestos fibers and also determined that the length/width distribution of the reported fibers was not that of an asbestos population.

RJLG addresses the key questions relevant to EPA Region 9's question below:

What fibers should be counted and considered in the risk assessment? If EPA Region 9 and ATSDR intend to base the risk assessment on its QAPP and on IRIS, the answer to the question is: *the asbestos concentration of airborne samples is determined by measuring the number of asbestiform fibers meeting the counting rules of the method employed to evaluate the risk under IRIS. In the case of environmental samples, this requires the use of PCM (NIOSH 7400) as the base analysis under which the fiber concentration is determined and TEM (NIOSH 7402) to determine the proportion of fibers counted that are asbestos. In this instance the upper limit on the width of asbestos fibers to be counted is 3 μm.*

If EPA Region 9 and ATSDR intend to use the more modern risk analysis written by Berman and Crump[58], the answer to the question is: *only fibers whose width (whether or not they are asbestiform) is less than 0.4 μm, and whose length is longer than 10 μm would be counted.*

If there is a concern about the potential interference in the asbestos count and/or risk presented by cleavage fragments, the analyses could have been done as the New Jersey Department of Health and EPA Region 2 did in the Southdown study.[52] In that case, the answer is: *Both asbestiform fibers and cleavage fragments should be identified, photographed and counted, enabling a formal risk assessment for the two populations of particles, using the criteria deemed appropriate by the risk experts.*

The size distribution of the measured particle population is inconsistent with that of asbestos particles; therefore the use of the IRIS risk model (which is based on exposure to commercial asbestos) is inappropriate. As a result, risk estimates for the El Dorado data set should be based on the model outlined in the Berman-Crump Technical Support Document for a Protocol to Assess Asbestos-Related Risk, which is far more applicable to mixed dust environments than is IRIS.

The question about the character of fibers reported as asbestos, however, goes beyond the El Dorado data set (whose interpretation is limited by the nature of the data collected) to a more general question of what is asbestos.

What dimensions should be counted? Asbestos is a recognized carcinogen for which specific models to estimate the health risk resulting from exposures have been developed. As pointed out in the Berman-Crump technical support document[58] published as a follow-up to the peer consultation report[26], to be valid, risks estimated using such models must meet two criteria: 1) the measurements must be made in the same manner as those used to develop the risk model, and 2) the measurements must reflect the characteristics of asbestos that cause disease. *This means that fibers that have the dimensions of the asbestos fibers to which the original study cohort was exposed should be counted in the El Dorado study.*

The technical support document states that the optimal exposure index assigns a single potency to fibers that are longer than 10 μm and less than 0.4 μm in width and zero potency to fibers outside these dimensions. The potency index would be different for lung cancer than for mesothelioma. *This means that the concentration of long thin fibers is the most relevant to describing the potential hazard of a dust.*

The peer consultation panel generally accepted these concepts with some qualifications and proposals for additional study. One of the suggestions for consideration was that thicker fibers (0.5 μm to 1.5 μm in width) not be excluded when considering lung cancer, although it was recognized that they have a much lower probability of penetrating into the lung. Another suggestion was that only thin (<0.5 μm) fibers be included for mesothelioma, but that fibers from 5 to 10 μm long be included in the index for lung cancer.

Another panel suggestion was that lacking other specific information, nonasbestos amphibole (cleavage) fragments of the same size and dimension as asbestos fibers be treated as though they were as potent as asbestos. One panelist emphasized that there are distinct morphological and chemical differences between naturally formed asbestos fibers and fragments whose surfaces were produced by fracture. Members of the panel noted that numerous studies have shown no asbestos disease amongst miners heavily exposed to nonasbestos amphiboles including the

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[26] USEPA (U.S. Environmental Protection Agency) (2003). Report on the Peer Consultation Workshop to Discuss a Proposed Protocol to Assess Asbestos-Related Risk, Final Report. Office of Solid Waste and Emergency Response, Washington D.C.

New York Talc miners, gold miners in Homestake, South Dakota and Minnesota iron ore workers. *This means that while the panel voiced opinions on the significance of cleavage fragments, no peer-reviewed EPA, OSHA, NIOSH, or ATSDR document has found that cleavage fragments cause asbestos disease.*

The panel emphasized the need to reconcile the disparity between the Quebec miners and South Carolina textile worker exposures. *The recent NIOSH reanalysis of the South Carolina[27] data reinforces the concept that the presence of long thin (>40 μm long, <0.3 μm wide) asbestos fibers in an aerosol is the most important measure of the potency of the dust.*

The clear and unambiguous message in the health and risk data is that long, thin airborne fibers are a prerequisite for asbestos disease – *absent long, thin airborne fibers, the data indicate zero potency.* The second important consideration is to ensure that particles with those size characteristics are reliably measured. While there is no specific evidence that long, thin cleavage fragments (> 10 μm long, thinner than 0.5 μm) present a risk of asbestos disease, they are a rare occurrence, and prudent public policy may indicate they should be counted as asbestos.

Should cleavage fragments be counted as asbestos? EPA Region 9 set forth a project plan specifically in response to concerns voiced by a citizen over potential exposures to asbestos in and around the El Dorado Hills School District. EPA Region 9, in conformance with past EPA practice as well as that of other agencies, recognized the mineralogical and geological vocabulary and defined asbestos for the purpose of the study as "fiber bundles made up of extremely long and thin fibers that are readily separated from one another."[28] EPA Region 9 did not differentiate between regulated asbestos and non-regulated asbestos minerals.

EPA Region 9's original project goals and definitions were consistent with the known characteristics of asbestos, both mineralogically and as a recognized hazardous substance. Counting nonasbestos particles as if they were asbestos is a deviation from the project plan.

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[27] NIOSH South Carolina Reference

[28] Ecology & Environment (2004). page ix.

The character of fibers counted as asbestos by a laboratory should be that of asbestos, unless a specific exception to the method is noted. In an environmental analysis all particles meeting the method counting criteria are identified, measured and reported appropriately as amphibole, amphibole asbestos, serpentine, chrysotile, or other mineral. These are the requirements of the Yamate and the NIOSH 7402 methods. Neither places a restriction on the counting of structures with diameters between 0.5 and 1.5 μm. The answer to EPA Region 9's question is that all particles meeting the counting criteria specified by the method should be counted, reported and classified as asbestos or nonasbestos. If risk criteria implicating nonasbestos amphiboles in disease are identified, their concentrations should be subject to a formal risk assessment as is asbestos. However, given they do not, in general, have equiaxed cross-sections, and that they have different aerodynamic qualities and deposition characteristics than their asbestiform counterparts, the risk analysis of nonasbestos particles should be independent of any asbestos risk analysis.

It is not a laboratory decision to determine whether or not cleavage fragments are to be counted from a risk perspective. It is a laboratory responsibility to Certify that the particles counted and reported as asbestos are in fact asbestos. Lab/Cor signed a contract to identify and count asbestos particles and signed reports indicating they had found asbestos when in fact they did not.

At a higher policy level, EPA Region 9 used their authority under CERCLA to assess the significance of potential asbestos exposures. Given the recognized differences between asbestos and nonasbestos amphiboles, both at the mineralogical and regulatory levels, any response action ought to be based on measurements of asbestos as it was defined by the QAPP.

Setting public policy by using the peer consultation report to bootstrap an equivalent asbestos exposure to mineral fragment exposure is inappropriate. Further bootstrapping the process by using risk estimates derived for long, thin commercial asbestos fibers to evaluate the significance of short, wide nonasbestos fragments is inappropriate.

7.2 Iron Valence State

How the R.J. Lee Group [sic] in the R.J. Lee Report distinguished between the presence of Fe^{3+} and Fe^{2+} found in amphibole minerals when performing an Energy Dispersive Spectroscopy ("EDS" or "EDXA" in the R.J. Lee Report) analysis.

The identification of valence state for iron should be determined using Mossbauer spectroscopy or by wet chemistry as described in the literature.[29] No such data was produced by Lab/Cor; RJLG has not performed such an evaluation for the amphibole particles in the soil samples examined.

The valence state (Fe^{3+} or Fe^{2+}) was evaluated in accordance with the procedure described in Leake, Appendix A beginning with step 6.[30] As implemented, the procedure does not require the actual ratio when determining the name to assign to a mineral. If the "all ferrous iron" name and "all ferric iron" name are the same, there is no need to determine the actual ratio of iron valence states.

RJLG evaluated the names assigned to 341 actinolite/tremolite particles by assuming the observed iron to be either all Fe^{3+} or Fe^{2+}. Only 47 showed resulted in a different name (generally to a hornblende).

7.3 Clay Contamination of Amphibole Particles

How the R.J. Lee Group [sic] in the R.J. Lee Report distinguished between the signal of an amphibole structure from the aluminum signal from aluminum-rich clay particles adhered to the amphibole structures when performing an EDS analysis.

RJ Lee Group analyzed the selected soil samples by XRD and determined the aluminum-bearing minerals in the samples to be hornblende, chlorite, plagioclase feldspar and muscovite/vermiculite.

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[29] Hawthorne from vol 9b

[30] Leake, B.E. et al. (1997). Nomenclature of the amphiboles: Report of the subcommittee on amphiboles of the International Mineralogical Association, Commission on New Minerals and Mineral Names: Canadian Mineralogist, 35, p. 219-246.

As part of the limited electron microscopy (EM) analyses performed by RJLG, the selected soil samples were prepared for analysis using an indirect preparation procedure. During this procedure, the soil is suspended in a liquid and is agitated using ultrasonication. This procedure separates adhering particles. The samples were carefully examined in the EM to determine if any particles were adhering to the target particle and, if so, whether the adhering particles would interfere with the analysis of the target particle. We determined that this was not an issue in our analyses. This is documented in the photographs taken during these analyses.

When evaluating the Lab/Cor data, RJLG accepted the reported data at face value. It is the obligation of the microscopist to obtain an EDS spectrum from a portion of the particle that is most representative and to avoid interferences from adhering particles.

Clay particles are generically alumino-silicates; their elemental composition is aluminum, silicon and other elements. As shown in Deer, et al (vol 3, 1962), the silcon:aluminum ratio for the atoms in clay minerals ranges from 1:1 for the kaolinite group, from 1:1 to 3:1 for the illite group, and from 2:1 to 6:1 for the montmorillinite group of clay minerals. For every atom of aluminum that must be accounted for from the clay mineral, at least one (and up to six) silicon atoms must also be accounted for from the clay minerals.

Thus, for there to be an exclusion of aluminum due to the presence of clay, there must also be an exclusion of silicon. This exclusion of silicon was apparently not done by Lab/Cor, therefore one must conclude there was no interference in the EDS spectra due to the presence of adhering clay minerals.

EPA contends that the mineral nomenclature complies with the Leake protocol.[31] RJLG has not, contrary to EPA statements (EPA page 5), challenged the assigned nomenclature but has noted that the amount of aluminum in the minerals precludes the formation of asbestos fibers.

Meeker (page 3) suggests that a hornblende mineral (fluoro-edenite) that reportedly occurs as asbestos is proof that amphibole particles can contain

[31] EPA, 2006, page 6.

significant quantities of aluminum and still be asbestos. Fluoro-edenite is not the same mineral as the actinolite/tremolite that is reported in the Lab/Cor data. It should be noted that "Edenitic compositions are rare in amphiboles, and their paucity might suggest a structural instability".[32]

The real issue is the low aluminum content of the tremolite/actinolite amphibole minerals – the minerals at issue in El Dorado Hills. Deer et al note (page 141) that in "most tremolite-actinolites, the replacement of Si by Al is small (<0.3 Al pfu)" and (page 182) that "Electron probe analyses showed that specimens that contain more than a very small amount of aluminum do not have asbestiform habit." Deer cites Dorling and Zussman[33] for the low aluminum content. Dorling and Zussman show (Figure 16 of their paper) that aluminum atoms in the asbestos samples analyzed were present at less than 0.1 apfu. The Dorling findings were supported by Verkouteren and Wylie[34] who showed 85% of their asbestos samples contained 0.1 Al apfu or less.

7.4 Variation of SAED with Particle Size

How the R.J. Lee Group [sic] in the R.J. Lee Report distinguished or otherwise considered in its comparison based on zone axis indices the EPA soil samples containing mixed-sized particulates from the reference of asbestos sample standards.

Tremolite/actinolite asbestos fibers have limited widths (0.1 to 0.5 μm), but can vary in length from microns to inches. The length of the asbestos fiber will have no effect on the observed zone axis[35] pattern. Nonasbestos amphibole minerals, however, will display varying zone axes depending on the fracture characteristics and orientation of the particle in the

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[32] A. Gianfagna and R. Oberti (2001). Fluoro-edenite from Biancavilla (Catania, Sicily, Italy): Crystal chemistry of a new amphibole end-member, American Mineralogist, 86, p. 1489-1493.

[33] M. Dorling and J. Zussman (1987). "Characteristics of asbestiform and non-asbestiform calcic amphiboles", *Lithos*, 20, p. 469-489.

[34] Verkouteren, J.R., and Wylie, A.G. (2000). "The tremolite - actinolite - ferro - actinolite series: Systematic relationships among cell parameters, composition, optical properties, and habit, and evidence of discontinuities", *American Mineralogist*, 85 p. 1239 – 1254.

[35] A "zone axis" is a way of describing the common direction of the intersections of the faces of a crystal. As used here, the "zone axis" describes a particular selected area electron diffraction pattern.

microscope. This variation in zone axis is one of the defining characteristics of the nonasbestos minerals.

It is well known that amphibole asbestos fibers have a tendency to orient in a preferred manner. This has been reported by Lee[36], Nord[37], Yamate[38], Ring[39], and Stewart[40] and is recognized in all TEM analytical methods as nearly diagnostic of asbestos. This preferred orientation gives rise to the so-called characteristic 0.53 nm row spacing referred to in EPA methods for TEM analysis of asbestos.[54] In contrast, nonasbestos amphiboles tend to have far greater variability in orientation, and do not predominantly show the 0.53 nm spacing. Rather as shown in Figure xx, the nonasbestos amphiboles in this study cluster around a different direction (110).

Examples of zone axis patterns for actinolite are shown in the following Figure 9.

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[36] R. Lee, J. Lally, and R. Fisher (1977). "Identification and Counting of Mineral Fragments", proceedings of the Workshop on Asbestos: Definitions and Measurement Methods held at NBS, Gaithersburg, MD, July 1977. NBS Special Publication 506, pp. 387.

[37] G. Nord and R. Lee (2003). "Characterization of Fibrous Particles by Analytical Transmission Electron Microscopy", Contained within "Program and Abstracts for International Symposium on the Health Hazard Evaluation of Fibrous Particles Associated with Taconite and the Adjacent Duluth Complex ", March 30-April 1, 2003.

[38] Yamate, G., S. C. Agarwal, R. D. Gibbons (1984). "Methodology for the Measurement of Airborne Asbestos by Electron Microscopy." EPA Contract No. 68-02-3266, July 1984.

[39] S.J. Ring (1981). Identification of Amphibole Fibers, Including Asbestos, Using Common Electron Diffraction Patterns. In Russell P.A. and Hutchings A.E. (Eds), Electron Microscopy and X-ray Applications to Environmental and Occupational Health Analysis, Vol. 2:175-198, Ann Arbor Science Publ., Inc.

[40] Stewart, I. (1988). "Asbestos - Analytical Techniques Definitions and Mineralogy of Asbestos Basic Crystallography and Electron Diffraction." Presented at JEOL TEM training courses.

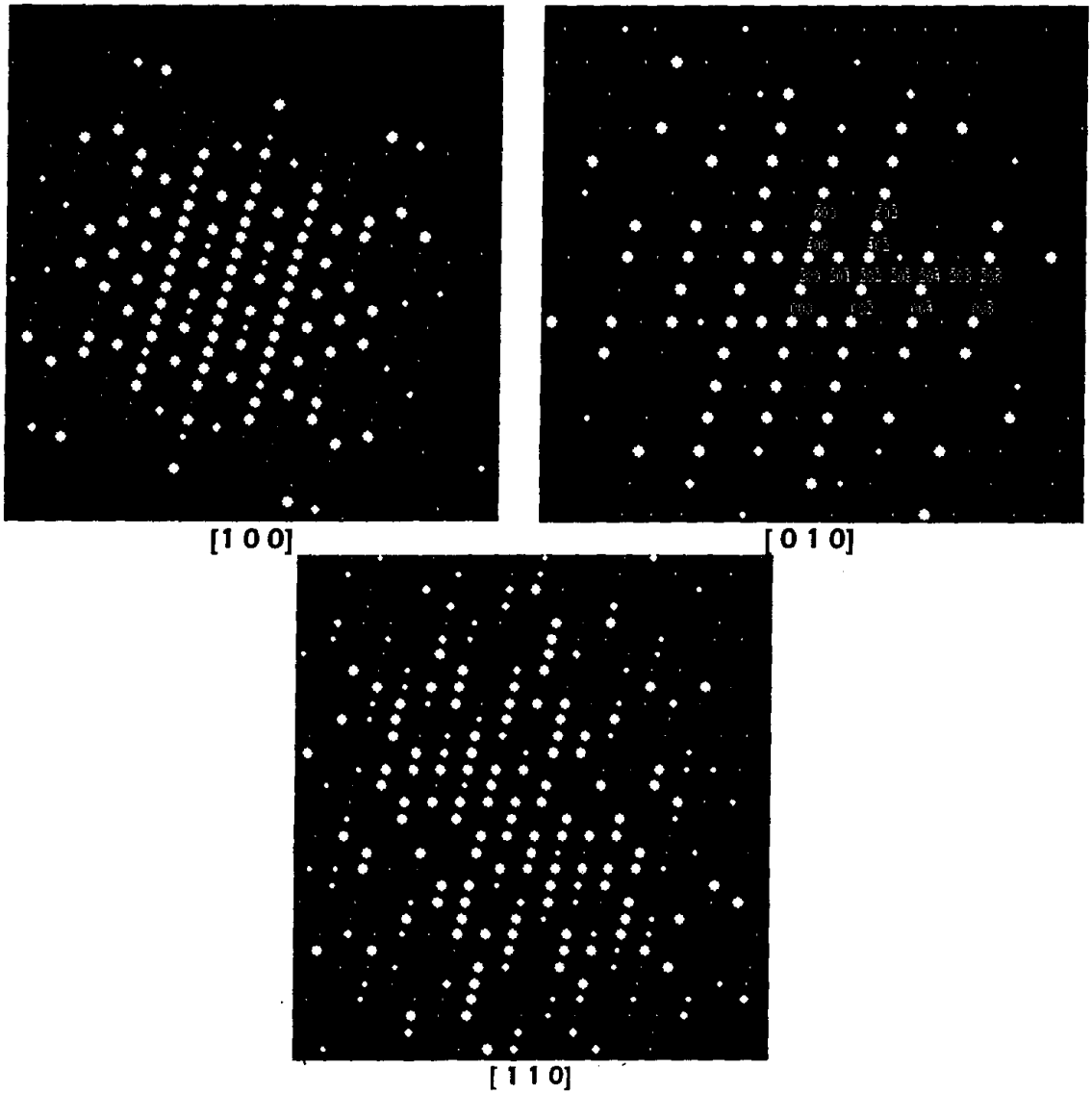

Figure 9. XX

7.5 Different Morphologies of Amphibole Particles

How, during a Polarized Light Microscopy ("PLM) analysis, the R.J. Lee Group [sic] in the R.J. Lee Report allowed for the presence of both asbestiform and nonasbestiform habits of the same mineral to be present in a rock or soil sample.

Within a geological setting where asbestos fibers are found, nonasbestos forms of the same mineral will always be observed. The converse is not true because specific geological conditions are needed for the development of asbestos fibers. RJLG "allows" for this occurrence, by

reporting the presence of all forms of the amphibole minerals. The characteristics of asbestos as described in the PLM methods are used to identify a particle as asbestos, supplemented by the descriptions found in Campbell[22], McCrone[41], Wylie, and others.

7.6 Analysis of Amphibole Minerals

Whether the asbestos amphibole fibers that the R.J. Lee Group [sic] counted for the R.J. Lee Report only included the six regulated asbestos mineral types that exhibit an asbestiform habit (>20:1 or 50:1 aspect ratio) and exhibit parallel extinction.

When analyses are performed on commercial product material to determine conformance with various regulations, then the asbestos types are limited to the six regulated minerals in accordance with the analytical methods and our laboratory certifications. For samples of raw materials, RJLG will not limit the reporting of asbestos minerals to those that are regulated.

In the analyses of the El Dorado samples, RJLG applied the PLM methods as described in the analytical methods and Campbell, McCrone, Wylie, and others for additional descriptions of asbestos mineralogy. These analyses were not limited to the regulated asbestos types.

7.7 Parallel Extinction of Asbestos Fibers

During a normal PLM analysis, whether the R.J. Lee Group [sic] in the R.J. Lee Report would consider parallel extinction to be a definitive indicator that an amphibole fiber is an asbestos fiber.

The use of parallel extinction is a defining characteristic of asbestos fibers as noted by Verkouteren and Wylie[42] and in the analytical protocols[55] when the fibers also exhibit other characteristics of asbestos. However, the observation of parallel extinction without other asbestos characteristics does not absolutely define a particle as asbestos. Asbestos is characterized by bundles of easily separated fibers, very thin fibers (less than 0.5 μm), fibers showing curvature, and fibers with very high aspect ratios. When a population of particles does not exhibit any of these characteristics and

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[41] W. C. McCrone, Particle Atlas.

[42] J. R. Verkouteren, A. G. Wylie (2002). "Anomalous optical properties of fibrous tremolite, actinolite, and ferro-actinolite," American Mineralogist, 87, p 1090-1095.

does not show parallel extinction, then the population of particles is clearly nonasbestos.

The PLM methods state that asbestos fibers have parallel extinction while nonasbestos particles have oblique extinction. As noted in the PLM method used by the EPA (NIOSH 9002), tremolite-actinolite will have oblique extinction (10° - 20°) for fragments. As noted in OSHA ID-191 (section 3.5): "...cleavage fragments of the monoclinic amphiboles show inclined extinction under crossed polars with no compensator. Asbestos fibers usually show extinction at zero degrees or ambiguous extinction if any at all." The draft ASTM method (P236) that was circulated by NIST to all NVLAP laboratories states: tremolite asbestos and actinolite asbestos has extinction "parallel in most fibers". The EU method (1997) states that "polarized light microscopy (PLM) can be used to exclude some elongated cleavage fragments on the basis of their non-parallel extinction angle" (page 13). As noted in EPA's own 1993 PLM method refractive indices are to be measured on tremolite-actinolite when the fiber exhibits extinction at a zero degree orientation (page 15).

Wylie[43], Dorling and Zussman[33], and Verkouteren and Wylie[42] report that asbestos fibers have parallel extinction or (if too thin) anomalous extinction properties.

7.8 SRM 1867

Whether asbestos fibers supplied by the National Institute of Standards and Technology ("NIST") Standard Reference Materials ("SRM") 1867 and 1867a, as referenced in the R.J. Lee Report, ever exhibit inclined extinction angles.

The National Institute of Standards and Technology (NIST) certificates accompanying Standard Reference Materials SRM 1867[44] and 1867a indicate the tremolite and actinolite standards in each are from the same batches of material and indicate that "a small amount of material may be massive" (tremolite) or "a considerable amount of material may be massive" (actinolite). The certificates note these minerals are "mine-grade

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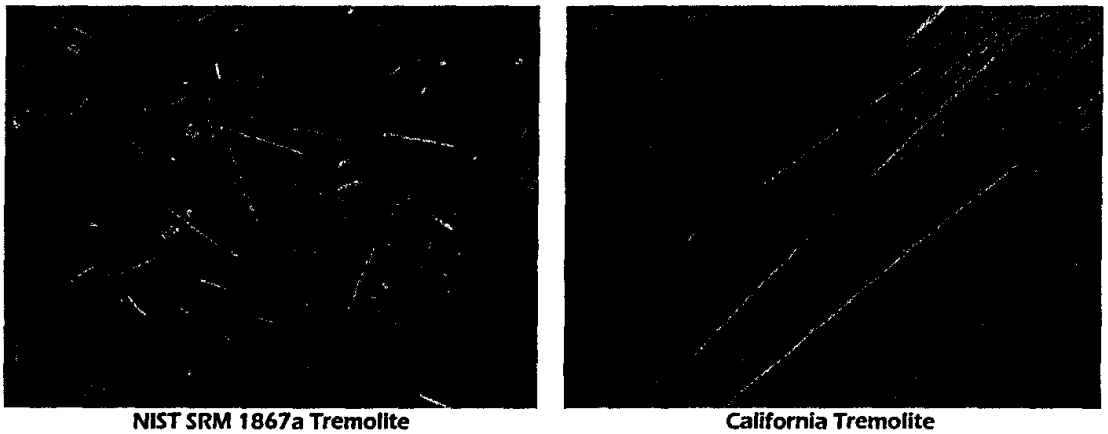
[43] A. Wylie (1979). "Optical properties of the fibrous amphiboles", Ann NY Acad Sci, 330, p. 611-619.

[44] NIST SRM 1867 and 1867a contain samples of tremolite, actinolite, and anthophyllite and are referred as "Uncommon Commercial Asbestos".

asbestos materials". The asbestos in each sample is described as having asbestiform characterisitcs: "Asbestiform: crystallizes with the habit of asbestos. These asbestos minerals possess properties such as long fiber length and high tensile strength. Under the light microscope, [some portion of][45] these samples exhibit the asbestiform habit as defined by several of the following characteristics: 1) mean aspect ratios ranging from 20:1 to 100:1 or higher for fibers longer than 5 μm, 2) very thin fibrils, usually less than 0.5 μm in width, 3) parallel fibers occurring in bundles, 4) fiber bundles displaying splayed ends, 5) fibers in the form of thin needles, 6) matted masses of individual fibers, and 7) fibers showing curvature" (page 2).

The following Figure xx illustrates the morphologies of particles found in the NIST SRM 1867a tremolite and compares those to tremolite found near San Andres, California. Both images were taken using a PLM and are at the same magnification. The highly fibrous nature of the California tremolite is evident compared with the NIST tremolite.



NIST SRM 1867a Tremolite

California Tremolite

Figure 10. XX

NIST SRM 1867a tremolite was examined to determine the extinction angle for those particles that are clearly asbestos and those that are clearly not asbestos. The asbestos fibers had extinction angles less than 10°, while the nonasbestos particles ranged up to 22°. The following Figure xx illustrates the differences in extinction angle between these two different types of particles.

[45] The words in brackets are from the SRM 1867a certificate.

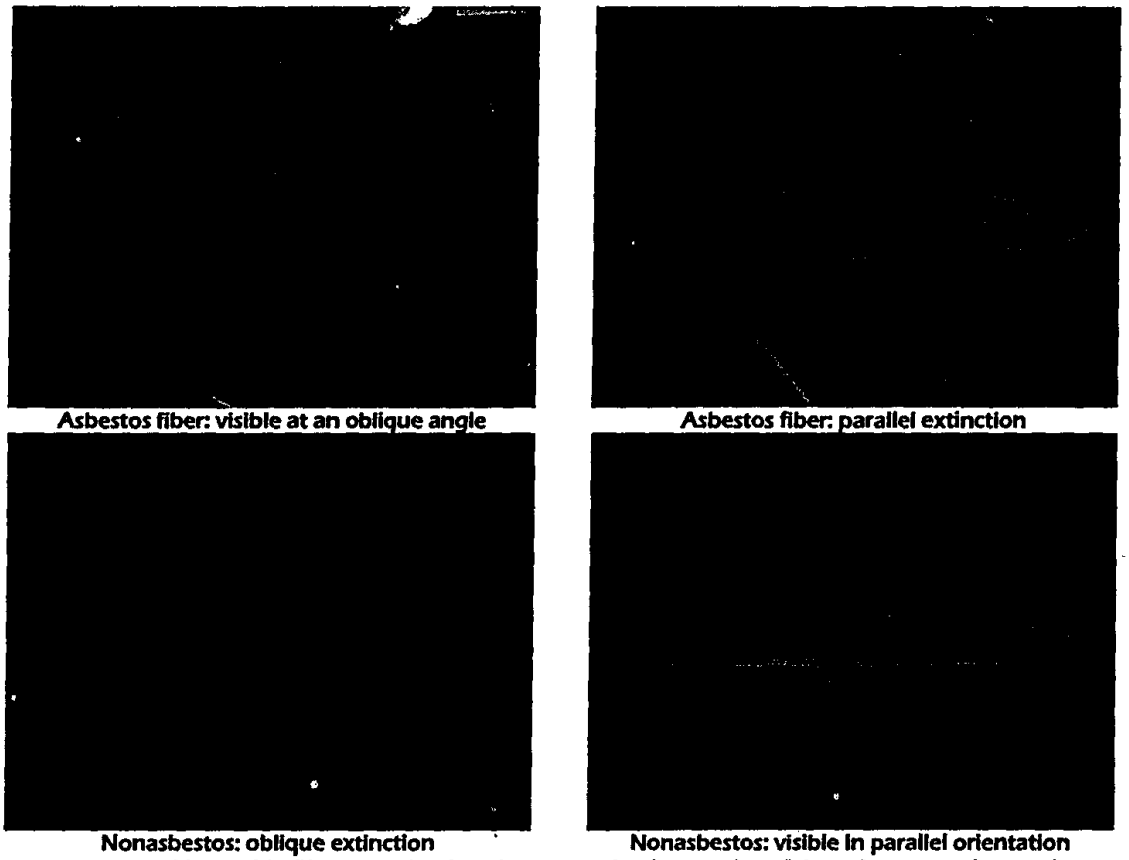


Figure 11. Photographs showing the extinction angles of the asbestos and nonasbestos particles observed in NIST SRM 1867a.

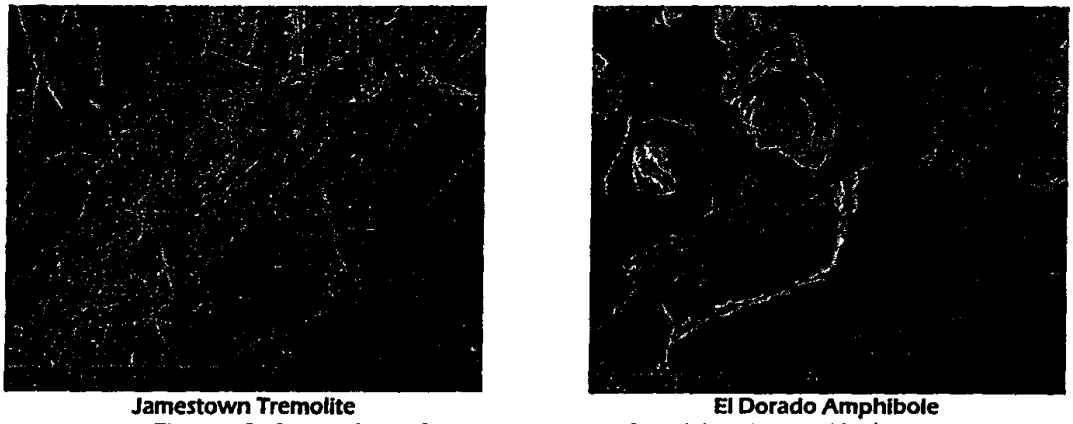
7.9 Fiber Terminations

During transmission Electron Microscopy ("TEM) or PLM, whether the R.J. Lee Group [sic] in the R.J. Lee Report would consider rounded terminations to be a definitive indicator that amphibole fiber is not an asbestos fiber.

Most fibers, at a sufficient magnification to observe the ends, have squared terminations. RJLG would not classify a particle with "rounded terminations" as nonasbestos solely on the basis of the terminations. If the particle exhibited the characteristics of asbestos (1) mean aspect ratios ranging from 20:1 to 100:1 or higher for fibers longer than 5 μm, 2) very thin fibrils, usually less than 0.5 μm in width, 3) parallel fibers occurring in bundles, 4) fiber bundles displaying splayed ends, 5) fibers in the form of thin needles, 6) matted masses of individual fibers, and/or 7) fibers showing curvature), RJLG would not generally evaluate the conditions of the particle terminations to determine whether it was asbestos or

nonasbestos. RJLG is interested in seeing pictures of particles with rounded terminations.

The following Figure 12 illustrates the morphology of tremolite fibers from Jamestown, CA as well as particles from El Dorado.



Jamestown Tremolite | El Dorado Amphibole

Figure 12. Comparison of the morphology of particles observed in Jamestown asbestos with that o f the amphibole particles observed in the El Dorado soil samples.

8.0 Verified Analyses

Please provide a step-by-step description of the method and all the information sources used to perform the verified count in the R.J. Lee Report for its allocation of true and false positive values and provide supporting documents.

Verified analysis is the comparison of TEM data generated from at least two independent analyses of the same grid opening(s), using the same counting methodology. By comparing the following characteristics of the counted particles, it is possible to determine if the same particle was observed in the independent analyses of the grid opening: 1) relative location within a grid opening, 2) appearance, 3) orientation of the particle in the grid, 4) size of the particle, 5) particle morphology, 6) EDS and/or SAED information for the particle, and 7) particle identification by the analysts. Procedures describing these evaluations have been written by personnel from the National Institute of Standards and Technology.[46,47,48]

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[46] S. Turner and E. B. Steel (1994). Airborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy – Version 2.0, NISTIR 5351.

[47] E. B. Steel and J. A. Small (1985). Accuracy of Transmission Electron Microscopy for the Analysis of asbestos in Ambient Environments, *Anal. Chem.*, 57, p. 209-213.

In performing the evaluation of the Lab/Cor data from repeat analyses of the same grid openings, the count sheets for the original and QC analysis were located from within the produced materials. Both count sheets were converted to spreadsheets. After data entry and verification that the data entry was correct, the QC data was matched to the original data in the following steps:

1) Data from the same grid openings were placed side-by-side.
2) Data from within each grid opening were placed side-by-side on the basis of mineral identification (chrysotile or amphibole).
3) Within a mineral type, the data were arranged by particle morphology (as either individual fibers or bundles or as part of a matrix or cluster). Where multiple matrices or clusters were found within a grid opening and mineral type, the types of substructures and size of the substructures were used to place the particles side-by-side within the spreadsheet.
4) Matching data were identified and enumerated. The count was based on the number of matched primary structures.

The following example is given for sample SRA-R04-100104. Only the data from the grid opening identified as C10 are included in this illustration.

After the data were placed side-by-side, as shown in the following Figure xx, the particle dimensions were compared. Both analyses report a diffuse matrix that contains one fiber longer than 5 μm (MD1-1). While there are some variation in the dimensions of the fiber (7.5 μm vs 6.5 μm), the overall descriptions of this particle are similar enough to consider this as the same particle. However, the original analysis reported an additional fiber (F, 2.4 μm x 0.6 μm) that was not reported in the QA analysis.

	Original Analysis				QA Analysis			
Loc.	Class	Length	Width	Aspect Ratio	Class	Length	Width	Aspect Ratio
C10	MD1-1	7.5	4	1.9	MD1-1	7.8	3.8	2.1
C10	MF	7.5	0.7	11	MF	6.5	0.7	9.3
C10	F	2.4	0.6	4	No Structure Reported			

[48] S. Turner and E. B. Small (1991). Accuracy of Transmission Electron Microscopy Analysis of Asbestos on Filters: Interlaboratory Study, Anal. Chem., 63, p. 868-872.

Table 1. Alignment of grid openings from two analyses of the same grid openings.

It is not possible to determine where the error occurred in the fiber count from this grid opening – was the fiber actually present but not verified or was it an error by the original analyst in reporting a structure that does not exist? However, one can conclude that there is a discrepancy with the fiber count on this grid opening.

The remainder of the grid openings were examined in a similar manner. Non-matching particle counts in the original analyses were highlighted as shown in the following Figure 13.

			Original Analysis						QA Analysis					
Gr	No	Loc.	Prim	Tot	Class	Len	Wid	Asp	Prim	Tot	Class	Len	Wid	Asp
A	1	A12	1	1	F	3.8	0.3	13	6	6	F	3.8	0.38	10
A	2	B22	[redacted]						No Structure Reported					
A	3	B10	3	3	F	7.5	1	7.5	1	1	F	7.5	1	7.5
A	3	B10	4		MD1-0	4	1.8	2.2	2		MD1-0	4.3	1.5	2.9
A	3	B10		4	MF	4	0.45	8.9		2	MF	3.5	0.22	16
A	3	B10	5	5	F	4.5	1.2	3.7	3	3	F	4	1	4
A	4	A30	No Structure Reported						No Structure Reported					
A	5	D2	[redacted]											
A	5	D2							7	7	F	2	0.6	3.3
A	6	D31	7	7	F	2.5	0.7	3.6	9	9	F	2.5	0.4	6.2
A	7	D23	8	8	F	4	1.1	3.6	8	8	F	3	0.25	12
A	8	C10	9		MD1-1	7.5	4	1.9	4		MD1-1	7.8	3.8	2.1
A	8	C10		9	MF	7.5	0.7	11		4	MF	6.5	0.7	9.3
A	8	C10	[redacted]						No Structure Reported					
A	9	C41	11	11	F	5	1	5	5	5	F	4.5	1	4.5
A	10	C22	No Structure Reported						No Structure Reported					
A	11	C14	No Structure Reported						No Structure Reported					
B	12	A30	No Structure Reported						No Structure Reported					
B	13	A11	No Structure Reported						No Structure Reported					
B	14	D21	No Structure Reported						11	11	F	5.8	1	5.8
B	15	C40	12	12	F	3.7	0.5	7.4	10	10	F	3.8	0.45	8.4
B	16	C11	No Structure Reported						No Structure Reported					
B	17	C24	[redacted]						No Structure Reported					
B	18	B1	No Structure Reported						No Structure Reported					
B	19	B20	No Structure Reported						Grid Opening Not Analyzed					

Figure 13. Illustration of the alignment of the particles from two analyses of the same grid openings on a sample showing the correlation of particle counts.

In the above example, no particle was observed in grid openings B22, C10, and C24 during the QC analysis. A second fiber was identified in grid opening D2 that was substantially shorter and thinner than the original analysis; the original fiber was not observed during the QC analysis.

There is some question as to whether the particle in grid D23 is the same in each analysis primarily due to the difference in particle width.

9.0 Reference Minerals

Please provide all of the spectral data and supporting references for the R.J. Lee Group's [sic] mineral identifications relevant to the R.J. Lee Report, including all documentation of raw data, calculations, equations, and the supporting references.

RJLG utilized a number of reference standards, including rock specimens collected at El Dorado Hills. The standards also include the powder diffraction files published by the JCPDS, reference minerals NIST SRM 1866 and 1867, and rock samples sold by Ward Scientific.

10.0 Optical Properties of Asbestos

Please provide a copy of any written procedures employed by the R.J. Lee Group [sic] that describe how reference standards are used to verify the accuracy of an analyst's ability to correctly determine the optical properties of asbestos.

The procedures used to evaluate the accuracy of an analyst to determine the refractive indices of mineral fibers are contained in the laboratory QA/QC procedures. RJLG utilizes the NVLAP round robin tests (and the New York ELAP tests) to document the accuracy of this analysis.

11.0 Air Sample Analysis Procedures

Please provide any written procedures or instructions given to analysts when the R.J. Lee Group [sic] performs a National Institute of Occupational Safety and Health (NIOSH) 7400 method analysis, an Asbestos Hazard Emergency Response Act (AHERA) method analysis or a California Air Resources Board AHERA- modified method analysis, including procedures regarding which aspect ratios are included in the count, whether or not all chrysotile or amphibole particles with the ratios that meet the method definition are included in the count, and any modifications to these methods. Please provide supporting documents, including any laboratory analysis bench sheets and reports with laboratory-identifying information redacted.

RJLG did not analyze any air samples during its review of the Lab/Cor data. However, if we had, the analyses would comply with the written procedures. An example of a report and count sheet for a PCM and TEM analysis is Attachment xx.

Given the concern expressed by EPA[49], that RJLG arbitrarily modifies procedures or otherwise ignores certain particles of interest during its analyses; RJLG would welcome EPA observers to its laboratory to monitor the air sample preparation and analysis if the grids or filters analyzed by Lab/Cor are made available.

If air samples are provided by the EPA, RJLG will analyze the El Dorado air samples using the NIOSH 7400[50] method supplemented by the NIOSH 7402[51] method using the TEM to apportion fibers into asbestos and nonasbestos fractions. Differentiating asbestos from nonasbestos particles is essential in mixed mineral environments where cleavage fragments or other nonasbestos particles may result in an increased PCM count. Using the TEM (NIOSH 7402 method) particles with a greater than 3:1 aspect ratio will be analyzed; energy dispersive x-ray spectra (EDS) and selected area electron diffraction (SAED) patterns will be recorded so that counted particles can be identified as asbestos or nonasbestos using the procedure approved by EPA Region 2.[52]

RJLG would supplement the NIOSH 7402 method by recording photomicrographs and SAED patterns additional to that required by the NIOSH 7402 method. Since the IRIS risk assessment procedures are based on the counting of asbestos, as defined in the EPA's El Dorado List of Terminology[53], RJLG would use the criteria in Yamate[54], OSHA[6], and in

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[49] Sacramento Bee, April 2, 2006.

[50] NIOSH Manual of Analytical Methods, Asbestos and Other Fibers by PCM, Method 7400, Issue 2, August 15, 1994, http://www.cdc.gov/niosh/nmam/pdfs/7400.pdf.

[51] NIOSH Manual of Analytical Methods, Asbestos by TEM, Method 7402, August 15, 1994, http://www.cdc.gov/niosh/nmam/pdfs/7402.pdf.

[52] P. Lioy, et al (2001). "Quality Assurance Project Plan: Assessment of Population Exposure and Risks to Emissions of Protocol Structures and Other Biologically Relevant Structures from the Southdown Quarry", January 24, 2001.

[53] Ecology & Environment (2004). El Dorado Hills Naturally Occurring Asbestos Multimedia Exposure Assessment El Dorado Hills, California, Quality Assurance Project Plan, Working Draft, EPA Contract No.: 68-W-01-012.

the EPA bulk PLM methodology[55], and supplement the analysis with SEM photomicrographs to document the morphology of individual particles. This is important because the NIOSH 7402 method was designed for measuring asbestos in a commercial environment, not for measurement of asbestos in mixed mineral dust environments.

Figure 14 illustrates the difference in particles from El Dorado Hills (mixed mineral dust environment) compared with particles that are actual asbestos fibers.

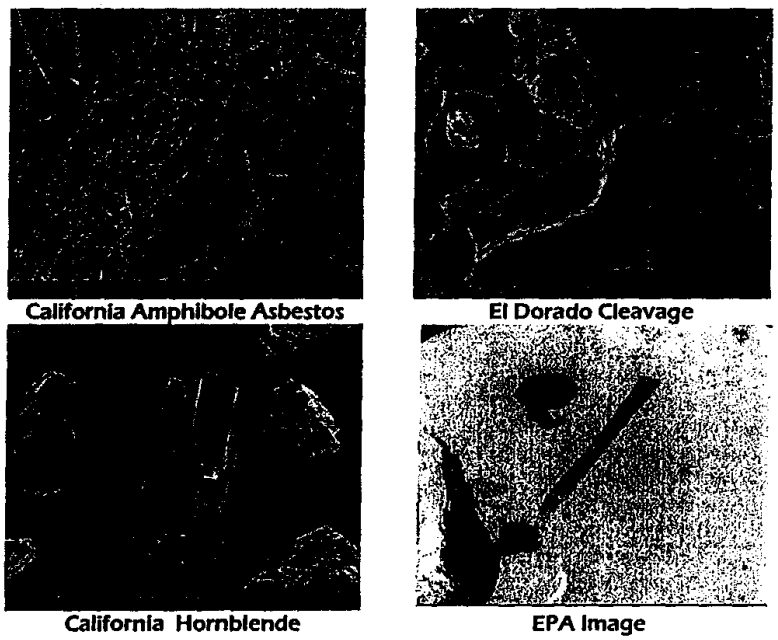

Figure 14. Comparison of actual asbestos fibers with particles observed in the El Dorado Hills samples illustrating the morphological differences between these particles *and those particles that LabCor included with non-parallel sides, which are outside the method.*

EPA Region 9 has suggested (Meers 2006[1]) that the definition of asbestos fibers within established asbestos analysis methods was not of particular interest – that any mineral particle that was at least 3 times longer than it was wide would be considered to be "asbestos". It is clear from our

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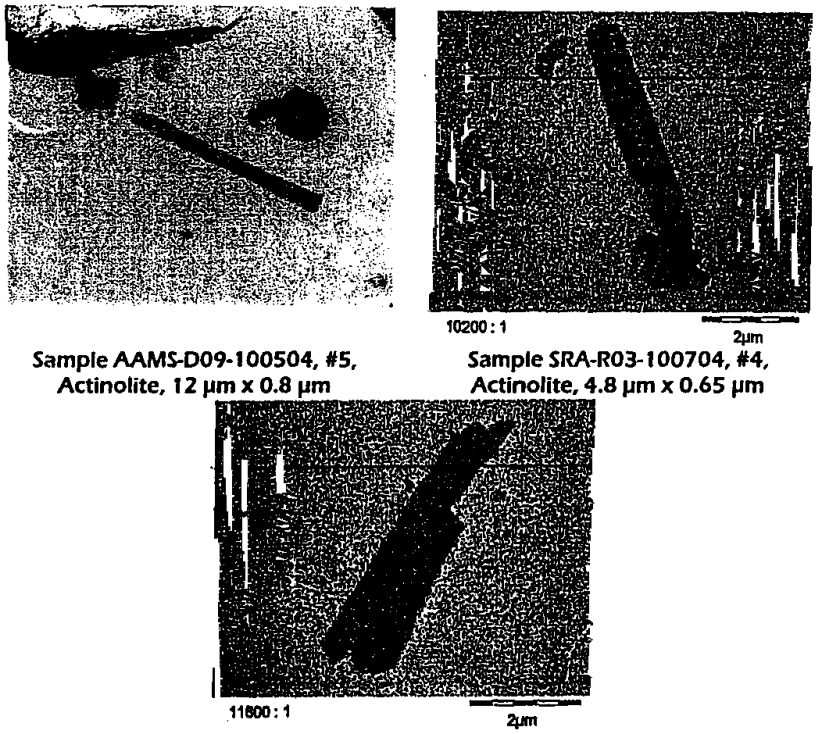
[54] G. Yamate, S. C. Agarwal, R. D. Gibbons (1984). "Methodology for the Measurement of Airborne Asbestos by Electron Microscopy", IIT Research Institute, Contract No. 68-02-3266, July 1984.

[55] R. L. Perkins and B. W. Harvey (1993). "Method for the Determination of Asbestos in Bulk Building Materials", U.S. Environmental Protection Agency, EPA/600/R-93/116, July 1993.

review of the data provided that Lab/Cor, EPA's contract laboratory, counted the particles in this manner and relaxed the definition of asbestos fiber specified in the ISO 10312 method from particles which had substantially parallel sides to any particle having a 3:1 aspect ratio, and some semblance of parallel sides, see Figure 2. ISO does not distinguish asbestos from nonasbestos particles; therefore it is necessary that this analytical method be modified for this mixed particle environment in order to differentiate asbestos from nonasbestos.



Sample AAMS-D09-100504, #5, Actinolite, 12 µm x 0.8 µm

Sample SRA-R03-100704, #4, Actinolite, 4.8 µm x 0.65 µm

Sample NRA-R05-101004, #1, Actinolite, 5.2 µm x 1.2 µm

Figure 15. Exemplars of particles observed by Lab/Cor during the analysis of EDH air filters Highlighting particle boundaries illustrates that LabCor did not follow ISO 10312 counting rules..

It has been recognized by ATSDR[56], NIOSH[57] and Berman-Crump[58] that long, thin fibers have the most potential medical significance. RJLG would therefore additionally supplement the NIOSH methods by providing separate tabulations of countable structures into different size categories (i.e., longer than 10 µm, and less than 0.4 µm in width, regardless of whether they are asbestiform or nonasbestos in nature; and longer than 40 µm and less than 0.4 µm, as have been suggested by Berman et al[59], and more recently NIOSH[57], as the principal components of

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[56] ATSDR Short Fiber report

[57] NIOSH analysis of SC Cohort samples

[58] D. W. Berman and K. Crump (2003). Technical support document for a protocol to assess asbestos-related risk," EPA, U.S. Environmental Protection Agency, Revision of original from September 4, 2001, Peer-reviewed consultation held in San Francisco on February 25-26, 2003.

[59] D. W. Berman, K. S. Crump, Eric J. Chatfield, John M.G. Davis, and Alan D. Jones (1995). "The Sizes, Shapes, and Mineralogy of Asbestos Structures that Induce Lung

an airborne fiber population correlating with disease). (Note that this is different than suggesting that only fibers longer than 40 μm in the lung are relevant, rather, that airborne particle populations with measurable quantities of very long, thin fibers drive the statistical relationship with disease.)

RJLG will make all grids it analyzes available for review by EPA, either at RJLG or at EPA's designated laboratories. The latter recognizes that transfer of grids to another laboratory may result in damage to the grid and potential loss of particles.

The following sections (11.1 – 11.14) are provided in anticipation of RJLG receiving air filters analyzed by Lab/Cor.

11.1 Laboratory Count Sheets

As observed by Mr. Smith in the EPA's 1995 audit of RJLG's Monroeville Laboratory (attached), RJLG has not maintained handwritten count sheets for a number of years. RJLG instead uses electronic recording at point–of-entry to document countable structures and produce reports. When EPA provides air filters, RJLG would, of course, maintain all data recorded electronically and provide copies of count sheets and grid maps.

11.2 Laboratory Notes

Any notes made by analysts during the course of analysis of air filters are electronically recorded and can be made available.

11.3 Logbook Pages

RJLG maintains an electronic logbook and tracking system and will make the records available.

11.4 Sketches

RJLG does not normally maintain sketches of the particles analyzed but can do so in special circumstances. Since RJLG expects that the TEM samples prepared from the EPA El Dorado air filters will be transferred from the TEM to the SEM for additional imaging, RJLG will maintain and make available copies of electronic or handwritten sketches.

Tumors or Mesothelioma in AF/HAN Rats Following Inhalation," Risk Analysis, 15, p. 181-195.

RJLG requests that any sketches recorded by Lab/Cor, but which have not been produced in response to previous requests, be made available for review.

11.5 Images

All images recorded are saved electronically. RJLG will make copies of any images recorded available.

RJLG requests that copies of images recorded by Lab/Cor, but which have not been produced in response to previous requests, be made available for review.

11.6 Spectra

All EDS spectra are electronically recorded and maintained. RJLG will provide copies of all recorded spectra.

11.7 Diffraction Patterns

Selected area electron diffraction (SAED) patterns are recorded electronically and processed to determine orientation relative to the electron beam, major crystal faces and potential mineral species of particles. These data are stored electronically. RJLG will make copies of SAED data available

More than 30% of the SAED patterns provided by Lab/Cor were illegible scans of printed documents. It is apparent that Lab/Cor maintains electronic copies of most, if not all, SAED patterns. Given EPA's position[60] that RJLG failed to obtain all relevant information and samples before commenting on the laboratory analysis, RJLG requests that all such electronic copies be produced for review.

11.8 Chain of Custody Forms

RJLG will maintain and provide copies of all chain of custody documentation.

11.9 Sample Tracking Sheets

RJLG maintains an electronic tracking system and will make the records available.

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[60] EPA 2006 Response, page 3

11.10 Quality Control ("QC") Sample Information

RJLG will maintain and provide information for QC samples analyzed.

11.11 Required Calibrations

RJLG maintains all required calibration records as part of its laboratory certification requirements for the NVLAP, AIHA, NELAC, and CA ELAP certification processes and will provide calibrations relevant to the time frame in which EPA air samples are analyzed for the instrumentation used in processing the samples.

Lab/Cor's data production did not include any relevant calibration data, including camera constants for SAED analysis, reference asbestos mineral photographs or EDS patterns used to calibrate their EDS system(s). During its review of Lab/Cor's data, RJLG went to extraordinary lengths to determine such information given the data provided. Given EPA's concern[61] that RJLG's analysis was lacking or limited by the available information or samples, RJLG renews its request that all such data and information be produced by Lab/Cor.

11.12 Technical Notes

RJLG will provide copies of technical notes made for any samples analyzed that are produced by EPA.

11.13 Instrumentation Descriptions

The list of relevant instrumentation is attached.

11.14 Qualifications

The list of relevant staff qualifications is attached.

12.0 Bundles

How would the R.J. Lee Group,[sic] when using an asbestos regulatory air analytical method (i.e., NIOSH or AHERA) or International Standards Organization ("ISO") method 10312, count bundles of asbestos fibers?

The referenced analytical methods provide definitions of bundles and instructions as to how these asbestos structures are to be counted. RJLG complies with these instructions.

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[61] EPA 2006, page 3.

EPA Region 9 suggests (2006, page 7) that RJLG ignored the presence of bundles in the Lab/Cor data when performing its evaluation of fiber dimensions. Our experience with data from other laboratories indicates some record the width of the component fiber while others record the overall width of the bundle. It was not clear what Lab/Cor's procedure was in recording the dimensions of bundles. Therefore, bundles were not included in the analysis of fiber dimensions.

The available data suggest the "bundles" identified by Lab/Cor may not even be bundles but that they may be cleavage fragments with evidence of striations. Fewer than 5% of all amphibole structures counted were bundles, hardly indicative of an asbestos fiber population. Eighty-five percent (85%) of the "bundles" were wider than 0.5 μm and 60% were wider than 1 μm (they basically had the same dimensions as the "fibers"). There were no photographs of bundles produced by EPA and very few diffraction patterns. RJLG requests that Lab/Cor's TEM grid preparations with reported bundles be produced so that the actual nature of these particles can be determined.

13.0 Selected Area Diffraction Patterns

How the R.J. Lee Group, [sic] when analyzing the selected area electron diffraction ("SAED) pattern of an amphibole mineral fiber, would distinguish between asbestos and non-asbestos mineral fibers?

It is recognized that amphibole asbestos fibers preferentially orient along the {100} crystal face. (The symbol {hkl} represents the Miller indices of all symmetrically equivalent faces). The principal cleavage direction in amphiboles is parallel to {110}. An amphibole crystal that grew as a fiber should show {100} faces but lack {110} faces. An amphibole crystal that is a cleavage fragment will show {110} faces and may or may not show {100} faces. Indexing the faces of a crystal and documenting a {110} face, will help to identify the crystal as a cleavage fragment.

Selected area electron diffraction (SAED) patterns are measured to determine the angles and distances (d-spacings) between atomic planes [hkl] in a crystal. This information is used for mineral identification by comparison of these results with standard reference patterns. Once a pattern is indexed, the crystal zone axis [uvw] parallel to the direction of the incident electron beam can be determined.

The relationship between the zone axis, angular tilt and crystal faces are illustrated in Figure xx. An example of measured crystal faces are shown in Figures xx and xx.

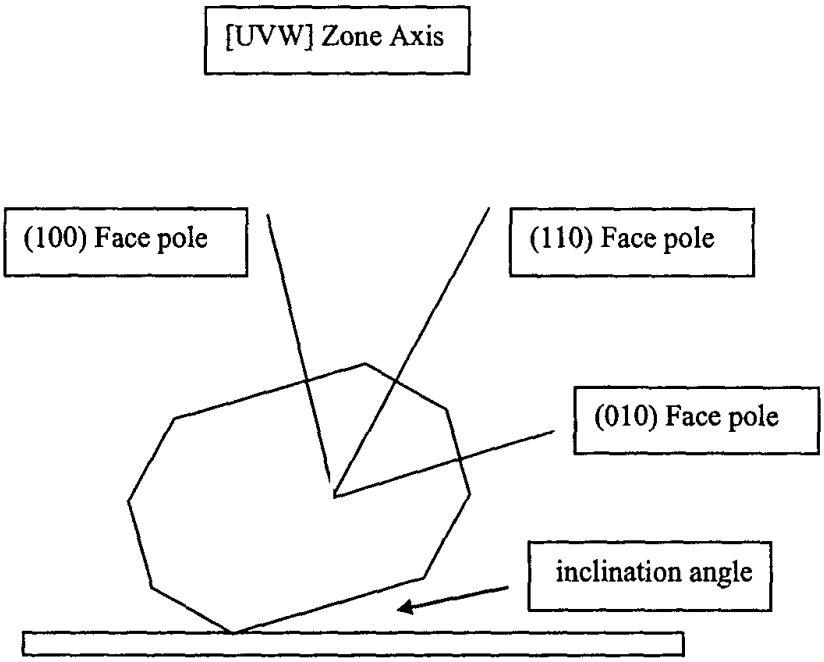

Figure xx. Example of relationship between TEM zone axis and crystal faces

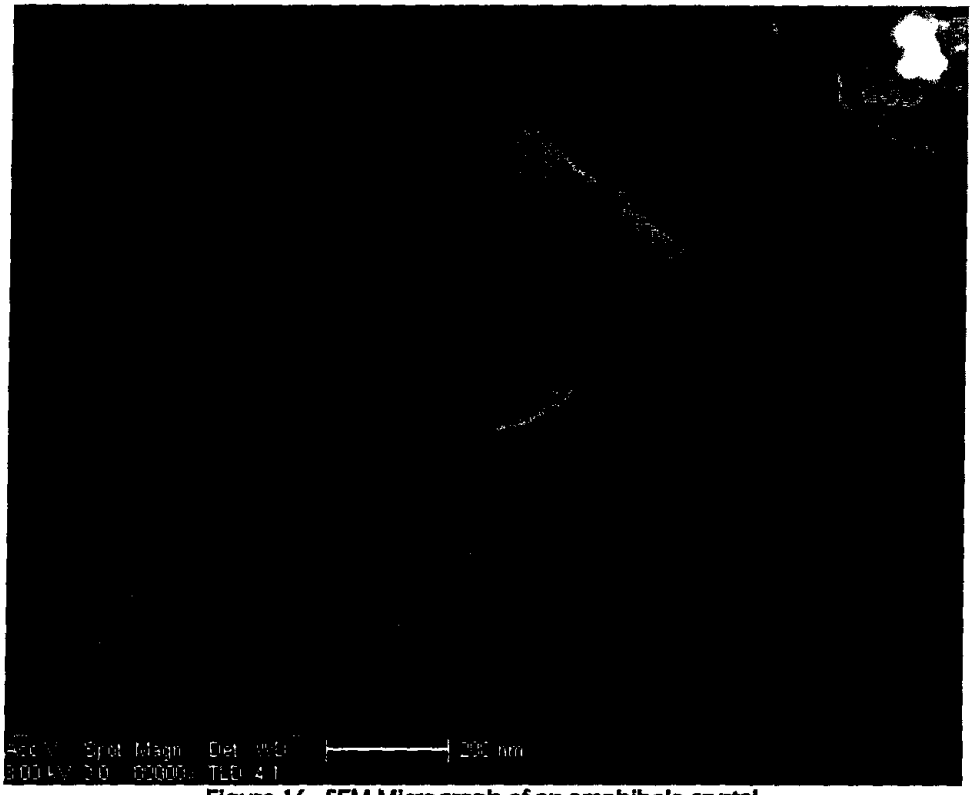

Figure 16. SEM Micrograph of an amphibole crystal

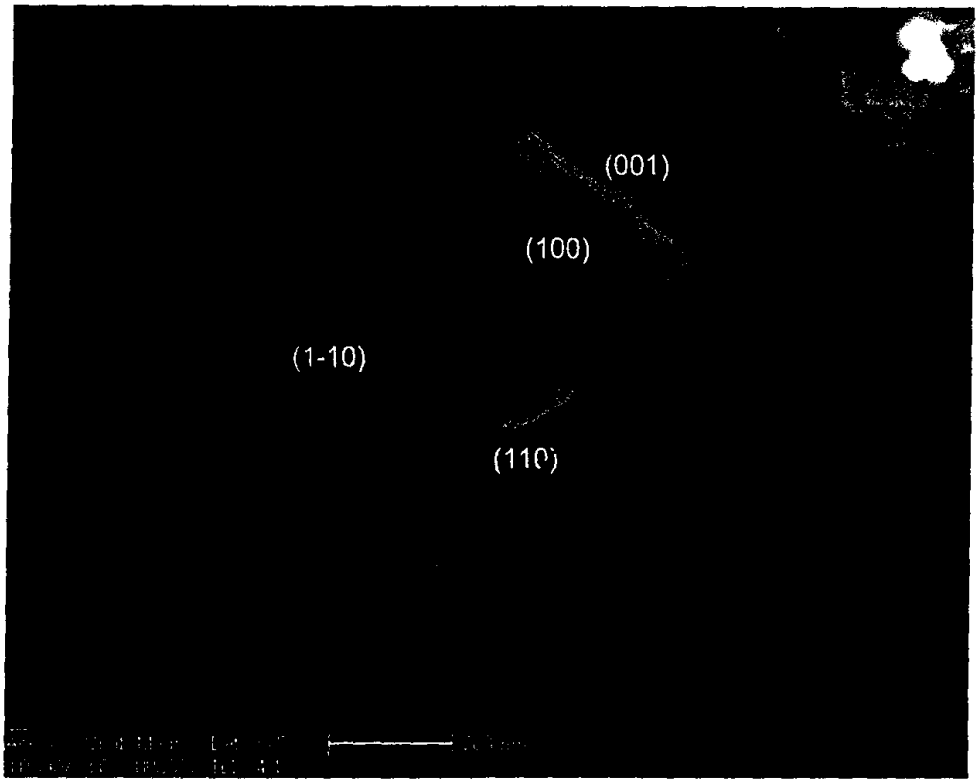

Figure 17. SEM Micrograph of an amphibole crystal with superimposed crystal faces.

The crystallographic zone axes from the Lab/Cor data are plotted in stereographic projection in Figure xx. Since the same zone axes were found multiple times, the data in each Figure are contoured to show the concentration of the zone axes. The average zone (vector mean of the data) plots essentially along the (110) face pole. The majority of the zone axes (~70%) lie in the region between the (110) face pole and the [110] zone axis. This is indicative that the majority of the crystals analyzed are not asbestos fibers. For comparison, Figure xx shows the clustering of the zones for tremolite asbestos from Jamestown, CA (a tremolite used in various animal studies). The zone axes are clustered around the (100) face poles with a few near (1-10), indicating these are from a population of asbestos fibers. Note: SAED patterns that exhibit patterns near (100) are not, in and of themselves, indicative of an asbestos fiber, but do present evidence that the fiber may be asbestos. Additional information, such as particle morphology and/or chemistry, is needed for a final determination.

June 6, 2006

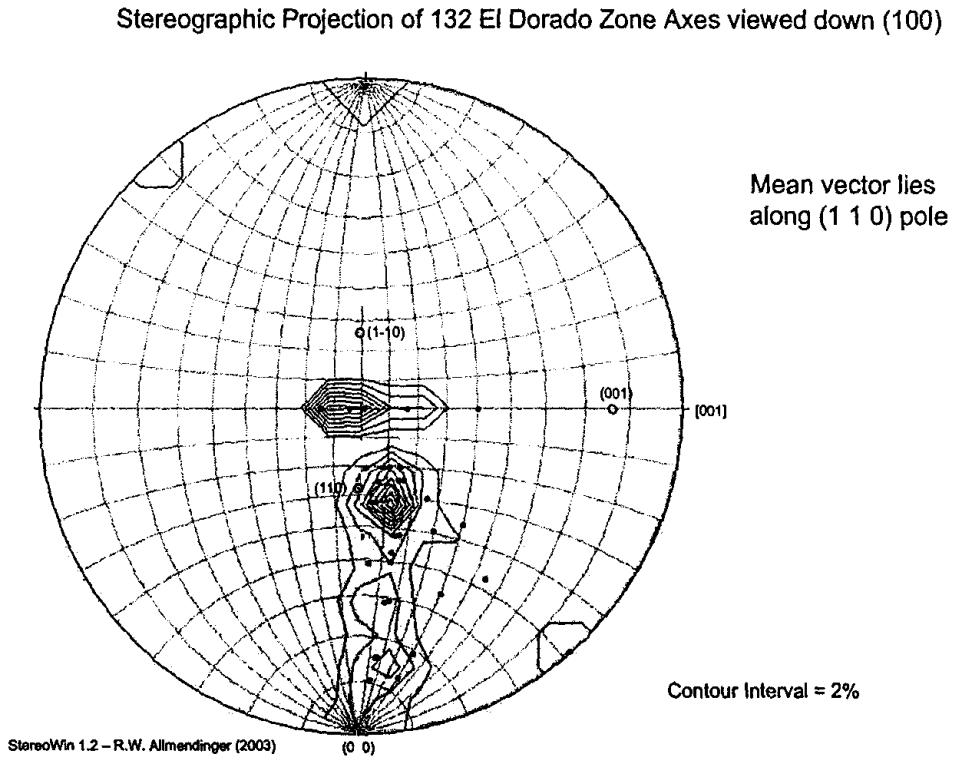

Figure 18. Stereographic projections of 132 identified zone axis patterns from the Lab/Cor data set. The graph is indicative of a population of nonasbestos particles.

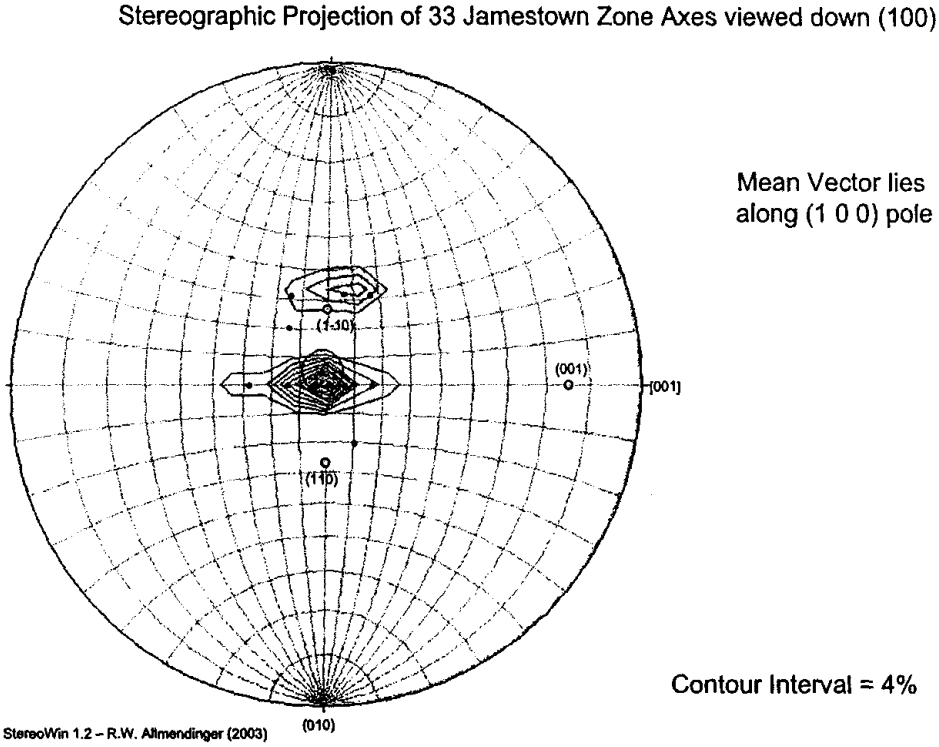

Figure 19. Stereographic projection of the zone axes from a population of tremolite asbestos from Jamestown, CA. The zones are centered around the {100} pole.

14.0 Rock Samples

The rock samples collected in and around the El Dorado Hills test site on January 10, 2006 were analyzed using polarized light microscopy in general accordance with the principals of geology. Verification of the mineral identification was performed using PLM (40 CFR Part 763, Appendix E to Subpart E). Minerals that could potentially be amphibole were removed from the rocks, lightly ground in a mortar and pestle and deposited onto SEM stubs. The particles on the stubs were examined in an SEM to obtain images of representative particles. In addition, finely ground particles were deposited onto a filter and examined in the TEM for elongated particles. EDS spectra of representative particles that were at least 3:1 (aspect ratio) were collected.

14.1 Documents, count sheets, EDS spectra

Documents, count sheets, EDS spectra and photographs generated during the analyses of the rock samples are Attachment xx to this response.

14.2 Images

All photographs related to the rock samples are Attachment xx to this response. These include field shots taken at EDH as well as PLM, SEM, and TEM photographs of representative particles.

14.3 Chain-of-Custody

The chain-of-custody for the rock samples is Attachment xx to this response.

15.0 Elutriator Tests

Four soil samples were evaluated using an elutriator procedure described in "Modified Elutriator Method for the Determination of Asbestos in Soils and Bulk Materials", by Berman and Kolk, May 23, 2000. Generated filters from the elutriator were analyzed in general accordance with ISO 10312.

15.1 Counts Sheets and Laboratory Notes

TEM count sheets for these analyses are Attachment xx. Laboratory notes related to the generation of the air filter for each sample are included.

15.2 Chain-of-Custody

The chain-of-custody for the soil samples are Attachment xx as part of response item 2.

Attachment B Qualifications of Richard J. Lee, Ph.D.

Richard J. Lee, Ph.D., has been involved in the development of methods for the identification of asbestos and other airborne particles for more than 30 years. Dr. Lee authored the first computer software for the analysis of electron diffraction patterns and the automated sizing and chemical analysis of asbestos and other particles. Dr Lee was an active participant in the first ASTM committee whose goal was to develop and test a TEM method for the analysis of asbestos, and was a co-author of the publication that resulted.

Dr Lee has served, from time to time, as an advisor to EPA for more than 25 years, beginning with the design of an EPA laboratory for the analysis of asbestos and other particulate, and serving as a peer reviewer/consulting expert in the development and writing of EPA's Yamate method. Dr. Lee authored the TEM analysis method instituted as part of the AHERA rules. Dr. Lee has performed the laboratory analysis underlying more than 30 EPA projects, including EPA's assessment of the airborne asbestos concentrations in public buildings, and has served as a peer reviewer on other EPA projects. Dr. Lee served on the HEI peer review panel to assess the significance of asbestos in public buildings, and was one of the authors of the landmark report that resulted from that review.

Dr. Lee and his staff actively consulted with and supported EPA Region 2 in evaluating contamination in NYC buildings that were impacted by the Events of 9/11. Dr. Lee and his staff designed, implemented, provided oversight and conducted sampling and laboratory analyses for building remediation. His group also planned, organized, developed protocols and conducted health and safety and community air quality monitoring programs in and around the impacted area and during deconstruction/demolition of one of the buildings. Dr. Lee and his staff reported project status and results to EPA Region 2 on a weekly basis during the course of the multi-year study. Dr. Lee and his staff actively supported EPA's Office of Research and Development in the development of a method to evaluate dusts from the Lower Manhattan district associated with the World Trade Center disaster. Dr. Lee and his staff supported EPA's investigation of potential emissions of tremolite asbestos

from the Southdown Quarry in New Jersey. Dr. Lee has served on ASTM committees to define methods for the analysis of asbestos.

Dr Lee has served as an expert witness in state and federal courts in numerous asbestos related cases over the last twenty years. Dr Lee is currently retained by W. R. Grace & Co. in the matter of United States of America v. W. R. Grace, et al (CR-05-070M-DWM D. Montana). Dr. Lee has published the most extensive surveys of asbestos concentrations in public buildings ever performed as well as more than 100 xxx

Dr Lee supervised the data review and the laboratory analysis conducted in conjunction with RJLG's review of EPA's analytical data from the El Dorado Hills Asbestos Evaluation Project.

Dr Lee holds a Ph. D. in theoretical physics, and was previously employed by US Steel Research where he was active in the development of asbestos analysis methods before forming RJ Lee Group in 1985.

AMERICAN JOURNAL OF INDUSTRIAL MEDICINE 32:592-594 (1997)

Major and Trace Element Compositions of the UICC Standard Asbestos Samples

D.R. Bowes, DSc,* and C.M. Farrow, PhD

Major and trace element compositions for chrysotile (2 samples), amosite, crocidolite, and anthophyllite UICC standard asbestos samples have been determined using UV-visible spectrophotometry, atomic absorption spectometry, flame photometry, volumetric analysis, and gravimetric analysis for major elements and x-ray and optical spectrometry for trace elements. The trace element data are for Li, S, Cl, Sc, V, Cr, Co, Ni, Cu, Zn, Ga, Rb, Sr, Zr, Nb, Ba, La, Ce, Pb, and Th and distribution in the various mineral phases is discussed. Am. J. Ind. Med. 32:592–594, 1997. © *1997 Wiley-Liss, Inc.*

KEY WORDS: amosite; anthophyllite; asbestos; chemical composition; chrysotile; crocidolite; major element; trace element

INTRODUCTION

With information relating to the type and quantity of mineral impurities in the UICC asbestos samples (chrysotile A and B, crocidolite, amosite, and anthophyllite) and fiber size distributions provided by Kohyama et al. [1996], the significance of their chemical compositions can be more readily evaluated. To assist this, new major element analyses, including H_2O+ determined directly and not from loss on ignition, together with data for Li, S, Cl, Sc, V, Cr, Co, Ni, Cu, Zn, Ga, Rb, Sr, Zr, Nb, Ba, La, Ce, Pb, and Th are presented. These provide a basis for comparison with major element analyses Kohyama et al. [1996, Table I], obtained mainly using different analytical techniques, and the limited trace element data also given by these authors. They also provide a basis for discussion of trace element distribution in the various mineral phases.

METHODS

The samples were reduced to 100 mesh in an agate mill for measurement of major elements, and a proportion reduced to 250 mesh in an agate ball mill for determination of H_2O+, CO_2, and trace elements. This meant that SiO_2 was the only possible contaminant and the extremely small effect on the agate after crushing of many thousands of mineral and rock samples is indicative of negligible contamination for an individual sample. The powders were then dried at 110°C so eliminating H_2O- determination and errors inherent in variations in hygroscopic water content with varying atmospheric conditions.

Most major elements were determined using rapid methods of analysis [Riley, 1958a; Potts, 1987, pp 66–67]. Following solution of the products of a NaOH fusion of 100 mesh powder in a silver crucible, SiO_2 was determined by UV-spectrophotometry. Following solution of the products of digestion of 100 mesh powder with HF and HPO_4 in a platinum crucible, TiO_2, Al_2O_3, total Fe (as Fe_2O_3), MnO, and P_2O_5 were determined by UV-visible spectrophotometry, CaO, and MgO by atomic absorption spectrometry [Potts, 1987, pp 122–125] and Na_2O and K_2O by emission spectrometry [Potts, 1987, pp 62–64] from aliquots of the same solution. FeO was determined by titration with potassium dichromate, using diphenylamine sulfonate as indicator, following attack by HF and H_2SO_4 on 100 mesh powder [Pratt, 1894; Potts, 1987, pp 67–70]; Fe_2O_3 was calculated by subtraction of the Fe_2O_3 equivalent of FeO from total Fe (as Fe_2O_3). H_2O+, and CO_2 were determined directly from 250 mesh powder using the method of Riley [1958b] with these components released at 1,100°C in an atmosphere of nitrogen and collected separately in absorption tubes. In this way errors associated both with the variable release of (OH)

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Accepted 1 July 1997

TABLE 1. Major (wt%) and Trace (ppm) Element Compositions of the UICC Standard Asbestos Samples

	Chrysotile A		Chrysotile B		Amosite		Crocidolite		Anthophyllite	
	Shibani, Zimbabwe		Canada		Penge, S Africa		Koegas, S Africa		Paakilla, Finland	
SiO_2	39.62[a]	40.24[b]	38.58[a]	38.35[b]	49.51[a]	50.63[b]	48.47[a]	49.01[b]	57.99[a]	56.04[b]
TiO_2	0.16	0.02	0.14	Trace	0.40	—	0.08	0.02	0.08	0.03
Al_2O_3	0.83	0.77	0.59	0.40	0.00	0.55	0.00	0.06	1.38	1.00
Fe_2O_3	2.14	1.99	2.70	2.41	2.83	1.90	18.97	19.14	0.39	—
FeO	0.41	0.49	1.12	1.15	33.86	35.41	18.76	20.02	5.12	5.83
MnO	0.07	0.06	0.07	0.06	1.80	1.82	0.12	0.11	0.15	0.17
MgO	40.00	42.97	40.49	43.54	6.76	6.44	2.72	2.33	28.93	31.56
CaO	0.52	0.33	0.20	0.17	0.39	0.51	0.92	1.08	0.33	0.29
Na_2O	0.25	Trace	0.29	0.02	0.12	0.02	5.57	5.60	0.22	0.03
K_2O	0.00	Trace	0.19	0.02	0.13	0.27	0.03	0.06	0.47	0.43
P_2O_5	0.01	—	0.01	—	0.01	—	0.03	—	0.01	—
H_2O+	13.38	12.69*	14.00	13.76*	2.72	2.32*	2.64	2.34*	4.33	4.28*
CO_2	2.26	—	—	—	1.55	—	1.41	—	0.34	—
Total	99.65	99.56	99.83	99.88	100.08	99.87	99.72	99.79	99.74	99.66
Li	<1	—	<1	—	2.5	—	11	—	7.9	—
S	1,290	1,240	735	420	350	960	140	120	3.510	2,840
Cl	230	500	1.420	2.500	130	300	90	Trace	120	100
Sc	2.1	—	4.6	—	3.6	—	5.2	—	3.6	—
V	17	—	8.5	—	7.7	—	9.2	—	14	—
Cr	2,600	1,580	900	480	<15	Trace	<15	—	1,330	880
Co	103	Trace	82	Trace	25	—	20	—	70	Trace
Ni	1,850	1,650	975	940	33	Trace	18	Trace	1,125	1,100
Cu	7	—	8	—	16	—	8	—	30	Trace
Zn	<3	Trace	25	Trace	16	—	14	—	234	240
Ga	<1	—	<1	—	<1	—	2	—	<1	—
Rb	<5	—	<5	—	20	—	5	—	20	—
Sr	<10	—	<10	—	<10	—	20	—	<10	—
Zr	15	—	15	—	20	—	10	—	15	—
Nb	<3	—	4	—	<3	—	<3	—	3	—
Ba	<20	—	<20	—	<20	—	<20	—	<20	—
La	<10	—	<10	—	<10	—	<10	—	<10	—
Ce	<10	—	100	—	<10	—	<10	—	<10	—
Pb	38	—	15	—	11	—	6	—	<5	—
Th	<5	—	<5	—	<5	—	<5	—	<5	—

[a]Analyzed at University of Glasgow; methods given in text.
[b]From Kohyama et al. [1996, table I]; recalculated to H_2O- free and trace elements to parts per million for comparison with a; * from loss on ignition.

and other volatile constituents, and only partial oxidation of FeO to Fe_2O_3 [Potts, 1987, pp 72–73] were avoided. By applying this method, the calculations used by Kohyama et al. [1996, p 519] in the estimation of H_2O+ were eliminated. Trace elements, except for Li, Sc, and V, were determined from pressed pellets of 250 mesh powder and an organic binder resin using a Phillips PW 1220 semiautomatic spectrometer with a Mo tube target [Leake et al., 1969; Harvey and Atkin, 1982]. Li, Sc, and V were determined by optical spectrography using a large Hilger-Watts quartz-glass spectrograph [Potts, 1987, pp 200–201, 206–209].

Standards used were 110°C dried (spec pure or A R) SiO_2, TiO_2, ammonium alum (for Al), iron wire, $MnSO_4.4H_2O$, magnesium ribbon, $CaCO_3$, Na_2SO_4, K_2SO_4, and KH_2PO_4 (for P). The major element data in Table 1 represent either corresponding duplicate analyses or average values of two closely corresponding analyses. As internal checks, each batch of analyses included a repeatedly ana-

lyzed international standard silicate rock powder. Reproducibility of results from these internal checks forms the basis for confidence in the new data, as does the corresponding reproducibility of results for standard powders analyzed in the batches of hundreds of other silicate rock and mineral powders analyzed each year in the same laboratory by the same methods.

DATA AND ASSESSMENT

The new analytical data are set out in Table I in columns labeled a. In columns labeled b are the data from Kohyama et al. [1996, table I] recalculated to H_2O- free and trace elements to parts per million to assist comparison. As the data given as H_2O+ by Kohyama et al. [1966] were determined by loss on ignition and include Cl, S, and other volatile constituents such as CO_2, they are designated by an asterisk (*) to distinguish from the directly determined H_2O+ in the new data

For most elements, there is general correspondence in the oxide percentages determined in the present work and those given by Kohyama et al. [1996]. However, there are marked differences for TiO_2 analysis for which our laboratory has obtained consistent results for standard powders. There are also marked differences for Na_2O at low concentrations but correspondence (for crocidolite) at high concentrations. Our data also show lower values of MgO for chrysotile A, chrysotile B, and anthophyllite than those of Kohyama et al. [1996]. This could account for the assessment of these authors [p 519] that MgO in chrysotile B is slightly greater than expected in an ideal chemical composition. Reassessment of conclusions based on H_2O+ values from loss on ignition is also required as a result of our direct determination of volatile constituents. In addition, the presence of CO_2 suggests the presence of a carbonate phase(s).

While the conclusion of Kohyama et al. [1996] that both the amosite and the crocidolite were 99% pure with the only other mineral phase identified being quartz (<1%) may require minor revision if a carbonate phase is identified, it does point to the trace element content being at least mainly in the crystal lattices. The 1.8% MnO in the amosite is consistent with the well-established $(Mg, Fe)^{2+}$–Mn^{2+} substitution in amphiboles with the composition $(Mg, Fe^{2+}, Mn)_7[Si_8O_{22}](OH)_2$ (cummingtonite–grunerite series) [Deer et al., 1963, p 235, table 36; 1997] and Co, Ni, Cu, Zn, and Pb would be corresponding substitutions. However the 5.6% Na_2O in the crocidolite ($Na_2Fe_3^{2+}Fe_2^{3+}[Si_8O_{22}](OH, F)_2$—now referred to as riebeckite-asbestos by mineralogists [Leake et al., 1997])—which is consistent with other published data [Deer et al., 1963, table 54; 1997], represents a major lattice-forming constituent. Cl would be substituted for (OH) as would any F (not determined). The S could be distributed unbonded in the lattice or as minute grains of (?Fe) sulfide.

With chrysotile A and B estimated to contain 94% and 92% chrysotile ($Mg_3[Si_2O_5](OH)_4$), respectively, most of the trace elements are also likely to represent lattice substitution, particularly Ni^{2+} (and Mn, Co, Cu, Zn, and Pb) with Mg^{2+}. Cr could be unbonded and distributed through the relatively weakly bonded lattice which is of a layered type similar to that in some clay minerals [Deer et al., 1992, pp 345–346]. This could also be the case for S, or it could be present in very fine-grained dispersed sulfide grains. Cl would be substituted for (OH).

With the anthophyllite standard estimated by Kohyama et al. [1996, table II] to contain as little as 60% anthophyllite $[(Mg,Fe^{2+})_7[Si_8O_{22}](OH)_2]$, further studies are needed to permit assessment of the trace element distribution in this and the other mineral phases present. However, regarding the major elements, the high MgO is consistent with the estimated 30% talc $[Mg_6[Si_8O_{20}](OH)_4]$, and the >5% FeO and >1% Al_2O_3 with the presence of 5% of each of chlorite $[(Mg,Al,Fe)_{12}[(Si,Al)_8O_{20}](OH)_{16}]$ and phlogopite $[K_2(Mg,Fe^{2+})_6[Si_6Al_2O_{20}](OH,F)_4]$. The presence of phlogopite would also account for the ca 0.5% K_2O. The S (ca 3500 ppm) could be present in dispersed sulfide grains.

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ERRATUM

Bowles DR, Farrow CM (1997): Major Trace Element Compositions of the UICC Standard Asbestos Samples Am J Ind Med 32:592-594

Table 1, p. 593, is incorrect. The third column, Glasgow analysis for Chrysotile B should include 1.45 CO_2.

The corrected table follows.

TABLE I. Major (wt%) and Trace (ppm) Element Compositions of the UICC Standard Asbestos Samples

	Chrysotile A		Chrysotile B		Amosite		Crocidolite		Anthophyllite	
	Shibani, Zimbabwe		Canada		Penge, S Africa		Koegas, S Africa		Paakilla, Finland	
SiO_2	39.62[a]	40.24[b]	38.58[a]	38.35[b]	49.51[a]	50.63[b]	48.47[a]	49.01[b]	57.99[a]	56.04[b]
TiO_2	0.16	0.02	0.14	Trace	0.40	—	0.08	0.02	0.08	0.03
Al_2O_3	0.83	0.77	0.59	0.40	0.00	0.55	0.00	0.06	1.38	1.00
Fe_2O_3	2.14	1.99	2.70	2.41	2.83	1.90	18.97	19.14	0.39	—
FeO	0.41	0.49	1.12	1.15	33.86	35.41	18.76	20.02	5.12	5.83
MnO	0.07	0.06	0.07	0.06	1.80	1.82	0.12	0.11	0.15	0.17
MgO	40.00	42.97	40.49	43.54	6.76	6.44	2.72	2.33	28.93	31.56
CaO	0.52	0.33	0.20	0.17	0.39	0.51	0.92	1.08	0.33	0.29
Na_2O	0.25	Trace	0.29	0.02	0.12	0.02	5.57	5.60	0.22	0.03
K_2O	0.00	Trace	0.19	0.02	0.13	0.27	0.03	0.06	0.47	0.43
P_2O_5	0.01	—	0.01	—	0.01	—	0.03	—	0.01	—
H_2O+	13.38	12.69*	14.00	13.76*	2.72	2.32*	2.64	2.34*	4.33	4.28*
CO_2	2.26	—	1.45	—	1.55	—	1.41	—	0.34	—
Total	99.65	99.56	99.83	99.88	100.08	99.87	99.72	99.79	99.74	99.66
Li	<1	—	<1	—	2.5	—	11	—	7.9	—
S	1,290	1,240	735	420	350	960	140	120	3,510	2,840
Cl	230	500	1,420	2,500	130	300	90	Trace	120	100
Sc	2.1	—	4.6	—	3.6	—	5.2	—	3.6	—
V	17	—	8.5	—	7.7	—	9.2	—	14	—
Cr	2,600	1,580	900	480	<15	Trace	<15	—	1,330	880
Co	103	Trace	82	Trace	25	—	20	—	70	Trace
Ni	1,850	1,650	975	940	33	Trace	18	Trace	1,125	1,100
Cu	7	—	8	—	16	—	8	—	30	Trace
Zn	<3	Trace	25	Trace	16	—	14	—	234	240
Ga	<1	—	<1	—	<1	—	2	—	<1	—
Rb	<5	—	<5	—	20	—	5	—	20	—
Sr	<10	—	<10	—	<10	—	20	—	<10	—
Zr	15	—	15	—	20	—	10	—	15	—
Nb	<3	—	4	—	<3	—	<3	—	3	—
Ba	<20	—	<20	—	<20	—	<20	—	<20	—
La	<10	—	<10	—	<10	—	<10	—	<10	—
Ce	<10	—	100	—	<10	—	<10	—	<10	—
Pb	38	—	15	—	11	—	6	—	<5	—
Th	<5	—	<5	—	<5	—	<5	—	<5	—

[a]Analyzed at University of Glasgow; methods given in text.
[b]From Kohyama et al. [1996, table I]; recalculated to H_2O- free and trace elements to parts per million for comparison with a; * from loss on ignition.

The publisher regrets this error.

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1867a

Uncommon Commercial Asbestos

This standard reference material (SRM) is comprised of three uncommon commercial asbestos materials (tremolite asbestos, actinolite asbestos, and anthophyllite asbestos) intended for use in the identification of these minerals by polarized light microscopy (PLM) [1,2]. The certified values for SRM 1867a are identical to the original certified values provided in the previous issuance of this material, SRM 1867. Additional reference values and information values are provided with this reissue. The materials used in SRM 1867a came from the original lots of material used for SRM 1867 and represent the last of the original supply. Each unit of SRM 1867a consists of a set of three bottles, each containing several grams of one of the three mine-grade asbestos materials, with certified optical properties that can be measured by PLM.

#1

The three materials contained in this SRM are single representatives of their mineral types and cannot represent all the variability inherent to these mineral species. The changes in optical properties due to differences in chemical composition for tremolite and actinolite from the materials contained in this SRM are detailed in Reference 3. The unique morphology of asbestos may alter the optical properties of tremolite, actinolite, and anthophyllite asbestos from those reported for the materials contained in this SRM, as described in Reference 4.

#2

#3

Certified Properties and Uncertainties: Refractive indices were measured in the range of visible wavelengths by using the double variation technique on individual fibers oriented with a spindle stage [5,6] in immersion liquids calibrated by an independent technique (minimum deviation). Refractive indices were fit to a Cauchy equation to calculate the refractive index at 589.3 nm (n_D) (Table 1) and to provide the dispersion constants (Table 2). Expanded uncertainties were calculated as Working-Hotelling confidence intervals [7,8] with a coverage factor 2 corrected for bias from calibration measurements.

Reference Values: Extinction angles (Table 1) were measured from oriented fibers with uncertainties on the means calculated as expanded uncertainties with a coverage factor 2 [8]. The compositions of the materials were determined by electron probe microanalysis (EPMA) on polished specimens and are given in Table 3 in % mass fraction of the oxide and in atoms per formula unit (apfu) calculated on the basis of 23 oxygens. The unit cell dimensions (Table 4) of the materials were determined by X-ray diffraction (XRD) on powdered specimens using an internal standard. Reference values are non-certified values that are the best estimate of the true value; however, the values, which are based on determinations done with a single reliable method, do not meet the NIST requirement for certification and are provided with associated uncertainties that may reflect only measurement precision and may not include all sources of uncertainty.

Expiration of Certification: The certification of this SRM is valid until **31 December 2017**, within the uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given. However, the certification will be nullified if the SRM is damaged, contaminated, or otherwise modified.

The support aspects involved in the certification and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald.

Richard R. Cavanagh, Chief
Surface and Microanalysis Science Division

Gaithersburg, MD 20899
Certificate Issue Date: 12 March 2003

John Rumble, Jr., Chief
Measurement Services Division

Characterization of the Standard Reference Material was performed in the NIST Surface and Microanalysis Science Division by J.R. Verkouteren.

Data from the original issue (SRM 1867) were collected by J.R. Verkouteren, J.M. Phelps, E.S. Windsor, D.M. Hues, and E.B. Steel of the NIST Surface and Microanalysis Science Division; and by R.L. Perkins, B.W. Harvey, S.S. Doorn, and T.F. Bergin of Research Triangle Institute, Research Triangle Park, NC.

Statistical analysis of the certification data was provided by S.D. Leigh of the NIST Statistical Engineering Division.

Source and Packaging of Materials:[1] The anthophyllite was purchased from Ward's Natural Science Establishment, Incorporated, who report the sample origin as the Rakabedo Mines near Udaipur, India. The actinolite was collected by Eric Steel and John Phelps of NIST at a construction site in Fairfax County, VA. The tremolite was collected by Stephen Bezore of the State of California Department of Conservation from the Conda deposit [9] near Barstow, CA. The materials were prepared and packaged by Research Triangle Institute, Research Triangle Park, NC, under the direction of R.L. Perkins.

INSTRUCTIONS FOR USE

Use: The optical properties of the fibers can be measured by immersing the fibers in liquids of known refractive index, either in random orientation or more precisely with fibers oriented on a spindle stage. There are specific guidelines included with this SRM for the measurement of refractive index from randomly oriented fibers in grain mounts. These guidelines may not be applicable to the measurement of unknown fibers in bulk insulation materials, soils, mined products, etc., because they assume relatively pure materials for which the variation in refractive index is due solely to fiber orientation (see *Measurement Guidelines* section).

Special Handling Requirements: Proper procedures for the safe handling of asbestos must be employed during preparation, analysis, and storage of this SRM. Store this SRM in the original bottles, tightly closed, and within the range of normal room temperature and humidity.

CERTIFIED PROPERTIES

Table 1. Refractive index (n) at the sodium D line (589.3 nm) for principal orientations. The uncertainties of the values of n_D are ± 0.0007. For orientation of principal refractive indices with fiber morphology, see the *Measurement Guidelines* section .

Asbestos type	$\gamma\ n_D$	$\beta\ n_D$	$\alpha\ n_D$	Birefringence ($\gamma\ n_D - \alpha\ n_D$)	Extinction angle[a]
Anthophyllite	1.6362	1.6273	1.6148	0.021	0 (parallel extinction)
Tremolite	1.6343	1.6230	1.6063	0.028	16.6 ± 0.3°
Actinolite	1.6393	1.6288	1.6126	0.027	15.9 ± 0.2°

[a] Provided as reference values only.

Table 2. To calculate the refractive index (n) at any wavelength (λ) in the range 470 nm to 620 nm, substitute

[1]Certain commercial equipment, instruments, or materials are identified in this certificate in order to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose

parameters a and b into the equation: $n(\lambda) = a + b/\lambda^2$. The uncertainties in the calculated refractive indices are not necessarily symmetric and vary with wavelength as follows: + 0.0005 and – 0.0015 @ 434 nm, + 0.0005 and – 0.0010 @ 486.1 nm, ± 0.0007 @ 589.3 nm, ± 0.0007 @ 656.3 nm. Uncertainties at all other wavelengths within the range 470 nm to 620 nm can be interpolated from these values.

	$n(\lambda) = a + b/\lambda^2$		
Asbestos type	γ	β	α
Anthophyllite	a = 1.61762 b = 6442	a = 1.61084 b = 5701	a = 1.59808 b = 5812
Tremolite	a = 1.61713 b = 5953	a = 1.60832 b = 5088	a = 1.59236 b = 4824
Actinolite	a = 1.61973 b = 6802	a = 1.61137 b = 6041	a = 1.59653 b = 5574

NOTE: The optical properties leading to certification were measured from the larger, single crystal fibers present in the samples. Some portion of the fibers in each sample will show anomalous optical properties due to the unique fibrillar structure of asbestos and {100} twinning. The anomalous properties of tremolite asbestos and actinolite asbestos include lower extinction angles or parallel extinction and slightly altered refractive indices, as detailed in [4].

REFERENCE VALUES

Table 3. Elemental compositions determined by EPMA calculated in % mass fraction of oxide (assuming all Fe as Fe^{2+}) and in atoms per formula unit (apfu) based on 23 oxygens. The ideal formulas for the minerals are: $Ca_2(Mg,Fe)_5Si_8O_{22}(OH)_2$ for tremolite and actinolite, $(Mg,Fe)_7Si_8O_{22}(OH)_2$ for anthophyllite. The mass fraction of each oxide with a concentration > 1 % mass fraction has a relative error of ≤ 10 %. The reference values are the means of results obtained by NIST using one analytical technique. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor is 2, (95 % confidence level). The concentrations of H_2O, F, and Cl were not determined.

Asbestos Type	% SiO_2	apfu Si	% FeO	apfu Fe	% MgO	apfu Mg	% CaO	apfu Ca	Mg/(Mg+Fe+Mn)
Anthophyllite	58.4	8.01	8.3	0.95	29.2	5.97	0.4	0.06	0.86
Tremolite[a]	56.3	7.88	1.3	0.16	23.6	4.93	13.1	1.97	0.96
Actinolite[b]	55.6	7.92	6.5	0.77	20.3	4.32	12.6	1.90	0.84

[a] Also contains 0.4 % mass fraction Al_2O_3 (0.07 apfu Al), 0.5 % mass fraction MnO (0.06 apfu Mn), and 0.2 % mass fraction Na_2O (0.05 apfu Na).

[b] Also contains 0.5 % mass fraction Al_2O_3 (0.09 apfu Al), 0.3 % mass fraction MnO (0.03 apfu Mn), and 0.1 % mass fraction Na_2O (0.02 apfu Na).

Table 4. Unit cell dimensions determined by XRD. The reference values are the means of results obtained by NIST using one analytical technique. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor is 2, (95 % confidence level).

Asbestos type	a, Å	b, Å	c, Å	β °
Anthophyllite	18.543 ± 0.004	18.010 ± 0.005	5.285 ± 0.005	---------
Tremolite	9.843 ± 0.002	18.063 ± 0.002	5.278 ± 0.004	104.75 ± 0.1
Actinolite	9.852 ± 0.002	18.088 ± 0.002	5.283 ± 0.002	104.69 ± 0.1

SUPPLEMENTAL INFORMATION

Table 5. Descriptive information from observations with low-power microscopy and polarized light microscopy and approximate concentrations of accessory phases determined by XRD analysis are provided for information purposes only in Table 5.

Anthophyllite

Texture: asbestiform[a]

Color: tan in hand specimen, colorless in plane polarized light

Concentration of anthophyllite: > 80 % mass fraction Talc is estimated to be present at concentrations of approximately 5 % mf to 15 % mf.

Tremolite

Texture: asbestiform[a] Some of the fibers are loose and others are more tightly bound together. A small amount of material may be massive.

Color: white to pale green in hand specimen, colorless in plane polarized light

Concentration of tremolite: > 90 % mass fraction Talc is estimated to be present at concentrations less than 5 % mf.

Actinolite

Texture: asbestiform[a] Some of the fibers are loose and others are more tightly bound together. A considerable amount of material may be massive. The massive material may contain significant clinochlore (chlorite).

Color: white to green in hand specimen, colorless in plane polarized light

Concentration of actinolite: > 80 % mass fraction of the fibrous material Clinochlore (chlorite) is estimated to be present at concentrations that can exceed 20 % mf if a considerable amount of massive material is present. The massive material can be easily segregated, leaving primarily actinolite in the fibrous portion. A small amount (< 5 % mass fraction) of talc may also be present in the fibrous component.

[a] Asbestiform: crystallizes with the habit of asbestos. These asbestos minerals possess properties such as long fiber length and high tensile strength. Under the light microscope, some portion of these samples exhibit the asbestiform habit as defined by several of the following characteristics: 1) mean aspect ratios ranging from 20:1 to 100:1 or higher for fibers longer than 5 µm, 2) very thin fibrils, usually less than 0.5 µm in width, 3) parallel fibers occurring in bundles, 4) fiber bundles displaying splayed ends, 5) fibers in the form of thin needles, 6) matted masses of individual fibers, and 7) fibers showing curvature.

MEASUREMENT GUIDELINES

Anthophyllite, actinolite, and tremolite are biaxial, and therefore, have three principal vibration directions that correspond to three principal refractive indices: α, β, and γ. Amphibole asbestos fibers are elongated along the *c* crystallographic axis, and tend to lay on surfaces parallel to *c* (*hk*0) in immersion slide mounts. The principal refractive indices are found only on the (100) and (010) surfaces, as shown in Figure 1. Since the largest refractive index, γ, is closest to the fiber axis, anthophyllite, tremolite, and actinolite are all optically length slow (positive sign of elongation).

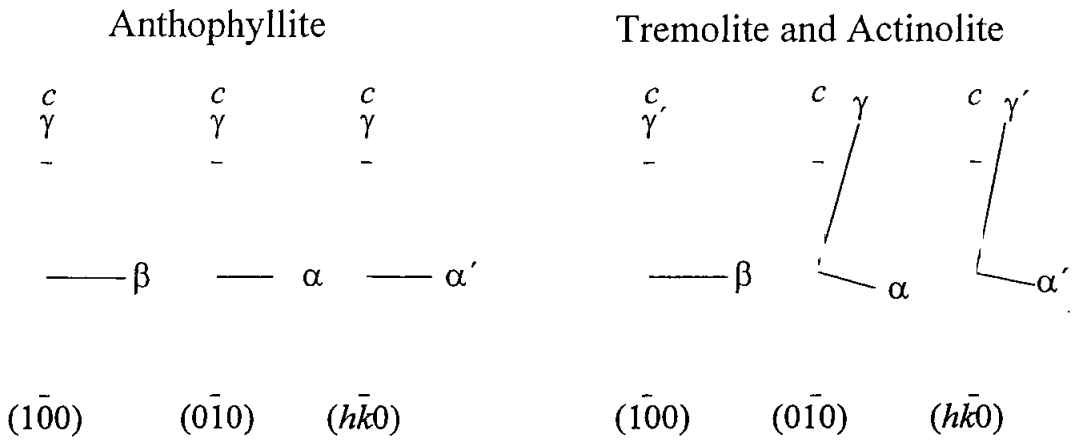

Figure 1. Possible orientations of amphibole asbestos fibers in immersion slide mounts.

The most common orientation that fibers will express in immersion slides mounts is shown by the (*hk*0) orientation. For anthophyllite, this means that γ can be measured from any fiber, but that the index perpendicular to fiber elongation for most fibers will have a value between α and β, represented by α'. For tremolite and actinolite, an (*hk*0) orientation will yield γ' (a value smaller than γ), α' (a value between α and β), and an extinction angle that may not be the true extinction angle (see Reference 4 for more detail).

To measure principal refractive indices for anthophyllite:

From slide immersion mounts, measure γ from any fiber by proper orientation of the fiber with respect to the polarizer. Observe a large number of fibers to determine the minimum refractive index perpendicular to elongation, which will correspond to α. Use the certified value of birefringence to estimate the immersion liquids that should be used to measure α. A relatively small number of fibers may display α, as an (010) orientation may not be particularly common. The maximum refractive index perpendicular to elongation will correspond to β. The (100) orientation in asbestos fibers may be more common than the (010) orientation.

To measure principal refractive indices for tremolite and actinolite:

From slide immersion mounts, determine γ by measurement of the index closest to the fiber elongation axis for approximately 50 fibers. Always place the fiber at the extinction position to measure refractive index. There should be a range in values of about 0.005 and the maximum value should correspond to the best estimate for γ. The number of fibers that display the maximum value should be a minority of the total number of fibers observed. Record the extinction angle for each fiber; use the extinction angle from the fibers that exhibit the maximum refractive index (γ) as the best estimate of the true value of extinction angle. As for anthophyllite, α and β are measured by determining the minimum and maximum value perpendicular (or near perpendicular) to elongation, respectively, by observation of a large number of fibers. Use the certified value of birefringence to estimate the immersion liquids that should be used to

measure α. Always place the fiber at the extinction position to measure refractive index. A relatively small number of fibers may display α, as an (010) orientation may not be particularly common. The maximum refractive index perpendicular (or near perpendicular) to elongation will correspond to β, and those fibers will show parallel extinction. The (100) orientation in asbestos fibers may be more common than the (010) orientation.

NOTE: The fibrillar structure and {100} twinning common to asbestos can produce anomalous optical properties in monoclinic amphiboles, such that some portion of the tremolite and actinolite fibers in this SRM will show parallel extinction for orientations other than (100) and/or will not exhibit the true extinction angle at any orientation. The fibrillar structure can alter the refractive indices observed perpendicular (or near perpendicular) to elongation such that a single value that is intermediate between α and β is observed. These, and other anomalous optical properties, are detailed in Reference 4. The anomalous effects of the fibrillar structure are expected to be more prevalent in the very thin fibers.

REFERENCES

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