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ASBESTOS IDENTIFICATION AND MEASUREMENT

CPL Sent to WR Benoit 5-9-83

Chron

PROCEEDINGS OF A TOPICAL SYMPOSIUM
APRIL 9, 1979
DES PLAINES, IL.

Sponsored by
THE AMERICAN CONFERENCE OF GOVERNMENTAL INDUSTRIAL HYGIENISTS



The American Conference of Governmental Industrial Hygienists
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ATT-174

LLA 002048

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Evaluation of Asbestos in Insulation

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The Environmental Protection Agency has undertaken the coordination of a nationwide effort to eliminate a possible threat to the health of school children posed by the past use of asbestos-containing materials in the nation's schools. The purpose, extent, and form of this effort can be best ascertained by contacting the EPA (800/424-9065; in Washington, D.C. call 554-1404). It is therefore important to have the tools and techniques available for rapid and dependable analysis of insulating, sound-proofing, and other materials used in schools for asbestiform minerals.

The task of cleaning up the nation's schools makes Hercules' task of cleaning the Augean stables seem like a Sunday picnic, and the cost will be monumental. Each school and each material in each school must be considered by informed experts who can decide the best solution to the problem. Removal is very costly and could, in some cases, actually increase the hazard. Leaving well enough alone should be the recommendation unless contraindicated, for example by high friability or a high probability of disturbance. Then encapsulation (sealing in with spray finishes) or isolation (such as installing a lowered ceiling) should be considered. Each situation must, however, be considered individually.

Anyone who must decide what to do must have reliable information on which to base his decision. In particular, he must know the composition of each possible asbestos-containing material. Ideally, he should know:

1. what asbestiform substances are presents;
2. in what proportion;
3. what other substances are present (e.g., cellulosic fibers, mineral wool, other glass fibers, vermiculite, talc, perlite, diatomaceous earth, organic fibers, clays, glass powder, quartz, gypsum,e tc.); and
4. size ranges for each substance.

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The third of these points is important because some of these substances are already known to be unhealthful and, the way matters are going, the others may well be declared hazardous in the future. Knowing what is present permits a more intelligent evaluation of the overall situation and may eliminate the need for a later more complete analysis.

Even the chemical analytical problem is formidable and many laboratories will be involved. McCrone Associates' two laboratories in Chicago and London have analyzed thousands of samples of insulation, acoustical tile, wallboard, and other construction and decorative materials. Our staff has sampled hundreds of locations and advised many of the institutions involved as to what we think they should do based on our analyses. Finally, the McCrone Research Institute teaches the methods we feel best serve the analyst who wishes to analyze these materials.

Polarized light microscopy

Our method of choice is polarized light microscopy. There is, in fact, no other method capable of doing the job. Even the minimal job of detecting chrysotile, amosite, and crocidolite can only be done microscopically. Certainly the identification of ground glass, mineral wool, glass wool, diatomaceous earth, micas, clays, perlite, lizardite, antigorite, other amphiboles, cellulosic fibers, organic fibers, etc. requires the microscope.

Part of the reason is that almost the only differences between many substances are microscopic shape or optical features: e.g. lizardite, antigorite, and chrysotile; perlite, diatoms, and quartz; cotton, wood fibers, and linen; or silk, human hair, and horse hair. X-ray diffraction may help in some cases but it does not differentiate between fibrous and nonfibrous varieties of the same minerals. Furthermore, it can't identify amorphous substances such as diatoms, perlite, glass or organic fibers; nor is it useful for particle size measurement. All of these analyses are quickly accomplished microscopically.

Another reason is that asbestos minerals were discovered, characterized, and named before x-ray or electron diffraction and other modern instrumental methods were invented. In fact, only chemical analysis and microscopy were then available; the differentiation of the various amphiboles and the three serpentines can only be quickly, confidently, and conclusively done by polarized light microscopy. The determination of chemical composition is not very practical on single particles in a mixture even aside from the fact that many different substances can have identical chemical composition, e.g. riebeckite and crocidolite; grunerite or cummingtonite and amosite; lizardite or antigorite and chrysotile; fibrous and nonfibrous tremolite, fibrous and nonfibrous actinolite, or fibrous and nonfibrous ferroactinolite.

Sampling

The first and a major problem faced by the microscopist is sampling. Insulation, especially in microscopic samples, is far from a uniform material containing a precise percentage of fibers throughout. The wide variation often found in the results from different laboratories or even the same laboratory can in most cases be explained by sample variation. The initial sample should be large enough to be representative and the microscopist must make every effort to take a representative milligram range sample. This is very difficult.

A sampling procedure recommended by the EPA that works very well for friable samples is to press a 35 mm film cannister completely through all layers to yield a core of material. This cannister should be carefully labeled and sent to the laboratory. There each sample should be divided in half (with careful attention to layering if present). One-half (after drying and weighing) should be placed in a small 100 ml beaker with about 50 ml of 10% H_2SO_4. Only the cementicious components will dissolve, leaving the fibrous and other more inert materials in suspension. The latter can be allowed to settle for several minutes, the solution decanted, and water washed by resuspension and decantation several times. Finally, the washed particles should be resuspended by agitation to give a uniformly mixed composition. A few drops can then be removed by eyedropper and several one-drop samples can be placed on microscope slides. These, after drying, can be used for microscopical identification of the particles present. To give more quantitative results the remaining sample, after removal of the eye-dropper samples, should be filtered, dried, and weighed. This will allow calculation of the % cementicious material and, by microscopy on the residue, the percentages of the remaining components.

When the budget allows, the above procedures would yield more accurate and more reproducible quantitative results. In most cases, the budget does not permit the luxury of doing such a complete job.

Instead, the original ounce sample submitted to the laboratory is usually sampled at 10-20 regularly spaced locations with fine forceps, and the accumulated sample is dispersed on a microscope slide in an appropriate immersion liquid. It is the variation in composition of such samples that often leads to corresponding variations in analytical results. One should expect that repeat analyses made in this way may vary by up to 50%. Any single 1-5 mg sample often contains no asbestos in samples containing as much as 50% of that substance in the overall sample.

Identification

No matter how the final microscope preparation is obtained, we now have the problem of identifying each component. This is best done by classical polarized light methods together with dispersion staining, a specialized petrographic procedure. A well-trained mineralogist may not need dispersion staining although most would find it easier to apply to asbestos identification than classical optical crystallography. Any microscopist not trained in mineralogy will find dispersion staining far simpler and, indeed, the

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only method that can be learned in a 3-5 day course or on one's own in any reasonable time.

Are asbestiform minerals present?

Generally, this means—Is chrysotile, amosite (fibrous cummingtonite or grunerite), or crocidolite (fibrous riebeckite) present? The actual analysis proceeds by adding a Cargille refractive index liquid (nD 1.550, high dispersion) to one of the slide samples *after* complete drying. The particles should be dispersed in this liquid by tearing aggregates apart, using two fine needles before covering with a coverslip. In this liquid, chrysotile has distinctive shape—very fine flexible fibrils (often curly) plus straight bundles of such fibrils—and distinctive dispersion staining colors*—usually blue-magenta parallel to the fiber axis and blue perpendicular. Different samples of chrysotile, however, may vary somewhat in λ_o, the wavelength at which particle and liquid have the same refractive index. Parallel to the length λ_o ranges from about 440-560 nm and perpendicular to the length from about 560-660 nm. $\Delta\lambda_o$ for $n_1 - n_u$ is usually close to 100-120 nm.

The presence of chrysotile and other fibrous particles is often obscured by a covering of large numbers of other particles. This is especially troublesome because most such particles are so different in refractive index from the mounting liquid that they appear bright white with the central stop. Trying to see the asbestos is like trying to see while driving at night with an oncoming stream of cars with high-beam headlights. Often one can be pretty sure the particles are covering obscured fibers because of their pattern. I have described this as the milky way effect. Sometimes a stray fibril may poke its way out into the liquid to show dispersion staining colors. It is always a help in such situations to examine the "milky way" with crossed polars since the underlying fibers often then become visible and recognizable as fibers. Crossed polars also help to locate smaller fibers and small percentages of fibers. Every sample should be quickly scanned with crossed polars before the absence of asbestos is reported.

Amosite and crocidolite are both very pale yellow to white by central stop dispersion staining in liquid 1.550, because their refractive indices are so much higher. Crocidolite, of course, also usually shows a blue absorption color. If no anisotropic fibers with refractive indices much higher than 1.550 are present, the analysis is completed.

If higher index anistropic fibers are present, another sample is mounted in Cargille liquid nD =1.680 and examined with dispersion staining. Most amosites used in buildings will show a λ_o of about 460 nm (golden yellow) parallel to the length and about 600 nm (blue-magenta) > 660 nm (pale blue) perpendicular to the length depending on fiber orientation. A perpendicular λ_o of > 660 nm corresponds to the α vibration direction which usually shows oblique extinction of about 15°; a λ_o 600nm perpendicular to the length corresponds to β on the view showing parallel extinction. Higher or lower values for any one of the three vibration directions should mean higher or lower values for all three. As with most silicate minerals, substitutional solid solution—in this case Fe^{2+} with Mg^{2+} —can cause α, β, and γ to vary.

Although the refractive indices of amosite and other amphiboles may vary over a wide range, most of these minerals are not from commercial sources. Nearly all amosite actually used in insulation commercially has the optical properties given above.

If, in liquid n_D=1.680 low birefringent fibers show lower λ_o colors close together in the yellow to golden magenta, crocidolite is strongly indicated. If they show a negative sign of elongation (higher λ parallel to the length) and blue absorption colors with pleochroism (blue parallel, gray-blue perpendicular), crocidolite is present. Further confirmation can be obtained by mounting a third sample in Cargille refractive index liquid n_D=1.70. Crocidolite will show λ_o colors close to 485 nm (golden magenta) parallel to the length and about 455 (golden yellow) perpendicular. Again, some parallel movement of the α, β, γ colors should be expected for crocidolites from different sources.

We should emphasize that dispersion staining is a method for rapid refractive index determination. To be certain the colors observed mean a particular asbestos is present one must be certain the dispersion staining data are consistent with particle size and shape as well as the relationship between the optical properties and the crystallographic axes. With amosite, for example, the crosswise index must be α for oblique extinction views and β for views showing parallel extinction.

Are other asbestiform minerals present?

If, during the above examination, anomalous results were observed, that is, highly fibrous with λ_o colors in other than the prescribed ranges for chrysotile, amosite, and crocidolite, then fibrous tremolite, fibrous actinolite, or anthophyllite may be present. These are rarely found, however, in insulation. When these anomalous results were obtained, you should have characterized the fibers in those liquids as to refractive indices relative to those liquids and extinction angles. If all of the fibers show parallel extinction, they are anthophyllite, if the possibility of organic fibers is first eliminated.

Tremolite will show strong colors in all orientations in 1.605 high dispersion liquid. The oblique extinction view (ca. 15-20°) will show α perpendicular (λ_o ca. 440 nm; yellow) and γ nearly parallel (λ_o ca. 680 nm; pale blue). The parallel extinction view shows γ parallel to the length (λ_o ca. 460 nm; golden yellow) and β perpendicular (λ_o ca. 530 nm; red magenta).

Actinolite has similar morphology and optics except that the indices are all higher than tremolite. Actinolite may also show pleochroism (green to colorless). It is best studied in high dispersion liquid 1.630 in which α and γ on the oblique extinction view show magenta (λ_o ca. 565 nm) and golden yellow (λ_o ca. 445 nm), respectively. On the parallel extinction view γ (lengthwise) shows λ_o ca. 470 nm or golden magenta and β (perpendicular) shows λ_o ca. 495 nm, also golden magenta although with more red.

Tremolite, actinolite, and ferroactinolite are

*The dispersion staining colors mentioned throughout this paper are those obtained by using the central stop rather than the annular.

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parts of a continuous solid solution series in the same manner as the amosite minerals, cummingtonite and grunierite. The name to use for a given amphibole depends directly on the optical properties, as shown in Table 1.

Identification of interfering substances

A few common substances show dispersion staining colors similar to those of chrysotile, and some are elongated as well. These include antigorite and lizardite (polymorphs of chrysotile), quartz, talc, paper fibers, and hairs. All of these show dispersion staining colors in 1.550 and all except talc and paper fibers show colors similar to chrysotile. Antigorite in 1.550 high dispersion liquid shows λ_o for γ, parallel to the length, about 465 nm (golden yellow), λ_o for α and β crosswise are ca. 500 nm (golden magenta) and 520 nm (red-magenta), respectively. Lizardite is platelike (often a lamellar aggregate) with α ($\lambda_o$ca. 700 nm, pale blue) perpendicular to the plate; it generally shows undulose extinction. The β and γ indices lie in the plane of the plate and both show a near 570 nm in the 1.550 high dispersion liquid (red magenta).

Quartz, although usually glassy flakes, shows blue and magenta central stop colors very similar to chrysotile in the 1.550 liquid. The shape is very different, however, and some isotropic views show only blue.

Talc fibers are derived by cleavage from large talc plates; I have never seen rolled talc plated as fibers. The dispersion staining colors are therefore always very pale yellow ($\lambda_o$ca. 360 nm) parallel to the length. They may also show λ_o=360nm for the perpendicular direction but, if on edge, they will show λ_o =645 nm (blue-green). Tapping gently on the coverslip with a needle will usually bounce these needles from the 360-360 nm view to the 360-645 nm view. This is a very useful technique for quartz and other mineral grains as well.

Animal and human hair may also have refractive indices in the same range as chrysotile, and if finely fibrilated, by electric razor for example, can be confusingly similar. Such fibrilated fibers are, however, rare; they also usually show melanin pigment particles and sufficiently different indices to avoid confusion. Paper fibers also show a crosswise index close to 1.55 (but lower) and the lengthwise index is much higher, hence shows a yellow color. Usually also the morphology of paper fibers is distinctive.

Identification of other asbestos substitutes

A number of substances are often used as asbestos substitutes, e.g. wollastonite, glass wool, mineral wool, polyester and paper fibers. Wollastonite, a low birefringent (0.014) mineral, is triclinic and therefore shows oblique extinction in all views. It has refractive indices in the same range as tremolite and anthophyllite but, fortunately, the β index is nearly parallel to the length, hence some views show positive and some negative signs of elongation (anthophyllite and tremolite always show a positive sign). The dispersion staining colors for wollastonite in 1.605 liquid are: parallel, 429 nm (yellow); perpendicular, 410 nm (pale yellow), and 532 nm (red-magenta).

Glass fibers, usually as mineral wool, are often found in insulation. Different samples vary widely in refractive index and may show dispersion staining colors in any of the standard liquids from 1.55 to 1.68. Generally, however, mineral wool has low indices < 1.55, and is often coated with a colored (yellow, orange to red) resin. It is always isotropic or very slightly birefringent due to strain.

Another unusual constituent found in several recent samples is a polyester fiber. The very high birefringence, uniform cylindrical crossection, and considerable length make this easy to distinguish. The indices are about 1.58

TABLE 1

Optical Properties of the Amphiboles

Mineral	Refractive indices α	Refractive indices γ	$\gamma-\alpha$ (aver.)	Extinction angle	Sign elonga'n	Sign optic	$2V_\alpha$
Tremolite	1.603-1.620	1.627-1.642	0.023	19-21	+	-	86-80
Actinolite	1.620-1.667	1.642-1.686	0.020	15-19	+	-	80-70
Ferroactinolite	1.667-1.683	1.686-1.702	0.019	10-15	+	-	70-65
Cummingtonite	1.633-1.664	1.654-1.687	0.022	15-21	+	+	103-92
Grunerite	1.664-1.686	1.687-1.729	0.033	10-15	+	+,-	92-82
Riebeckite	1.654-1.698	1.666-1.712	0.014	10-20	-	-	40-90
Anthophyllite	1.606-1.648	1.626-1.670	0.021	0	+	+,-	66-120

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perpendicular (less than chrysotile) and 1.71 parallel (greater than crocidolite).

Identification of other possible nonfibrous constituents of insulation

Possible additional constituents of insulation etc. include ground glass, perlite (a heat-expanded volcanic lava), diatomaceous earth, vermiculite, and mica. Of these, ground glass, perlite, and diatoms are isotropic and characteristically shaped. Vermiculite, a clay, and the micas are very thin, flat plates often nearly isotropic but with turned-up higher birefringent edges. Vermiculite will have indices less than 1.55 and the micas above 1.55 and often above 1.605. If colored brownish-gray the mica is biotite with higher indices than colorless muscovite, another common mica.

Other materials may well be found as time goes on but the observations made during the analytical procedure described above should uncover such substances, since they will not fit the data given here.

All things considered, a careful microscopist, confident with dispersion staining, should have no difficulty in identifying asbestos and most other substances associated with it.

Acknowledgement

Although fully responsible personally for the ideas expressed here, the author acknowledges with gratitude the constructive appraisal by his colleagues at McCrone Associates: John Delly, Lucy McCrone, Mark Palenik, and Ian Stewart.

Asbestos and the Electron Microscope

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I first learned about this meeting two weeks ago, when I saw the provisional program, and my immediate reaction was that the ACGIH was going to wish on us in electron microscopy the same "raw deal," if I may call it that, as they had wished on us with light microscopy. I noted that there was to be a paper on the use of the scanning electron microscope to characterize asbestos but not one on transmission electron microscopy. I felt that this was similar to using phase contrast microscopy to tackle a mineralogical problem which is more appropriately tackled by a petrographic microscope, so I called Mr. Kelley to ask him what he was doing to us. He very kindly suggested that I come along and present my views in the general discussion at this meeting and so I came prepared with one or two slides to do this. When I arrived, however, Jim Ferguson told me that I was to present my views a little more formally, so here they are.

Criteria for Asbestos Identification

Let me say from the outset that I should not be regarded as an anti-scanning electron microscope man. There are many situations in which the scaning electron microscope is the appropriate tool to use, but I do not believe that the asbestos situation is necessarily one of them. My reasons for saying this are that, like Dr. McCrone, I believe that the main criteria by which one must identify asbestos are crystallographic with chemistry as a secondary consideration, and the scaninng electron microscope is unfortunatly lacking in its ability to give crystallographic characterization from particles of the sizes which are going to be of interest to us.

An additional problem in many scanning electron microscopes is that of resolution. Although manufacturers currently will claim that resolutions are better than 100 A° (10nm) for their microscopes, they do reserve the right to select the specimens on which they demonstrate this resolution, and their samples are generally those with very clearly defined features and very high contrast. This situation does not normally prevail with asbestos fibers down to unit fibril dimensions.

Having brought in the term "unit fibril," let me define it. This term is applied generally to chrysotile asbestos. It is the smallest chrysotile fiber which can exist as a single entity and has an approximate diameter of 300-350 A° (30-35 nm). Fibers of these dimensions are quite common in environmental samples but one does generally see larger fibers in the workplace. However, as control procedures improve, it is to be hoped that the larger fibers will be less prevalent in the workplace, in which case we will have to concentrate on the very fine fibers which are not visible and thus cannot be characterized by the light microscope.

How then do we go about characterizing these fine fibrils with the electron microscope? As I have mentioned, the principal criterion is a crystallographic one. However, one may summarize the three main criteria for identification of a fiber as asbestos under three headings: morphology, crystallography, and chemistry.

Morphology

Clearly, since you are interested in controlling asbestos fibers, the particle in question must have the morphology of a fiber. I will not at the present time go into the various semantics of defining a fiber. But I will say that, at this time, everyone concerned with this problem is utilizing (I almost said accepting) the federal definition of a fiber as a particle with an aspect ratio greater than 3:1. It is quite possible that this may stimulate some discussion. However, though one may argue the semantics of a fiber from a mineralogist's point of view, the final criterion to decide what will be called a fiber for regulatory purposes must only be the biological significance of the particles's aspect ratio. This is a subject on which I am not qualified

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