

May 15, 1984

MSHA Visit - May 3, 1984

Background

Cyprus Industrial Minerals Company, South Plainfield Mill personnel were monitored on May 17, 1983, by the Mine Safety and Health Administration (MSHA). Personal air filter samples from South Plainfield workers were sent to Denver MSHA and analyzed by optical and electron microscopic methods. The filters were reported to contain 71.2% fibrous talc and 5.8% anthophyllite, an asbestiform amphibole.

Explanation of the MSHA Results

Before going any further, it is important to underscore that MSHA's report also confirms that CIMC is in compliance with U. S. Department of Labor - MSHA federal regulations. A detailed explanation of MSHA's findings is as follows:

1. After examination of the air filters with optical microscopy (phase contrast and dispersion staining), a filter sample (with greater than or equal to 2 fibers/cm²) was examined with the analytical electron microscope (AEM*).
2. An Image Analysis* system was then used to examine the sample to locate and analyze via XRF* all particulates, including those that satisfy MSHA's definition of a fiber (any particulate greater than 5 micrometers in length having a 3:1 aspect ratio).
3. In the case of CIMC's sample, the Image Analysis system found a total of 52 fibers in a field of approximately 500 (MSHA estimate) particulates. Three of the 52 fibers (5.8%) were identified as anthophyllite. Thirty-seven of the 52 fibers (71.2%) were identified as fibrous talc. The 52 fibers were classified by the Image Analysis system as follows:

<u>Identification</u>	<u># of Fibers</u>	<u>% of Total Fibers**</u>
Fibrous Talc	37	71.2
Not Asbestos	8	15.3
Unknown Asbestiform	3	5.8
No X-Ray	1	1.9
Sub Total	49	94.2
Anthophyllite Asbestos	3	5.8
Total	52	100.0

* These methods will be discussed in more detail later.

** Note that these results are reported in terms of a percentage of the total number of fibers observed in the sample in contrast to a weight percentage such as we would use to report XRD results.

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The above data was taken by R.L. ("Red") Clark at MSHA headquarters here in Denver. The anthophyllite data was confirmed by "Red" using standard SAED* procedures. Photographs were taken of the SAED patterns. No photographs were taken of the actual anthophyllite fibers. "Red" summarized the data shown above and reported it to MSHA as follows:

<u>Identification</u>	<u>% of Total Fibers</u>
Fibrous Talc	71.2
Not Asbestos	23.0
Anthophyllite Asbestos	5.8
	<u>100.0</u>

MSHA reported "Red's" data to CIMC as follows: (see Attachment 1)

<u>Identification</u>	<u>% Total</u>
Fibrous Talc	71.2
Anthophyllite	5.8
	<u>77.0</u>

According to "Red", the author (unknown) of the MSHA report to CIMC took certain "liberties" with his ("Red's") data. "Red" emphatically pointed out that the fibrous talc should have been placed in the Not Asbestos category ("Red" takes some responsibility for the confusion) and that the MSHA report to CIMC should have read as follows:

<u>Identification</u>	<u>% of Total Fibers</u>
Not Asbestos	94.2
Anthophyllite	5.8
	<u>100.0</u>

Even though we are not out of compliance in this report, I recommend that CIMC request an amended report from MSHA. The reasons for this include:

1. It is not in the best interests of CIMC to have the term "fibrous talc" included in any MSHA report.
2. The 77% total (see MSHA report) is used in both the "Full Shift" and the "Total Additive Value" calculations, when in fact a 5.8% total (for fibers) should have been used. This would dramatically lower the "Full Shift" and "Total Additive Value" results.

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It is worth noting that the category "Unknown Asbestiform" (Image Analyzer results) is included in the "Not Asbestos" category in the final report. It is also worth noting that the final report may not contain nearly as much data as is contained in the SAED confirmed Image Analyzer results. It is worth questioning whether the same number (77%, 5.8%, or whatever) should be used to calculate the "Full Shift" and "Total Additive Value" results on all three workers since the sample L-620 for Jorge Aguilar (see MSHA report) was the only sample analyzed, since it was the only sample analyzed as having greater than or equal to 2.0 fiber/cm³ by optical microscopy.

It can be questioned at this point why CIMC did not detect the anthophyllite in this Italian talc. The total fibers represent approximately 10% of the bulk material. Assuming that the percentage of fibers in an air sample from Italian talc is the same as that in a bulk sample of Italian talc (it is recognized that this assumption may not be valid), this means approximately 0.6% of the bulk Italian talc is anthophyllite. Why did it escape detection by XRD? The answer is that the detection limit for anthophyllite by XRD is only about 2.0%, as compared to 0.2% to 0.5% for tremolite.

Trip Report

On May 3, 1984, C.B. Grimm and I visited MSHA. We were cordially greeted by G.W. Sutton and R.L. ("Red") Clark of the Toxic Materials Branch. We began the meeting by explaining that we routinely perform XRD analyses on all of our bulk sample lots of talc, but, that for the most part, air samples must be analyzed by outside laboratories. It was explained that we would prefer to perform these analyses ourselves. We gave them a copy of the CTFA procedure. We then asked them to explain their analysis scheme so that we could use it as a model to set up our own procedures. At this point, Sutton and Clark opened up to us completely explaining the smallest details of their procedures. This approach was taken by us for two reasons. First, we wanted to test the validity of their South Palinfeld report. Second, we did, in fact, want to get ideas as to how to set up an analytical facility for asbestos identification if CIMC were to become interested in such a facility.

We learned from "Red" Clark that he performs all of the electron microscopy analyses (asbestos, free silica, etc.) for MSHA here in Denver. We also learned that all of their optical microscopy (except for one facility in New England) is also done in Denver by Bill Pitt. Bill Pitt was absent the day of our visit, so we were unable to see MSHA's optical microscopy facilities. However, we were invited to tour these facilities on another day.

During the course of our discussions we also learned that MSHA follows a specific plan of analysis (Attachment 2) for asbestiform minerals. They supplied us with their analysis flow diagram for asbestos. I had the feeling first, that this was a very complete analysis scheme, and second, that this scheme had been tested in court. They emphasized that a "false positive" analysis for asbestos was not possible using this scheme.

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Other topics covered, too detailed to mention here, included:

1. Type of sampling device used in personal sampling.
2. Type of filter used in personal sampling.
3. Certification of purity for the filters used.
4. Supervision of the worker during the sampling period by MSHA inspectors.
5. Loss of particulate matter during shipment to Denver from remote locations.
6. Contamination prevention procedures utilized during sampling.
7. Contamination prevention procedures used by MSHA inspectors.
8. Contamination prevention procedures used by MSHA technicians prior to shipment of samples to Denver.
9. Contamination prevention procedures used in Denver after the samples are received.
10. Optical and electron microscopy procedures used to prevent sample contamination.
11. Field blank filters.
12. Laboratory blank filters.
13. Laboratory glassware cleaning procedures.
14. Where to obtain high purity reagents, including water.
15. Recommended sample coating devices.
16. Recommended sample handling enclosures (laminar flow hoods, dry boxes, etc.).
17. Filter storage procedures.

Next we concentrated on the actual analytical method, instrumentation, and personnel used at MSHA. It is important to note that the MSHA Standard Method for Fiber Identification by Electron Microscopy was written by "Red" Clark. He developed the MSHA method from the existing EPA method. Being familiar with the EPA method, I was impressed with the improvements "Red" made in the areas where the EPA method was obviously weak. For instance, "Red" vastly improved the EPA sample preparation procedure. He also extended the credibility of MSHA's analytical results by going far beyond the EPA's simple TEM/SAED technique. "Red", a Certified Electronic Technician, is an experienced microscopist and has served as an expert witness and friend of the court during the course of various litigations.

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*Definition of Terms

SEM	Scanning Electron Microscope
TEM	Transmission Electron Microscope
EDS	Energy Dispersive Spectrometry - A form of X-ray fluorescence (XRF)
SAED	Selected Area Electron Diffraction - Similar to XRD, but uses electrons instead of X-rays in the diffraction process
STEM	Scanning Transmission Electron Microscope - A combination TEM and SEM with SAED capability
AEM	Analytical Electron Microscope - A STEM with EDS capability

Basically, MSHA uses an analytical electron microscope (AEM)/Image Analysis system for fiber identification. The AEM is a STEM/EDS combination instrument. MSHA highly recommends the use of an AEM for fiber identification work. MSHA recommends that all analyses be performed in a single instrument. This reduces contamination problems and saves a great deal of operator time. It was emphasized that an AEM is designed to be a TEM first, and an SEM second. This is important so that adequate TEM/SAED can be performed. "Red" emphasized that a 100 Kev (minimum) instrument is required to perform TEM/SAED analysis on serpentines and amphiboles which are electron dense minerals. Cyprus does not have an electron microscope with 100 Kev capability.

Most important, MSHA, in order to provide absolutely defensible data in court, as well as save operator time, is using an Image Analysis system with their AEM. An Image Analysis/EDS system under computer control can be programmed to:

1. Scan a specified field of particulates.
2. Size each particle.
3. Find the centroid of each particle.
4. Perform an EDS analysis (XRF chemistry) on each particle.
5. Store all information in memory.
6. Compute the shape of each particle.
7. Compute the surface area of each particle.
8. Compute the elemental ratios for each particle from the XRF analysis.

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9. Identify all particles (mineralogically) on the basis of size, shape, and chemistry.
10. Note the coordinates of each particle so that TEM/SAED confirmation analysis can be performed on each particle if necessary.

It is important to note that the Image Analyzer/EDS system represents an alternate means of obtaining mineralogy on a sample. This mineralogy is on a particle-by-particle basis similar to TEM/SAED, rather than on a bulk basis (XRD). The advantage of an Image Analyzer is that it needs neither operator attention nor operator interpretation, since it is completely under computer control. This is not the case with TEM/SAED analysis. It should be emphasized (see flow diagram) that all Image Analyzer asbestos identifications are verified by MSHA using the TEM/SAED technique, described as "the bottom line" by MSHA. An Image Analyzer may be used to provide complete scans on large fields of particulates. This particulate screening technique provides the analyst with absolutely defensible data, since the AEM/Image Analyzer computer neither misses nor is biased toward any particular type of particulate.

While we were at MSHA, we also discussed bulk sampling, as opposed to air sampling methods. "Red" felt that electron microscopy techniques would be limited in bulk analysis because of the problems associated with getting a representative sample of the bulk material. I suggested the use of an aerosol mixing chamber, from which aerosol samples could be drawn. "Red" seemed more receptive to this suggestion, although he said that there probably would be problems in obtaining reproducible results from the mixing chamber. However, I believe that with careful design, such a mixing chamber would provide reproducible results. The mixing chamber would allow the use of a greater amount of sample (hence reducing the bulk sampling problem) but, most important, it could provide valuable information concerning the composition of the respirable size range of particulates in our products.

In the afternoon, we were able to tour the electron microscopy laboratory at MSHA. Unfortunately, the AEM/Image Analysis system was not operational due to problems with the electronics. However, we were invited to return another day for a complete tour. MSHA uses a Philips STEM with an Ortec EDS system. They use a LeMont Image Analysis system, which, according to "Red", is the best and one of the least expensive units on the market. "Red" again emphasized that the microscope used must have 100 Kev capability, and he also discouraged the use of hybrid systems. For asbestos work, he advised that the AEM is by far the best instrument to use, the TEM the next best, and the SEM the worst.

We also discussed vendors with "Red." He said that the only two vendors worth considering are JEOL and Philips. He reminded us that his equipment was six years old, and that important improvements have been made since his equipment was purchased. For instance, he spends three to eight hours on each air sample depending on the fiber loading. The reason for this is that he is not really able to fully automate his AEM due to the design of the STEM. This means that he must manually scan each sample. Today's

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instruments, however, allow full automation of the Image Analyzer system as previously described. Image Analyzer/EDS analysis can truly be performed automatically independent of the manual TEM/SAED work. Automated TEM/SAED systems are also commercially feasible, according to Philips.

We also discussed other laboratories which might have state-of-the-art capabilities relative to asbestiform mineral identification. Rich Lee at U. S. Steel was recommended, as was Manville and McCrone.

All in all, MSHA personnel were very supportive, cordial, and cooperative. I feel that a good relationship has been established between them and us on a technical level. MSHA offered to run some samples for us (time permitting). They would prefer to run air samples rather than bulk samples, if possible. They also are willing, on the suggestion from C.B. Grimm, to co-author a technical paper with us.

Recommendations

Having worked in the enforcement branch of the EPA myself, I know that when analytical results become an issue in court, they are judged on the basis of analytical procedure used and analytical instrumentation used, as well as on the credentials of the analyst. I believe that it is imperative that CIMC, through the use of equivalent or superior analytical methods, equivalent or superior analytical instrumentation, and equivalent or superior analysts, be able to present defensible data in court. I also believe that it is important that CIMC start a comprehensive asbestos analysis program as soon as possible, in order to establish a "track record" which would be viewed favorably in court.

I believe that we should visit other laboratories competent in the analysis of asbestiform minerals. C.B. Grimm and I have formulated a list of organizations that we feel would provide valuable information in this area, and aid in developing a facility for the analysis of asbestiform minerals at CIMC.

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