

## Procedure for the Analysis of Talc for Asbestos

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### ABSTRACT

The analysis of talc powder for asbestos is most appropriately done with a combination of polarized light microscopy (PLM), transmission electron microscopy (TEM) and in some cases a screening by X-ray diffraction (XRD). Low levels of thin asbestos fibers in talc may only be seen using the TEM analysis. Although never formally adopted by the U.S. Environmental Protection Agency (EPA), the 1993 EPA bulk method (EPA R-93) for asbestos provides the basis for the PLM portion of the method, as it is a good description of the light microscopy techniques available. The consensus method D6281 balloted and published by ASTM International provides the basis for the TEM portion of the method. The method described here has been used to investigate vintage talcum powders and talcum products currently available. Some asbestos has been found in vintage powders but with the exception of one Chinese product, asbestos was not detected in currently available powders using the talc-asbestos method described here.

**Keywords:** talcum, asbestos, polarized light microscopy (PLM), transmission electron microscopy (TEM), X-ray diffraction (XRD), light microscopy, National Institute for Occupational Safety and Health (NIOSH), U.S. Environmental Protection Agency (EPA), ASTM International, International Standards Organization (ISO), phase contrast microscopy (PCM), McCrone Research Institute, New York University Department of Chemistry, tremolite, chrysotile,

anthophyllite, pyrophyllite, asbestiform, fibers, selected area electron diffraction (SAED), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), Asbestos Hazard Emergency Response Act (AHERA) U.S. Pharmacopeia (USP) Talc monograph, infrared spectroscopy (IR), Walter C. McCrone, Lucy McCrone

### INTRODUCTION

In 1968, Cralley et al. (1), from the Occupational Health Program, National Center for Urban and Industrial Health in Cincinnati, Ohio (predecessor of the National Institute of Occupational Safety and Health – NIOSH) reported that they had examined 22 talcum products purchased off-the-shelf (representing body powder, bath powder, and all purpose powder) for fibrous and mineral content. Cralley et al. used phase contrast microscopy (PCM) and found that all of the 22 talcum products had an appreciable fiber content that ranged from 8% to 30% by count of the total talcum particulates. Although the specific fibrous materials were not identified by PCM, XRD analysis by the authors led them to believe that the fibers were predominantly fibrous talc, with the probable presence in minor amounts of other fibrous minerals, such as tremolite, anthophyllite, chrysotile and pyrophyllite. The authors remarked that the electron microscope, with its higher power of resolution, showed a number of submicron diameter particulates not visible by means of PCM, but they did not identify any of the

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fibers by electron microscopy. The authors concluded that cosmetic talcum products should be included as a source of fibers from which may be derived ferruginous bodies observed in the lungs of humans.

A number of independent scientists were involved with analyzing talcum powders in the 1970s. Walter C. McCrone Associates, Inc., in Chicago analyzed talcum powders for various groups, including NIOSH. They used PLM, XRD and TEM in their investigations. They reported finding asbestos fibers in a number of talc samples (2–5).

At the New York University Department of Chemistry one sample of talcum powder sample (referred to as #1615) was tested in 1972 (6). They reported that their initial test by XRD showed “some features in its X-ray pattern that suggested that it might contain some tremolite” and “accordingly, the specimen was subjected to a detailed microscopic examination. Both tremolite and chrysotile fibers were found to be present in the sample. It is estimated the tremolite content is about 2% by weight, and the chrysotile about 0.5%” (6).

In 1974, Rohl and Langer (7) reported on the analysis of consumer talcum powders using analytical methods for identification, characterization and quantitation of asbestos fibers that included PLM, XRD, and TEM with selected area electron diffraction, and electron microprobe techniques. They remarked that the light microscope methods had severe limitations imposed by the ultimate size resolution of the light-optical system. They reported that small particles can go unresolved and most optical properties, e.g., refractive indices, are difficult to measure on small particles. They recommended light microscopy for use only as a preliminary tool for the analysis of consumer talc. Their detection limits for XRD analysis of consumer talcum products were as low as 0.1% by weight for tremolite, 0.25% for chrysotile but only 2.0% for anthophyllite. They concluded that the unique characterization of amphibole fibers (anthophyllite and tremolite versus fibrous talc) required TEM structural analysis (selected area electron diffraction — SAED) and micro-chemical characterization. Rohl and Langer recommended both XRD and TEM with SAED for analysis of consumer talc for their asbestos fiber content.

In another article published in 1974, Rohl (8) remarked, “Talc deposits include asbestos minerals such as chrysotile and amphiboles that may be carried over into consumer products. Optical [light] microscopy and X-ray diffraction analyses may not reveal their presence.” Rohl reported that even at the detection limit for chrysotile by XRD (0.25%), there would be about a billion ( $10^9$ ) fibers per mg of talc. He concluded that a

sample of cosmetic talcum powder, which had been found negative for chrysotile when checked only by XRD, might contain billions of fibers that could be released during dusting with a half-gram dose.

In 1976, Rohl and Langer (9) reported on their testing of 20 consumer products labeled as “talc” or “talcum powder,” including body powders, baby powders, facial talcums and one pharmaceutical talc. Of those 20 products, 10 were found to contain detectable amounts of tremolite and anthophyllite, principally asbestiform. The samples were analyzed by XRD, PLM, scanning electron microscopy (SEM) and TEM equipped with energy dispersive X-ray spectroscopy (EDS) and SAED capabilities. The authors noted that while some asbestos was resolvable by light microscopy, most samples were too fine-grained, with particle dimensions too small for light microscopy. By comparing the results of PLM and quantitative XRD with those from TEM analysis, they noted that large numbers of fibers could go undetected when using only the less sensitive techniques of PLM and XRD.

In 1990, Kremer and Millette (10) published a TEM procedure for the analysis of powdered talc for asbestos that had been in use in the McCrone laboratory in Atlanta since 1985. The method began by preparing an aqueous suspension of talc treated with the wetting agent, methylcellulose. Particles were transferred to a TEM grid via the “drop mount” method, where a drop of the talc-water suspension is placed on a carbon-coated formvar grid. Asbestos fibers were identified based on morphology as seen in the TEM, crystal structure as determined by SAED and elemental composition using an EDS system. Elongated particles with parallel sides and an aspect ratio of greater or equal to 3:1 were counted. Fibrous particles that needed to be distinguished from asbestos were listed as enrolled talc, ribbon talc, antigorite, talc fragments, silica and iron oxide fibers, and organic additives such as perfumes that may crystallize as fibers or needle-shaped crystals. The published method had a theoretical detection limit of 0.00005% ( $10^{-5}$ ) weight percent based on a fiber 3  $\mu\text{m}$  long by 0.2  $\mu\text{m}$  wide by 0.06  $\mu\text{m}$  thick as an asbestos fiber thought to be representative at the time of the smaller asbestos fibers found in some talc.

For lack of better statistical information at the time in 1990, the publication stated a rule of thumb that the detection of five or more asbestiform minerals of one variety in an analysis constituted a quantifiable level of detection. Subsequent method development in the area of TEM analysis for asbestos has shown that the detection of less than five fibers in a sample can provide a statistically valid result.



Although SEM is used to monitor asbestos in several European countries, it is not accepted in the U.S. for any analysis method of asbestos in talc. Davis, 1991 (11) tried to use the SEM to differentiate asbestos fibers from non-asbestos fibers. They reported: "This proved impracticable to do subjectively with any degree of reproducibility and had to be abandoned..." (11).

## EXISTING METHODS FOR TALCUM POWDERS

The two historical methods for the analysis of talcum powders for asbestos are known as the CTFA-J4-1 (12) and USP-Talc (13). They are not considered up-to-date and are in need of revision.

The CTFA-J4-1 stands for the "Cosmetic, Toiletry and Fragrance Association method for Asbestiform Amphibole Minerals in Cosmetic Talc" first published in 1971. Part 1 is an XRD method. If an amphibole mineral is detected at a level greater than 0.5%, then the sample must be analyzed by Part 2 using (light) microscopy coupled with dispersion staining. To be counted, the fibers must have at least a 5:1 aspect ratio, be less than 3  $\mu\text{m}$  in diameter and less than 30  $\mu\text{m}$  in length. The document states that TEM with SAED offers greater sensitivity, but that it was not included because it was not thought to be suitable for normal quality-control application (based on time of analysis, expertise required and expense of equipment).

USP-Talc refers to the existing U.S. Pharmacopeia (USP) talc monograph published before 1983, which includes a test for "Absence of Asbestos." The asbestos test (which is currently pending revision) began with either an infrared spectroscopy (IR) test (USP-191) or an XRD test (USP-941). If the result of the IR or XRD test is negative, then no further analysis is required. If the IR or XRD test option gives a positive result, then an optical microscopy test (USP-776) must be done to confirm asbestos. The optical microscopy procedure does not require the use of polarized light.

## SUMMARY OF A METHOD FOR THE ANALYSIS OF TALCUM POWDER FOR ASBESTOS

The method for the investigation for asbestos in talc described here is based on the early work of Walter and Lucy McCrone, the work of Kremer and Millette published in 1990 and the subsequent asbestos analytical procedures for PLM developed for the EPA, and the TEM methods standardized and published by the ASTM International (formerly American Society for Testing and Materials).

In the asbestos-talc method presented here, the

sample is initially examined under a stereomicroscope at magnifications ranging from 7X to 40X. Portions of the particulate found in the sample are mounted in appropriate Cargille refractive index liquids for analysis by PLM using a polarized light microscope with a magnification range from 100X to 1,000X. The PLM analysis follows the procedures for bulk analysis of building materials described in the EPA 1993 bulk method (14). General SEM imaging of the sample using a scanning electron microscope can be done as an option to judge the extent of fibers in the sample. As a screening, XRD analysis is performed by scanning over a range of 3° to 45° 2 $\theta$  using 40kV, 25mA Cu K $\alpha$  radiation. Mineral phases are identified with the aid of computer-assisted programs accessing a CD-ROM powder diffraction database. Mineral concentrations are based on relative peak heights and reference intensity ratios.

A transmission electron microscope equipped with EDS X-ray analysis system and capable of SAED is used to analyze the talc and asbestos fibers in the sample including tilting of talc/anthophyllite fibers. The TEM asbestos fiber counting criteria of fibers greater than 0.5 micrometer in length with at least a 5:1 aspect ratio as described in the Asbestos Hazard Emergency Response Act (AHERA) (15) and ASTM methods: D6281 (16), D5755 (17), D5756 (18) and D6480 (19) as well as in ISO 10312 (20) and 13794 (21) are used. The d-spacing/interfacial angle tables of Shu-Chun Su (22) are used when the option to index zone-axis patterns of amphibole minerals obtained by SAED in the TEM is chosen. The results of the TEM analysis are recorded using the procedures described in ASTM D6281.

## TEM NOTES

The procedures for counting asbestos fibers with TEM described in ASTM D6281 and ISO 10312 (which are essentially the same) are the most fully developed of any of the TEM methods. The major difference between ASTM D6281 and ISO 10312 is that D6281 contains inter-laboratory precision data. Both methods have been vetted, debated and approved through the ASTM International or International Standards Organization procedures involving multiple ballots by experienced and knowledgeable scientists. Although ASTM D6281 and ISO 10312 were published as methods for asbestos in air, the basic counting procedures are the same for any sample once that sample material has been placed on a TEM grid. Since they are the most developed methodologies and have been accepted internationally, D6281 was chosen as the basis for the TEM part of this talc analysis method.

**TABLE 1** Examples of the Minimum Number of Grid Openings Required to Achieve a Particular Analytical Sensitivity for a Collection Filter Area of 385 mm<sup>2</sup> and TEM Grid Openings of 85 μm (0.0072 mm<sup>2</sup>)

Analytical Sensitivity	Volume of Air Sampled, L						
	500	1000	1200	2000	3000	4000	5000
0.1	1066	533	444	267	178	134	107
0.2	533	267	223	134	89	67	54
0.3	356	178	148	89	60	45	36
0.4	267	134	112	67	45	34	27
0.5	214	107	89	54	36	27	22
0.7	153	77	64	39	26	20	16
1.0	107	54	45	27	18	14	11
2.0	54	27	23	14	9	7	6
3.0	36	18	15	9	6	5	4
4.0	27	14	14	7	5	4	4
5.0	22	11	13	6	4	4	4
7.0	16	8	7	4	4	4	4
10.0	11	6	5	4	4	4	4

**Figure 1.** Table 1, reprinted from ASTM D6281-09 Standard Test Method (16), contains examples of the minimum number of grid openings required for certain analysis situations, ranging from four to 1,066 openings.

In both ISO 10312 and D6281 methods, one sentence has been interpreted by one scientist as indicating that the method is presumptive of asbestos present. The claim is that the fibers determined during the analysis using the method cannot be considered to be asbestos unless bulk analysis has been performed previously and asbestos identified in a product. This is not the case. The sentence contains two independent phrases that describe the applicability of the method. The first phrase describing the application of the method is for “the measurement of airborne asbestos in a wide range of ambient air situations.” This expression is general, and there is absolutely no suggestion contained within it that asbestos is presumed to be present or presumed to be absent. The second phrase in the sentence is “for detailed evaluation of any atmosphere in which asbestos structures are likely to be present.” This second phrase was intended to show an example of one of the many types of situations where the method might be used. D6281 is applicable for a detailed evaluation of any atmosphere for asbestos.

#### Number of Grid Openings to Be Counted

It is clear from examination of the equation used to calculate the concentration of asbestos fibers in a sample that the level of analytical sensitivity improves with the number of grid openings analyzed. ASTM D6281 does not specify a maximum number of grid openings that should be examined. Table 1 (see Figure 1) of D6281 contains examples of the minimum

number of grid openings required for certain analysis situations that range from four to 1,066 openings. While the “rule of thumb” guideline of using 10 full-grid openings represents a judicious compromise between a reasonable experimental effort and a fairly low value of the detection limit, using two or more TEM grids (to analyze more grid openings) reduces the detection limit further and improves the precision of the estimates (23).

#### Differentiation of Asbestos Fibers from Non-asbestos Fibers

In 1990, Wylie (24) published some suggested characteristics of a population of particles with the asbestiform mineral habit. These included a mean aspect ratio of 20:1 or greater for fibers longer than 5 μm. Asbestos was characterized by very thin fibrils, usually less than 0.5 μm in width, and two or more of the following:

- Parallel fibers occurring in bundles
- Fiber bundles displaying splayed ends
- Fibers in the form of thin needles
- Matted masses of individual fibers
- Fibers showing curvature

Subsequently, the draft EPA R-93 (14) repeated most of the characteristics in a glossary providing a definition of a population of asbestos fibers as observed with light microscopy in a bulk sample. The EPA draft deleted the characteristic of fibers in the form of thin needles as being indicative of asbestiform.

TABLE 2-2. OPTICAL PROPERTIES OF ASBESTOS FIBERS

Mineral	Morphology and Color <sup>1</sup>	Refractive Indices <sup>2</sup>		Birefringence <sup>d</sup>	Extinction	Sign of Elongation
		$\alpha$	$\gamma^5$			
Chrysotile (asbestiform serpentine)	Wavy fibers. Fiber bundles have splayed ends and "kinks". Aspect ratio typically >10:1. Colorless <sup>3</sup>	1.493-1.546 1.532-1.549 1.529-1.559 1.544-1.553	1.517-1.557 1.545-1.556 1.537-1.567 1.552-1.561	0.004-0.017	Parallel	+ (length slow)
Amosite (asbestiform grunerite)	Straight to curved, rigid fibers. Aspect ratio typically >10:1. Colorless to brown, nonpleochroic or weakly so. <sup>4</sup> Opaque inclusions may be present	1.657-1.663 1.663-1.686 1.663-1.686 1.676-1.683	1.699-1.717 1.696-1.729 1.696-1.729 1.697-1.704	0.021-0.054	Usually parallel	+ (length slow)
Crocidolite (asbestiform riebeckite)	Straight to curved, rigid fibers. Aspect ratio typically > 10:1. Thick fibers and bundles common, blue to dark-blue in color. Pleochroic.	1.693 1.654-1.701 1.680-1.698	1.697 1.668-1.717 1.685-1.706	0.003-0.022	Usually parallel	- (length fast)
Anthophyllite- asbestos	Straight to curved fibers and bundles. Aspect ratio typically > 10:1. Anthophyllite cleavage fragments may be present with aspect ratios <10:1. Colorless to light brown.	1.598-1.652 1.596-1.694 1.598-1.674 1.6148 <sup>7</sup>	1.623-1.676 1.615-1.722 1.615-1.597 1.6362 <sup>7</sup>	0.013-0.028	Parallel	+ (length slow)
Tremolite- Actinolite- asbestos	Straight to curved fibers and bundles. Aspect ratio typically > 10:1. Cleavage fragments may be present with aspect ratios <10:1. Colorless to pale green	Tremolite 1.600-1.628 1.625-1.655 1.604-1.612 1.627-1.635 1.599-1.612 1.625-1.637 1.6063 <sup>7</sup> 1.6343 <sup>7</sup>		0.017-0.028	Parallel and oblique (up to 21°); Composite fibers show parallel extinction.	+ (length slow)
Actinolite 1.600-1.628 1.625-1.655 1.612-1.668 1.635-1.688 1.613-1.628 1.638-1.655 1.6126 <sup>7</sup> 1.6393 <sup>7</sup>						

<sup>1</sup>Colors cited are seen by observation with plane polarized light.

<sup>5</sup>] to fiber length, except  $\perp$  to fiber length for crocidolite only.

<sup>2</sup>From references 2, 11, 12, and 18, respectively. Refractive indices for  $n_x$  at 589.3nm.

<sup>d</sup>Maximum and minimum values from references 2, 11, 12, and 18 given.

<sup>3</sup>Fibers subjected to heating may be brownish. (references 13, 14, and 15)

<sup>7</sup> $\pm$  0.0007

<sup>4</sup>Fibers subjected to heating may be dark brown and pleochroic. (references 13, 14, and 15)

Figure 2. Table 2.2, reprinted from EPA Test Method R-93 (14), suggests using an aspect ratio of 10:1 in distinguishing between asbestos and non-asbestos fibers when considering optical properties.



Although these mineralogical population characteristics serve as a useful index in screening products and materials that contain fibers that might cause asbestosis, the criteria are not very useful when dealing with individual fibers. The characteristics of parallel fibers occurring in bundles, fiber bundles displaying splayed ends, matted masses of individual fibers and fibers showing curvature are not related to the disease causing potential of asbestos fibers. Microscope analysis of individual fibers found on air sample filters produced from standard reference amosite (grunerite) asbestos fibers found very few parallel fibers occurring in bundles, fiber bundles displaying splayed ends, matted masses of individual fibers or fibers showing curvature. Trying to use two or more of those mineralogical characteristics would result in misclassifying up to 80% of the asbestos fibers.

The aspect ratio (AR) of a fiber, as determined by dividing its length by its width, has been used in discriminating between asbestos and non-asbestos fibers. Table 2.2 (see Figure 2) in the draft EPA R-93 method suggests using an aspect ratio of 10:1 in distinguishing between asbestos and non-asbestos fibers when considering optical properties. However, while research has shown that a population of cleavage fragment particles has a smaller average AR than a population of commercial asbestos fibers, the AR distributions of the two populations overlap, and on an individual basis, some fibers can be classified either way. Research by Wylie (25) reported in 1985 showed that 50% of the fibers in a known amosite (grunerite) asbestos sample would not be counted if a 20:1 aspect ratio were used as a criterion. Comparison of the aspect ratio plots in the 1977 Bureau of Mines Circular (26) shows that a criterion of about 5:1 aspect ratio appears to be the best aspect ratio discriminator for asbestos versus non-asbestos fibers. The 5:1 aspect ratio is used in AHERA; ASTM methods D6281, D5755, D5756 and D6480; and ISO 10312 and 13794.

The width of the fiber was found in inter-laboratory testing by Harper (27) to be the best discriminator for asbestos fibers, and that using a criterion of width that is less than or equal to one micrometer provides the least number of false negatives when dealing with asbestos and non-asbestos fibers. At the time of this writing, this information has not been incorporated into any standard method.

### Elemental Analysis

The X-ray elemental spectrum collected from individual fibers is compared to data collected from known

asbestos minerals. It is noted that the elemental compositions of talc and anthophyllite can be very similar. Although NIST-standard anthophyllite contains a small amount of iron, end-member anthophyllite, which contains very low or non-detectable amounts of iron, is reported in a standard mineralogical text (28) and documented in at least one talc deposit (29).

### Zone Axis Indexing

Using ASTM D6281 allows for the option of indexing a portion of the SAED patterns and then comparing the values determined to calculated zone axis values. This is not possible with all fibers. Method D6281 (or any other TEM asbestos method) does not dictate the tolerance required for a positive match between observed and calculated values. Because of the known variability among the same mineral types found in different sources, it has been suggested that a tolerance of 10% might be used. Testing in the 1970s at the EPA research laboratory of chrysotile asbestos fibers from many sources showed that 5% tolerance was necessary when matching chrysotile asbestos SAED "d" values for the (002), (110) and inter-row spacing to account for the variability between different chrysotile fiber sources. This 5% criterion has been the standard taught during TEM asbestos analysis classes since 1987. This value is in line with early XRD data such as the 3.43% difference between the observed talc (002) measurement of 9.278 angstroms when compared to the calculated value of 8.96 angstroms by Gruner (30) and the 4.24% difference in the measured value for talc (002) by Gruner (30) of 8.960 angstroms and that measured by Stemple (31) of 9.34 angstroms. Table 4 (see Figure 3) in the draft Yamate document (23) shows a 16% difference between the  $d_1$  of the SAED Internal Standard File Data and the  $d_1$  from the X-ray Powder Diffraction File Data for the [101] zone axis for crocidolite (XRD File Index: 19-1061).

### Talc Pseudo-Hexagonal Pattern

Table 4 in the draft Yamate document (23) lists  $[-1\ 4\ 2]$  as a reference zone axis for anthophyllite. With  $d_1$  and  $d_2$  both at 4.56 angstroms and an angle of  $60^\circ$ , this pattern is very close to the zone axis measured on a typical pseudo-hexagonal pattern obtained from a talc plate. Therefore, a fiber cannot be considered to be anthophyllite on the basis of a zone axis index match of the  $[-1\ 4\ 2]$  alone. Fortunately, a talc fiber can be differentiated from an anthophyllite fiber because the talc pattern remains evident as the talc particle is tilted, but the pattern changes when an anthophyllite fiber is tilted.

TABLE 4. COMPARISON OF d-SPACINGS FROM SAED FILE  
AND POWDER DIFFRACTION FILE (EXAMPLE)

Amphibole type	Zone axis	Internal Standard File Data				Powder Diffraction File Data (1975)		
		d <sub>1</sub> (Å)	d <sub>2</sub> (Å)	θ (deg)	Interrow spacing, R (Å)	d <sub>1</sub> (Å)	d <sub>2</sub> (Å)	File index no.
Amosite	[100]	5.3	9.14	90.0	5.3	5.22	9.20	17-725
	[30 $\bar{1}$ ]	1.79	9.26	84.0	—	1.76	9.20	17-725
	[101]	4.88	9.23	74.0	5.17	4.84	9.20	17-725
	[ $\bar{1}$ 01]	4.14	9.11	78.0	4.21	4.10	9.20	17-725
	[ $\bar{3}$ 10]	5.22	5.13	95.0	—	5.22	5.12	17-725
Crocidolite	[100]	5.22	8.97	90.0	5.22	5.20	9.02	19-1061
	[101]	4.94	9.05	75.0	5.19	5.89	9.02	19-1061
	[ $\bar{1}$ 10]	4.79	8.19	79.0	5.23	4.89	8.40	19-1061
	[30 $\bar{1}$ ]	1.75	8.97	83.5	—	1.76	9.02	19-1061
	[ $\bar{3}$ 10]	5.12	5.12	96.0	—	—	—	19-1061
Tremolite	[100]	5.04	9.03	90.0	—	5.07	8.98	13-437
	[101]	4.83	9.03	75.0	—	4.87	8.98	13-437
	[20 $\bar{1}$ ]	2.59	8.97	80.5	—	2.59	8.98	13-437
	[30 $\bar{1}$ ]	1.72	8.98	83.5	—	1.69	8.98	13-437
Anthophyllite	[100]	—	—	90.0	5.24	5.28	8.90	9-455
	[142]	4.56	4.56	60.0	—	4.50	4.50	9-455

Figure 3. Table 4, reprinted from the EPA Draft Report Contract #68-02-3266 by Yamate et al. (23), shows a 16% difference between the d<sub>1</sub> of the SAED Internal Standard File Data and the d<sub>1</sub> from the X-ray Powder Diffraction File Data for the [101] zone axis for crocidolite (XRD File Index: 19-1061).

### Fibers with Kinks

When using the zone-axis indexing option, a few rare fibers with kinks in them that would normally be dismissed as talc ribbons by morphology may show a zone axis that match anthophyllite. Because the crystal structure matches anthophyllite and the fiber has substantially parallel sides for the majority of the fiber length, the fiber is counted as anthophyllite in this method.

### RESULTS FROM USING THIS TALC METHOD

The method described here has been used to analyze both vintage talcum powders and some currently available. The analyses of samples of one brand of vintage talcum powder by this method showed the presence of asbestos fibers was described in Gordon (32). Analyses of one modern talcum powder product and a set of current cosmetic talc source samples from one

supplier using the same method did not detect any asbestos present. These later findings with the modern talcum powder are consistent with the results of a recent FDA sponsored study. During 2011–2012, the FDA contracted with AMA Analytical Services, Inc. to examine 28 cosmetic-grade talc samples from four suppliers and examine 34 off-the-shelf cosmetics for asbestos (33). Samples were received from suppliers who voluntarily sent samples; off-the-shelf samples were purchased directly from various stores based on a list of products determined by the FDA. AMA used a modified version of the New York State ELAP method 198.6/198.4 (non-friable bulk samples by PLM and TEM [34, 35]). AMA did not detect asbestos in any of the 28 talcs provided in 2011 from the suppliers or in 34 the talc-containing cosmetic products that were purchased in stores during the same period. In fact, AMA reported that all the talc materials tested contained only talc plates and no fibrous particles. Therefore, no specific testing procedures such as dispersion staining for PLM or SAED/EDS for TEM were needed. The limit of detection for the PLM portion of the AMA testing was based on one point out of 400 points multiplied by any loss during gravimetric reduction. Because there wasn't much loss for talcum powder samples, the PLM detection was reported as "around 0.21% to 0.23%." The AMA reported a limit of detection for TEM of "about 0.0000020% to 0.0000030%" based on the equation:  $(EFA \times DF \times M)/(AA \times IM)$ , where M was the mass of the smallest countable chrysotile asbestos fiber ( $1.60 \times 10^{-15}$  grams), EFA was the effective filter area, DF was the dilution factor, AA was the area analyzed and IM was the initial sample mass. The result of the equation was multiplied by 100, to convert it to a percentage.

## DISCUSSION

The methodology presented here updates the 1990 publication by Kremer and Millette and provides some information that may be helpful in updating the USP talc method. The analysis of talc powder for asbestos is most appropriately done with a combination of PLM, TEM and in some cases a screening by XRD. Low levels of asbestos fibers in talc, especially those too thin to be seen by light microscopy, may only be seen using the TEM analysis.

In 2014, Block et al. (36) discussed the modernization of the asbestos testing required in the USP talc monograph. The U.S. Food and Drug Administration (FDA) through the FDA Monograph Modernization Task Group asked the USP and National Formulary (USP-NF) to modernize the USP talc monograph in

November 2010. This FDA request included updating the monograph to assure that talc used for cosmetic and pharmaceutical products is not sourced from mines that are known to contain asbestos, and asked that USP consider revising the current tests for asbestos to ensure adequate specificity. The expert panel that was charged with modernizing the USP talc monograph by the USP-NF recommended that the revision of the test for "Absence of Asbestos" omit the IR test and include a revised XRD procedure, in combination with one or more microscopic evaluations (PLM, TEM or SEM). The expert panel determined that the IR and XRD methods, as currently written, could lead to false-negative results, which could allow talc samples with asbestos contamination to pass. The panel also found that even with the additional light optical microscopy test (which currently does not include PLM), the analyst could not rule out the presence of hazardous fibers in the talc sample. In addition, the lack of identification procedures in the light optical microscopy section could lead to false-positive results. The 2014 report concluded that there was a need to modernize the current USP monograph because both the IR and the XRD methods have relatively high detection limits for asbestos, and there is no known "safe" level of asbestos exposure.

## DISCLOSURE

The author has worked for both plaintiffs and defendants in lawsuits involving asbestos contamination. No client funds were received for the writing of this research article.

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