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The Tenth-Meter

In a recent (1) piece about the test diatom "Nitzschia firthii Fuge" I quoted Spitta's comments (2) which included the tenth-meter in reference to the wavelength of light (4700 tenth-meters) recommended to best resolve the lines (striae) on the diatom valve face.

I feel an explanation of this term as used by Spitta is owed to readers of this column. In a footnote on p. 267 of his text I find "It should be mentioned here perhaps, although hardly in logical sequence, that even the μ or micron is not small enough a unit for the physicist when dealing with the measurement of the wavelengths of light. In this case the German savants employ what is called the double mu (written μμ), which is the thousandth part of the micron; but the English scientist adopts a smaller unit still, called the tenth-meter, which is the ten thousandth part of the micron, the raison d'étre of the term being that 10^{10} (10 at the tenth power) go to a meter".

From this then we realize that the tenth-meter is equivalent to the Angstrom Unit, named after A.J. Angström (1814-74), a Swedish physicist, and defined as one-ten-thousandth of a micron. However in this age of enlightenment, after great strides in clarifying scientific terminology, we have dropped the micron in favor of the micrometer, and the millimicron in favor of the nanometer, and so since the Angstrom is one ten thousandth of a micron (oops...micrometer), Spitta was recommending light of 470 nanometers. Got that?

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A Standard TEM Procedure for Identification and Quantitation of Asbestiform Minerals in Talc

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Keywords: Asbestiform, asbestos, MSHA, OSHA, talc, TEM, amphiboles, mineral fibers, microscopy, electron microscopy.

ABSTRACT

A standard method for the preparation of powdered talc for transmission electron microscope (TEM) analysis has been developed to identify and quantify small quantities of asbestos and related minerals. The method utilizes an aqueous suspension treated with methyl cellulose. Particles are mounted by transferring a drop of the talc-water suspension to a TEM grid bearing a formvar film which has been coated with evaporated carbon. Once dry, two grids per sample are examined in the TEM at magnifications of 5,000X and 20,000X for elongate and

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fibrous particles. Morphology, crystal structure and elemental composition are used to identify and characterize particles of interest. Calculations are included to determine a detection limit and/or determine concentration in weight percent.

INTRODUCTION

The regulatory definition of asbestos was established in 1972 when the National Institute of Occupational Safety and Health (NIOSH) published its definitions and analysis methods for asbestos (1). Under their method, asbestos was defined as any fiber of chrysotile, crocidolite, amosite, anthophyllite, tremolite or actinolite. What has come to be known as a "federal fiber" was defined as a particle with a length to width (aspect ratio) of at least 3:1 and a length greater than 5 micrometers (μm) (1). Although NIOSH has set forth phase contrast microscopy (PCM) as the analytical tool for regulating airborne asbestos, it has acknowledged the need for and has used analytical electron microscopy (AEM) to identify asbestos in air samples and bulk talc samples (2,3).

Both the Occupational Safety and Health Administration (OSHA) and the Mine Safety and Health Administration (MSHA) refer to NIOSH methods. As regulatory agencies, OSHA and MSHA are responsible for monitoring the safety of workers in the work and mine environments, respectively, which includes setting limits on the levels of hazardous materials to which a worker may be exposed. The primary route of asbestos exposure is inhalation, therefore air concentration limits were established (4). A primary source of airborne dust is fine particle bulk materials. Consequently, manufacturers have been concerned with the potential presence of these minerals in their products in any measurable quantity. In February, 1990 OSHA promulgated a Hazard Communication Standard (HCS) (5). The HCS requires producers to label as a carcinogen, bulk materials and bulk minerals and products which contain 0.1% or more of asbestos and, pending supplemental rulemaking to be completed November, 1990, non-asbestiform anthophyllite, tremolite and actinolite. To meet this standard, a more sensitive method to detect asbestos is needed. Such low levels are not quantifiable by polarized light microscopy and the morphology of fibers is not discernible by x-ray diffractometry. Transmission

electron microscopy is capable of detecting these low levels and has been recognized by both government and industry for several years (3,6).

Given the NIOSH definition of asbestos and the low levels to which it must be detected, the need arose for quantitative TEM analysis of powdered talc. To meet this need, McCrone Environmental Services developed the following method, which has been in use since 1985.

Standard Operating Procedure

Analysis of Powdered Talc for Asbestiform Minerals by Transmission Electron Microscopy

1. Scope and Purpose

This method is applicable to the identification and quantitation of small (typically 1-20 micrometer) elongate minerals in powdered talc, and was designed for chrysotile and the elongate forms of tremolite, actinolite and anthophyllite. Samples may be previously screened by light microscopy or x-ray diffraction techniques.

2. Principle of Method

The combined techniques of transmission electron microscopy (TEM), selected area electron diffraction (SAED) and energy dispersive x-ray spectrometry (EDS) permit the detection of asbestiform minerals based on morphological characteristics, followed by a definitive mineralogical identification of each fiber. These techniques are currently the best analytical tools for the determination of fine asbestos minerals in a talc matrix.

3. Interferences

Interferences caused by other fibrous particles must be distinguished from positively identifiable asbestos. Large particle or particle aggregates may obscure fibers. Positively identified non-asbestos fibers include enrolled talc, ribbon talc (Figures 1A and B), antigorite (Figures 2A,B and C), talc fragments (Figures 3A and B), silica and iron oxide fibers. Organic additives such as perfumes may crystallize as fibers or needle-shaped crystals in

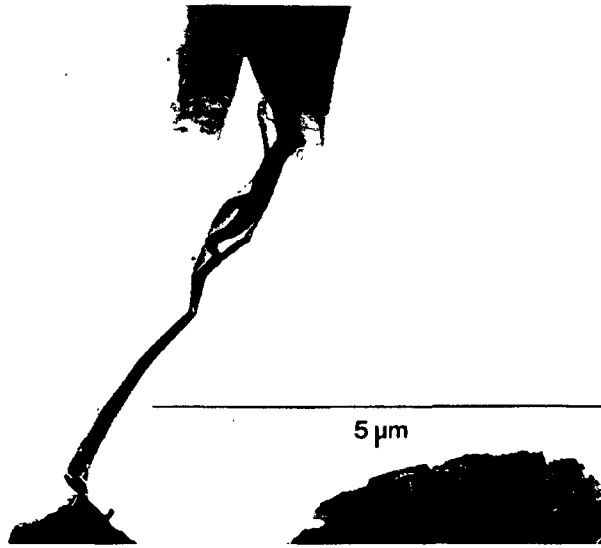

Figure 1a. Talc platelets and attached ribbon.

Figure 1b. SAED pattern of a talc ribbon with its (001) face oriented approximately parallel with the electron beam.

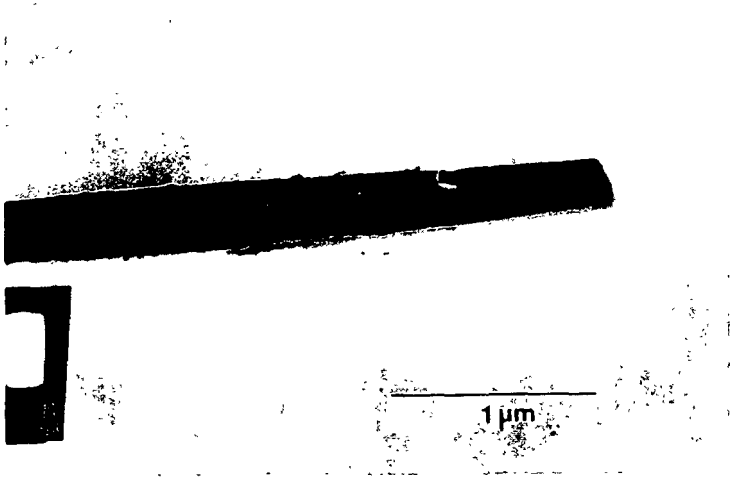

Figure 2a. Antigorite: rectangular lath with its superimposed electron diffraction pattern.

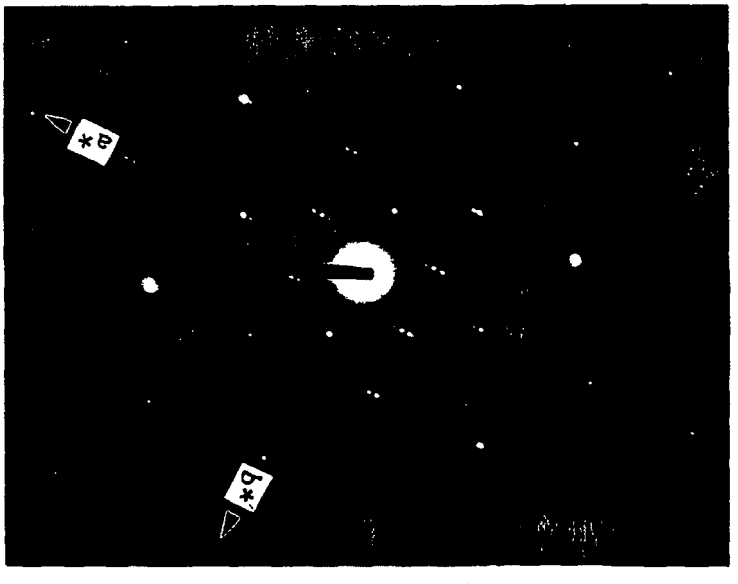

Figure 2b. SAED pattern with a diagnostic 35 to 40 A spacing in the a-crystallographic direction.

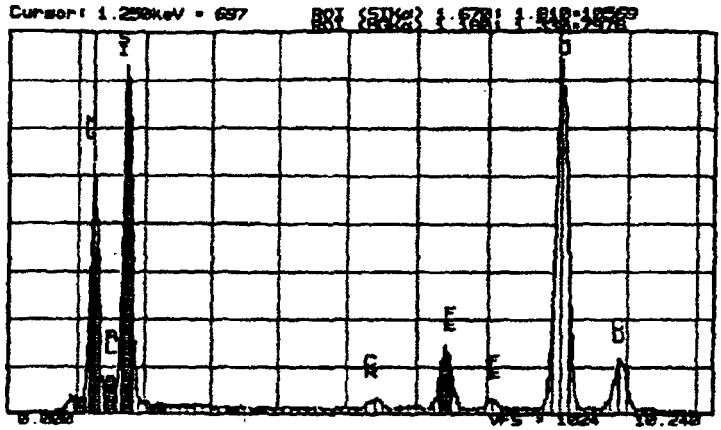

Figure 2c. The elemental composition, determined by EDXRA, is characteristic of a serpentine mineral.

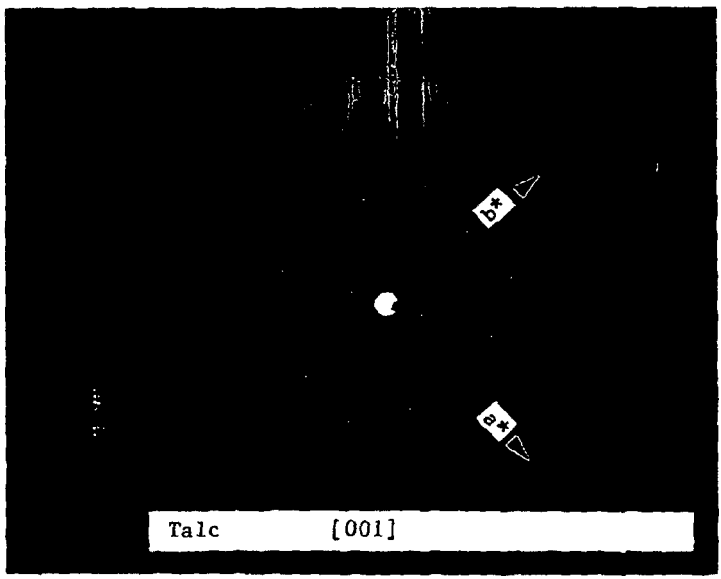

Figure 3a. SAED pattern of talc, showing the typical [001] zone axis of a phyllosilicate mineral, with the (001) face oriented perpendicular to the electron beam.

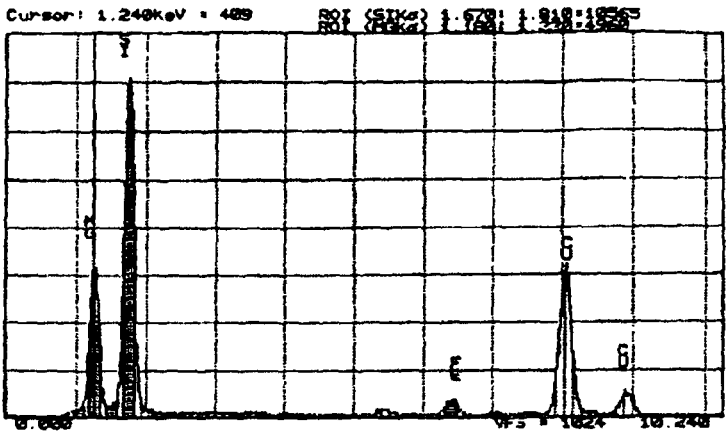

Figure 3b. EDXRA spectrum, characteristic of talc.

finished cosmetic products. In the absence of positive identification, all other fibers must be classified as unidentified.

4. Instrumental Conditions

The talc specimen grids are examined in the TEM at an accelerating voltage of 120 kV and at magnifications of 5,000X and 20,000X.

5. Sensitivity

This method can detect a single fiber as small as 1 micrometer (µm) long by 0.075 µm wide in the entire TEM field, which results in a theoretical detection limit of 10^{-5} weight percent. Such fibers usually can be identified readily by SAED and EDS. The mass of a fiber with the above dimensions is about 1.1×10^{-14} g for chrysotile and about 1.9×10^{-14} g for amphiboles.

6. Limit of Quantifiable Detection

The detection of five or more asbestiform minerals of one variety in an analysis constitutes a quantifiable level of detection. When no asbestiform minerals are detected a representative fiber size is used to calculate a detection limit. A representative fiber size is 3 µm long by 0.2 µm wide by 0.06 µm thick, which is

considerably larger than the smallest fiber than can be detected (see Section 5, Sensitivity), but it is more typical of small asbestos fibers that are detected in talc analyses. The mass of five such fibers is calculated as follows:

$$3\ \mu m \times 0.2\ \mu m \times 0.06\ \mu m = 0.036\ \mu m^3 \text{ per fiber}$$
$$\times 3.3 \times 10^{-12}\ g/\mu m^3 = 1.2 \times 10^{-13}\ g \text{ per fiber}$$
$$\times 5 \text{ fibers} = 6 \times 10^{-13} \text{ grams}$$

The limit of quantifiable detection for most talc analyses is approximately 6×10^{-4} weight percent. The theoretical and quantifiable detection limits assume homogeneity of the material being sampled.

7. Quality Assurance

Blank suspensions are routinely prepared and tested in order to monitor potential residual contamination from the sample jars. Blank carbon-coated grids are routinely tested to monitor the ambient fiber count. If greater than 4 fibers per grid are present, the jars are pre-cleaned or new carbon-coated grids are prepared, respective of the test.

For each analysis, one grid opening is examined by another analyst as a quality control check.

8. Background Correction

As of the time of this writing, background correction has not been necessary. Blank contamination is very rare and corrective steps are taken before the testing of material proceeds.

9. Preparation and Analysis Time

Preparation time per sample (including preparation of related materials) is one hour. Analysis search time per sample is a maximum of two hours.

10. Apparatus

A. Analytical balance with 0.0001 gram sensitivity
B. Weighing boats
C. Narrow spatula

D. Wide mouth polyethylene jars (125 mL)
E. Mild ultrasonic bath, 50-60 watts
F. Micropipette (5-10 µL range) with disposable tips
G. Standard 3 mm diameter, 200 mesh, copper TEM grids, covered with a carbon-coated formvar film
H. Transmission electron microscope (TEM) with an 80-120 kV accelerating voltage and energy dispersive x-ray analyzer

11. Reagents

A. Methyl cellulose, powder, USP 4000 cps - Fisher Certified Reagent #M-352 or equivalent
B. Water: deionized, particle free (<0.2 µm filtered)
C. Methyl cellulose solution: 0.002% (wt/vl) (20 ppm). Dissolve 20 ±0.5 mg of methyl cellulose in 500 mL of deionized particle-free water to make a 0.004% stock solution. Dilute 1:1 to make a working solution.
Note: Methyl cellulose acts as a wetting agent to aid in maintaining a uniform particle distribution as the sample dries.

12. Sample Preparation

12-1. Transfer 30 to 50 mg of talc powder to a clean 125 mL polyethylene jar.

12-2. Add 80 mL of 20 ppm methyl cellulose solution, cap and shake vigorously for one minute.

12-3. After shaking, loosen cap and ultrasonicate for 10 minutes in order to disperse the finer particles. Then shake again for one minute to produce a uniform suspension.

12-4. Immediately after shaking, uncap and remove 9.2 µL by micropipette.

12-5. Transfer 9 µL drop to a carbon film covered TEM grid. (Grid is first lightly anchored by 2 parallel strips of double-stick tape mounted about 2.5 mm apart on a clean glass microscope slide.) Repeat to make two sample grids per talc sample.

Note: Do not expel the remaining 0.2 µL suspension from the

micropipette tip. It tends to sputter and frequently destroys the stability of the sample drop.

12-6. Transfer slide with grids to a desiccator. (Drying time is 2-3 hours.) Do not leave the grids on the slide for more than one day as the double-stick tape may adhere too tightly.

Note: The talc:water ratio may need to be varied for some samples. Preparation of talc samples with significantly finer or coarser particles results in large differences in particle coverage on the TEM grid.

13. TEM Analysis

13-1. Definition of fiber: an elongated particle with parallel sides and an aspect ratio ≥3:1. When this definition is employed, fibers which fit the OSHA (4), EPA (7) or client's definition can be selectively extracted from the total fiber data for each analysis.

13-2. Scan sample at 120-150X magnification to check for even dispersion of particles and to locate grid squares with optimum particle density. (Optimum particle density is particle coverage over 15-35% of the field of view.)

13-3. Scan three grid squares on each grid at 20,000X magnification and seven grid squares on each grid at 5,000X for asbestiform minerals. Each asbestiform mineral is recorded as to type (chrysotile, tremolite, anthophyllite, etc.), structure (bundle, clump, fiber) and dimensions (length x width).

13-4. Questionable fibers are examined first by SAED. The chrysotile SAED pattern is unique and diagnostic. Amphibole SAED patterns are variable but usually characteristic. Additional analysis and measurement of amphibole SAED patterns are done if warranted.

13-5. Ten percent of chrysotile fibers are checked by EDS for further confirmation. If the SAED pattern is not clearly diagnostic, or if it is consistent with an amphibole SAED pattern, then it is examined by EDS to confirm the identification or to identify

the type of amphibole.

14. Calculation of Results

14-1-A. Mass of chrysotile fibers: M(f)
$M(f) = \pi r^2 l \times d$

where: r = fiber radius
l = fiber length
d = density of chrysotile = 2.55×10^{-12} g/μm^3
$\pi = 3.14159$

14-1-B. Mass of asbestiform amphibole particles: M(a)
$M(a) = l \times w \times th \times d$
l = length
w = width
th = thickness = 0.3 width (approximation)
d = density of amphiboles = 3.3×10^{-12} g/μm^3

14-2-A. Mass of talc deposited on each TEM grid (step 12-5)
$M(s) = T \times (V/H)$
T = amount of talc sampled (step 12-1)
V = volume of aliquot transferred to TEM grid (step 12-5)
H = volume of methyl cellulose solution (step 12-2)

14-2-B. Total estimated talc mass examined: M(t)
$M(t) = M(s) \times (N \times A(s))/A(g)$
N = number of grid squares examined
A(s) = area of a single TEM grid square
A(g) = area of an entire TEM grid (effective area over which a 9 µL drop of suspension dries)

14-3. Weight percent:

$$\frac{\text{sum total of M(f) or M(a)} \times 100}{M(t)}$$

15. Calculation of a Detection Limit

15-1. M(dl) = A minimum quantifiable mass of asbestos fibers, based on the detection of 5 fibers (approximately 6×10^{-13} grams, from Section 6)

15-2. Detection Limit (Weight Percent) = $\frac{M(dl) \times 100}{M(t)}$

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"GOD BLESS THE MICROSCOPE", *A History of the Royal Microscopical Society over 150 years, Gerald L'e Turner, published by the Society, 116 pages, 48 figures.*

On 3 September 1839, 17 microscopists met at No. 50 Wellclose Square at the invitation of Edwin Quekett, the owner. They met to consider "forming a society for the promotion of microscopical investigation, and for the introduction and improvement of the microscope as a scientific instrument". The timing was right, because during that decade Joseph Jackson Lister had published his classic paper on the achromatization of microscope lenses. Only then was the compound microscope able to out-perform the simple (Leeuwenhoek-type) microscope. (Note: Brian Ford tells us that Antony only followed the directions of Hooke, a Britisher who had earlier produced "Leeuwenhoek-type" microscopes.)

In any case, the Society was formed by Quekett and his guests among whom were Joseph Jackson Lister, James S. Bowerbank and Nathaniel Ward. These men had met informally over the previous several years and this culminated in the decision taken on 3 September 1839 to form "The Microscopical Society of London". Philologists will be interested to learn that Joseph B. Read, one of the 17, advised the use of microscopical rather than microscopic to prevent "the possibility of ourselves being mistaken for microscopic objects".

Dr. Turner's book covers little microscopical science but there is much biographical data and a complete and very readable account of the history of the (after 1867) Royal Microscopical Society. It is a great success story with many trials and tribulations, all overcome with renewed and enhanced status. The book is well written, well illustrated and should be required reading for microscopists.

Walter C. McCrone