DOC TYPE: TEST METHOD SPECIFICATION

SUBJECT: ANALYSIS OF POWDERED TALC FOR ASBESTIFORM MINERALS BY TRANSMISSION ELECTRON MICROSCOPY

LOCATION: ROYSTON, FLUID, KOLMAR

| REVISION | AUTHORIZATION | DESCRIPTION OF CHANGE |
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## 1.0 SCOPE & PURPOSE

This method is applicable to the identification and quantitation of small (typically 1-20 micrometer) asbestiform minerals in powdered talc. Samples may be previously screened with light microscopy or x-ray diffraction techniques.

## 2.0 PRINCIPLE OF METHOD

The combined techniques of transmission electron microscopy (TEM), selected area electron diffraction (SAED) and energy dispersive x-ray analysis (EDXRA) permit the detection of asbestiform minerals based on morphological characteristics, followed by a definitive mineralogical identification of each fiber.

## 3.0 INTERFERENCES

Interferences are caused by fibrous particles which must be distinguished from positively identifiable asbestos, and by large particles or particle aggregates which may obscure fibers. Positively identified non-asbestos fibers include rolled talc, ribbon talc, antigorite, silica fibers and iron oxide fibers. Organic additives such as perfumes may crystallize out as fibers or needle-shaped crystals in finished cosmetic products. In the absence of positive identification, all other fibers must be classified as unidentifiable.

## 4.0 INSTRUMENTAL CONDITIONS

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

## 5.0 SENSITIVITY

This method is capable of detecting a single fiber as small as 1 micrometer (mm) long by 0.075 mm 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 EDXRA. The mass of a fiber with the above dimensions is $1.1 \times 10^{-14}$ g for chrysotile and $1.5 \times 10^{-14}$ g for amphibole.




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## 6.0 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 mm long by 0.2 mm wide by 0.06 mm thick, which is considerably larger than the smallest fiber that can be detected (see section 5, SENSITIVITY), but is more typical of small asbestos fibers that are detected in talc analyses. The mass of five such fibers is calculated as follows:

3 mm x 0.2 mm x 0.06 mm = 0.036$mm^3$ per fiber
x 3.3E-12 g / $mm^3$ = 1.2 E-13 g per fiber
x 5 fibers = 6E-13 grams per 5 fibers.

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

## 7.0 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.

## 8.0 BACKGROUND CORRECTION

As of the time of this writing, background correction has not been necessary. The amount of background asbestos detected has been insignificant in comparison to the levels of asbestos found in contaminated samples.

## 9.0 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.

10.0 APPARATUS

10.1 Analytical balance with 0.0001 gram sensitivity

10.2 Weighing boats

10.3 Narrow spatula

10.4 Wide mouth polyethylene jars (125 ml)

10.5 Mild ultrasonic bath, minimum 50 watts

10.6 Micropipettor (5-10 ml range) with disposable tips

10.7 Standard 3 mm diameter, 200 mesh, copper TEM grids, covered with a carbon-coated formvar film.

10.8 Transmission electron microscope (TEM) with an 80-120 kv accelerating voltage and energy dispersive x-ray analyzer.

11.0 REAGENTS

11.1 Methyl cellulose, powder, USP 4000 cps - Fisher Certified Reagent #M-352 or equivalent

11.2 Water: deionized, particle free (+0.2 mm filtered)

11.3 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, by greatly reducing the surface tension of water.

12.0 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 microliters with a micropipette.

12.5 Transfer a 9 ml drop to a carbon film covered TEM grid. (Grid was first lightly anchored by 2 parallel strips of double-stick tape mounted 3 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 ml 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 a significantly finer or coarser particle size results in large differences in particle coverage on the TEM grid.

13.0 TEM ANALYSIS

13.1 Definition of fiber: An elongated particle with parallel sides and an aspect ratio ≥3:1. The definition employed may vary with the needs of the client.

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 EDXRA 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 EDXRA to confirm the identification or to identify the type of amphibole.

14.0 CALCULATION OF RESULTS

14.1 Mass of chrysotile fibers: M(f)

$M(f) = \pi r^2 l \times d$

$\pi = 3.14159$

r = fiber radius

l = fiber length

d = density of chrysotile = $2.55 \times 10^{-12}$ g/mm$^3$

14.2 Mass of asbestiform amphibole particles: M(a)

$M(a) = l \times w \times th \times d$

l = length

w = width

th = thickness $\underline{Z}$ 0.3 width (approximation)

d = density of amphiboles = $3.3 \times 10^{-13}$ g/mm$^3$

14.3 Mass of talc deposited on each TEM grid: M(s)

$M(s) = T \times (V/H)$

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)

14.4 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 microliter drop of suspension dries)

14.5 Weight percent:

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

15.0 CALCULATION OF A DETECTION LIMIT

15.1 M(dl) = A minimum quantifiable mass of asbestos fibers, based on the detection of 5 fibers (approximately 6E-13 grams, from Section 6).

15.2 Detection Limit (Weight Percent) = $\frac{M(dl) \times 100}{M(t)}$