

COSMETIC TALC

Minerals of Interest

The asbestiform minerals of primary concern in regard to cosmetic talc are fibrous serpentine (chrysotile) and fibrous amphiboles (tremolite and anthophyllite).

Analytical Techniques Used at FDA

The main techniques used at FDA are differential thermal analysis for the determination of serpentine minerals and x-ray diffraction for the determination of amphibole minerals. Because these techniques do not distinguish between fibrous and non-fibrous (massive) forms of asbestiform minerals, optical microscopy is used to confirm the presence of fibers.

A summary of the analytical methods used for the determination of asbestiform minerals in cosmetic talc along with estimated levels of detection are shown in Table 1. A brief discussion of each technique is presented below.

Differential Thermal Analysis

Chrysotile, a serpentine mineral, when present in a relatively pure talc matrix can be detected by differential thermal analysis. Under these conditions chrysotile can be detected at a level of 0.5%. A detection level of 1% is more realistic for most samples due to certain minerals which do

not directly interfere with the determination but adversely affect the level of detectability.

Differential thermal analysis is not applicable for the determination of tremolite or anthophyllite because amphiboles do not exhibit intense thermal transitions distinguishable from talc.

Chrysotile was never found in any commercial cosmetic talc samples analyzed by FDA.

X-Ray Diffraction Analysis

X-ray diffraction is suitable for the determination of tremolite and anthophyllite at levels of 0.1% and 3% respectively.

The technique employed at FDA is similar to the CTFA method J4-1, "Asbestiform Amphibole Minerals in Cosmetic Talc."

Chrysotile can be detected by x-ray diffraction provided chlorite, an interfering mineral, is not present.

X-ray diffraction does not differentiate between fibrous and non fibrous forms of asbestiform minerals. Therefore, the detection of amphiboles by x-ray diffraction requires further analysis by optical microscopy to determine if the mineral is fibrous or not.

Optical Microscopy

Optical microscopy is used to confirm the presence of asbestiform minerals whose presence is indicated by differential thermal or x-ray diffraction analysis. This is accomplished by using refractive indices, optical crystallographic properties and morphology as determined with a polarizing microscope as a means of characterizing mineral species. The method used appeared in the Federal Register (38 FR 27079, 9/28/73).

Optical microscopic examination can distinguish between fibrous and non-fibrous amphiboles. Chrysotile can also be distinguished from fibrous looking talc particles by use of appropriate refractive index liquids.

However, optical microscopy is limited to observing particle sizes which fall into the range of the resolving power of the scope and is dependent on the wavelength of incident light. For a visible microscope particle sizes of less than 0.5 micrometers (μm) would not be seen.

Fibers meeting the following criteria are counted by this method; a length to width ratio of 3 or greater, a length of 5 μm or greater and, a width of 5 μm or less.

Optical microscopic examination of a talc sample which is contaminated with asbestiform minerals will require from 4-5 hours of time per sample.

Talc Analyses Results

Of 348 cosmetic talc products analyzed to date not a single sample was found to contain chrysotile (fibrous serpentine) at a detectability level of 1-2%. In the most recent analytical survey of 61 commercial cosmetic talc products no chrysotile was found, 9 samples were found to contain tremolite at levels of 0.1 to 0.8% and 3 contained traces of anthophyllite.

Sample Preparation

The selection of talc samples for asbestiform analysis presents special problems. Some are associated with variability of the composition of talc as it is mined and the manner of the processing (crushing and grinding) to prepare talc powder. All steps in process are continuous and therefore the composition of each bag of talc produced may vary if the mineral, as mined, varies in composition. Mineralogists are in agreement that such variability is more prevalent in some mining locations than others. Therefore any given analysis performed on talc whether on the raw material or on a finished product is subject to the possibility that the sample is not representative of more than a very limited number of product batches or perhaps even retail units. Therefore any methodology developed would have to take into account the variability in product composition. To get a representative analysis the number of samples of a given product analyzed would have to be increased to compensate for possible non-homogeneity.

Talc Dusting Studies

Talc dusting experiments have been performed to estimate the level of exposure to infants during applications. These experiments were performed by the Division of Cosmetics Technology and Dr. F. Pooley, Department of Mineral Exploitation, University College, Cardiff, Wales, U.K. The work by Dr. Pooley was by contractual agreement with Johnson and Johnson.

FDA Talc Dusting Experiment

The purpose of this study was to determine, by means of simulated dusting experiments, the amount of talc an infant may inhale while the powder was being applied. The experimental data obtained was then used to calculate the low, high and average talc concentration per unit volume. The apparatus used to conduct these experiments is shown in Figure 1. A talc dispenser (T) was located a height (D) above a flat surface (S) at a horizontal distance (D) from the collection filter (F). A flow meter (M) was connected to a vacuum source (S) to maintain airflow equal to infant respiration rate. Talc was dispensed with vibrator (V).

A total of 24 talc dusting experiments were conducted using different values of H and D. The variations of H and D resulted in four different groups of data. The actual average talc concentration varied within groups from 0.07

mg/liter to 0.10 mg/liter. The largest group of experiments were conducted at H = 10 and D = 9. The data for this group is summarized as follows:

Conditions:

H = 10" (25 cm)

D = 9" (23 cm)

Flow rate (respiration rate) = 0.5 liters/minute

Sample size = 200 gms/application

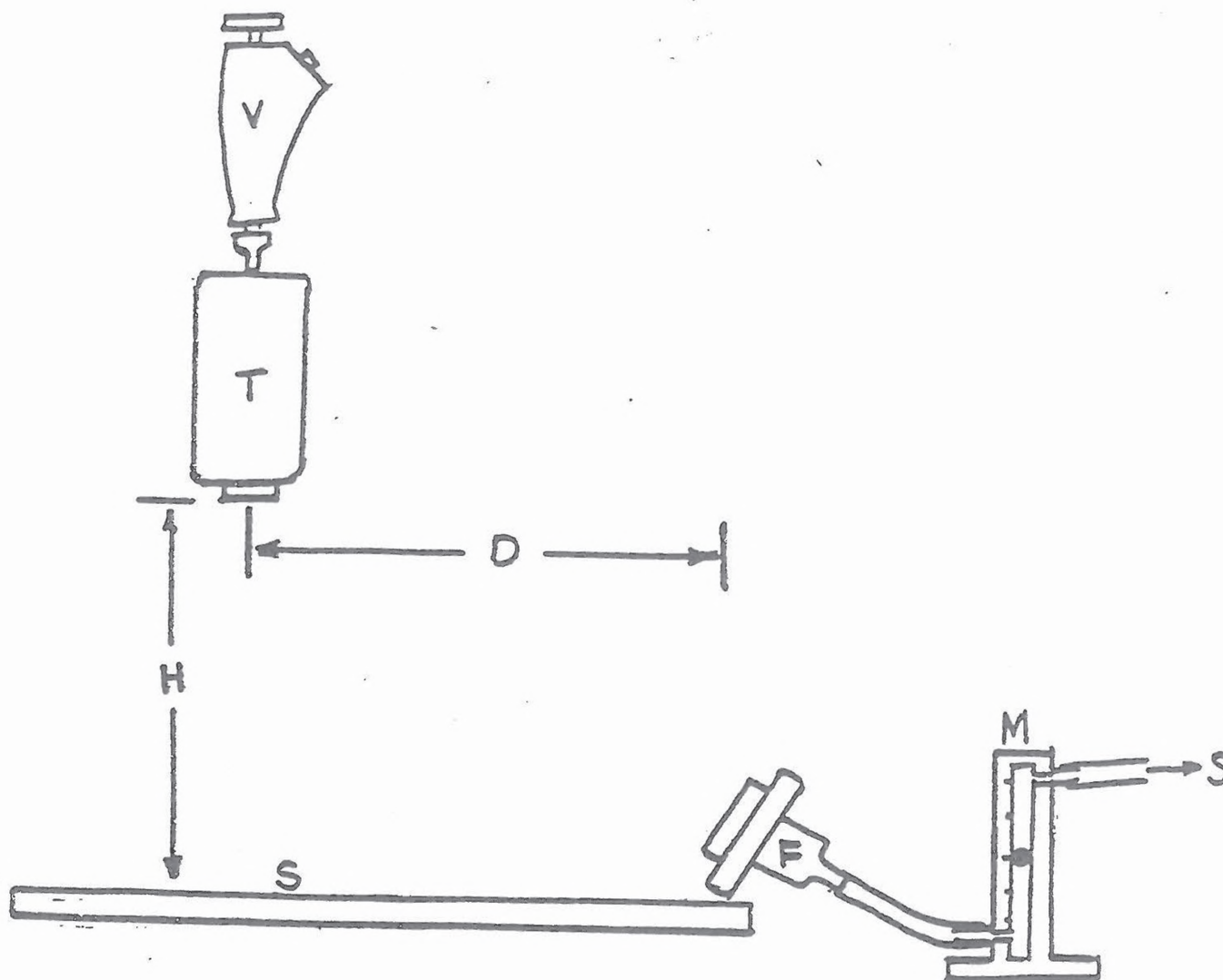
Experimental results (10 determinations):

Time of application: Varied from 15 to 40 minutes with
average time of 30 minutes

Weight of talc collected: High - 2.0 mg
Low - 0.1 mg
Average - 1.0 mg

Calculated concentration of talc: High - 0.16 mg/liter
Low - 0.01 mg/liter
Average - 0.08 mg/liter

FIGURE 1



Future Efforts

Scanning electron microscopy has not been used at FDA to analyze talc for asbestiform minerals. Specific methodology is not, as yet, available. It was not given consideration earlier because of its complexity, and limited availability.

SEM - Qualitative Analysis

Scanning electron microscope is capable of visually detecting fibers at very high magnifications and, when the instrument is fitted with an energy dispersive x-ray assembly, to obtain an elemental analysis of the fiber which identifies the asbestiform mineral. This capability makes it a valuable tool for fiber identification. Magnifications on the order of 10,000 X are suitable for this type of qualitative analysis. At this magnification particle sizes on the order 0.03 micrometers can be observed.

Qualitative analysis time is estimated to be 3 hours per sample and requires only a few milligrams of talc sample.

SEM - Quantitative Analysis

No quantitative analysis of talc for asbestiform minerals has been carried out by FDA using scanning electron microscopy. It is believed that using this method for

quantitative analysis would require scanning a series of samples from each product (perhaps as many as 10-25) and examining a large number of fields in each such sample.

To explore the use of the SEM as a tool for the analysis of talc for asbestiform minerals would require the investment of 2 man years of time over a 2 year period.

Based on a review of the asbestos contract activities in the food area we estimate that \$250,000 to \$500,000 would be required to fund a contract to develop suitable methodology for the quantitative determination of asbestiform minerals in cosmetic talc products by SEM.

	<u>X-Ray Diffraction and Optical Microscopy(OM)</u>	<u>Differential Thermal Analysis (DTA) and Optical Microscopy</u>	<u>Scanning Electron Microscopy (SEM) Fitted with X-ray Probe</u>
Equipment Cost	X-Ray \$60,000 OM 12,000	DTA \$16,000 OM 12,000	\$60,000
Time/Sample	X-Ray 3 hours OM 5 hours	DTA 2 hours OM 5 hours	Qualitative 3 hours Quantitative several days
Asbestiform Minerals detected	Tremolite and anthophyllite	Chrysotile	Chrysotile, tremolite, anthophyllite
Limits of detectability	X-Ray 0.1% tremolite 3% anthophyllite OM* Limited to parti- cles of 0.5 μ m diameter.	DTA 1% OM* not known	Essentially none-very low limit of detection possible but analysis time may take several days. Fiber diameter down to 0.03 micrometers can be observed.
Sample size	X-Ray 1 gm OM 1 mg	DTA 100 mg OM 1 mg	SEM 1-5 mg
Cost/per samples	\$500	\$450	\$1000**
Advantages	Qualitative and quantitative for amphiboles	Same as X-Ray/OM (for serpentine)	Qualitatively identifies fibers.
Disadvantages	Particle sizes below 0.5 μ m diameter there- fore result inconclusive	Same as X-Ray/OM	Quantitative Qualitative capability not known. Analysis time may be days per sample.

*Limit of resolution of optical microscope with condenser is 0.5 micrometers (μ m) diameter

**Based on a contract cost estimate for 200 samples