FILE NAME:

DATE: 1975 Oct 7

DOC#:

DOCUMENT DESCRIPTION:

COSMETIC, TOILETRY and FRAGRANCE ASSOCIATION, INC.

F-873

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# ASBESTIFORM AMPHIBOLE MINERALS IN COSMETIC TALC

## Part I: X-ray Diffraction Method
## Part II: Optical Microscopy and Dispersion-Staining Method

### Introduction

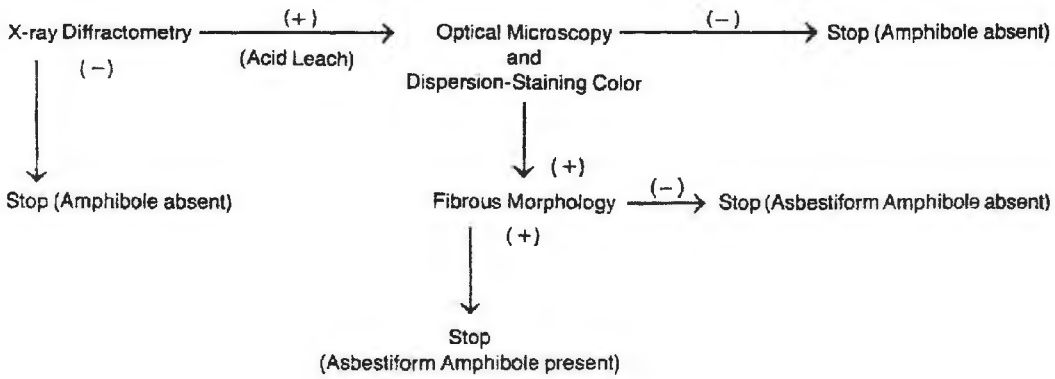
The method which has been adopted for the detection of amphibole minerals in cosmetic talc is the generally accepted method of x-ray diffraction. Methods which appear in the literature for the detection of fibrous amphibole, such as, transmission electron microscopy with selected area diffraction[1] and electron microprobe,[2] have also been considered since they are capable of a lower level of detection than by x-ray diffraction. However, they have not been adopted since they suffer from the drawbacks, that the amount of material under examination is quite small (less than a microgram) and the time for analysis, expertise required, and expense of equipment eliminates them as routine methods.

The methodology presented is the most practical available, based on current technology. The use of Transmission Electron Microscopy with Selected Area Electron Diffraction offers greater sensitivity, but is not presented since it is unsuitable for normal quality control application.

Enrichment or concentration techniques using flotation cells have been tried as a means of improving the detection level; however, all efforts so far have been unsuccessful.

### Principle

The x-ray diffraction method is based upon the principle that when a crystalline material is placed in an x-ray beam, a portion of the x-rays are diffracted by each set of atomic planes within the crystal. The diffracted rays strike a scintillation counter as the sample is scanned through a prescribed angle with the resulting development of peaks corresponding to each interplanar distance (d). A peak with d value in the range of 8.04 to 8.85A° for a sample talc is strong evidence for the presence of amphibole in that talc. The level of detection of amphibole by this method is 0.5% and above. The variability of detection is caused by such factors as age and manufacturer of x-ray diffractometers, sample homogeneity, specific amphibole mineral present, morphology of amphibole, particle size, preferred orientation, etc. For these reasons the level of detection should be reported for levels above 0.5%, since below this level the data has been found to be not reproducible. If a statistically significant peak is found of intensity equal to or greater than that obtained for the 0.5% standard in the d range for amphibole, described above, then the sample must be put through the following confirming scheme:



## Part I: Amphibole Minerals by X-ray Diffractometry

### Apparatus

1. X-ray diffractometer, employing nickel-filtered copper K-alpha radiation, horizontal or vertical goniometer with variable scan speed capability, suitable talc pellet sample holder, variable speed recorder, electronic panel including ratemeter and variable attenuation and time constant settings
2. Hydraulic press, capable of attaining a pressure of 15,000 to 24,000 lb calculated on a 3" ram
3. Mortar and pestle or grinding mill (Note 1)
4. Waring Blendor,* or equivalent blender
5. Spex Mixer/Mill,* or equivalent mechanical mixer
6. Sieve, 325-mesh
7. Optical microscope (Note 2)
8. 1¼" pellet press

### Reagents

1. Standard talc sample, containing no detectable amphibole minerals
2. Standard tremolite sample, at least 80% pure
   Sample of tremolite standard may be obtained by ordering from The Cosmetic Toiletry and Fragrance Association, Inc., 1133 Fifteenth Street, N.W., Washington, D.C. 20005.
3. Denatured ethanol
4. Boric acid

### Procedure

The procedure consists of slow-scanning, under previously determined conditions, a compressed pellet of the sample talc in the 11.0 to 10.0°2$\theta$ (8.85 to 8.04Å) region for the presence of an amphibole peak. There are times when it is difficult to discriminate a possible peak for amphibole over the background noise level.

*Registered Trademark

Should the presence of a small amphibole peak above the background "noise" be in question, it will be necessary to statistically evaluate the scan. A timer/scaler is required on the electronic panel of the x-ray diffractometer. In order for a peak to be statistically significant, the peak intensity must equal or exceed three standard deviations ($3\sigma$) above the average background intensity (N):

$N + 3\sigma$ = minimum peak intensity
N = average background count

Where:

$$\sigma = \sqrt{N}$$

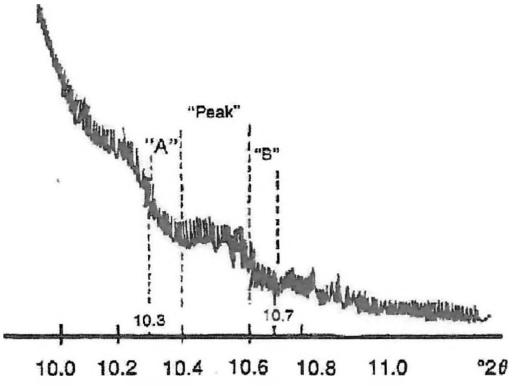



**Figure 1.**

Determine the region of the scan in question: in the Figure 1 scan, a peak appears to be present in the 10.40 to 10.60 °2$\theta$ region.

Slow scan with cumulative pulse counting through the peak region three separate times and average the number of counts.

Determine a background count by scanning a region equal to ½ of the °2$\theta$ region covered by the peak, immediately before and after the peak. The counting time for each of these background regions will equal ½ the total counting time used for the peak. Count each background region three times. Then average each region and add the two averages to obtain the background count (N).

Example:

In Figure 1.

| | *Region (°2θ)* | *Time (sec.)* |
|---|---|---|
| Peak | 10.40 to 10.60 | 120 |
| Background | | |
| Region A | 10.30 to 10.40 | 60 |
| Region B | 10.60 to 10.70 | 60 |

| *Peak* | | *Background* | | | |
|---|---|---|---|---|---|
| | | *Region A* | | *Region B* | |
| *10.40 to 10.60 °2θ* | | *10.30 to 10.40 °2θ* | | *10.60 to 10.70 °2θ* | |
| *time secs.* | *counts* | *time secs.* | *counts* | *time secs.* | *counts* |
| 120 | 60,332 | 60 | 28,784 | 60 | 28,506 |
| 120 | 59,870 | 60 | 28,943 | 60 | 28,368 |
| 120 | 60,105 | 60 | 28,634 | 60 | 28,204 |
| Average | 60,102 | | 28,787 | | 28,359 |

$N = 28{,}787 + 28{,}359 = 57{,}146$

$\sigma = \sqrt{57{,}146} = 239 \quad 3\sigma = 717$

$N + 3\sigma = 57{,}146 + 717 = 57{,}863$

The actual number of counts obtained for the integrated peak intensity was 60,102; therefore, the "suspect" peak is statistically present in the scan.

**Standard Preparation**

Optimal instrument conditions must first be determined with the use of tremolite standards: 1.0%, 0.75%, 0.5% tremolite by weight, prepared in a standard talc which is free of interfering peaks in the 11.0 to 10.0°2$\theta$ region.

Weigh out appropriate amounts of standard talc and tremolite both of which have been ground to pass a 325-mesh sieve. Transfer to a Waring Blendor.* Add 100 ml of ethanol to the blender and blend at low speed for 5 minutes.

Carefully transfer the contents of the blender, with repeated ethanol washings, into a large beaker. Evaporate the ethanol on a steam bath.

Shake the sample in a plastic vial for 5 minutes on a Spex Mixer/Mill* to remove clumps and caked sample resulting from the evaporation of ethanol.

Determine by microscopy the homogeneity of the prepared standard previous to the x-ray diffraction analysis.

Press the homogeneous standard into a 1¼" pellet with a backing of boric acid. Transfer 2 (± 0.2) g of standard to the die-holder and evenly distribute on a polished, scratch-free die. Distribute 4 (± 0.2) g of boric acid evenly on the talc layer. Press the mixture into a pellet under conditions suitable for obtaining a smooth planar surface (for example, a pressure of 15,000 to 24,000 lb calculated on a 3" ram has been found to produce suitable pellets). The resulting pellet must have a talc face which is free of flaws; if not, the pellet must be discarded (Note 3). Prepare two acceptable pellets from each standard.

*Registered Trademark

### Sample Preparation

Prepare two pellets from each sample in the manner described for the standard pellets. Make a qualitative scan from 4 to 50 °2$\theta$ on one of these pellets to ascertain the presence of amphibole above the 2% level or the presence of mineral impurities having interfering peaks in the 11.0 to 10.0 °2$\theta$ (8.85 to 8.04 Å) region of the scan. The presence of such interference will eliminate use of the x-ray diffraction method for the sample, and one will have to proceed directly to the microscopical procedure.

### Instrumentation

Instrumental variables are optimized on the 1% standard. Lower standards are then analyzed under the optimum conditions to determine the lower level of detection. Of major importance in obtaining maximum instrument sensitivity are a slow diffractometer speed combined with compatible recorder speed, and high attenuation combined with a statistically acceptable time constant on the ratemeter. Under appropriate instrumental conditions the peak obtained for the 0.5% standard should be detectable above background noise as shown in Figure 2.

Typical instrumental conditions employed for the Siemens Diffractometer (Model No. M386-X-A4), and Counter and Recorder Unit (Type T) are:

Radiation: Cu with $K_b$ filter at 40KV and 24 ma
Divergence slit: 1° Receiving slit: 0.2 mm
Goniometer speed: $^1/_{10}$°2$\theta$/minute
Recorder speed: 300 mm/hour
Attenuation: $1 \times 10^3$ impulses/second
Time constant: T (s) = 4

Statistical error of 1.1% under these conditions

Rise Time = 0.18
Attenuator = 20

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