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CTFA Method J4-1
Asbestiform Amphibole Minerals in Cosmetic Talc
Part II; Microscopy and Dispersion-Staining Method

TENTATIVE REVISION

Apparatus

1. Polarizing microscope. Best results will be obtained if the instrument includes the following:
 - a. Individually centering objectives or centerable stage.
 - b. Bertrand lens
 - c. High-intensity light source
 - d. Centering condenser/substage
2. Dispersion-staining device (Note 4)
3. Vacuum filtration equipment, including either a porcelain cone with glass fiber filter mat or a porous glass bottom cup

Reagents

1. Hydrochloric acid, 10% v/v
2. Cargille immersion liquid Series HD, $n_D^{25^\circ} = 1.605$ (Note 5)
3. Cargille immersion liquid, $n_D^{25^\circ} = 1.650$

Procedure

Acid Treatment

Because of the interference caused by some carbonates (e.g. calcite) in the detection of asbestiform amphiboles in talc by optical microscopy/dispersion-staining, it is necessary to first remove these carbonates by a simple acid leaching procedure:

Weigh out 2 g of the talc into a 100 ml beaker. Add 25 ml of 10% v/v HCl slowly (to prevent excessive evolution of gas if carbonates are present) and heat, with occasional stirring on a steam bath for 30 minutes.

Filter with vacuum filtration equipment, and wash several times with hot water. Dry the talc.

Optical Microscopy and Dispersion-Staining

Instrument Preparation

Carefully adjust the dispersion staining objective for optimum viewing of central stop colors. The microscope is first arranged for Köhler illumination: lamp iris (field diaphragm) in focus in the field of view; lamp filament in focus in objective back focal plane. The lamp, if movable, should be placed close to the mirror so that the image of the lamp filament is as small as possible in the objective back focal plane. This along with overvolting the lamp by 15-20% and removing the daylight blue filter will ensure bright central stop colors.

The stage should next be centered and the central stop should be precisely centered with respect to the image of the substage iris (aperture diaphragm) in the objective back focal plane. The substage iris is then closed just behind the central stop and the Bertrand lens (or equivalent viewing system) is removed. The preparation is scanned for central stop colors in the visible; critical study of an area or a given particle is carried out with the field diaphragm nearly closed around the center portion of the field of view.

Identification of Tremolite and Anthophyllite Minerals in Talc by Dispersion-Staining

Mount a representative sample (about 0.1 mg.) in each of the Cargille refractive index liquids and examine carefully for tremolite and anthophyllite particles. The sample is mounted in the drop of Cargille liquid between slide and coverslip. The talc sample is best dispersed by sliding the coverslip with a rotary motion of a pencil eraser "gripping" the coverslip. The final prep should be thin and the coverslip should be parallel to the slide (this avoids a "prism" shaped prep that will decenter the image of the substage iris relative to the central stop).

IMPORTANT: Since dust and other foreign debris can cause interference in the identification of small particles by dispersion-staining, great care must be taken that both slide and coverslip are thoroughly clean before use. This is accomplished by wiping briskly with isopropyl alcohol until dry, followed with a lens tissue (lint-free) as final wipe. The pencil eraser must be dipped in

diluted rubber cement and dried before use to avoid transfer of particles to the top of the coverslip.

Minerals, especially silicates such as those under consideration here, vary in refractive index. Table I shows average refractive index values for talc, tremolite, and anthophyllite.

TABLE I

MINERAL	REFRACTIVE	INDICES	(AVERAGE VALUES)
	α	β	γ
Talc	1.545	1.591	1.595
Tremolite	1.599	1.612	1.622
Anthophyllite	1.645	1.658	1.668

The analysis of a talc sample proceeds as follows:

Examine the sample, immersed in Cargille HD liquid, $n_{25}^D = 1.605$, in the dispersion-staining central stop mode. Under these conditions, the minerals will show the following dispersion-staining colors:

Talc - Pale blue remaining the same with rotation of the sample relative to the polarizer, or

Pale blue changing to yellow with rotation. The variation of the color change is due to the fact that a mineral may lie in one of two positions relative to its principal optical orientation.

Tremolite - Yellow changing to magenta with rotation, or

Yellow changing to orange with rotation.

Anthophyllite - Orange changing to magenta with rotation, or

Orange-red (or yellow) changing to blue with rotation.

In order to prevent the misidentification of a talc particle (plate, shard, or lath-shaped) on edge as being an amphibole mineral, the following additional test must be applied to the particle in question, along with the dispersion-staining technique:

This test applies only to the talc sample immersed in Cargille HD liquid, $n_D^{25} = 1.605$.

Using a higher magnification (200-250 X) and plane polarized light, determine the refractive indices (R.I.) of the particle with respect to this Cargille HD immersion liquid, using the Becke line method. Then -

If both R.I. < 1.605 , the particle is not amphibole and is probably talc.

If R.I. ≥ 1.605 , in at least one optical direction, the particle may be an amphibole mineral.

Next, the sample is examined in the same manner, immersed in another Cargille liquid, $n_D^{25} = 1.650$ to provide additional dispersion-staining evidence. The following central spot colors will be observed:

Talc - dark blue (yellow edges) remaining the same with rotation, or

dark blue changing to yellow with rotation, for talc plates "on edge".

Tremolite - dark blue (yellow edges) remaining the same with rotation, or

yellow changing to blue with rotation.

Anthophyllite - Orange changing to magenta with rotation, or

yellow-orange changing to blue with rotation.

The combination of crystal morphology and optics is a considerable help in making sure of the identity of a given mineral component. One should look for different views of a given mineral and/or vary the view on a single crystal by tapping gently on the top of the coverslip with a needle while observing dispersion-staining colors. In this way, fiber-like talc plates or shards on edge can be tipped into the plate view. Both morphology and optics must be noted in order to make identification of asbestiform tremolite or anthophyllite.

In order for an amphibole mineral to be considered asbestiform (fibrous) it must meet the following definition (Reference 4).

1. Particles must appear to be fibrous rather than as crystals or slivers.
2. The maximum diameter of a fiber to be counted is 3 microns.
3. The maximum length of a fiber to be counted is 30 microns.
4. The length to width ratio must be 5 or more to 1, that is, 5 times or more longer than wide.
5. The separate or individual fibers must contain fibrils or the "bundle of sticks" effect, unless they are at a nondivisible stage. A fibril cannot be subdivided and would be counted, if it meets the other criteria. The length to width ratio of 5 or more to 1 is not meant to imply that other particles are not hazardous.

Report results as "Asbestiform Amphibole Present" or as "Asbestiform Amphibole Absent".

It is imperative that both dispersion-staining color and fibrous morphology criteria be satisfied, as well as the Becke line determination of refractive index, before identifying a particle as asbestiform amphibole, since other substances may show dispersion-staining results similar to those described. (Note 6)

Notes

1. Talc to be analyzed and the tremolite used to prepare standard samples must be finer than 325 mesh (maximum particle size of 44 microns). The Tekmar Analytical Mill (Model A-10) is recommended. It is available from:

Tekmar Company
P.O. Box 37202
Cincinnati, Ohio 45222

2. It is important that the homogeneity of the prepared talc-tremolite standard samples be verified by optical microscopy.
3. This requirement is critical since excessive surface scatter will cause abnormally high background counts.
4. The only commercially available dispersion-staining device is sold by:

Walter C. McCrone Associates, Inc.
2820 South Michigan Avenue
Chicago, Illinois 60616

5. Available from:

R. P. Cargille Laboratories, Inc.
Cedar Grove, New Jersey 07009

- or from laboratory suppliers.

6. Method J4-1, Part II, may not be applicable for analysis of cosmetic talcs having extremely fine particle size (as is the case for some facial powders). It may also be difficult to apply the Becke line method when very fine fibers are detected. In these instances, an instrumental technique such as transmission electron microscopy/selected area electron diffraction is recommended.

References

1. Rohl, A.N., Langer, A.M., Environmental Health Perspectives 9,95 (1974)
2. Rubin, I.B., Maggiore, C.J., Environmental Health Perspectives 9,81 (1974)
3. L.S. Birks, X-Ray Spectrochemical Analysis, pages 54-55, Interscience Publishers (1959)
4. "Tremolite and Talc." U.S. Department of Labor, Occupational Safety and Health Administration, Field Information Memorandum #74-92, November 21, 1974.