

MEMORANDUM

DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE
PUBLIC HEALTH SERVICE
FOOD AND DRUG ADMINISTRATION

TO : George H. Boone
Director of Science, HFR-2160
New York District

DATE: October 5, 1976

FROM : Product Composition Branch, HFF-446
Division of Cosmetics Technology

SUBJECT: Asbestos in Talc

*Yes You approved at
last 4th. res. meeting.
WMP
10-14-76*

This memorandum is in reply to your telephoned offer to provide us with assistance in the development of analytical methods for the detection and determination of asbestos in cosmetic talcs.

It is my understanding that you expressed interest in the development of methods for the detection of chrysotile and tremolite by optical microscopy and that you would like to receive some samples for analysis. Per your request we are sending you, under separate cover, the following samples:

DCST-77-1371
DCST-77-1372
DCST-77-1373
DCST-77-1374
DCST-77-1375
DCST-77-1383
DCST-77-1384
DCST-77-1385

These samples consist of talc with known amounts of added chrysotile or tremolite. The talc used to prepare these samples is a commercial product which was analyzed by x-ray diffractometry and differential thermal analysis. The analysis indicated the absence of tremolite, chrysotile and chlorite at the limit of detection. To make the samples as uniform in composition as possible, the talc and asbestiform mineral were mixed in a blender using alcohol as a mixing aid. The alcohol was then removed by evaporation and the sample remixed.

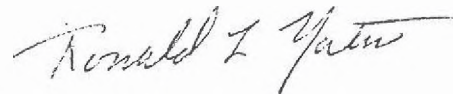
George H. Boone

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We would appreciate receiving the results of your analyses when you have completed the examination of these samples. We would like to know the identity of the asbestos in each sample and, to the extent possible, the amount present. We would also appreciate receiving a copy of your methodology or a reference.

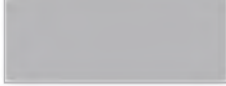
Enclosed are some reprints of material dealing with the microscopic identification of amphibole and serpentine minerals.

Thank you for your cooperation, assistance and interest.



Ronald L. Yates

(b) (5)



Fr. J. Stuart

TO: Members of the CTFA Task Force on Methodology for the
Detection of Asbestos in Talc

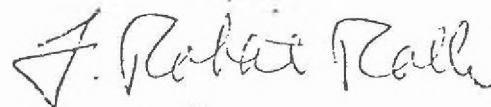
At the September 11, 1974, meeting of the CTFA Task Force on methodology for the detection of asbestos in talc for cosmetic use, the results of our round robin analysis were discussed. The two "cookbook" procedures for the detection of chrysotile and asbestiform amphibole in cosmetic grade talc have now been modified as a result of this evaluation. The two methods would assure that a talc is 99.0 to 99.5% free of asbestiform minerals.

A shortcoming of the round robin analysis for chrysotile in talc is that no one performed the necessary optical microscopy-dispersion staining back up method to the DTA procedure. This method is of great importance since the primary method (DTA) is specific only to the serpentine class of minerals; that is, antigorite, chrysotile, etc. which have different morphologies.

Enclosed you will find three coded talc samples possibly containing chrysotile. Would you please examine these samples strictly in accordance with our proposed optical microscopy-dispersion staining test method, a copy of which is attached.

I will contact you by telephone on or around October 31, for your results and comments.

Thank you,



F. Robert Rolle, Ph.D.
Chairman, Task Force on
Methodology for the Detection
of Asbestos in Talc

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Optical Microscopy and Dispersion Staining

Having failed the DTA test for serpentine minerals, the sample must next be tested by optical microscopy/dispersion staining or by transmission electron microscopy.

The following optical method uses the Becke line procedure for determining relative refractive index.

1. Prepare a microscope slide by dispersing approximately 0.3 mg. of talc in Cargille refractive index liquid 1.550, under a 22 x 22 mm. cover glass.
2. On examination with the polarizing microscope any chrysotile fibers or fiber bundles in the prepared slide will exhibit:
 - a. in the crossed polars mode, low order (first order, grey-white) retardation colors and straight extinction characteristics.
 - b. distinctive fibrous morphology - in most cases a length: diameter ratio of several hundred to one.
 - c. in the plane-polarized light mode a refractive index match when the long direction of the fiber is parallel to the plane of polarization. In the plane perpendicular to this the fibers will exhibit a refractive index less than that of the liquid.
3. Repeat 1 and 2 using Cargille refractive index liquid 1.544. Under these conditions for 2.c. (above) the refractive index match will occur when the short (diameter) direction of the fiber is parallel to the plane of polarization. The other index exhibited will be greater than that of the liquid.
4. Using the slide prepared in 1, examine the sample in the dispersion-staining mode with central stop (dark-field).

Any chrysotile fibers present will exhibit blue colors, distinctly separating them from the non-chrysotile particles.

If this test is carried out with plane polarized light as the illuminating source the color exhibited will vary with fiber orientation, so that as the stage is rotated the chrysotile fibers will change color: blue→blue-purple.

Again, for positive confirmation, both color and morphology (fibers, large l:d) criteria must be satisfied.

5. If optical microscopy/dispersion staining gives negative results, chrysotile may be present with a diameter too small to be detected by optical microscopy, thus, transmission electron microscopy with SAED must be employed.

Following is the transmission microscope procedure. If chrysotile serpentine is detected at the same level found by DTA by either microscopical method, then the talc fails.

Microscopic Detection of Asbestos in Talc

by

Arnold E. Schulze and William V. Eisenberg
U.S. Food and Drug Administration, Washington, D.C.

The six kinds of asbestos can be distinguished from talc and from each other by their refractive indices, other optical crystallographic properties, and morphology. Talc occurs mainly in the form of thin plates, which may appear to be fibrous when seen edgewise in microscopic view. Beta and gamma indices of talc vary from about 1.575 to 1.590, beta being very close to gamma. All of the principal refractive indices of chrysotile are less than 1.574, and those of the amphibole types of asbestos are greater than 1.590. One can, therefore, distinguish chrysotile asbestos from fibrous looking talc particles in a 1.574 refractive index liquid, and the amphibole types from talc in a 1.590 liquid. The accompanying table of optical crystallographic properties for talc and the asbestos minerals shows refractive indices which are usually encountered in these minerals, but occasional samples may have indices which are somewhat higher or lower.

Method

Weigh out 1 mg. of a representative portion of talc on each of two microscope slides. Mix the talc on one slide with a drop of 1.574 refractive index liquid, and the other with 1.590 liquid, and place on each a cover glass sufficiently large so that the liquid will not run out from the edge (18 mm. in diameter or larger). Observe the material with a polarizing microscope at a magnification of about 400 X, using the optical crystallographic technique to detect the presence of asbestos. In the 1.574 liquid look for asbestiform fibers with indices less than 1.574 in both extinction positions, and in the 1.590 liquid look for fibers with indices exceeding 1.590. If any such particles are found, check type of extinction and sign of elongation to confirm tentative identification as asbestos. Count and record the number of asbestiform fibers found in 1 mg. as determined from a scan of either or both slides at a magnification of about 400 X. To determine specific identity of asbestiform fibers, make mounts in appropriate refractive index liquids, and refer to the optical crystallographic data in the accompanying table.

OPTICAL CRYSTALLOGRAPHIC CHARACTERISTICS OF ASBESTOS MINERALS AND TALC

Substance	Reference for n	$n\alpha$	$n\beta$	$n\gamma$	Extinction	Elongation
Actinolite (variety of tremolite)	(2) p. 172 (8) p. 285	1.614 1.615-1.655	1.630 1.625-1.665	1.641 1.64-1.68	Inclined	Positive
Amosite	(4) p. 261 (4) p. 261	1.663* 1.675*	-- --	-- --		
Anthophyllite	(2) p. 170 (2) p. 222 (5) p. 298 (2) p. 222 (2) p. 172 (6) p. 236 (2) p. 173 (2) p. 116	1.598 1.598 1.598-1.674 1.608 1.619 1.619-1.633 1.629 1.633	1.623 -- 1.605-1.685 -- 1.630 1.630-1.642 1.635 1.638	-- 1.623 1.615-1.697 1.631 1.640 1.640-1.657 1.640 1.652	Parallel.	Positive
Chrysotile	(2) p. 99 (2) p. 100 (3) p. 154 (8) p. 260 (6) p. 290 (8) p. 260 (2) p. 104 (1) p. 40 (1) p. 40	1.493 1.508 1.529-1.559 1.53-1.54 1.542 1.542 1.546 1.546 1.548	1.504 1.512 1.530-1.564 -- -- 1.543 (calc.) 1.550 1.557 1.560	1.517 1.522 1.537-1.567 1.54-1.55 1.555 1.555 1.557 1.557 1.560	Parallel	Positive

$$* \frac{n\alpha + n\beta}{2}$$

Substance	Reference for n	n α
Crocidolite (Riebeckite)	(2) p. 125	--
	(2) p. 187	--
	(6) p. 241	1.693
	(2) p. 226	1.695
	(2) p. 226	1.695
	(2) p. 127	1.697
Talc	(8) p. 259	1.538-1.545
	(2) p. 164	1.539
	(3) p. 167	1.539
	(6) p. 281	1.539
	(8) p. 259	1.540
	(1) p. 41	1.541
	(1) p. 41	1.544
	(1) p. 41	--
Tremolite	(7) p. 758	1.545
	(2) p. 222	1.599
	(8) p. 285	1.599
	(3) p. 169	1.599-1.612
	(2) p. 169	1.600
	(2) p. 222	1.602
	(2) p. 222	1.602
	(6) p. 237	1.602-1.623
	(7) p. 766	1.604
	(2) p. 222	1.604
	(2) p. 222	1.609
	(2) p. 222	1.609
	(2) p. 222	1.613

CHARACTERISTICS OF ASBESTOS MINERALS AND TALC

n_{β}	n_{γ}	Extinction	Elongation
1.687	--	Parallel	Negative
1.695	--		
1.695	1.697		
--	1.703		
1.698	--		
1.700	1.703		
--	1.575-1.590	Parallel or 2° or 3°	Positive
1.589	1.589		
1.589	1.589		
1.589	1.589		
nearly = n_{γ}	1.575		
1.585	1.585		
1.592	1.592		
1.594	1.594		
1.584	1.584		
1.613	1.625	Inclined	Positive
1.613	1.625		
1.613-1.626	1.625-1.637		
1.616	1.627		
1.614	1.635		
1.618	1.631		
1.613-1.638	1.624-1.650		
1.612	1.628		
1.617	1.630		
1.622	1.636		
1.623	1.636		
1.621	1.634		

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