

September 17, 1974

H. Nazarewicz

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TO: R. E. Norwood
FROM: H. D. Stanley
RE: Meeting Notes of CTFA Talc Subcommittee
CC: J. K. Barr W. M. Stephenson
J. P. Bartels D. B. Vanderheiden
D. W. Holstein G. A. Wells
W. Nazarawicz

Enclosed please find the notes from the CTFA Talc Subcommittee meeting held August 14, 1974. This report can be divided into three main sections:

First, a section in which Dr. Rolle of Johnson and Johnson describes the work of the Methodology Committee (Pfizer is an active member of this).

Second, a report by Mr. J. N. Sivertson of Johnson and Johnson on an estimate of a safe level of detection of asbestos and asbestiform minerals in talc. A copy of the source of the data used in this report is also attached. The report proposes a 1% level of asbestos is safe and well below the 1976 OSHA standards for talc miners.

Third, the lack of definitive data on the carcinogenic character of tremolite was brought up. Apparently, there is not general agreement on this aspect. I don't know what, if anything, we can contribute here.

Hal

Harold D. Stanley

HDS/jmb

attachments

Meeting Date: August 14, 1974
Release Date: SEP 6 1974

MEETING NOTES OF CTFA TALC SUBCOMMITTEE

PRESENT:

Mr. George Sandland, Chairman
Dr. Murray Berdick
Dr. George L. Cohen
Mr. Sal DiBianca
Mr. Ray Krammes
Mr. George Lee
Mr. Fred Roesch
Dr. Robert F. Rolle
Mr. Jack Sivertson
Dr. C. A. Thompson
Dr. John Travers
Mr. Thomas Wolf

Bristol Myers Products
Chesebrough Ponds
Bristol Myers Products
Mennen Company
Whittaker, Clark & Daniels
Johnson & Johnson
Whittaker, Clark & Daniels
Johnson & Johnson
Johnson & Johnson
R. T. Vanderbilt
Avon Products
Colgate Palmolive

COPY:

Dr. Tryggve Baak
Dr. Christopher Costello
Mr. Harold D. Stanley
Mr. Ronald Yakupcin
Mr. Alan M. Harvey

United Sierra
Colgate Palmolive
Pfizer
Kolmar Laboratories
R. T. Vanderbilt

Scientific Advisory Committee

Covington and Burling

REPORT OF AD HOC METHODOLOGY COMMITTEE

(Dr. F.R. Rolle, Chairman)

Dr. Rolle described the status of the work of the Methodology Committee:

The following samples were submitted to members of the committee who are able to perform analyses by Differential Thermal Analysis (for serpentine) and X Ray Diffraction (for amphibole).

- 1.) Sample spiked with chrysotile
- 2.) Sample spiked with tremolite
- 3.) Two samples supplied by FDA
- 4.) Two samples from Cyprus Mines (Unperfumed)
- 5.) One sample from Pfizer (Unperfumed)
- 6.) Sample of Avon's Rapture
- 7.) Sample of Ammens Powder
- 8.) Sample of Cashmere Bouquet
- 9.) Sample of Johnson's Baby Powder

Items 6, 7, 8 & 9 were purchased from the market, and since they might have been recognized by odor, were masked by the addition of musk oil.

All samples were submitted with a code number as the only identification. Dr. Rolle alone, has the cross referenced identification. Methodology involves DTA for Chrysotile and X Ray diffraction for tremolite with microscopy as a follow up secondary method in the case of a positive for serpentine. IR is an alternate method for tremolite. Copies of the methods are attached.

Not all of the participants in the round robin have completed the tests, however, there is very good agreement of data among the submissions received so far. One sample from FDA showed the presence of serpentine particles.

The DTA method for serpentine has a limit of 0.5 to 1.0%. One limitation in the method occurs when a positive reading is found, requiring a follow-up using microscopy. The method requires reading an exothermal peak at 800° and an endothermal peak at 650°C.

Antigorite will be detected as serpentine, but shows up as platey by microscope. Many times chrysotile is so fine that it can't be seen by optical microscopy at 300X to 1000X, but it will be detected by SEM and still requires TEM for positive identification and determination.

A talc sample containing 0.75% tremolite failed using the x ray diffraction method. Avon was able to detect tremolite at this level also, by means of I.R. spectroscopy. One advantage in the IR method is its specificity for tremolite.

Dr. Rolle stated that FDA resurrected the optical microscopic method in May and requested samples to be run by J & J and McCrone, both of whom came up with results similar to those obtained in our "round robin" evaluation in 1973. The same criticisms of the method were likewise offered.

Dr. C. S. Thompson gave a report of discussions with the N.Y. Bureau of Mines and their consideration of proposals submitted by R.T. Vanderbilt:

- 1.) It has been proposed that the certification of an ore body, or mine, be required in order for it to be used to supply talc for cosmetic, food, or similar uses.
- 2.) Suggestions have been made to get around "aspect ratio" as the definition of a fiber. An aspect ratio of 3 to 1 and length greater than 3 to 5 M has been pushed by FDA. ASTM on the other hand is considering a 100 to 1 aspect ratio.

Dr. Thompson stated that RTV is well on the way to getting NIOSH to work on this problem along with a review of an RTV petition.

* * * * *

Mr. J.N. Sivertson, Manager of Statistical and Computer Operations at J & J then presented "An Estimate of a Safe Detection Level of Asbestos Fiber in Baby Powder Talc." A copy of the report is attached. Included attached also is Reference (1) which represents the source of the data used.

The data presented by Mr. Sivertson indicate clearly that a 1% limit each for asbestos and tremolite in talc would offer significantly (48,300 times) better protection for the users of talc based products, than would be provided, using the 1976 OSHA standards for talc miners. In addition, we now have adequate, dependable analytical methodology to make the application of the specification realistic.

We recommend, therefore, that a recommendation be made by CTFA to FDA that a proposal of the 1% limits be published in the Federal Register. This recommendation should be well accepted this time since they favor the methods approach and also the very substantial safety factor based on the J. & J. data should lend weight to the proposal. FDA has already received the Reference (1) material and has had ample opportunity to review it.

The J & J data are based on work done by Dr. Pooley using J. & J. Baby Powder. The CTFA Talc Subcommittee has compiled a sedimentation rate comparison of J & J Baby Powder with several brands of talcum powders representing 80% of the total market. This comparison was described in our meeting notes of January 4, 1974 (Release date - April 4, 1974), and should justify the application of the J & J data to all talcs.

In a review of literature available on the subject of tremolite, all investigators whose work has been published appears to demonstrate that tremolite is not carcinogenic. We refer this to the Pharmacology and Toxicity Committee for an opinion regarding the need for any additional substantiation of the non-carcinogenicity of tremolite. A copy of a literature survey is appended for reference. (SAC Committee only)

If indeed, tremolite is not a carcinogen, it would appear that a specification limit of 1% may be unnecessary, and we should recommend either a higher limit, or perhaps no limit at all in our petition to FDA.

The time is particularly propitious for us to petition FDA. There is no regulation, (only a proposal which has been refuted not only by industry but also by academic experts) and unless FDA comes up with some form of regulation soon, the individual states will be making up their own. If we permit this to happen, there will be most certainly a lack of uniformity across the board.

It is requested that this subject be discussed as part of the agenda for the next meeting of the Scientific Advisory Committee on September 18, 1974.

G. W. Sandland
Chairman, CTFA Talc Subcommittee

DETECTION OF CHRYSOTILE IN TALC BY DTA

Johnson & Johnson
Avon

1. This method permits the detection of chrysotile and other serpentine minerals in pharmaceutical grade talc containing less than 10% by weight chlorite. The minimum level of detection is 0.5 - 1% by weight of chrysotile asbestos.
2. The instrumental parameters must be determined for the particular differential thermal analyzer which is employed, with regard to geometry of the sample holder, sensitivity of the differential thermocouple, etc., such that 0.5 - 1% by weight chrysotile may be easily detected.
3. The successful development of the DTA method is, therefore, based on the preparation of homogeneous chrysotile-talc standard samples. Standards containing 0.5, 1, 3, and 5% by weight chrysotile in talc are prepared as follows:
 - a. Five gm. of the standard talc is slurried in about 100 ml. of ethanol in a Waring Blender.
 - b. The appropriate weight of chrysotile asbestos of at least 95% purity is then added to the talc slurry, and the mixture is blended for 5 minutes at low speed.
 - c. The solution is washed into a beaker with ethanol, and the solvent is evaporated on a steam bath.
 - d. The resulting caked sample is broken and mixed in a plastic vial by shaking for 5 minutes in a mixer such as the Spex Mixer/Mill.
 - e. The homogeneity of the chrysotile-talc standards should be verified by optical or electron microscopy.
4. The experimental conditions, for example, for the Stone DTA unit (Models LA-XYH and RC-202C) are as follows:

High temperature powder sample holder of nickel or stainless steel construction (Model SH-8BE2) with exposed-loop differential thermocouple (Platinel II)

Temperature range: ambient to 1000°C.

Reference thermocouple: Platinel II or Chromel/Alumel

Sample: 130-140 mg. talc, using a loose, consistent packing technique.

Reference material: alumina, ground to pass a 325-mesh sieve (maximum particle size of 44 microns)

Inert dynamic atmosphere: helium or nitrogen at 0.05 SCFH

Heating rate: 10°C./minute

Sensitivity: 40 μ volts full scale

Furnace: LTF, water cooled to 1200°C

The purpose of the inert dynamic atmosphere is to prevent oxidative reactions and to sweep out gaseous mineral decomposition products. The Stone powder sample holder yields maximum sensitivity by permitting direct contact of thermocouple and sample. True dynamic gas flow through the sample is also attained. It may be possible to use a more rapid heating rate with some DTA instruments, if it can be ascertained that constant thermal equilibrium exists between sample holder and furnace. Reproducibility of the method is based on standardizing the experimental conditions.

5. Initially, a thermogram of the blank standard sample is run in order to determine the thermal profile of the talc.
6. A thermogram is run on chrysotile asbestos by diluting the material approximately 1:4 in fine alumina. The thermal transitions involve a dehydroxylation endotherm at about 650°C and a recrystallization exotherm at about 820°C. These two peaks establish the presence of serpentine mineral.
7. The 5, 3, 1, and 0.5% chrysotile in talc standards are then analyzed. Both thermal transitions of the chrysotile in the 1% standard should be easily detectable. If not, the experimental conditions must be altered to improve detectability.
8. Normally, pharmaceutical grade talcs will not require additional grinding for DTA. The analysis of talc rock or the presence of lumps or gritty material will, however, necessitate grinding the sample to pass a 325-mesh sieve.

9. When there is insufficient talc available to fill the entire sample cavity or cup, a lesser amount of the talc may be placed in the bottom of the cup and covered with fine alumina, or packed as a "sandwich" surrounding the thermocouple, between layers of fine alumina. In this case, the standard samples should be packed and run by the same technique to determine the corresponding decrease in peak intensities.
10. If serpentine mineral is detected in a talc sample by DTA, the sample must be subjected to TEM to establish the presence of fibrous chrysotile. If chrysotile serpentine is detected at the same level by the proposed TEM method, then the talc fails.

Addendum

Experimental conditions for the du Pont DTA unit (Models 900 and 990) would be as follows:

Temperature range: ambient to 1000°C.

Furnace: 1200°C.

Sample: about 40 mg. talc

Reference Material: alumina

Atmosphere: static air

Heating rate: 30° C./minute

Sensitivity: ΔT 0.004 mv./in.

T 0.8 mv./in.

DETECTION OF AMPHIBOLE IN TALC BY X-RAY DIFFRACTOMETRY

Cyprus Mineral Company
Pfizer
JOHNSON & JOHNSON

This continuous scanning X-ray diffraction method will detect the presence of amphibole minerals in talc and has a lower limit of detection of approximately 0.2% amphibole, ^{0.2-0.5%} based upon analysis of tremolite standards.

The procedure consists of slow-scanning, under previously determined conditions, a compressed pellet of the sample talc in the $11.0-10.0^\circ 2\theta$ ($8.85-8.04^\circ$) region for the presence of an amphibole peak.

1 - Standard Preparation

Optimal instrument conditions must first be determined with the use of tremolite standards - 1.0%, 0.5%, 0.25%, tremolite by weight prepared in a standard talc which is free of interfering peaks in the $11.0-10.0^\circ 2\theta$ region. The tremolite standards are prepared in the following manner:

a. Weigh out appropriate amounts of standard talc and tremolite of at least 95% purity, both of which have been ground to pass a 325-mesh sieve. Transfer to a Waring Blender (or equivalent). Add 100 ml. of ethanol to the blender and blend at low speed for 5 minutes.

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

c. The dried sample is then shaken for 5 minutes on a Spex Mixer/Mill or equivalent to remove clumps and caked sample resulting from the evaporation of ethanol.

d. The homogeneity of the prepared standard is determined by microscopy before the X-ray diffraction analysis.

e. The homogeneous standard is pressed into a 1½" pellet backed with boric acid. 2 gram of standard is transferred to the die-holder and evenly distributed on a polished, scratch-free die. 4 gram of boric acid is then distributed evenly on the talc layer. The mixture is pressed into a pellet under a pressure of 24,000 lb. (calculated on a 3" ram) for 5-10 minutes. The resulting pellet must have a talc face which is free of flaws; if not the pellet must be discarded. (This requirement is critical since excessive surface scatter will cause excessively high background counts.) Three acceptable pellets are prepared from each standard.

2 - Sample Preparation

Three pellets are prepared from each sample in the manner described for the standard pellets (1. Step e.). A

qualitative scan from $4-50^{\circ}2\theta$ is made 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 $11.0-10.0^{\circ}2\theta$ ($8.85-8.04\text{\AA}$) region of the scan. The presence of the latter will eliminate use of this method for the sample.

3 - 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 a compatible recorder speed and high attenuation with a statistically acceptable time constant on the ratemeter.

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

Radiation:	Cu with K_{β} filter at 40 KV and 24 Ma	
Divergence slit:	1°	Receiving slit: 0.2 mm
Goniometer speed:	1/ $10^{\circ}2\theta$ /minute	
Recorder speed:	300 mm/hr	
Attenuation =	4×10^2 impulses/second	Statistical error of 1.1% under these conditions.
Time constant, T(s) =	10	
Rise Time =	0.18	
Attenuator =	20	

4 - X-Ray Procedure

The pellet is placed in a suitable X-ray holder and slow-scanned from $11.0-10.0^{\circ}2\theta$. All three pellets are scanned in this manner. The presence of a reproducible peak or peaks between 10.75 and $10.10^{\circ}2\theta$ ($8.75-8.25\text{\AA}$) is due to the presence of amphibole mineral(s) in the talc. Identification of an amphibole mineral as tremolite must be performed by optical microscopy/dispersion staining or electron microscopy. A typical scan for a 0.25% tremolite in talc standard sample is included.

