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Minutes of the meeting of the CTFA Task Force on Methodology for the Detection of Asbestos in Talc held at the Johnson & Johnson Research Center, June 21, 1974.

Present were the following:	Affiliation
F. Robert Rolle, Task Force Chairman	Johnson & Johnson
Ian Stewart	McGrone Associates
D. Harris	Cyprus Industrial Minerals
C. S. Thompson	R. T. Vanderbilt
Dave Hamer	Johnson & Johnson
Walter Luckewicz	Avon
John Schelz	Johnson & Johnson
George Sandland	Bristol Myers

1. Mr. David Hamer discussed the most recent version of the FDA proposed optical microscope method for the determination of asbestos fibers in talc. The only difference between this method and the earlier method is that the area of the sample to be scanned is reduced, thereby reducing the time of analysis. The method is still unacceptable, however, for all the reasons given in the December 10, 1973 CTFA report, "Report of CTFA Talc Sub-committee on Method to Detect Chrysotile and Tremolite in Talc".

2. The Rose[1] dye procedure for the detection of chrysotile in talc has been evaluated by Dr. C. S. Thompson, and he finds it non-specific. For example, the serpentine minerals antigorite and lizardite also show adsorption of the dye. He further pointed out that the method is very time consuming. Dr. T. Baak (Cyprus Ind. Minerals) in an earlier communication reported that the mineral palygorskite would act in a manner similar to chrysotile by this method. Mr. W. Luckewicz reported that the particle size of the chrysotile would have a dramatic effect on any attempted quantitation of chrysotile by this analysis.

3. The infrared method proposed by Mr. W. Luckewicz and evaluated by Bristol Myers has been shown to be specific for the detection of tremolite in talc. The level of detectability is about 1% by weight.

4. The "cookbook" transmission electron microscope procedure of Pfizer for the detection of chrysotile in talc was found acceptable, however, Mr. Ian Stewart would prefer to quantify by area count rather than "eyeballing" against a spiked standard.

5. The "cookbook" Differential Thermal Analysis (DTA) procedure of Johnson & Johnson and Avon, for the detection of serpentine in talc, was accepted by the committee. The minimum level of detection is 0.5-1% by weight.

6. The "cookbook" x-ray fluorescence procedure for the determination of tremolite in talc, followed by dispersion staining in the event of a positive reading, was accepted by the committee.

7. The "cookbook" x-ray diffraction method for the detection of amphibole minerals in talc by Cyprus and Pfizer was accepted by the committee. The lower limit of detection was set at 0.2-0.5% by weight.

[1] Rose, H.A., "Detection and Determination of Chrysotile in Talc USP", J. of Pharm. Science 63, 248 (1974).

Mr. George Sandland, Chairman of the CTFA Sub-committee on Asbestos in Talc, reviewed a meeting held on June 12 and 13, between the C.T.F.A. Liaison Committee and the FDA. This meeting was requested by the FDA to review a presentation on cosmetics made to Commissioner Schmidt on May 29, for the industry. The FDA has under review three methods for the detection of asbestos in talc:

1. X-Ray Diffraction for tremolite.
2. Differential Thermal Analysis for chrysotile.
3. Optical microscopy for both tremolite and chrysotile.

It was the opinion of the CTFA Task Force committee that the x-ray fluorescence, electron microscope, and infrared method were all viable methods, but that in order to keep in step with the FDA, as well as to save time in the evaluation of methodology, only the x-ray diffraction and DTA procedures would be put to a round robin test at this time. These "cookbook" methods are attached. Coded round robin talc samples are now available for anyone interested in participating in this test of the proposed methods.

John P. Scheetz

gm

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 μvolts 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°2θ (8.85-8.04Å) 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°2θ 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.

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 μvolts 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.

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 1¼" 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 <u>critical</u> 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°2θ 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°2θ (8.85-8.04Å) 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.

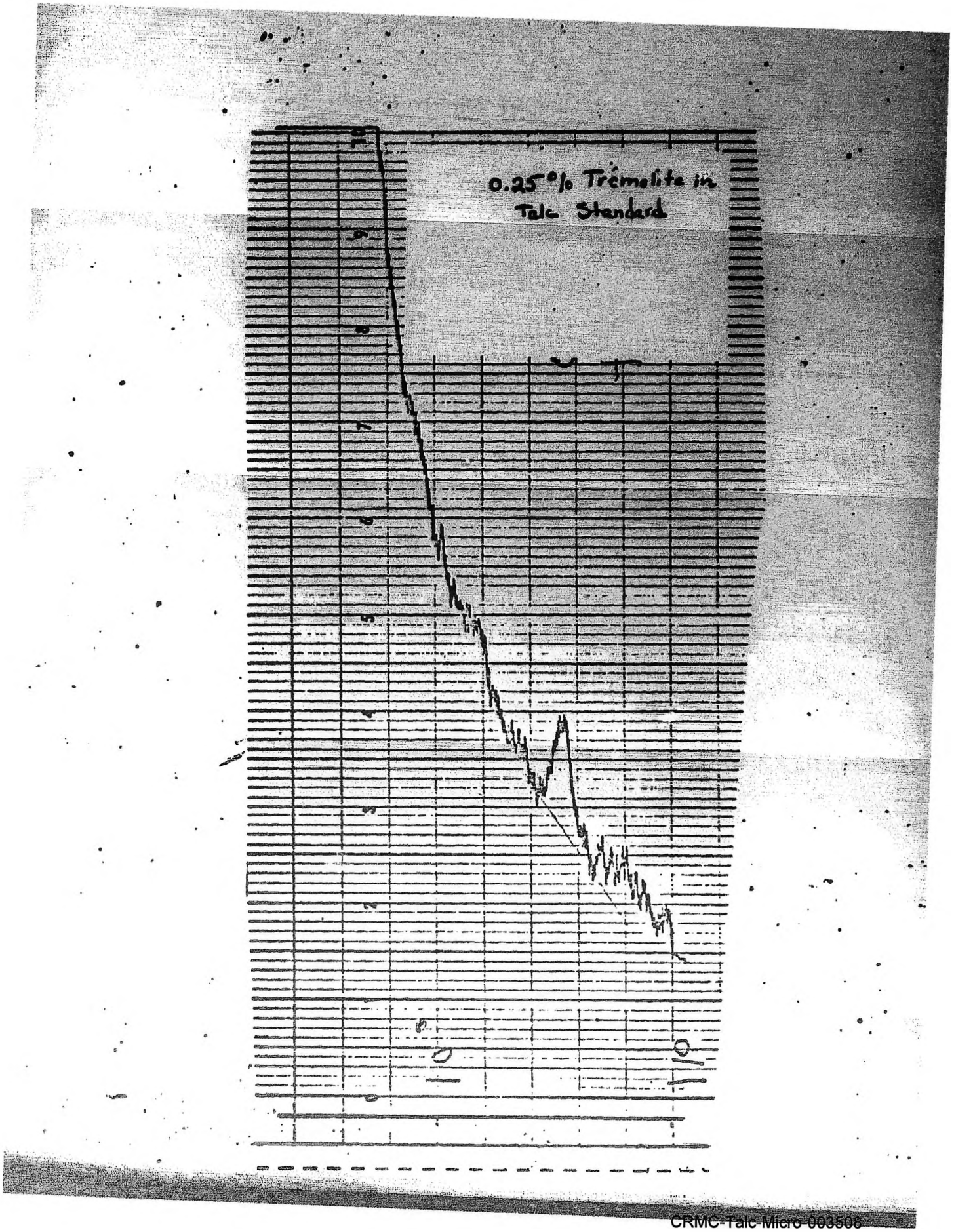
Instrumental conditions employed for the Siemens Diffractometer (Model No. M386-X-A4), and counter and Recorder Unit (Type T) are:

Radiation: Cu with K_B filter at 40 KV and 24 Ma
Divergence slit: 1° Receiving slit: 0.2 mm
Goniometer speed: 1/10°2θ/minute
Recorder speed: 300 mm/hr
Attenuation = 4 x 10^2 impulses/second
Time constant, T(s) = 10
Rise Time = 0.18
Attenuator = 20

Statistical error of 1.1% under these conditions.

4 - X-Ray Procedure

The pellet is placed in a suitable X-ray holder and slow-scanned from 11.0-10.0°2θ. All three pellets are scanned in this manner. The presence of a reproducible peak or peaks between 10.75 and 10.10°2θ (8.75-8.25Å) 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.



Johns-Manville

Internal Correspondence

To: R. S. Preston

Date: July 8, 1974

From: T. M. Jackson

Copies: R. Rees, J. P. Leineweber, DHF, D/C

Subject: Winthrop Laboratories
Rensselaer, New York 12144
Your Letter 6/17/74

The paper "Detection of Chrysotile in Talc" was written by one Harry Rose of Eli Lilly. It reports on a technique of detecting chrysotile by dye adsorption and microscopic inspection. The chrysotile is supposed to selectively adsorb the dye in preference to the talc present.

Our expert in detection of chrysotile in anything is Jim Leineweber of Research and Development. From him, I learn that chrysotile from different sources adsorbs dyes differently--and so do talc from different sources. The article describes work with carefully selected pure chrysotile and pure talcs.

We would not regard the proposed technique as reliable as a standard test for all types of talc or chrysotile.

Detection of chrysotile by X-ray defraction would be more reliable, and is easier to perform if you have the equipment.

Low levels of chrysotile in talc can be measured only microscopically--and an electron microscope is necessary.

If Mr. Ellis wants more detailed information or amplification of this brief comment, I suggest you have him call Leineweber, (303) 770-1000, Ext. 83-553.

T. M. Jackson

TMJ:ccr