Minutes of the Meeting of the CTFA Task Force on Methodology for the Detection of Asbestos in Talc held at the Johnson and Johnson Research Center, Tuesday, February 19, 1974.

Present were the following:

	Affiliation
F. Robert Rolle, Task Force Chairman	Johnson and Johnson
Hal Stanley	Pfizer
William V. Eisenberg	FDA/BF/OS/DM
John W. Stuart	FDA Cosmetics
Walter Luckewicz	Avon Products
David H. Hamer	Johnson and Johnson
John P. Schelz	Johnson and Johnson
John M. Facq	Colgate-Palmolive Co.
C. J. Parmentier	Cyprus Industrial Minerals
Ian M. Stewart	Walter C. McCrone Associates
Trygve Baak	Cyprus Industrial Minerals
J. P. Simko, Jr.	Colgate-Palmolive Co.
D. Harris	Cyprus Minerals Corp.
George Sandland	Bristol Myers Co.
Herb Conrad	Johnson and Johnson

OBJECTIVES

Dr. Rolle outlined the objectives of the task group to be: "development of methods for the detection of fibrous amphibole and fibrous chrysotile in cosmetic grade talc."

The methods must be applicable to finished products and must meet the following criteria:

a) must be specific for detection of asbestos in talc and it, or a follow up method, must discriminate between fibrous and non-fibrous forms of asbestos.

b) must have definable detection limits.

c) must be reproducible among different workers with different instruments.

d) must result in a pass or fail on a talc.

Before proceeding to a detailed discussion of methods submitted by participants, details of talc inhalation studies were reported by Dr. Rolle.

INHALATION STUDIES

This work was done by Dr. F. D. Pooley in England to determine the amount of talc a baby might breathe during powdering. The model system consisted of a doll which was sprinkled with talc to simulate powdering. The amount of airborne talc was measured using an M.R.E. personal air sampler which separates collected dust into respirable and non-respirable fractions. The point was made that 8 minutes of constant sprinkling were required to allow enough talc (microgram amounts) to be collected for measurement. These data were then used to calculate the average number of talc particles a baby could be exposed to during a week. The calculation was outlined in detail and the data were compared with presently accepted industrial exposure limits for asbestos. It was concluded from these tests that even if talc were pure asbestos the levels of exposure of a baby during normal powdering are far below the accepted tolerance limits. The results and details of this experiment are being prepared for submission to the FDA and CTFA.

DEFINITION OF A FIBER

Considerable time was devoted to discussion of alternative definitions of a fiber other than the 3:1 axial ratio as specified in the Federal Register which is felt to be unrealistic. It was decided, however, to stay with this definition because of a lack of medical evidence correlating geometric factors with potential health hazards.

PARTICLE SIZE

Hal Stanley reported that particle size studies of various talc samples submitted by participating CTFA members have been completed and that all are generally similar. These data will be submitted to the FDA together with the inhalation studies reported by J. and J.

ANALYTICAL METHODS:

Optical Microscopy:

A promised method from Johns-Manville is under modification and was not submitted.

Dispersion Staining - [redacted] (b)(4)

Principles of the dispersion staining method were presented by David Hamer. From his experience the method was reported to be suitable for tremolite down to levels of 0.1 percent but was not suitable for chrysotile. The principal reason is the size of chrysotile particles which are generally below the limits of resolution of the method. The minimum dimensions of visible tremolite particles were estimated at 10 µm x 2 µm. One experiment was cited where up to 7% chrysotile in a known spiked sample was missed. It was pointed out that in finished product talcs other added ingredients might interfere with the method. A combination of color and geometric factors must be considered when identifying a tremolite particle by this method.

X-ray Diffraction:

X-ray diffraction procedures for tremolite were submitted by representatives from Pfizer, Johnson and Johnson, and Cyprus. Hal Stanley indicated that Pfizer's technique for tremolite, based on step-scanning of the 110 reflection at 10.56° two-theta, has a detection limit of 0.2%. There is general agreement that this detection limit can be reached by step-scanning procedures, but some disagreement as to the detection limits achievable by normal scanning. Since the 10.56 two-theta peak is characteristic of amphiboles and the method is thus not specific for tremolite, the question of back-up methods was raised. Pfizer and Cyprus have used acid leaching followed by x-ray fluorescence for Ca as a back-up for tremolite. X-ray fluorescence is capable of detecting to limits of 0.1%. The possibility of interferences by Ca-containing substances, particularly in finished products, must be considered. Dispersion staining is also an alternate provided size criteria are met.

Differential Thermal Analysis:

Differential thermal analysis as a method for serpentine minerals in talc was discussed by J. Schelz of Johnson and Johnson. This method was reported to detect 0.5 to 1.0% chrysotile which exhibits two thermal events, an endotherm at 650°C and an exotherm at 830-840°C. The method is specific for minerals of the serpentine class. The method does not depend on morphology and in the case of positive responses must be checked by microscopy. Walter Luckewicz of Avon confirmed J. & J. experience with DTA. Excellent agreement on samples exchanged between the two laboratories was reported. Some question as to the type of instrument used was raised. Avon uses DuPont and J. and J. uses Stone instrumentation. Hal Stanley of Pfizer reported no success using Fisher equipment. Trygve Baak of Cyprus indicated reservations with respect to reproducibility among different instruments and laboratories.

X-Ray Fluorescence

An x-ray fluorescence method capable of detecting 0.1% tremolite based on Ca analysis was reported by Cyprus. Acid leaching is used to remove acid soluble interfering Ca-containing minerals. The question of interferences by Ca compounds, especially in finished products, by flow agents or other additives, was raised. Positive responses must be followed up by another method such as S.E.M., dispersion staining or TEM with SAED. X-ray fluorescence is considered by Cyprus to be easier to quantify than X-ray diffraction. Atomic absorption can be used as an alternative for greater sensitivity.

Electron Microscopy

The Pfizer method for chrysotile in talc utilizing TEM and SAED was presented in detail by Mr. Stanley. Chrysotile is estimated easily at 0.1% by weight. Fiber counts for chrysotile in unknowns are compared with known mixtures similarly prepared. Identification of fibers as chrysotile is made by SAED.

Cyprus techniques are similar but include aluminum shadowing to get a better idea of particle shape and as an internal standard for SAED. Levels of chrysotile are determined by count in comparison with standard.

[redacted] methods were discussed by [redacted] They carry out diffraction studies at potentials of 150-200 kv. utilizing talc itself as internal standard. Quantitation is based on area percent determinations versus standards. The procedure requires several hours to carry out. It was pointed out that at 0.1% chrysotile the fibers are readily apparent in every exposure and can be readily estimated in a matter of minutes. SAED is necessary to identify fibers as chrysotile. Materials with similar morphology thus are eliminated from the count. McCrone's method is sensitive to well below the 0.1% level for chrysotile. The method is suitable in situations where a great number of different talc specimens are to be studied. (b)(4)

It was agreed that the Pfizer method would be used by the task group since it is the most accurately specified. Sample size will be at the operator's discretion but must be consistent. Aluminum shadowing will be included. The test will be pass or fail at the 0.1% level.

Infrared Spectroscopy

An infrared method, proposed by W. Luckewicz of Avon and based on an absorption at 765 cm-1, specific for tremolite, was discussed. Chlorite is an interference and must be eliminated by heating. Although the method was deemed useful for qualitative indication of the presence of tremolite, it was felt that additional study of the quantitative aspects was needed before considering the procedure further.

Summary

The following methods will be used in round-robin studies:

1) X-ray diffraction for amphibole; if positive, to be followed up by SEM or dispersion staining to see if it is fibrous. Two positives and talc fails.

2) Differential thermal analysis for serpentine; if positive, to be followed up by TEM to see if it is chrysotile. Two positives and talc fails.

3) Transmission electron microscopy for chrysotile utilizing a spiked sample at the 0.1% chrysotile level. If chrysotile is at the 0.1% level or higher, talc fails.

4) X-ray fluorescence for calcium in amphibole; if positive, to be followed up by SEM or dispersion staining to see if it is fabrous. Two positives and talc fails.

J. P. Simko, Jr.

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