Johnson & Johnson

Talc Ask.

BABY PRODUCTS COMPANY

RARITAN, N. J. 08869

February 23, 1978

Mr. R. N. Miller, President
Windsor Minerals, Inc.
P. O. Box 680
Windsor, Vermont 05089

Dear Roger:

As you know, Windsor Minerals and the Baby Products Company have already authorized the documentation of a "no detectable asbestos" requirement in the Windsor 66 Talc Material Specification. In this regard, the testing requirement is solely for fibrous amphibole by the CTFA Method J4-1 and is intended to make the specification wholly consistent with the CTFA standard for cosmetic grade talc.

However, we need to recognize that Windsor Minerals and Johnson and Johnson have exercised more extensive controls and testing in the past than just meeting the J4-1 requirement. Furthermore, we intend continuing to surpass the industry testing as reflected by CTFA's J4-1. During the July 15, 1977 meeting in your office, we had agreed to the need of documenting the entire audit protocol which has been your standard operating policy and procedure since August 1973 and will continue to be practised by Windsor Minerals for Windsor 66 Talc. These are the following:

Examination for Non-Detection of Asbestos

Asbestos is defined to be the fibrous serpentine, chrysotile and the fibrous forms of the amphibole group as represented by amosite, anthophyllite, crocidolite, tremolite asbestos and actinolite.

Material will be tested for conformance on an audit basis, frequencies noted according to sample types described and tests required:

Mr. R. N. Miller
Windsor Minerals, Inc.
Windsor, Vermont

Sample Type	Tests	Frequency
Ground Ore	TM 7024	biweekly composite samples by Windsor
Flash Dried Talc	CTFA J4-1 TM 7019	weekly composite samples by J&J

Characteristic	Test	Requirement
Fibrous amphibole forms	CTFA J4-1	none detected
Serpentine forms	TM 7019	none detected
Asbestiform minerals	TM 7024	none detected

Windsor 66 talc shall be manufactured from a previously approved mine site known to contain an acceptable cosmetic grade talc ore which has been tested by and has met the requirements for asbestos according to the test methods and sampling plan described above. Windsor Minerals will assure mine site evaluation data to include analysis of diamond drill core samples and deposit testing of composite ore samples removed from the mine site during the development phase prior to production of cosmetic talc to be used for JOHNSON'S Baby Powder.

Sincerely,

George Lee

/by
Attachment: TM 7019
TM 7024

cc: D. R. Petterson
J. E. Runnells
B. M. Deavenport

Johnson & Johnson BABY PRODUCTS COMPANY

STANDARD TEST METHOD

T.T.

SUBJECT	NO.	REV.	PAGE
DETECTION OF SERPENTINE MINERALS IN WINDSOR 66 TALC BY DIFFERENTIAL THERMAL ANALYSIS	7019		1
	DATE 4/5/77		SUPERSEDES ADL-1297

1. SCOPE & PURPOSE: This method is applicable to the detection of serpentine minerals (including chrysotile asbestos) in Windsor 66 Talc.

2. UNUSUAL SAFETY PRECAUTIONS: None.

3. PRINCIPLE OF METHOD: In differential thermal analysis (DTA), a sample is heated at a constant rate within a portion of the temperature range between -150°C. and 1,000°C. The physical and chemical changes which occur are accompanied by the absorption or release of energy in the form of heat. These transitions and thermally induced reactions are measured against an inert reference material and recorded as thermal peaks.

Serpentine minerals, $Mg_3Si_2O_5(OH)_4$, such as chrysotile asbestos, undergo endothermic dehydroxylation at approximately 650°C. and exothermic decomposition to forsterite, Mg_2SiO_4, at approximately 820°C. Since talc, $Mg_3Si_4O_{10}(OH)_2$, undergoes no first-order transitions in the ambient to 900°C. temperature range, differential thermal analysis may be used to detect the presence of small quantities of serpentine mineral (including chrysotile asbestos) in a talc matrix.

4. FUNDAMENTAL EQUATIONS: $2Mg_3Si_2O_5(OH)_4 \longrightarrow 3Mg_2SiO_4 + SiO_2$ (amorphous) $+ 4H_2O$

5. INTERFERENCES: No minerals commonly found as trace contaminants in cosmetic talc are known to exhibit thermal transitions which would interfere with the detection of serpentine minerals by DTA. However, 10% w/w or more of chlorite $Mg_5Al_2Si_3O_{10}(OH)_8$, a common talc ore accessory mineral, interferes with detection of trace levels of serpentine minerals by this method.

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6. SENSITIVITY: Approximately 0.5% w/w of serpentine mineral in talc may be detected by DTA. This method is not specific as to the variety of serpentine mineral present, i.e., whether it is antigorite, chrysotile or lizardite.

7. ANALYSIS TIME:

	Man-hours (hr.)	Overall time (hr.)
First sample	2.0	2.0
Each additional sample	2.0	2.0

8. APPARATUS:

A. Stone DTA, Model #RC-202 or Model #LA-XYH or equivalent DTA instrument having the required sensitivity.

B. Waring Blender

C. Spex Mixer/Mill

9. REAGENTS:

A. Reference Material ..... Alumina (Al_2O_3), 325 mesh.

B. Standard Chrysotile ..... 325 mesh - International Union against Cancer, P. O. Box 4788, Johannesburg, So. Africa(1)

C. Standard Talc ..... Windsor Talc 66, reference 3553-79, lot #772-432-79 or Windsor Talc 66 of comparable purity.

D. Ethanol

10. INSTRUMENTAL CONDITIONS:

For Stone Model #RL-202 ..... SH-8BE2 type sample holder (stainless steel or nickel).

Pt/Platinel II or comparable thermocouple which may be used up to 900°C.

Heating rate ..... 10°C/min.

Sensitivity ..... 40μ volts F/S.

Temperature range ..... 1000°C.

Atmosphere ..... Static air.

Upper Temp. Limit Switch ..... Approx. 80% of x-axis (corresponding to 850°C.)

(1) Purity of standard must be determined by x-ray diffraction to be 80% or greater.

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11. STANDARDIZATION:

Step 1. Prepare approximately 5 gm. each of 0.5 and 1.0% w/w standards by slurrying the appropriate amounts of standard talc and chrysotile in ethanol in the Waring Blender for 10-15 minutes. Evaporate the ethanol on a steam bath and disperse the resulting caked sample by shaking in a plastic vial in Spex Mixer/Mill.

Step 2. Fill reference cavity of DTA sample holder with alumina and sample cavity with the reference standard talc, tapping lightly several times to insure even sample distribution about the thermocouples.

Step 3. Obtain heating thermogram of Windsor 66 reference talc to 850°C.

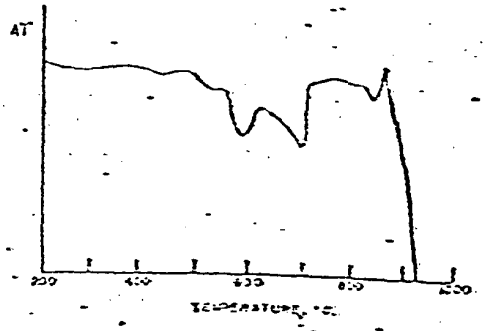

Fig. 1 DTA curve for talc used to prepare chrysotile talc standards

Step 4. Obtain heating thermogram of chrysotile standards to 850°C.

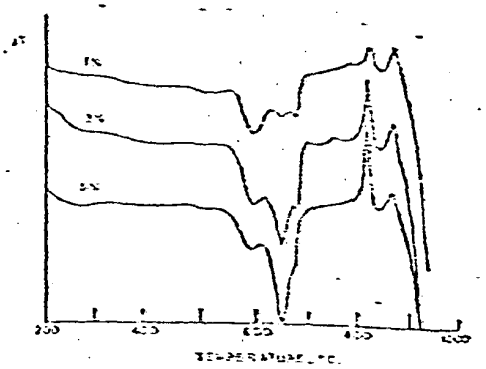

Fig. 2 Typical thermograms of a reference talc spiked with various amounts (%w/w) of chrysotile asbestos

12. SAMPLE:

Step 1. Pack sample cavity with the Windsor 66 talc sample as in Sec. 11, Step

Step 2. Obtain heating thermogram to 850°C.

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13. RESULTS:

The presence of an endothermic peak at approximately 650°C., accompanied by an exothermic peak at approximately 820°C., indicates the presence of serpentine mineral in the talc. In this case, the variety of serpentine mineral must be determined by transmission electron microscopy/selected area electron diffraction, performed by a consulting analytical laboratory. The absence of the two peaks indicates absence of serpentine mineral at the 0.5% w/w minimum detectable level, and the sample is reported as "None Detected".

Johnson & Johnson
BABY PRODUCTS COMPANY

STANDARD TEST METHOD

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DETERMINATION OF ASBESTOS MINERALS IN WINDSOR 66 TALC BY TRANSMISSION ELECTRON MICROSCOPY	7024		1
	DATE 4/29/77		SUPERSEDES ADL-1305

1. SCOPE AND PURPOSE

This method is applicable to the detection and, if necessary, quantitative determination of asbestos minerals in Windsor 66 talc.

2. UNUSUAL SAFETY PRECAUTIONS

Normal safety precautions applicable to the operation of electron microscopes must be observed.

3. PRINCIPLE OF METHOD

The combined techniques of transmission electron microscopy (TEM) and selected area electron diffraction (SAED) permit the detection of asbestiform minerals based on morphological characteristics (TEM), followed by a definitive mineralogical identification of each fiber (SAED). These techniques are currently the ultimate analytical tools for unequivocal determination of asbestos minerals in a talc matrix.

4. FUNDAMENTAL EQUATIONS

None

5. INTERFERENCES

In any TEM field in which fibrous materials are observed, selected area electron diffraction patterns must be taken unless these fibers can be readily identified as rolled talc. Every effort is made to positively identify any fiber present since there are several mineral species which may be associated with talc and which can be fibrous in habit. In finished cosmetic products, organic additives such as perfumes may crystallize out as fibers or needle-shaped crystals. Only those fibers which can be positively identified as asbestos will be counted as an asbestos fiber. In the absence of positive identification, all other fibers must be classified as unidentifiable.

6. SENSITIVITY

This method is capable of detecting a single fiber in the entire TEM field, a negligible level in terms of weight percentage. TEM/SAED is an ideal technique when the asbestos fiber size is $\leq 0.2\mu m$. The shape of fibers down to $0.005\mu m$ is immediately apparent on scanning a sample and the diffraction pattern is immediately discernible.

7. ANALYSIS TIME

	Man-hours (hr.)	Overall Time (hr.)
First Sample	1.0	1.0
Each Additional Sample	1.0	1.0

8. APPARATUS

A. Transmission electron microscope (RCA Model EMU-4 or equivalent).

B. Standard 3-mm. diameter TEM grids, coated with carbon support film.

C. Ultrasonerator

9. REAGENTS

A. Isopropanol

10. INSTRUMENTAL CONDITIONS

The talc specimen grids are examined in the TEM at accelerating voltages of 100-200 kV. Certain fields selected at random are photographed at a magnification in the range of 25,000X - 30,000X with an accelerating voltage of 100 kV.

11. STANDARDIZATION

None required.

12. BACKGROUND CORRECTION

In all talc examinations by TEM, a blank carbon support grid is run to determine whether a significant ambient fiber count is present.

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13. SAMPLE PROCEDURE

A. Ultrasonic dispersion of the talc powder in a carrier liquid is utilized as the sample preparation technique. Approximately 0.1 g of the powder is suspended in 25 ml. of isopropanol. After manual shaking of the suspension, it is held in an ultrasonerator for 10 min. During this time, the mineral flakes, generally those larger than 5-10 μm across, settle toward the bottom of the tube, leaving the finer portion in suspension.

B. Immediately place 1-2 drops of this suspension on a standard 3-mm. diameter TEM carbon support grid which is supported on a glass slide. Prepare several grids for each sample.

C. Transfer the glass slid to a hot plate, where the isopropanol carrier liquid will evaporate.

D. Scan the dried talc specimen grid in the TEM for fibrous particles.

E. Obtain a SAED pattern for any fiber which is detected, in an effort to make positive identification.

14. RESULTS

The fiber content of a talc sample after correction for ambient count, can be expressed in two ways:

A. As a percentage by particle number or

B. As a percentage by particle area which, to a first order approximation, is equivalent to a weight percentage. In general, expressing the content by number will give a high value for the percentage; expressing it by area and inferring a weight percentage are to be preferred as more realistic indications of the asbestor fiber content. In the absence of fibrous, asbestos minerals, the results are reported as "No Asbestiform Minerals Detected".