A Procedure to Examine Talc for the Presence of Chrysotile and Tremolite-Actinolite Fibers

Prepared for

Johnson & Johnson
501 George Street
New Brunswick, New Jersey

By

Colorado School of Mines Research Institute
Golden, Colorado

Project C10704

December 27, 1973

APPROVED:

Herman Ponder
Director

Jerry Krause
Senior Scientist
Mining Division

James M. Link
Director
Mining Division

CONTENTS

	Page
Introduction	1
Objective	2
Summary and Conclusions	3
Discussion	4
Details of the Procedure	4
Samples	4
Separation Details	4
Microscopy	6
Tremolite-Actinolite	7
Chrysotile	8
X-Ray Diffraction	8
Appendix	

INTRODUCTION

The purpose of this document is to report the methods used at the Colorado School of Mines Research Institute for detection of chrysotile and/or tremolite-actinolite in samples predominantly composed of talc. The methods described herein have evolved over a period of time, with the aid of suggestions from many individuals, and are frequently subjected to review.

As the impurity level becomes very low (<<1%), it is necessary to examine increasingly larger amounts of sample in order to detect the impurity. As a result of the requirement to detect the proverbial "needle in a haystack," we have evolved a procedure which preconcentrates the impurities prior to examination. The net effect is that a large initial sample is fractioned in order to reject the majority from further examination.

OBJECTIVE

The objective of this work was to develop a procedure to screen talc for the presence of chrysotile and tremolite-actinolite asbestos minerals. Based on past experience with detecting and identifying minerals when present at low levels, a concentration of the phases to be detected was considered essential to the success of any suggested procedure. Once concentrated the impurities could be detected by conventional methods of examination.

SUMMARY AND CONCLUSIONS

A procedure to detect the presence of chrysotile and/or tremolite-actinolite fibers in talc is presented. The procedure involves two heavy liquid separations to concentrate any chrysotile and tremolite-actinolite which may be present. The heavy liquid concentrates are examined by optical microscopy for the presence of optical size (greater than approximately 2 microns in length) fibers of chrysotile and/or tremolite-actinolite. The procedure is capable of detecting fibers present at a level of approximately 10 ppm or less.

DISCUSSION

DETAILS OF THE PROCEDURE

The optical and physical properties of talc, chrysotile, and tremolite-actinolite important to their separation, concentration, and identification are listed in the table on the following page.

The separation and concentration technique involves heavy liquid separations and is therefore dependent upon specific gravity differences. Identification of the phases thus separated and concentrated is based upon their optical and morphological properties. It is estimated that the following procedure will allow the detection of chrysotile and/or tremolite-actinolite when each is present at a level of approximately 10 ppm or less.

Samples

This method may be applied to a variety of samples ranging from raw ore to final metallurgical concentrates. Raw ore samples should ideally be crushed and sized to -200+325 mesh to liberate talc and other minerals. Metallurgical process samples containing a large proportion of -325 mesh material can be handled in the same manner although the centrifuging and filtering times will be increased.

Separation Details

Five-gram samples are added to each of two 125-ml separatory funnels which contain approximately 75 ml of heavy liquid (2.90 sp gr).[1]

(1) Centigrav; commercially available from American Mini-Chem Co., Corapolis, Penn., 15108.

Relevant Optical and Physical Properties of Talc, Chrysotile, and Tremolite-Actinolite(1)

	Optic Sign	Optic Orientation	Refractive Indices α	β	γ	Specific Gravity	Morphology
Talc	(-)	$Z \wedge a \cong 10°$, $X \cong b$	1.539-1.550	1.589-1.594	1.589-1.600	2.59-2.83	Plate Fiber(2)
Chrysotile	(-)	X = C	1.532-1.549	--	1.545-1.556	~2.55	
Tremolite-Actinolite	(-)	$Z \wedge c$ = 10-21°	1.599-1.688	1.612-1.697	1.622-1.705	3.02-3.44	Fiber

(1) Data from Deer, Howie, and Zussman, Rock Forming Minerals, vol. 2, 1962; vol. 3, 1963.
(2) Fiber -- any material having a form such that it has a minimum length to average maximum width of 3:1.

Each sample is well dispersed by thorough shaking of the loaded stoppered funnels, and then centrifuged at 800 rpm for two intervals of 1/2 hr. The float material is agitated slightly between centrifuge intervals to aid in releasing high specific gravity particles which may be trapped in the tightly packed floating fraction. The heavy and light fractions are collected separately on 0.45μ millipore filters, washed with ethanol or isopropyl alcohol, dried, and carefully weighed. The heavy fraction (sp gr >2.90) will be examined for tremolite-actinolite.

The light fraction (sp gr <2.90) collected above is reprocessed in an identical manner in a liquid of sp gr 2.65. The light fraction with sp gr <2.65 will be examined for chrysotile. The fraction with sp gr >2.65 and <2.90 is assumed to be predominantly talc and therefore is not subjected to further examination. This fraction could of course contain fragments of other minerals locked to the talc.

The 2.65 sp gr liquid is prepared by diluting Certigrav 2.90 sp gr liquid with n, n dimethylformamide having a specific gravity of 0.95. The heavy liquid can be recovered from the alcohol-n, n dimethylformamide washings by extraction with large volumes of water.

The fractions recovered from the heavy liquid separations generally amount to 20 mg or less.

Microscopy

Optical examination of the heavy liquid separates for the presence of fibers is a sensitive examination method. Optical microscopy can detect fibers with a length greater than approximately 2μ, when present at a level

of approximately 0.1% or greater. If optical examination at magnifications up to approximately 625X does not reveal the presence of fibrous particles, the sample can be passed as being clean. If fibrous material is detected optically, then specific identification of the fibers must be attempted. Optical identification is difficult and subject to numerous errors, especially when working with small particles which are near the resolution limit of the microscope. Electron microscopic examination employing selected area electron diffraction and/or x-ray emission spectrography may be required in order to specifically identify small fibrous particles.

The following optical identification schemes require a great deal of expertise, and are subject to errors introduced by small particle size, the presence of talc fibers, plates lying on edge thereby appearing to be fibers, overlap in optical properties, and variable reaction of chrysotile to the iodine stain.

Tremolite-Actinolite

The heavy liquid separate having a sp gr >2.90 is mounted in immersion oil of refractive index 1.600 for transmitted light examination under a petrographic microscope. All amphiboles have refractive indices appreciably greater than 1.600 and will be readily visible. All observations are made at magnifications of 125X and 250X. Single particles are occasionally examined at a magnification of 625X. Tremolite-actinolite fibers are identified by having length to width ratios greater than or equal to 3:1; refractive indices greater than or equal to 1.600; and extinction angle varying between 10° and 21°.

JNJ000268037

Metadata

BegAttach	JNJ 000268034	ORIGINAL
Confidentiality	Y	ORIGINAL
Custodian	Legacy 1	ORIGINAL
DocumentType	Physical	ORIGINAL
EndAttach	JNJ 000268045	ORIGINAL
PgCount	9	ORIGINAL
Text Path	TEXT\0276\JNJ 000268037.txt	ORIGINAL
Trial_Ex_Number	Pltf_JNJ_00040942	ORIGINAL