September 16, 1975

Mr. George W. Sandland
Chairman CTFA Talc Subcommittee
Bristol-Myers Products
225 Long Avenue
Hillside, NJ 07207

Dear Mr. Sandland:

Pursuant to your request, we advise that in August 1971 a test program was instituted by our company to insure customers using our cosmetic grade talcs that they are free of asbestos fibers. It was determined that the use of X-ray diffraction would be the most practical method of detecting the possible presence of asbestos fibers and thus assure us of a reliable and workable means of periodic monitoring our talcs. In the event that the results of this testing showed possible positive results, the sample was further subjected to optical microscopy. In all cases the testing of our products was performed by outside independent laboratories.

Our file contains approximately 275 reports on 20 to 25 grades of cosmetic talc from areas in Alabama, North Carolina, Montana, Italy, South Korea and Vermont. These reports were based onapproximately 57 ground ore samples analyzed over a period of four years. Additionally, separate tests made during this period by many of our customers supported and substantiated our test results.

We feel that the continuation of this program provides the best assurance that our cosmetic customers will receive cosmetic talcs which are free of detectable chrysotile asbestos.

Very truly yours,

WHITTAKER, CLARK & DANIELS, INC.

Frederick F. Roesch
Executive Vice President

FFR:ME


# The Cosmetic, Toiletry and Fragrance Association, Inc.

1133 15th STREET, N.W., WASHINGTON, D.C. 20005 • 202/331-1770 • TELEX 89-2673

James H. Merritt
President

Norman F. Estrin, Ph.D.
Vice President—Science


March 15th, 1976

Mr. Heinz J. Eiermann
Food and Drug Administration
200 C Street, S.W.
Washington, D.C. 20204

On March 11th, 1976, Mr. Eiermann --

-- the CTFA Talc Subcommittee met to discuss the current status of talc. It was recognized that a need exists to bring to your attention a profile of the analyses for asbestos form materials in talc used in the U.S. production of cosmetics and toiletry products.

The attached letters demonstrate responsibility of industry in monitoring its talcs. We are certain that the summary will give you assurance as to the freedom from contamination by asbestos form materials of cosmetic talc products.

Individual companies are more than willing to discuss the detailed data with you at your convenience.

Cordially,

Norman F. Estrin, Ph.D.
Vice President - Science

NFE:jj
Attachments

COSMETIC, TOILETRY and FRAGRANCE ASSOCIATION, INC.
FORMERLY THE TOILET GOODS ASSOCIATION, INC

# News Release

FOR IMMEDIATE RELEASE
March 8, 1976

FOR FURTHER INFORMATION CONTACT: Norman F. Estrin, Ph.D.
Anita Curry
202/331-1770

## ASBESTOS IN TALC

The CTFA has an expert Talc Subcommittee which has been working for the past two years on the detection of asbestos in talc. This Subcommittee has developed reproducible research methods for the detection of fibrous minerals in cosmetic talc. CTFA has performed repeated analyses on the talcs representing more than 85% of the volume of cosmetic talc sold in the United States. Chrysotile Asbestos has not been found in any of these talcs.

CTFA has also developed a specification for cosmetic grade talc and has made its specifications and test methods available to the FDA and the appropriate OTC panels.

Although CTFA has not had the opportunity to review the Mt. Sinai data for methodology, we believe the results of the analysis on asbestos fibers can be misinterpreted because: (1) the dates of the samples are unknown and don't represent currently manufactured products, (2) other raw materials represented in the formulation can confuse the test results, (3) previous Mt. Sinai test results on some United Kingdom formulated talc products have been disputed by well recognized experts in this field.

For these reasons the CTFA critically questions the validity of the Mt. Sinai conclusions on the alleged presence of asbestos like particles.

On the issue of nickel detected in talc, one of our member companies has extensively studied their talc. They have reported that the nickel is permanently bound within the molecular lattice of the talc and therefore cannot be extracted to react with body tissues.

The work by Dr. Wagner on the occurence of fibrosis in rats was only one of several studies on the safety of cosmetic talcs revealed last September. A lifetime inhalation study in hamsters exposed to talc reported no fibrosis. An epidemiological study on talc workers showed no evidence of increased cancer despite high occupational exposure over a 30-year period. All of this information has been presented at world-wide scientific meetings, published in the proceedings and forwarded to the FDA.

The CTFA does not feel that consumer talc products represent any hazard to the consumer.

-30-

The Cosmetic, Toiletry and Fragrance Association, Inc.

1133 15th STREET, N.W., WASHINGTON, D.C. 20005 • 202/331-1770 • TELEX 89-2673

James H. Merritt
*President*

Norman F. Estrin, Ph.D.
*Vice President– Science*

# M I N U T E S

## CTFA TALC TASK FORCE

A meeting of the CTFA Talc Task Force was held on March 11th, 1976 at the CTFA offices in Washington, D.C. Those in attendance were:

George Sandland, Bristol-Myers Company (Chairman)
John E. Clements, Pfizer, Inc.
Ray Krammer, Whittaker, Clark & Daniels
George Lee, Johnson & Johnson Baby Products Company
Monroe Messinger, Chesebrough-Pond's Inc.
Roderick A. Mundy, Sterling Drug Inc.
Louis D. Murino, Cyprus Industrial Minerals Company
D.R. Petterson, Johnson & Johnson Baby Products Company
Fred Roesch, Whittaker, Clark & Daniels
Robert Rolle, Johnson & Johnson Baby Products Company
John P. Schelz, Johnson & Johnson Baby Products Company
Maurice Siegel, Faberge, Inc.
J.C. Simko, Jr., Colgate-Palmolive Company
Harold Stanley, Pfizer, Inc.
Ian Stewart, Walter C. McCrone Associates
Robert Suffis, The Mennen Company
S. Thompson, R.T. Vanderbilt & Company
John J. Travers, Avon Products, Inc.
William C. Waggoner, Johnson & Johnson Baby Products Company
Norman F. Estrin, CTFA

Mr. Sandland opened the meeting and proceeded with the agenda.

1. Status Review

   A. Talc Standards and Methods

      Copies of the proposed specification and methods, plus copies of a letter to Dr. Schaffner were distributed to task force members.

   B. A status report (dated March 8th, 1976), prepared by Dr. Petterson, on cosmetic talc status was distributed to those present. A copy of an article by

CONTINUED ON NEXT PAGE

Dr. Rohl and Dr. Arthur M. Langer on "Identification and Quantitation of Asbestos in Talc" was distributed as it appears in Environmental Health Perspectives, Volume 9, 1974, pp. 95-109. Also, a copy of the correction to the March 8, 1976 Washington Post article on talc and the summary of the draft Talk Paper anticipated from the FDA was distributed to those present.

C. Lewin Samples

Mr. Sandland summarized the contents of the November, 1975 letter from Henry Davis (FDA) requesting CTFA apply its methodology to several of Dr. Lewin samples provided on a coded basis. The results of the analyses were described by Dr. Rolle.

2. Mt. Sinai Analyses on Old Production Samples

Dr. Petterson gave detailed background on Dr. Langer's work. He stated the objective of the task force should be to develop and collate data that would lead to a presentation before the Miscellaneous External Panel, in order to attempt to have talc placed in Category I. The results of Dr. Pooley's analyses, sponsored by Johnson & Johnson, were summarized.

It was reported that Dr. Langer performed his anaylses on samples purchased in 1973. Mr. Simko described a meeting between himself and Dr. Langer on Tuesday. He estimated the sample of "Cashmere Bouquet" tested was probably a pre-1970 sample. Mr. Mundy (Sterling) reported on his contacts with Dr. Langer and stated the sample Dr. Langer tested was from 1970 production.

3. Recent Production Data

It was agreed by the task force that it would be extremely beneficial if induvidual companies could provide data to the FDA on recent production. Most companies present indicated that they possess such data and would be willing to provide the data. Mr. Messinger (Chesebrough-Pond's) stated he would have to confirm this willingness with his management. It was suggested each company prepare a letter, addressed to Dr. Estrin, with a summary of the data on recent production for their products and a commitment to set-up a subsequent meeting with FDA to discuss this data, in detail, if FDA so desires. This letter should be delivered to Dr. Estrin by Monday, March 15th. Subsequently, a meeting was set-up for that Monday, March 15th, at the CTFA offices, with a meeting at FDA to be held in the afternoon.

4. CTFA Study on Current Production

Advantages and disadvantages of having CTFA contract an independent laboratory to do tests on current production samples was discussed. It was noted that such data would no doubt be useful, not only in discussions with FDA, but also as part of a presentation to the Miscellaneous External Panel and, perhaps, to other panels as well. It was suggested the CTFA methodology be utilized for such a task, but that TEM be used on those samples that have been shown to be positive

CONTINUED ON NEXT PAGE

| DCST # | LEWIN # | PRODUCT |
|---|---|---|
| 1338 | 72 | Lady Ester Face Powder, Rachel |
| 1339 | 77 | Touch and Glow Face Powder |
| 1340 | 96 | Blanchard's Dusting Powder |
| 1341 | 97 | Born Wild Dusting Powder, Del Labs |
| 1342 | 101 | Tai Winds Spray Talc, Avon |
| 1343 | 102A | Tosca Dusting Powder |
| 1344 | 143 | Pin-Zow Talque, Perf. Beauty Prod. |
| 1345 | 144B | Overton's "High Brown" Face Powder |
| 1346 | 151 | Early American Old Spice Talcum Powder |
| 1347 | 154 | Bismoline Medicated Powder |
| 1348 | 157A | Mavis Imported Talcum |
| 1349 | 160 | Avon Beauty Dust Refill - Charisma |
| 1350 | 163 | Pinaud Clubman Talc |
| 1351 | 75 | OH! de London Talc, Yardley |
| 1352 | 173A | Corsage Dusting Powder |

| TREMOLITE | | CHRYSOTILE | |
|---|---|---|---|
| LEWIN X-RAY | DCST (OM) | LEWIN X-RAY | DCST |
| ND | 2,800 f/mg. | ND | ND |
| 3 | 5,600 | ND | ? |
| 8 | 13,000 | 10 | - |
| 12 | 60,000 | 15 | ND |
| 15 | 8,900 | ND | ND |
| 10 | 1,800 | 7 | ND |
| 5 | 400 | 10 | - |
| 5 | ND | ? | ND |
| ND | 2,000 | ND | - |
| 2 | 9,000 | ND | - |
| 6 | ND | 8 | ND |
| ND | 9,000 | trace | ND |
| 10 | ? | 10 | - |
| ND | - | ND | ? |
| ND | ND | ? | ? |

by CTFA methodology and also be performed on a random basis on other samples. Company representatives were polled at this meeting and previously by Mr. George Lee to determine whether they would be willing to participate in such a study by assuring that current production samples would be provided and that financial support would be given. The results would be made known to the FDA and probably published in some form. The following companies indicated they would be willing to participate:

Avon Products, Inc.
Bristol-Myers Company
Colgate-Palmolive Company
Coty, Inc.
Faberge, Inc.
Johnson & Johnson
Mennen
Sterling

In addition, Chesebrough-Pond's, Mem, and Yardley would probably be interested.

The task force agreed to await the results of the meetings with CTFA before deciding on whether a study on current production should be initiated.

The meeting adjourned in order for a small group to meet with FDA at 2:00 p.m. to discuss the results of CTFA analyses of Dr. Lewin's samples.

Norman F. Estrin /asc

Norman F. Estrin, Ph.D.
Vice President - Science

NFE:jj
March 12th, 1976

# AVON PRODUCTS, INC.

SUFFERN • NEW YORK 10901

March 12, 1976

Dr. Norman Estrin
CTFA
1133 15th Street, N.W.
Washington, D.C. 20005

Dear Norm:

This is to confirm our indication yesterday of our willingness to disclose to you and/or the Food and Drug Administration the results of our analyses of all lots of talc we have used in our products during the past three years.

During 1973 all of our talc receipts were sampled, and aliquots were sent to McCrone Associates for X-ray diffraction analyses with the specific request for confirmation of the presence or absence of chrysotile. We did not specify other amphiboles at that time because our earlier data indicated that they were present only rarely in the talcs we examined. A total of 170 samples of talcs used in our products were examined by McCrone during that year. The X-ray diffraction patterns are on file at McCrone and can be reviewed by authorized people. The results were negative.

Since February, 1974, we have analyzed about 250 samples of talc receipts in-house by DTA for chrysotile and IR for tremolite. As mentioned above, we felt that IR for tremolite was adequate for screening purposes, because of the rareness of other amphiboles. The data from these analyses can be made available, also, if needed. The results were negative.

Please let us know if there is anything else we can do to help resolve the question of asbestos in cosmetic talc.

Yours truly,

John J. Travers

JJT:mc
cc: C. S. Rowland
G. A. Sprott
R. C. Reardon
Central File

Chesebrough-Pond's Inc.
RESEARCH LABORATORIES
TRUMBULL INDUSTRIAL PARK, TRUMBULL, CONNECTICUT 06611

PHONE: 203 377-7100

March 12, 1976

Dr. Norman Estrin
Cosmetic, Toiletry & Fragrance Association
1133 - 15th Street, N.W.
Washington, D.C. 20005

Dear Norman:

For some years, Chesebrough-Pond's Inc. has been concerned about reports that some talcs might contain asbestos as a naturally-occurring contaminant. As you know, we have cooperated with CTFA, and through CTFA with the FDA in developing suitable and practical methodology to resolve the very complex and difficult task of identifying asbestiform fibers in talc. We have also worked closely with responsible talc suppliers to identify high quality sources of talc.

By the beginning of 1973, we felt that practical methodology for the purpose was applicable. Since then, we have utilized the techniques described in the attachment to monitor incoming cosmetic talcs of three types that we have been using. These three are from three different geographical areas.

I have reviewed the results of these analyses on the incoming lots of these talcs used in recent production of cosmetic and toiletry products in the United States. The last 84 reports, covering a three-year period, are, without exception, negative for chrysotile and negative for fibrous amphiboles.

If it can be useful, we would be pleased to discuss these data with scientists at the Food and Drug Administration.

Sincerely,

Murray Berdick
Director of Regulatory Affairs

MB/bm
encl.

COLGATE-PALMOLIVE COMPANY
909 River Road
Piscataway, N.J. 08854

March 15, 1976

Norman F. Estrin, Ph.D.
Vice President
The Cosmetic, Toiletry and
Fragrance Association, Inc.
1133-15th Street, N.W.
Washington, D.C. 20005

Dear Dr. Estrin:

Attached please find a summary of Analytical Data on 42 Talc Samples (representing 20 raw materials and 17 finished products) covering the period 1971 to present and examined by various techniques by the Colgate-Palmolive Research and Development Analytical Department and McCrone Associates.

Asbestos minerals were not detected in any of these samples, with the exception of insignificant trace amounts found by microscopic methods in three samples from 1971-1972, which is believed due to laboratory contamination. Other methodologies indicate the absence of any asbestos. The results of these analyses indicate to us that talc products produced by the Colgate-Palmolive Company since 1972 are free of asbestos minerals when subjected to the most sophisticated methodology available.

We have ascertained by inspection of the package and analyses of the product that the sample of Colgate-Palmolive's Cashmere Bouquet examined by Dr. Langer of Mt. Sinai Hospital was manufactured prior to 1970. The Bauer and Black Talc reported by Dr. Langer has not been manufactured since the end of World War II. This company was acquired by Colgate-Palmolive approximately three years ago.

Very truly yours,

Christopher H. Costello

Christopher H. Costello, Ph.D.
Administrator Regulatory Affairs

CHC:mp
Attachments

March 12, 1976

To: Dr. C. H. Costello

From: J. P. Simko, Jr.

Subject: Analytical Data on Talc Samples 1972-Present

Since late 1972 when the possibility of asbestos in talc was first raised, R&D Analytical has been engaged in re-analyzing U.S. talc raw material and finished products for asbestos minerals by the most sophisticated methodology available at that time. Analyses were made on a spot-check basis. Techniques used in-house are x-ray diffraction followed by optical and scanning electron microscopy, if necessary. X-ray is the basic screening tool and examination by microscopy is made when positive responses occur. Additional tests on random samples were made by transmission electron microscopy and selected area electron diffraction by an outside consulting firm.

Forty-two samples (from 20 raw materials and from 17 finished products), covering the period 1971 to the present, have been examined by these various techniques. Asbestos minerals were not detected in any of these with the exception of trace amounts in 3 samples from 1971-2. These levels are considered generally below the normal background levels encountered when this microscopic methodology is employed. It is believed that these trace amounts are due to laboratory contamination. A detailed tabulation is attached.

JPS/ed
Att.

## No. of Samples Examined for Asbestos Minerals 1972-76

| Method | Raw Talc | Cashmere Bouquet |
|---|---|---|
| TEM-SAED | *5 | **7 |
| Sample dates (yrs.) | 1971-72 | 1972-76 |
| X-ray plus microscopy (optical - SEM) | 12 | 11 |
| Sample dates (yrs.) | 1971-76 | 1972-76 |
| DTA | 6 | 1 |
| Sample dates (yrs.) | 1972 | 1972 |

*1 sample 1971 raw talc contained 10-100 ppm. chrysotile

*1 sample 1971 raw talc contained 1-10 ppm. chrysotile

These levels represent possible background contamination

**1 sample 1972 Cashmere Bouquet talc contained <5 ppm. chrysotile

No amphibole detected in any of these.

JPS/ed
3-12-76

# The Cosmetic, Toiletry and Fragrance Association, Inc.

1133 15th STREET, N.W., WASHINGTON, D.C. 20005 • 202/331-1770 • TELEX 89-2673

James H. Merritt
President

Norman F. Estrin, Ph.D.
Vice President—Science


COTY / TAC

## MEMORANDUM

TO: Norman F. Estrin, Ph.D.

FROM: Anita S. Curry

DATE: March 12th, 1976

Jim Jenkins called regarding the letter you have asked for on current talc analyses. He wishes to inform you as to what they have been doing:

1. Mr. Clements contacted Dr. Langer who gave him the code on the Coty Airspun that was tested. It turns out that this was a July, 1969 production item.

2. In 1970 Coty changed to a completely different talc; since that time the supplier (WC&D) has supplied letters to the effect that they do continuous monitoring of production, even though individual batches sold to Coty may not be individually tested.

3. Coty has had tests run on intermittent batches at the Pfizer Laboratory in Easton, Pennsylvania. These were done by X-ray diffraction and TEM. The results were all negative. Also, in the last week they have tested 1974 and 1975 lots. These too were negative.

ASC:jj

CTFA WSH
WU WSH
TLXA146 VAF139(1427)(1-018216AB72)PD 03/12/76 1424
TWX CIM123 LSA
20 LOSA CA
PMS THE COSMETIC, TOILETRY AND FRAGARNCE ASSOCIATION, IN.
1133 FIFTEENTH STREET, N. W.
WASHINGTON, D. C. 20005

MARCH 12, 1976
DR. NORMAN ESTRIN
VICE PRESIDENT - SCIENCE

DEAR DR. ESTRIN:

AS A RESULT OF DISCUSSIONS I HAVE HAD WITH LOUIS MURINO CONCERNING THE MARCH 11, 1975 CTFA TALC SUB-COMMITTEE MEETING, I WOULD LIKE TO STATE THAT WE HAVE A CONTINUING PROGRAM IN EXISTENCE TO TEST OUR MONTANA GRADES OF TALCS THAT ARE BEING OFFERED TO THE COSMETIC INDUSTRY.

PRIOR CORRESPONDENCE BETWEEN OURSELVES AND MR. GEORGE W. SANDLAND, CHAIRMAN OF THE TALC SUB-COMMITTEE, HAS ESTABLISHED THAT WE HAVE TESTED 2,839 SAMPLES FROM OUR CORE DRILLING PROGRAM AND BY X-RAY DIFFRACTION PATTERNS HAVE NO SEEN ANY DETECTABLE LEVELS OF CHRYSOTILE AND TREMOLITE. OUR X-RAY DIFFRACTION TECHNIQUES ARE IN ACCORDANCE WITH THE CTFA J-4-1 METHOD OUTLINED FOR USE UNDER CTFA SPECIFICATIONS FOR COSMETIC TALC.

SINCE OUR ORIGINAL INVESTIGATION AND SUBMISSION (AUGUST 8, 1975), WE HAVE EVALUATED AN ADDITIONAL FIFTY MONTANA

PRODUCTION SAMPLES WITH THE SAME EXCELLENT RESULTS. WE CONTINUE TO USE X-RAY DIFFRACTION TECHNIQUES AS OUTLINED BY CTFA J-4-1.

WE SHALL BE HAPPY TO CONTINUE TO COOPERATE WITH CTFA IN OUR TESTING PROGRAM.

VERY TRULY YOURS,

H. T. MULRYAN
PRESIDENT

CYPRUS INDUSTRIAL MINERALS COMPANY
555 SOUTH FLOWER STREET
LOS ANGELES, CALIFORNIA 90071
TELEPHONE (213) 489-3700
END
NNNN

323PEST
CTFA WSH

## TALC INVESTIGATIONS

Our investigations of talc dates back to November 1972, when the following samples were analyzed by McCrone Associates and found to be free of any asbestos content:

| | |
|---|---|
| Music Talc | 9-26-72 |
| Aphrodisia Women's Talc | 9-26-72 |
| Aphrodisia Men's Talc | 9-26-72 |
| Flambeau Talc | 9-26-72 |
| Woodhue Men's Talc | 9-26-72 |
| Straw Hat Talc | 9-26-72 |
| Tigress Talc | 9-26-72 |
| Brut Talc | 9-26-72 |
| Kiku Talc | 9-26-72 |
| Woodhue Women's Talc | 9-26-72 |
| Xanadu Talc | 9-26-72 |
| Zizanie Talc | 9-26-72 |

In November 1975, the following samples were again analyzed by McCrone Associates and found to be free of any asbestos content:

| | |
|---|---|
| Brut Talc | 3.4.75 |
| Brut Talc | 20.6.75 |
| Kiku Talc | 11.11.75 |
| Kiku Talc | 24.4.75 |
| Kiku Talc | 3.7.75 |
| Xanadu Talc | 20.6.75 |

On March 9, 1976, McCrone Assoc. purchased in a Chicago store, one 4 oz. container of Brut Talc, Lot #2585, which was produced on September 15, 1975. This sample was also found to be free of any asbestos content.

Johnson & Johnson

BABY PRODUCTS COMPANY NEW BRUNSWICK, N. J. 08903

March 15, 1976

Dr. Norman F. Estrin
Vice President - Science
Cosmetic, Toiletries, and
Fragrance Association
1133 15th Street, N.W.
Washington, D.C. 20005

Re: Examination for Asbestos In Talc

Dear Dr. Estrin:

The talc which is used by Johnson & Johnson is solely derived from its own mine in Vermont, where the ore is mined selectively and then beneficiated by the process of flotation.

Shipments of this highly purified talc are routinely examined by our own laboratory to verify the absence of asbestos minerals.

Historically, Johnson & Johnson has always been concerned about the mineralogical integrity of its talcs and even its earliest specifications have required the examination for acicular particulates. When it was erroneously reported in 1971 that our powder contained asbestos, we sponsored a scientific seminar of mineralogical and analytical experts for the FDA to prove that our talc is not contaminated with asbestos. At that time numerous samples were analyzed by experienced consultants such as McCrone Associates, Colorado School of Mines, Professor F. D. Pooley (of the University College, Cardiff), Professor G. Brown (of Princeton), and Professor M. Buerger (of M.I.T.). These examinations included X-ray diffraction analysis, differential thermal analysis, and transmission electron microscopy.

During the period December 1972 to October 1973, 93 lots were individually sampled and examined by X-ray diffractometry for the presence of asbestos minerals. No amphiboles or serpentine minerals were detected in any sample.

Beginning in October 1973, differential thermal analysis was added to our specified requirement. Both, differential thermal analysis and X-ray diffractometry were instituted for the routine examination of sequential lots. A further 100 lots have since been examined, applying both methods to each sample. Again, no amphibole or serpentine minerals have been detected.

I would like to advise that we are continuing to employ differential thermal analysis and X-ray diffractometry in the examination of sequential lots for the detection of amphibole and serpentine minerals.

It should be pointed out that we also maintain a worldwide monitoring of talcs used by our Johnson & Johnson affiliates, employing these same techniques.

Finally, let me add that periodically, we also submit sub-samples for Transmission Electron Microscope examination and these have always verified the absence of asbestiform minerals.

It is our understanding that you would wish to submit this information to the FDA. Please regard this letter as non-confidential.

Sincerely yours,

George Lee
Director of Applied Research

GL/mm

Johnson & Johnson

BABY PRODUCTS COMPANY

NEW BRUNSWICK, N. J. 08903

March 14, 1976

# EXAMINATION OF AMERICAN & BRITISH TALCUM POWDER PRODUCTS BY DR. F. D. POOLEY

A total of 38 samples originating from the laboratory of the Department of Environmental Health, Mt. Sinai Hospital were examined by Dr. F. D. Pooley in an effort to substantiate results reported by staff of the Mt. Sinai Hospital.

## American Talcum Products

A total of 20 samples (corresponding to 19 American products) were subjected to examination by X-ray and electron microscopy. Dr. Pooley verified that five products contained several percent, by weight, of fibrous anthophyllite asbestos.

This is in contrast to the Mt. Sinai report that five of the products contained 8 to 20% asbestos and that another five of the products contained less than 5% asbestos.

## British Talcum Products

Similarly, 18 products were examined by Dr. Pooley. He verified one product as containing anthophyllite. On the other hand, the Mt. Sinai laboratory reported finding tremolite in three products and anthophyllite in a fourth product. They further reported that tremolite or anthophyllite were "indicated at or near the lower limit of detectability" in five other samples. It was also reported that "serpentine phase was found in two, and possibly in several other samples," but "electron microscopic examination would be required to resolve the possiblity of the existence of chrysotile." However, Dr. Pooley's subsequent examination with the transmission electron microscope has refuted the existence of any chrysotile.

It should be emphasized that these American products were sampled in 1973 and are not necessarily representative of current American talcum production.

walter c. mccrone associates, inc.
CONSULTING: ULTRAMICROANALYSIS • MICROSCOPY • SMALL PARTICLE PROBLEMS • SOLID-STATE CHEMISTRY

12 March 1976

Dr. Norman Estrin
Cosmetics Toiletries and Fragrances Association
1133 15th Street, N.W.
Washington, D.C. 20005

Dear Dr. Estrin:

This letter will amplify our recent conversation on talc analyses for asbestos.

Since 1971, Walter C. McCrone Associates, Inc., has worked with several producers and users of talc, the majority of them involved with cosmetic grades of talc. During this period we have analyzed several hundred talc samples for the presence of asbestiform minerals. The methodologies used have been principally X-ray diffraction and light microscopy using dispersion staining, although extensive transmission electron microscopy examinations have been carried out for some companies. Subject to the approval of our clients, we are prepared to make available to the FDA, through CTFA, the results of these analyses. We also have available retained samples for most of the analyses.

Verbal agreement to release this data has been already given by clients accounting for probably 80-90% of the samples; the others have not yet been approached. It would, however, be improper for us to comment on specific samples until such time as written approval is received from our clients, together with identification of those materials used in cosmetic production. The latter point is stressed because several of our analyses represent the results of raw material assessments which some companies initiated and might include talc ore bodies which are unacceptable for cosmetic applications.

I do not think, however, that I would be violating any company confidences if I were to comment on the asbestos in talc situation as we saw it over the last 5 years. I think our view can be summarized quite briefly as follows.

2820 SOUTH MICHIGAN AVENUE • CHICAGO, ILLINOIS 60616 • 312/842-7100 • CABLE CHEMICRONE

12 March 1976

Dr. Norman Estrin
Cosmetics Toiletries and Fragrances Association

Page Two

Some cosmetic talcs prior to about mid-1972 to early 1973 were found to contain amphiboles — principally of the Tremolite-Anthophyllite type at levels, determined by X-ray diffraction, of the order of 5-15% by weight.

Microscopical examination of these talcs, however, indicated that the bulk of this amphibole content (>90%) was non-fibrous, even applying the 3:1 aspect ratio criterion for a fiber, and hence not asbestos.

No chrysotile asbestos was found in these talcs.

Since 1973 none of the talcs which we have examined and which have been identified to us as production materials have shown any detectable levels of either chrysotile or asbestiform amphibole.

Yours sincerely,

Ian M. Stewart

Ian M. Stewart
Manager, Electron Optics Group

Companies contacted and granting verbal approval to release information include:

Avon Products, Inc.
Bristol-Myers Products
Chesebrough-Ponds, Inc.
Colgate-Palmolive, Inc.
Faberge, Inc.
Johnson & Johnson
Windsor Minerals, Inc.
Whittaker, Clark & Daniels, Inc.

lmh

STERLING DRUG INC.

90 PARK AVENUE, NEW YORK, N. Y. 10016

EXECUTIVE OFFICES

March 12, 1976

Corporate Director of Quality

Dr. Norman Estrin
Vice President - Science
The Cosmetic, Toiletry & Fragrance Association, Inc.
1133 Fifteenth Street N.W.
Washington, D.C. 20005

Dear Norman:

Sterling Drug Inc. initiated testing in November 1971 of the talc used in "ZBT Baby Powder" for the presence of asbestiform minerals. Since that time we have had tested by x-ray diffraction analysis about one-third of the lots received. In all instances the results indicated there was no detectable asbestiform minerals present in these samples. We also have had assurance from our supplier, Whittaker, Clark & Daniels, Inc., that they routinely monitor the shipments of talc supplied us for the presence of asbestiform minerals and have found no detectable amounts.

We are willing to meet with FDA at some future date to present the detailed results of this testing.

As I told you the code on the package of "ZBT Baby Powder" tested by the Mount Sinai Hospital group was coded and was produced in June 1970. We are of the opinion that the testing we and our suppliers have performed assures us that the "ZBT Baby Powder" produced since late 1971 does not contain asbestiform minerals.

Sincerely,

Roderick A. Mundy

RAM:mm

# The Cosmetic, Toiletry and Fragrance Association, Inc.

1133 15th STREET, N.W., WASHINGTON, D.C. 20005 • 202/331-1770 • TELEX 89-2673

James H. Merritt
President

Norman F. Estrin, Ph.D.
Vice President - Science


## M I N U T E S

## TALC MEETING AT FDA

A meeting was held on March 11th, 1976 in the Commissioner's Conference Room, Food and Drug Administration, 200 C Street, S.W., Washington, D.C., to discuss results of the CTFA analyses of coded samples selected by FDA and those used by Dr. Lewin for his analyses. Those in attendance were:

George Sandland, Bristol-Myers Company (Chairman)
Henry M. Davis, FDA
George Lee, Johnson & Johnson Baby Products Company
Robert Rolle, Johnson & Johnson Baby Products Company
John P. Schelz, Johnson & Johnson Baby Products Company
Ronald L. Yates, FDA
Norman F. Estrin, CTFA

Dr. Estrin opened the meeting by introducing the Chairman of the CTFA Talc Task Force, Mr. George Sandland, and by noting to Mr. Davis that CTFA is planning a meeting on Monday, March 15th to discuss other data collected on talc.

Mr. Sandland noted the November letter from Mr. Davis and the fact that the analyses by Dr. Rolle have now been completed. Dr. Rolle's letter of December 29th, 1975 and the results of the analyses were submitted to Mr. Davis and to others present. Mr. Davis then critically compared the results with those of FDA and Dr. Lewin. Substantial agreement was found with FDA analyses. It was noted the method used was developed for raw materials and not finished products. A copy of the CTFA method was distributed to those present. Dr. Estrin emphasized the work done on these old production samples is valuable from the methodological point of view, but is not valuable if one's goal is to ascertain the present quality of cosmetic talcs.

During the meeting, Mr. Davis noted an article by Gordon Everett entitled, "Analyses for Asbestos in Environmental Samples" (Environmental Health Perspectives, Volume 9, 1974, pp. 181-182).

There being no further business, the meeting adjourned.

Norman F. Estrin, Ph.D.
Vice President - Science

NFE:jj
March 12th, 1976
Attachment

Johnson & Johnson
BABY PRODUCTS COMPANY

For CTFA Talc Meeting
March 11, 1976

March 8, 1976

# COSMETIC TALC STATUS

The safety of consumer talcum products has been under continued attack, primarily led by the Mt. Sinai group, because of three alleged concerns. These allegations are as follows:

1. Commercial talcs often contain asbestos as naturally occurring contaminants.

2. Talc contains other impurities, minerals and metals which may have potential biological significance.

3. Talc as a mineral itself may be hazardous.

## I. FACTS ON TALC

### A. Asbestos and Cosmetic Talcs

- chrysotile asbestos has never been detected in cosmetic talcs or finished talcum products.

- amphibole asbestos, e.g. tremolite and anthophyllite, has been found in a few U. S. consumer talcum products (by Mt. Sinai and confirmed by a very reliable expert consultant).

- disagreement existed between Sinai scientist and an expert consultant in recent analyses for asbestos in British OTC talcum products. This resulted in withdrawal of highly controversial allegations of asbestos in British talcum products intended for presentation at the 4th International Symposium on Inhaled Particles & Vapours.

- Mt. Sinai disclosed statements in February '76 during private interview with the Canadian Broadcasting Corporation that their analysis of U. S. products has found asbestos.

March 8, 1976

B. Biological Testing of Cosmetic Talcs

- extensive animal testing data is only available for Windsor 66 talc and Italian 00000 talc.
- Battelle hamster inhalation testing on Windsor 66 talc with chronic exposures up to 185 times median baby dosage has demonstrated no exposure-related pathology.
- MRC (British Medical Research Council) testing with Italian 00000 talc indicated no tumours resulted from intrapleural inoculation or ingestion.
- MRC testing with Italian talc showed fibrosis at high and very long exposures (up to 1000 times median baby dosage).

C. Human Medical Studies

- Rubino Mortality Study indicated no excess deaths due to cancer amongst talc miners and millers exposed to Italian cosmetic talc.
- Green prospective Pulmonary Study with Windsor talc miners and millers have shown no respiratory disease.
- Kleinfeld has published that deaths were due to cancer of lung and pleura amongst talc workers receiving high exposure to industrial talcs mixed with appreciable amounts of tremolite, anthophyllite and small amount of silica.

D. Conclusions

- all of the data which argues strongly that cosmetic talcs are not a hazard to health under normal-use conditions are based on studies with Windsor 66 and Italian 00000 talcs, both of which have a talc purity of better than 90%, and no asbestos content.
- amphibole asbestos contaminants have been observed in finished talcum products. Tight specifications should be written to preclude sources which are marginal as to potential contaminants.

March 8, 1976

II. FDA/OTC PANEL REVIEWS OF TALC

FDA - LEWIN PRODUCT ANALYSES

- Professor Lewin's mis-identification of chrysotile in store shelf talcum products highlighted to FDA the need to develop analytical methodology for talc.

OTC CONTRACEPTIVES & VAGINAL PRODUCTS PANEL

- After a brief look at the safety of talc, the September 18-19, 1975 meeting decided to defer definitive action on talc to the OTC Antiperspirant Panel.

OTC SEDATIVE PRODUCTS PANEL

- This panel originally proposed that talc, a non-essential ingredient be banned from OTC products that are meant to be ingested. Because of a J & J response, this Panel altered its position to urge that talc containing asbestos not be permitted to be formulated in OTC products meant to be ingested.

OTC ANTIPERSPIRANT PRODUCTS PANEL

- Talc was cleared for use in antiperspirants only on the basis of the low exposure in use. This panel has stated that it has not cleared talc for other uses.

OTC MISCELLANEOUS EXTERNAL DRUG PRODUCTS PANEL

- Because all three of the above Panels have not issued an unequivocal conclusion in the safety of talc, this fourth Panel will be required to provide an ultimate and definitive safety review on talc. With the previous history of attacks in the public media and the erosion of consumer confidence, all means should be taken to obtain a Category I for cosmetic grade talc. The publicity of category III would do irreparable harm to talc's already tarnished image.

PRESENT PROBLEM FOR CTFA & INDUSTRY

- Present allegations from Sinai that they have analyzed and found asbestos in a substantial number of U. S. talcum products is particularly damaging to CTFA's Panel presentation testimony that means are presently being taken by industry to self-regulate.

March 8, 1976

Unless this Sinai data is refuted, it will adversely influence the Miscellaneous External Panel's forthcoming safety review for talc and undermine the tenet that safe cosmetic grade talcs are now being responsibly utilized by the industry.

## III. RECOMMENDATIONS FOR CTFA ACTION

- CTFA should immediately provide refuting data by organizing a test of current talcum products and conduct examination for asbestos by the best method through independent experts. These same product samples should be offered to the FDA and the Mt. Sinai Laboratory for verification of analyses.

- to support an effective and persuasive presentation to the OTC Miscellaneous External Panel, CTFA should issue a strict revised talc standard which would very clearly distinguish cosmetic from industrial grades of talc. Such a specification will be credible to the consumerist advocates only if it fulfills the following requirements:

  1. It must specify the absence of asbestos constituents by the most sensitive method.

  2. It must adhere to a talc purity level which is closely similar to that already used in toxicological studies; namely, at least 90% talc.

- CTFA should identify conforming sources of talc as CTFA Cosmetic Grade Talcs and encourage manufacturers using this grade to label their finished products with this designation.

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WHITTAKER, CLARK & DANIELS, INC.

1000 COOLIDGE STREET · SOUTH PLAINFIELD, N.J. 07080

March 11, 1976

Dr. Norman Estrin
CTFA Headquarters
1133 Fifteenth Street, N.W.
Washington, D.C. 20005

Dear Dr. Estrin:

In August 1971 a test program was instituted by our company to insure customers using our cosmetic grade talcs that they are free of fibrous asbestos. It was determined that the use of X-ray diffraction would be the most practical method of detecting the possible presence of asbestos and thus assure us of a reliable and workable means of periodic monitoring our talcs. In the event that the results of this testing showed possible positive results, the sample was further subjected to optical microscopy. In all cases the testing of our products was performed by outside independent laboratories.

We have also used other test methods such as Differential Thermal Analysis and Optical Microscopy, however X-ray diffraction gave us the best reproducable results.

Our file contains reports on various grades of cosmetic talc from areas in Alabama, North Carolina, Montana, Italy, South Korea and Vermont. These reports were based on approximately 74 ground ore samples analyzed over a period of four years all of which show non-detectable amounts of fibrous asbestos form minerals. Additionally, separate tests made during this period by many of our customers supported and substantiated our test results.

All of the testing for the presence of asbestos form minerals in talc has been performed by "outside experts" including the following:

Walter C. McCrone Associates, Chicago, Illinois
Ernest Fullam Associates, Schenectady, New York
E.S. Laboratories, Marlton, New Jersey
Prof. Charles R. Manning, North Carolina University
Prof. Seymour Z. Lewin, New York University
Dr. Gordon Brown, Princeton University

continued .....

De Bell & Richardson, Enfield, Connecticut
Columbia Scientific Industries, Austin, Texas

We are willing to submit this information to the committee in detail to help substantiate their claims.

Very truly yours,

WHITTAKER, CLARK & DANIELS, INC.

Frederick F. Roesch
Executive Vice President

FFR/dm

The Cosmetic, Toiletry and Fragrance Association, Inc.

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James H. Merritt
*President*

Norman F. Estrin, Ph.D.
*Vice President—Science*

E. Edward Kavanaugh
*Vice President—Congressional Relations*

Margaret W. Smith
*Executive Secretary*

M E M O R A N D U M

TO: The CTFA Talc Subcommittee

FROM: Norman F. Estrin, Ph.D.

DATE: November 2nd, 1976

SUBJECT: CTFA Talc Methodology

McCrone has performed a very useful service by comparing and evaluating CTFA methodology.

Their suggestions with regard to methodology and their other suggestions should be discussed at the November 10th Talc Subcommittee meeting.

NFE:jj
Attachment
cc: Walter C. McCrone

walter c. mccrone associates, inc.
CONSULTING ULTRAMICROANALYSIS • MICROSCOPY • SMALL PARTICLE PROBLEMS • SOLID-STATE CHEMISTRY

3 September 1976

Dr. Norman F. Estrin
CTFA
1133 15th St., N.W.
Washington, DC 2005

Dear Dr. Estrin:

As you know, we, at McCrone Associates, have done considerable work on talc and especially on various mineral impurities in talc. As a result we have accumulated data, procedures (and prejudices) concerning these analyses.

I have recently had occasion to look at the proposed CTFA procedures for asbestiform minerals in talc and can't help feeling that our routine procedures are, I regret to say, considerably more sophisticated. Since I was about to commit our methods to a paper for another publication (unfortunately, for the FDA) I felt that, as an intellectual exercise, I would recast the same material as a CTFA method. I am enclosing this horrendous document for your perusal and sympathize if you choose to quickly burn it at the stake. Note first, however, that I have retained some of the features of your method that I liked.

Still, in our hands, the methods we propose work very well. In addition, they are rapid and reasonably inexpensive. We are interested in improving the competence of microanalysts generally and, as you may know, teach about 40 courses a year in these procedures both here and abroad. As a matter of fact, I leave this weekend where I will be teaching 3 back-to-back courses in the identification of asbestiform minerals by the methods covered in the infamous enclosure.

Seriously, I don't propose you adopt these methods in the detail I've suggested but I do suggest that you urge each CTFA member to take steps now that will lead to their ability to make such analyses. The more each company knows about the products it makes and the raw materials it uses the better able will that company be to stay out of trouble and represent its best interests if trouble arises. We can help here through our teaching arm. I suggest we offer a course either directly through the CTFA or simply to the member companies. This course would train one or more qualified microanalysts in each company in the use of the proposed x-ray diffraction and dispersion staining methods for the study of cosmetic talc and possible contaminating substances.

We would further aid each company in setting up (purchasing, if necessary) the equipment, supplies and mineral standards required for such studies. The entire package would include a one-week intensive course in Chicago, evaluation of the company laboratory and equipment, furnishing sets of

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standard samples and test material and a follow-up over a several-month period of progress in each company by means of a series of round-robin sample analyses. We will sign off each company when we are satisfied their personnel and equipment can do a professional job of analyzing talc samples. The cost of this package would be $1500 per company.

I look forward to your response and hope I haven't scared you away.

Yours Sincerely,

Walter C. McCrone

Walter C. McCrone

WCM:cdb
Enclosure

# COSMETIC TALC

**DEFINITION:** Cosmetic Talc is a white, essentially odorless, fine powder, ground from naturally occurring rock ore, consisting mainly of hydrated magnesium silicate having the ideal formula $Mg_6[Si_8O_{20}] \cdot (OH_4)$ with lesser amounts of naturally associated minerals such as calcite, chlorite, dolomite, kaolin and magnesite, and containing no asbestos minerals detectable by x-ray diffraction.

| TEST | SPECIFICATION | METHOD |
|---|---|---|
| Color..... | As specified by the buyer and showing no change after heating | Heat 1 to 2 g at 200°C for 5 minutes |
| Odor..... | As specified by the buyer | CTFA G 3-1 |
| Identification..... | Positive:<br>1. Close match to CTFA Spectrum with no indication of foreign materials | CTFA G 3-1 |
| | OR | |
| | 2. (Alternate) Close match Powder Diffraction File No. 19-770, published by ASTM, showing the most intense reflections at d values about 9.35, 1.53 and 4.59 Å | ASTM D 934-74 |
| Slip..... | As specified by the buyer | |
| Lustre..... | Do. | |
| Water-Soluble Iron..... | Passes test | USP XIX, page 487 |
| Screen Test..... | 100% through 100 mesh<br>95% minimum through 200 mesh<br>Finer grades: as specified by the buyer | CTFA C 6-1 |
| Water Soluble Substances... | 0.1% maximum | USP XIX, page 487<br>See test for "Reaction and Soluble Substances" |

| | | |
|---|---|---|
| Acid Soluble Substances.... | As specified by the buyer 6.0% maximum | CTFA E 32-1 |
| Loss of Ignition..... | 5.0% maximum | USP XIX, page 487 |
| Arsenic (as As)..... | 3 ppm maximum | CTFA F 1-1, Parts 1-A and 11 |
| Lead (as Pb)..... | 20 ppm maximum | CTFA F 2-1, Parts 1-A and 11 |
| Amphiboles..... (Asbestiform Tremolite et al) | None detected by XRD | CTFA J 4-1 |
| Free Crystalline Silica.... (Quartz) | As specified by the buyer | CTFA J 5-1 (DTA) Alternate: CTFA J 6-1 (X-ray) |

## ACID SOLUBLE SUBSTANCES IN TALC

Principle

A sample of talc is mixed with diluted hydrochloric acid and the insoluble portion is removed by centrifugation and filtration. The filtrate is sulfated, evaporated to dryness and ignited. The acid soluble matter is determined gravimetrically as the sulfate.

Apparatus

1. Controlled temperature water bath
2. Beaker, Griffin, 100 ml
3. Volumetric flask, 50 ml
4. Centrifuge
5. Centrifuge tubes (2), 50 ml
6. Graduated Cylinder, 50 ml
7. Sintered glass funnel, ultra fine porosity, 60 ml (0.9 to 1.4 μm) Corning #36060-UF or equivalent
8. Vacuum flask, 125 ml

9. Pipette, 10 ml

10. Platinum or Vycor evaporating dish or equivalent

Reagents

1. Hydrochloric Acid, diluted, 10% v/v

2. Sulfuric Acid, diluted, 10% v/v

Procedure

Weigh 2 g (±0.001 g) of sample into a 100 ml beaker, add 40 ml diluted hydrochloric acid, and place in water bath at 55° (±2.5°C), with occasional stirring for 30 minutes. Immediately remove beaker from bath and pour contents into a 50 ml centrifuge tube, rinsing beaker and stirring bar with 2 to 3 ml of distilled water. Centrifuge at 5000 RPM for 30 minutes (Note 1).

Filter supernatant through ultrafine porosity sintered glass funnel into vacuum flask, using 2 to 3 ml of distilled water to rinse the centrifuge tube and funnel, and using care to avoid dislodging the packed talc at the bottom of the tube. Filtrate must be clear (Note 1). Pour filtrate into 50 ml volumetric flask, rinsing with 3 to 5 ml distilled water. Adjust to volume with distilled water.

Pipette 25 ml of adjusted filtrate into tared, ignited evaporating dish. Add 2 ml of diluted sulfuric acid and evaporate to dryness.

Ignite at 800° (±25°C) for 1 hour. Cool and weigh.

Calculation

$$\% \text{ Acid Soluble Substances} = \frac{\text{Weight of Residue in g} \times 200}{\text{Weight of Sample in g}}$$

Note

1. Examine the centrifuged sample carefully to assure good clarity before proceeding with filtration. If haziness is detected, centrifuge the sample for an additional 15 to 30 minutes as necessary.

## ASBESTIFORM MINERALS IN COSMETIC TALC

The presence of asbestiform minerals in talc is determined by x-ray diffraction with a sensitivity of about 0.5% by weight. The mineral identification is confirmed and the asbestiform habit is determined by polarized light microscopy with the added help furnished by dispersion staining.

If >0.5% of asbestiform mineral is present the sample is rejected. If the non-asbestiform habits are present, e.g., lizardite, antigorite, grunerite, cummingtonite and riebeckite the sample passes. If <0.5% of the asbestiform habits are present the sample passes. (Note: The polarizing microscope with dispersion staining will detect 0.01% of any mineral with not more than 30 minutes scanning time and if the particles are, at least, 0.2 x 3 μm.)

## PART I — ASBESTIFORM MINERALS BY X-RAY DIFFRACTION

### Principle

The x-ray diffraction method is based upon the principle that when a crystalline material is placed in an x-ray beam, a portion of the x-rays are diffracted by each set of atomic planes within the crystal. The diffracted rays strike a scintillation counter as the sample is scanned through a prescribed angle with the resulting development of peaks corresponding to each interplanar distance (d). A peak with a d value in the range of 8.04 to 8.65 Å for a talc sample is strong evidence for the presence of amphibole. The limit of detection of amphibole by careful application of this method is 0.5%.

### Apparatus

1. X-ray diffractometer, employing nickel-filtered copper K-alpha radiation, horizontal or vertical goniometer with variable scan speed capability, suitable talc pellet sample holder, variable speed recorder, electronic panel including ratemeter, variable attenuation and time constant settings
2. Hydraulic press, capable of attaining a pressure of 15,000 to 24,000 lb applied to a 3" ram
3. Mortar and pestle or grinding mill (Note 1)
4. Waring Blendor, or equivalent blender
5. Spex Mixer/Mill, or equivalent mechanical mixer
6. Sieve, 325-mesh
7. Optical microscope (Note 2)
8. 1-1/4" pellet press

Reagents

1. Standard talc sample, containing no detectable amphibole minerals
2. Standard tremolite sample (at least 95% pure), may be obtained by ordering from The Cosmetic Toiletry and Fragrance Association, Inc., 1133 Fifteenth Street, N.W., Washington, D.C. 20005
3. Denatured ethanol
4. Boric acid

Procedure

The procedure consists of scanning (1°/min) under otherwise high tivity conditions, a compressed pellet of the sample talc over the 2 range from 4° to 54° (about 20 Å to 2.6 Å) with Cu Kα radiation. Check the recorded trace for the peaks tabulated in Table 1.

TABLE 1

Possible Characteristic Strong Peaks in the X-Ray Spectrum of Talc

| 2θ, degrees | d, Å | I | Mineral |
|---|---|---|---|
| 6.31 | 14.0 | 80 | chlorite |
| 9.45 | 9.35 | 100 | talc |
| 9.66 | 9.15 | 20 | cummingtonite (amosite) |
| 10.52 | 8.40 | 100 | crocidolite |
| 10.52 | 8.40 | 100 | hornblende |
| 10.55 | 8.38 | 100 | tremolite, actinolite, anthophyllite |
| 10.61 | 8.33 | 100 | cummingtonite |
| 12.01 | 7.36 | 100 | chrysotile |
| 12.04 | 7.34 | 100 | lizardite |
| 12.20 | 7.25 | 100 | antigorite |
| 12.51 | 7.07 | 8 | talc |
| 18.99 | 4.67 | 8 | talc |
| 19.32 | 4.59 | 45 | talc |
| 19.45 | 4.56 | 25 | talc |
| 19.48 | 4.53 | 12 | talc |
| 20.49 | 4.33 | 6 | talc |
| 20.83 | 4.26 | 35 | quartz |

| | | | |
|---|---|---|---|
| 23.02 | 3.86 | 12 | calcite |
| 24.30 | 3.66 | 100 | chrysotile |
| 24.50 | 3.63 | 60 | lizardite |
| 24.57 | 3.62 | 60 | antigorite |
| 25.25 | 3.541 | 80 | chlorite |
| 25.88 | – | 40 | apatite |
| 26.64 | 3.343 | 100 | quartz |
| 27.26 | 3.268 | 75 | tremolite |
| 27.33 | 3.26 | 30 | hornblende |
| 27.59 | 3.23 | 50 | anthophyllite |
| 28.59 | 3.121 | 100 | tremolite |
| 28.60 | 3.12 | 40 | talc |
| 28.60 | 3.12 | 55 | crocidolite |
| 28.68 | 3.11 | 100 | actinolite |
| 28.77 | 3.10 | 70 | hornblende |
| 29.16 | 3.06 | 100 | anthophyllite, amosite |
| 29.55 | 3.02 | 100 | calcite |
| 30.96 | 2.886 | 100 | dolomite |
| 31.60 | 2.829 | 20 | chlorite |
| 31.76 | 2.814 | 100 | apatite |
| 31.82 | 2.81 | 100 | calcite |
| 32.19 | 2.778 | 60 | apatite |
| 32.46 | 2.756 | 70 | amosite |
| 32.63 | 2.742 | 100 | magnesite |
| 32.85 | 2.726 | 40 | crocidolite |
| 32.90 | 2.720 | 60 | apatite |
| 33.09 | 2.705 | 90 | tremolite |
| 33.15 | 2.70 | 50 | actinolite |
| 33.19 | 2.697 | 20 | hornblende |
| 35.60 | 2.52 | 90 | antigorite |
| 35.85 | 2.503 | 18 | magnesite |
| 36.04 | 2.49 | 100 | lizardite |
| 36.56 | 2.456 | 80 | chrysotile |
| 38.49 | 2.337 | 2 | talc |
| 39.40 | 2.285 | 18 | calcite |
| 41.40 | 2.192 | 30 | dolomite |

| | | | |
|---|---|---|---|
| 41.98 | 2.15 | 60 | lizardite |
| 42.99 | 2.102 | 45 | magnesite |
| 45.08 | 2.095 | 18 | calcite |
| 46.81 | 1.94 | 12 | magnesite |
| 46.05 | 1.892 | 50 | tremolite |
| 48.65 | 1.870 | 4 | talc |
| 50.16 | 1.817 | 17 | quartz |
| 50.55 | 1.804 | 20 | dolomite |
| 51.10 | 1.786 | 30 | dolomite |
| 51.25 | 1.781 | | |
| 53.88 | 1.700 | 35 | magnesite |
| 59.26 | 1.558 | 2 | talc |
| 60.24 | 1.535 | 60 | chrysotile |
| 60.41 | 1.531 | 70 | lizardite |
| 60.50 | 1.529 | 55 | talc |
| 61.80 | 1.500 | 60 | lizardite |

Some minerals, e.g., carbonates, quartz, hornblende etc. have been listed here not because of any known harmful effects but because they may be observed in talc samples.

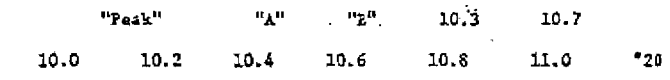
Under ideal conditions 0.5-1.0% of all of the above minerals should be indicated by a possible peak in the 4°-34° range. Appropriate peak regions for the indicated mineral should then be scanned at 0.1°/min as indicated below.

Should the presence of a small peak above the background "noise" be in question, it will be necessary to statistically evaluate the scan made at 0.1°/min in the suspected peak region(s). A timer/scaler is required on the electronic panel of the x-ray diffractometer. In order for a peak to be statistically significant, the peak intensity must equal or exceed three standard deviations ($3\sigma$) above the average background intensity (N):

$N + 3\sigma$ = minimum peak intensity,

N = average background count $\sigma = \sqrt{N}$

Figure 1




Example:

In Figure 1.

| | Region (°2θ) | Time (sec.) |
|---|---|---|
| Peak | 10.40 to 10.60 | 120 |
| Background | | |
| Region A..... | 10.30 to 10.40 | 60 |
| Region B..... | 10.60 to 10.70 | 60 |

| Peak 10.40 to 10.60°2θ | | Background Region A 10.30 to 10.40°2θ | | Region B 10.60 to 10.70°2θ | |
|---|---|---|---|---|---|
| time secs. | count | time secs. | count | time secs. | counts |
| 120 | 60,332 | 60 | 28,784 | 60 | 28,506 |
| 120 | 59,870 | 60 | 28,943 | 60 | 28,368 |
| 120 | 60,105 | 60 | 28,634 | 60 | 28,204 |
| Average | 60,102 | | 28,787 | | 28,359 |

$$N = 28,787 + 28,359 = 57,146$$

$$\sigma = \sqrt{57,146} = 239 \qquad 3\sigma = 717$$

$$N + 3\sigma = \sqrt{57,146} + 717 = 57,863$$

The actual number of counts obtained for the integrated peak intensity was 60,102; therefore, the "suspect" peak is statistically present in the scan."

Standard Preparation

Optimal instrument conditions must first be determined with the use of standards containing 1.0%, 0.75%, 0.5% by weight of the suspected contaminant prepared in a standard talc which is free of interfering peaks.

Weigh out appropriate amounts of the standard talc and the mineral of interest both of which have been ground to pass a 325-mesh sieve. Transfer to a Waring Blendor. Add 100 ml of ethanol to the blender and blend at low speed for 5 minutes.

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

Shake the sample in a plastic vial for 5 minutes on a Spex Mixer/Mill to remove clumps and caked sample resulting from the evaporation of ethanol.

Determine by microscopy the homogeneity of the prepared standard previous to the x-ray diffraction analysis.

Press the homogeneous standard into a 11/4" pellet with a backing of boric acid. Transfer 2 (±0.2) gram of standard to the die-holder and evenly distribute on a polished, scratch-free die. Distribute 4 (±0.2) grams of boric acid evenly on the talc layer. Press the mixture into a pellet under conditions suitable for obtaining a smooth planar surface (for example, a pressure of 15,000 to 24,000 lb calculated on a 3" ram has been found to produce suitable pellets). The resulting pellet must have a talc face which is free of flaws; if not, the pellet must be discarded (Note 3).

Sample Preparation

Prepare two pellets from each sample in the manner described for the standard pellets. Make a qualitative scan from 4 to 34°2θ on one of these pellets to ascertain the presence of non-talc minerals having interfering peaks.

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 compatible recorder speed, and high attenuation combined with a statistically acceptable time constant on the ratemeter. Under appropriate instrumental conditions the peak obtained for the 0.5% standard should be detectable above background noise as shown in Figure 2 for tremolite.

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

Radiation: Cu with Ka filter at 40KV and 24 mA
Divergence slit: 1° Receiving slit: 0.2 mm (.007")
Goniometer speed: 1/10°2θ/minute
Recorder speed: 300 mm/hour
Attenuation: $1 \times 10^3$ impulses/second
Time constant: T (s) = 4

Statistical error of 1.1% under these conditions

Rise Time = 0.18
Attenuator = 20

## PART II: ASBESTIFORM AMPHIBOLE MINERALS BY POLARIZED LIGHT MICROSCOPY AND DISPERSION STAINING

### Apparatus

1. Polarizing microscope. Best results will be obtained if the instrument includes the following:

   a. Individually centering objectives or centerable stage
   b. Bertrand lens
   c. High-intensity light source
   d. Centering condenser/substage

2. Dispersion staining device (Note 4)

3. Vacuum filtration equipment, including either a porcelain cone with glass fiber filter mat or a porous glass bottom cup

### Reagents

2. Cargille immersion liquids: $n_D^{25} = 1.550$ (HD series); (Note 5)

   $n_D^{25} = 1.605$ (HD " )

   $n_D^{25} = 1.660$ (RF " )

   $n_D^{25} = 1.710$ (H " )

### Optical Microscopy and Dispersion Staining

Carefully adjust the dispersion staining objective for optimum viewing of central stop colors. The microscope is first arranged for Köhler illumination: lamp iris (field diaphram) in focus in the field of view; lamp filament in focus in objective back focal plane. The lamp, if movable, should be placed close to the mirror so that the image of the lamp filament is as small as possible in the objective back focal plane. This along with overvolting the lamp by 15-20% and removing the daylite blue filter will ensure bright central stop colors and detection of submicrometer particles.

The stage should next be centered and the central stop should be precisely centered with respect to the image of the substage iris (aperture diaphram) in the objective back focal plane. The substage iris is then closed just behind the central stop and the Bertrand lens (or equivalent viewing system) is removed. The preparation is scanned for $\lambda_0$ colors in the visible; critical study of an area or of a given particle is carried out with the field diaphram nearly closed around the center portion of the field of view.

Identification of non-talc minerals by dispersion staining.

Mount a representative sample (about 0.1 mg) in the following Cargille refractive index liquids and examine carefully for non-talc substances. The particles are mounted in the drop of Cargille liquid between slide and coverslip. The particles are best dispersed by sliding the coverslip with a rotary motion of a pencil eraser "gripping" the coverslip. The final prep should be thin and the coverslip should be parallel to the slide (this avoids a "prism" shaped prep that will decenter the image of the substage iris relative to the central stop). The slide and coverslip must be carefully cleaned before use and the pencil eraser must be dipped in diluted rubber cement and dried before use to avoid transfer of particles to the top of the coverslip. The minerals to be checked for include:

| | | Refractive Indices | | |
|---|---|---|---|---|
| | | α or ε | β | γ or ω |
| Chrysotile | | 1.545 | - | 1.556 |
| Lizardite | | 1.545 | - | 1.556 |
| Antigorite | | 1.564 | ≅1.566 | 1.570 |
| Chlorite | | 1.600 | 1.603 | 1.610 |
| Quartz | | 1.544 | | 1.553 |
| Calcite | | 1.658 | | 1.486 |
| Dolomite | | 1.679 | | 1.500 |
| Magnesite | | 1.700 | | 1.509 |
| Hornblende | | 1.655 | 1.665 | 1.672 |
| Forsterite | | 1.640 | 1.656 | 1.674 |
| Talc | | 1.540 | 1.589 | 1.595 |
| Anthophyllite | | 1.525 | 1.635 | 1.650 |
| Tremolite | | 1.599 | 1.612 | 1.622 |
| Actinolite | | 1.628 | 1.640 | 1.650 |
| Amosite | Cummingtonite | 1.648 | 1.658 | 1.674 |
| | Grunerite | 1.680 | 1.700 | 1.715 |
| Crocidolite | Glaucophane | 1.630 | 1.640 | 1.645 |
| | Riebeckite | 1.695 | 1.704 | 1.710 |

Minerals, especially silicates but also the isomorphous series of carbonates (calcite, dolomite, magnesite etc.) vary in refractive index. Figure 2 a. and b. show average indices for the minerals which might be found in talc. Generally, the dispersion staining curves for these minerals move parallel to themselves as the composition changes. The birefringence values ($\gamma-\beta$, $\beta-\alpha$ and $\varepsilon-\omega$) will generally remain nearly constant for a given mineral although the indices themselves may vary considerably.

The combination of crystal morphology and optics is also a great help in making sure of the identity of a given mineral. One should look for different views of a given mineral and/or vary the view on a single crystal by tapping gently on the top of the coverslip with a needle. In this way fiberlike talc plates on edge can be tipped into the plate view and quartz can be made to show $\varepsilon$ as well as $\omega$.

Both morphology and optics are noted in order to identify the asbestiform minerals. To be asbestiform requires that they be fibers, not slivers, measuring <3 µm in diameter, <30 µm in length and have a length to width ratio of at least 5:1.

Examine the sample for asbestiform or fibrous amphibole minerals

In order for an amphibole mineral to be considered asbestiform or fibrous it must meet the following OSHA definition (Reference 4).

1. Particles must appear to be fibrous rather than as crystals or slivers.
2. The maximum diameter of a fiber to be counted is 3 micrometers.
3. The maximum length of a fiber to be counted 30 micrometers.
4. The length to width ratio must be 5 or more to 1, that is, 5 times or more longer than wide.

5. The separate or individual fibers must contain fibrils or the "bundle of sticks" effect, unless they are at a non-divisible stage. A fibril cannot be subdivided and would be counted, if it meets the other criteria. The length to width ratio of 5 or more to 1 is not meant to imply that other particles are not hazardous.

The analysis proceeds by mounting the sample in successive Cargille liquids with identification of each mineral showing visible $\lambda_0$ colors in each liquid. The first liquid is $n_D = 1.550$

Cargille high dispersion liquid $n_D^{25} = 1.550$:

A representative sample, about 0.1 mg, mounted in this liquid will show characteristic $\lambda_0$ colors for any of the following: talc, quartz, chrysotile, lizardite, antigorite and many fibers (paper, silk, viscose rayon and human hair etc.). The crystallographic data in Figure 2 should ensure identification of most of these substances or allow the conclusion that any other substance showing colors in this liquid is not one of those listed (Figure 3). Characterization of this extraneous substance will often be possible by referring to the Particle Atlas.

Cargille high dispersion liquid $n_D^{25} = 1.605$:

Chrysotile and all other substances giving dispersion staining colors in liquid 1.550 will be white or pale blue in 1.605 (Figure 4). Talc is the only substance in this group that shows a $\lambda_0$ close to the visible (ca 700 nm). All talc plates show both n's ($\beta$ & $\gamma$) close to 1.605 in the red hence the central stop shows a pale blue in all directions lying in the talc plate and white corresponding to the c direction. Other minerals showing colors in $n_D = 1.605$ include tremolite, chlorite and actinolite (Figure 5).

Cargille Liquid $n_D = 1.660$ (HF series)

Non-talc minerals showing dispersion staining colors in $n_D = 1.660$ include actinolite, forsterite, hornblende, calcite, cummingtonite and dolomite (Figure 6).

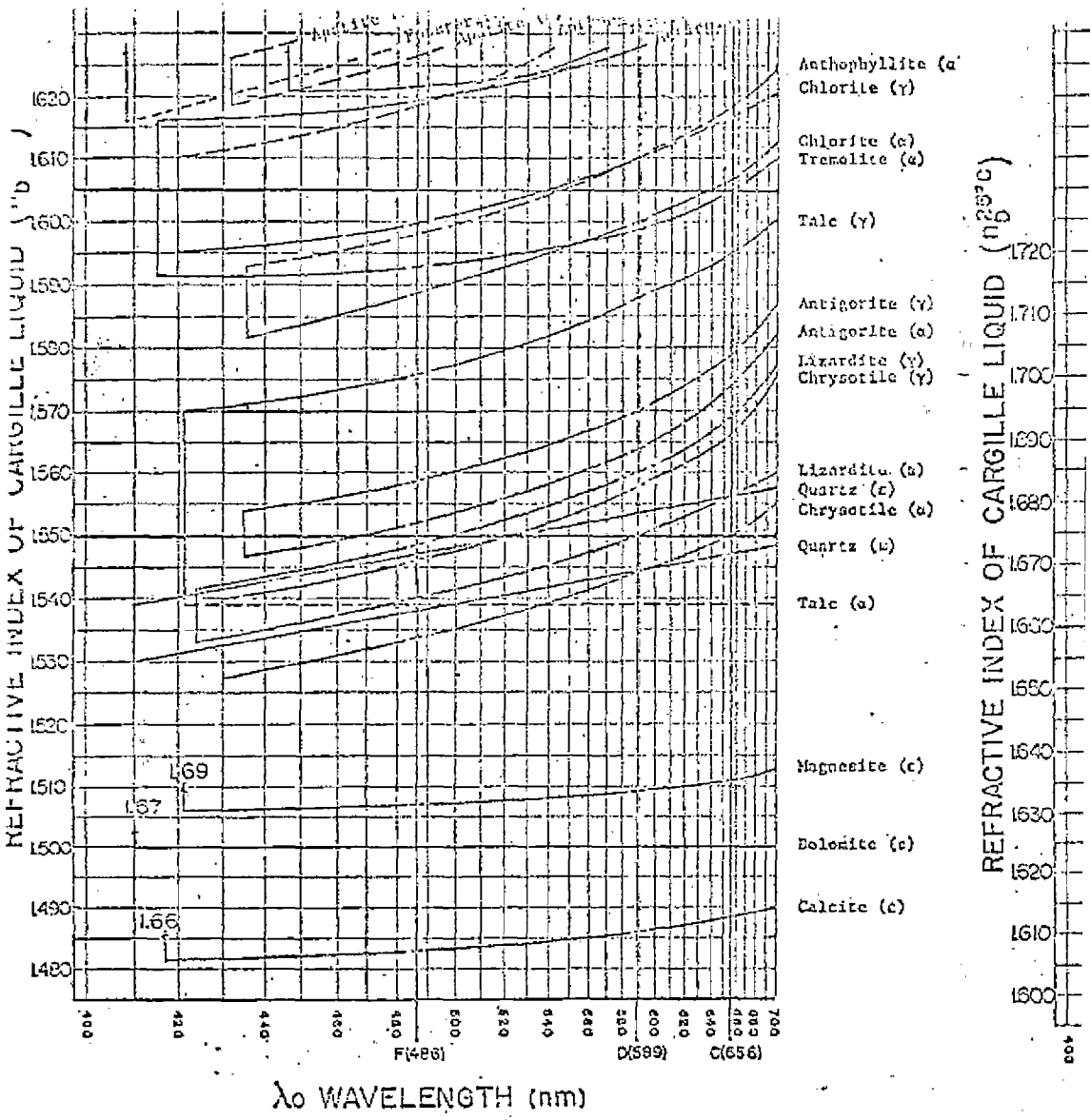



Figure 2a. Dispersion staining curves.

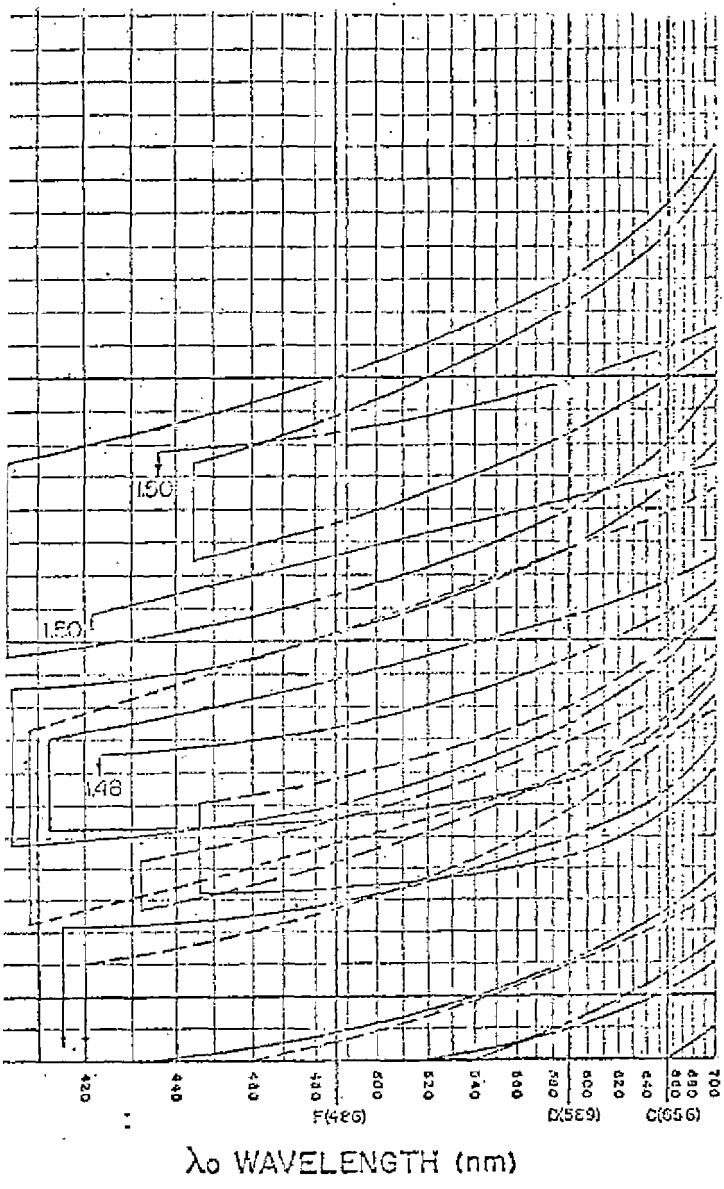
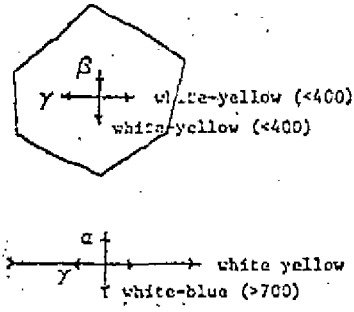



Figure 2b. Dispersion staining curves.

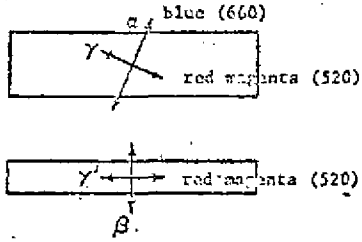
Talc:




Quartz:

conchoidal flakes, all show $\lambda_0$ ca 680 nm corresponding to ω on rotation of the stage; 90° from the ω orientation either ε ($\lambda_0$ = 590 nm) or any ε' (590 <$\lambda_0$ <680).

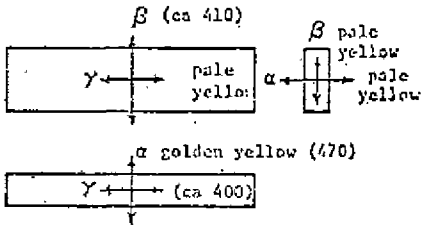
Chrysotile:




Lizardite:

Aggregates of very fine plates, n's slight > chrysotile, β & γ in plane of plates $\lambda_0$ ~600 nm (blue magenta).

Antigorite:




Paper fibers:

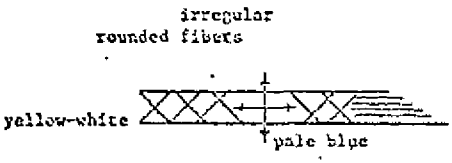



Figure 3. Central stop dispersion staining colors in $n_D$ = 1.550 (HD).

Cargille Liquid $n_D$ = 1.605:

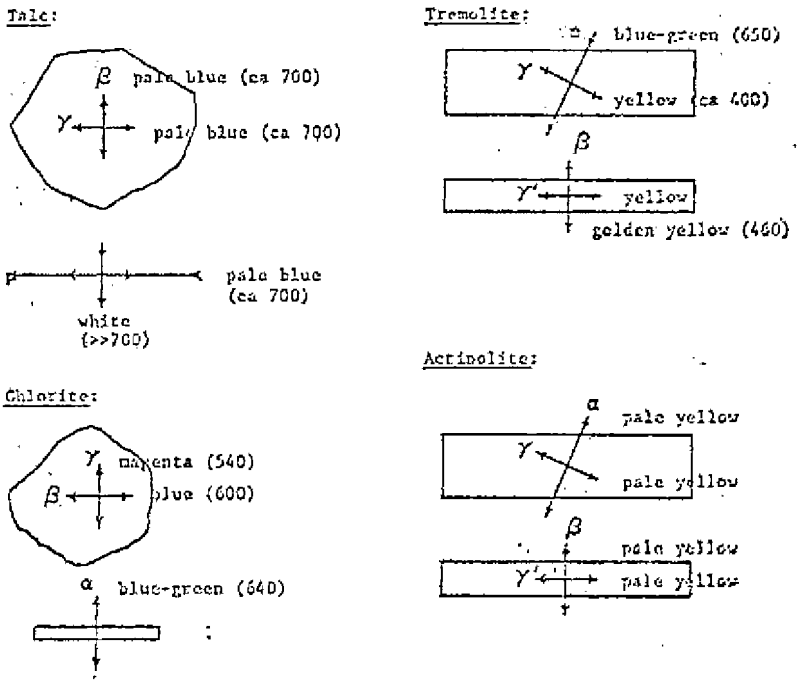



Figure 4. Central stop dispersion staining colors in Cargille Liquid $n_D$ = 1.605 (HD series).

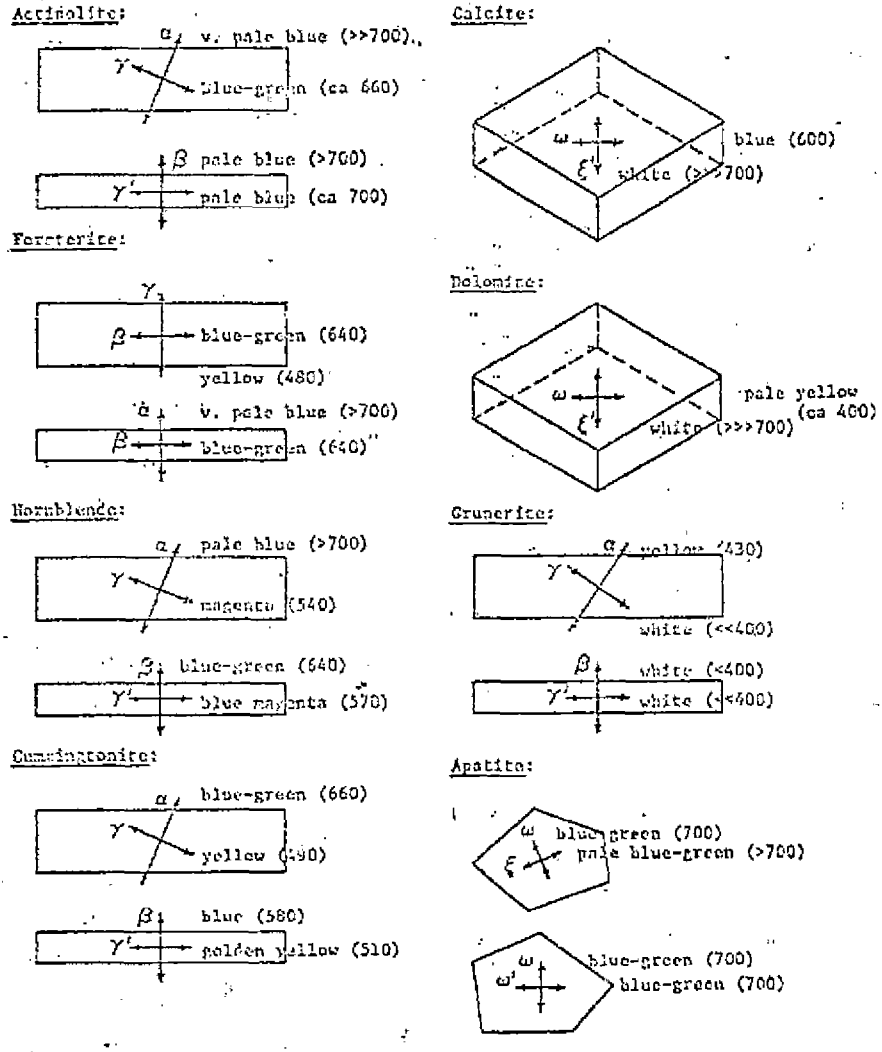
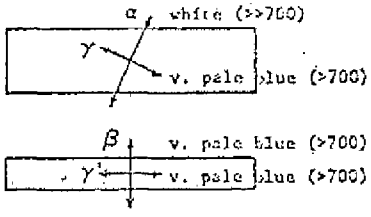


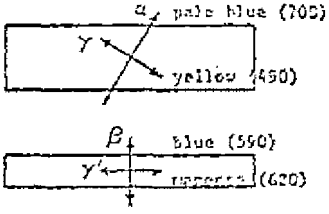

Figure 5. Central stop dispersion staining colors in Cargille liquid $n_D$ 1.660 (RF series).

Cargille liquid $n_D$ = 1.700

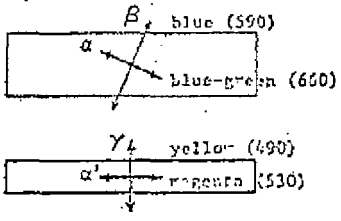
Cummingtonite:




Grunerite:




Crocidolite:




Magnesite:

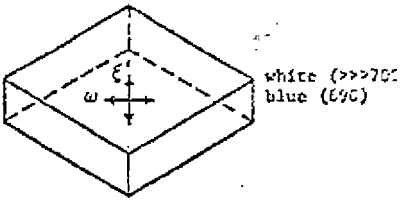



Figure 6. Central stop dispersion staining colors in Cargille liquid $n_D$ = 1.700 (M series).

Cargille Liquid $n_D$ = 1.700 (M series)

Minerals to look for in $n_D$ = 1.700 include cummingtonite, grunerite, crocidolite and magnesite.

Report results as "Asbestiform Amphibole Present" or as "Asbestiform Amphibole Absent."

It is imperative that both dispersion-staining color and fibrous morphology criteria be satisfied before identifying a particle as asbestiform amphibole, since other substances may show colors similar to those described.

Notes

1. Talcs to be analyzed and the standard minerals used to prepare standard samples must be -325 mesh (maximum particle size of 44 micrometers). Tekmar Analytical Mill (Model A-10) is recommended. It is available from:

   Tekmar Company
   P. O. Box 37202
   Cincinnati, OH 45222

2. It is important that the homogeneity of the prepared talc-tremolite standard samples be verified by optical microscopy.

3. This requirement is critical since excessive surface scatter will cause abnormally high background counts.

4. The only commercially available dispersion staining device is sold by:

   Walter C. McCrone Associates, Inc.
   2820 South Michigan Avenue
   Chicago, IL 60616

5. Available from:

   R. P. Cargille Laboratories, Inc.
   Cedar Grove, NJ 07009

   or from laboratory suppliers

## References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2. Rubin, I. B., M. J. Maggiore, Environmental Health Perspectives 9, 81 (1974).

3. L. S. Birks, X-Ray Spectrochemical Analysis, pages 5455, Interscience Publishers (1959).

4. "Tremolite and Talc." U.S. Department of Labor, Occupational Safety and Health Administration, Field Information Memorandum #74-92, November 21, 1974.

5. McCrone, W. C. and J. D. Delly, Particle Atlas, 2nd Edition, Ann Arbor Science Publishers, Inc. (1973).