U.S. Department of Labor

Occupational Safety and Health
Salt Lake Technical Center
Sandy, Utah 84070

Report of Evaluation of Cosmetics and Cosmetic Talc for FDA
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23 February 2019

This report is a response to an Agency Technical Assistance Request dated 23 March 2018 between

The Office of Cosmetics and Colors (OCAC), Center for Food Safety and Applied Nutrition (CFSAN), US Food and Drug Administration (FDA)
and
The Salt Lake Technical Center, Directorate of Technical Support and Emergency Management, Occupational Safety and Health Administration (OSHA), US Department of Labor

This report presents data and evaluation of the materials examined. It does not express any opinions of OSHA regarding any issues within the purview of the requesting Agency.

Ten different materials were submitted to the Salt Lake Technical Center (SLTC) for evaluation for the presence of asbestos. Eight of the samples were commercial cosmetics. Two samples were talc from different sources. SLTC was instructed to hold one of the samples back without analysis. (Claire's Highlighting Pallet, 945287). Appendix A contains photos and chemistry of each sample. Appendix B documents the samples as received.

The samples were examined by light microscopy, scanning electron microscopy with energy dispersive analysis by X-ray, and X-ray diffraction.

The samples were examined by phase contrast with polarized light illumination (PCM-PLM). The test used is standard at the Salt Lake Technical Center for screening talc samples for amphibole asbestos.[1,2] Samples are mounted in index of refraction liquid n = 1.605 and analyzed using central stop dispersion staining (CSDS). Generally, if any fibers are present which have indices of refraction above 1.605, the samples are analyzed in scanning electron microscopy (SEM) with x-ray energy dispersive analysis (EDX). In this investigation, all samples were analyzed by SEM whether or not fibers were present with indices greater than n = 1.605.

Samples were mounted for examination by SEM and analyzed for the presence of fibers. When a fiber was found, a chemical spectrum was obtained using EDX to determine the chemistry of the mineral. Representative fibers were photo-documented. Initially, the samples were not coated

[1] PCM-PLM analysis procedure given in OSHA Method ID-191 and 29 CFR 1910.1001 Appendix J
[2] Screening test outlined in Dixon, W.C., Applications of Optical Microscopy in Analysis of Asbestos and Quartz, Analytical Techniques in Occupational Health Chemistry, edited by D.D. Dollberg and A.W. Verstuyft. Wash. D.C.: American Chemical Society, (ACS Symposium Series 120) 1980. pp. 13-41.

with a conductive metal (gold) in order that the gold not interfere with the chemical evaluation. The samples were coated with gold and high-resolution photos were obtained. The SEM images in Appendix A are of coated samples, except as noted.

Sub-samples of each submitted sample were examined by wide-angle scan using a Cubix Pro Panalytical X-ray Diffractometer (XRD) and the data evaluated using Rigaku PDXL software for best match and also for forced match to actinolite (card 9001922), anthophyllite (card 9016381), chrysotile (card 1010960), grunerite (card 9000000), riebeckite (card 9004132), and tremolite (card 9003673). In addition to software search, a manual visual search for the presence of the highest three tremolite peaks was performed for each of the submitted samples. None of the XRD results was positive for the presence of any of these regulated asbestos minerals.

Table 1 summarizes the results for the 9 analyzed samples.

Appendix C provides definitions. A fiber is an elongate particle, longer than 5 micrometers and at least three times longer than it is wide. Minerals fitting this criterion are known as elongate mineral particles (EMP).

The second column of Table 1 indicates the presence of elongate mineral particles (EMPs) in the sample. The third column of Table 1 indicates if the EMPs have indices greater than n = 1.605. These have the potential to be amphibole asbestos.

Column 1 is the FDA-assigned number and description of material.
Column 2 indicates whether there are EMPs (possible regulatory fibers) present in the sample
Column 3 indicates whether the EMPs have a morphology consistent with asbestos.
Column 4 indicates the presence of fibers in the SEM.
Column 5 indicates if there are fibers with chemistry consistent with a regulated amphibole.
Column 6 indicates whether or not a regulated mineral was detected by XRD
Column 7 indicates the name of a mineral.

Table 1

Sample Number	Fibers Present in PCM-PLM	Possible amphibole fibers present in PCM-PLM	Fibers Present in SEM	SEM + EDX (chemistry) consistent with regulated minerals	XRD	Regulated asbestos name
761227 Eye shadow	Yes	Yes	Yes	Yes (talc fibers also noted)[1]	ND	Tremolite asbestos
761228 Love	Yes	No	Yes	No (hornblende, other?)[2]	ND	
761230 Compact Powder	Yes	Yes	Yes	Yes (other fiber)[3]	ND	Tremolite asbestos
761231 Contour	Yes	Yes	Yes	Yes	ND	Tremolite asbestos
945288 Shadow & Highlight	Yes	Yes	Yes	No (Ti fiber)[4]	ND	
1027488 Just Shine	Yes	Yes	Yes	Yes (other, hornblende?)[5]	ND	Tremolite asbestos
1036658 NVF talc	Yes	Yes	Yes	No (talc)[6]	ND	
1036659 NVF Talc	Yes	Yes	Yes	Yes (talc)[7]	ND	
5063618 Ombre	Yes	No	Yes	No (Unidentified mineral)[8]	ND	

[1] Both tremolite and talc fibers were found
[2] Amphibole not seen. EMP present that may be a hornblende.
[3] Tremolite was found as well as other unidentified EMP
[4] No amphibole found, titanium oxide fibers found.
[5] Tremolite was found as well as possible EMP that may be hornblende
[6] PCM-PLM showed amphibole, SEM-EDX did not.
[7] Talc fibers found
[8] No amphibole found.

All of the samples had fibers present in PLM.

Samples generally have EMP fibers of multiple minerals.

Samples 761227, 761230, 761231, and 1027488 are positive for elongate particles of tremolite. These fibers appear morphologically fibrous, and likely asbestos. No other species of regulated mineral were found.

All of the results by XRD are ND, none detected. The amount of potential regulated fiber in the samples as noted by PCM/PLM and SEM/EDS is very small and likely below the limit of detection for the XRD, which is generally taken as 1% in practice.

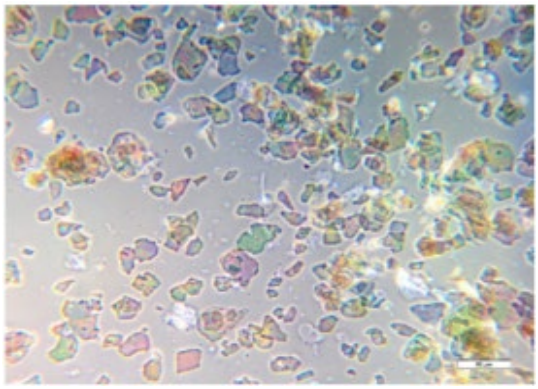
Appendix A contains the photos and chemistry of the samples
Appendix B contains the sample receiving information
Appendix C contains definitions
Appendix D contains preparation and analytical description for PCM and PLM
Appendix E contains preparation and analytical description for SEM and EDS

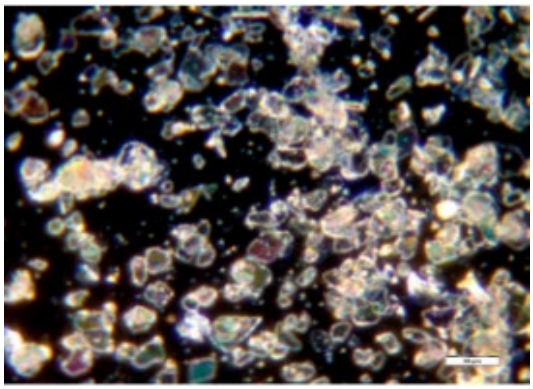
Appendix A

The following are photos and spectra of the samples. Not every type of photo is provided for every sample. Scale bars are included in the light microscopy samples.

761227

1





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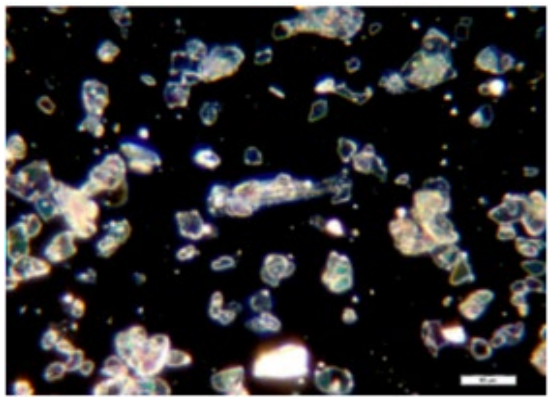

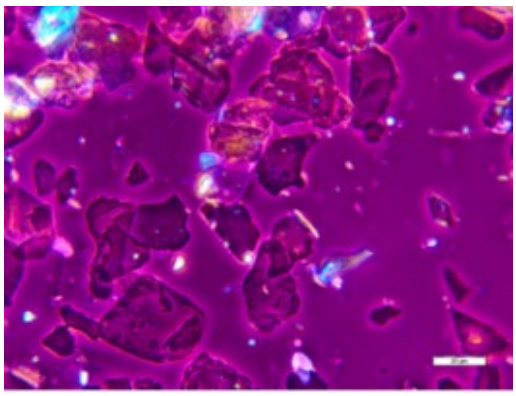

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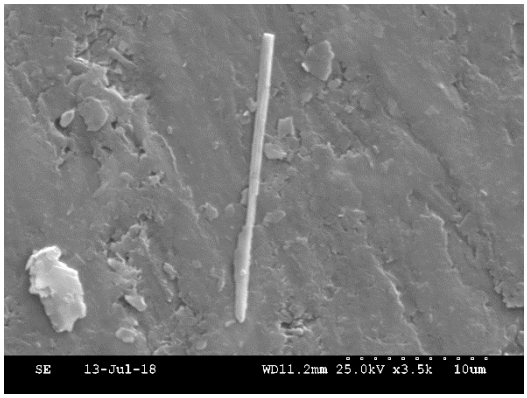
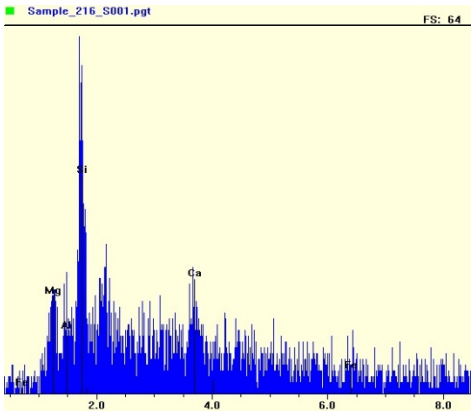




6

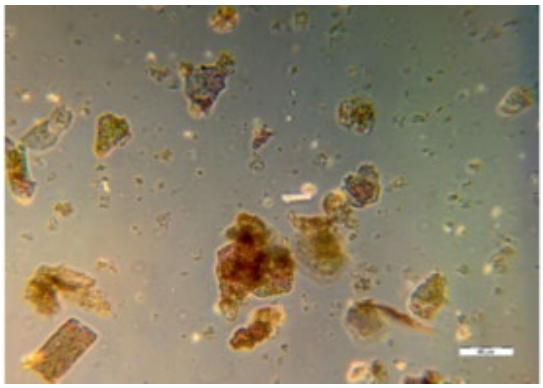
1. Bright Field (PCM) of sample showing a fiber in the center 160X
2. Central Stop Dispersion Stain of the fiber in figure 1. 160X
3. Central Stop Dispersion Stain of a fiber bundle. 160X
4. Crossed polarized light with first order red plate of a fiber in the center 400X
5. SEM of asbestiform tremolite 21 micrometers x 0.7 micrometers
6. EDX spectrum of the fiber in 5, consistent with tremolite.

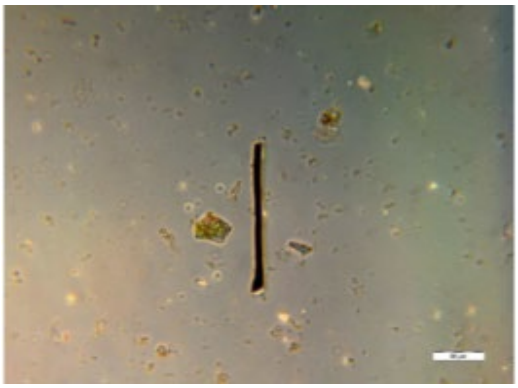
Appendix A

The following are photos and spectra of the samples. Not every type of photo is provided for every sample. Scale bars are included in the light microscopy samples.

761228

1





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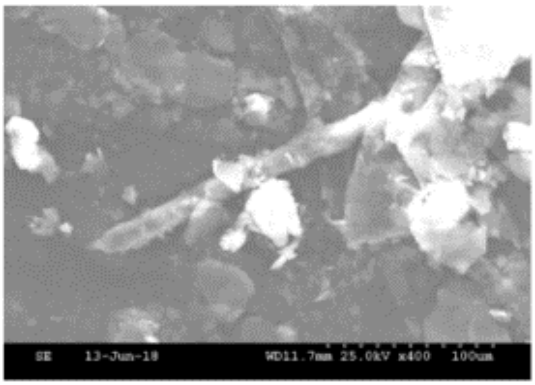

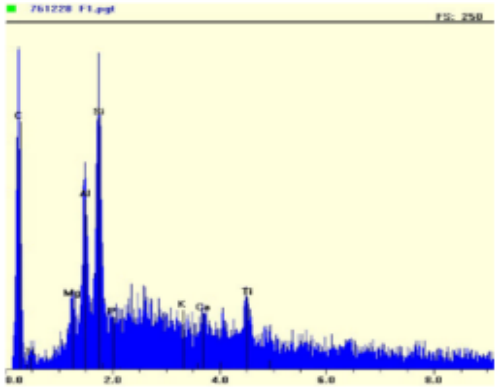

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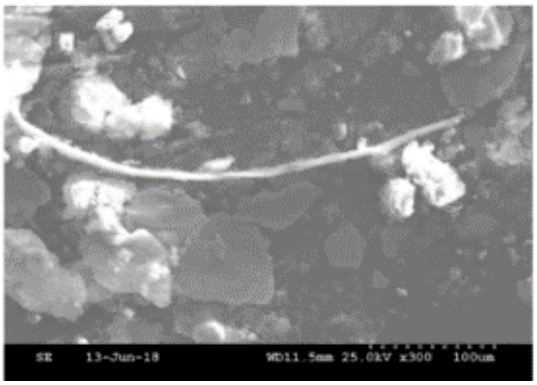
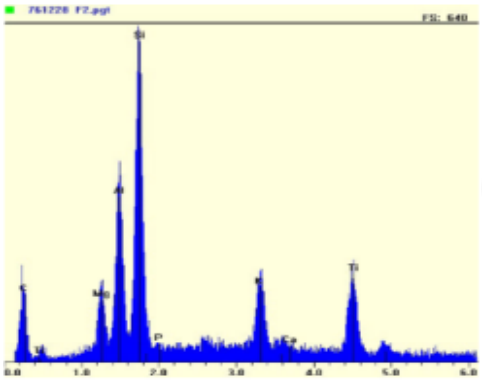




6

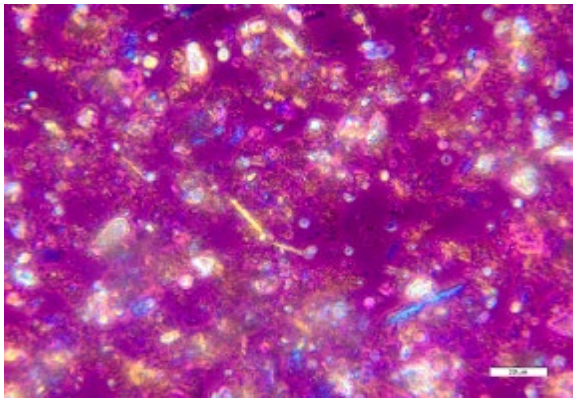
1. Bright Field (PCM) of sample showing a fiber in the center 160X
2. Bright Field (PCM) of sample showing a fiber in the center 160X
3. SEM of non-asbestiform, non-regulated EMP (uncoated with gold)
4. EDX showing Mg, Al, Si, Ti, Ca Not consistent with regulated mineral
5. SEM of non-asbestiform, non-regulated EMP. (uncoated with gold)
6. EDX showing Mg, Al, Si, Ti, Ca Not consistent with regulated mineral. Morphology and chemistry are consistent with organic (plant) fiber.

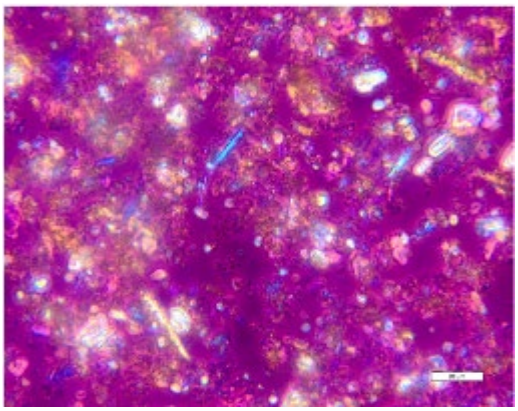
Appendix A

The following are photos and spectra of the samples. Not every type of photo is provided for every sample. Scale bars are included in the light microscopy samples.

761230

1





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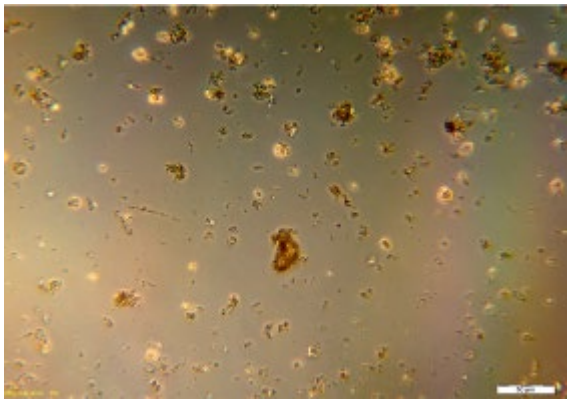

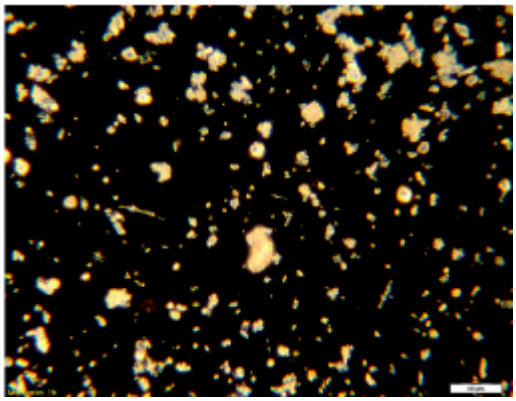

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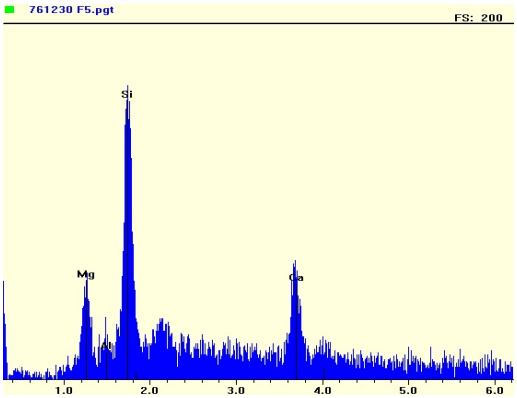


6

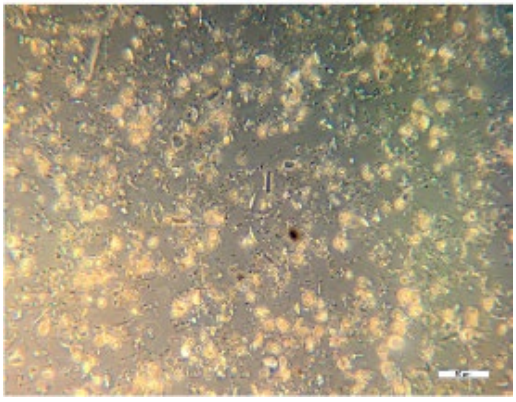
1. PCM-PLM at 400X With several EMPs visible.
2. PCM-PLM with the microscope stage rotated 90º. Note that fibers in photo 1 which appear yellow, appear blue in photo 2 and vice-versa.
3. Bright Field (PCM) 160Xshowing several fibers.
4. Field of Photo 3 in CSDS showing fibers with indices greater than n=1.605 (yellow)
5. SEM photograph of tremolite fiber at 3000X. 23 micrometers x 0.7 micrometers.
6. EDX spectrum of top fiber in Photo 5. It is consistent with tremolite.

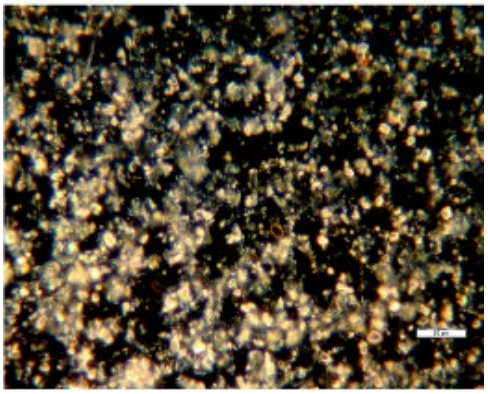
Appendix A

The following are photos and spectra of the samples. Not every type of photo is provided for every sample. Scale bars are included in the light microscopy samples.

761231

1





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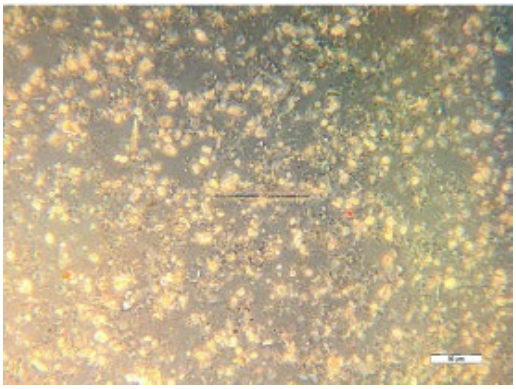
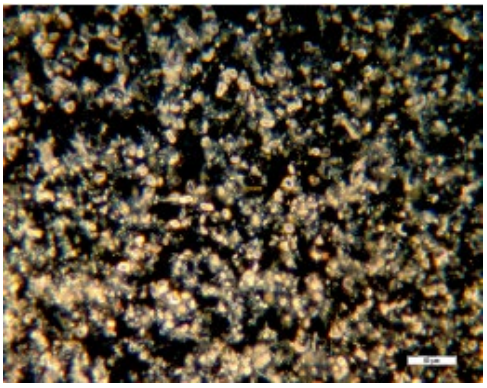
3





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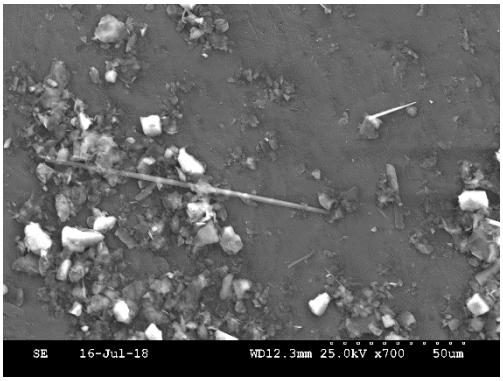
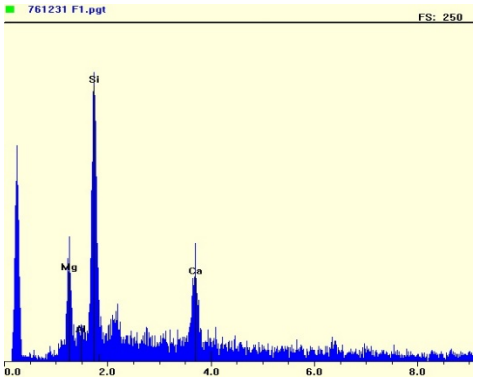




6

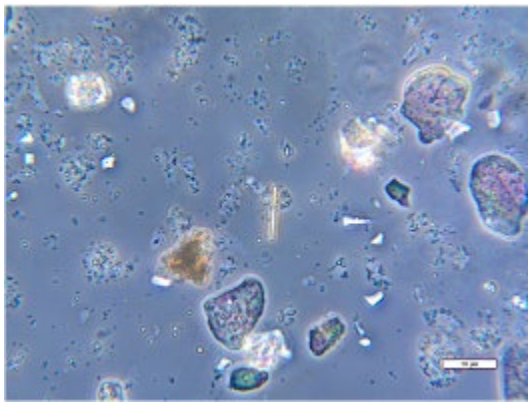
1. Bright Field (PCM) 160Xshowing several fibers
2. Field of Photo 1 in CSDS showing fibers with indices greater than n=1.605 (yellow)
3. Bright Field (PCM) 160Xshowing a long fiber.
4. Field of Photo 3 in CSDS showing fibers with indices greater than n=1.605 (yellow)
5. SEM of asbestiform tremolite. 700X. 109 micrometers x 0.4 micrometers.
6. EDX spectrum of the fiber in 5, consistent with tremolite.

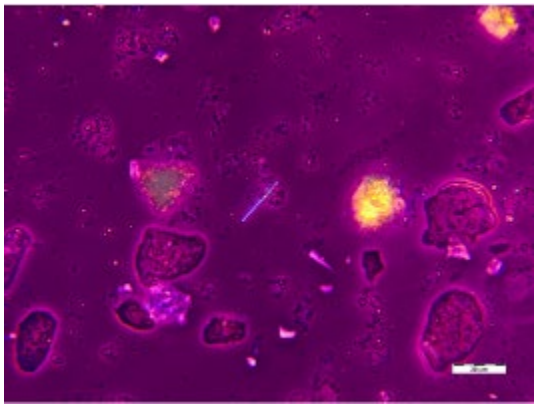
Appendix A

The following are photos and spectra of the samples. Not every type of photo is provided for every sample. Scale bars are included in the light microscopy samples.

945288

1





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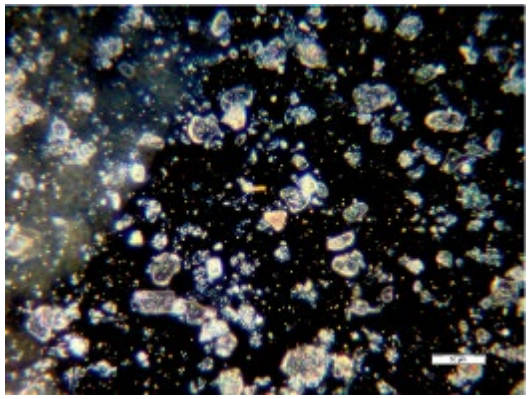

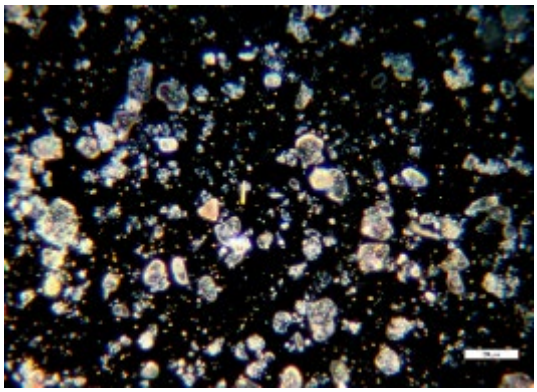

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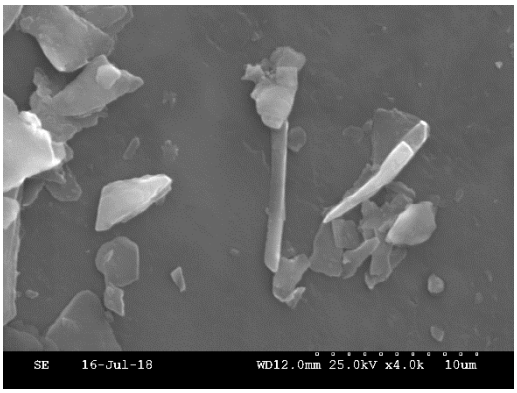
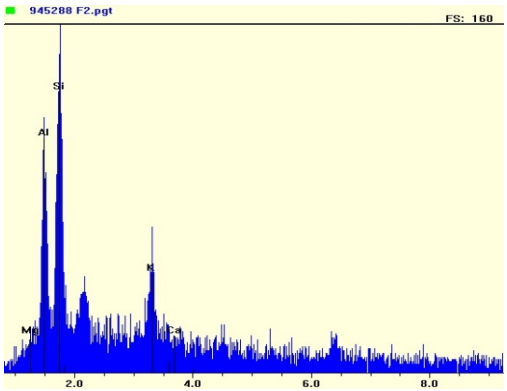




6

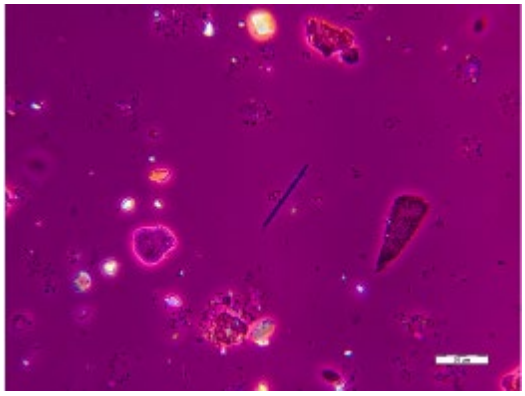
1. Bright Field (PCM) 160Xshowing several fibers 400X
2. PCM-PLM of fiber in Photo 1 160X
3. CSDS of fiber in Photo 1 showing index greater than n=1.605
4. CSDS of fiber rotated 90º to Photo 1 showing index greater than n=1.605
5. SEM of typical fiber
6. EDX of the long fiber in Photo 5 showing K with Al and Si. Not a regulated mineral.

Appendix A

The following are photos and spectra of the samples. Not every type of photo is provided for every sample. Scale bars are included in the light microscopy samples.

1027488

1



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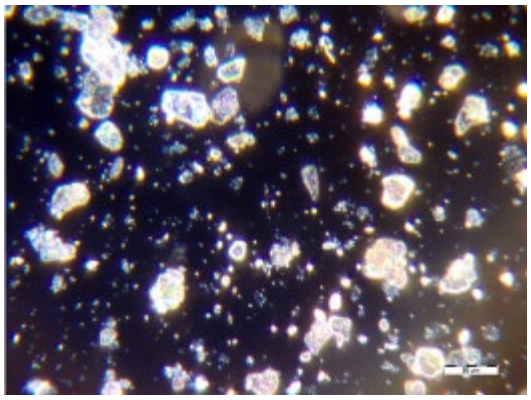

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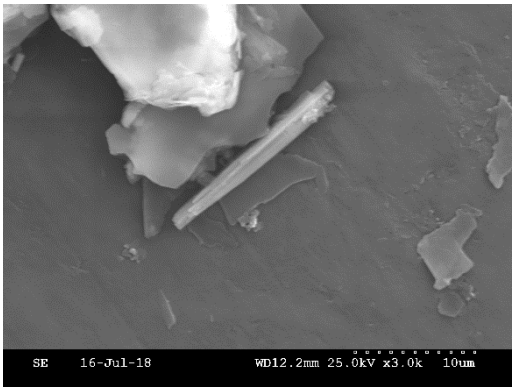

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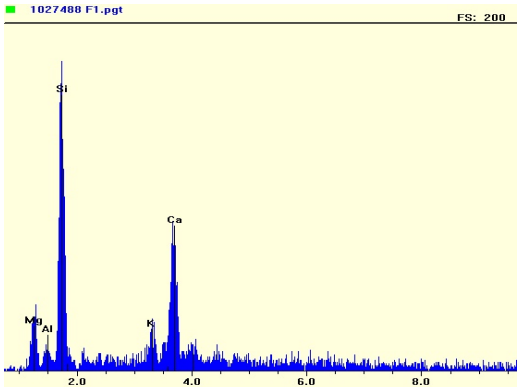

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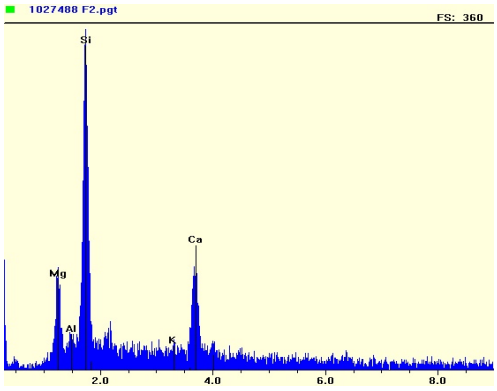
6



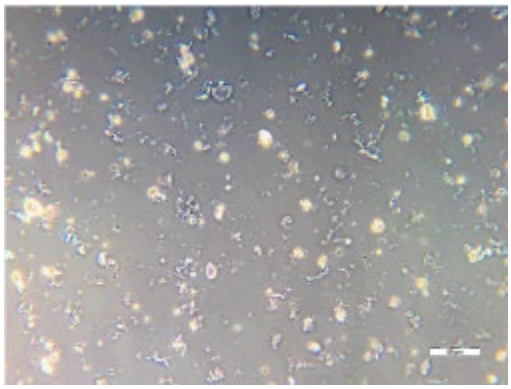
1. PCM-PLM of EMP at 400X
2. CSDS of fiber in Photo 1 showing index of refraction greater than n=1.605
3. SEM of tremolite fiber3000X 17.3 micrometers x 1.8 micrometers.
4. EDX of the tremolite fiber in Photo 3
5. SEM of tremolite fiber 5000 X 14.5 micrometers x 0.9 micrometers.
6. EDX spectra of the fiber in 5.

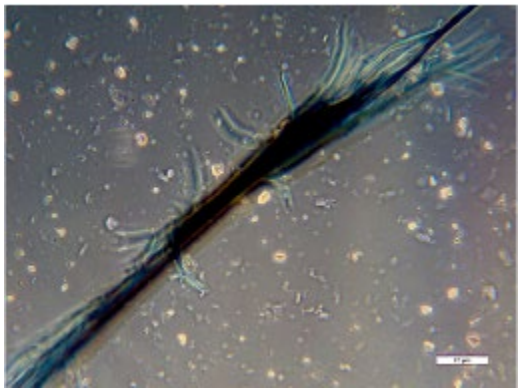
Appendix A

The following are photos and spectra of the samples. Not every type of photo is provided for every sample. Scale bars are included in the light microscopy samples.

1036658

1





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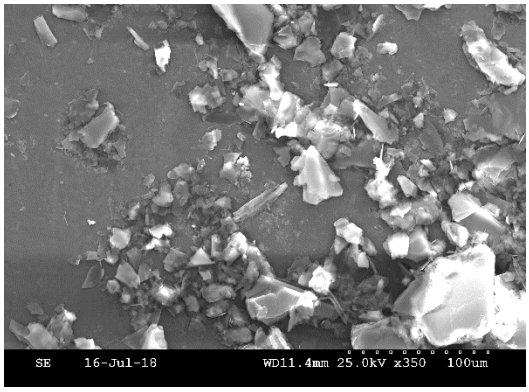
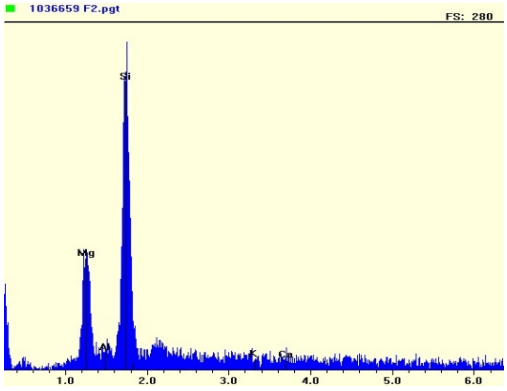




4

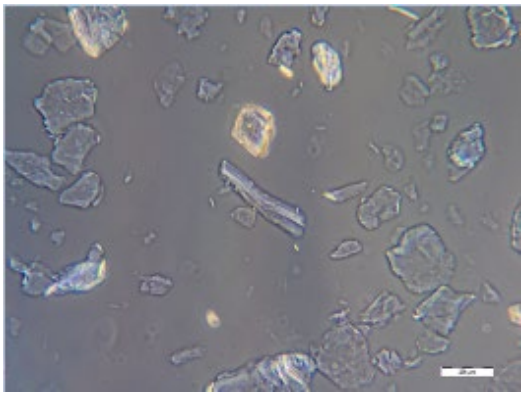
1. Bright Field PCM at 160X
2. Bright Field PCM at 160X organic (plant) fiber
3. SEM Elongate particle
4. EDX of particle in Photo 3. The particle is talc.

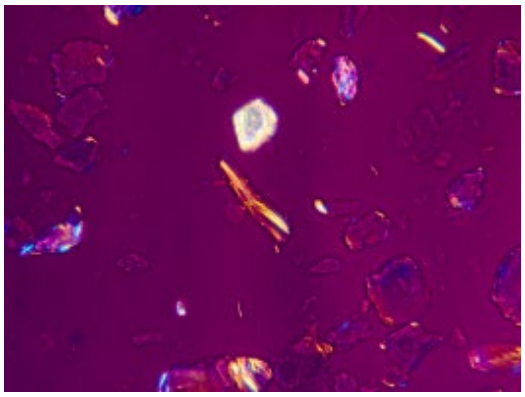
Appendix A

The following are photos and spectra of the samples. Not every type of photo is provided for every sample. Scale bars are included in the light microscopy samples.

1036659

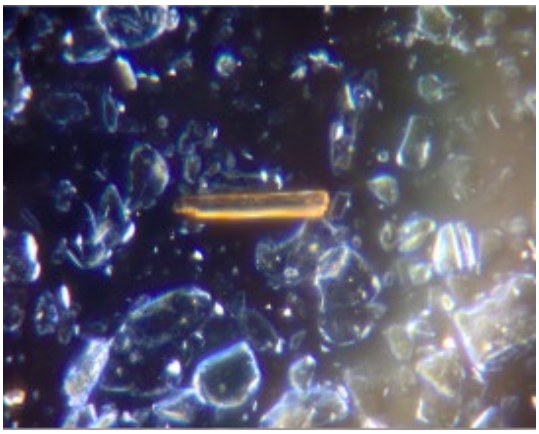
1

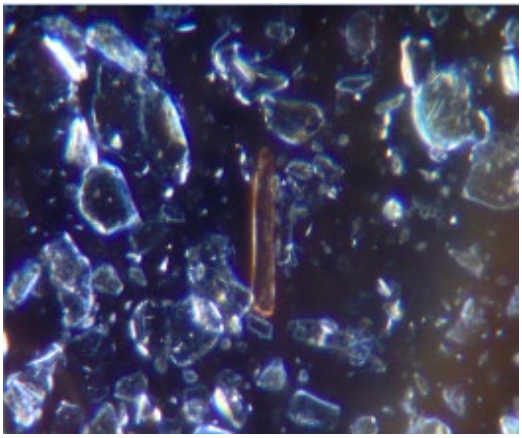




2

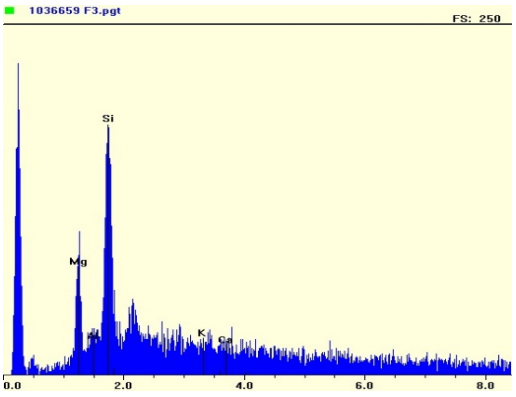
3





4

5



6

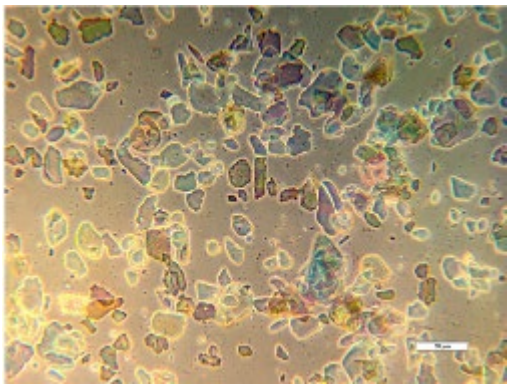
1. Bright Field PCM 400X asbestiform
2. PCM-PLM 400X same fiber as photo 1
3. CSDS of fiber 160X index greater than n=1.605
4. CSDS of fiber 160X index greater than n=1/605
5. SEM of EMP
6. EDX of fiber in Photo 5. The particle is talc.

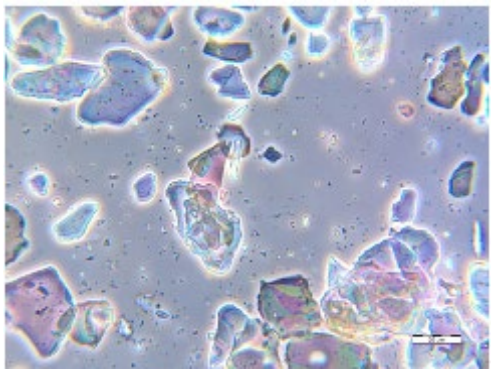
Appendix A

The following are photos and spectra of the samples. Not every type of photo is provided for every sample. Scale bars are included in the light microscopy samples.

5063618

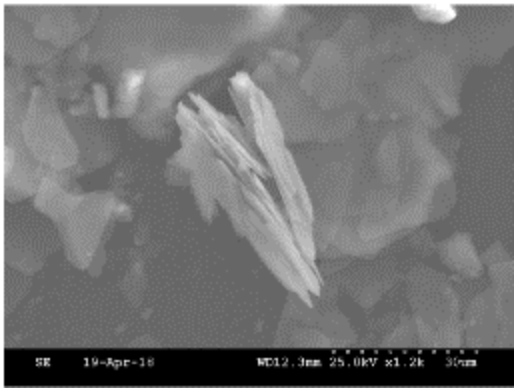
1

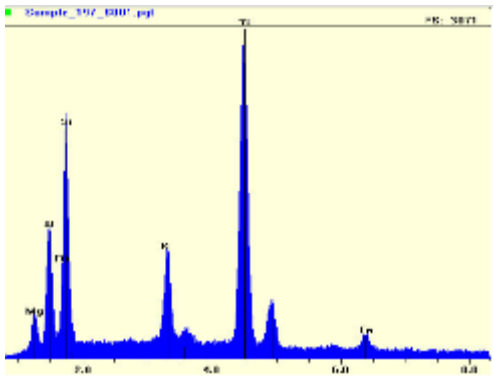




2

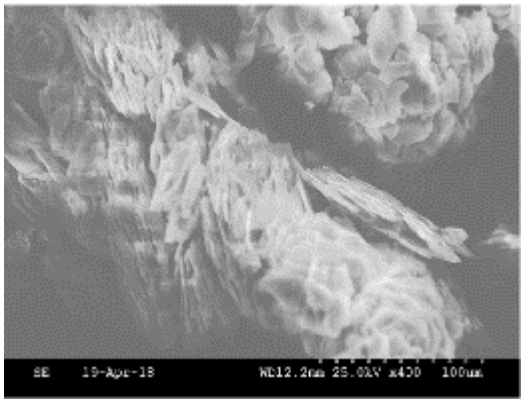
3

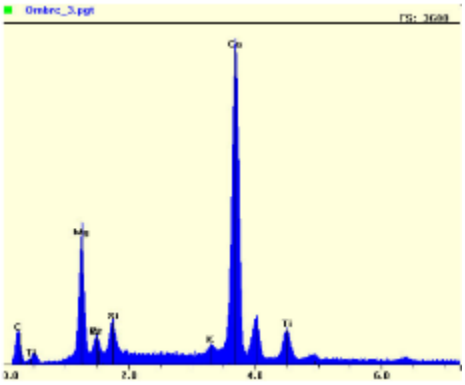




4

5





6

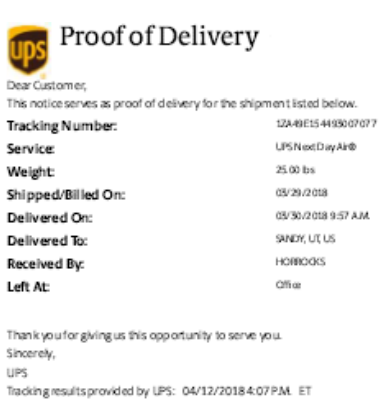
1. Bright Field PCM 160X some EMP
2. Bright Field PCM 400X some EMP
3. SEM EMP, foliar morphology, (uncoated with gold)
4. EDX of particle in Photo 3. NOT regulated mineral
5. SEM EMP, foliar morphology, (uncoated with gold)
6. EDX of particle in Photo 5. NOT regulated mineral

Appendix B -- Receipt of Samples

Firm Name	Product Description	Shipping Information and Sample Identification
Claire's 761227	Eye shadow pallet SKU84716	UPS 1ZA49E154493007077 INV 761227 (rcd. 30 March 2018) (14 boxes)
Claire's 761228	Love labeled box of mixed product SKU33411	UPS 1ZA49E154492098463 INV 761228 (rcd. 30 March 2018) (12 boxes with 2 units each)
Claire's 761230	Compact Powder	UPS 1Z A49E150196670130 761230 (rcd 10 May 2018) (24 units)
Claire's 761231	Contour	UPS 1ZA49E150196670130 761231 (RCD 10 May 2018) (24 units)
Claire's 945288	Shadow & Highlight	UPS 1ZA49E150196670130 945288 (RCD 10 May 2018) (27 units)
Tween Brands 1027488	JUSTICE JUST SHINE SHIMMER POWDER	UPS 1Z2R3A560107281092 1027488 (rcd. 30 March 2018) (6 units)
Beauty Plus 5063618	CITY COLORS SHIMMER OMBRE HIGHLIGHT PINK OPAL C-0025A (SK-17061A)	UPS 1Z2R3A560107281092 1030370 (rcd. 20 March 2018) (6 boxes with 2 units each)
1036658 NVF talc	FDA sample and sampling equipment	UPS 1ZA4744W8591420444 (rcd 27 March 2018) 2 bottles and assorted implements
1036659 NVF talc	FDA sample and sampling equipment	UPS 1ZA4744W8591420444 (rcd 27 March 2018) 2 bottles and assorted implements
Claire's 945287 **NOT ANALYZED PER FDA REQUEST**	Highlight Highlighting Palette UPC 61960-1	UPS 1ZA49E154494870454 INV 945287 (rcd. 30 March 2018) (26 boxes with 2 units each)

Appendix B -- Receipt of Samples

Sample INV 761227 was received in a single box Salt Lake Technical Center on 30 March 2018. Sample INV 761227 consisted of 14 boxes (units).



Proof of Delivery

Dear Customer,

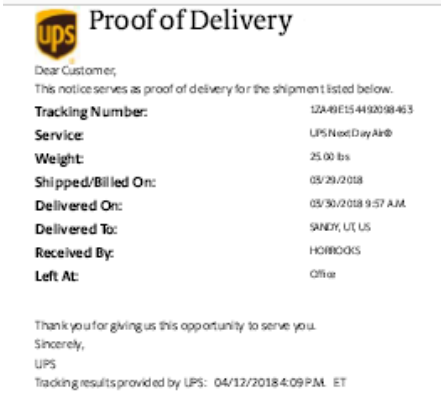
This notice serves as proof of delivery for the shipment listed below.

Tracking Number:	1ZA48E154490307077
Service:	UPS Next Day Air®
Weight:	25.00 lbs
Shipped/Billed On:	03/29/2018
Delivered On:	03/30/2018 9:57 A.M.
Delivered To:	SANDY, UT, US
Received By:	HORROCKS
Left At:	Office

Thank you for giving us this opportunity to serve you.
Sincerely,
UPS
Tracking results provided by UPS: 04/12/2018 4:07 P.M. ET

Appendix B -- Receipt of Samples

Sample 761228 was received in a single box Salt Lake Technical Center on 30 March 2018. Sample 761228 consisted of 12 boxes with 2 units each.



Proof of Delivery

Dear Customer,

This notice serves as proof of delivery for the shipment listed below.

Tracking Number:	1ZA49E154492098463
Service:	UPS Next Day Air®
Weight:	25.00 lbs
Shipped/Billed On:	03/29/2018
Delivered On:	03/30/2018 9:57 A.M.
Delivered To:	SANDY, UT, US
Received By:	HORROCKS
Left At:	Office

Thank you for giving us this opportunity to serve you.
Sincerely,
UPS
Tracking results provided by UPS: 04/12/2018 4:09 P.M. ET

Appendix B -- Receipt of Samples

Proof of Delivery

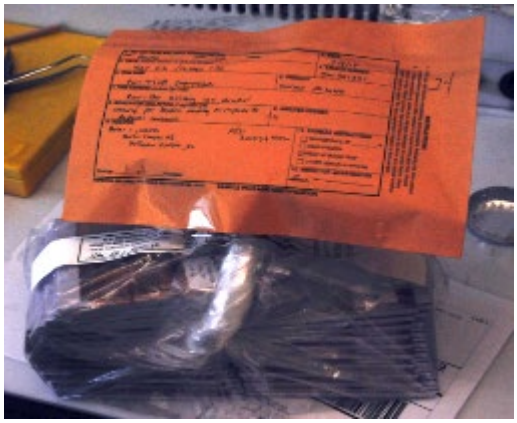
Dear Customer,

This notice serves as proof of delivery for the shipment listed below.

Tracking Number:	1ZA49E150196670130
Service:	UPS Next Day Air®
Weight:	25.00 lbs
Shipped/Billed On:	05/09/2018
Delivered On:	05/10/2018 9:57 A.M.
Delivered To:	SANDY, US
Received By:	HORROCKS
Left At:	Office

Sample 761230, 761231 and 945288 were received in a single box Salt Lake Technical Center 5on 30 March 2018. Sample 761230 consisted of 24 units, sample 761231 consisted of 24 units and sample 9455288 consisted of 27 units.

761230



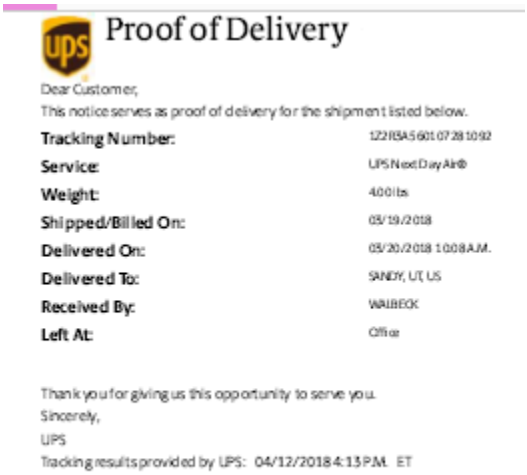
761231

Appendix B -- Receipt of Samples

945288

Appendix B -- Receipt of Samples

Samples 1027488 and 1030370 were received in a single box at the Salt Lake Technical Center on 20 March 2018. Sample 1027488 consisted of 6 units. Sample 1030370 consisted of 6 boxes with 2 units each



Proof of Delivery

Dear Customer,

This notice serves as proof of delivery for the shipment listed below.

Tracking Number:	1Z2R8A560107281092
Service:	UPS Next Day Air®
Weight:	4.00 lbs
Shipped/Billed On:	03/19/2018
Delivered On:	03/20/2018 10:08 A.M.
Delivered To:	SANDY, UT, US
Received By:	WALBECK
Left At:	Office

Thank you for giving us this opportunity to serve you.
Sincerely,
UPS
Tracking results provided by UPS: 04/12/2018 4:13 P.M. ET

Appendix B -- Receipt of Samples

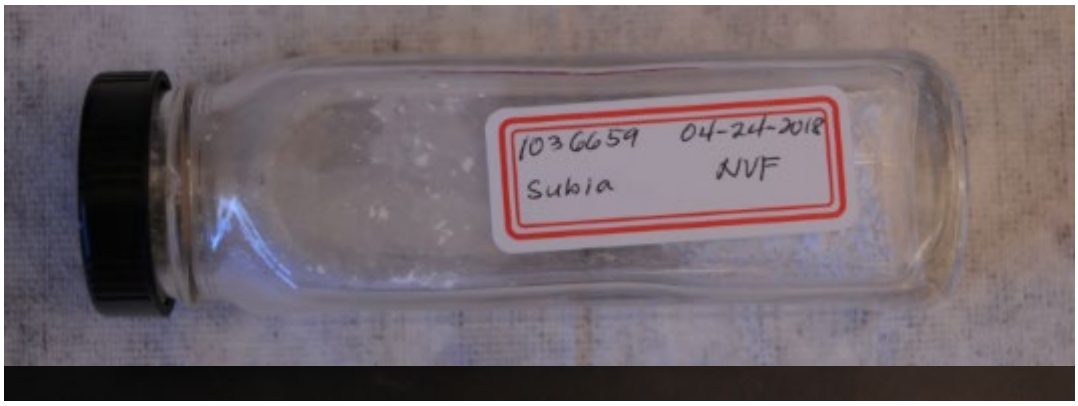
Samples 1036658 and 1036659 were received in a single box. Each consisted of two glass bottles of white powder and a bag containing sampling implements. The sampling implements were set aside and have not been analyzed.

Proof of Delivery

Dear Customer,

This notice serves as proof of delivery for the shipment listed below.

Tracking Number:	1ZA4744W8591420444
Weight:	7.00 lbs
Shipped/Billed On:	04/25/2018
Delivered On:	04/27/2018 9:41 A.M.
Delivered To:	SANDY, US
Received By:	HORROCKS
Left At:	Office



Note: Sampling implements not shown.

Appendix B -- Receipt of Samples

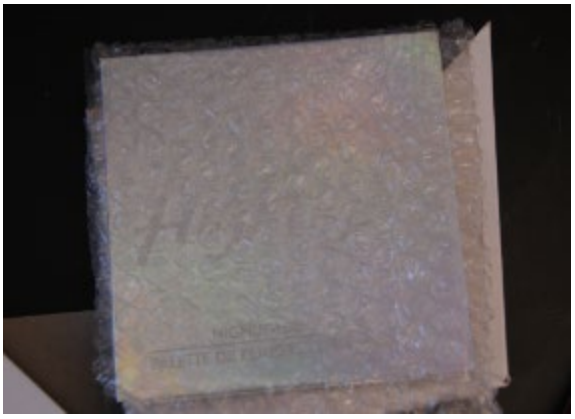
Sample INV 945287 was received in a single box Salt Lake Technical Center on 30 March 2018. Sample INV 945287 consisted of 26 boxes with 2 units each.

Proof of Delivery

Dear Customer,
This notice serves as proof of delivery for the shipment listed below.

Tracking Number:	1ZA481E54494870454
Service:	UPS Next Day Air®
Weight:	25.00 lbs
Shipped/Billed On:	03/29/2018
Delivered On:	03/30/2018 9:57 A.M.
Delivered To:	SANDY, UT, US
Received By:	HORROCKS
Left At:	Office

Thank you for giving us this opportunity to serve you.
Sincerely,
UPS
Tracking results provided by UPS: 04/12/2018 4:02 P.M. ET



Appendix C -- Definitions for Regulated Asbestos

In order to avoid any confusion, the following nomenclature is used. This is based upon the definitions that have been established by regulation and litigation.[3, 4]

Asbestos includes only the minerals chrysotile, Amosite, crocidolite, tremolite asbestos, anthophyllite asbestos, and actinolite asbestos.[5] An asbestos mineral has a fibrous growth habit.

Asbestiform The term "asbestiform" is not a growth habit. It is a description of a mineral which has a fibrous growth habit. The growth habit used in mineralogy is "fiber." Individual fibers of asbestos are held together by Van der Waals forces rather than ionic or covalent bonds.

> "It (asbestiform) is an inherent, fine-textured morphology in a mineral. Resulting from unequal relative development of the principle crystal faces, which ideally produces a predominant release of strong, flexible fibers having (1) microscopic to submicroscopic diameter and (2) and aspect ratio often exceeding 20:1, when the mineral is subjected to comminution."[6]

Fibrous is a mineralogical term for growth habit for crystals that are composed of long thin fibers. In mineralogy, there is no fixed aspect ratio (length to width ratio) that defines a fiber. Commonly used aspect ratios are 3:1, 5:1, 10:1, 20:1 and 100:1 depending upon use. In regulation, a 3:1 aspect ratio is established by regulation and case law.

A **regulatory fiber** is a fiber of asbestos

1. Having an aspect ratio greater than or equal to 3:1
2. Longer than or equal to 5 micrometers
3. Visible in a phase contrast microscope (PCM)

A **fibril** is a single crystal of an asbestiform mineral. A fibril, is, by definition, a fiber. Fibrils nominally have diameters of about 50 nm for chrysotile and about 100 nm for amphiboles (depending upon the species and growth conditions.).

A **bundle** is an assembly of fibrils and is usually considered to be a fiber.

A **matrix** is a matted mass of fibers, considered a regulatory fiber if one or more fibers extends 5 micrometers from the mass. (In the TEM, a matrix is considered equivalent to one fiber)

Elongate particle (EMP) is a particle with an aspect ratio generally longer than 3:1 regardless of whether it is fibrous or is elongate due to comminution (grinding).

Growth habit Crystal habit is the tendency for specimens of a mineral to repeatedly grow into characteristic shapes.[7]

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[3] Secretary of Labor v Borg Warner, OSHRC Docket No. 10757, 22 February 1978
[4] 29 CFR 1910.1001 and others, Occupational Safety and Health Administration,
[5] 29 CFR 1910.1001 (b) definitions
[6] Definitions for Asbestos and other Health-related silicates, Levadie, B., ASTM STP834, 1982
[7] https://geology.com/minerals/crystal-habit/

Appendix D
PCM-PLM-Dispersion Staining Go/No-Go Test for Amphibole Fibers in Talc

The PCM-PLM Dispersion Staining Go/No-Go test is used as a screening test to determine whether or not amphibole fibers are present in powdered samples of talc.

A sample is examined using a polarizing phase-contrast microscope (PCM-PLM) at 400X and 160X magnifications. Any fibers meeting the federal definition for asbestos will be visible because of the use of PCM. Fibers with diameters greater than 0.5 to 1 micrometer may be examined for the presence of retardation colors. If there are any visible fibers, they are examined using central stop dispersion staining (CSDS) using the same microscope.

A preparation of powdered sample is made in high-dispersion refractive index liquid with n = 1.605. The observable indices of refraction of all of the regulated amphiboles are greater than 1.605, while the indices of refraction for talc is less than n = 1.605.

Because of this, the central-stop dispersion staining (CSDS) colors observed for amphiboles will indicate that the particle indices are greater than the index of the liquid. Generally, this will be yellow orange to yellow, or yellow-white. Talc, brucite and chrysotile fibers will be blue to blue-white.

If there are no fibers with CSDS colors indicative of possible amphibole present (e.g. yellow to yellow white) then it is unlikely that any amphibole asbestos is present.

When fibers are visible that have CSDS colors indicating indices greater than n = 1.605, further examination in matching liquids as well as other measurements are required to identify the amphibole.

The index of refraction liquid n = 1.605 is not a matching liquid for talc or any amphibole and is insufficient to identify any of the regulated minerals.

The samples submitted for this investigation were either packed powder or loose powder. All were apparently dry with no apparent wetness, equilibrated to the local humidity.

For this examination, no gravimetric reduction was performed. (e.g. the samples were not subjected to acid, heat or chemicals designed to remove unwanted substances, and examined as received.).

Samples were prepared individually in a table-top laboratory hood, and all instruments and horizontal surfaces were cleaned in between samples. This is a measure to prevent cross-contamination between the samples.

At least three separate slide preparations were made and examined for each of the samples.

1. Two to three drops of high dispersion index of refraction liquid are allowed to drop by gravity onto the surface of a cleaned 1 x 3 in. glass slide.
2. A clean dissecting needle is used as a sampling probe. The tip is wetted in the index of refraction liquid on the slide and then dipped into the sample. A small amount of powder

clings to the dissection needle. The amount is determined by the diameter of the captured powder clump and is usually less than a 0.5 mm.

3. The dissection needle with the powder is introduced into the liquid on the slide and stirred gently to disperse the powder as evenly as possible.
4. A clean, No 1 ½ glass cover slip is gently lowered onto the liquid.

NOTE 1: The amount of index of refraction liquid placed on the slide in step 1 is determined by experience such that little to no liquid extends beyond the boundary of the cover slip.

NOTE 2: If the preparation is cloudy, it indicates that too much powder was on the dissection needle when the powder was sampled. Discard this preparation and perform steps 1 to 4 using a smaller amount of powder until a suitable preparation is made. Too much powder in the preparation will interfere with the optical tests. These tests generally depend on particles being dispersed in the liquid sufficiently far apart so that their images do not interfere with one another.

Examination

A phase contrast microscope which also has crossed polarizing elements and a first-order red compensator is used to examine the samples. (See OSHA ID-191 for a full description of PCM-PLM examination.)

A slide preparation is placed on the microscope stage and examined at 160X and 400X. (The sample may also be examined at 100X if the microscope is equipped for PCM at 100X. In this particular investigation, little use was made of the 100X magnification because the particles were very finely divided.)

An initial scan of the slide is made at 160X and 400X to note the presence of any elongate particles. The morphology of any observed elongate particle is evaluated whether it is, organic, synthetic or mineral, and if mineral, consistent with asbestiform growth habit or other growth habit. Any observed fiber is evaluated for the presence of birefringence observed as retardation colors.

Any mineral fibers are examined at 160X using CSDS. If the colors observed indicate that the fiber may be amphibole, further analysis of the sample is indicated. The sample prep is scanned at 160X to determine if any amphibole source mineral is present.

Appendix E

Preparation and Analysis of Samples by Scanning Electron Microscopy and X-ray Energy Dispersion

The samples were examined by scanning electron microscopy (SEM) and x-ray energy dispersive analysis (EDX). In combination, SEM and EDX provide visual evidence of the morphology of the fibers and a semi-quantitative measure of which elements are present in individual particles.

The SEM uses a beam of electrons accelerated to a high voltage focused to a point and scanned across the surface of the sample. Secondary electrons generated when the beam strikes the sample are used to form a dimensional image of the surface of the sample. At each point where the primary electron beam strikes the sample, x-rays are generated which have energies characteristic of the elements at that location. These x-rays are captured, and the energies analyzed to provide a spectrum representing the elements present in the particle.

Samples for SEM are made by applying a very small amount of powder on a spectrometrically pure carbon adhesive surface on a carbon SEM mount. The powder is smeared onto the surface in a very sparse layer to provide adequate separation between the particles. If the particles are too close, the analytical signal may be affected by other particles nearby.

For high resolution images, conductive coatings are evaporated or sputtered onto SEM samples to render the surface sufficiently conductive electrically that the surface cannot build up a surface charge and degrade the image.

In the first analytical trial, the samples were examined in the SEM without any conductive coating. This was to obtain particle spectra free of any interference from the conductive metal. However, images obtained without a metal coating do not have the resolution necessary to reveal the fine structure of the surfaces of non-conductive particles. During the analysis, it was noted that there were no elements intrinsic to the particulate present in the cosmetics that preclude the use of gold as a coating metal.

A second analytical trial looked at selected samples which have been coated with sputtered gold to a thickness of 5 to 10 Å (0.5 to 1 nm). This thickness is less than the analytical resolution of the SEM, but sufficient to provide a conductive surface and thereby a better evaluation of the morphology of the fibers.

The SEM used for this is a Hitachi S-3500 VP. It was operated in high vacuum mode at and acceleration voltage of 25kV and a working distance of 10mm. A variety of magnifications were used as noted on the photographs. Magnifications greater than about 2,000X were not usable for uncoated samples. Gold coating is necessary for higher magnification evaluation.

The surface of the samples were scanned at between 500 and 1000X to find any fibers. When found, the electron beam is focused on a single spot on the fiber and an EDX spectrum is collected and a determination as to the possible identification of the mineral. Fibers are photo-documented as well as a corresponding EDX spectrum.