AMA Analytical Services, Inc.
Focused On Results.

CERTIFICATE OF ANALYSIS

Chain of Custody: 308006
Client: US Food & Drug Adminitration
Address: Office of Cosmetics & Colors
4300 River Road
College Park, MD 20740
Attention: John Gasper

Job Name: Task 3 - Analysis of Official Samples
Job Location: 4th Group - 15 Samples
Job Number: CLIN 1 - Task 3
PO Number: HHSF223201810337P

Date Submitted: 7/24/2019
Date Analyzed: 8/20/2019-9/18/2019
Report Date: 10/3/2019
Date Sampled: Not Provided
Person Submitting: Goran Periz
Revised: 10/11/2019 (Revision #2)

SUMMARY OF ANALYSIS

AMA Sample ID	Client Sample ID	TEM LOD Using ASTM D5756 Mass Calculation	TEM LOQ Using ASTM D5756 Mass Calculation	% Tremolite by TEM Using ASTM D5756 Mass Calculation	% Chrysotile by TEM Using ASTM D5756 Mass Calculation	% Total Tremolite & Chrysotile by TEM Using ASTM D5756 Mass Calculation	% Asbestos by PLM	% Organics	% Acid Soluable	% Other	Comments
308006-6	D-58	0 00000169%	0.00000675%	ND	ND	ND	ND	0.3%	6.7%	93.1%	Gravimetric Loss from PLM Prep: Organics = 0.3%; Acid Soluable = 7.1%; Other = 92.6%
308006-6A	D-58	0 00000133%	0.00001485%	ND	< 0.00001%	< 0.00001%	ND	0.2%	19.5%	80.2%	Gravimetric Loss from PLM Prep: Organics = 0.2%; Acid Soluable = 8.5%; Other = 91.3%
308006-6B	D-58	0 00000135%	0.00000540%	ND	0.00002%	0.00002%	ND	0.2%	11.2%	88.6%	Gravimetric Loss from PLM Prep: Organics = 0.3%; Acid Soluable = 5.5%; Other = 94.2%

LOD = Limit of Detection **LOQ** = Limit of Quantification **ND** = Not Detected **PLM** = Polarized Light Microscopy **TEM** = Transmission Electron Microscopy

Analytical Method(s): PLM by Modified NY ELAP 198.6
TEM by Modified NY ELAP 198.4/ASTM D5756

Analyst(s): **PLM** (b) (6)
TEM (b) (6)

Technical Director: Andreas Saldivar

All results are to be considered preliminary and subject to change unless signed by the Technical Director or Deputy

Record Changes Report

Client: US Food & Drug Administration

Client Code: FDA

Chain of Custody: 308006

Date	Description
10/11/2019	308006 6, 6A, 6B/D 58: 1) added initials & dates to all strike throughs and additions to gravimetric bench sheets. 2) revised handwritten TEM bench sheet for 6B to break up the single cluster found on Grid B, GO I8 into its 3 component fibers 3) changed the word "fiber" to "structure" on p. 4 of Case Narrative under LoQ discussion for 6A & 6B & updated the basis of LoQ calculation for 6B. 4) changed the word "fiber" to "structure" in reference to chrysotile on p.4 of Case Narrative under the TEM Discussion and Interpretation of Analytical Findings. 5) Updated the picture for 308006 6B Chrysotile Structure 1 on p. 6 of Case Narrative. 6) revised reported LoQ, concentration of chrysotile & total cocentration for aliquot 6B based off of 4 structures (original concentration was based off of 2 structures). 7) added gravimetric loss data for PLM preparations to comments section of the certificate of analysis.
10/08/2019	308006 6, 6A, 6B/D58: 1) The Special Instructions section of the login sheet was revised to include the FDA's cancellation of a request for analyzing a 4th aliquot of D 58 (308006 6C). 2) The preparation date was added to pages 2 & 3 of the TEM gravimetric bench sheet and to page 2 of the PLM gravimetric bench sheet; an explanation for the date written in the right hand margin of both sets of bench sheets was added to them; added missing weights for 308006 16 and 308006 17. 3) The handwritten TEM Bench Sheet for 308006 6A was revised to explain that the 2nd Chrysotile structure was identified based upon tubular morphology; also the structure number count for the 2nd listed stricture was corrected to read "#2"

AMA Analytical Services, Inc.
Focused on Results www.amalab.com
AIHA-LAP (#100470) NVLAP (#101143-0) NY ELAP (10920)
4475 Forbes Blvd. • Lanham, MD 20706
(301) 459-2640 • (800) 346-0961 • Fax (301) 459-2643

CHAIN OF CUSTODY

(Please Refer To This Number For Inquires) 308006

Mailing/Billing Information:
1. Client Name: FDA
2. Address 1:
3. Address 2:
4. Address 3:
5. Phone #: Fax #:

Submittal Information:
1. Job Name: Task 3 - Analysis of Official Samples
2. Job Location: 4th Group - FD Samples
3. Job #: P.O. #: HHSF-223201810337P
4. Contact Person: FDA John Graster Cell:
5. Collected by: FDA Cell:

Reporting Info (Results provided as soon as technically feasible). If no TAT/Reporting Info is provided, AMA will assign defaults of 5-Day and email/fax to contacts on file.

AFTER HOURS (must be pre-scheduled)	NORMAL BUSINESS HOURS	REPORT TO:
❑ 4 Hours ❑ Late Night ❑ Immediate Date Due: ❑ 24 Hours Time Due: Comments:	❑ 4 Hours ❑ 3 Day ❑ Same Day ❑ 5 Day + ❑ Next Day Date Due: ❑ 2 Day ❑ Results Required By Noon	❑ Email: ❑ Email 2: ❑ Verbals:

Asbestos Analysis
*PCM Air – Please Indicate Filter Type:
- ❑ NIOSH 7400 (QTY)
- ❑ Fiberglass (QTY)

TEM Air* – Please Indicate Filter Type:
- ❑ AHERA (QTY)
- ❑ NIOSH 7402 (QTY)
- ❑ Other (specify) (QTY)

PLM Bulk
- ❑ EPA 600 – Visual Estimate (QTY) ❑ Pos Stop
- ❑ EPA Point Count (QTY)
- ❑ NY State Friable 198.1 (QTY)
- ❑ Grav. Reduction ELAP 198.6 (QTY)
- ❑ Other (specify) (QTY)

MISC
- ❑ Asbestos Soil PLM (Qual) PLM (Quan) PLM/TEM (Qual) PLM/TEM (Quan)

*It is recommended that blank samples be submitted with all air and surface samples

TEM Bulk
- ❑ ELAP 198.4/Chatfield (QTY)
- ❑ NY State PLM/TEM 15 (QTY) FDA MOD
- ❑ Residual Ash (QTY)
- ❑ Vermiculite

TEM Dust*
- ❑ Qual. (pres/abs) Vacuum/Dust (QTY)
- ❑ Quan. (s/area) Vacuum D5755-95 (QTY)
- ❑ Quan. (s/area)Dust D6480-99 (QTY)

TEM Water
- ❑ Qual. (pres/abs) (QTY)
- ❑ ELAP 198.2/EPA 100.2 (QTY)
- ❑ EPA 100.1 (QTY)

❑ All samples received in good condition unless otherwise noted.
TEM Water samples °C)

If field data sheets are submitted, there is no need to complete bottom section.

Metals Analysis
- ❑ Pb Paint Chip (QTY)
- ❑ *Pb Dust Wipe (wipe type) (QTY)
- ❑ *Pb Air (QTY)
- ❑ Pb Soil/Solid (QTY)
- ❑ Pb TCLP (QTY)
- ❑ Drinking Water ❑ Pb (QTY) ❑ Cu (QTY) ❑ As (QTY)
- ❑ Waste Water ❑ Pb (QTY) ❑ Cu (QTY) ❑ As (QTY)
- ❑ Pb Furnace (Media) (QTY)

Fungal Analysis
Collection Apparatus for Spore Traps/Air Samples:
Collection Media
- ❑ *Spore-Trap (QTY) ❑ Surface Vacuum Dust (QTY)
- ❑ *Surface Swab (QTY)
- ❑ *Surface Tape (QTY)
- ❑ Other (Specify) (QTY

SAMPLE INFORMATION				ANALYSIS						MATRIX							
CLIENT ID #	SAMPLE LOCATION/ ID	DATE/ TIME	VOL (L)/ Wipe Area	TEM	PCM	PLM	LEAD	MOLD	AIR	BULK	DUST	WATER AND OTHER	SPORE TRAP	TAPE	SWAB	COMMENTS / SPECIAL INSTRUCTIONS
	D23 thru D-67															

	Print Name	Signature	Date	Time	Shipping Information
Relinquished by:	GORDON PEARCE	[redacted]	7/24/2019	11:00	☐ UPS ☐ In Person ☐ Other
Received by:	[redacted]	[redacted]	7/24/19	1055	☐ FedEx ☐ Drop Box ☐ USPS ☐ Courier

ATTACHMENT B: CFSAN OFFICE OF COSMETICS AND COLORS CHAIN OF CUSTODY FORM

CFSAN
Office of Cosmetics and Colors
CHAIN OF CUSTODY FORM

Case/Lab No: _____

Submitter: Goran Periz

Assignment No./ Contract No.: HHSF223201810337P

Entry No./Lab No.: 3058004

Date Sealed: 7/23/2019 Sample Type: 15 samples D-53 to D67

Description of Evidence		
Item #	**Quantity**	**Description of Item** (Lab#, Lot #, Condition)
1	1	(b) (4) [redacted]
2	1	
3	1	
4	1	
5	1	
6	1	Sample ID #: D-58
7	1	(b) (4) [redacted]
8	1	
9	1	
10	1	
11	1	
12	1	
13	1	
14	1	
15	1	

Chain of Custody

Item #	Date	Received by (Print Name)	Received by (Signature)	Comments/Location
1-15	7/24/2019			

CHAIN OF CUSTODY FORM

(Continued)

Chain of Custody				
Item #	**Date/Time**	**Released by (Print Name)**	**Received by (Signature)**	**Comments/Location**
1-15	7/24/19 1000	GORAN PERIZ	(b) (6)	D/O @ AMA

Final Disposal Authority

Authorization for Disposal

Item(s) #: _____ on this document is/are no longer needed as evidence and is/are authorized for disposal by (check appropriate disposal method)

☐ Return to Submitter ☐ Destruction

Name of Authorizing Official: _____ Date: _____

Signature: _____

Witness to Destruction of Evidence

Item(s) #: _____ on this document were destroyed by (Name) _____ in my presence on (date) _____.

Name of Witness to destruction: _____ Signature: _____ Date: _____

Adapted from: Technical Working Group on Biological Evidence Preservation. *The Biological Evidence Preservation Handbook: Best Practices for Evidence Handlers*. U.S. Department of Commerce, National Institute of Standards and Technology. 2013.

Release to Lawful Owner

Item(s) #: _____ on this document was/were released by Evidence Custodian
_____ ID#: _____ to
Name _____
Address: _____ City: _____ State: _____
Zip Code: _____
Telephone Number: (_____) _____
Under penalty of law, I certify that I am the lawful owner of the above item(s).

Signature: _____ Date: _____

Copy of Government-issued photo identification is attached. ☐ Yes ☐ No

This form is to be retained as a permanent record by the Center for Food Safety and Applied Nutrition, Office of Cosmetics and Colors.

Adapted from: Technical Working Group on Biological Evidence Preservation. *The Biological Evidence Preservation Handbook: Best Practices for Evidence Handlers*. U.S. Department of Commerce, National Institute of Standards and Technology. 2013.

AMA Analytical Services, Inc.
Focused On Results.

NVLAP Lab Code 101143-0

NY ELAP Lab ID 10920

Case Narrative

Client Name:	FDA Office of Cosmetics & Colors	Contact:	John Gasper
PO Number:	HHSF223201810337P	Phone:	(240) 402-1133
Job Name/Location:	Task 3 – Analysis of Official Samples (4th Group – 15 Samples)	Email:	john.gasper@fdsa.hhs.gov
AMA COC Number:	308006-6, 6A, 6B/D-58	Date Received:	July 24, 2019

AMA Sample No.	Client Sample No.	Sample Description	Analytical Method
308006-6	D-58	Slightly clumpy, white powder with a matte appearance	Mod. PLM ELAP 198.6 /TEM ELAP 198.4
308006-6A	D-58		Mod. PLM ELAP 198.6 /TEM ELAP 198.4
308006-6B	D-58		Mod. PLM ELAP 198.6 /TEM ELAP 198.4

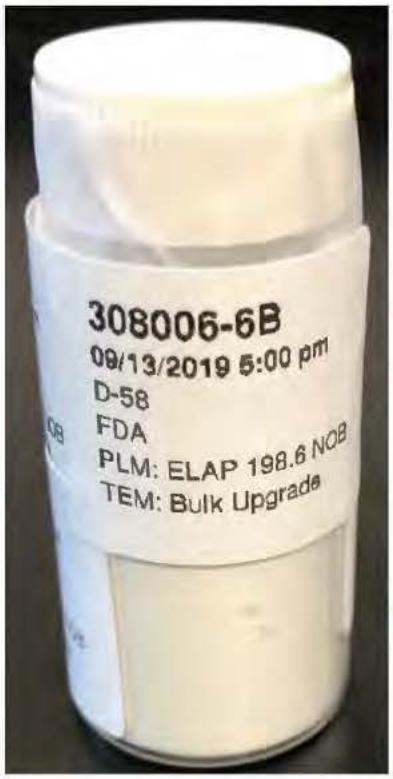
Requested Analyses: PLM and TEM Analysis for asbestos fibers conducted by Modified NY ELAP Method 198.6 and Modified NY ELAP Method 198.4

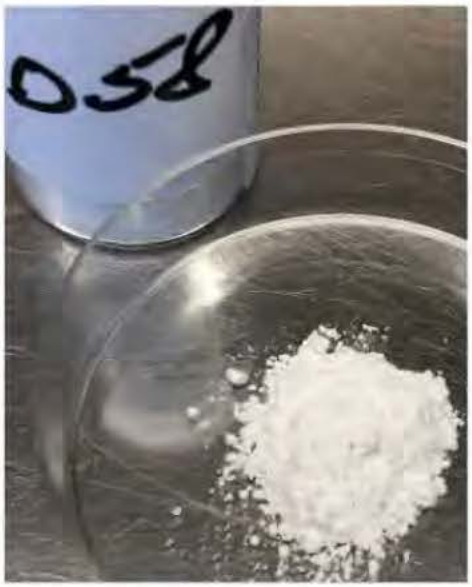
Sample Receipt:

The samples were received by AMA Analytical Services, Inc. on July 24, 2019 at 1058 via in-person drop-off by FDA representative, Goran Periz. The set consisted of 15 (fifteen) samples submitted in ~2oz, glass jars sealed with scotch tape. Conditions were checked upon receipt and all sample containers were intact. Most jars were filled approximately ½ to ¾ full. The sample set was processed on AMA Chain-of-Custody (COC) number 308006. This COC number served as the internal laboratory job number for tracking purposes. The samples were entered into the AMA laboratory database on August 12, 2019 at 1151 by (b) (6). The samples were logged in for analysis in triplicate and each sample aliquot was assigned a unique laboratory identification number as shown in the table above. After the sample login, the set was transferred to AMA's lock-box for storage.

The following pictures document the condition of each sample upon receipt at AMA:

308006-6, 6A, 6B/D58





Sample Preparation

Samples were prepared for PLM and TEM bulk analysis by (b) (6) on August 13, 2019 through September 5, 2019. Sample preparation consisted of the following steps:

1) Label and weigh two 8mL glass vials for each sample in the set – one vial for the PLM preparation and one vial for the TEM preparation.
2) Weigh out 0.1 to 0.8 grams of material and place in corresponding 8mL glass vial. Record weight.
3) Burn samples at 480° C for at least 12 hours.
4) Record Post-Ash Weight.
5) Treat ashed sample with concentrated hydrochloric acid.
6) Filter acid reduced material onto a pre-weighed 47mm 0.4um PolyCarbonate filter.
7) Place filter into drying oven for 30 minutes and then record Post-Acid Reduced weight.
8) Make four PLM slide preparations from the PLM residual ash for each sample in 1.550 dispersion oil. Make additional preparations in 1.605, 1.625, 1.680 and 1.700 dispersion oil as necessary for particle identification.
9) Weigh a portion of the residue from the TEM residual ash and place it into the corresponding pre-weighed 100ml jar.
10) Fill the 100ml jar with deionized water
11) Sonicate the jars for approximate 5-minutes.
12) Filter 0.2ml to 1ml of the solution onto a 47mm 0.22um MCE filter.
13) Dry the filter for 10 minutes then collapse, carbon coat, and place on a 3 TEM grids.

PLM Analysis

Analysis was performed in accordance with NY ELAP 198.6 protocols. The analysis was conducted using an Olympus BH-2 polarized light microscope (PLM) equipped with a dispersion staining objective. All four slide preparations for each aliquot were examined. 400-point count was performed for those samples on which asbestos was observed. If no asbestos was detected on any of the slides, the percentage of fibrous components was determined by visual estimation. The results of this analysis are detailed below in the *Discussion and Interpretation of Analytical Findings* section for each individual sample.

TEM Analysis

Analysis was performed in accordance with modified NY ELAP Method 198.4 protocols. The analysis was performed using a JEOL JEM-100CX II transmission electron microscope (TEM), equipped with a Thermo Fisher Quest Energy Dispersive X-Ray Analyzer (EDXA), at magnifications of 19,000x. Two grids for each aliquot were examined. Twenty (20) grid openings were examined per sample.

Modifications to the NY ELAP 198.4 Method were:

1) The residue was not placed in alcohol and prepared using the quick drop method. To obtain a more uniform preparation, the residue was placed in a jar and filled with 100ml of deionized water. The jar was sonicated, and a portion of the solution was filtered onto a 47mm 0.22um MCE filter.
2) The tremolite and chrysotile were not visually estimated. The length and width of the observed particles were measured, and the mass of each amphibole particle was calculated using the ASTM D5756 method.
3) All particles identified as tremolite were included with the counts/concentrations, regardless of size and aspect ratio.

The results of this analysis are detailed below in the *Discussion and Interpretation of Analytical Findings* section for each individual sample.

Calculations

ASTM D5756 Mass

$M = \pi/4\ L * W^2 * D * 10^{-12}$

M = mass
L = length

W = width
D = density
Percent Calculation

$$\frac{EFA(mm^2) * 100ml * MA(g) * RW(g)}{VF(ml) * IW(g) * AA(mm^2) * RJ(g)}$$

The calculated value is then multiplied by 100 to convert it to percent.

EFA – Effective filter area
MA – Mass of asbestos
RW – Weight of residue
VF – Volume filtered
IW – Initial weight of the sample
AA – Area analyzed
RJ – Weight of residue placed into the jar

Limit of Detection and Quantification

We used the mass of a 0.5 x 0.04-micron tremolite or chrysotile fiber, depending on what was found in each sample, as the basis for our calculations. Limit of detection was defined as 1 fiber and limit of quantification was defined as 4 fibers.

Some aliquots of sample D58 contained very small amounts of asbestos that were either at or below our 4-fiber limit of quantification. For these samples we defined our limit of quantification as follows:

308006-6A: mass of the two observed chrysotile structures plus the mass of two chrysotile fibers measuring 0.5 x 0.04 microns
308006-6B: mass of 4 chrysotile fibers measuring 0.5 x 0.04-micron

Discussion and Interpretation of Analytical Findings:

308006-6, 6A, 6B Client Sample D-58

PLM

All three aliquots of sample D-58 were analyzed by (b) (6) on September 13, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

308006-6	NAD
308006-6A	NAD
308006-6B	NAD

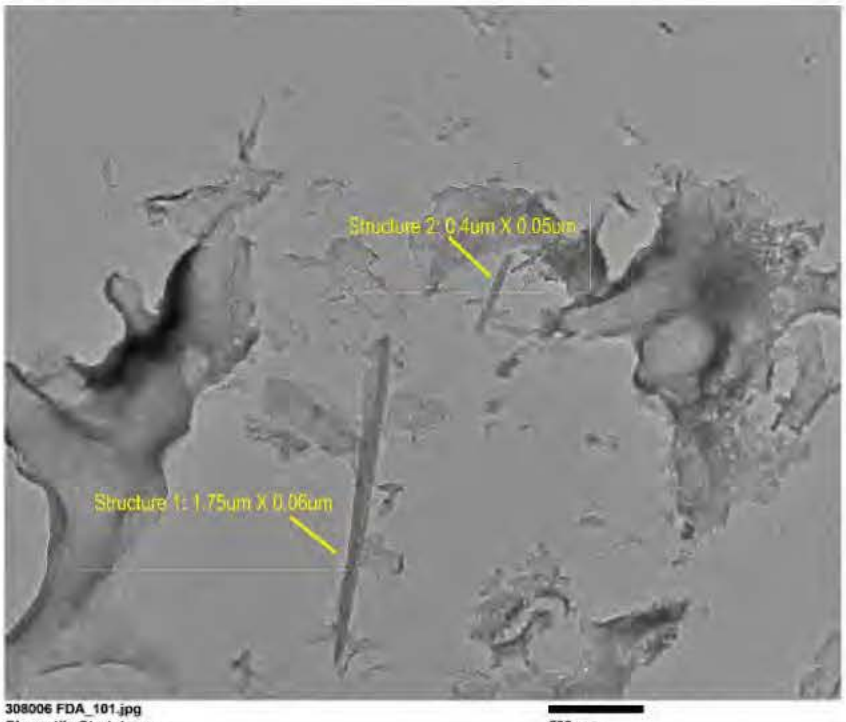
TEM

Sample 6 was analyzed by (b) (6) on September 3, 2019. Samples 6A and 6B were analyzed by (b) (6) (b) (6) on September 7, 2019. The primary particle observed was talc along with a few talc fibers, talc ribbons and mica particles. Two Chrysotile structures were detected on the aliquot for 6A and four chrysotile structures were detected on the aliquot for 6B. The results were calculated using the equations detailed in the calculations section.

308006-6	NAD
308006-6A	<0.00002%
308006-6B	0.00002%

Below are pictures, diffraction patterns, and chemistry from some of the observed particles. The unidentified peaks in chemistry spectra are copper, zinc, and carbon. Those peaks are from the TEM specimen holder and specimen grid.

Sample 308006-6A, Chrysotile Structures



308006 FDA_101.jpg
Chrysotile Strutctures
308006-6a
Cal: 0.001774 µm/pix
14:06 9/7/2019
TEM Mode: Imaging
Microscopist: CD
Camera: NANOSPRTS, Exposure: 800 (ms) x 5 std. frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

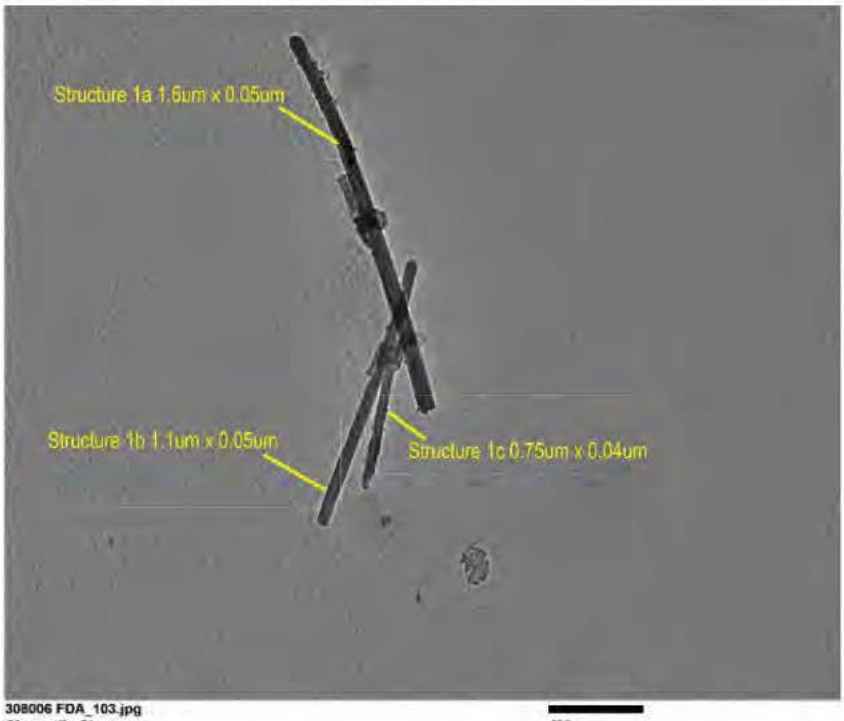
500 nm
HV=100kV
Direct Mag: 5800 x
AMA Analytical Services, Inc

Diffraction Pattern from Chrysotile Structure 1 pictured above

308006 FDA_100.jpg
Chrysotile Dif
308006-6a
14:03 9/7/2019
TEM Mode: Diffraction
Microscopist: CD
Camera: NANOSPRTS, Exposure: 800 (ms) x 5 std. frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

100 (1/Å)
HV=100kV
Cam Len: 0.2200 m
AMA Analytical Services, Inc

Sample 308006-6B, Chrysotile Structure 1



308006 FDA_103.jpg
Chrysotile Cluster
308006-6b
Cal: 0.001429 µm/pix
15:33 9/7/2019
TEM Mode: Imaging
Microscopist: CD
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 std. frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

400 nm
HV=100kV
Direct Mag: 7200 x
AMA Analytical Services, Inc

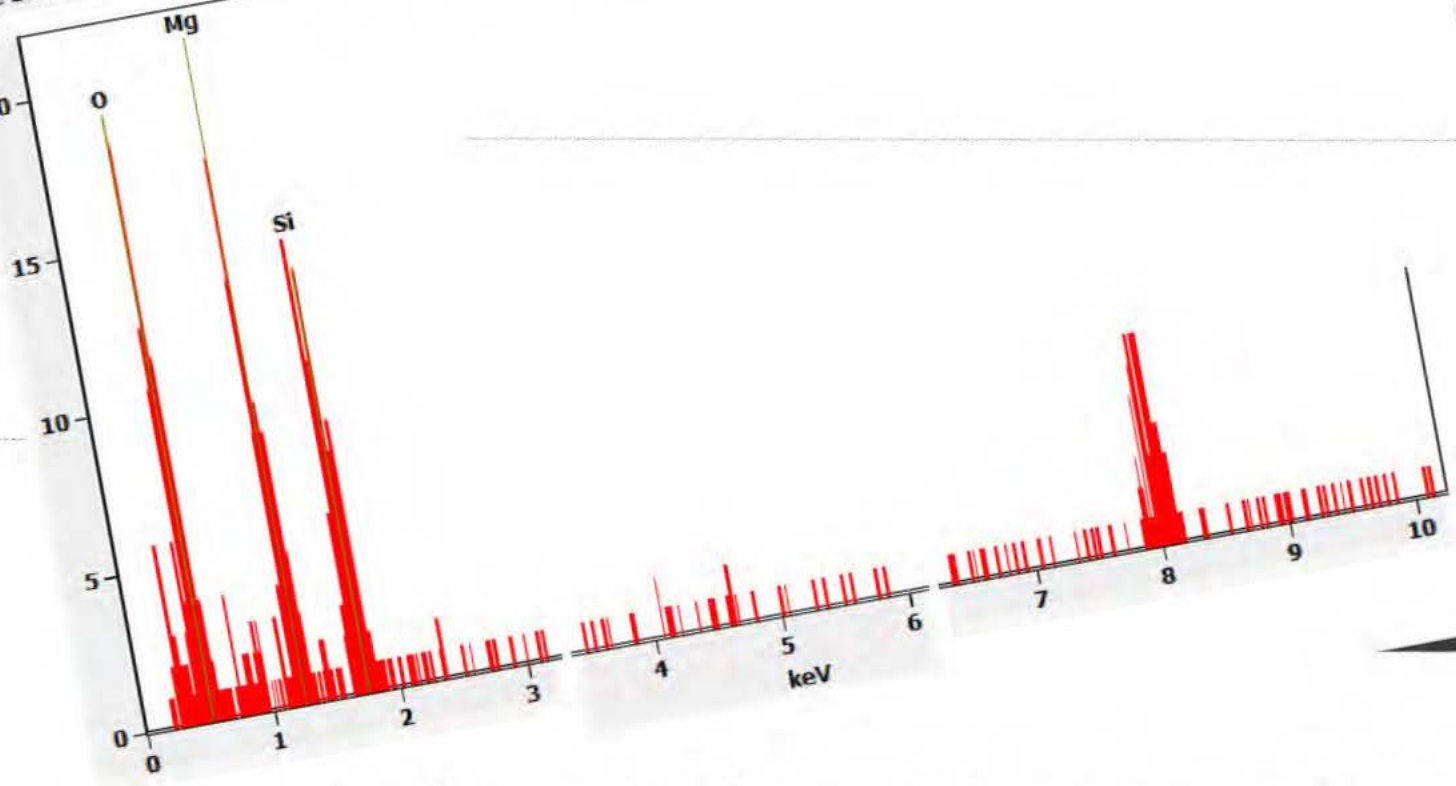
Diffraction Pattern from Chrysotile Structure pictured above

308006 FDA_102.jpg
Chrysotile Dif
308006-6b
15:32 9/7/2019
TEM Mode: Diffraction
Microscopist: CD
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 std. frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

100 (1/Å)
HV=100kV
Cam Len: 0.2200 m
AMA Analytical Services, Inc

mistry from Chrysotile Structure pictured above

scale counts: 22



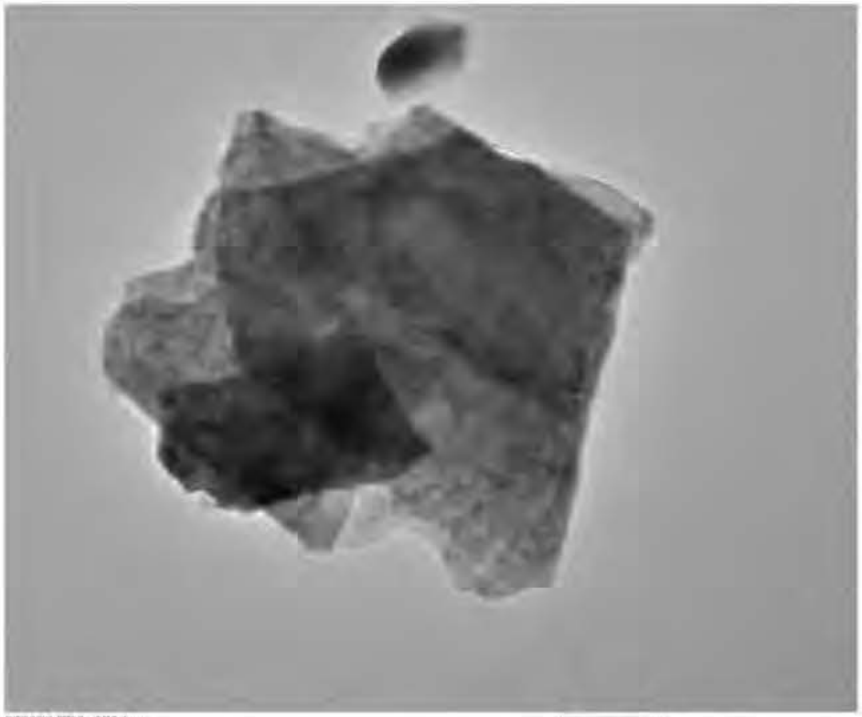
308006-6B, Chrysotile Structure 2

Diffraction Pattern from Chrysotile Structure pictured above

308006 FDA_104.jpg
Chrysotile Dif
308006-6b
16:03 9/7/2019
TEM Mode: Diffraction
Microscopist: CD
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 std. frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

100 (1/Å)
HV=100kV
Cam Len: 0.2200 m
AMA Analytical Services, Inc

308006-6, Talc Particle



308006 FDA_052.jpg
Talc Particle
Cal: 0.001774 µm/pix
17:18 9/3/2019
TEM Mode: Imaging
Microscopist: MG
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

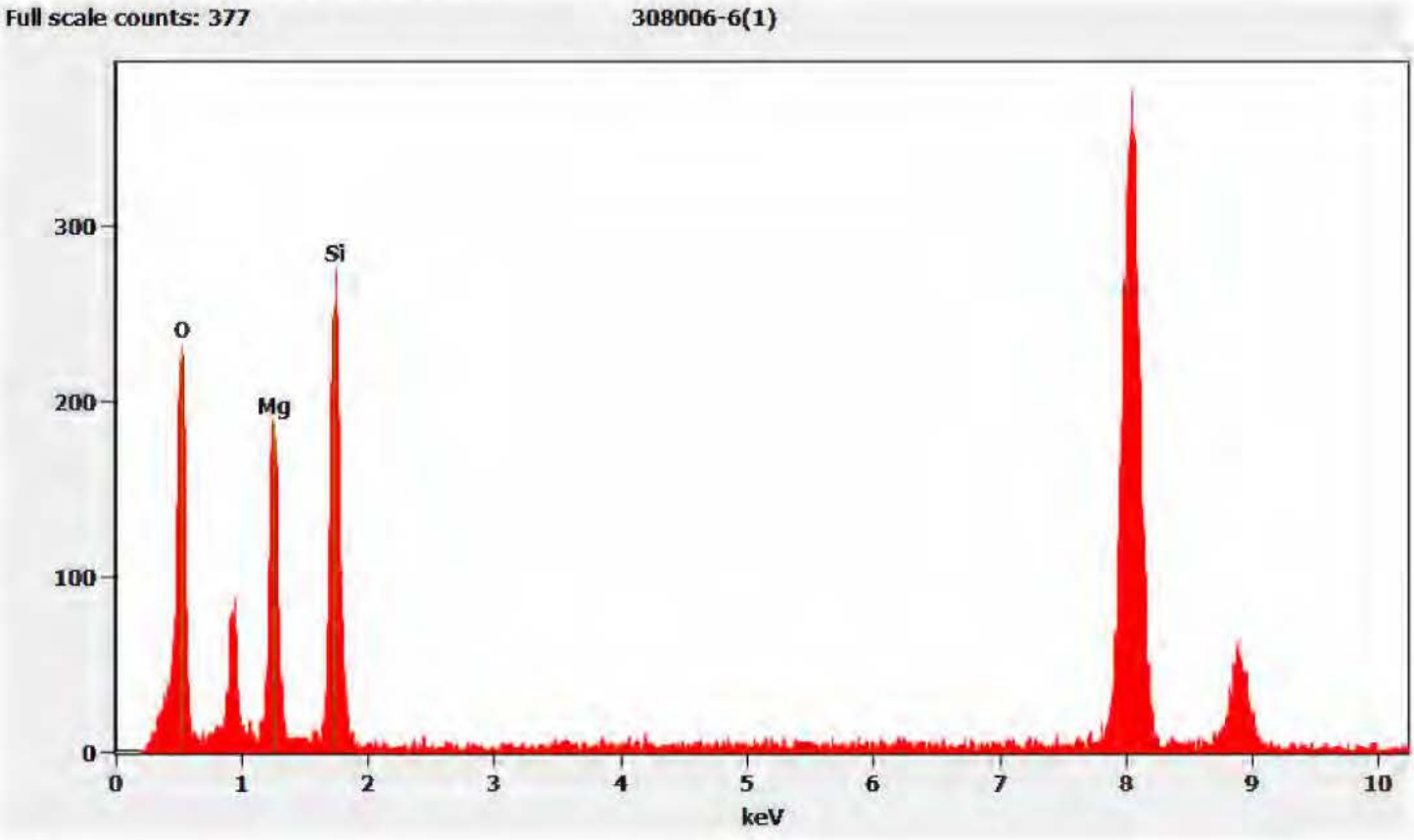
500 nm
HV=100kV
Direct Mag: 5800 x
AMA Analytical Services, Inc

Hexagonal Diffraction Pattern from Talc Particle pictured above

308006 FDA_053.jpg
Talc Particle
17:19 9/3/2019
TEM Mode: Diffraction
Microscopist: MG
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

100 (1/Å)
HV=100kV
Cam Len: 0.2200 m
AMA Analytical Services, Inc

Chemistry from Talc Particle pictured above



306008-6, Mica Particle

308006 FDA_054.jpg
Mica Particle
Cal: 0.001429 µm/pix
17:21 9/3/2019
TEM Mode: Imaging
Microscopist: MG
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

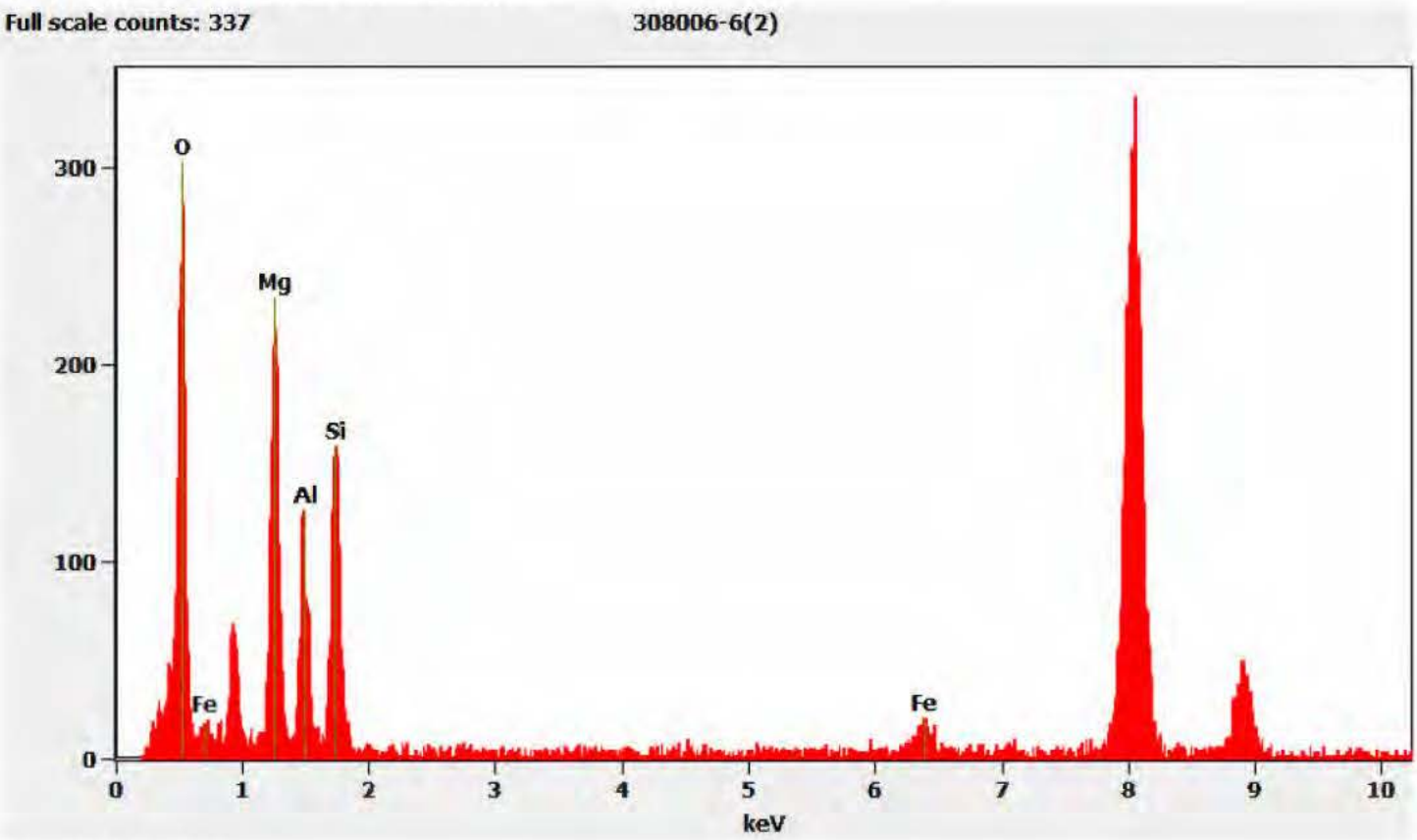
400 nm
HV=100kV
Direct Mag: 7200 x
AMA Analytical Services, Inc

Diffraction Pattern from Mica Particle pictured above

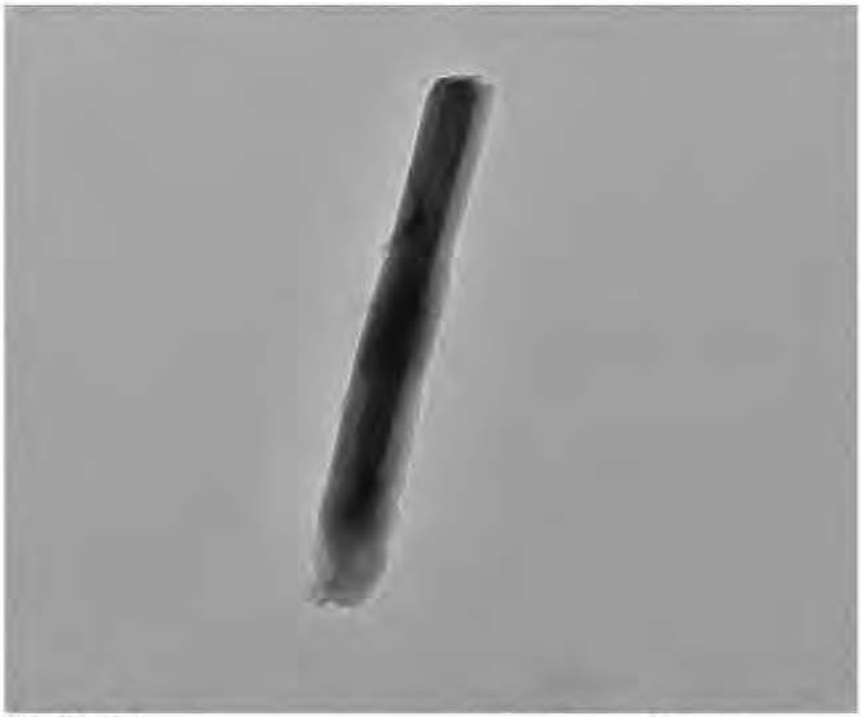
308006 FDA_056.jpg
Mica Particle
17:22 9/3/2019
TEM Mode: Diffraction
Microscopist: MG
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

100 (1/Å)
HV=100kV
Cam Len: 0.2200 m
AMA Analytical Services, Inc

Chemistry from Mica Particle pictured above

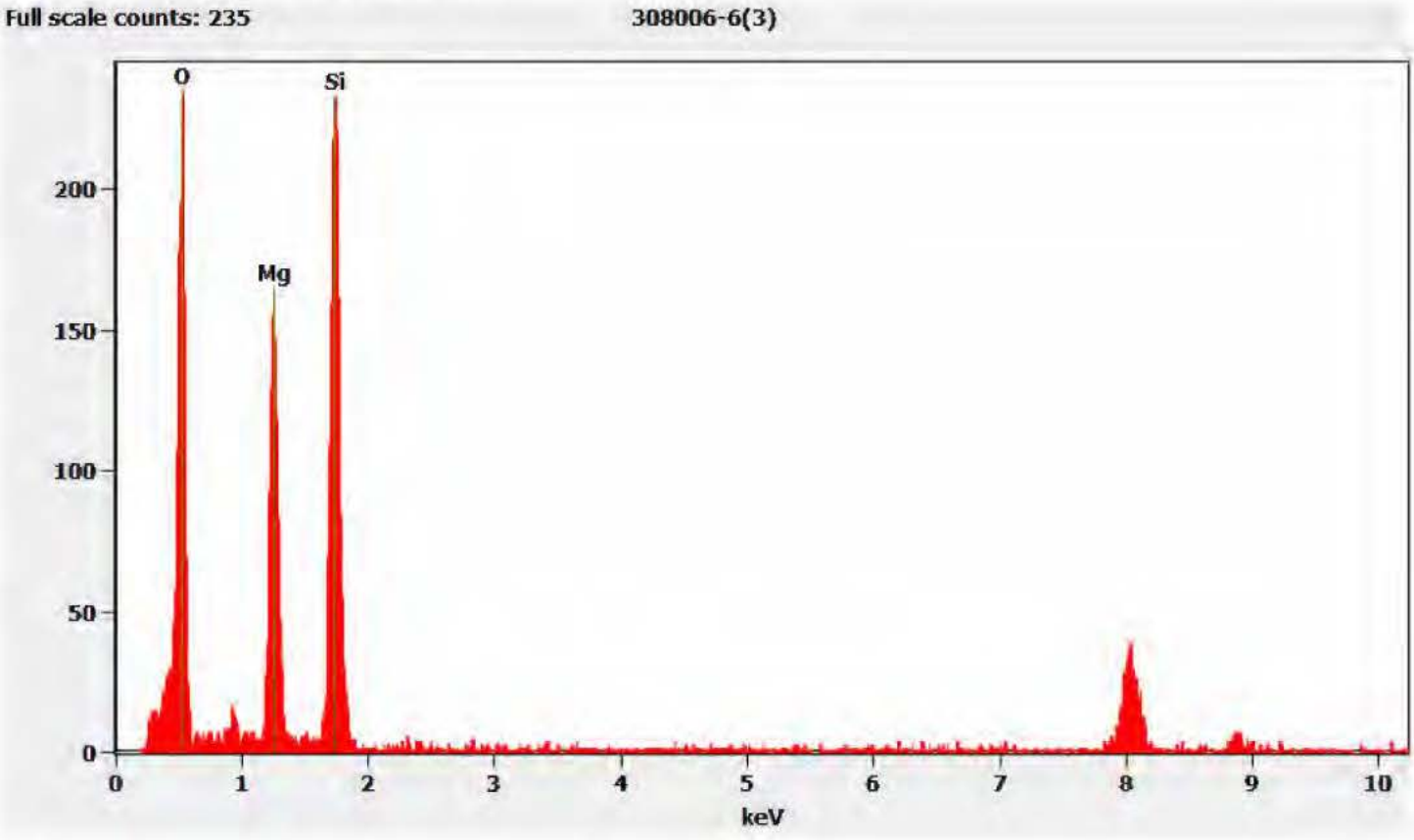


308006-6, Talc Fiber

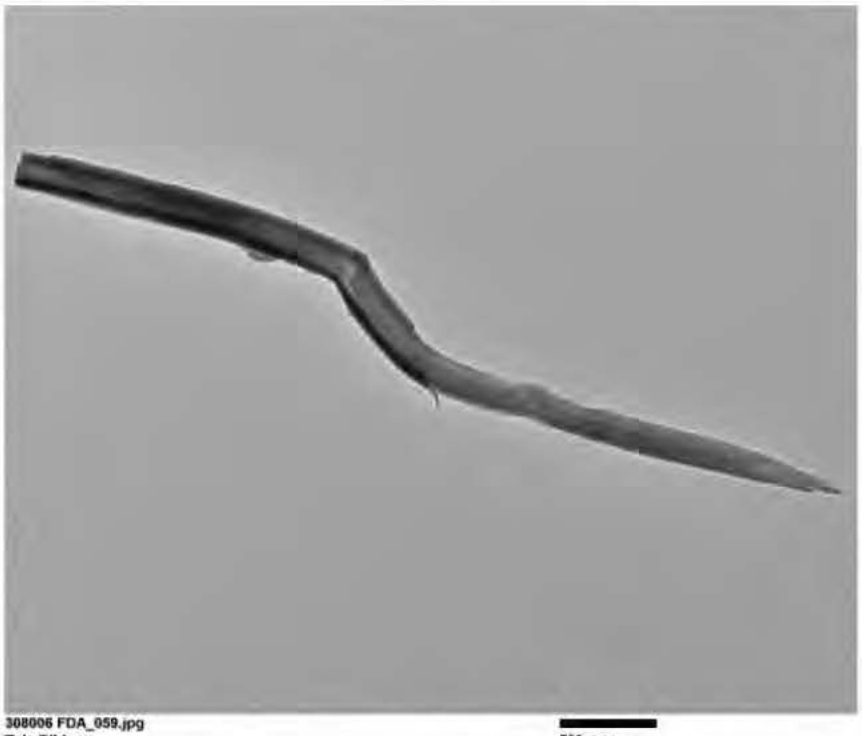


Diffraction Pattern from Talc Fiber pictured above

Chemistry from Talc Fiber pictured above



308006-6, Talc Ribbon



308006 FDA_059.jpg
Talc Ribbon
Cal: 0.001774 µm/pix
17:37 9/3/2019
TEM Mode: Imaging
Microscopist: MG
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

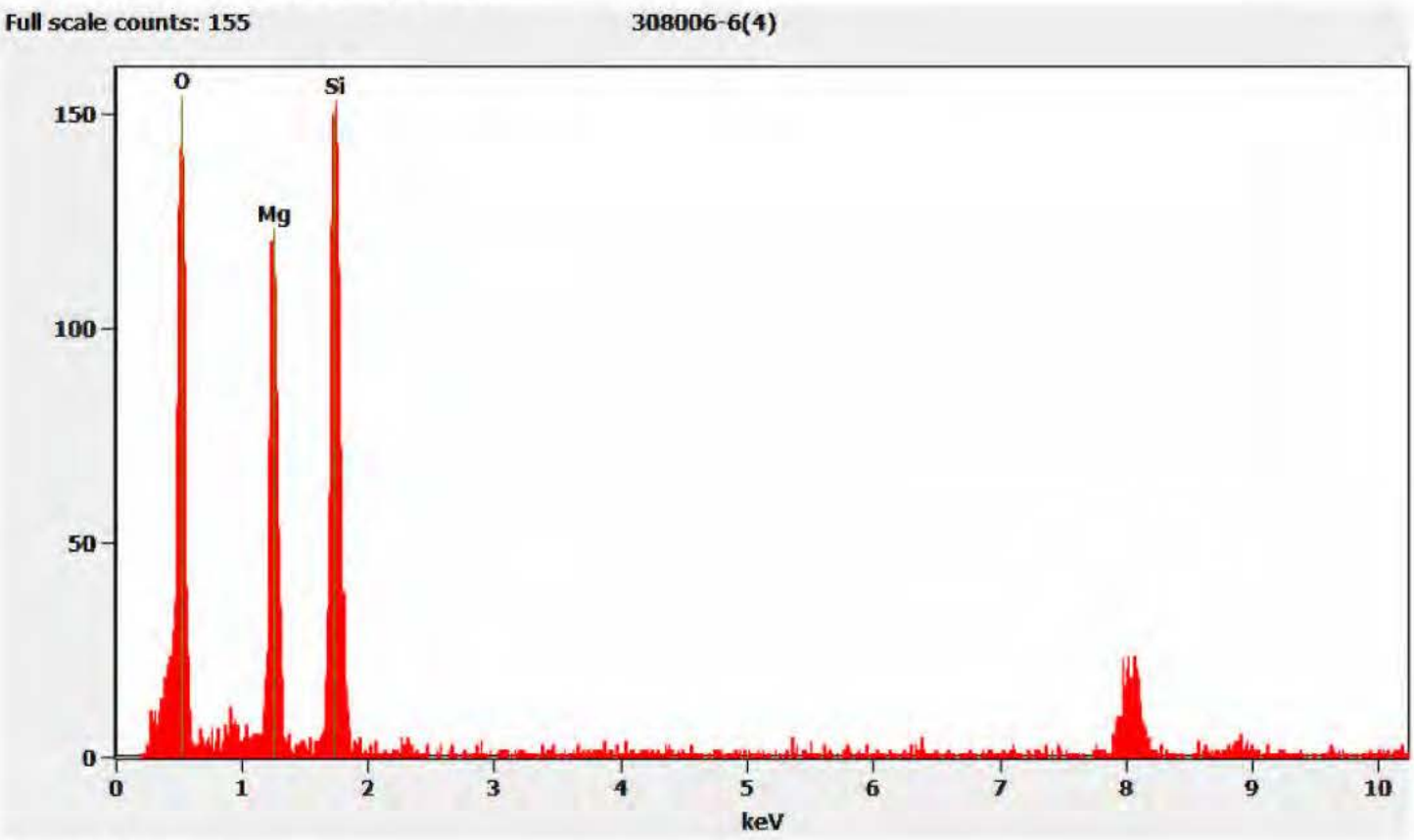
500 nm
HV=100kV
Direct Mag: 5800 x
AMA Analytical Services, Inc

Diffraction Pattern from Talc Ribbon pictured above

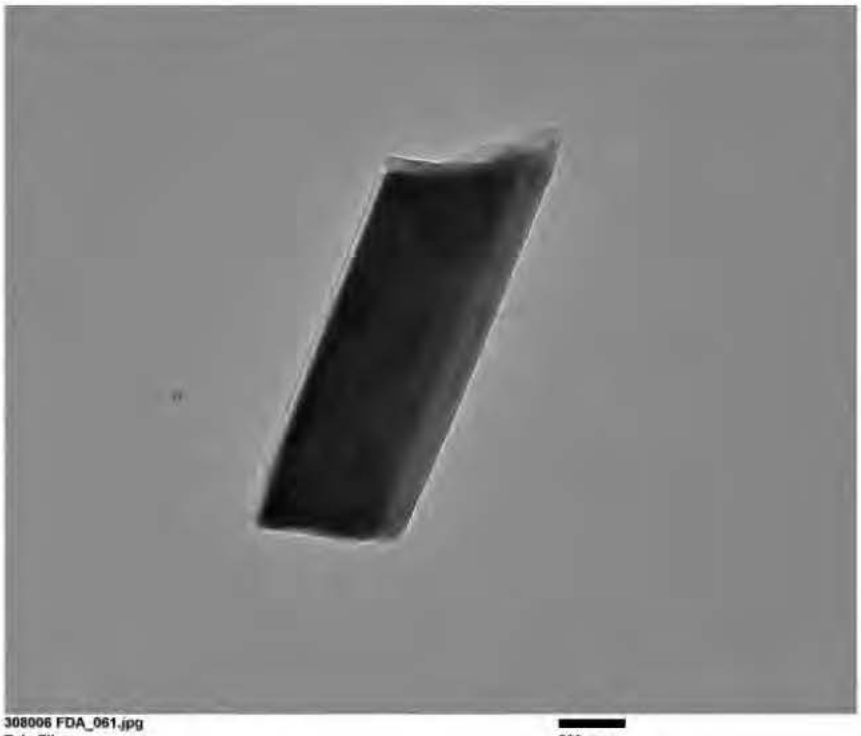
308006 FDA_060.jpg
Talc Ribbon
17:38 9/3/2019
TEM Mode: Diffraction
Microscopist: MG
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

100 (1/Å)
HV=100kV
Cam Len: 0.2200 m
AMA Analytical Services, Inc

Chemistry from Talc Ribbon pictured above

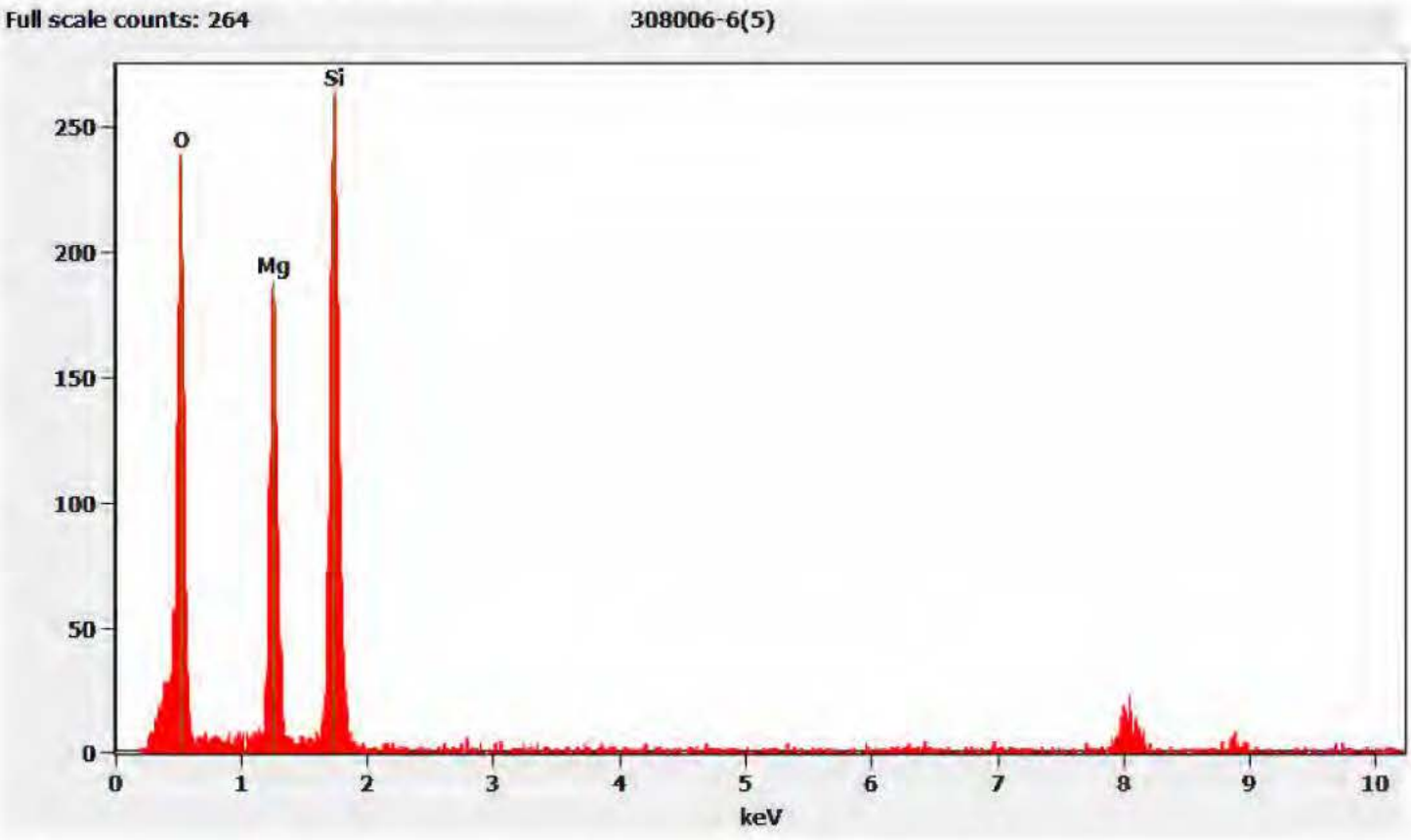


308006-6, Talc Fiber



Diffraction Pattern from Talc Fiber pictured above

Chemistry from Talc Fiber pictured above



QC Discussion:
During preparation, three blank control samples and one reference control sample were prepared. These samples were prepared alongside the customer samples. The blank samples were prepared using Sigma-Aldrich Talc Powder, <10 micron, and was analyzed by (b) (6) on September 18, 2019. No asbestos was detected on the blank samples. The reference sample was made from the same Sigma-Aldrich talc powder spiked with 10% Chrysotile. The reference sample was analyzed by (b) (6) on September 18, 2019 and found to be within acceptable limits. Additionally, filter blanks were prepared with each batch of carbon coated filters. Filter blank number EB-54155 was associated with the carbon coating for samples 308006-6, 6A, 6B/D-58. No asbestos was detected on the filter blank sample.

Our laboratory information management system (LIMS) randomly selected samples 308006-2/D-54 and 308006-15/D-67 for additional replicate QC analysis. Separate preparations were made for PLM and TEM analysis. The replicate QC analysis was performed by (b) (6) on September 13, 2019, 2019 for PLM analysis and by (b) (6) on September 18, 2019 for TEM analysis. The QC results matched the original analysis.

Attachments:
The following items are attached to this case narrative for your reference:

1) Sample Log-In Sheet
2) Daily PLM Scope Calibration Log
3) Refractive Index Oil Calibration Log
4) Daily TEM Scope Calibration Log
5) QC Results Summary
6) Replicate & Duplicate QC Chart for (b) (6) for samples analyzed between 1/1/2019 and 9/18/2019
7) Replicate & Duplicate QC Chart for (b) (6) for samples analyzed between 1/1/2019 and 9/18/2019
8) Replicate & Duplicate QC Chart for (b) (6) for samples analyzed between 1/1/2018 and 9/18/2019
9) Raw Data Sheets
    a. Gravimetric Data
    b. Filtration Worksheets
    c. PLM Analysis
    d. TEM Analysis
    e. QC Samples

I certify that all information contained in this report pertaining to laboratory events, procedures, and protocols is true and accurately describes the handling of this project by AMA Analytical Services, Inc. and its personnel.

10/11/2019

Andreas Saldivar — Date
Laboratory Director

Login Sheet

Client:	US Food & Drug Adm n s ra on	**Job Name:**	Task 3 - Ana ys s of Off c a Samp es	**Chain of Custody:**	308006
Date Submitted:	07/24/2019	**Job Location:**	4 h Group - 15 Samp es	**PO Number:**	SF225201810337P
Due Date:	09/13/2019 5:00 pm	**Job Number:**	CLIN 0001		

AMA Sample Number	Client Sample Number	Analysis Type(s) and Sample Type(s)
(b) (4)	(b)	(b) (4)

AMA Sample Number	Client Sample Number	Analysis Type(s) and Sample Type(s)
308006-6	D-58	PLM: ELAP 198.6 NOB TEM: Bu k Upgrade
308006-6A	D-58	PLM: ELAP 198.6 NOB TEM: Bu k Upgrade
308006-6B	D-58	PLM: ELAP 198.6 NOB TEM: Bu k Upgrade
308006-6C	D-58	PLM: ELAP 198.6 NOB TEM: Bu k Upgrade
(b) (4)	[redacted]	[redacted]

AMA Sample Number	Client Sample Number	Analysis Type(s) and Sample Type(s)
(b) (4)		

Special Instructions:
Use FDA Protocols. Samples are in Asbestos Sample Lock Box (See (b) (6) for Key). ALL PLM & TEM Analysts: Please record the date & amount of time spent analyzing each sample in the comments section of the bench sheet. Please save all pictures, graphs, etc. to L:\Case Narratives\FDA Project\308006

9/30/2019 by (b)(6): Client requested that we analyze a 4th aliquot for sample 308006 6/D58; this was added as 308006 6C
10/1/2019 by (b)(6): Client requested that we cancel their request to analyze 308006 6C, Preparation was mostly complete by the time we received the cancellation notice, but no analysis was performed.

Daily Calibrations for PLM Scope # 2

Date: 9/12/19
Analyst Initials: (b) (6)

Cleaning:
Oculars ✓ Objectives ✓ Field Lens ✓ Mechanical Stage ✓ Main Body ✓

Alignment:
✓ Kohler Illumination (or as close to it as scope allows)
✓ Polarizer & Analyzer at 90 degrees to one another
✓ Polarizer & Analyzer aligned with reticule cross hairs
✓ Axis of rotation of stage centered in field of view
✓ Central stop of the D.S. objective aligned with condenser aperture

Refractive Index Colors of Permanent 1,680 Amosite:
440 Parallel wavelength 660 Perpendicular wavelength

Comments/other procedures performed:

Daily Calibrations for PLM Scope # 2

Date: 9/13/19
Analyst Initials: (b) (6)

Cleaning:
Oculars ✓ Objectives ✓ Field Lens ✓ Mechanical Stage ✓ Main Body ✓

Alignment:
✓ Kohler Illumination (or as close to it as scope allows)
✓ Polarizer & Analyzer at 90 degrees to one another
✓ Polarizer & Analyzer aligned with reticule cross hairs
✓ Axis of rotation of stage centered in field of view
✓ Central stop of the D.S. objective aligned with condenser aperture

Refractive Index Colors of Permanent 1,680 Amosite:
440 Parallel wavelength 660 Perpendicular wavelength

Comments/other procedures performed:

Daily Calibrations for PLM Scope # 2

Date: 9/16/19
Analyst Initials: (b) (6)

Cleaning:
Oculars ✓ Objectives ✓ Field Lens ✓ Mechanical Stage ✓ Main Body ✓

Alignment:
✓ Kohler Illumination (or as close to it as scope allows)
✓ Polarizer & Analyzer at 90 degrees to one another
✓ Polarizer & Analyzer aligned with reticule cross hairs
✓ Axis of rotation of stage centered in field of view
✓ Central stop of the D.S. objective aligned with condenser aperture

Refractive Index Colors of Permanent 1,680 Amosite:
440 Parallel wavelength 660 Perpendicular wavelength

Comments/other procedures performed:

REFRACTIVE INDEX OIL CALIBRATION

DATE	ANALYST	RI OIL	BOTTLE ID	LAB ID	RI BEAD	TEMP (Deg. C)	Dispersion Staining Color/ Becke Line Observations	Matching Wavelength	Measured R.I.
9-3-19	(b) (6)	1.550	1.550X3		1.55	25°	Red, Blue, Margenta	580	1.5505
"		1.680	1.680X3		1.68	"	Blue, Green	620	1.6816
"		1.605	1.605X3		1.60	"	Blue, Light Green	660	1.6041
"		1.625	1.625X3		1.62	"	Blue, Margenta	560	—
"		1.700	1.700X3		1.70	"	Blue, Green, Margenta	580	—

Revision 2, February 19, 2007

DAILY TEM CALIBRATION LOG

Every analyst should confirm alignment prior to analyzing samples.
X-ray analyzer must be calibrated prior to each day's use.
Dewar for x-ray detector is to be filled each Tuesday and Friday.

Note: Please enter code letters in Type Column.

Type of Analysis:

Routine Analysis	A
Quality Control	QC
Training	T
Research	R
Other (Explain)	O

TRANSMISSION ELECTRON MICROSCOPE								X-RAY ANALYZER	
DATE	NAME	SYSTEM/ ALIGN. CHECK	ACTUAL "BEAM TIME" USED ON	OFF	TOTAL MINUTES	TOTAL # SAMPLES	TYPE	EDXA CAL. (AL/CU)	DEWAR LN2 (INIT)
9/3/19	(b) (6)	OK							
9/4/19	(b) (6)	ok	Filament Changed						
9/5/19	(b) (6)	OK						Cu OK	
9/6/19	(b) (6)	ok	0900			12	A		
9/7/19	(b) (6)	ok	1230			17	A		
9/8/19	(b) (6)	OK							
9/9/19	(b) (6)	OK							
9/10/19	(b) (6)	OK							
9/11/19	(b) (6)	OK							
9/12/19	(b) (6)	OK							

Version 2-1:9/90

Revision 0, Issued Oct. 2004

Chain Of Custody #308006

+ Add CoC

General Samples Documents QC Results

QC Samples

Date Analyzed	Sample Number	Original PLM Analyst	Original PLM Result	PLM QC Result	PLM QC Analyst	PLM R Value	Original TEM Analyst	Original TEM Result	TEM QC Result	TEM QC Analyst	TEM R Value	Comments
09/09/2019	308006-16RQC	(b) (6)	0.00		SW	0.00	MG	0.00	0.00	CD	0.00	Analysis 9/18/19
09/09/2019	308006-17RQC		0.00		SW	0.00	MG	0.00	0.00	CD	0.00	Analysis: 9/18/19

Reference Samples

Sample Number	Tile #	Analyst	Asbestos Type	Percent Asbestos	Result	Created Date	Comments
Talc Ref	Talc Ref 10%	(b) (6)	Chrysotile	10.00	Pass	09/18/2019	

Blanks

Blank Number	Date		Asbestos Percentage	Asbestos Type	Comments
NB19-646	09/18/2019	(b) (6)	0.0		
NB19-645	09/18/2019		0.0		
NB19-647	09/18/2019		0.0		

PLM Error(s)

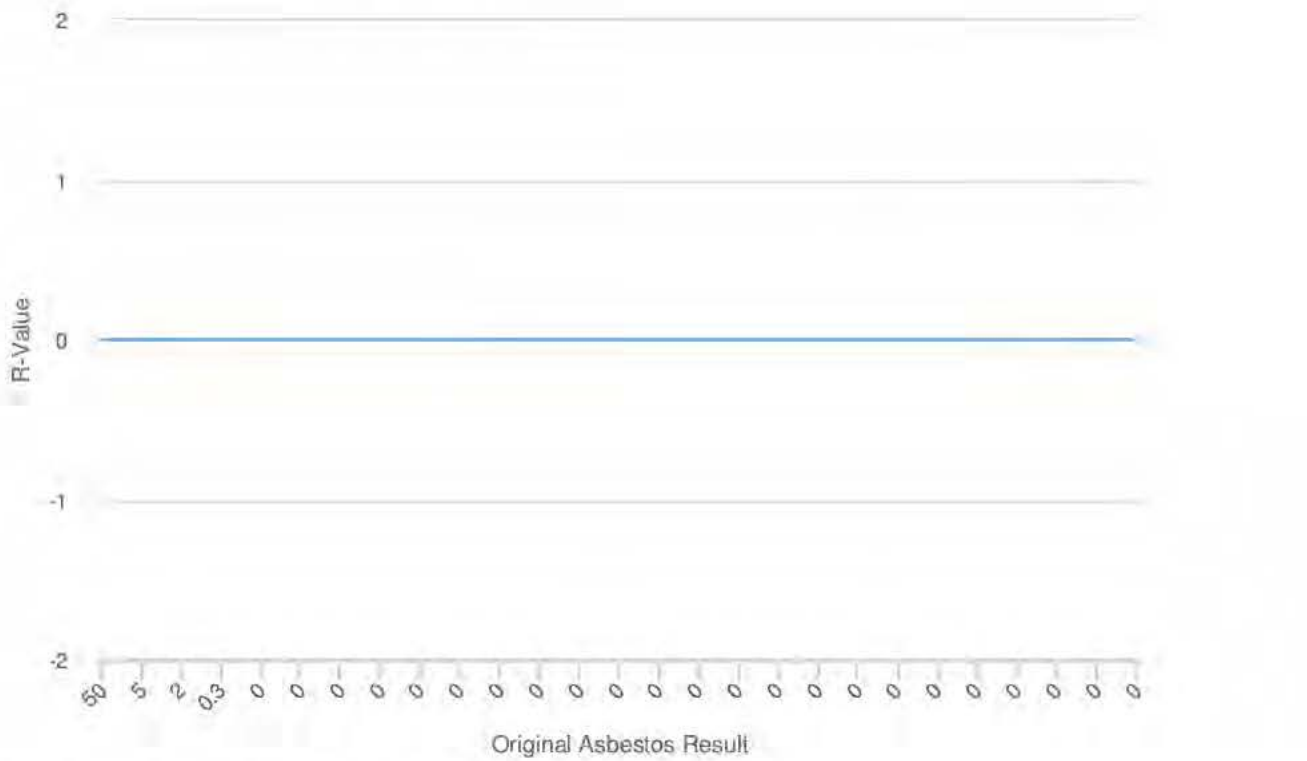
No Results

TEM NOB Error(s)

No Results

PLM QC Chart

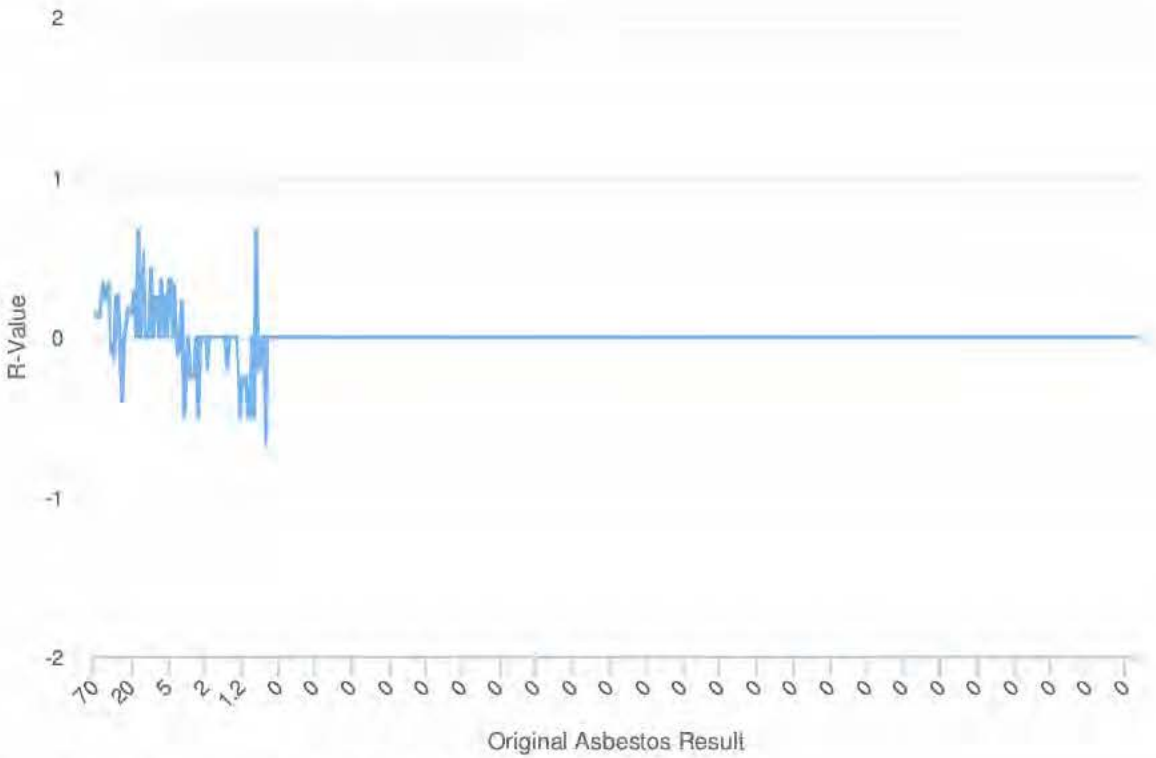
QC Type: Duplicate
Analyst: (b) (6)
Dates Analyzed: *01/01/2019 - 09/18/2019*



R = [Original Result] - [QC Result] / [Average]

PLM QC Chart

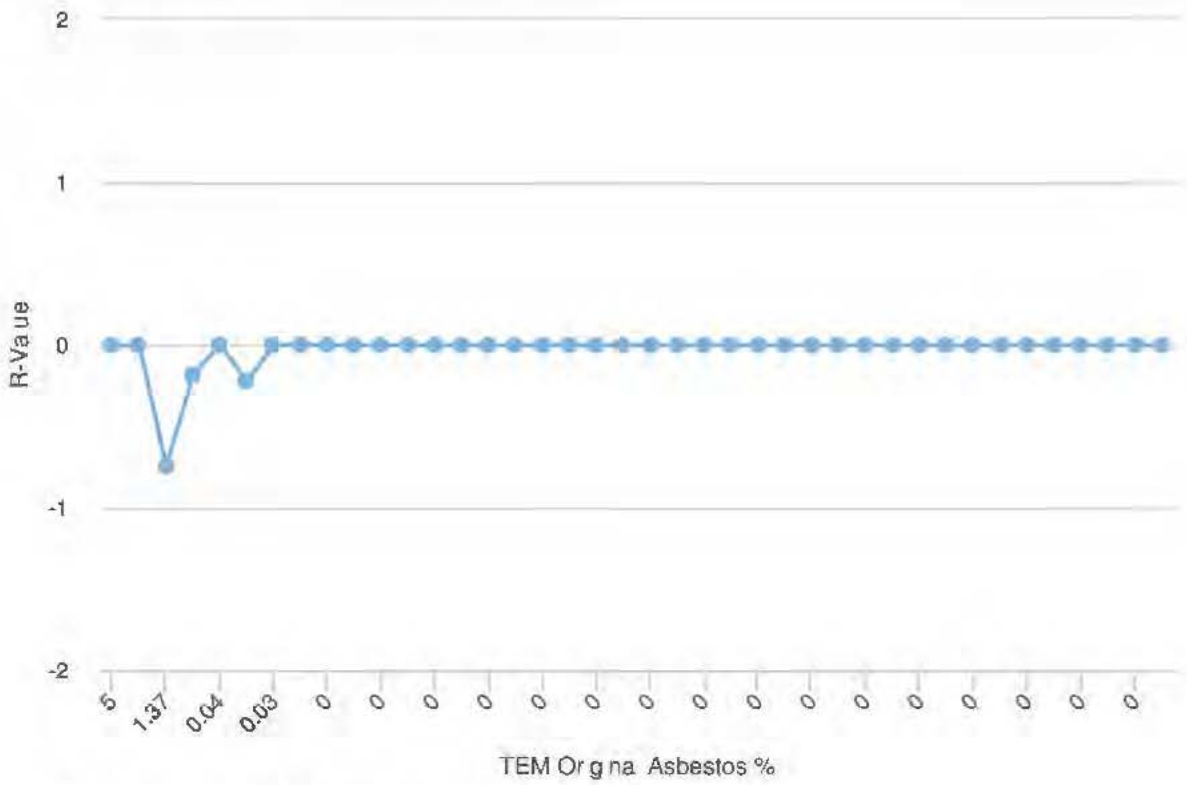
QC Type: Replicate
Analyst: (b) (6)
Dates Analyzed: *01/01/2019 - 09/18/2019*



R = [Original Result] - [QC Result] / [Average]

NOB QC Chart

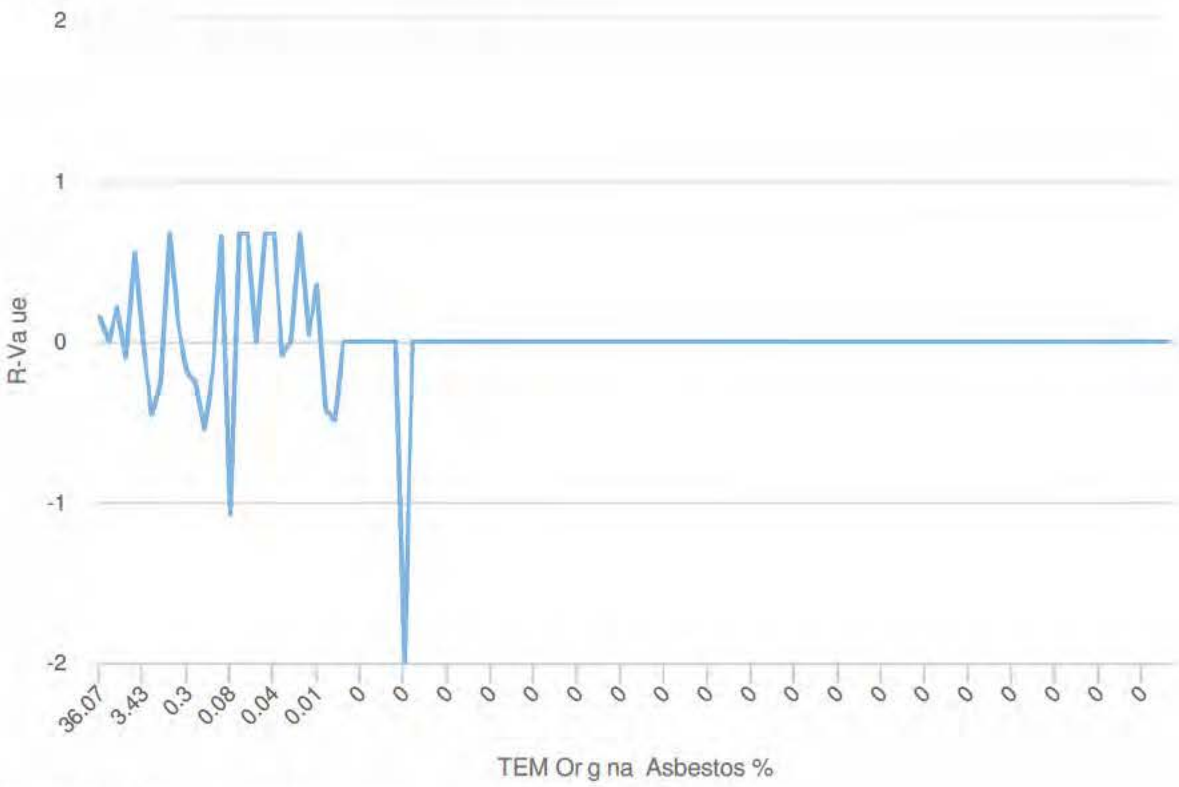
QC Type: Duplicate
Analyst (b) (6)
Dates Analyzed: *01/01/2019 - 09/18/2019*



R = [Or g na Resu t] - [QC Resu t] / [Average]

NOB QC Chart

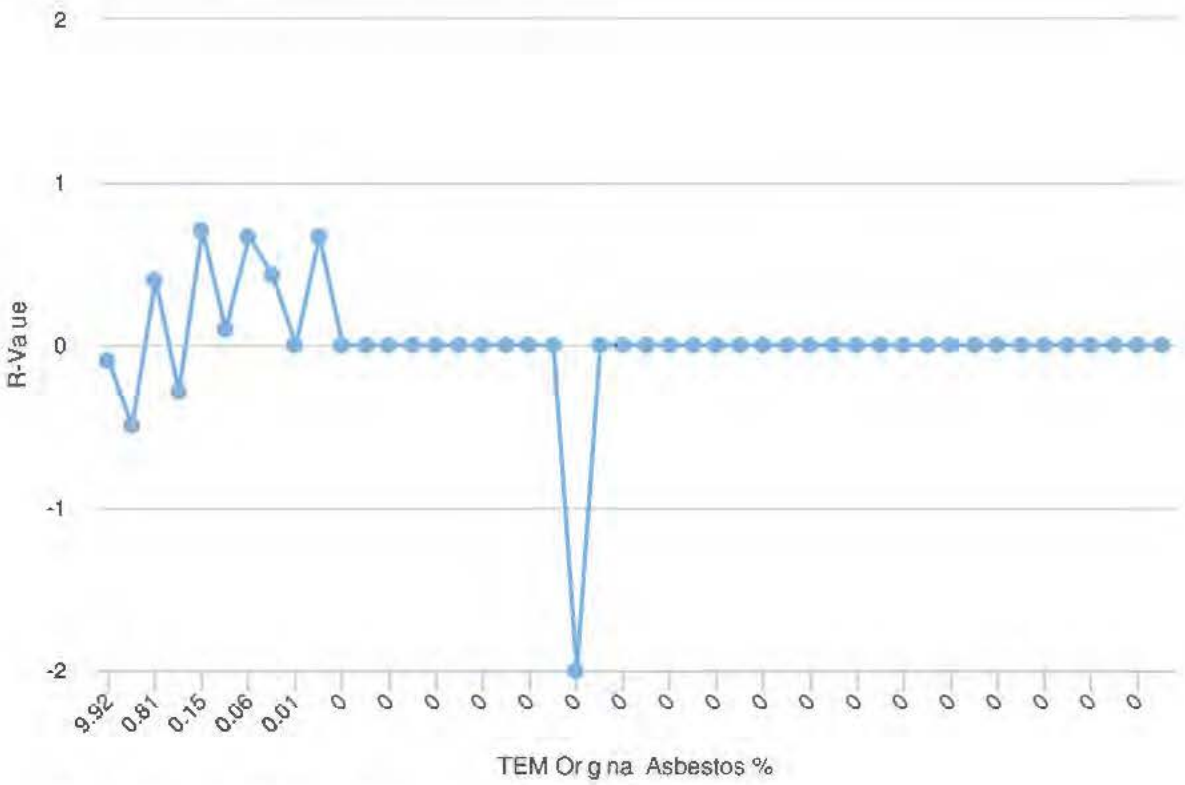
QC Type: Replicate
Analyst: (b) (6)
Dates Analyzed: *01/01/2019 - 09/18/2019*



R = [Or g na Resu t] - [QC Resu t] / [Average]

NOB QC Chart

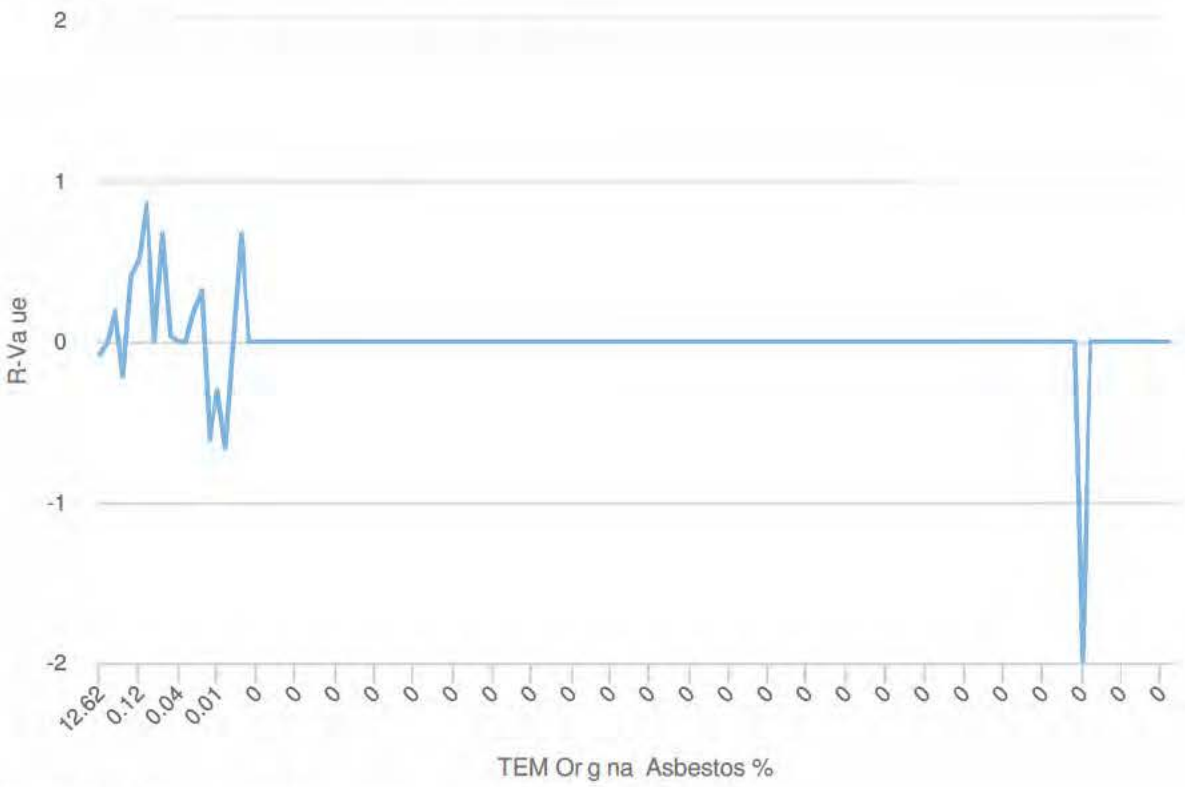
QC Type: Duplicate
Analyst: (b) (6)
Dates Analyzed: *01/01/2019 - 09/18/2019*



R = [Or g na Resu t] - [QC Resu t] / [Average]

NOB QC Chart

QC Type: Replicate
Analyst: (b) (6)
Dates Analyzed: *01/01/2019 - 09/18/2019*



R = [Or g na Resu t] - [QC Resu t] / [Average]

AMA Analytical Services, Inc.
Focused On Results.

Gravimetric Reduction and Filtration Bench Sheet for Modified ELAP 198.4

TEM Preparations

COC #: 3080066

Date: 8/13/2019 & 9/30/19

Client: USFDA

Prep By: (b) (6)

Filter Type: 47 mm, 0.22 µm, MCE EFA: 1047 mm²

Filtered By: (b) (6) Lot #: R9CA03145; Date: See margin 8/19/19

AMA Sample ID	Gravimetric Reduction Weights: Mass (g) Vial	Mass (g) Vial & Sample	Mass (g) Post Ash Vial & Sample	Filtration Weights: Mass (g) Filter & Petri Dish	Mass (g) Post Acid Wash Filter & Petri Dish	Mass (g) 100mL Jar w/ Lid	Mass (g) 100mL Jar w/ Lid & Sample Residue	Filtration Volumes: Initial Volume (mL)	Volume Filtered (mL)	Serial Dilution Initial Volume (mL) (If left blank, no serial dilution performed)	Serial Dilution Volume Filtered (mL) (If left blank, no serial dilution performed)	Serial Dilution Final Volume (mL) (If left blank, no serial dilution performed)
(b) (4)												=
												=
												=
												=
												=
												=
												=
												=
												=
												=
												=
												=
												=
												=
												=
3080066	7.2520	7.6844	7.6833	6.0877	6.4902	19.6991	19.8935	100	.2			=
-6A	7.1474	7.6335	7.6324	6.0891	6.4791	19.7148	19.8949	100	.2			=
-6B	7.1687	7.6634	7.6622	6.0903	6.5285	19.8223	20.0189	100	.2			=
(b) (4)												=

8/19/2019

9/30/2019

AMA Analytical Services, Inc.
Focused On Results.

Gravimetric Reduction and Filtration Bench Sheet for Modified ELAP 198.4

TEM Preparations

COC #: 308006

Client: USFDA

Date: 8/13/19 & 9/30/19

Prep By: (b) (6) - WSH 10/9/19

Filter Type: 47 mm, 0.22 µm, MCE EFA: 1047 mm²

Filtered By: (b) (6) Lot #: R9CA03145; Date: See margin - WSH 10/9/19

AMA Sample ID	Gravimetric Reduction Weights: Mass (g) Vial	Mass (g) Vial & Sample	Mass (g) Post Ash Vial & Sample	Filtration Weights: Mass (g) Filter & Petri Dish	Mass (g) Post Acid Wash Filter & Petri Dish	Mass (g) 100mL Jar w/ Lid	Mass (g) 100mL Jar w/ Lid & Sample Residue	Filtration Volumes: Initial Volume (mL)	Volume Filtered (mL)	Serial Dilution Initial Volume (mL) (if left blank, no serial dilution performed)	Serial Dilution Volume Filterd (mL) (if left blank, no serial dilution performed)	Serial Dilution Final Volume (mL) (if left blank, no serial dilution performed)
(b) (4)												=
												=
												=
												=
												=
												=
												=
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												=
												=

8/30/2019

9/5/2019

AMA Analytical Services, Inc.
Focused On Results.

Gravimetric Reduction and Filtration Bench Sheet for Modified ELAP 198.4

TEM Preparations

COC #: 308006

Client: USFDA

Date: 8/13/19 + 9/30/19

Prep By: (b) (6) - BMH 10/8/19

Filter Type: 47 mm, 0.22 µm, MCE EFA: 1047 mm²

Filtered By: (b) (6) Lot #: R9CA03145; Date: See margin - BMH 10/8/19

AMA Sample ID	Gravimetric Reduction Weights: Mass (g) Vial	Mass (g) Vial & Sample	Mass (g) Post Ash Vial & Sample	Filtration Weights: Mass (g) Filter & Petri Dish	Mass (g) Post Acid Wash Filter & Petri Dish	Mass (g) 100mL Jar w/ Lid	Mass (g) 100mL Jar w/ Lid & Sample Residue	Filtration Volumes: Initial Volume (mL)	Volume Filtered (mL)	Serial Dilution Initial Volume (mL) (if left blank, no serial dilution performed)	Serial Dilution Volume Filtered (mL) (if left blank, no serial dilution performed)	Serial Dilution Final Volume (mL) (if left blank, no serial dilution performed)
(b) (4)												=
												=
												=
												=
												=
												=
												=
												=
												=
												=
NB19-645	7.2023	7.5055	7.5051	6.0260	6.2879	19.6999	19.8381	100	.2			=
NB19-646	7.1965	7.4457	7.4452	6.0241	6.2595	19.8538	19.9748	100	.2			=
NB19-647	7.1488	7.5222	7.5218	6.0210	6.3629	19.8468	20.0132	100	.2			=
RB									10 %			=
9/30/19												=
308006-66	7.1387	7.5613	7.5601	61753	6.5876	20.8765	21.0656	100	.2			=
NB19-699	7.2313	7.5347	7.5335	6.2610	6.5578	19.8215	19.9581	100	.2			=
												=
												=

9/5/2019

1 page/VA

AMA Analytical Services, Inc.
Focused On Results.

Gravimetric Reduction Bench Sheet

Modified ELAP 198.6

PLM Preparations

COC #: 308006 Date: 8/13/2019

Client: USFDA Prep By: [redacted]

AMA Sample ID	Mass (g) Vial	Mass (g) Vial & Sample	Mass (g) Post Ash Vial & Sample	Mass (g) Filter & Petri Dish	Mass (g) Post Acid Wash Filter & Petri Dish
[redacted]					
308006-6	7.1829	7.6946	7.6932	6.2233	6.6973
-6A	7.1097	7.5536	7.5526	6.1705	6.5757
-6B	7.2309	7.7182	7.7167	6.2312	6.6900
[redacted]					

8/19/2019 = TEM Filtration Date - [illegible]

8/30/2019 = TEM Filtration Date - [illegible]

AMA Analytical Services, Inc.
Focused On Results.

Gravimetric Reduction Bench Sheet
Modified ELAP 198.6

PLM Preparations

COC #: 308006

Date: 9/13/2019 - BMH 10/5/19

Client: USFDA

Prep By: (b) (6)

AMA Sample ID	Mass (g) Vial	Mass (g) Vial & Sample	Mass (g) Post Ash Vial & Sample	Mass (g) Filter & Petri Dish	Mass (g) Post Acid Wash Filter & Petri Dish
(b) (4)					
NB19- 645	7.2023	7.5055	7.5051	6.0260	6.2879
NB19- 646	7.1965	7.4457	7.4452	6.0241	6.2595
NB19- 647	7.1488	7.5222	7.5218	6.0210	6.3629
RB					10 %

8/30/2019 - BMH Elevation Date - BMH 10/5/19

9/5/2019 - BMH Elevation Date - BMH 10/5/19

Edit Sample #308006-6 (D-58)

General | PLM: ELAP 198.6 NOB | TEM: Bulk Upgrade

Initial Sample Weight

Vial Weight	7.282
Vial + Sample Weight	7.8844
Initial Sample Weight	0.432

Post Ash Weight

Vial + Ashed Sample Weight	7.6833
Ashed Sample Weight	0.431

Post Acid Treatment

Filter Tare	5.8877
Gross Filter Weight	6.4902
Weight Residue	0.402

Visual Estimations

Estimated Asbestos PLM	0.5

Final Asbestos Percents

Percent Asbestos PLM	NAD

Final Non-Asbestos %

Percent Organics	0.254
Percent Acid Soluble	6.66
Percent Other	93.085

Material Type: Powder | Color: White | Homogeneity: Homogeneous

Sample Type: Whole | Texture: Powdery

- [ ] Sample was not analyzed
- [ ] Fibrous
- [x] Scanning Negative Option
- [ ] Stratified Point Count
- [ ] 400 Point Count

Macro Asbestos %		Macro Non Asbestos %		Macro Non Fibrous %	
Chrysotile Percent		Mineral Wool Percent		Particulate	100.0
Amosite Percent		Fiberglass		Other Non-Fibrous	
Crocidolite Percent		Organic			
Other Asbestos Percent		Synthetic			
		Other			

Macroscopic Comments

Temperature (°C): 26.0 | Microscope #: 2

Morphology	Color	Elongation	Est. Avg	Dispersion Staining 1	Dispersion Staining 2	Refractive Index 1	Refractive Index 2	Pleochroism	Birefringence	Refractive Index Oil	Mount

Add Row

Slide 1		Slide 2		Slide 3		Slide 4		Slide 5		Slide 6		Slide 7		Slide 8	
Mount	Fiber #	Mount	Fiber #	Mount	Fiber #	Mount	Fiber #	Mount	Fiber #	Mount	Fiber #	Mount	Fiber #	Mount	Fiber #

Add Row

Asbestos Points

Chrysotile Points		Pending	Trace Detected but no points
Amosite Points		Pending	Trace Detected but no points
Crocidolite Points		Pending	Trace Detected but no points
Other Asbestos Points		Pending	Trace Detected but no points
Total Asbestos Points	Pending	NAD	< 0.25%
Total Points	Pending		

Non Asbestos Fiber Percentages		Non Fibrous Percentages	
Mineral Wool + Fiberglass Combined		Particulate	100.0
Organic		Other Non-Fibrous	
Synthetic			
Other			

- [ ] Recommend TEM
- [ ] Recommend Matrix Reduction

Microscopic Comments

Report Comments

- [ ] Error(s) Found During Review

Edit Sample #308006-6 (D-58)

General | PLM: ELAP 198.6 NOB | TEM: Bulk Upgrade

Initial Sample Weight		Post Ash Weight		Post Acid Treatment	
Vial Weight	7.252	Vial + Ashed Sample Weight	7.6833	Filter Tare	6.0877
Vial + Sample Weight	7.6844	Ashed Sample Weight	0.431	Gross Filter Weight	6.4902
Initial Sample Weight	0.432			Weight Residue	0.402

Visual Estimations		Final Asbestos Percents		Final Non-Asbestos %	
Estimated Asbestos PLM	0.0	Percent Asbestos PLM	NAD	Percent Organics	0.254
Estimated Asbestos TEM	0.0	Percent Asbestos TEM	NAD	Percent Acid Soluble	6.66
				Percent Other	93.085

Sample Type	Material Type	Sample Color
Whole		

Grid Box	Row and Slots	Microscope #
A19-433	1ab	1

Working Mag. High (K)	Working Mag. Low (K)	Accelerating Voltage (KV)
15		100

☐ Sample was not analyzed

Structure Chrystallographic and Photographic Data

Structure #	SAED	Elements	Neg. #	Camera Length / Mag.	Ident.
5	Hex	Mg,Si, Talc Fiber		10	
4	Neg	Mg,Si- Talc Ribbon		5.8	
3	Hex	Mg,Si, Talc Fiber		14	
2	Hex	Mg,Al,Si,Fe- Mica F		7.2	
1	Hex	Mg Si- Talc Particle		5.8	

Add Row

Grid #1 Estimated Asbestos	Grid #2 Estimated Asbestos	Estimated Asbestos %
0.0	0.0	0.0%

Analyst Comments

f orientation - 250, 115
Grid A analyzed 9/3/19, Grid B analyzed 9/4/19,
Analytical time = 1.5hrs.

Report Comments

☐ Error(s) Found During Review

Edit Sample #308006-6A (D-58)

General | PLM: ELAP 198.6 NOB | TEM: Bulk Upgrade

Initial Sample Weight

Vial Weight	7.1474
Vial + Sample Weight	7.6236
Initial Sample Weight	0.486

Post Ash Weight

Vial + Ashed Sample Weight	7.6324
Ashed Sample Weight	0.485

Post Acid Treatment

Filter Tare	5.[illegible]1
Gross Filter Weight	5.4791
Weight Residue	0.390

Visual Estimations

Estimated Asbestos PLM	0.0

Final Asbestos Percents

Percent Asbestos PLM	NAD

Final Non-Asbestos %

Percent Organics	0.226
Percent Acid Soluble	19.543
Percent Other	80.232

Material Type: Powder | Color: White | Homogeneity: Homogeneous

Sample Type: Whole | Texture: Powdery

- [ ] Sample was not analyzed
- [ ] Fibrous
- [x] Scanning Negative Option
- [ ] Stratified Point Count
- [ ] 400 Point Count

Macro Asbestos %		Macro Non Asbestos %		Macro Non Fibrous %	
Chrysotile Percent		Mineral Wool Percent		Particulate	100.0
Amosite Percent		Fiberglass		Other Non-Fibrous	
Crocidolite Percent		Organic			
Other Asbestos Percent		Synthetic			
		Other			

[illegible] Comments

Temperature (°C): 26.0 | Microscope #: 2

Morphology	Color	Elongation	Est. Avg	Dispersion Staining		Refractive Index		Flow	Birefringence	Refractive Index Oil	Mount
											Add Row

Slide 1		Slide 2		Slide 3		Slide 4		Slide 5		Slide 6		Slide 7		Slide 8	
Mount	Fiber/5	Mount	Fiber/5	Mount	Fiber/5	Mount	Fiber/5	Mount	Fiber/5	Mount	Fiber/5	Mount	Fiber/5	Mount	Fiber/5
															Add Row

Asbestos Points

Chrysotile Points		Pending	Trace Detected but no points
Amosite Points		Pending	Trace Detected but no points
Crocidolite Points		Pending	Trace Detected but no points
Other Asbestos Points		Pending	Trace Detected but no points
Total Asbestos Points	Pending	NAD	< 0.25%
Total Points	Pending		

Non Asbestos Fiber Percentages		Non Fibrous Percentages	
Mineral Wool + Fiberglass Combined		Particulate	100.0
Organic		Other Non-Fibrous	
Synthetic			
Other			

- [ ] Recommend TEM
- [ ] Recommend Matrix Reduction

Microscopic Comments

Report Comments

- [ ] Error(s) Found During Review

Edit Sample #308006-6A (D-58)

General | PLM: ELAP 198.6 NOB | TEM: Bulk Upgrade

Initial Sample Weight

Vial Weight	7.1474
Vial + Sample Weight	7.6335
Initial Sample Weight	0.486

Post Ash Weight

Vial + Ashed Sample Weight	7.6324
Ashed Sample Weight	0.485

Post Acid Treatment

Filter Tare	6.0891
Gross Filter Weight	6.4791
Weight Residue	0.390

Visual Estimations

Estimated Asbestos PLM	0.0
Estimated Asbestos TEM	0.01

Final Asbestos Percents

Percent Asbestos PLM	NAD
Percent Asbestos TEM	0.008

Final Non-Asbestos %

Percent Organics	0.226
Percent Acid Soluble	19.543
Percent Other	80.222

Sample Type	Material Type	Sample Color
Whole		

Grid Box	Row and Slots	Microscope #
a19-433	2ab	1

Working Mag. High (K)	Working Mag. Low (K)	Accelerating Voltage (KV)
15		100

☐ Sample was not analyzed

Structure Chrystallographic and Photographic Data

Structure #	SAED	Elements	Neg. #	Camera Length / Mag.	Ident.	
5	hex					✖
4	uto (0.4x0.05) Grib	uto	101	5.8	Chrysotile	✖
3	pos (1.75x0.06) Gri	Si Mg	100	0.22/5.8	Chrysotile	✖
2	neg					✖
1	hex					✖

Add Row

Grid #1 Estimated Asbestos	Grid #2 Estimated Asbestos	Estimated Asbestos %
0.01	0.01	0.01%

Analyst Comments

Analysis: 9/7/19, time = 1hr 10 mins
F Orientation: 285,40

Report Comments

☐ Error(s) Found During Review

AMA Analytical Services, Inc.
Focused On Results.

Fiber Count Sheet for Transmission Electron Microscopy

Page 1 of 1

Date: 09/07/19
Client ID: D-58
Filter Size/Type/Porosity 47mm MCE 0.22 µm
Client: FDA
COC #: 308006
AMA ID #: 308006-6A
Analyst (b) (6) [redacted]
Working Mag: 15 K
Accel Voltage: 100 kV
Orientation of Letter F: F, F
Grid Box #: A19-433
Grid Acceptable: [✓ Y] N
Volume Filtered: 0.2 mL
Signed
Row #: 2
Grid: A / B /
Grid Openings to Observe: 20
Method: Mod. NY ELAP 198.4 (FDA Procedures)

Grid Opening # Location	Structure #	Length & Width (in microns)		SAED # Rows	Mineral Type	Elements ID'd (If left blank, no elements ID'd)	Filter Neg # (If left blank, no photo was taken)	SAED Neg # (If left blank, no photo was taken)	Magnification or Camera Length (If left blank, no photo was taken)	
C2 B	1	1.75	0.06	pos	Chrysotile	Si, Mg	101	100	5.8 K	cm
C2 B	2	0.4	0.05	Uto	Chrysotile		101		5.8 K	cm
☐									K	cm
☐									K	cm
☐									K	cm
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☐									K	cm

Legend: NSD = No Structures Detected UTO = Unable to Obtain ☐ = 1st Grid ☒ = 2nd Grid

# of Structures Counted: 2

Mineral Type: Chrysotile = Total 2
Mineral Type: =
Mineral Type: =
Mineral Type: =

Total # of Grid Openings Observed: 20 = 0.280 mm²
Mean Grid Opening Area: 0.0140 mm²
EM Serial #: S/N 156120-35

Notes: Structure #2 determined to be Chrysotile asbestos based on morphology (tubular). Chemistry was Uto due to the size of the fiber & surrounding particulate.

Edit Sample #308006-6B (D-58)

General | PLM: ELAP 198.6 NOB | TEM: Bulk Upgrade

Initial Sample Weight

Vial Weight	7.1887
Vial + Sample Weight	7.6834
Initial Sample Weight	0.495

Post Ash Weight

Vial + Ashed Sample Weight	7.6822
Ashed Sample Weight	0.494

Post Acid Treatment

Filter Tare	[illegible]
Gross Filter Weight	6.5285
Weight Residue	0.438

Visual Estimations

Estimated Asbestos PLM	0.0

Final Asbestos Percents

Percent Asbestos PLM	NAD

Final Non-Asbestos %

Percent Organics	0.243
Percent Acid Soluble	11.178
Percent Other	88.57

Material Type: Powder | Color: White | Homogeneity: Homogeneous

Sample Type: Whole | Texture: Powdery

- [ ] Sample was not analyzed
- [ ] Fibrous
- [x] Scanning Negative Option
- [ ] Stratified Point Count
- [ ] 400 Point Count

Macro Asbestos %		Macro Non Asbestos %		Macro Non Fibrous %	
Chrysotile Percent		Mineral Wool Percent		Particulate	100.0
Amosite Percent		Fiberglass		Other Non-Fibrous	
Crocidolite Percent		Organic			
Other Asbestos Percent		Synthetic			
		Other			

[illegible] Comments

Temperature (°C): 26.0 | Microscope #: 2

Morphology	Color	Elongation	Est. Avg	Dispersion Staining		Refractive Index		Fiber	Birefringence	Refractive Index Other	Mount
											Add Row

Slide 1		Slide 2		Slide 3		Slide 4		Slide 5		Slide 6		Slide 7		Slide 8	
Mount	Fiber #	Mount	Fiber #	Mount	Fiber #	Mount	Fiber #	Mount	Fiber #	Mount	Fiber #	Mount	Fiber #	Mount	Fiber #
															Add Row

Asbestos Points

Chrysotile Points		Pending	Trace Detected but no points
Amosite Points		Pending	Trace Detected but no points
Crocidolite Points		Pending	Trace Detected but no points
Other Asbestos Points		Pending	Trace Detected but no points
Total Asbestos Points	Pending	NAD	< 0.25%
Total Points	Pending		

Non Asbestos Fiber Percentages

Mineral Wool + Fiberglass Combined	
Organic	
Synthetic	
Other	

Non Fibrous Percentages

Particulate	100.0
Other Non-Fibrous	

- [ ] Recommend TEM
- [ ] Recommend Matrix Reduction

Microscopic Comments

Report Comments

- [ ] Error(s) Found During Review

Edit Sample #308006-6B (D-58)

General | PLM: ELAP 198.6 NOB | TEM: Bulk Upgrade

Initial Sample Weight		Post Ash Weight		Post Acid Treatment	
Vial Weight	7.1687	Vial + Ashed Sample Weight	7.6622	Filter Tare	6.0903
Vial + Sample Weight	7.6634	Ashed Sample Weight	0.494	Gross Filter Weight	6.5285
Initial Sample Weight	0.495			Weight Residue	0.438

Visual Estimations		Final Asbestos Percents		Final Non-Asbestos %	
Estimated Asbestos PLM	0.0	Percent Asbestos PLM	NAD	Percent Organics	0.243
Estimated Asbestos TEM	0.01	Percent Asbestos TEM	0.009	Percent Acid Soluble	11.178
				Percent Other	88.57

Sample Type	Material Type	Sample Color
Whole		

Grid Box	Row and Slots	Microscope #
A19-433	3ab	1

Working Mag. High (K)	Working Mag. Low (K)	Accelerating Voltage (KV)
15		100

☐ Sample was not analyzed

Structure Chrystallographic and Photographic Data

Structure #	SAED	Elements	Reg. #	Camera Length / Mag.	Ident.	
5	hex					✖
4	neg					✖
3	hex					✖
2	pos (1.0x0.05, Matr	Si Mg Al	104	0.22/10000	Chrysotile	✖
1	pos (1.6x0.05, Clus	Si Mg	102	0.22/7800	Chrysotile	✖

Add Row

Grid #1 Estimated Asbestos	Grid #2 Estimated Asbestos	Estimated Asbestos %
0.01	0.01	0.01%

Analyst Comments

Analysis: 09/07/2019, Time = 1hr 10 mins
F Orientation: 120,230
Al peak on the chrysotile matrix is likely from the

Report Comments

☐ **Error(s) Found During Review**

AMA Analytical Services, Inc.
Focused On Results.

Fiber Count Sheet for Transmission Electron Microscopy

Page 1 of 1

Date: 09/07/19
Client ID: D-58
Filter Size/Type/Porosity 47mm MCE 0.22 µm
Client: FDA
COC #: 308006
AMA ID #: 308006-6B
Analyst (b) (6) [redacted]
Working Mag: 15 K
Accel Voltage: 100 kV
Orientation of Letter F: 7, ✓
Grid Box #: A19-433
Grid Acceptable [✓ Y] N
Volume Filtered: 0.2 mL
Signed
Row #: 3
Grid: A / B /
Grid Openings to Observe: 20
Method: Mod. NY ELAP 198.4 (FDA Procedures)

Grid Opening # Location	Structure #	Length & Width (in microns)		SAED # Rows	Mineral Type	Elements ID'd (If left blank, no elements ID'd)	Fiber Neg # (If left blank, no photo was taken)	SAED Neg # (If left blank, no photo was taken)	Magnification or Camera Length (if left blank, no photo was taken)	
I8 B	1a	1.6	0.05	pos	Chrysotile	Si Mg	103	102	7.2 K	cm
I8 B	1b	1.1	0.05	pos	Chrysotile	Si Mg	103	102	7.2 K	cm
I8 B	1c	0.75	0.04	pos	Chrysotile	Si Mg	103	102	7.2 K	cm
E3 A	2	1.0	0.05	pos	Chrysotile	Si Mg (Al)	105	104	10 K	cm
☐									K	cm
☐									K	cm
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☐									K	cm

Legend: NSD = No Structures Detected UTO = Unable to Obtain ☐ = 1st Grid ☒ = 2nd Grid

# of Structures Counted: 4

Mineral Type	Total
Chrysotile	4

Total # of Grid Openings Observed: 20 = 0.280 mm²
Mean Grid Opening Area: 0.0 140 mm²
Notes: Al peak is likely from the surrounding particulate on structure #2
EM Serial #: S/N 156120-35

Edit TEM NOB Blank Result

Blank Number

NB19-645

Analyst

(b) (6)

Date Analyzed

09-18-2019

Percent Asbestos

0.0

Asbestos Type

Comments

Save Changes

Edit TEM NOB Blank Result

Blank Number

NB19-646

Analyst

(b) (6)

Date Analyzed

09-18-2019

Percent Asbestos

0.0

Asbestos Type

Comments

Save Changes

Edit TEM NOB Blank Result

Blank Number

NB19-647

Analyst

(b) (6)

Date Analyzed

09-18-2019

Percent Asbestos

0.0

Asbestos Type

Comments

Save Changes

Edit NOB Reference Sample Result

Sample Number	Analyst	Reference Sample Tile #
Talc Ref	Christopher D	Talc Ref 10%

Reference Value	Asbestos Type	Lower Limit	Upper Limit
10	Chrysotile	5	25

Vial Weight	0.0	Post Acid Weight	1.0	Asbestos Type	Chrysotile
Vial and Sample Weight	1.0	Filter Tare	0.0	Estimated Asbestos	10.0
Vial and Ashed Sample Weight	1.0	Ashed Weight	1	Percent Asbestos	10
Initial Sample Weight	1	Residue Weight	1	Result	Pass

Comments

Save Changes

AMA Analytical Services, Inc.
Laboratory Blank Log

Blank ID #	Initials	Prep Date	Chain of Custody #	AMA or Client Sample Numbers	Analysis Date	Asbestos Conc.	Client Name	Archive Box #
EB- 54140	(b) (6)	8/30/19	[redacted]					
EB- 54141		8/30/19						
EB- 54142		8/30/19						
EB- 54143		"						
EB- 54144		"						
EB- 54145		9/1/19						
EB- 54146		"						
EB- 54147		"						
EB- 54148		"						
EB- 54149		9/1/19						
EB- 54150		9/3/19						
EB- 54151		"						
EB- 54152		"						
EB- 54153		"						
EB- 54154		"						
EB- 54155		"	308006	308006 (6, 6A, 6B,	(b) (4)		USFDA	
EB- 54156		"	(b) (4)				USFDA	
EB- 54157		"					USFDA	
EB- 54158		9/4/19						
EB- 54159		"						
EB- 54160		"						
EB- 54161		"						

Ver:1-3 (4/91)

Revision 0, Issued Oct 2004

Edit Air Blank Result

Blank Number

54155

Analyst

(b) (6)

Date Analyzed

09-18-2019

Area Analyzed

0.07

Asbestos Structures

0

Asbestos Type

Result

< 14.286

Comments

Save Changes