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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

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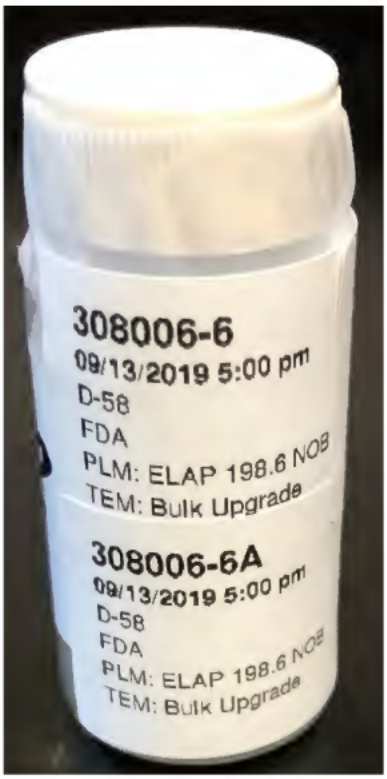
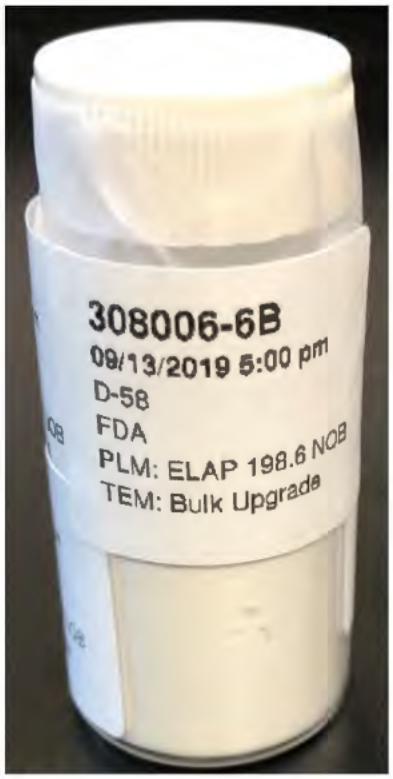
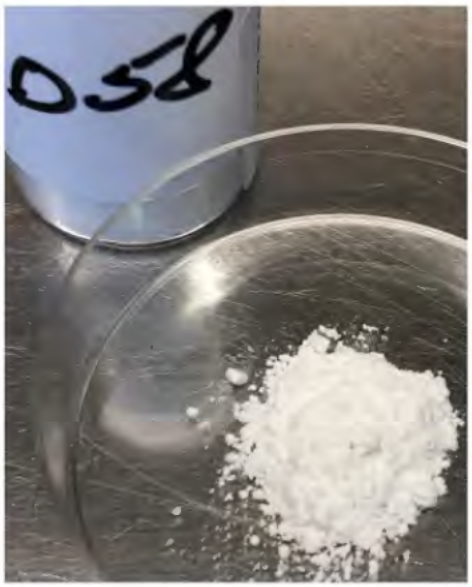
308006-6, 6A, 6B/D58







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.

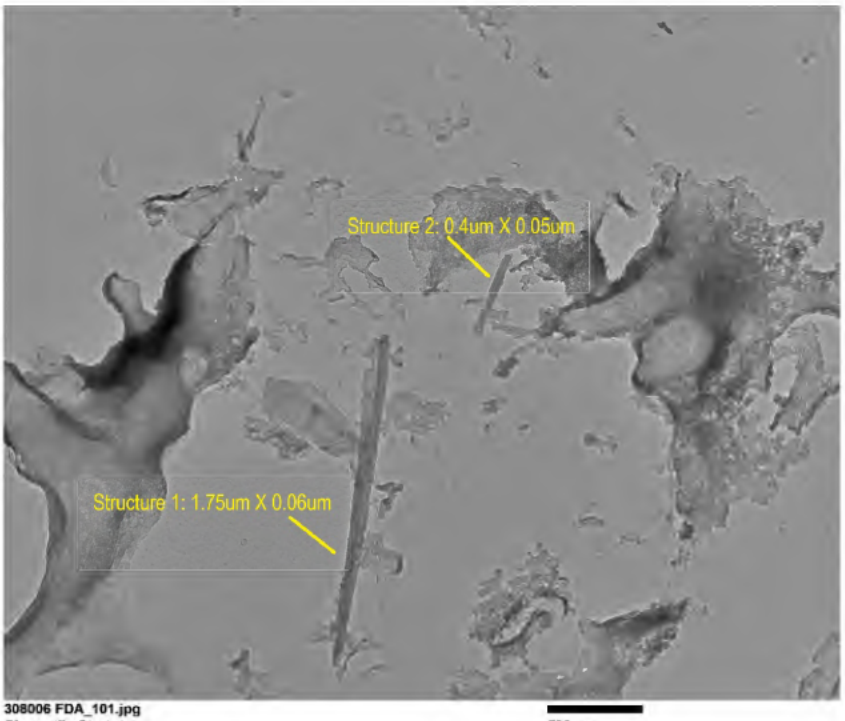
308006-6	NAD
308006-6A	NAD
308006-6B	NAD

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.

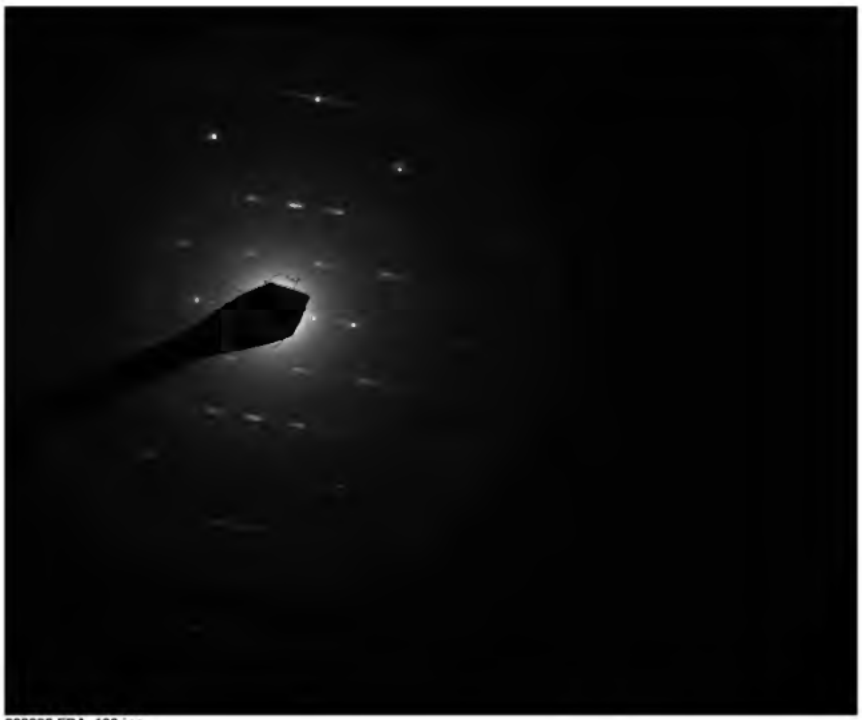
Sample 308006-6A, Chrysotile Structures



308006 FDA_101.jpg
Chrysotile Strutctures
308006-6a
Cal: 0.001774 µm/pix
14:06 9/7/2019
TEM Mode: Imaging
Microscopist: CD
Camera: NANOSPRT5, 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

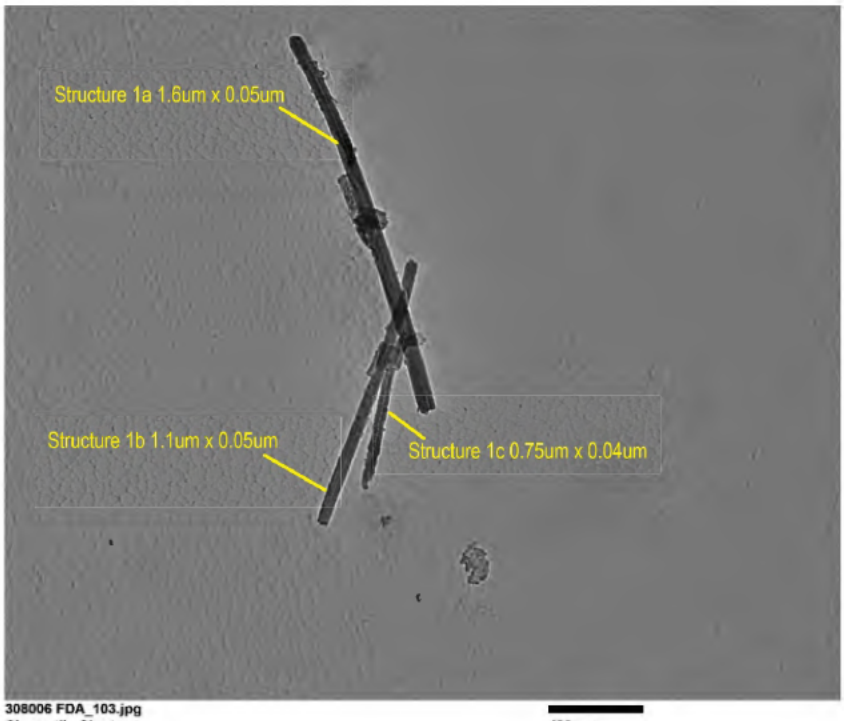
Diffraction Pattern from Chrysotile Structure 1 pictured above



308006 FDA_100.jpg
Chrysotile Dif
308006-6a
14:03 9/7/2019
TEM Mode: Diffraction
Microscopist: CD
Camera: 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

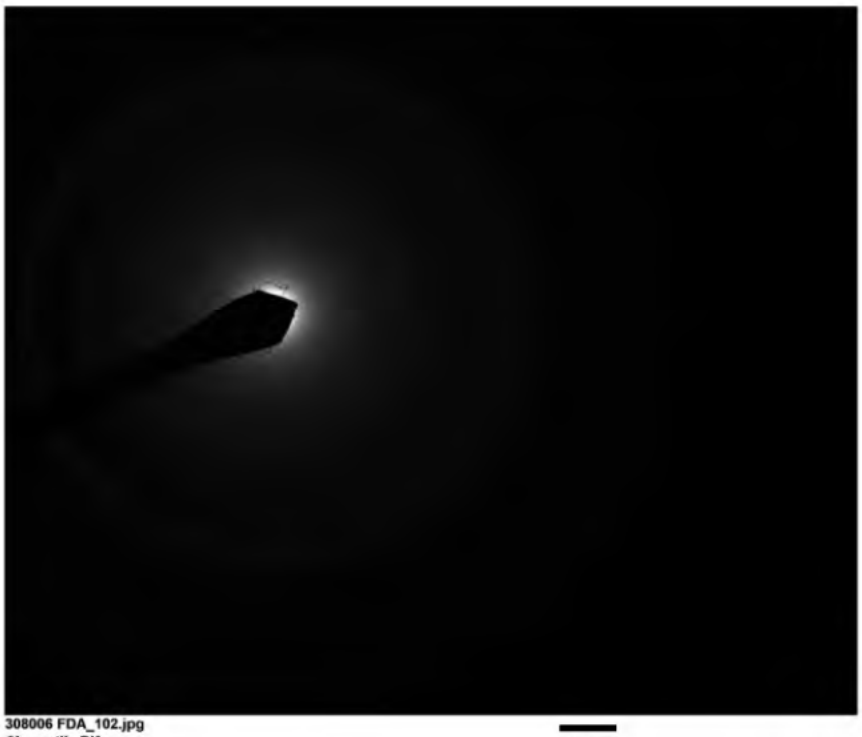
Sample 308006-6B, Chrysotile Structure 1



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

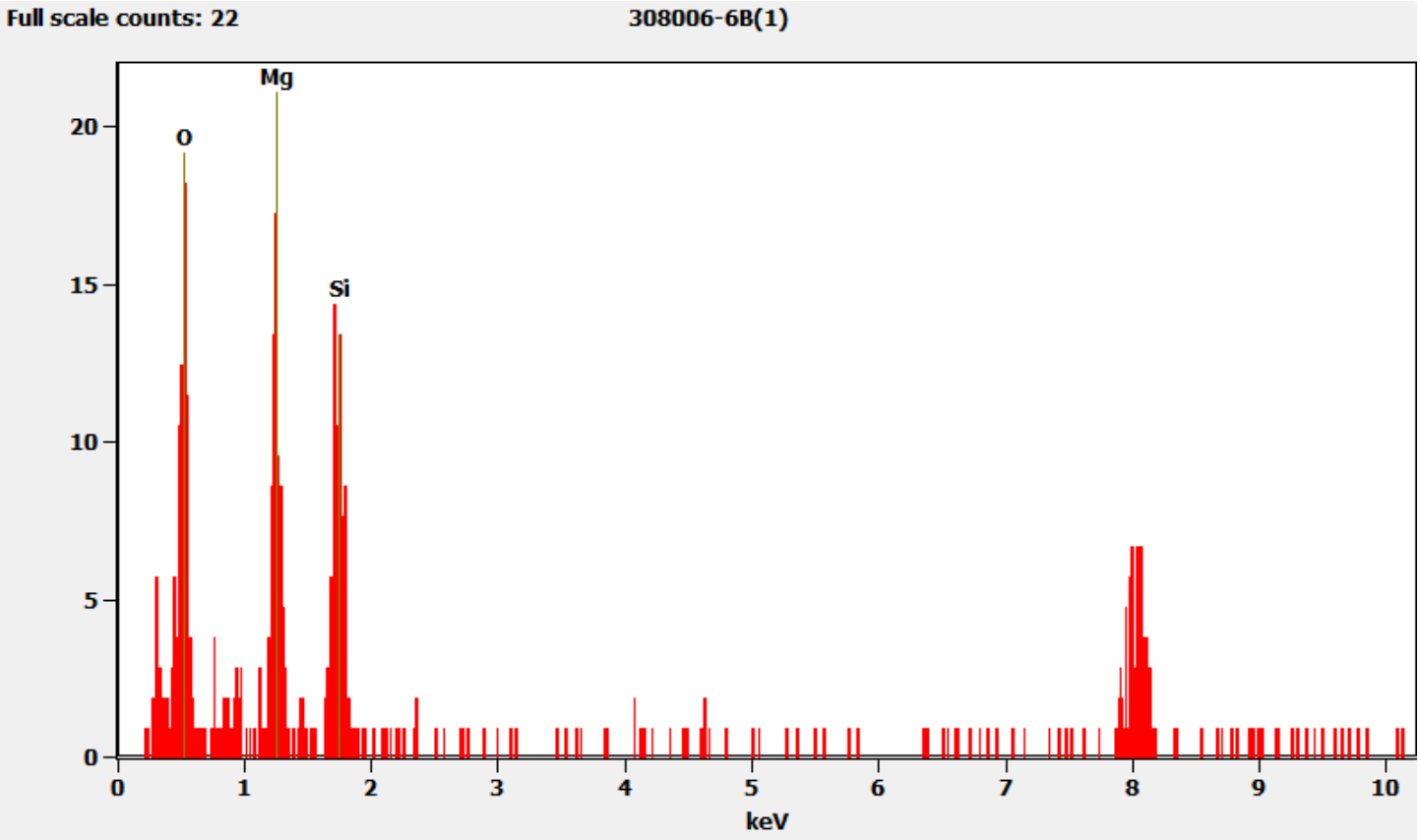
Diffraction Pattern from Chrysotile Structure pictured above



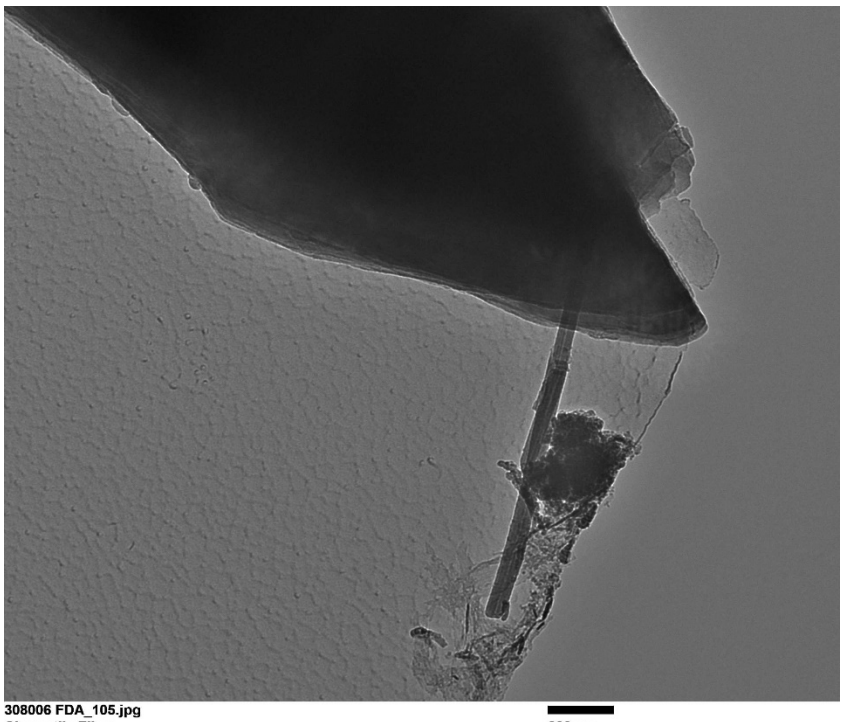
308006 FDA_102.jpg
Chrysotile Dif
308006-6b
15:32 9/7/2019
TEM Mode: Diffraction
Microscopist: CD
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 std. frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from Chrysotile Structure pictured above



308006-6B, Chrysotile Structure 2



308006 FDA_105.jpg
Chrysotile Fiber
308006-6b
Cal: 0.001029 µm/pix
16:05 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

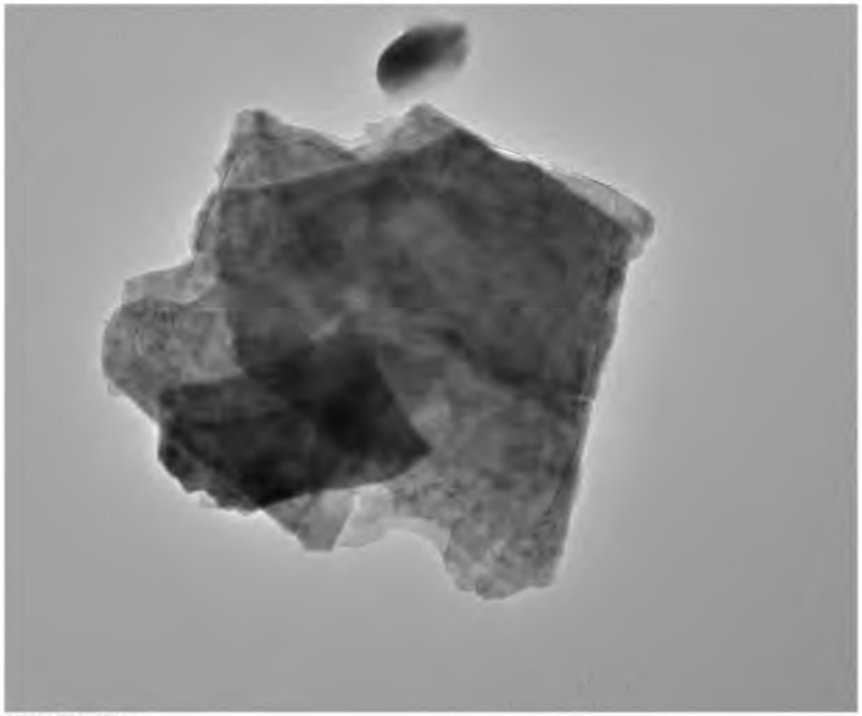
200 nm
HV=100kV
Direct Mag: 10000 x
AMA Analytical Services, Inc

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

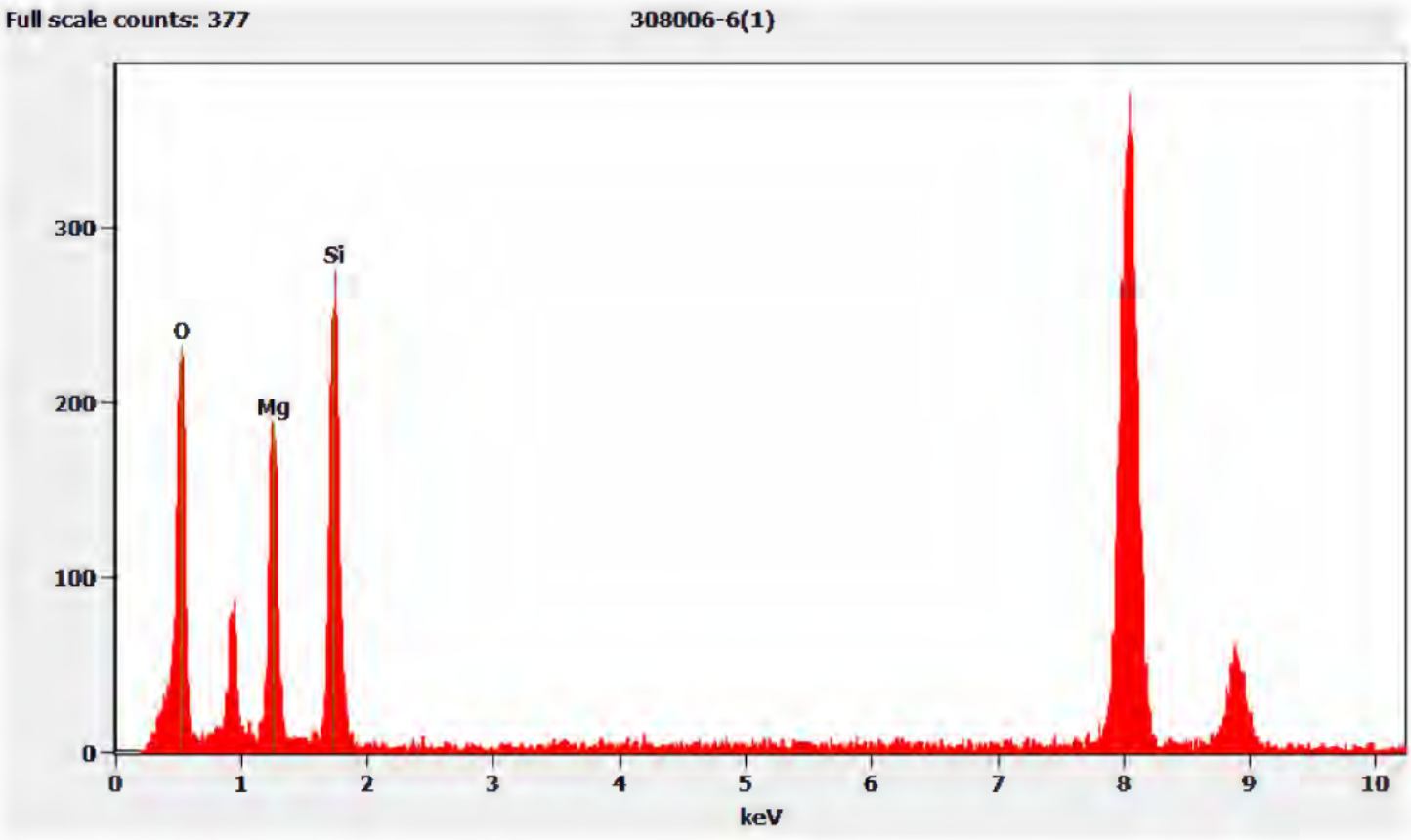


308006 FDA_052.jpg
Talc Particle
Cal: 0.001774 µm/pix
17:18 9/3/2019
TEM Mode: Imaging
Microscopist: MG
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Hexagonal Diffraction Pattern from Talc Particle pictured above

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

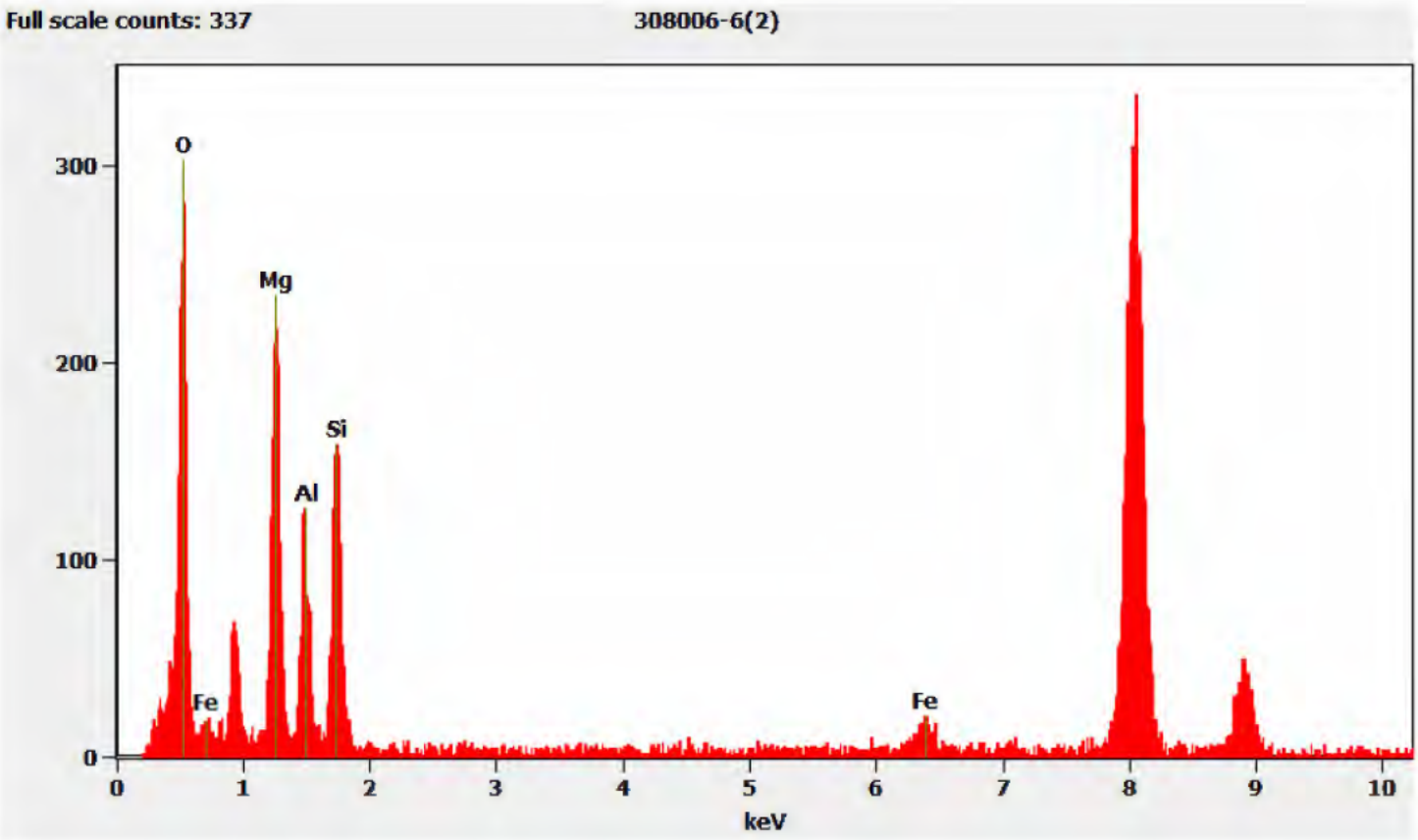
400 nm
HV=100kV
Direct Mag: 7200 x
AMA Analytical Services, Inc

Diffraction Pattern from Mica Particle pictured above

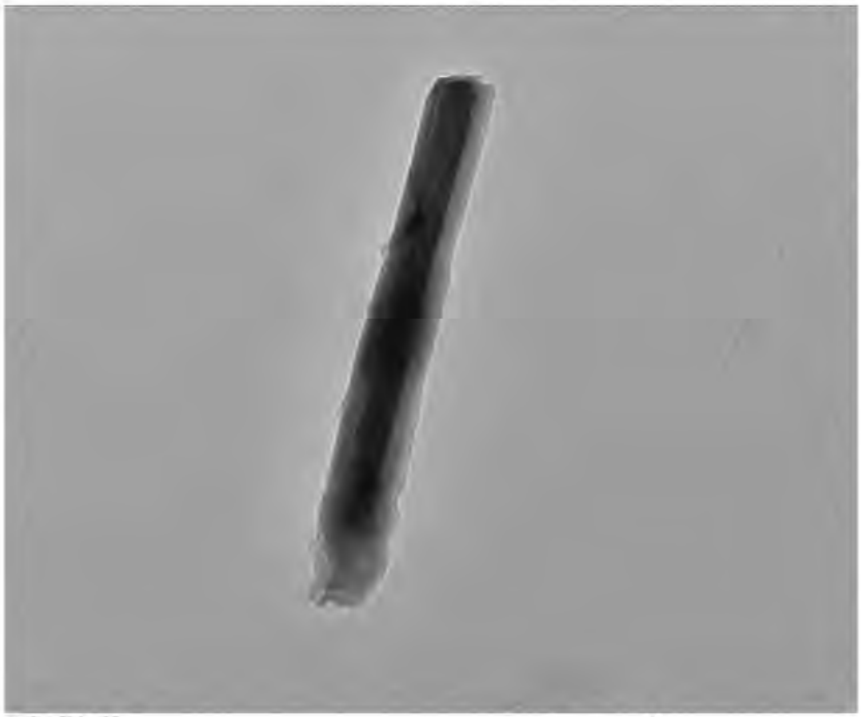
308006 FDA_056.jpg
Mica Particle
17:22 9/3/2019
TEM Mode: Diffraction
Microscopist: MG
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

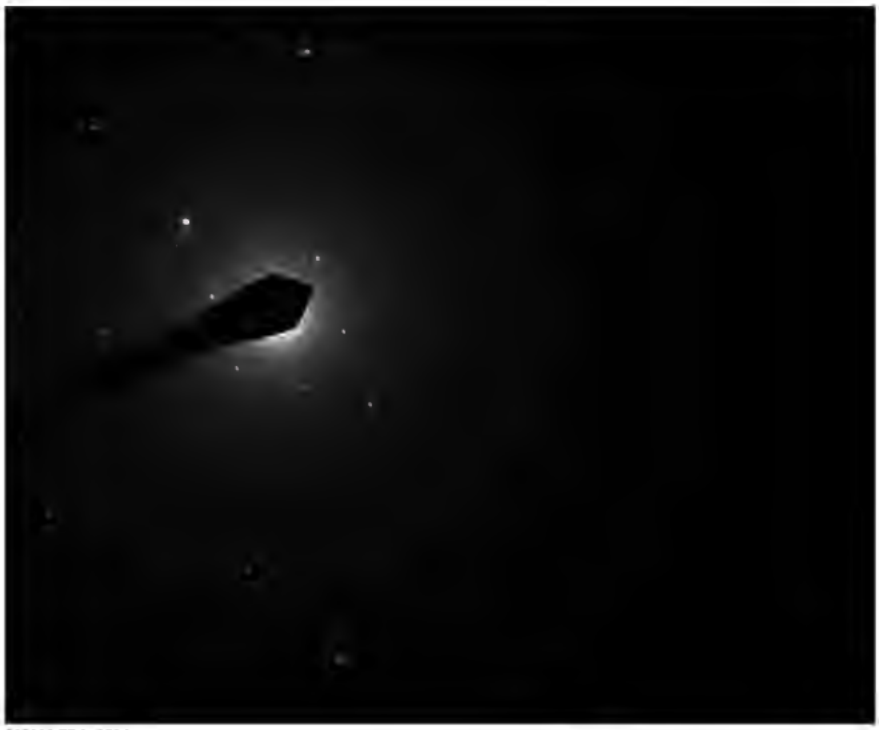
Chemistry from Mica Particle pictured above



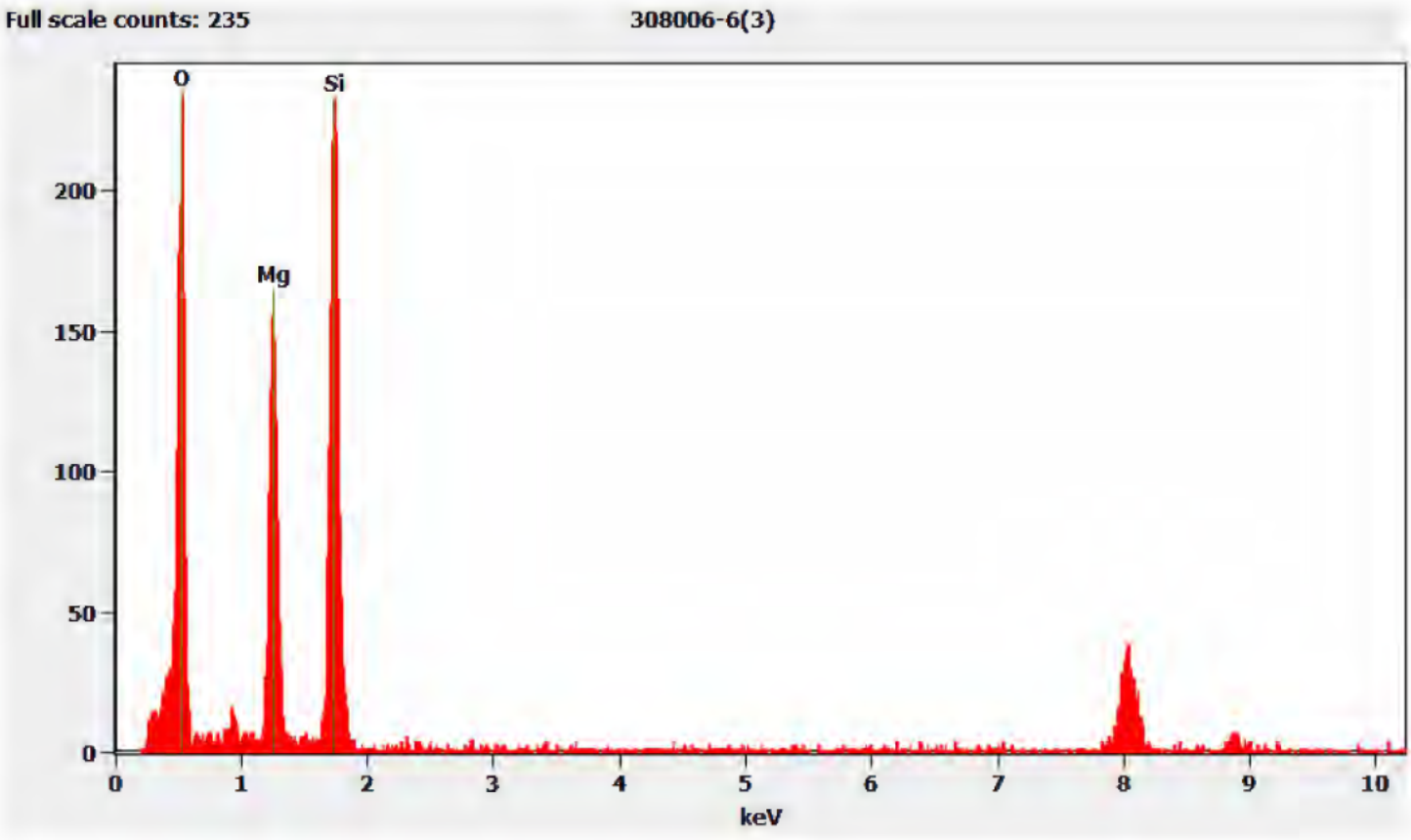
308006-6, Talc Fiber



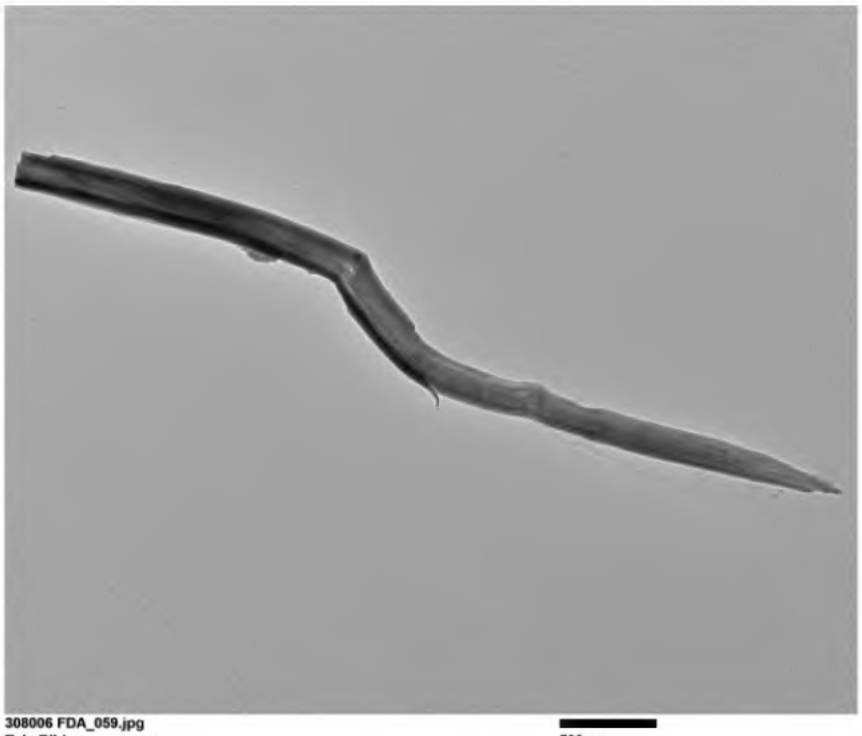
Diffraction Pattern from Talc Fiber pictured above



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

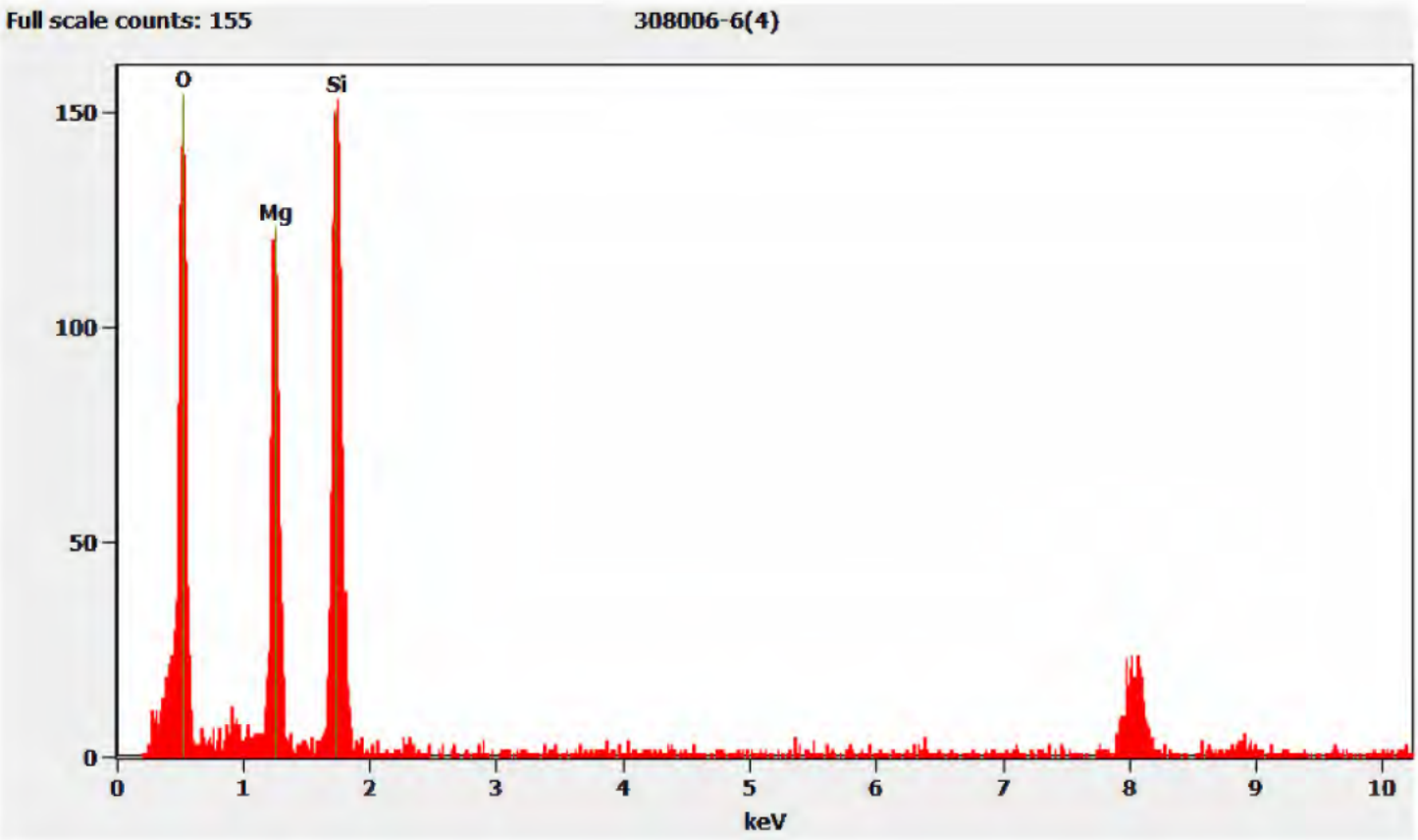
500 nm
HV=100kV
Direct Mag: 5800 x
AMA Analytical Services, Inc

Diffraction Pattern from Talc Ribbon pictured above

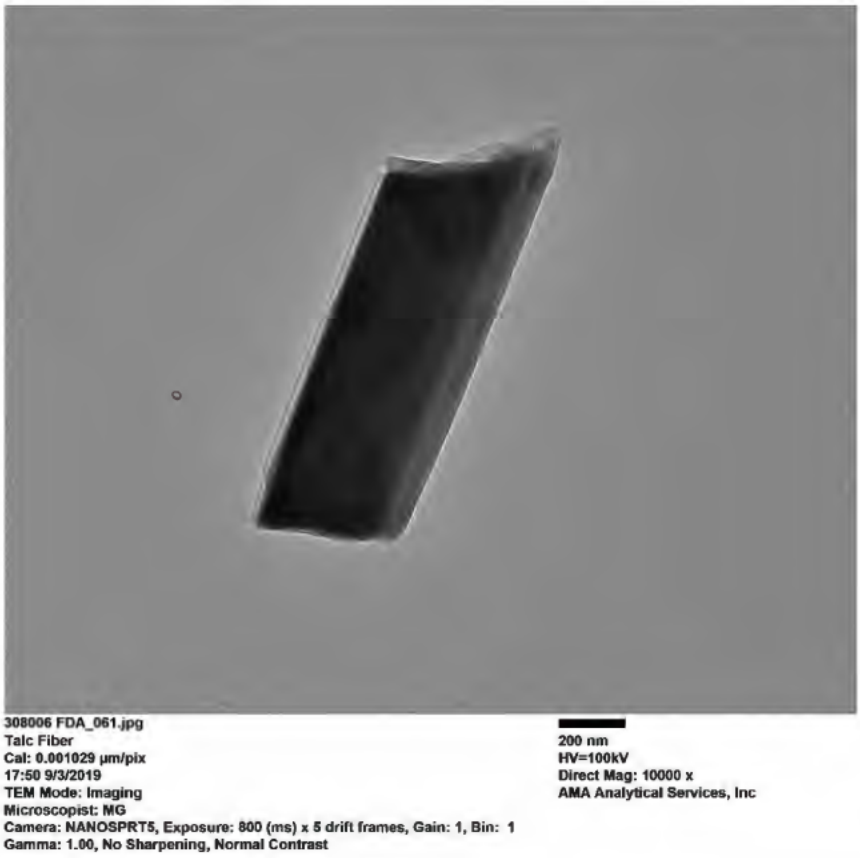
308006 FDA_060.jpg
Talc Ribbon
17:38 9/3/2019
TEM Mode: Diffraction
Microscopist: MG
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from Talc Ribbon pictured above

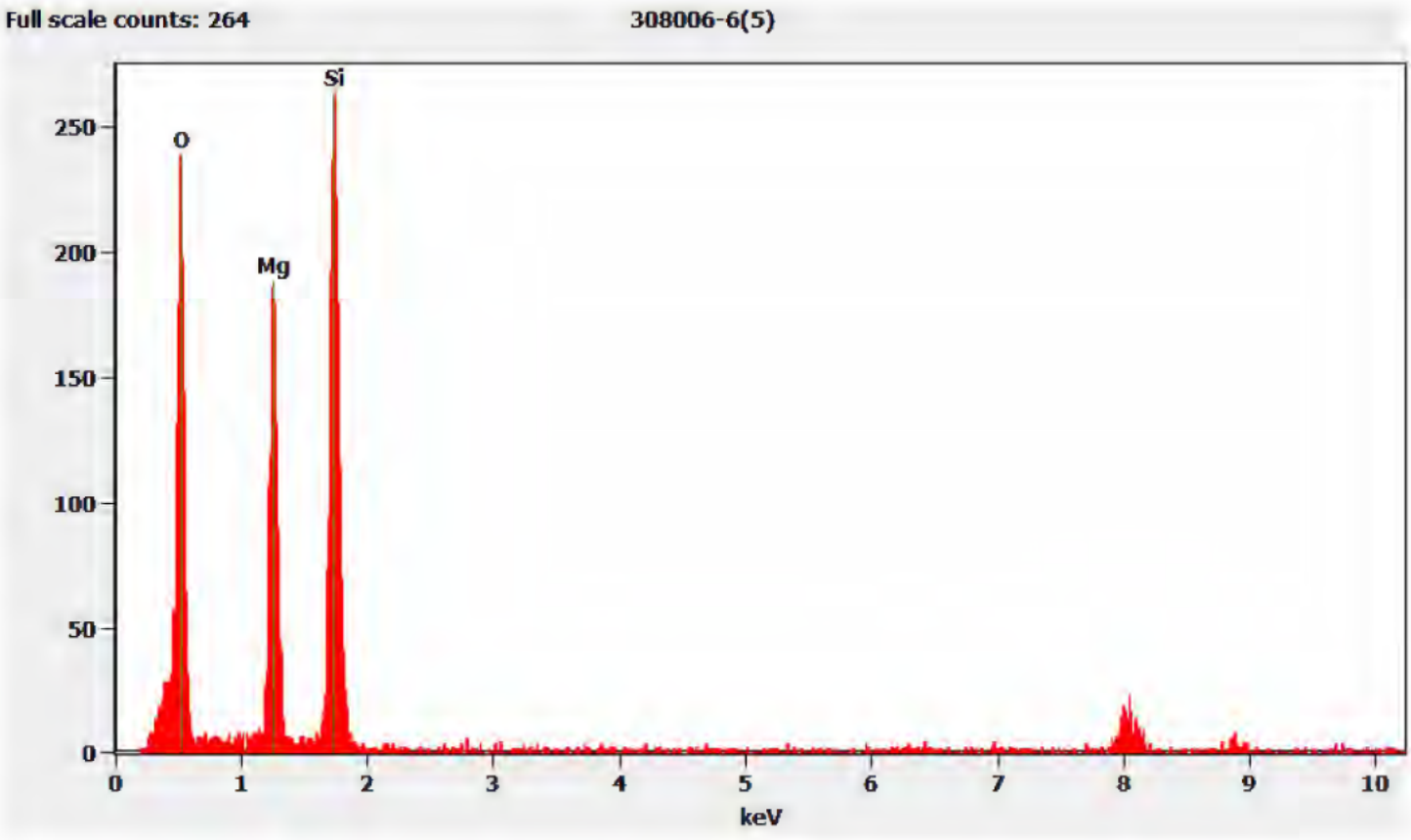


308006-6, Talc Fiber



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