



### CERTIFICATE OF ANALYSIS

**Chain of Custody:** 308004

**Client:** US Food & Drug Administration

**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:** 3rd Group - 2 Samples

**Job Number:** CLIN 1 - Task 3 (2 Samples)

**PO Number:** HHSF223201810337P

**Date Submitted:** 5/29/2019

**Date Analyzed:** 7/25/2019 - 8/1/2019

**Report Date:** 8/14/2019

**Date Sampled:** Not Provided

**Person Submitting:** Steve Wolfgang

**Revised:** 8/30/2019, 3rd Revision

#### 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
308004-1	D-49	0.00000115%	0.00000946%	0.13214%	< 0.00001%	0.13214%	ND	12.2%	16.5%	71.3%	
308004-1A	D-49	0.00000133%	0.00000532%	0.00018%	0.00002%	0.00020%	ND	12.4%	14.7%	72.8%	
308004-1B	D-49	0.00000153%	0.00000612%	0.20597%	0.00193%	0.20790%	ND	12.5%	14.3%	73.1%	

**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

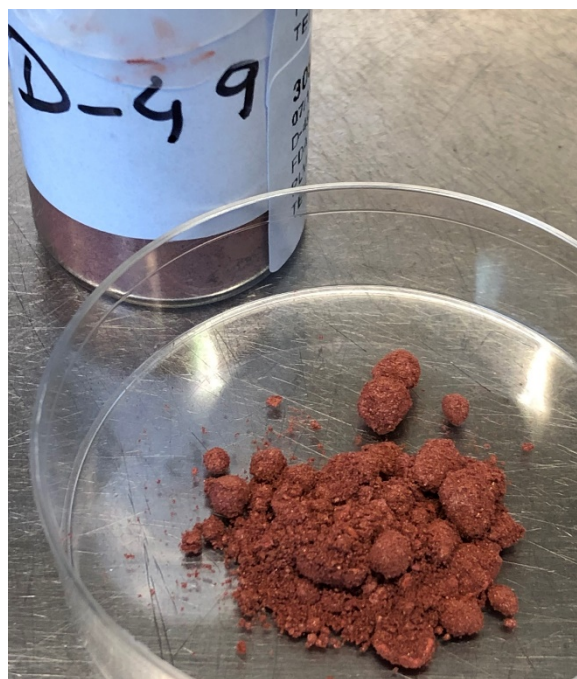
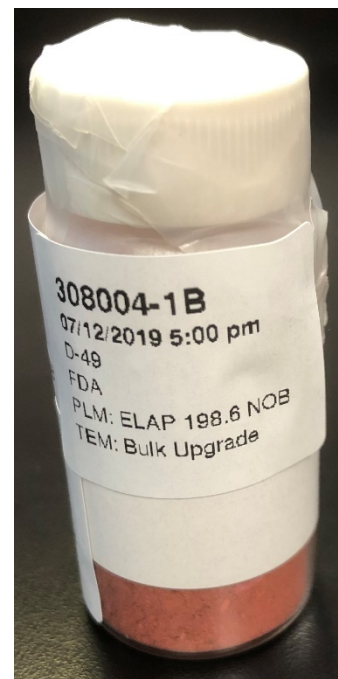
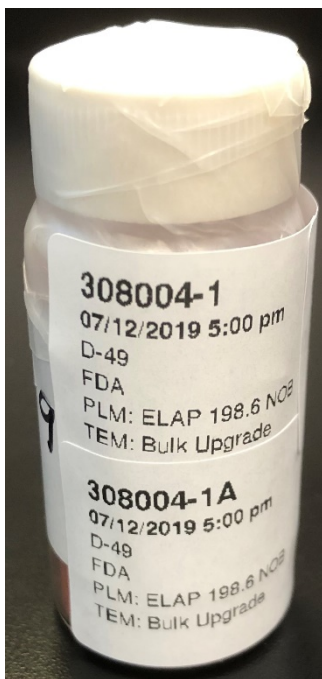
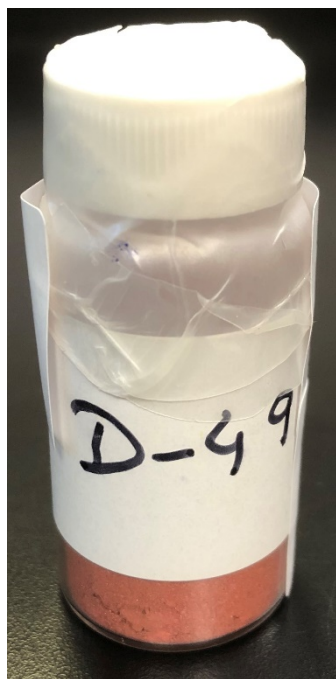
**Analyst(s):** PLM  
 TEM

(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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### Sample Preparation

Samples were prepared for PLM and TEM bulk analysis by Chon Simpha on July 2, 2019 through July 9, 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 or a regulated amphibole 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 particle was calculated using the ASTM D5756 method. 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 chrysotile fiber 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 samples D-49 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:

308004-1: mass of the single observed chrysotile fiber plus the mass of three tremolite fibers measuring 0.5 x 0.04 microns

#### Discussion and Interpretation of Analytical Findings:

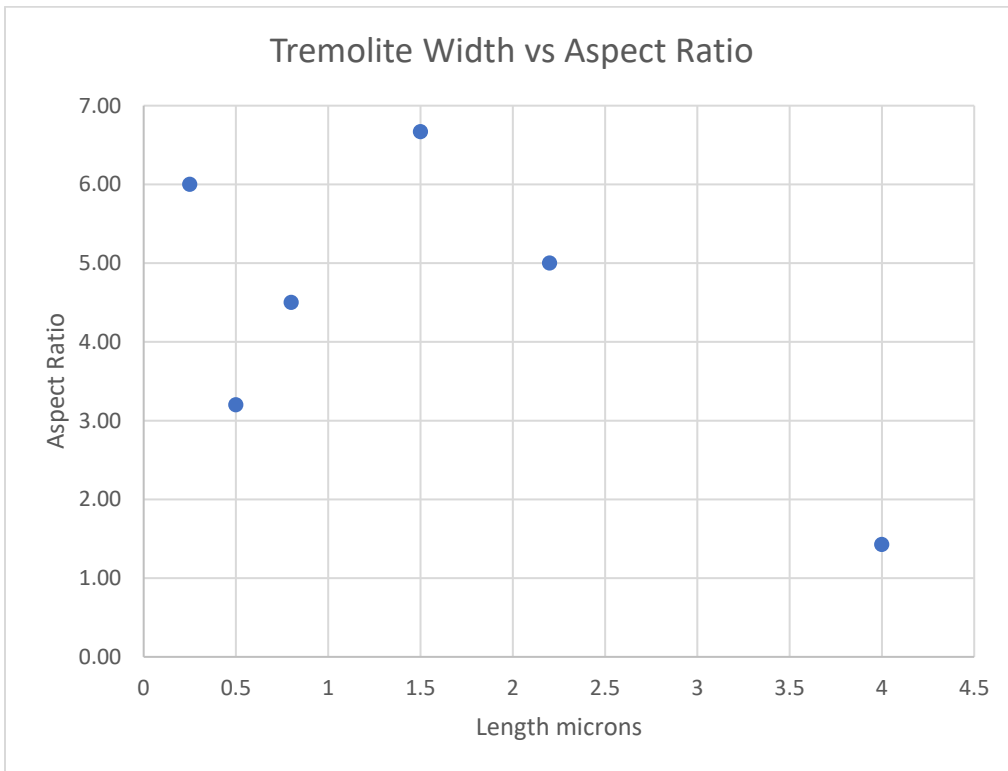
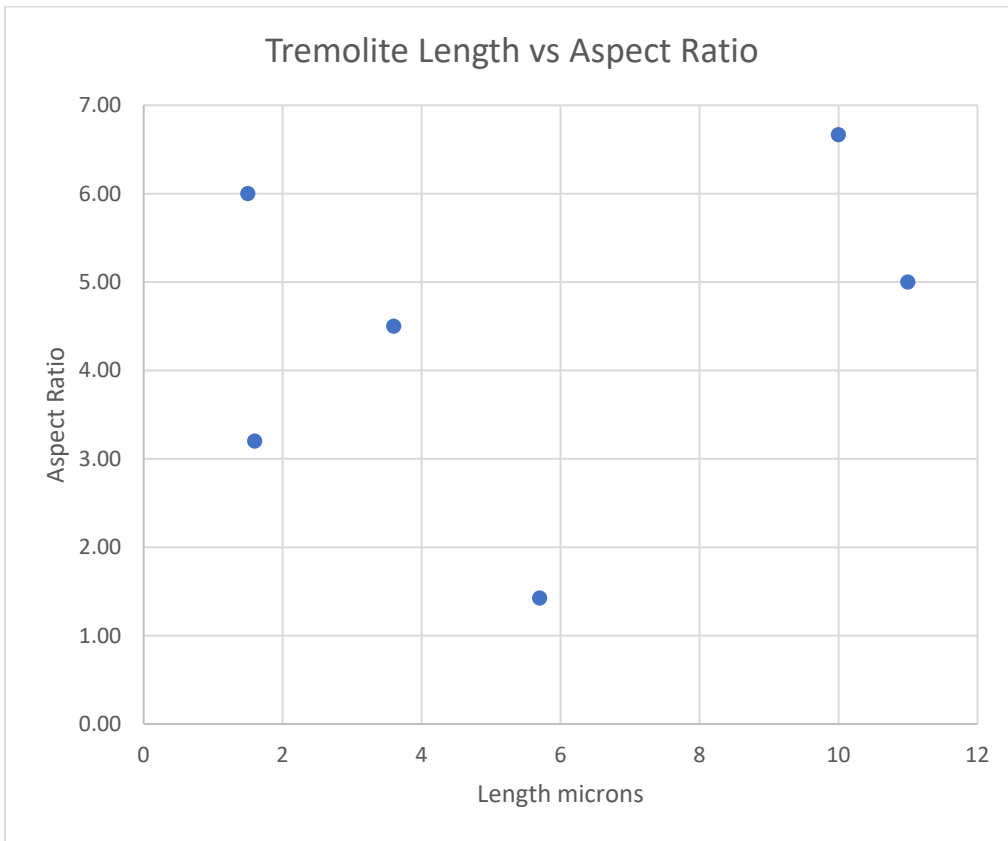
*PLM*  
All three aliquots of sample D-49 were analyzed by Peerawut Chaikenee on July 25, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

308004-1	NAD
308004-1A	NAD
308004-1B	NAD

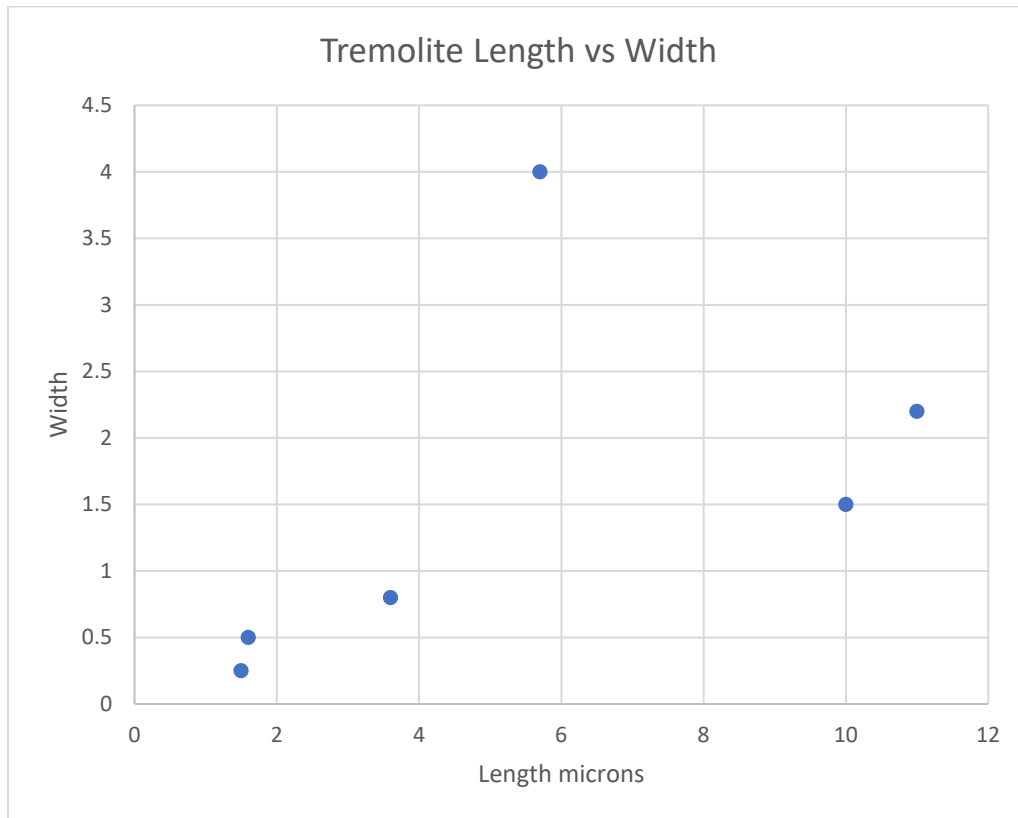
*TEM*  
Michael Greenberg analyzed sample 1 on July 29, 2019, 1A on July 30, 2019 and 1B on August 1, 2019. The sample consisted of a mix of talc and mica particles, with a few talc fibers, mica fibers and titanium fibers/particles. Chrysotile and tremolite were observed on all three aliquots. The results were calculated using the equations detailed in the calculations section.

308004-1	0.13214%
308004-1A	0.00020%
308004-1B	0.20790%

The following charts plot aspect ratio vs. length, aspect ratio vs. width, and length vs. width for all the tremolite particles counted over all three aliquots.

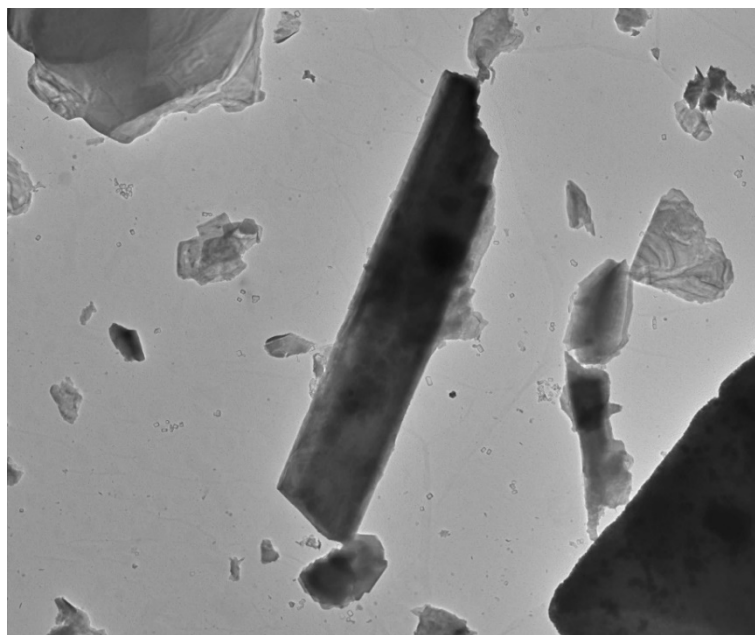






Below are representative pictures, diffraction patterns, and chemistry from the observed particles. The unidentified peaks in chemistry spectra are copper, zinc, and carbon. Those peaks are from the TEM specimen holder and specimen grid.

*Tremolite Particle from 308004-1*



308004 FDA\_020.jpg  
Tremolite 1  
Cal: 0.007349  $\mu\text{m}/\text{pix}$   
16:43 7/29/2019  
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

2  $\mu\text{m}$   
HV=100kV  
Direct Mag: 1400 x  
AMA Analytical Services, Inc

Zone-Axis Diffraction Pattern from the Tremolite Particle pictured above



308004 FDA\_017.jpg  
Tremolite Zone Axis  
[.8 2 .8]  
16:31 7/29/2019  
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

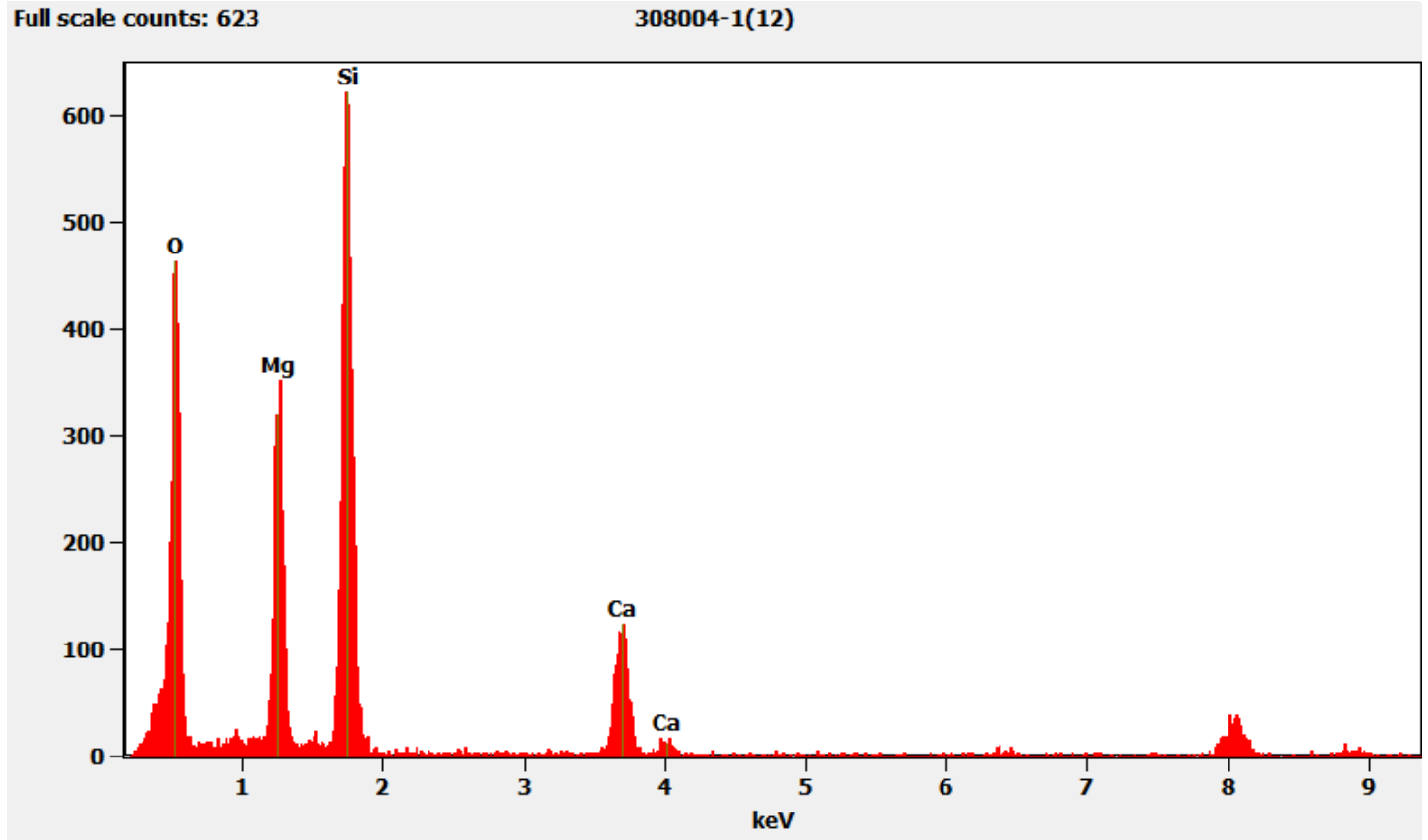
Diffraction Pattern from the Tremolite Particle pictured above



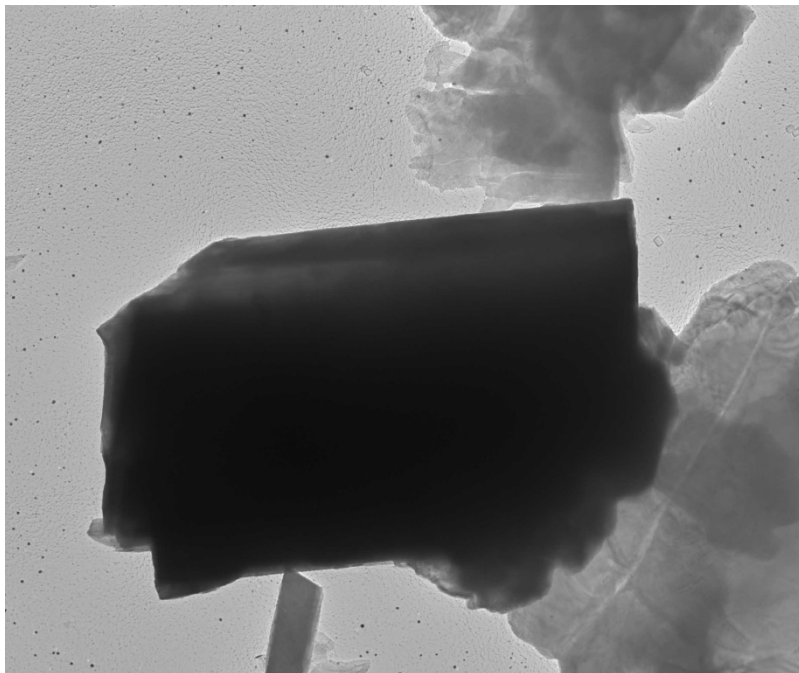
308004 FDA\_018.jpg  
Tremolite  
16:36 7/29/2019  
TEM Mode: Diffraction  
Microscopist: MG  
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1  
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from the Tremolite Particle pictured above



Tremolite Particle from 308004-1B



308004 FDA\_065.jpg  
Tremolite 2  
Cal: 0.003548  $\mu\text{m}/\text{pix}$   
15:29 8/1/2019  
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

1  $\mu\text{m}$   
HV=100kV  
Direct Mag: 2900 x  
AMA Analytical Services, Inc



Diffraction Pattern from Tremolite Particle pictured above



308004 FDA\_066.jpg

Tremolite 2

15:32 8/1/2019

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/A)

HV=100kV

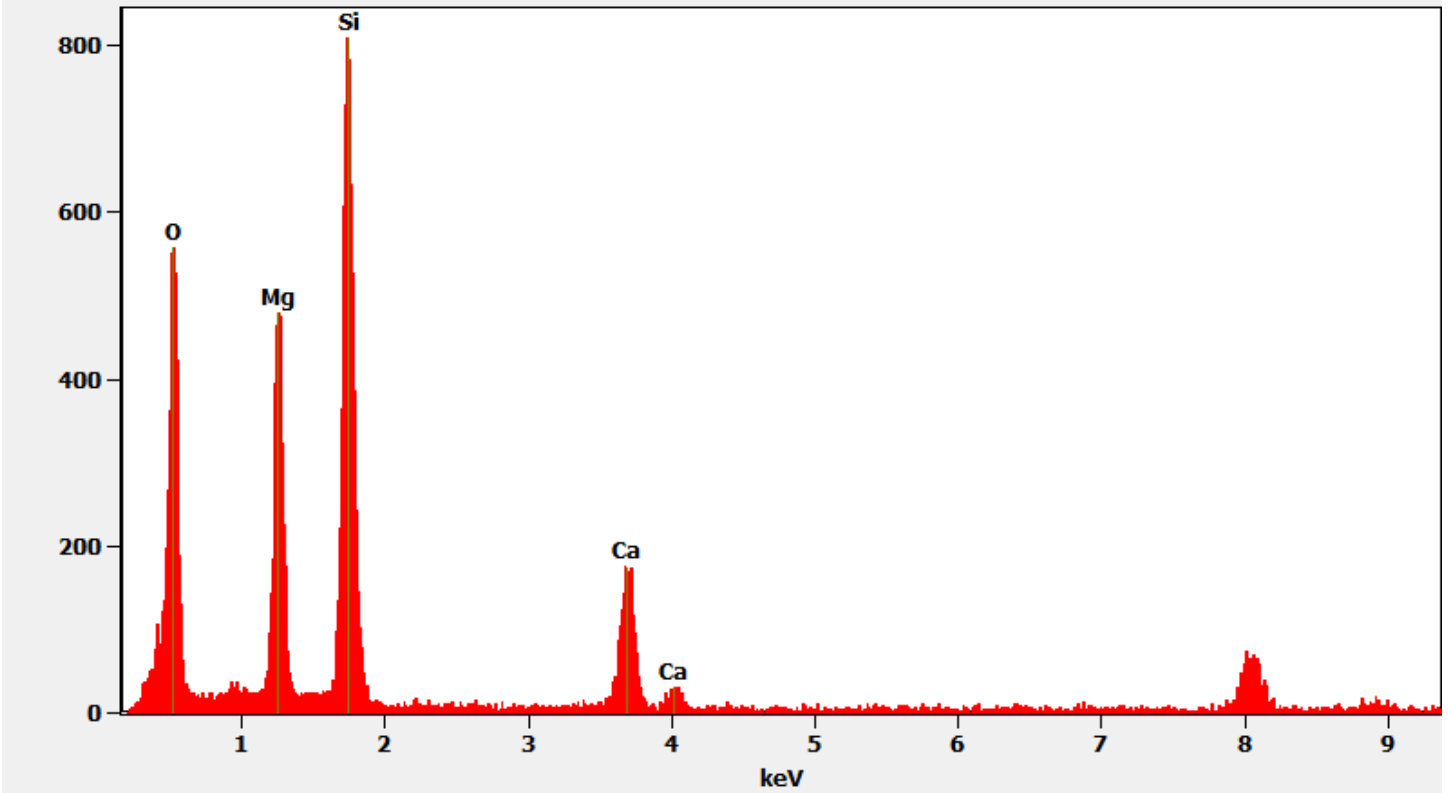
Cam Len: 0.2200 m

AMA Analytical Services, Inc

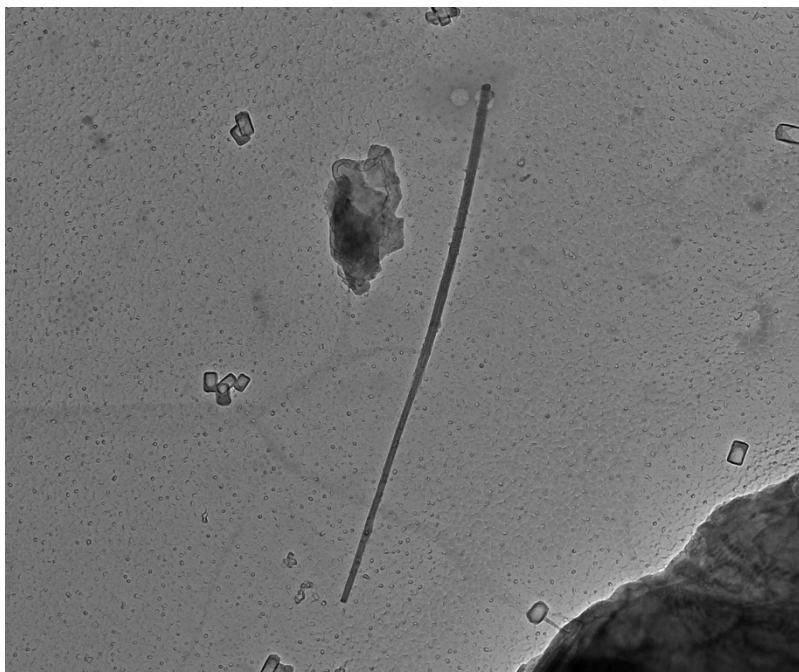
Chemistry from Tremolite Particle pictured above

Full scale counts: 810

308004-1B(3)



Chrysotile Fiber from 308004-1A



308004 FDA\_043.jpg  
Chrysotile 1  
Cal: 0.001774  $\mu\text{m}/\text{pix}$   
17:30 7/30/2019  
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

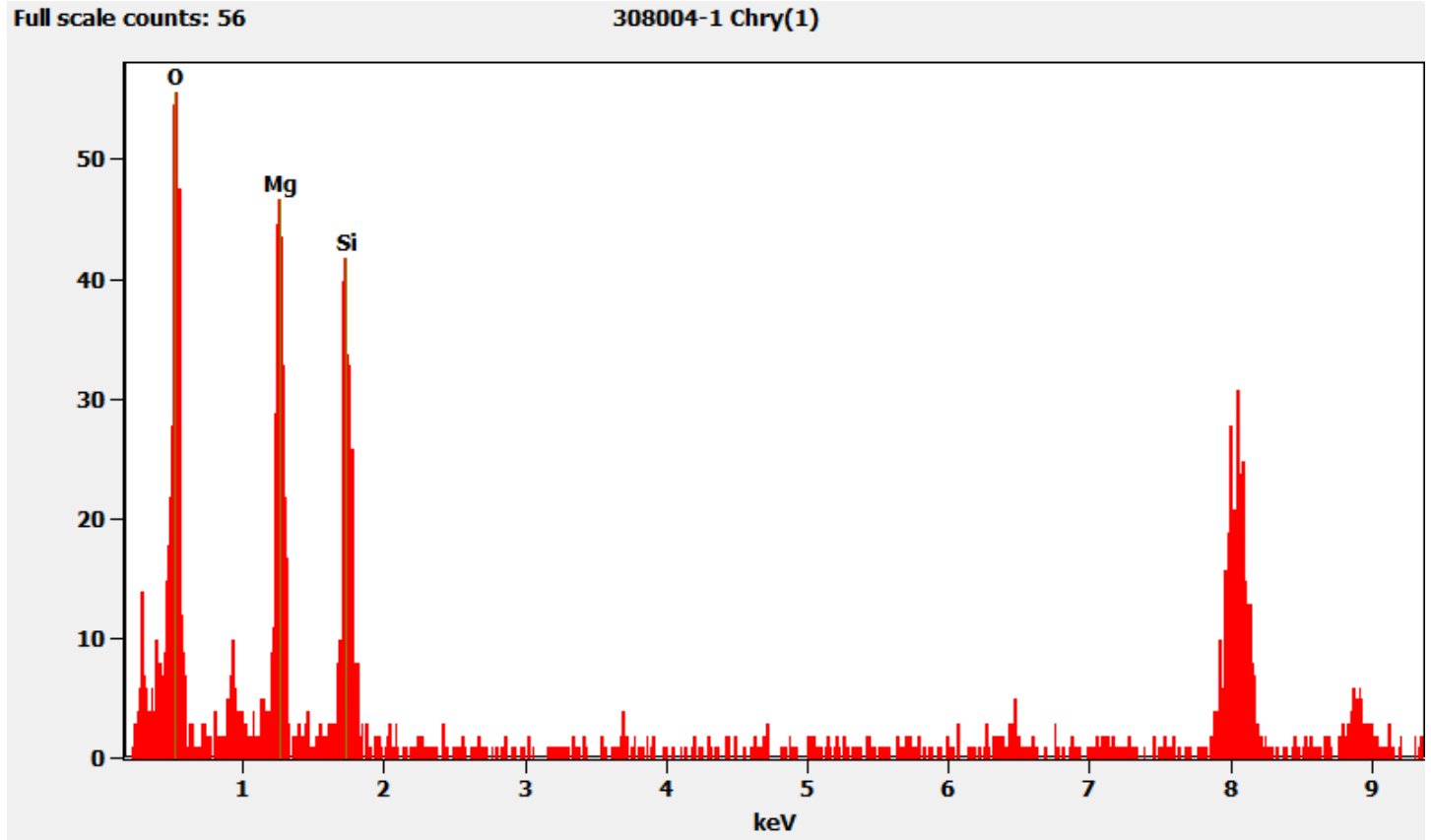
Diffraction Pattern from Chrysotile Fiber pictured above



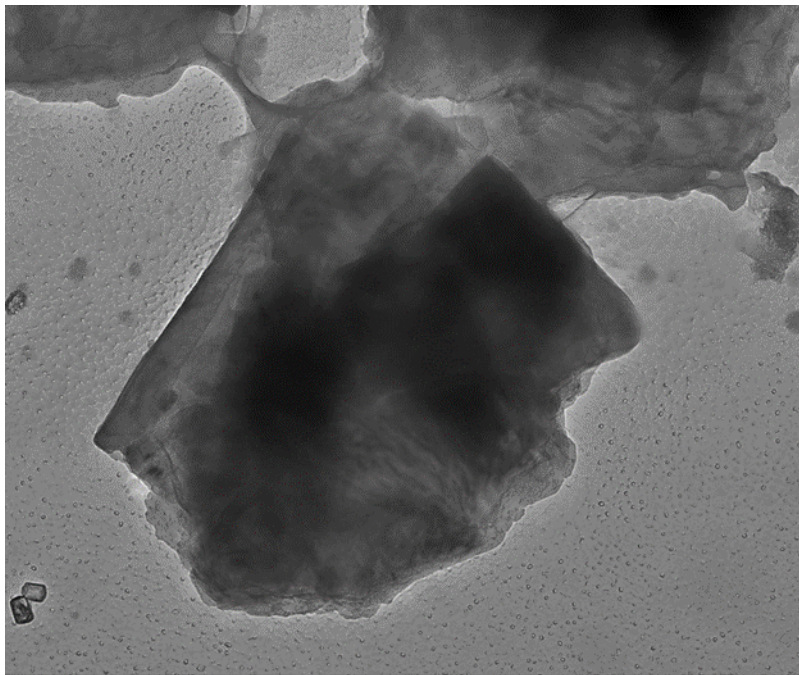
308004 FDA\_042.jpg  
Chrysotile 1  
17:28 7/30/2019  
TEM Mode: Diffraction  
Microscopist: MG  
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1  
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from Chrysotile Fiber pictured above



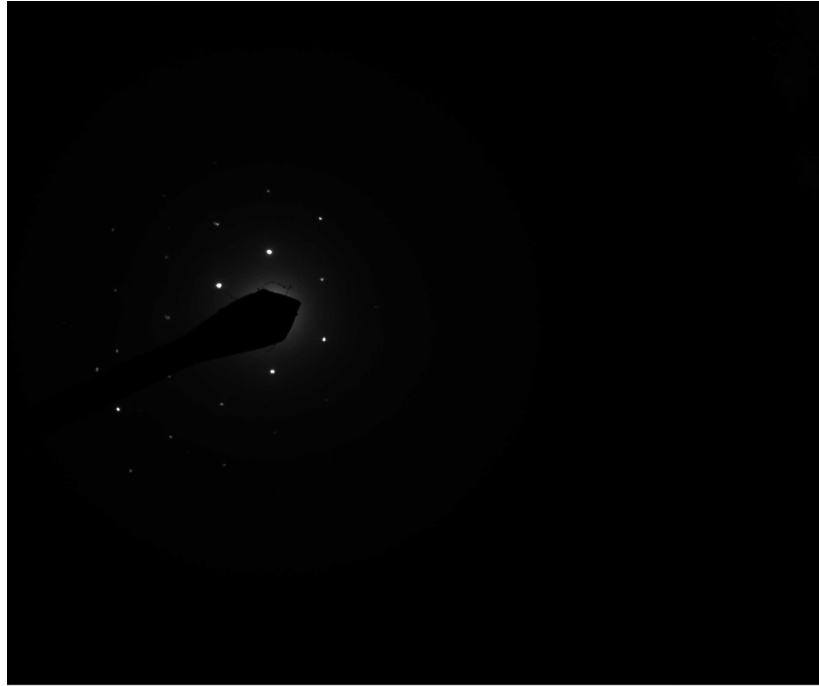
Talc Particle from 308004-1



308004 FDA\_005.jpg  
Talc  
Cal: 0.001429  $\mu\text{m}/\text{pix}$   
15:32 7/29/2019  
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

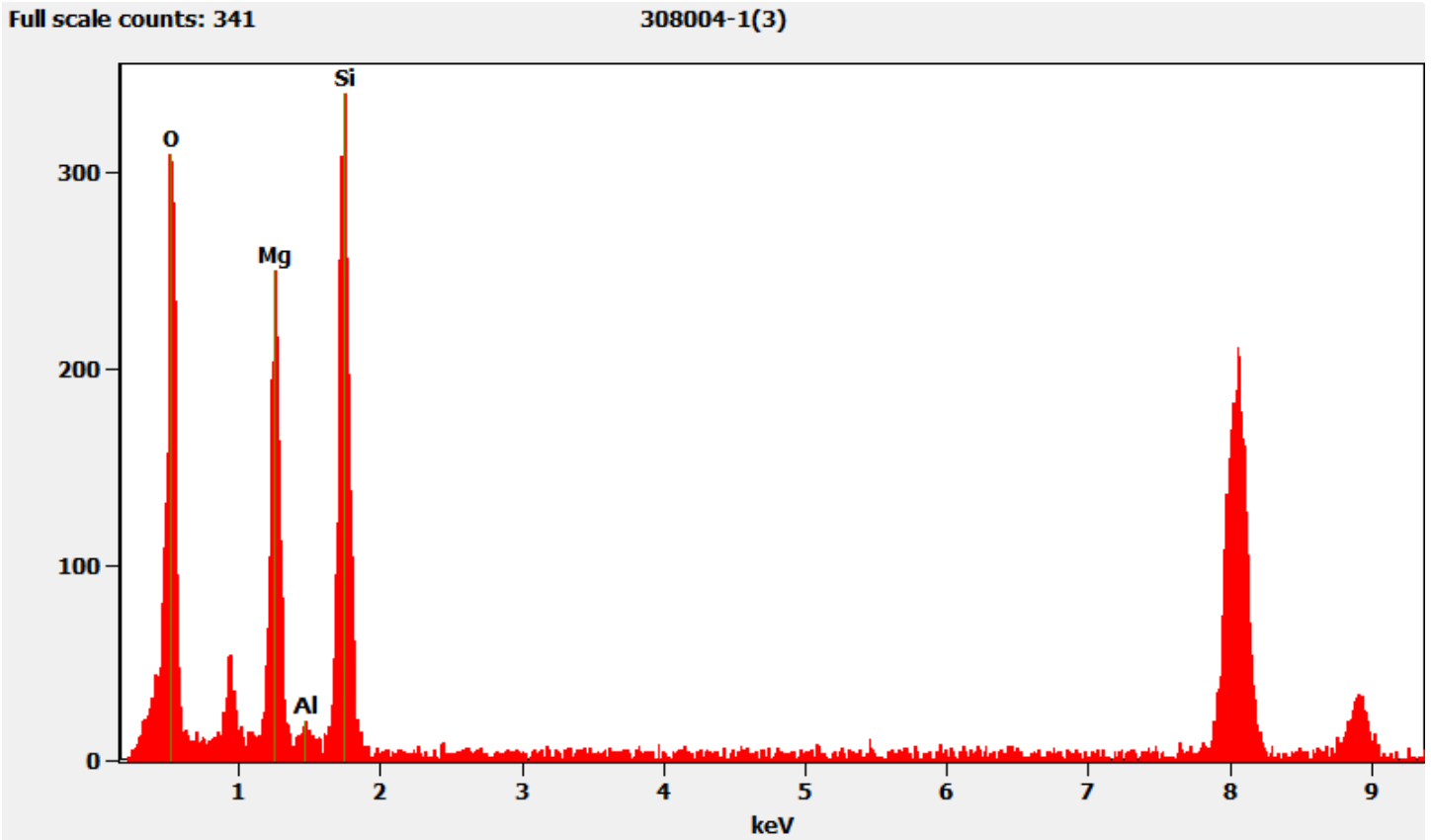
400 nm  
HV=100kV  
Direct Mag: 7200 x  
AMA Analytical Services, Inc

Diffraction Pattern from the Talc Particle pictured above

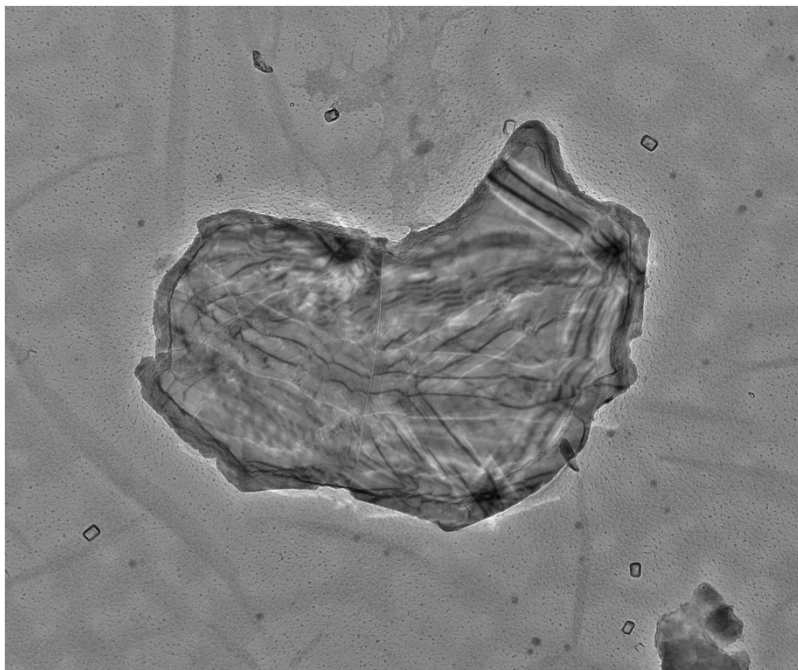


308004 FDA\_006.jpg  
Talc  
15:34 7/29/2019  
TEM Mode: Diffraction  
Microscopist: MG  
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1  
Gamma: 1.00, No Sharpening, Normal Contrast  
100 (1/A)  
HV=100kV  
Cam Len: 0.2200 m  
AMA Analytical Services, Inc

Chemistry from the Talc Particle pictured above



Mica Particle from 308004-1



308004 FDA\_011.jpg  
Mica  
Cal: 0.002858  $\mu\text{m}/\text{pix}$   
15:51 7/29/2019  
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

800 nm  
HV=100kV  
Direct Mag: 3600 x  
AMA Analytical Services, Inc

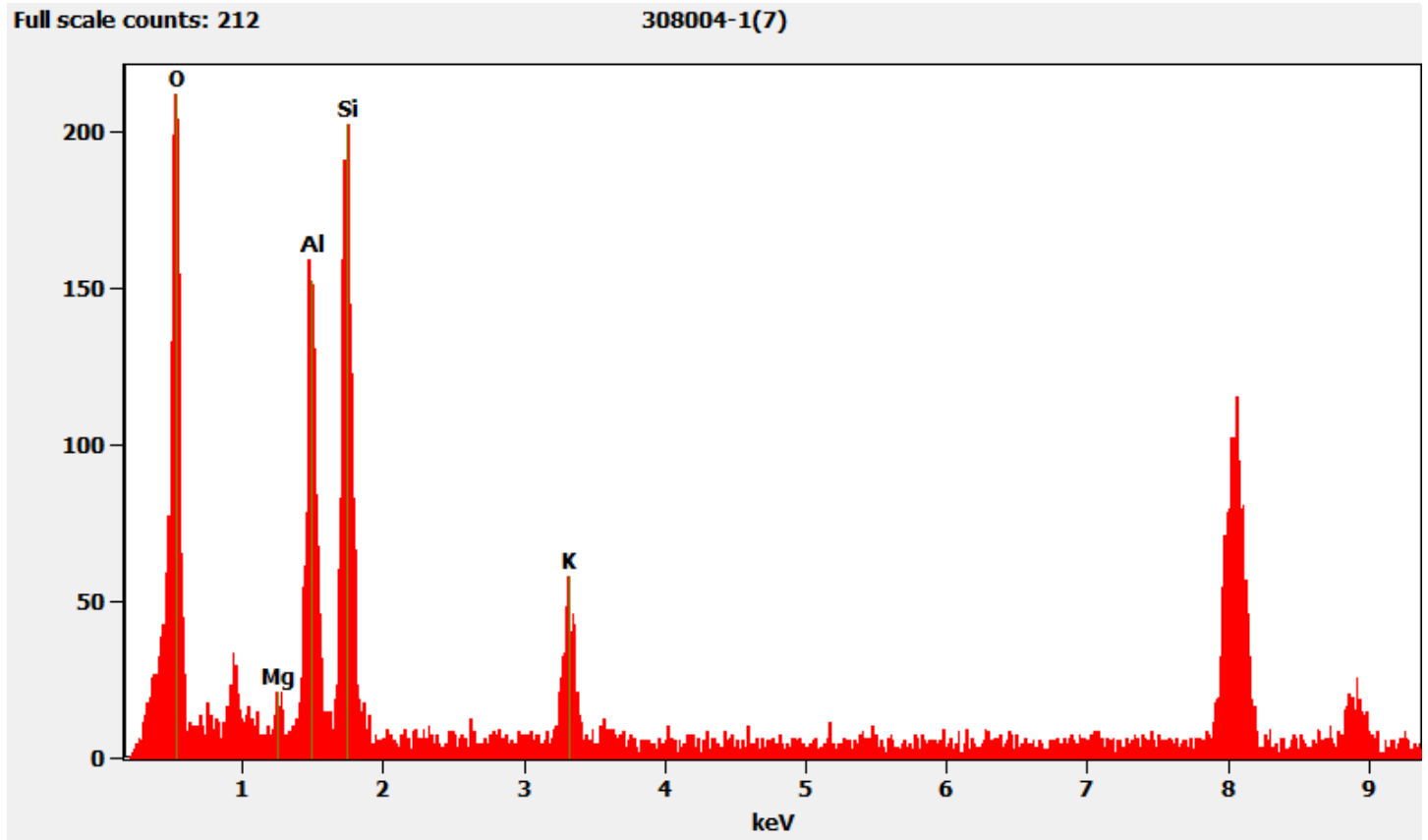
Diffraction Pattern from the Mica Particle pictured above



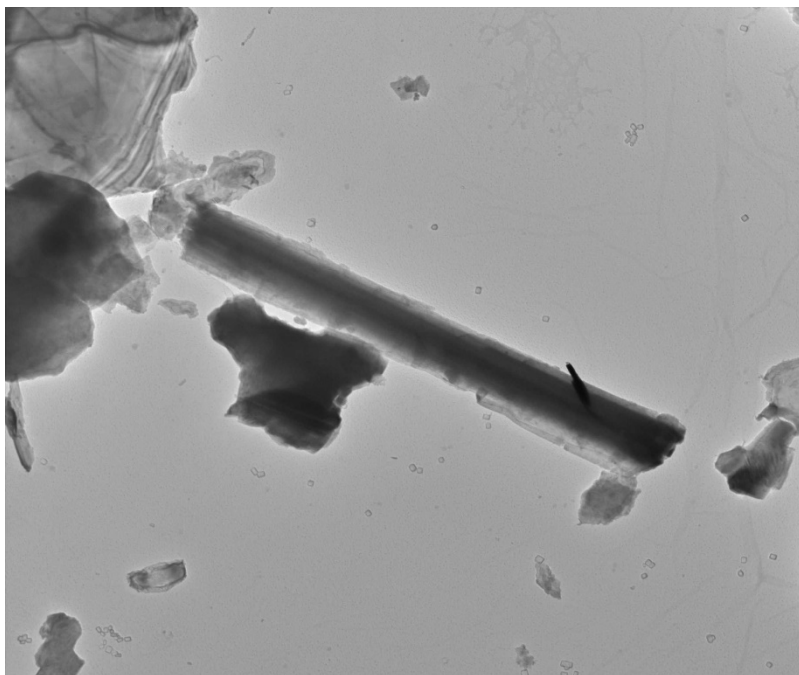
308004 FDA\_012.jpg  
Mica  
15:52 7/29/2019  
TEM Mode: Diffraction  
Microscopist: MG  
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1  
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from the Mica Particle pictured above



Talc Fiber from 308004-1



308004 FDA\_013.jpg

Talc Fiber

Cal: 0.005415  $\mu\text{m}/\text{pix}$

16:22 7/29/2019

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

1  $\mu\text{m}$

HV=100kV

Direct Mag: 1900 x

AMA Analytical Services, Inc



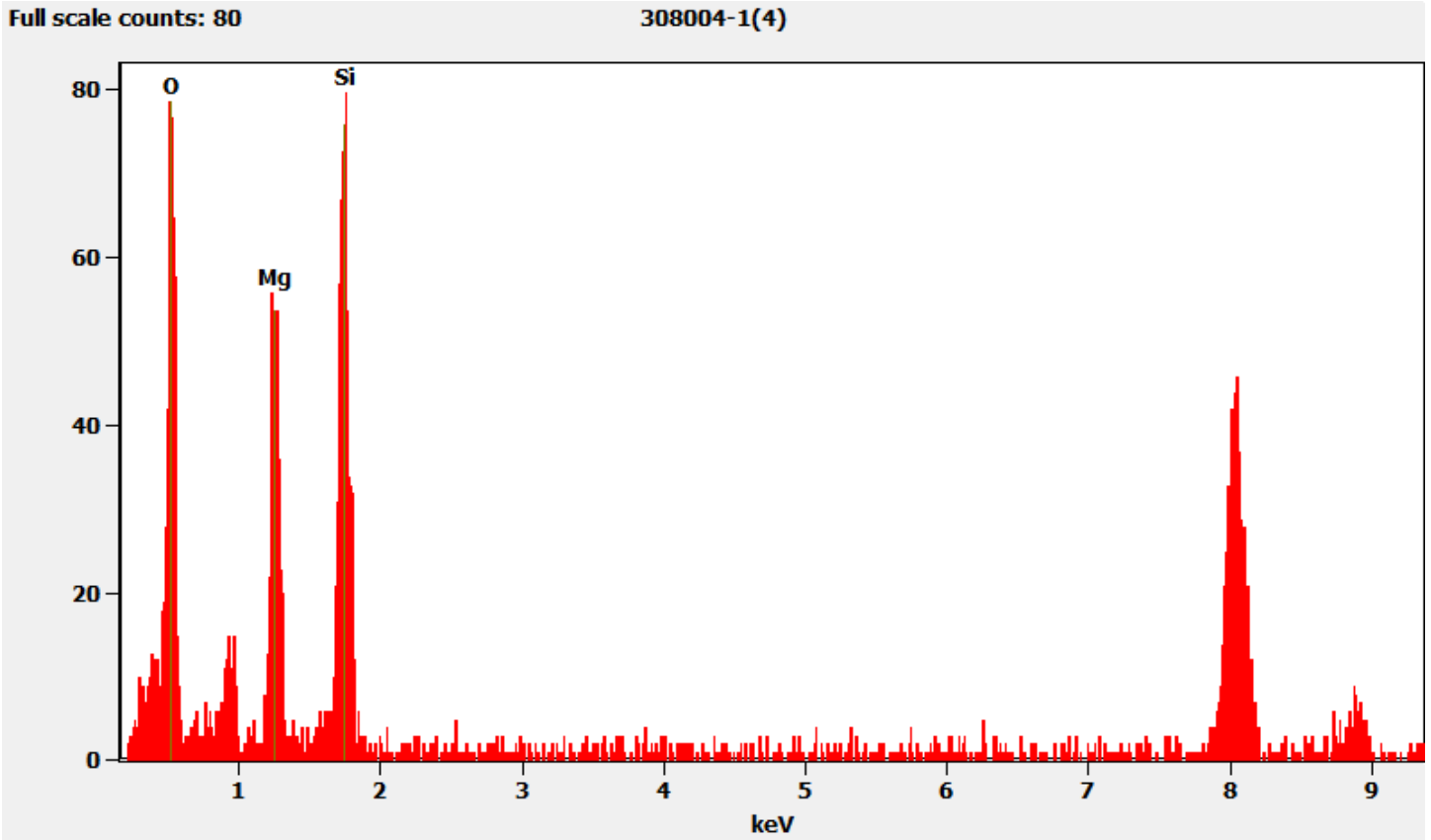
Diffraction Pattern from the Talc Fiber pictured above



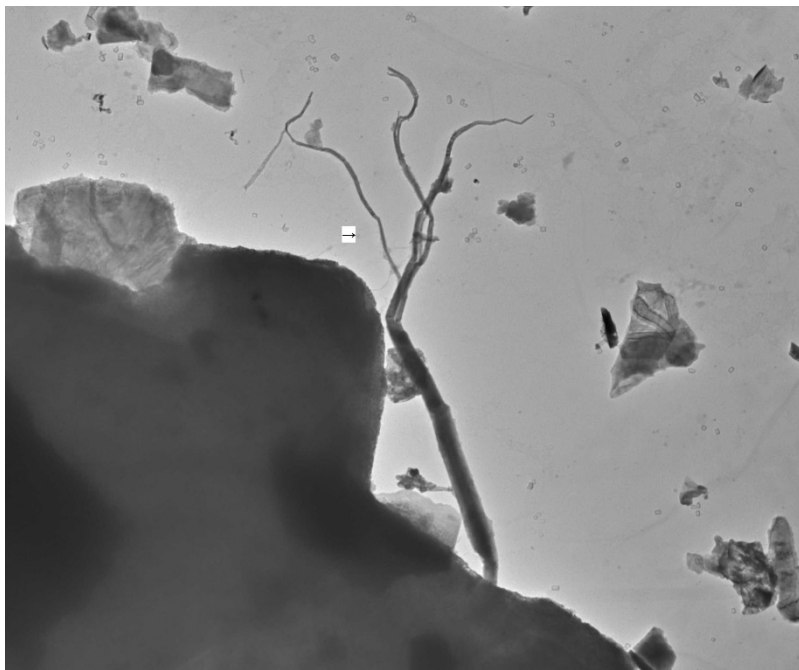
308004 FDA\_014.jpg  
Talc Fiber  
16:23 7/29/2019  
TEM Mode: Diffraction  
Microscopist: MG  
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1  
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from the Talc Fiber pictured above



Talc Ribbon from 308004-1



308004 FDA\_009.jpg  
Talc Ribbon  
Cal: 0.007349  $\mu\text{m}/\text{pix}$   
15:43 7/29/2019  
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

2  $\mu\text{m}$   
HV=100kV  
Direct Mag: 1400 x  
AMA Analytical Services, Inc

Diffraction Pattern from the Talc Ribbon pictured above



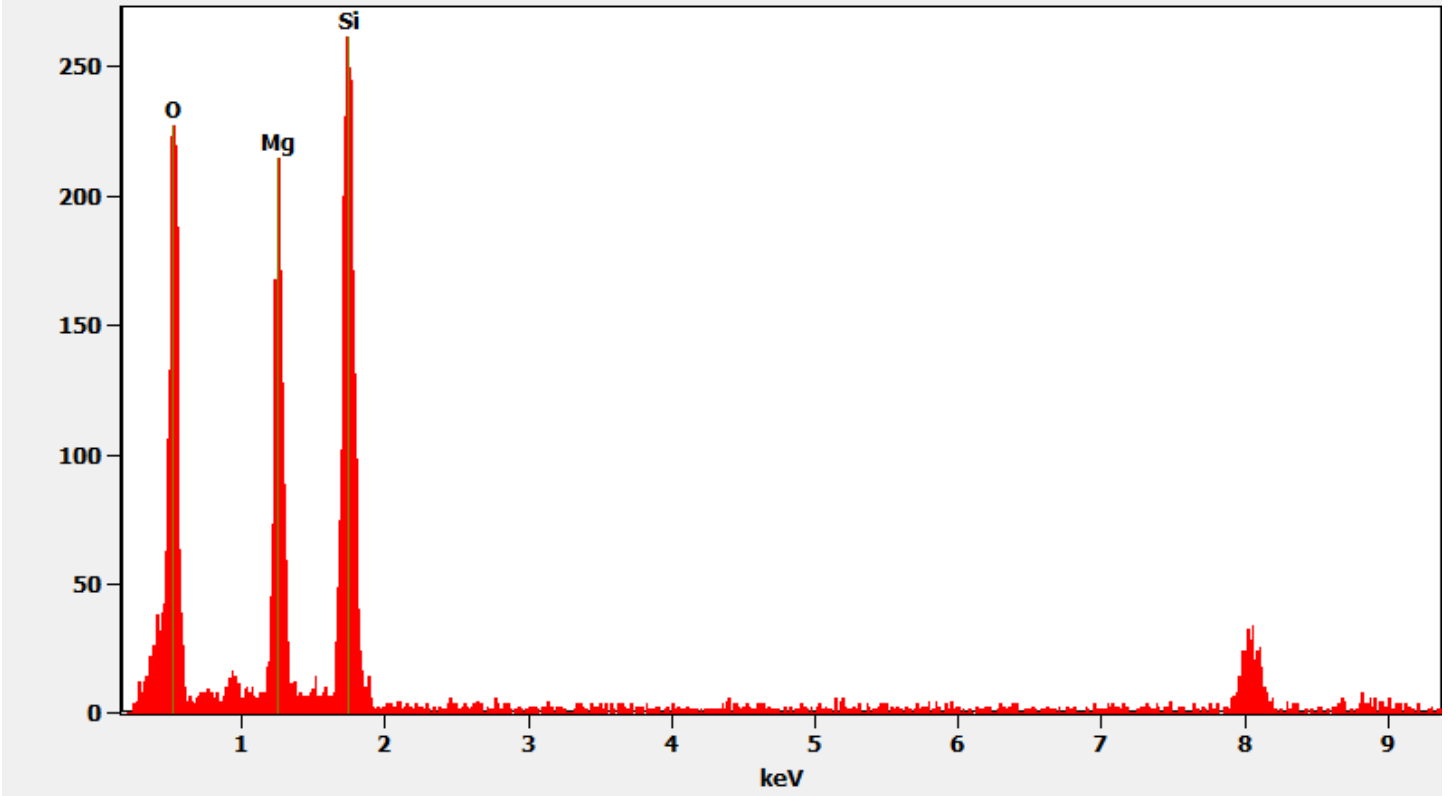
308004 FDA\_010.jpg  
Talc Ribbon  
15:45 7/29/2019  
TEM Mode: Diffraction  
Microscopist: MG  
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1  
Gamma: 1.00, No Sharpening, Normal Contrast

100 (1/Å)  
HV=100kV  
Cam Len: 0.2200 m  
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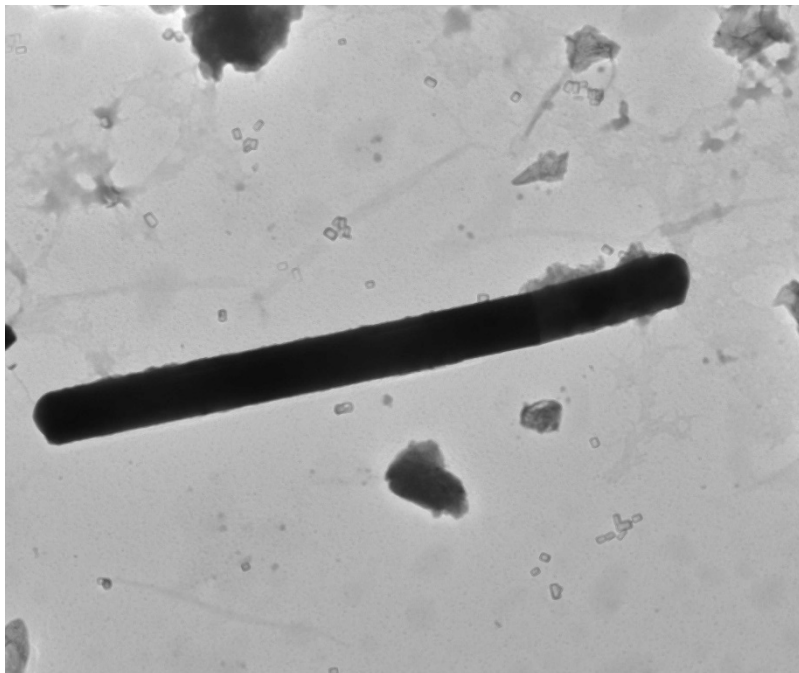
Chemistry from the Talc Ribbon pictured above

Full scale counts: 262

308004-1(6)



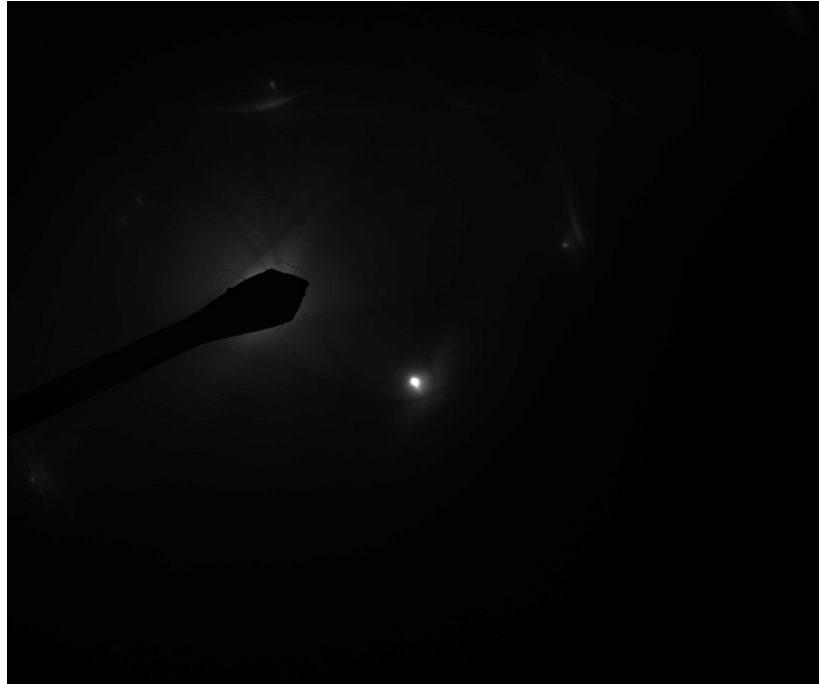
Titanium Fiber from 308004-1



308004 FDA\_001.jpg  
Titanium Fiber  
Cal: 0.003548  $\mu\text{m}/\text{pix}$   
15:25 7/29/2019  
TEM Mode: Imaging  
Microscopist: MG  
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 std. frames, Gain: 1, Bin: 1  
Gamma: 1.00, No Sharpening, Normal Contrast

1  $\mu\text{m}$   
HV=100kV  
Direct Mag: 2900 x  
AMA Analytical Services, Inc

Diffraction Pattern from the Titanium Fiber pictured above



308004 FDA\_002.jpg

Titanium Fiber

15:26 7/29/2019

TEM Mode: Diffraction

Microscopist: MG

Camera: NANOSPRT5, Exposure: 800 (ms) x 5 std. frames, Gain: 1, Bin: 1

Gamma: 1.00, No Sharpening, Normal Contrast

100 (1/A)

HV=100kV

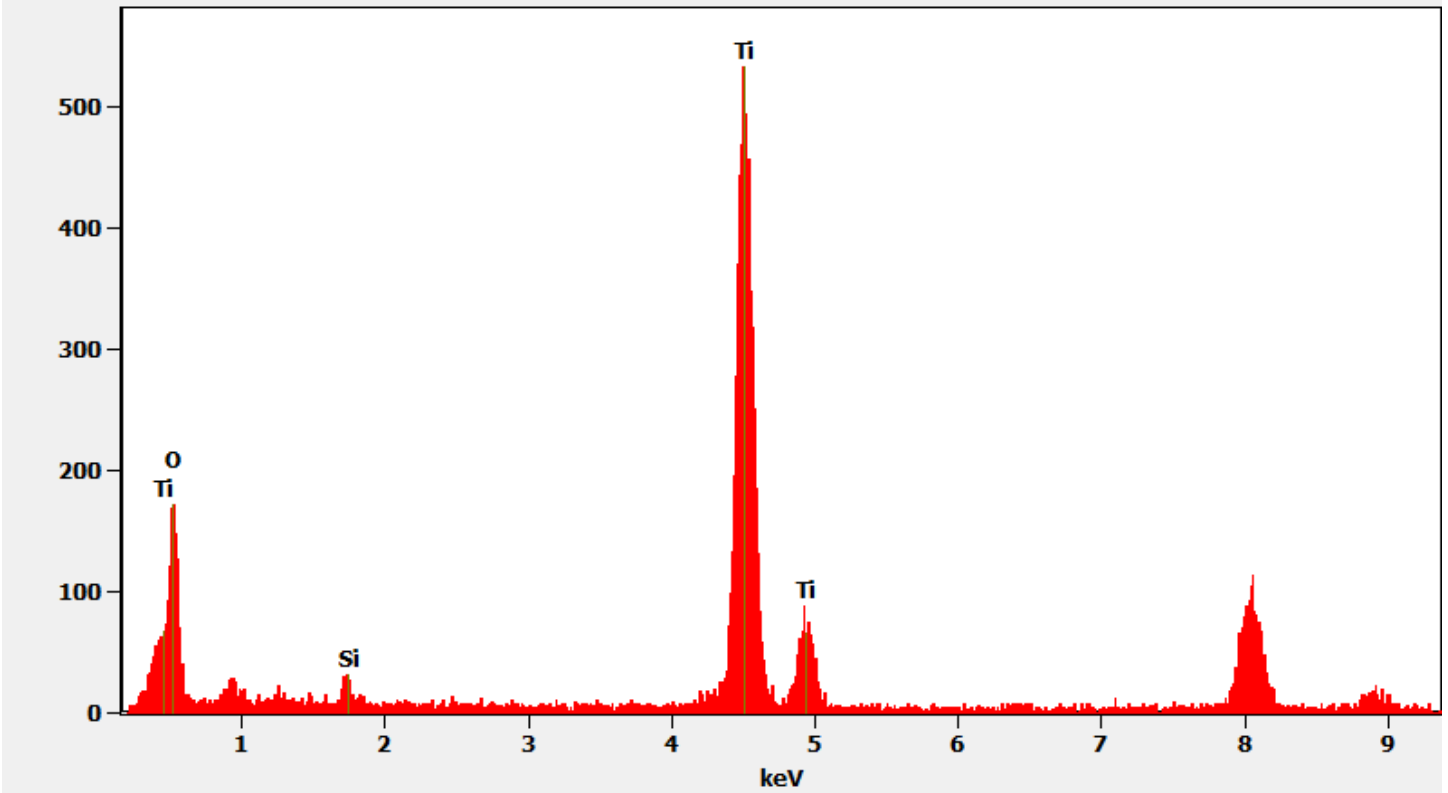
Cam Len: 0.2200 m

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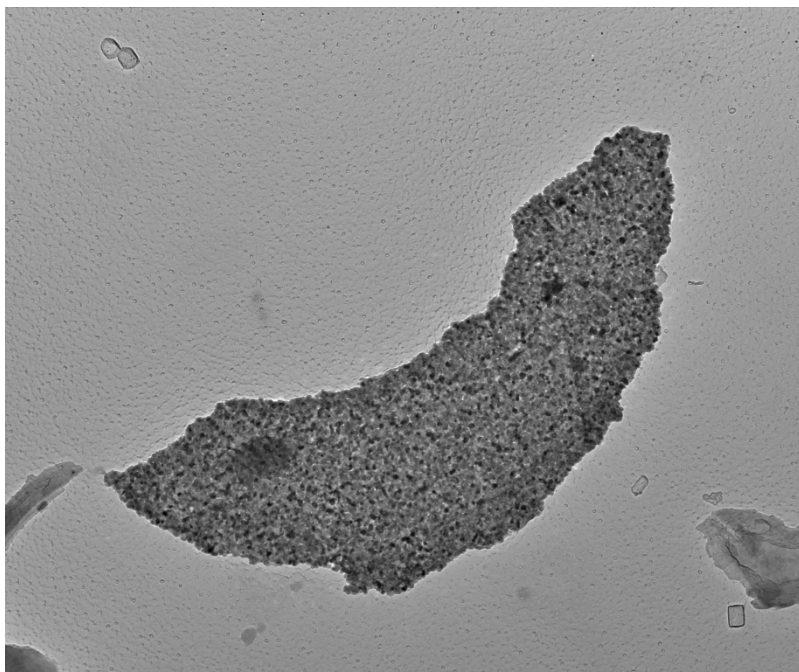
Chemistry from the Titanium Fiber pictured above

Full scale counts: 535

308004-1(1)



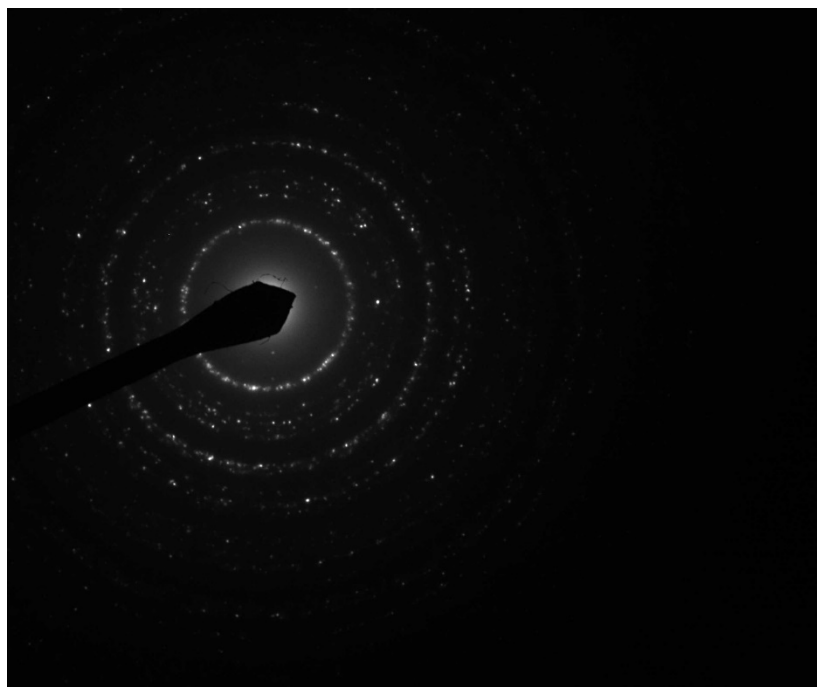
Particle coated with Titanium from 308004-1



308004 FDA\_015.jpg  
Titanium particle or titanium coated particle  
Cal: 0.001774  $\mu\text{m}/\text{pix}$   
16:25 7/29/2019  
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 the Titanium Coated particle pictured above



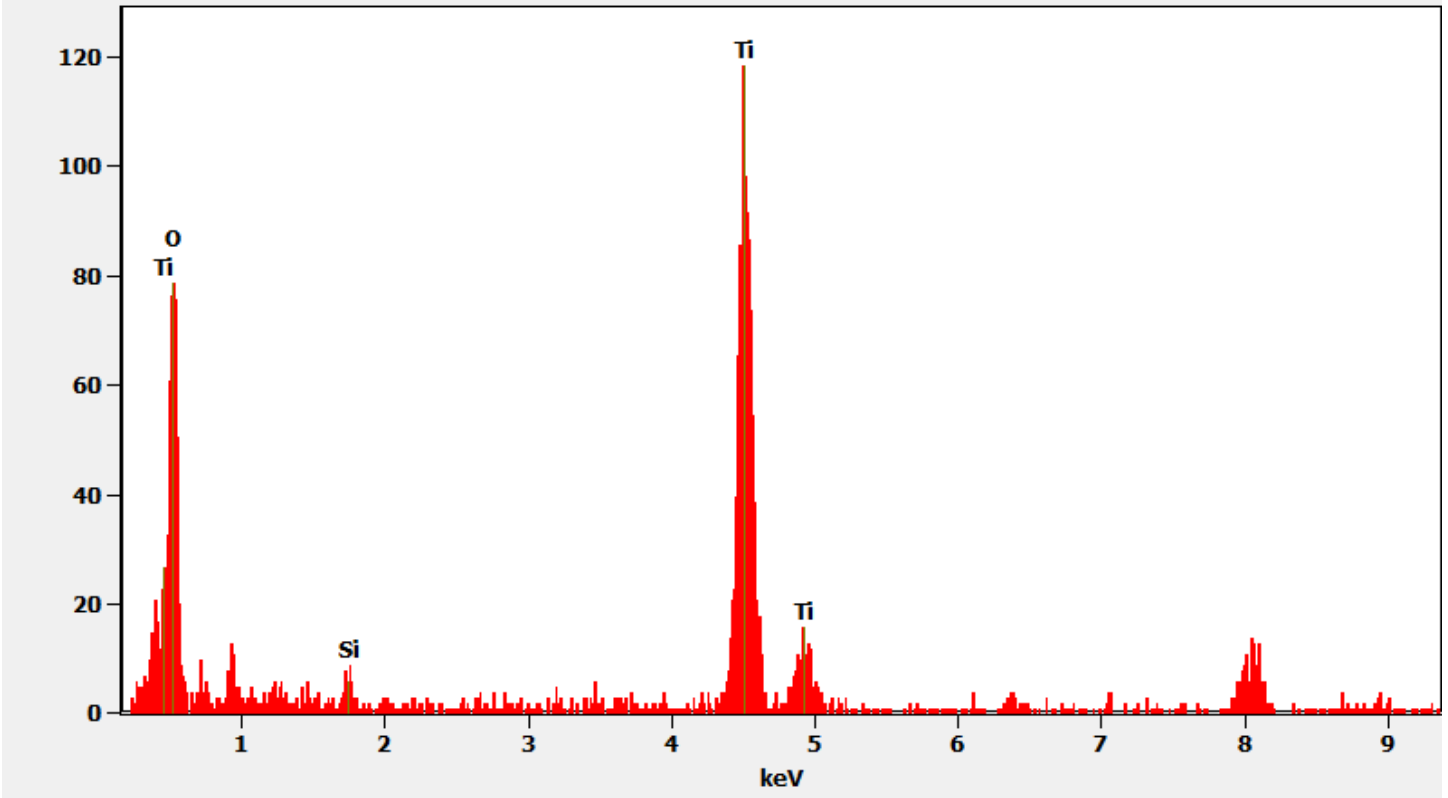
308004 FDA\_016.jpg  
Titanium particle or titanium coated particle  
16:26 7/29/2019  
TEM Mode: Diffraction  
Microscopist: MG  
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1  
Gamma: 1.00, No Sharpening, Normal Contrast

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

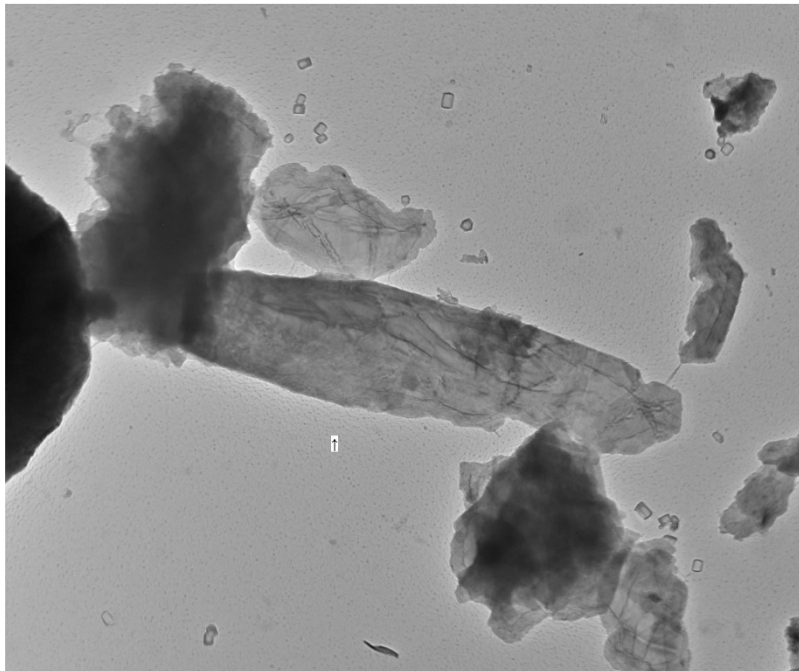
Chemistry from the Titanium Coated Particle pictured above

Full scale counts: 119

308004-1(11)



Elongated Mica Particle from 308004-1



308004 FDA\_112.jpg  
Elongated Mica Particle  
Cal: 0.002858 µm/pix  
13:12 8/12/2019  
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

800 nm  
HV=100kV  
Direct Mag: 3600 x  
AMA Analytical Services, Inc



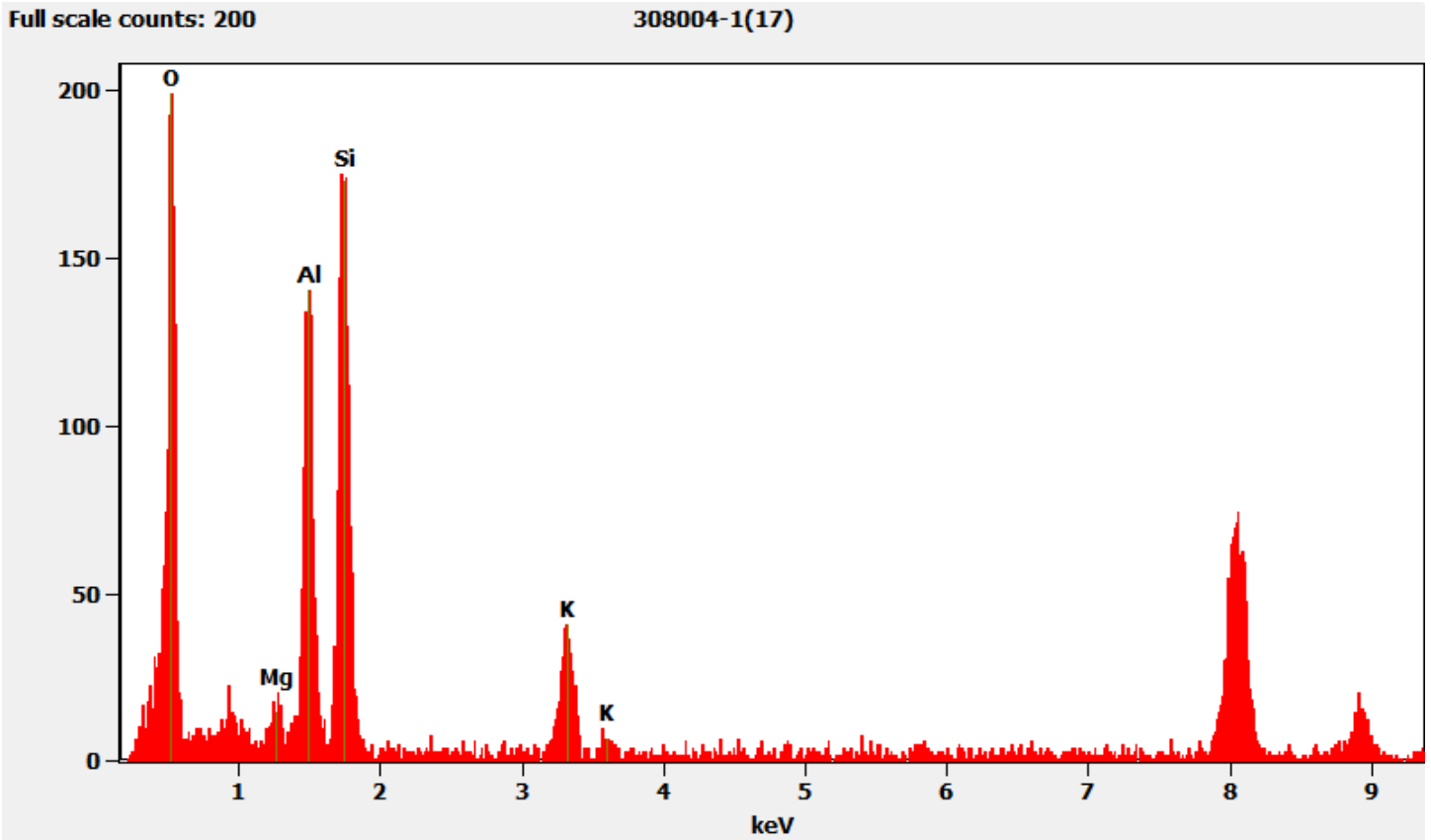
Diffraction Pattern from the Elongated Mica Particle pictured above



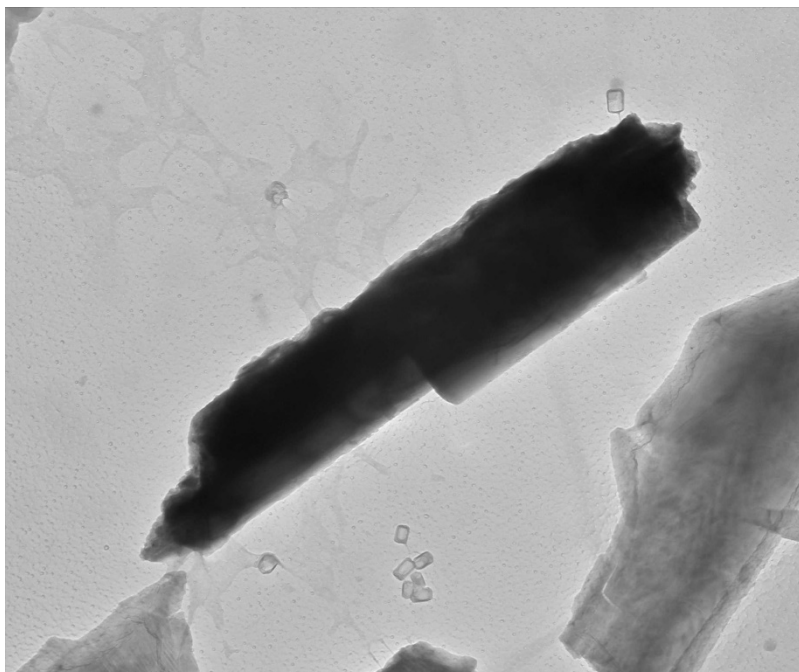
308004 FDA\_113.jpg  
Elongated Mica Particle  
13:13 8/12/2019  
TEM Mode: Diffraction  
Microscopist: MG  
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1  
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from the Elongated Mica Particle pictured above



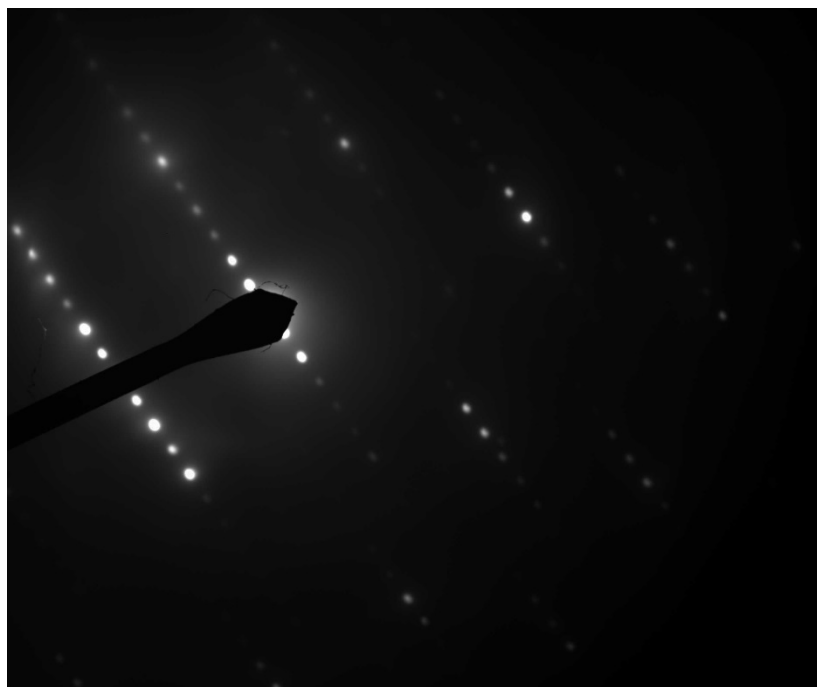
Tremolite Particle from 308004-1



308004 FDA\_021.jpg  
Tremolite 2  
Cal: 0.001774  $\mu\text{m}/\text{pix}$   
16:46 7/29/2019  
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 the Tremolite Particle pictured above



308004 FDA\_022.jpg  
Tremolite 2  
16:51 7/29/2019  
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

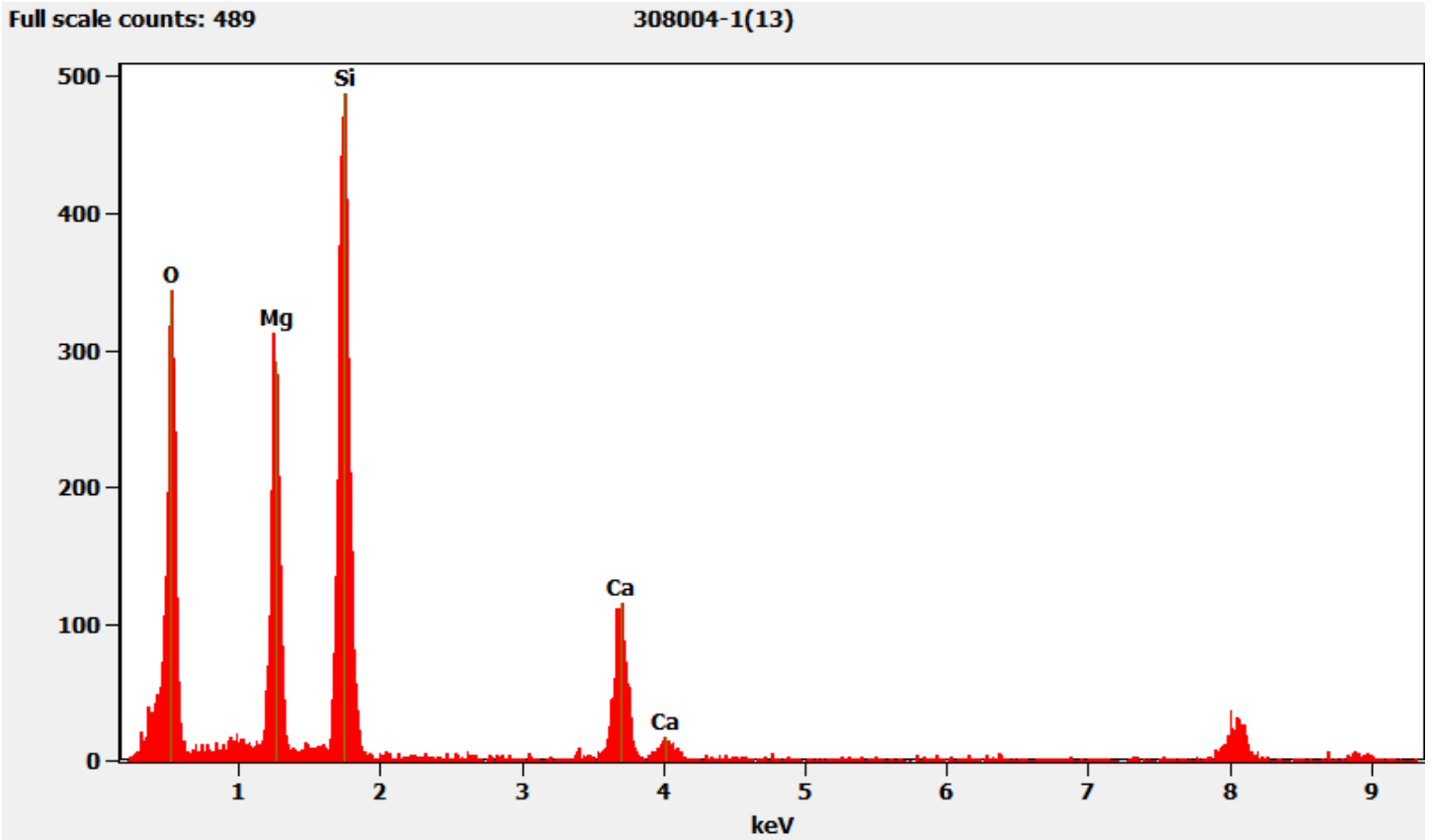
Diffraction Pattern from the Tremolite Particle pictured above



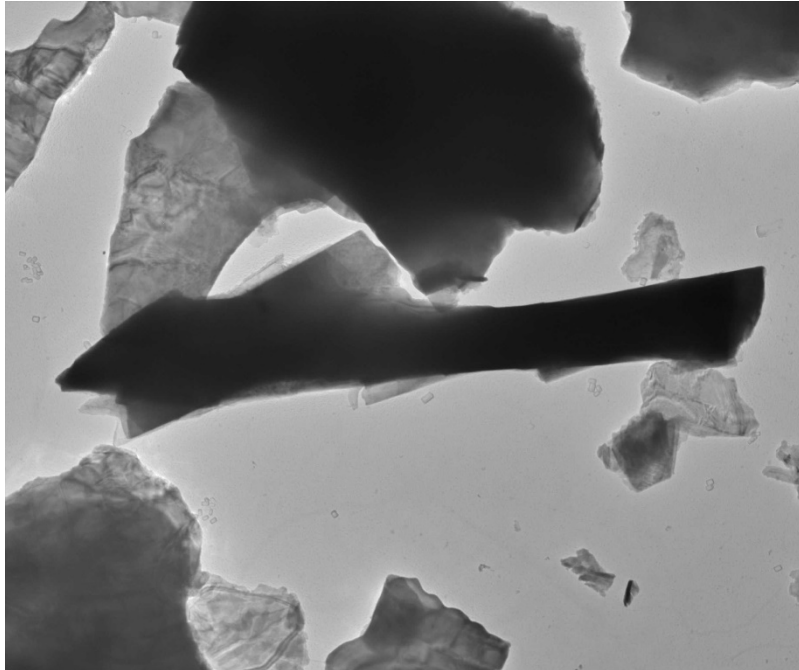
308004 FDA\_023.jpg  
Tremolite 2  
16:52 7/29/2019  
TEM Mode: Diffraction  
Microscopist: MG  
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1  
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from the Tremolite Particle pictured above



Tremolite Particle from 308004-1



308004 FDA\_024.jpg  
Tremolite 3  
Cal: 0.005415  $\mu\text{m}/\text{pix}$   
17:07 7/29/2019  
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

1  $\mu\text{m}$   
HV=100kV  
Direct Mag: 1900 x  
AMA Analytical Services, Inc

Diffraction Pattern from the Tremolite Particle pictured above



308004 FDA\_025.jpg  
Tremolite 3  
17:08 7/29/2019  
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

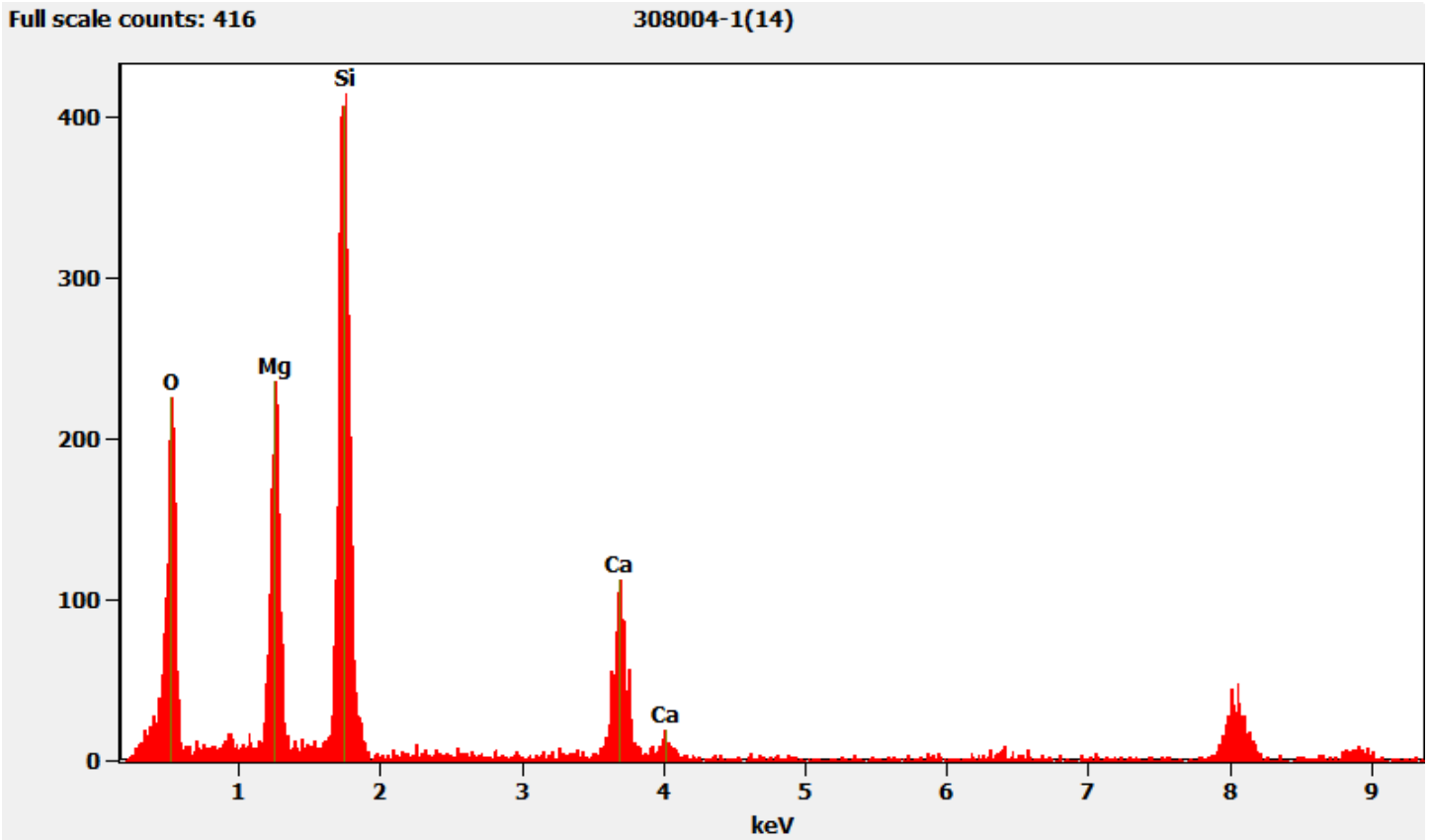
Diffraction Pattern from the Tremolite Particle pictured above



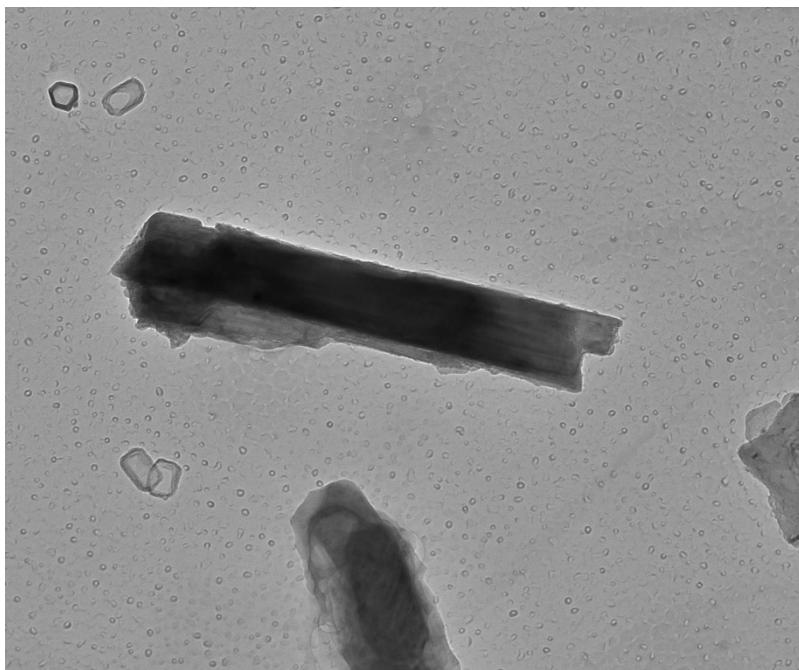
308004 FDA\_026.jpg  
Tremolite 3  
17:11 7/29/2019  
TEM Mode: Diffraction  
Microscopist: MG  
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1  
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from the Tremolite Particle pictured above



Tremolite Particle from 308004-1A



308004 FDA\_046.jpg  
Tremolite 1  
Cal: 0.001029  $\mu\text{m}/\text{pix}$   
18:11 7/30/2019  
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

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

Diffraction Pattern from the Tremolite Particle pictured above

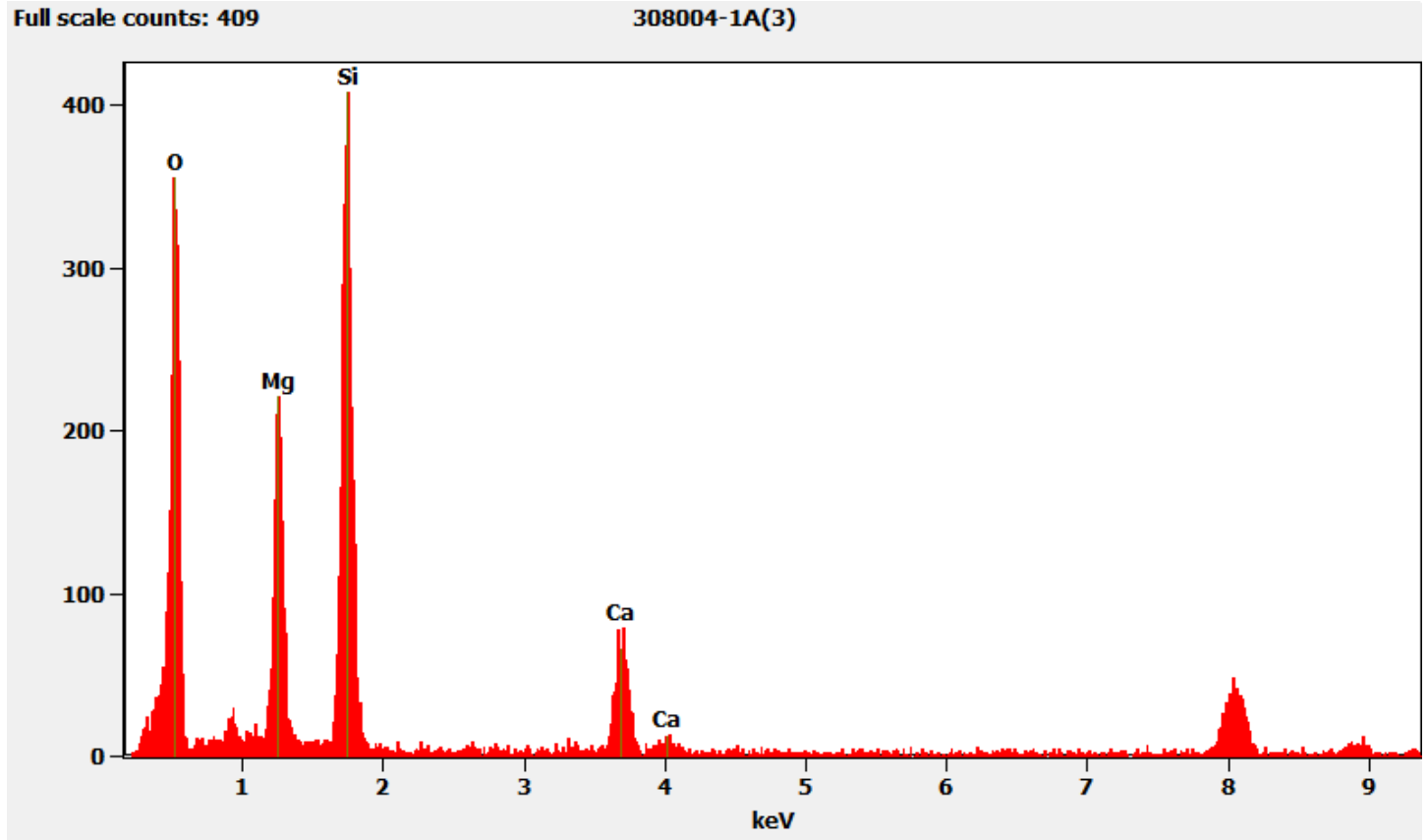


308004 FDA\_047.jpg  
Tremolite 1  
18:14 7/30/2019  
TEM Mode: Diffraction  
Microscopist: MG  
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1  
Gamma: 1.00, No Sharpening, Normal Contrast

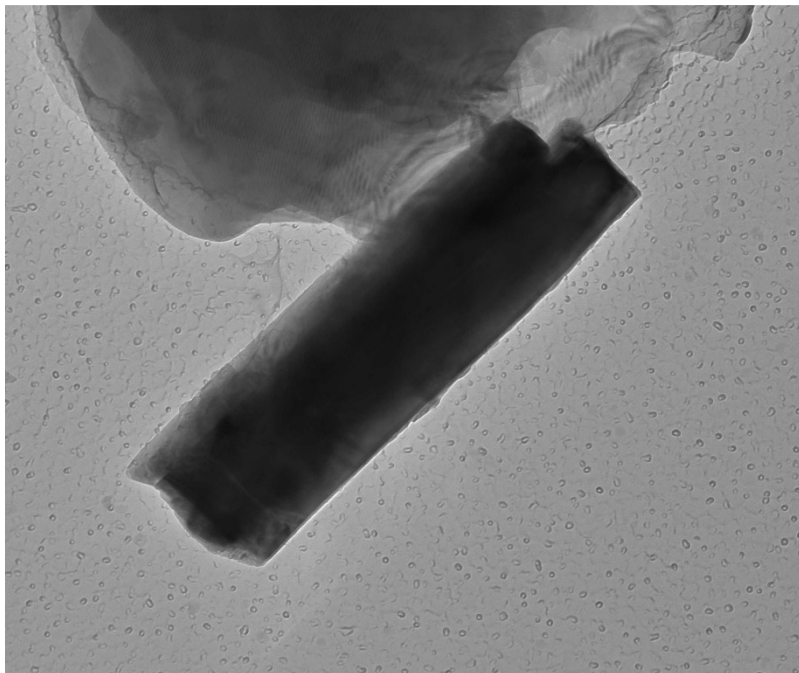
100 (1/Å)  
HV=100kV  
Cam Len: 0.2200 m  
AMA Analytical Services, Inc



Chemistry from the Tremolite Particle pictured above



Tremolite Particle from 308004-1B



308004 FDA\_055.jpg  
Tremolite 1  
Cal: 0.001029  $\mu\text{m}/\text{pix}$   
14:28 8/1/2019  
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

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

Diffraction Pattern from the Tremolite Particle pictured above



308004 FDA\_056.jpg  
Tremolite 1  
14:31 8/1/2019  
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/A)  
HV=100kV  
Cam Len: 0.2200 m  
AMA Analytical Services, Inc

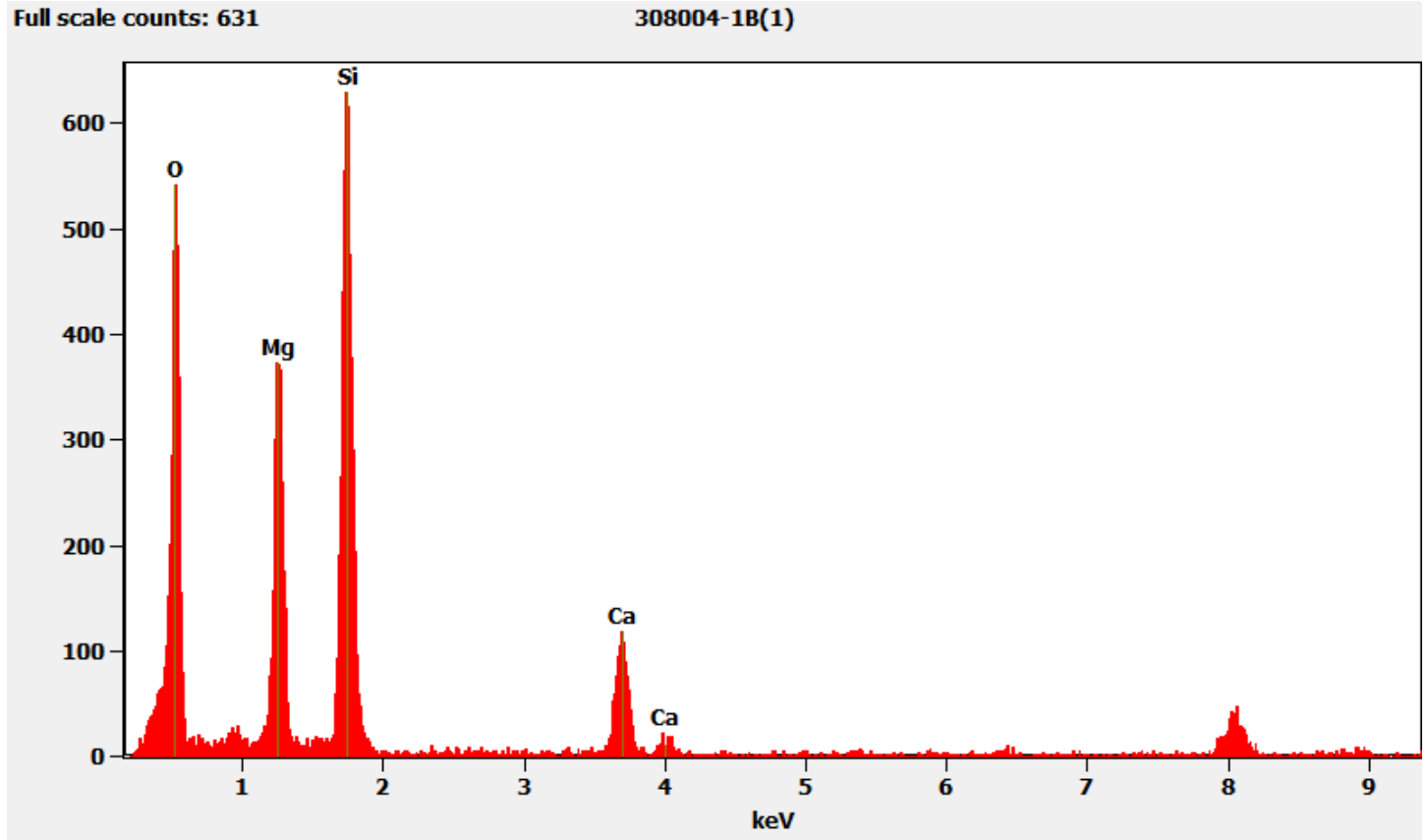
Diffraction Pattern from the Tremolite Particle pictured above



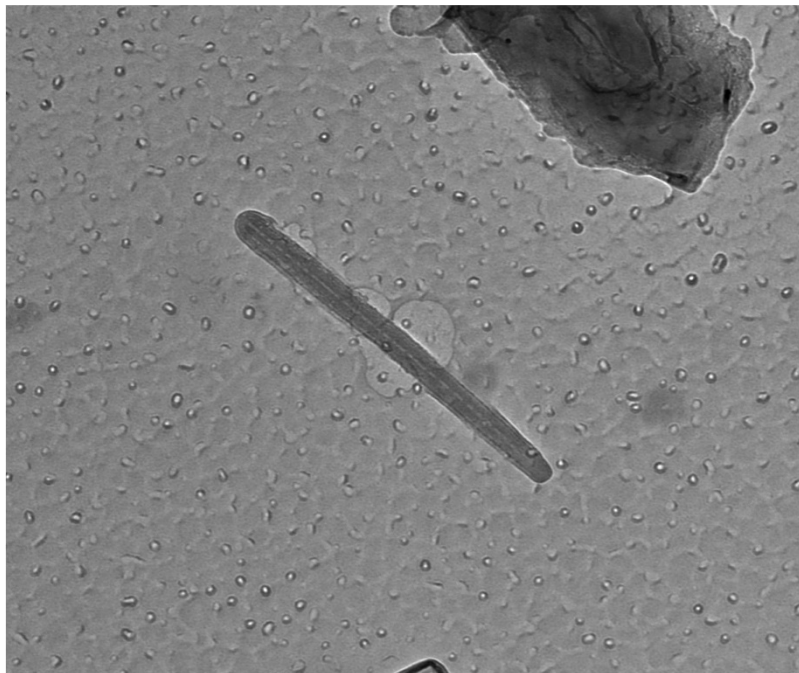
308004 FDA\_057.jpg  
Tremolite 1  
14:32 8/1/2019  
TEM Mode: Diffraction  
Microscopist: MG  
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1  
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from the Tremolite Particle pictured above



Chrysotile Fiber from 308004-1



308004 FDA\_052.jpg  
Chrysotile 4  
Cal: 0.541520 nm/pix  
18:35 7/30/2019  
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

100 nm  
HV=100kV  
Direct Mag: 19000 x  
AMA Analytical Services, Inc

*Diffraction Pattern from the Chrysotile Fiber pictured above*



308004 FDA\_050.jpg  
Chrysotile 4  
18:33 7/30/2019  
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/A)  
HV=100kV  
Cam Len: 0.2200 m  
AMA Analytical Services, Inc

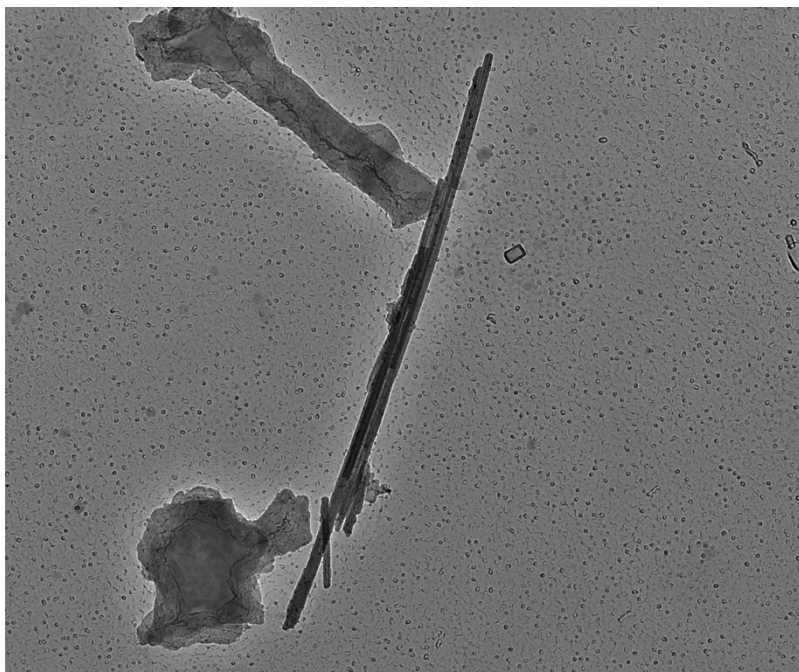
*Diffraction Pattern from the Chrysotile Fiber pictured above*



308004 FDA\_051.jpg  
Chrysotile 4  
18:34 7/30/2019  
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/A)  
HV=100kV  
Cam Len: 0.2200 m  
AMA Analytical Services, Inc

Chrysotile Structure from 308004-1B



308004 FDA\_062.jpg  
Chrysotile 3  
Cal: 0.001774  $\mu\text{m}/\text{pix}$   
14:54 8/1/2019  
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

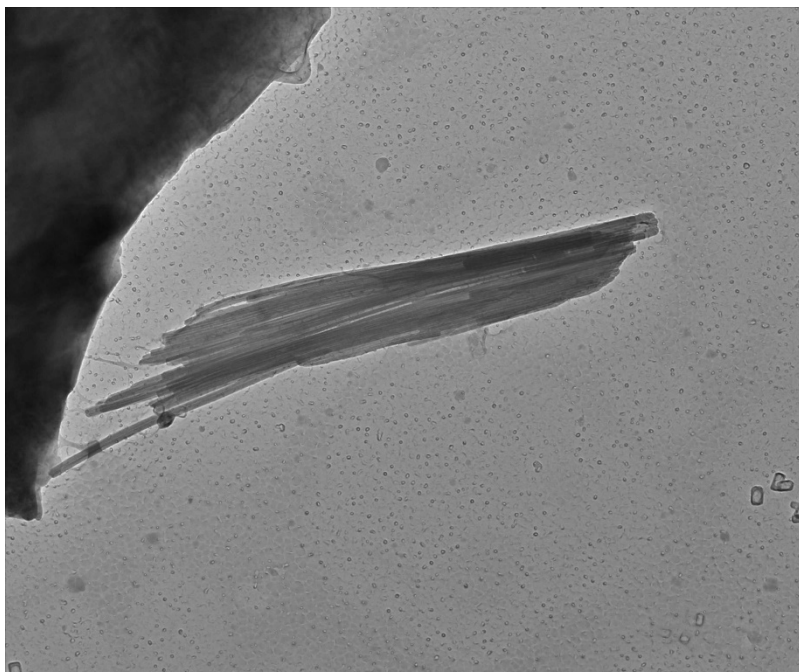
Diffraction Pattern from the Chrysotile Structure pictured above



308004 FDA\_061.jpg  
Chrysotile 3  
14:53 8/1/2019  
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

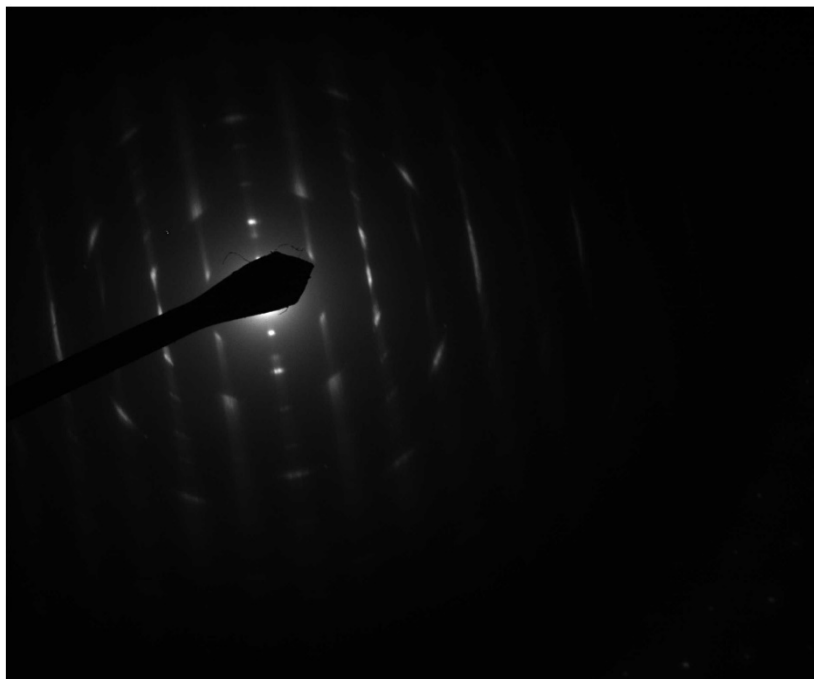
Chrysotile Structure from 308004-1B



308004 FDA\_064.jpg  
Chrysotile 4  
Cal: 0.001774  $\mu\text{m}/\text{pix}$   
14:59 8/1/2019  
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 the Chrysotile Structure pictured above



308004 FDA\_063.jpg  
Chrysotile 4  
14:58 8/1/2019  
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



**QC Discussion:**

During preparation, one blank control sample and one reference control sample were prepared. These samples were prepared alongside the customer samples. The blank sample was prepared using Sigma-Aldrich Talc Powder, <10 micron. No asbestos was detected on the blank sample. The reference sample was made from the same Sigma-Aldrich talc powder spiked with 1% Chrysotile. The reference sample was analyzed by and found to be within acceptable limits.

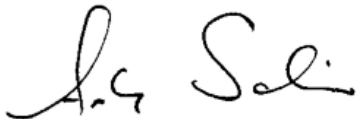
Our LIMS randomly selects samples for additional replicate and duplicate QC. 308004-1, 1A, and 1B/D-49 were not selected for any additional QC analysis.

**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 and Duplicate QC Charts for Peerawut Chaikenee for samples analyzed between 1/1/2019 & 8/8/2019
- 7) Replicate and Duplicate QC Charts for Michael Greenberg for samples analyzed between 1/1/2019 & 8/8/2019
- 8) 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.



8/14/2019

Andreas Saldivar

Date

Laboratory Director