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Focused On Results.

CERTIFICATE OF ANALYSIS

Chain of Custody: 308004
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: 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%	

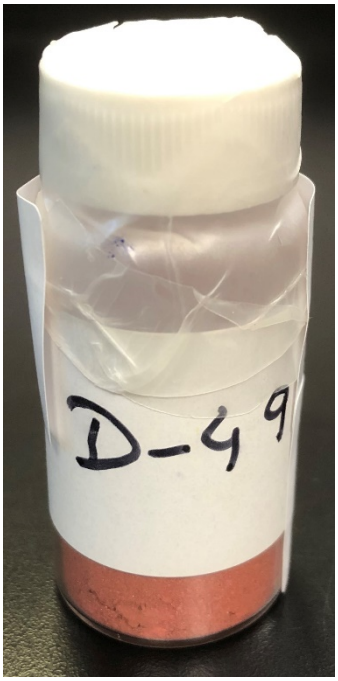
LOD = Limit of Detection **LOQ** = Limit of Quantification **ND** = Not Detected **PLM** = Polarized Light Microscopy **TEM** = Transmission Electron Microscopy

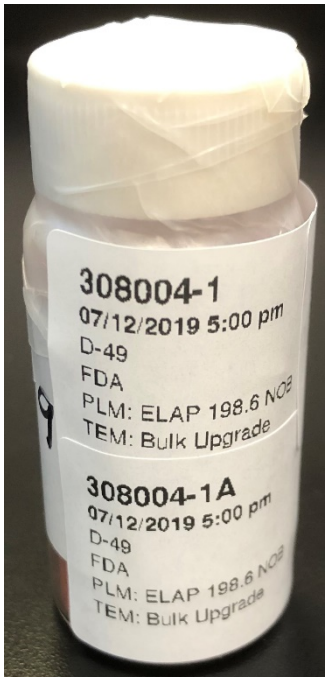
Analytical Method(s): PLM by Modified NY ELAP 198.6
TEM by Modified NY ELAP 198.4/ASTM D5756

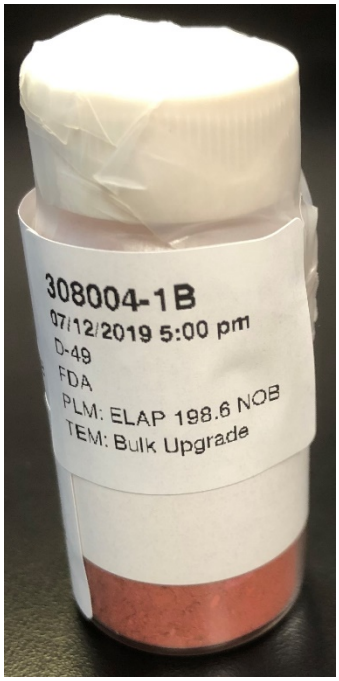
Analyst(s): **PLM** (b) (6)
TEM (b) (6)

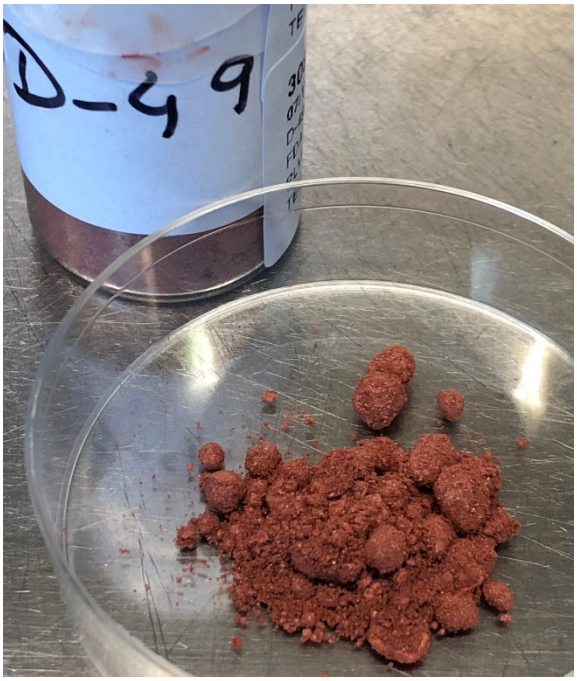
Technical Director: Andreas Saldivar

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









Sample Preparation

Samples were prepared for PLM and TEM bulk analysis by (b) (6) 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

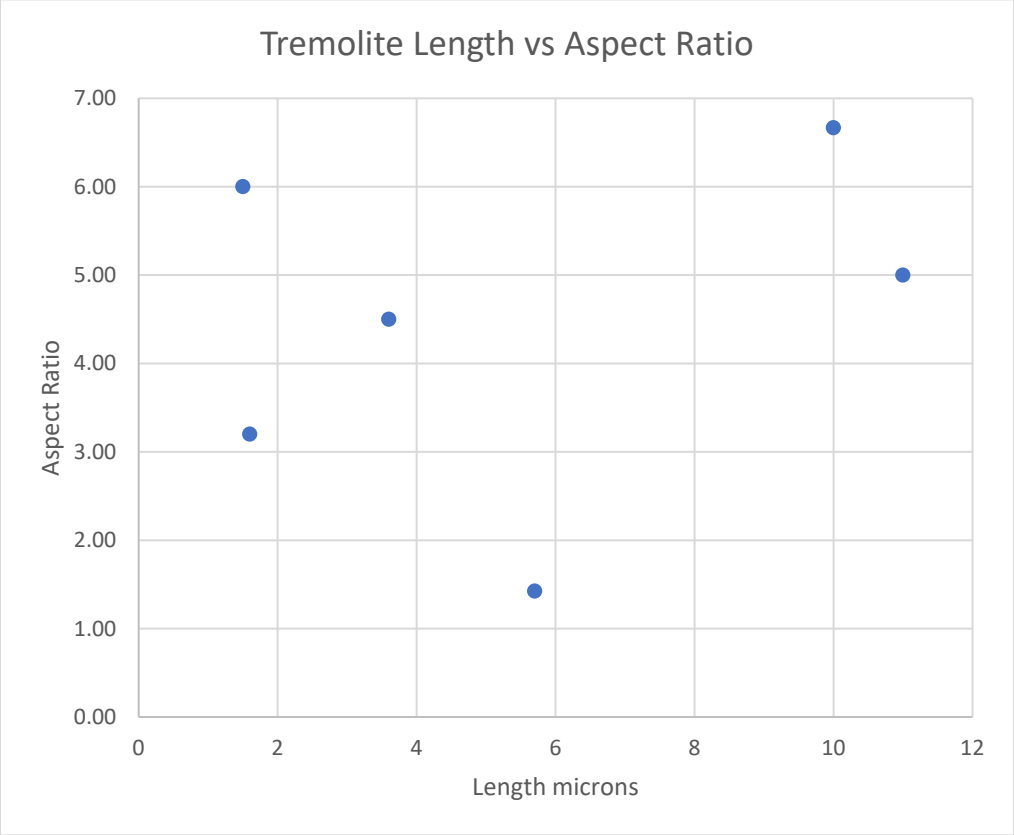
Discussion and Interpretation of Analytical Findings:
PLM
All three aliquots of sample D-49 were analyzed by (b) (6) on July 25, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

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

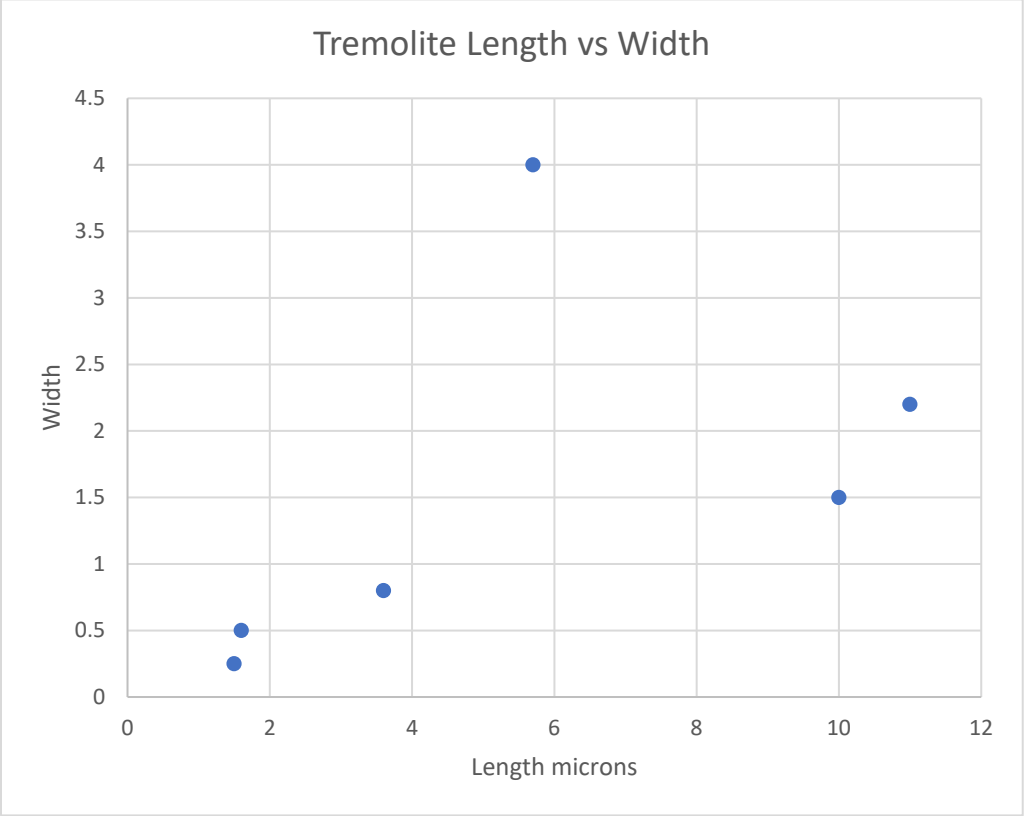
TEM
(b) (6) analyzed sample 1 on July 29, 2019, 1A on July 30, 2019 and 1B on August 1, 2019. The sample consisted of a mix of talc and mica particles, with a few talc fibers, mica fibers and titanium fibers/particles. Chrysotile and tremolite were observed on all three aliquots. The results were calculated 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.

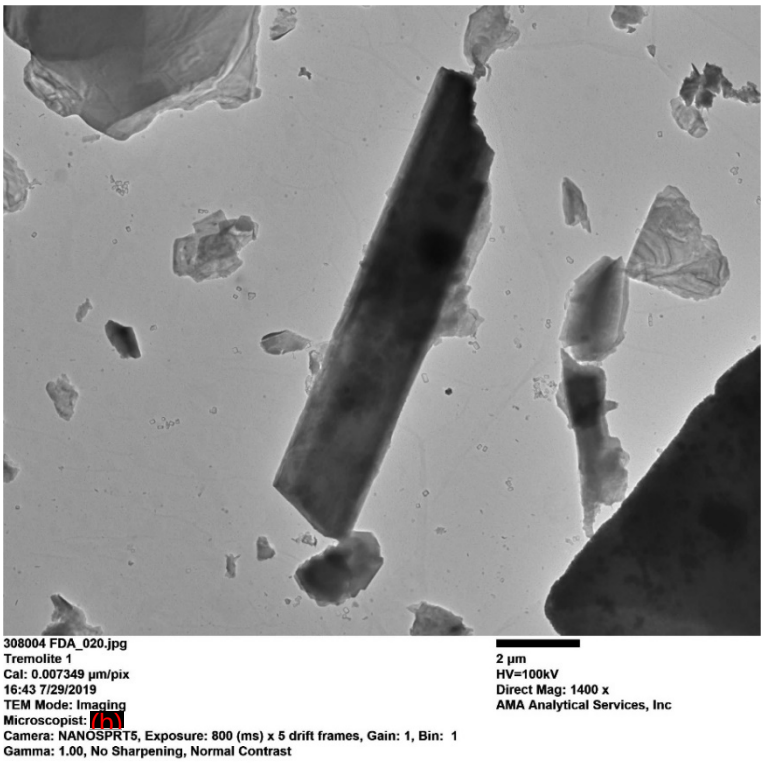

Tremolite Length vs Aspect Ratio
Aspect Ratio
7.00
6.00
5.00
4.00
3.00
2.00
1.00
0.00
0
2
4
6
8
10
12
Length microns


Tremolite Width vs Aspect Ratio
Aspect Ratio
7.00
6.00
5.00
4.00
3.00
2.00
1.00
0.00
0
0.5
1
1.5
2
2.5
3
3.5
4
4.5
Length microns

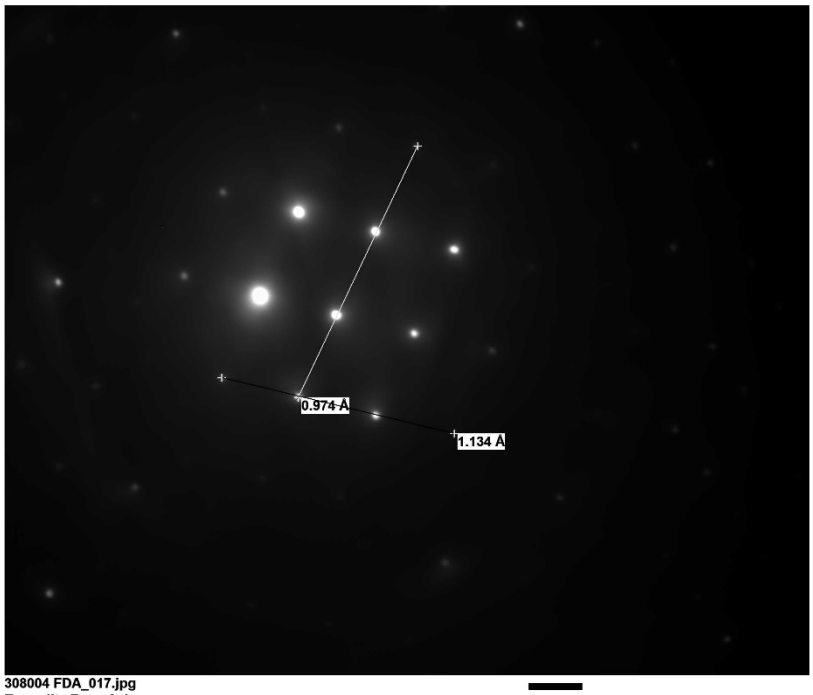


Below are representative pictures, diffraction patterns, and chemistry from the observed particles. The unidentified peaks in chemistry spectra are copper, zinc, and carbon. Those peaks are from the TEM specimen holder and specimen grid.

Tremolite Particle from 308004-1



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: (b)
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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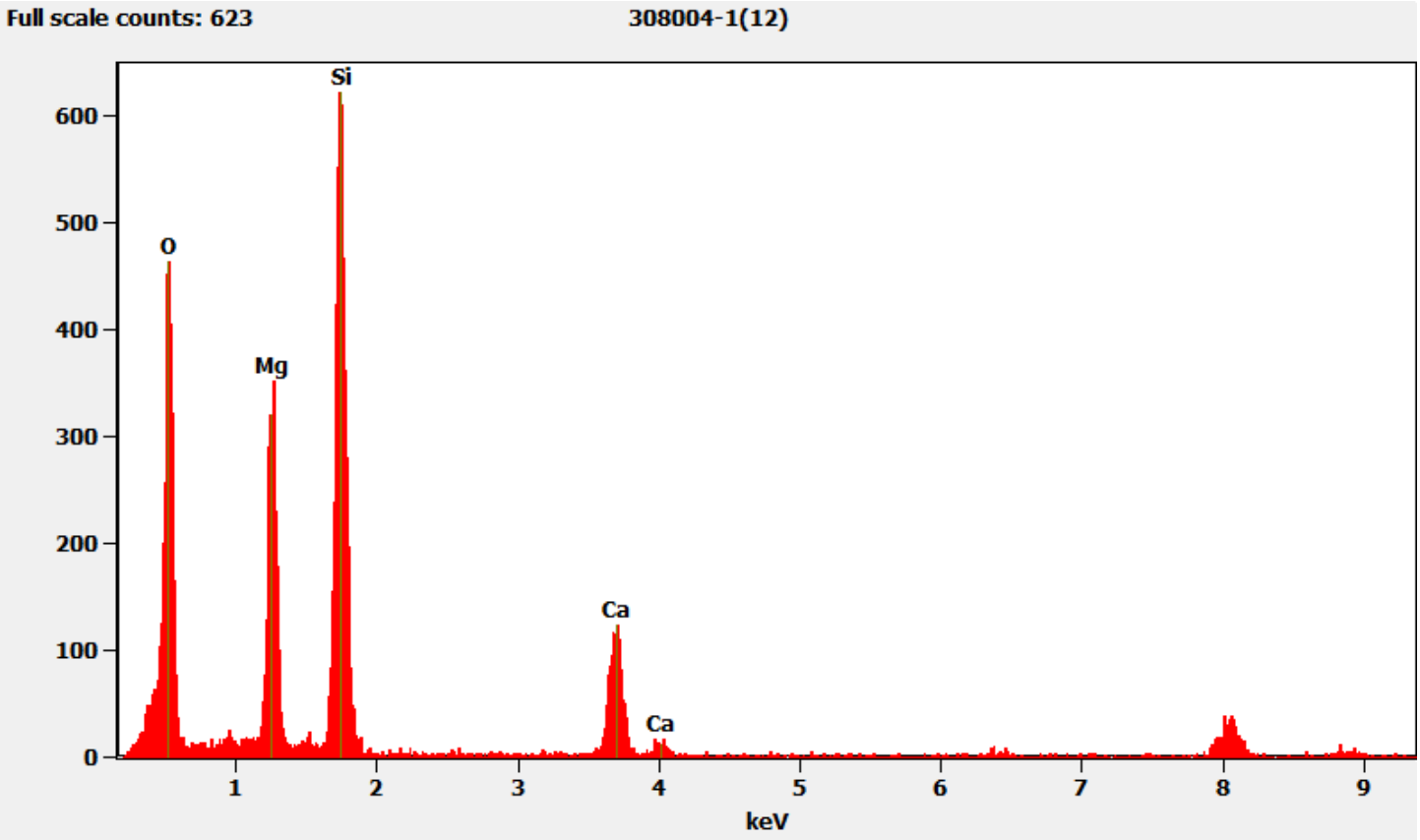
Diffraction Pattern from the Tremolite Particle pictured above

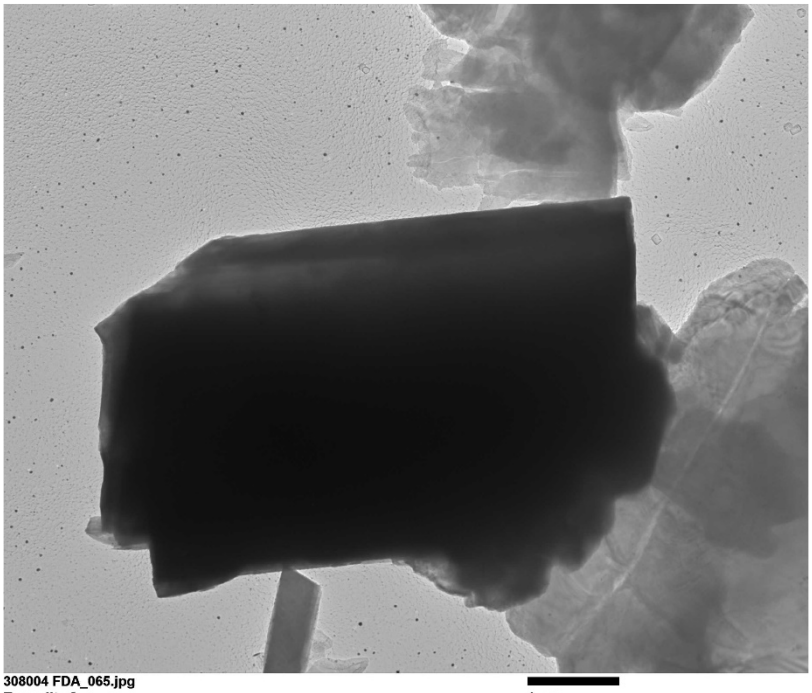
308004 FDA_018.jpg
Tremolite
16:36 7/29/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from the Tremolite Particle pictured above

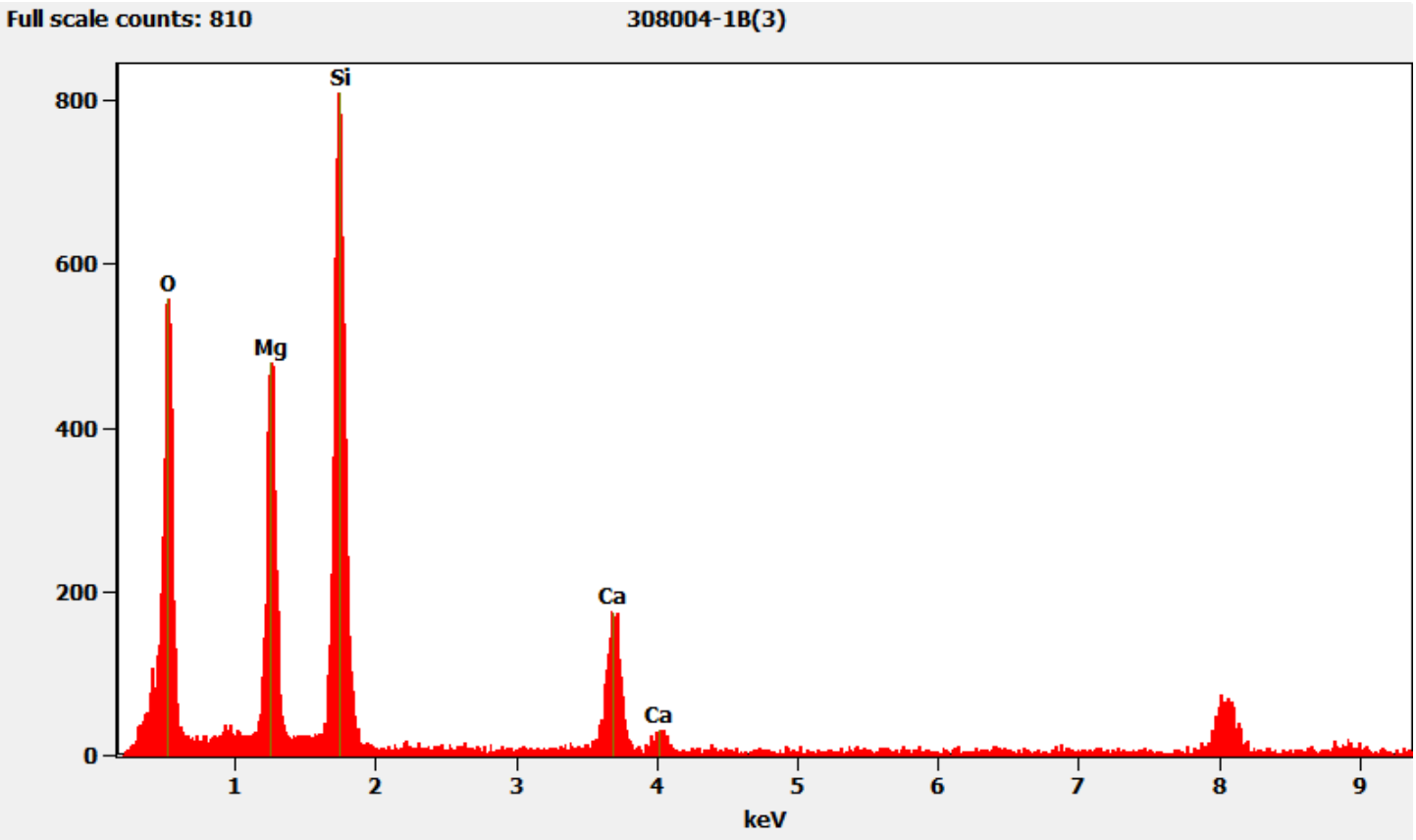


Tremolite Particle from 308004-1B

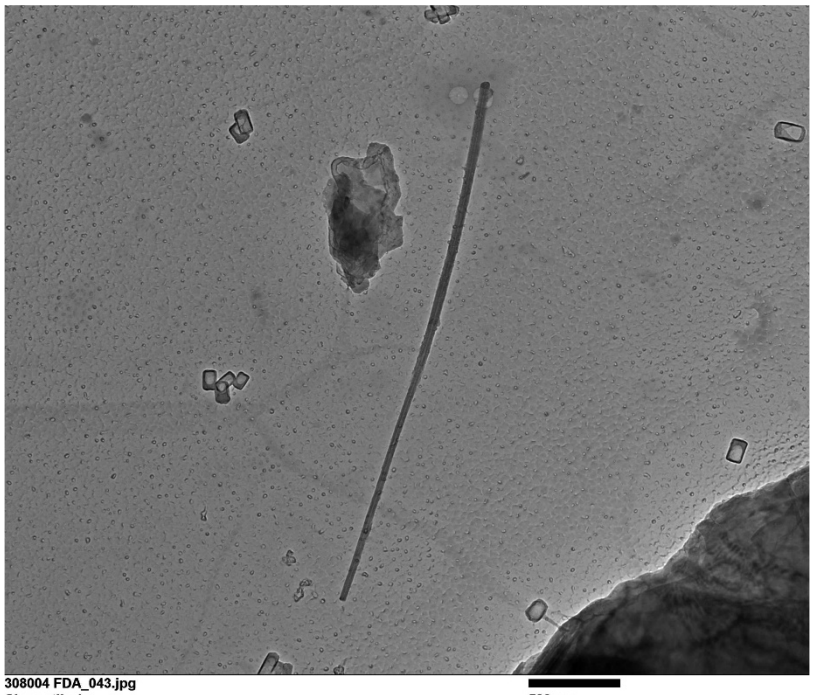


Diffraction Pattern from Tremolite Particle pictured above

Chemistry from Tremolite Particle pictured above



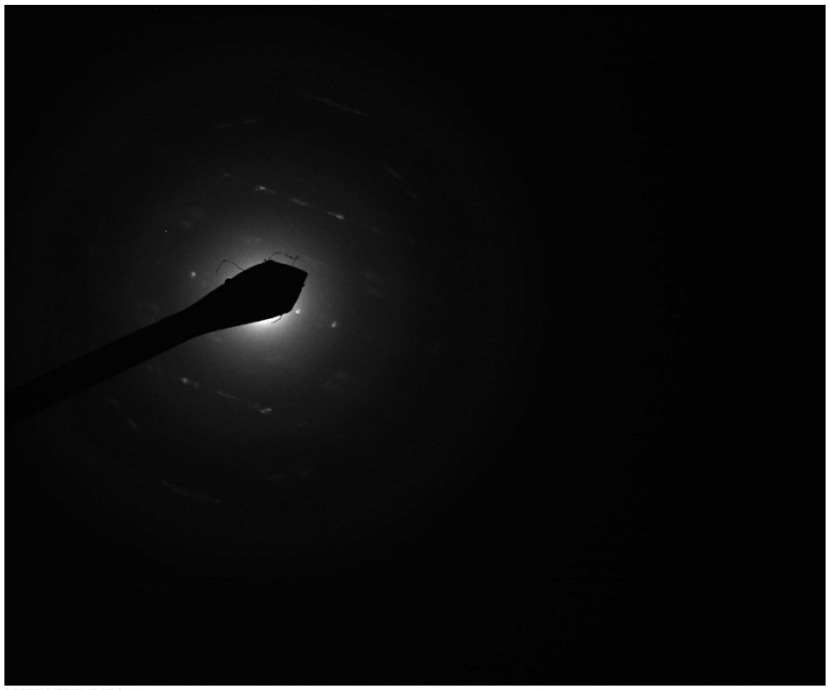
Chrysotile Fiber from 308004-1A



308004 FDA_043.jpg
Chrysotile 1
Cal: 0.001774 µm/pix
17:30 7/30/2019
TEM Mode: Imaging
Microscopist: [redacted]
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

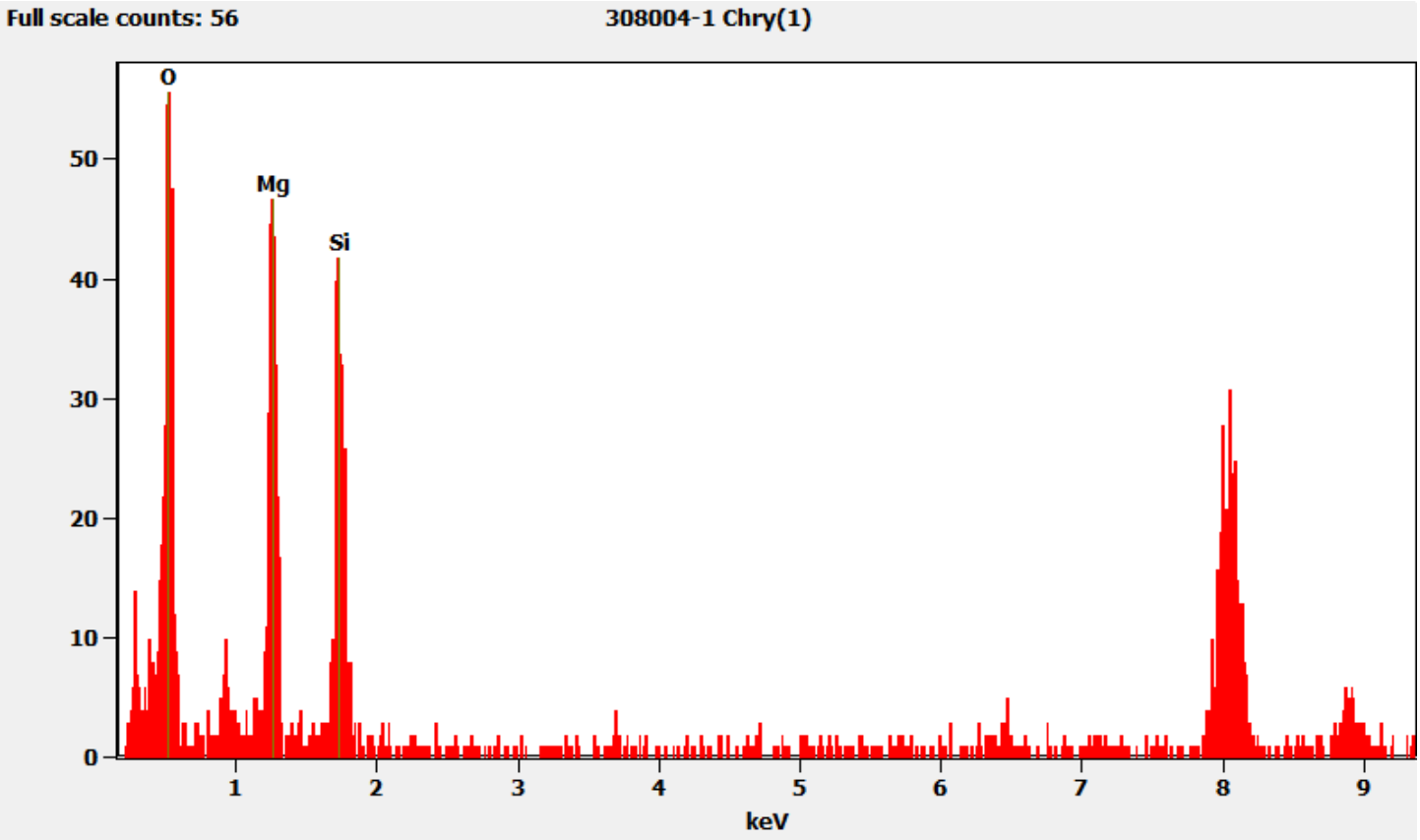
Diffraction Pattern from Chrysotile Fiber pictured above



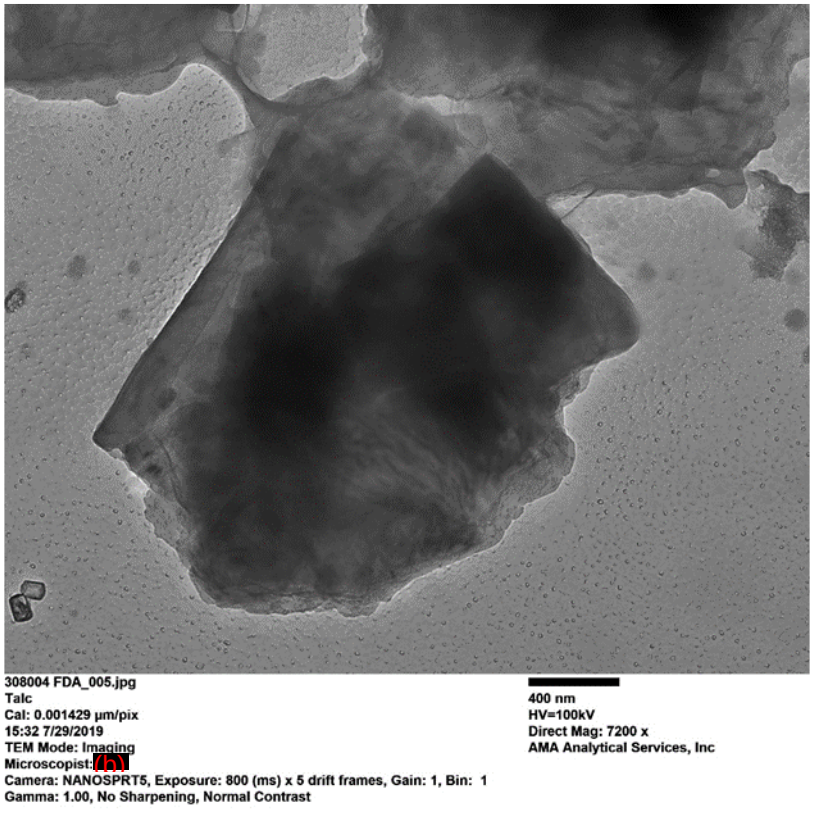
308004 FDA_042.jpg
Chrysotile 1
17:28 7/30/2019
TEM Mode: Diffraction
Microscopist: [redacted]
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from Chrysotile Fiber pictured above

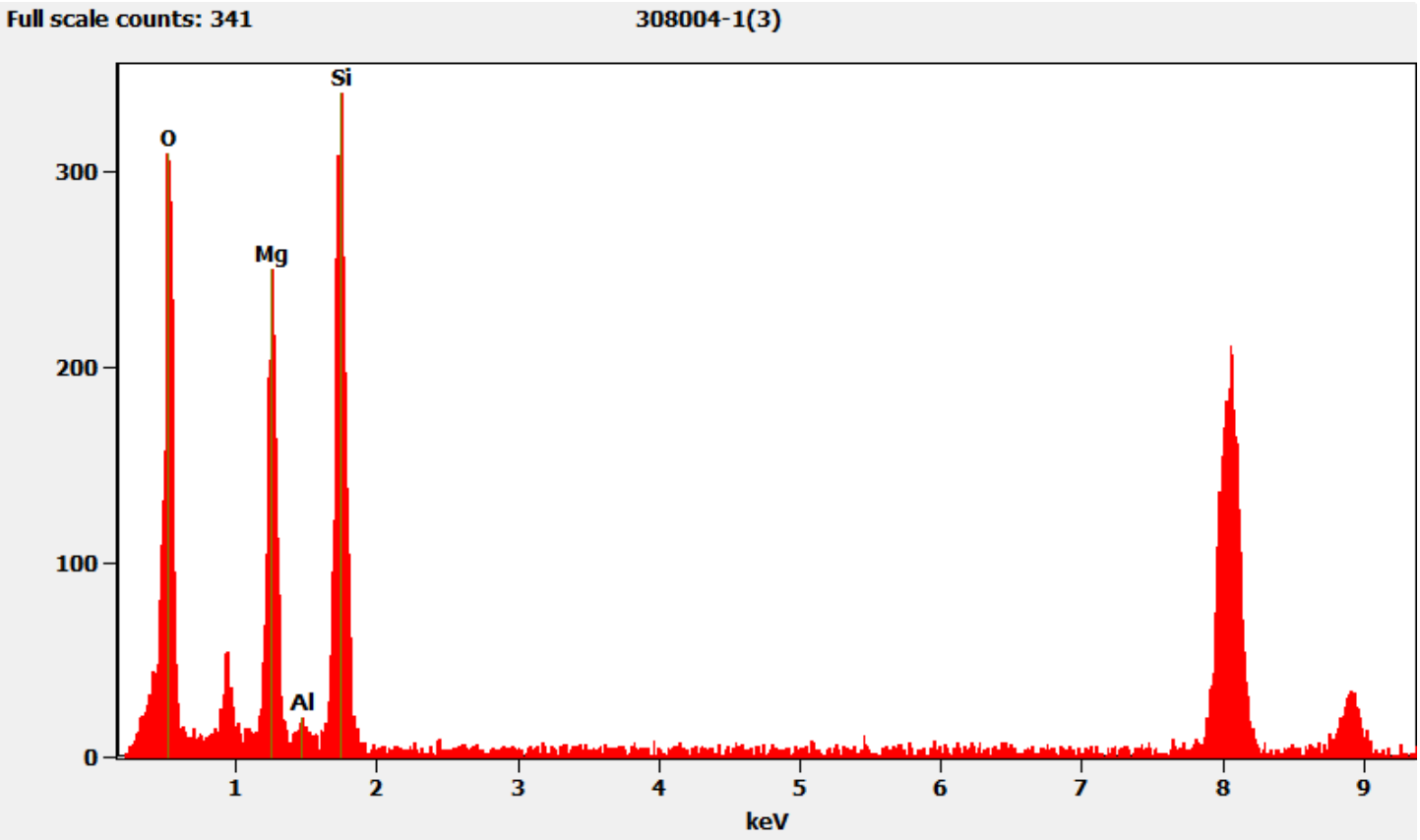


Talc Particle from 308004-1

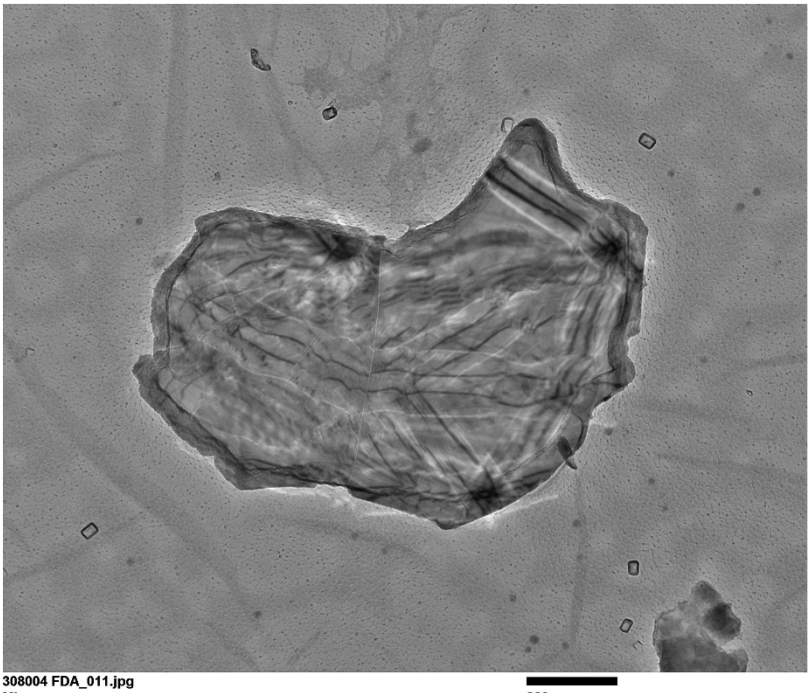


Diffraction Pattern from the Talc Particle pictured above

Chemistry from the Talc Particle pictured above



Mica Particle from 308004-1



308004 FDA_011.jpg
Mica
Cal: 0.002858 µm/pix
15:51 7/29/2019
TEM Mode: Imaging
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

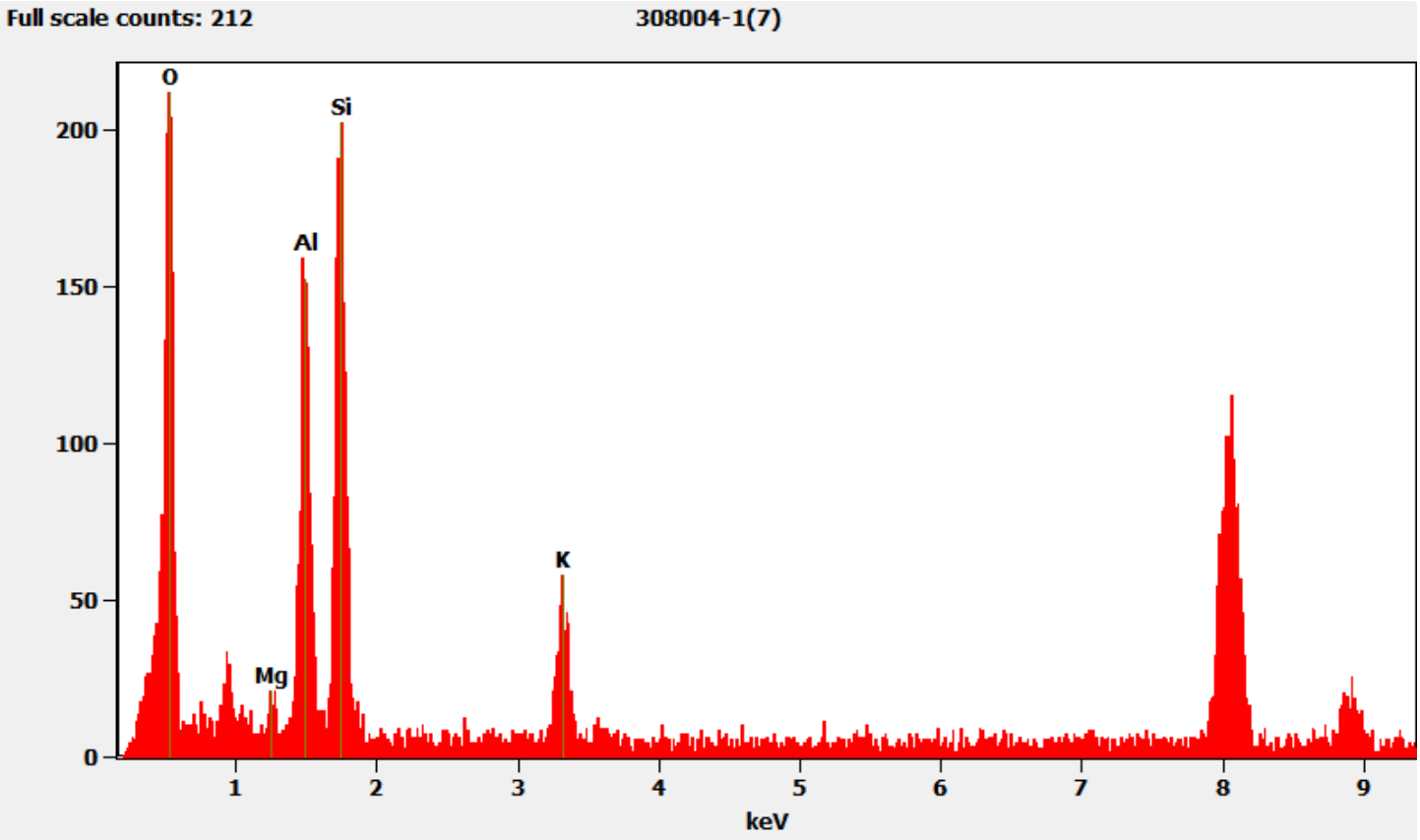
800 nm
HV=100kV
Direct Mag: 3600 x
AMA Analytical Services, Inc

Diffraction Pattern from the Mica Particle pictured above

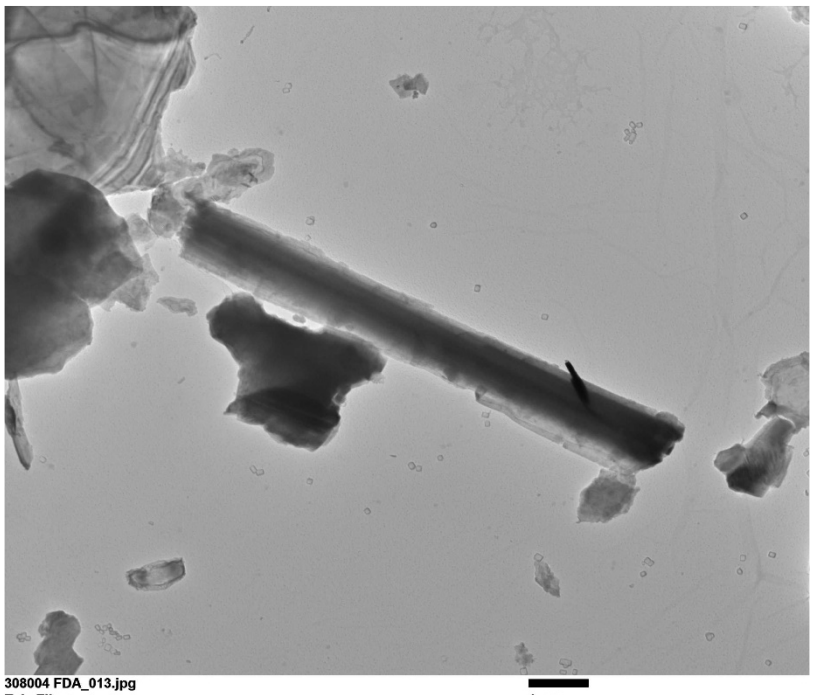
308004 FDA_012.jpg
Mica
15:52 7/29/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from the Mica Particle pictured above

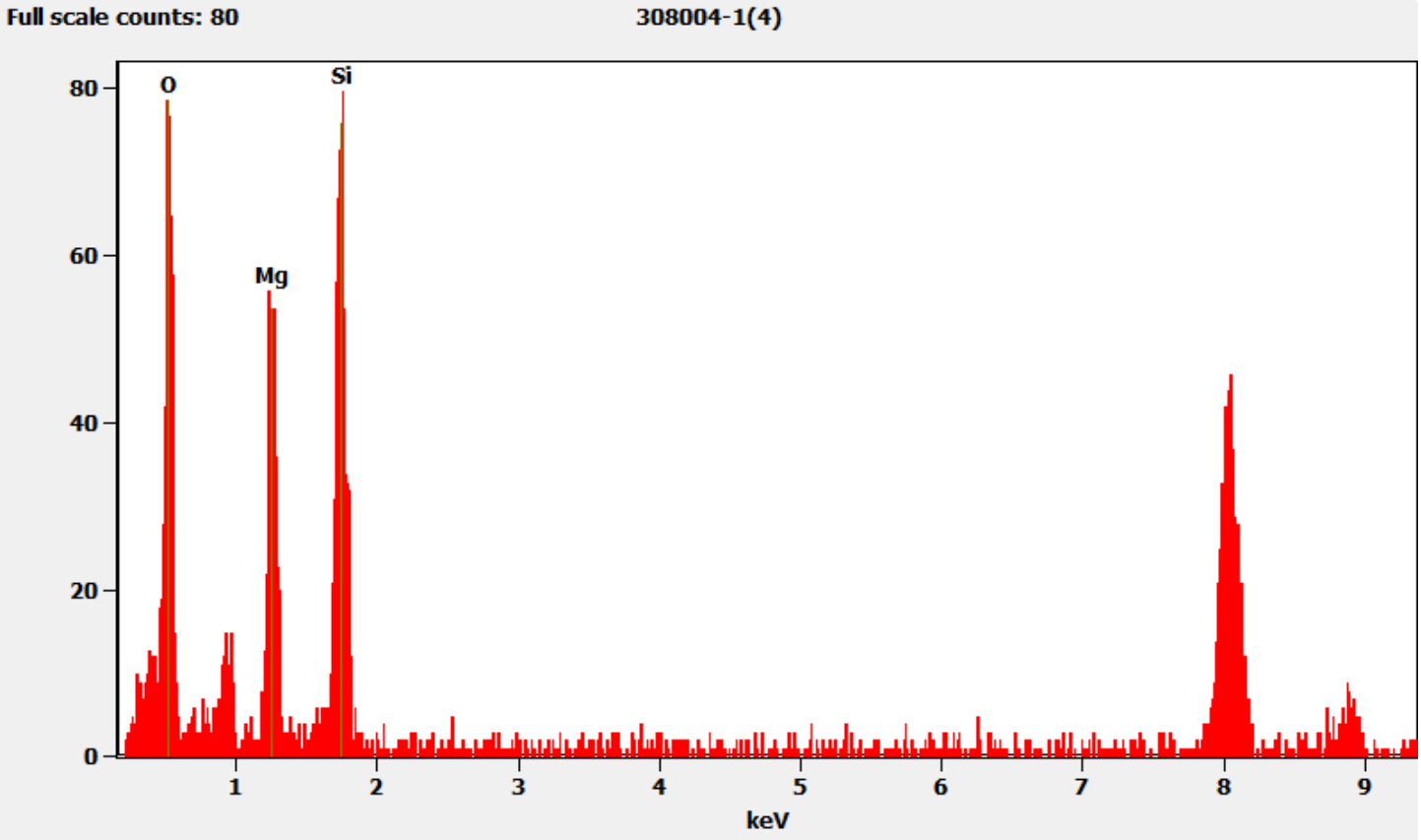


Talc Fiber from 308004-1

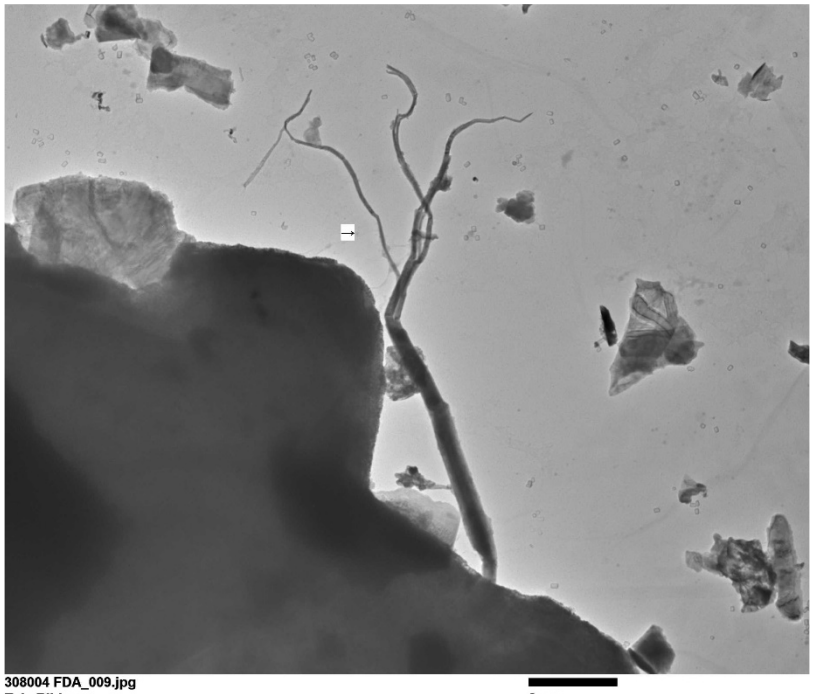


Diffraction Pattern from the Talc Fiber pictured above

Chemistry from the Talc Fiber pictured above



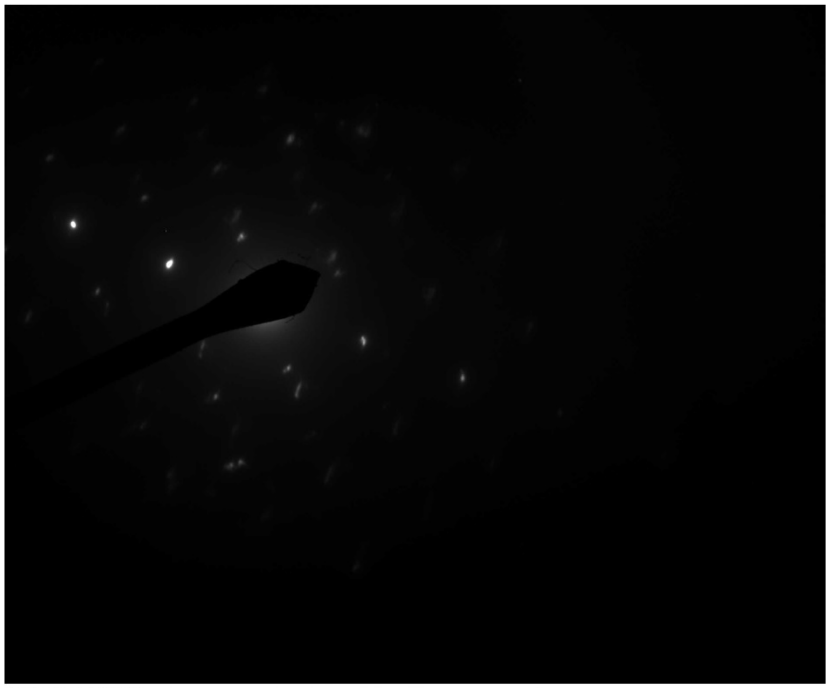
Talc Ribbon from 308004-1



308004 FDA_009.jpg
Talc Ribbon
Cal: 0.007349 µm/pix
15:43 7/29/2019
TEM Mode: Imaging
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

2 µm
HV=100kV
Direct Mag: 1400 x
AMA Analytical Services, Inc

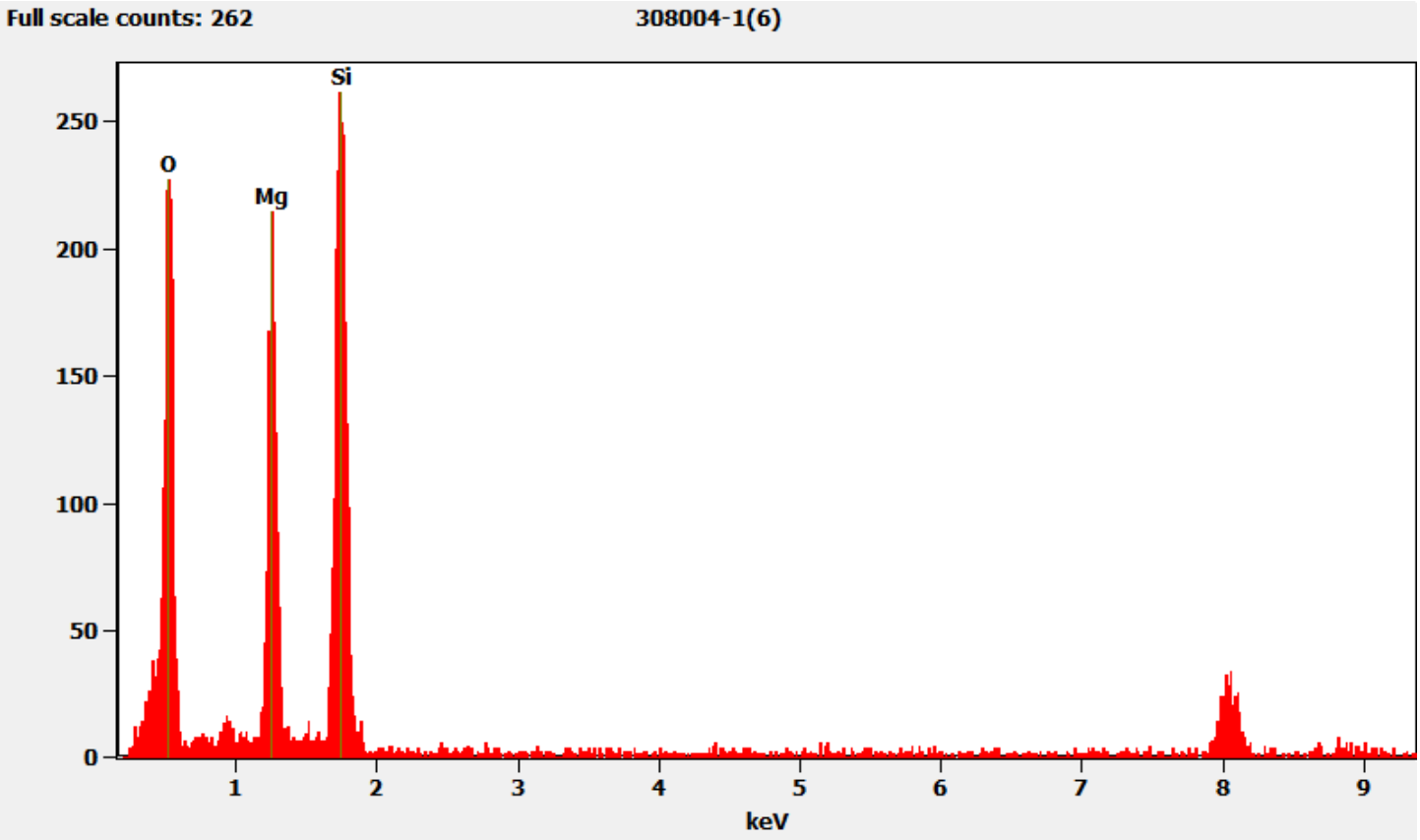
Diffraction Pattern from the Talc Ribbon pictured above



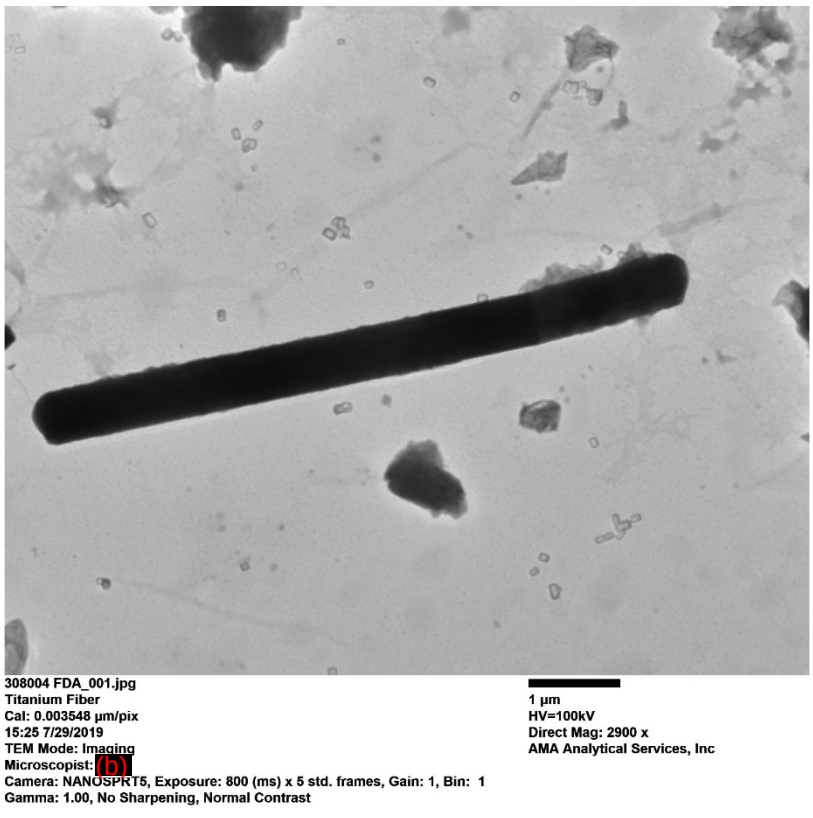
308004 FDA_010.jpg
Talc Ribbon
15:45 7/29/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

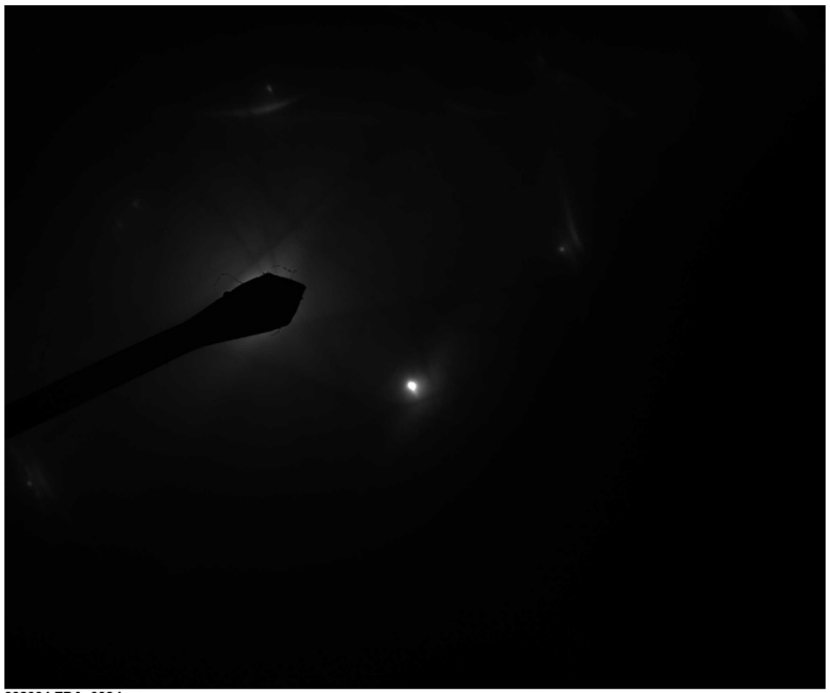
Chemistry from the Talc Ribbon pictured above



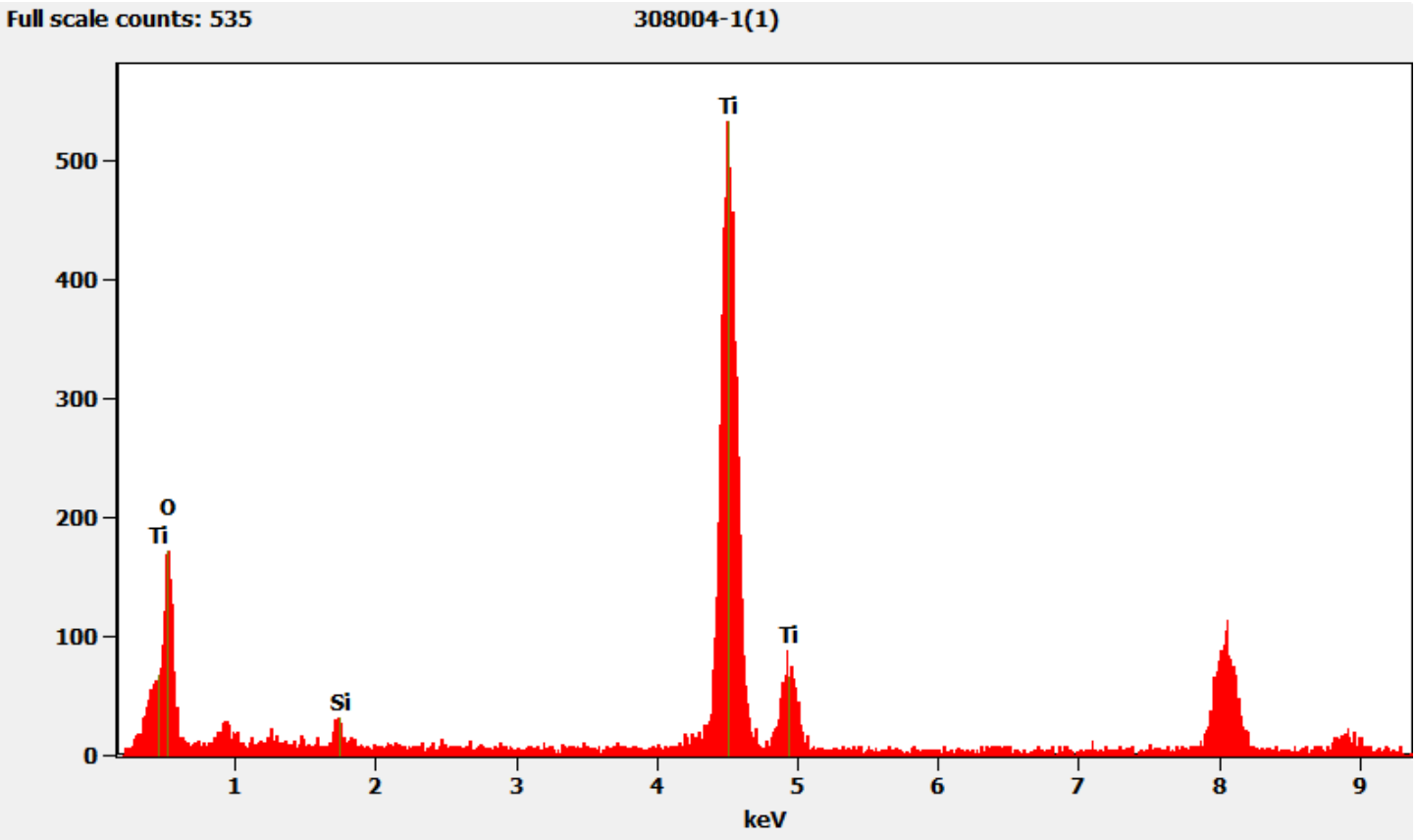
Titanium Fiber from 308004-1



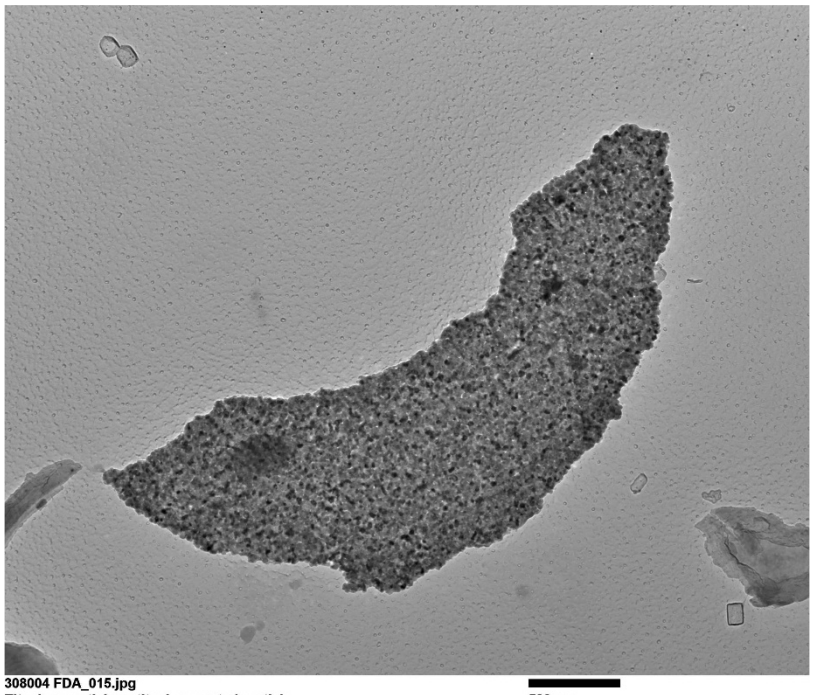
Diffraction Pattern from the Titanium Fiber pictured above



Chemistry from the Titanium Fiber pictured above



Particle coated with Titanium from 308004-1



308004 FDA_015.jpg
Titanium particle or titanium coated particle
Cal: 0.001774 µm/pix
16:25 7/29/2019
TEM Mode: Imaging
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

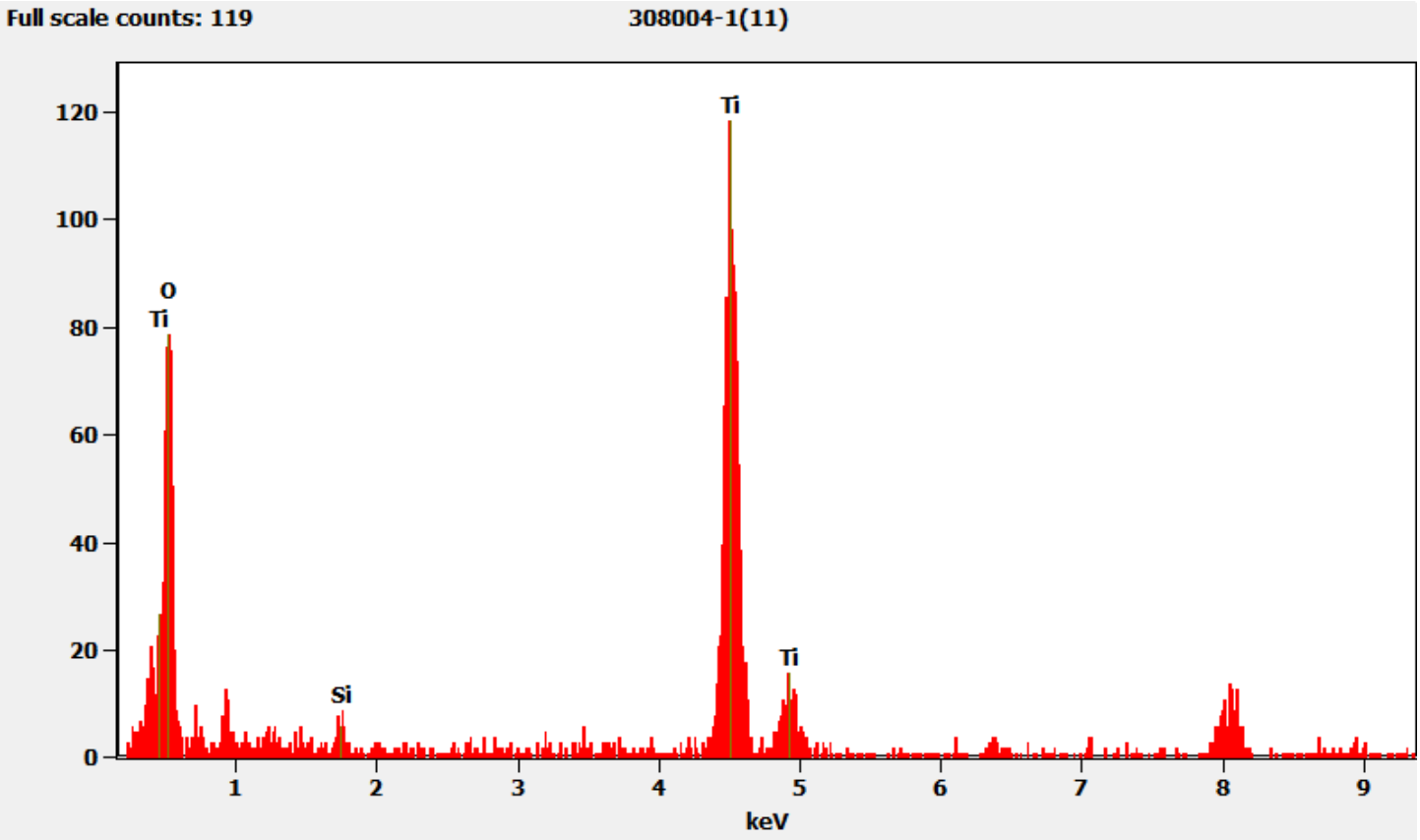
500 nm
HV=100kV
Direct Mag: 5800 x
AMA Analytical Services, Inc

Diffraction Pattern from the Titanium Coated particle pictured above

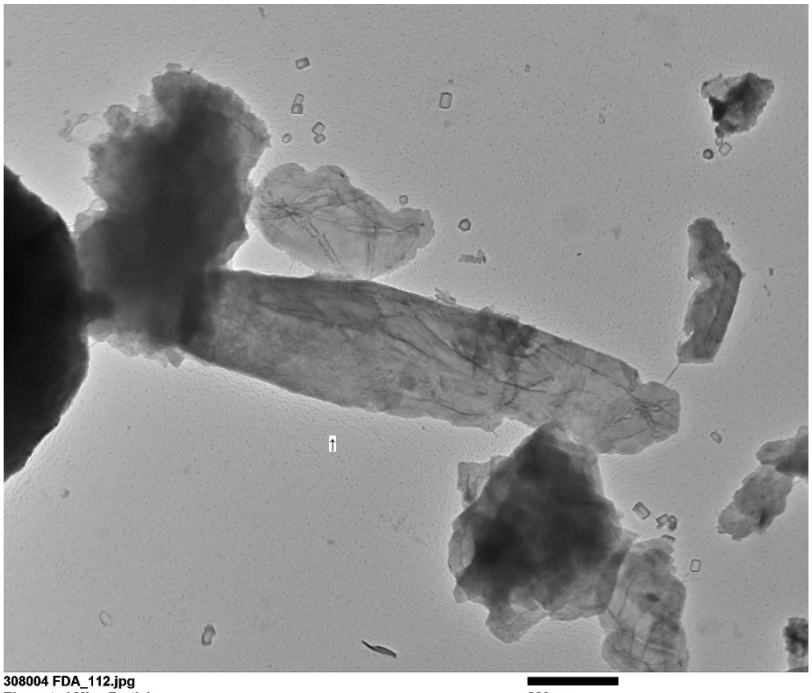
308004 FDA_016.jpg
Titanium particle or titanium coated particle
16:26 7/29/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from the Titanium Coated Particle pictured above

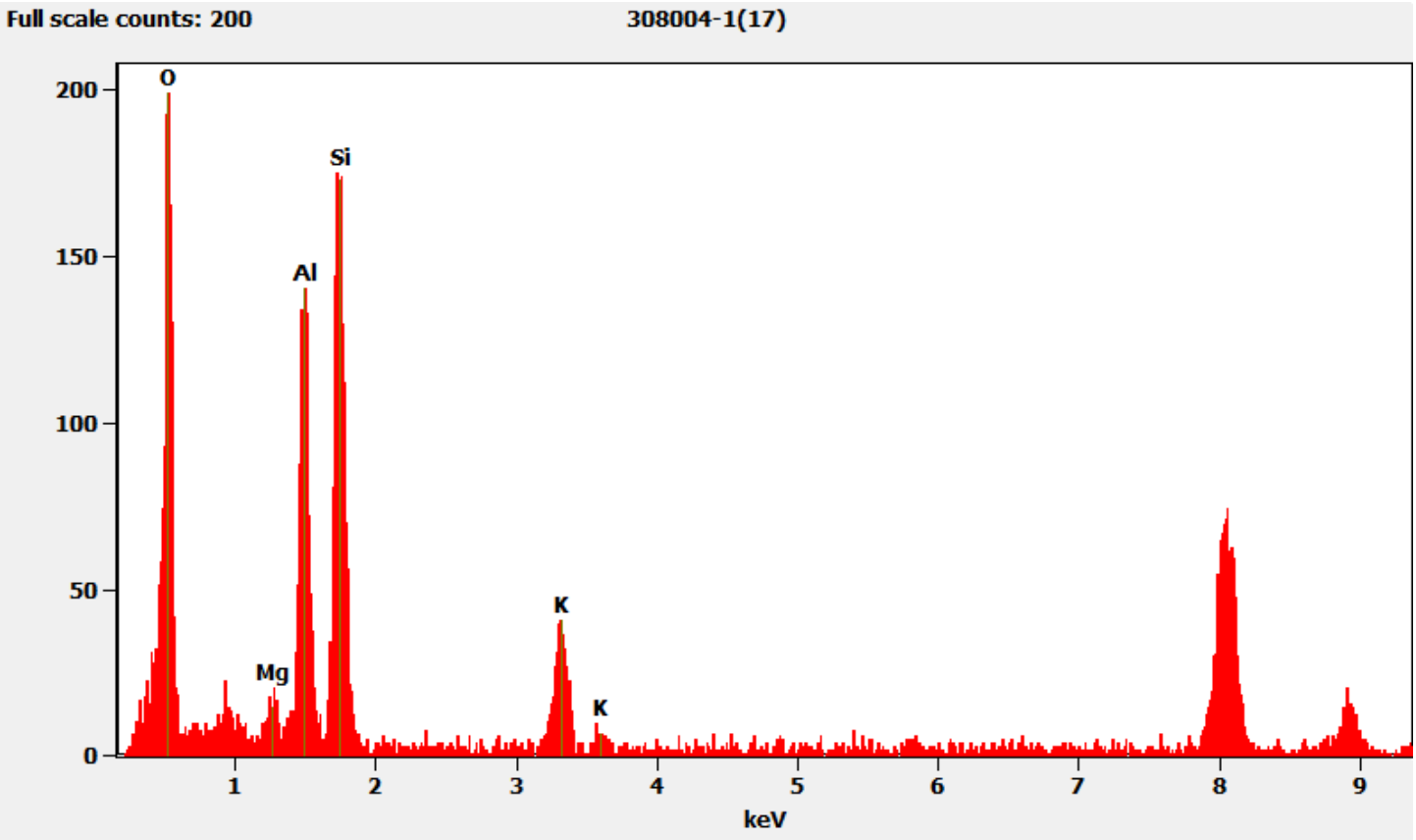


Elongated Mica Particle from 308004-1

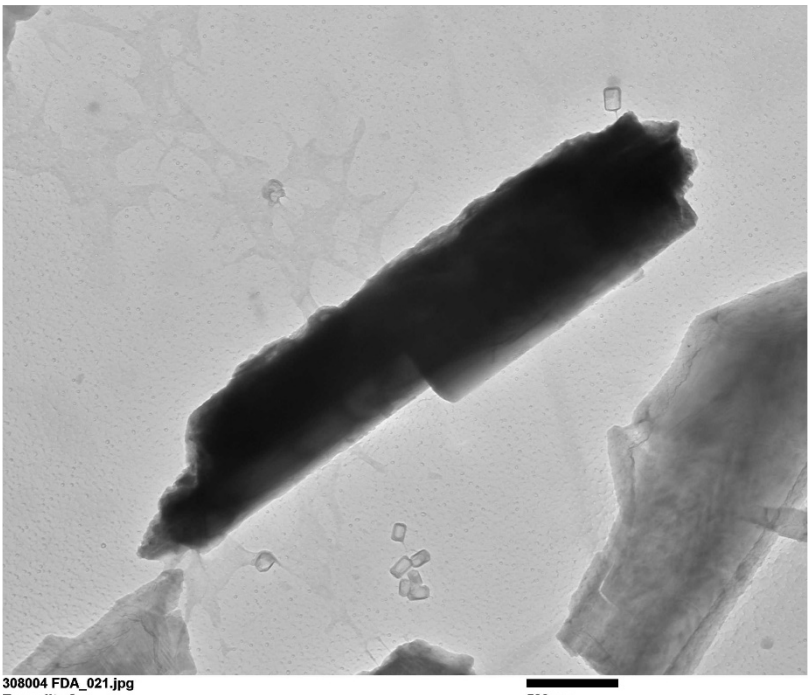


Diffraction Pattern from the Elongated Mica Particle pictured above

Chemistry from the Elongated Mica Particle pictured above



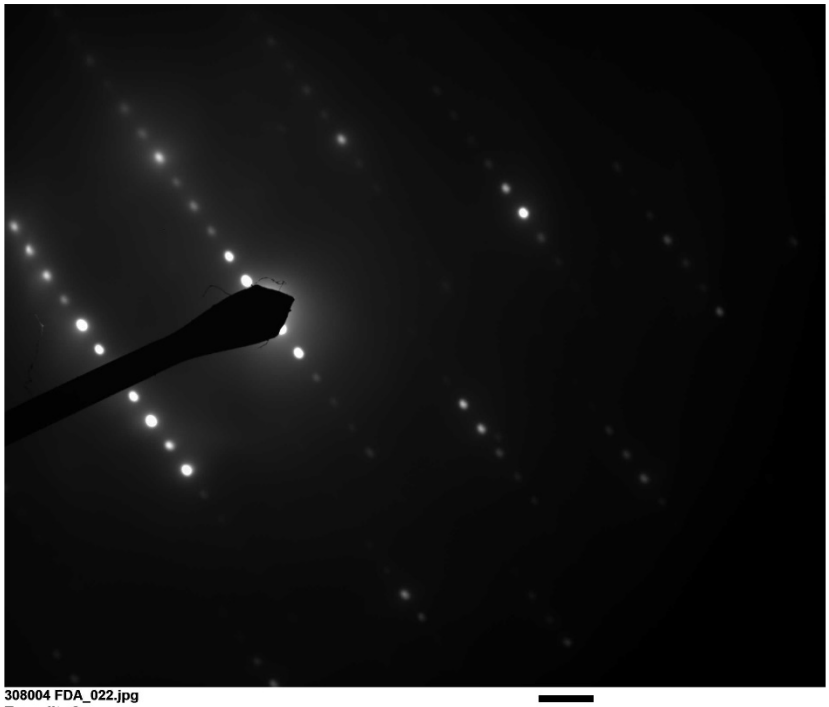
Tremolite Particle from 308004-1



308004 FDA_021.jpg
Tremolite 2
Cal: 0.001774 µm/pix
16:46 7/29/2019
TEM Mode: Imaging
Microscopist: (b)
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

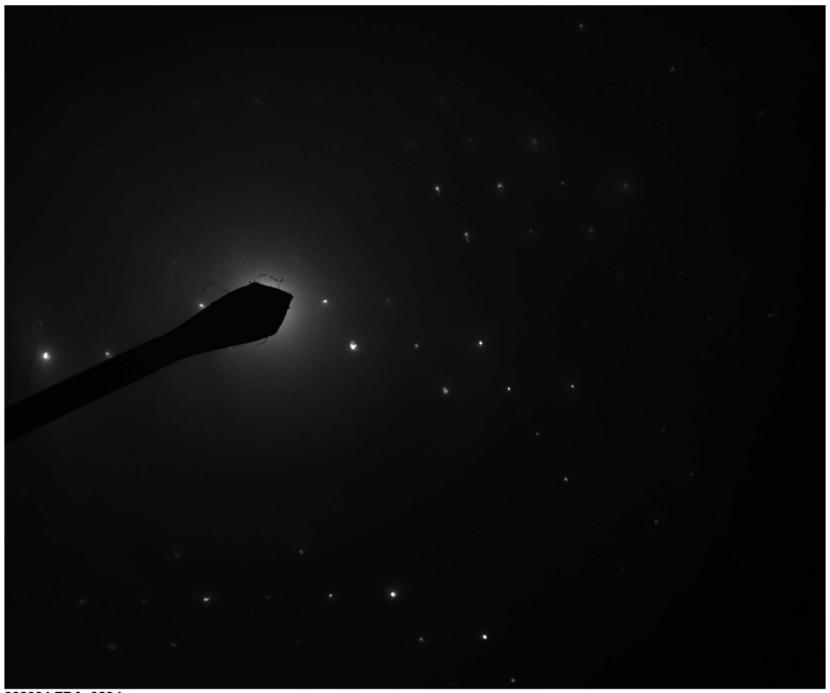
Diffraction Pattern from the Tremolite Particle pictured above



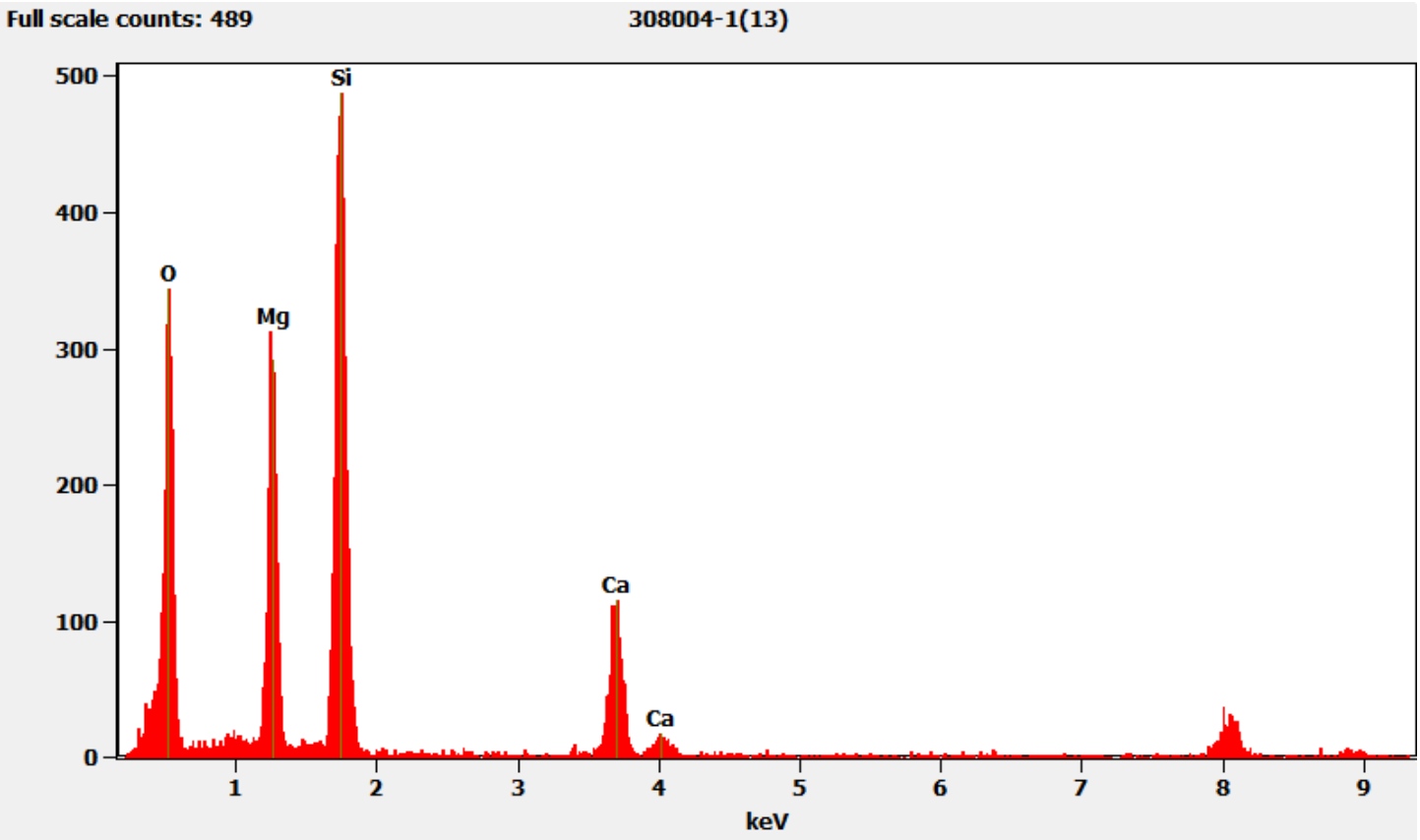
308004 FDA_022.jpg
Tremolite 2
16:51 7/29/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

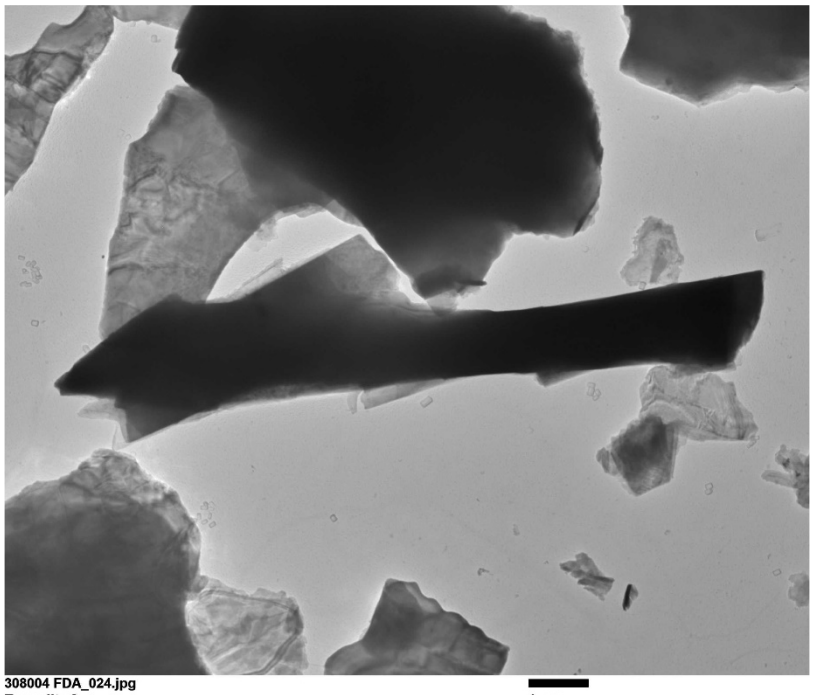
Diffraction Pattern from the Tremolite Particle pictured above



Chemistry from the Tremolite Particle pictured above



Tremolite Particle from 308004-1



308004 FDA_024.jpg
Tremolite 3
Cal: 0.005415 µm/pix
17:07 7/29/2019
TEM Mode: Imaging
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

1 µm
HV=100kV
Direct Mag: 1900 x
AMA Analytical Services, Inc

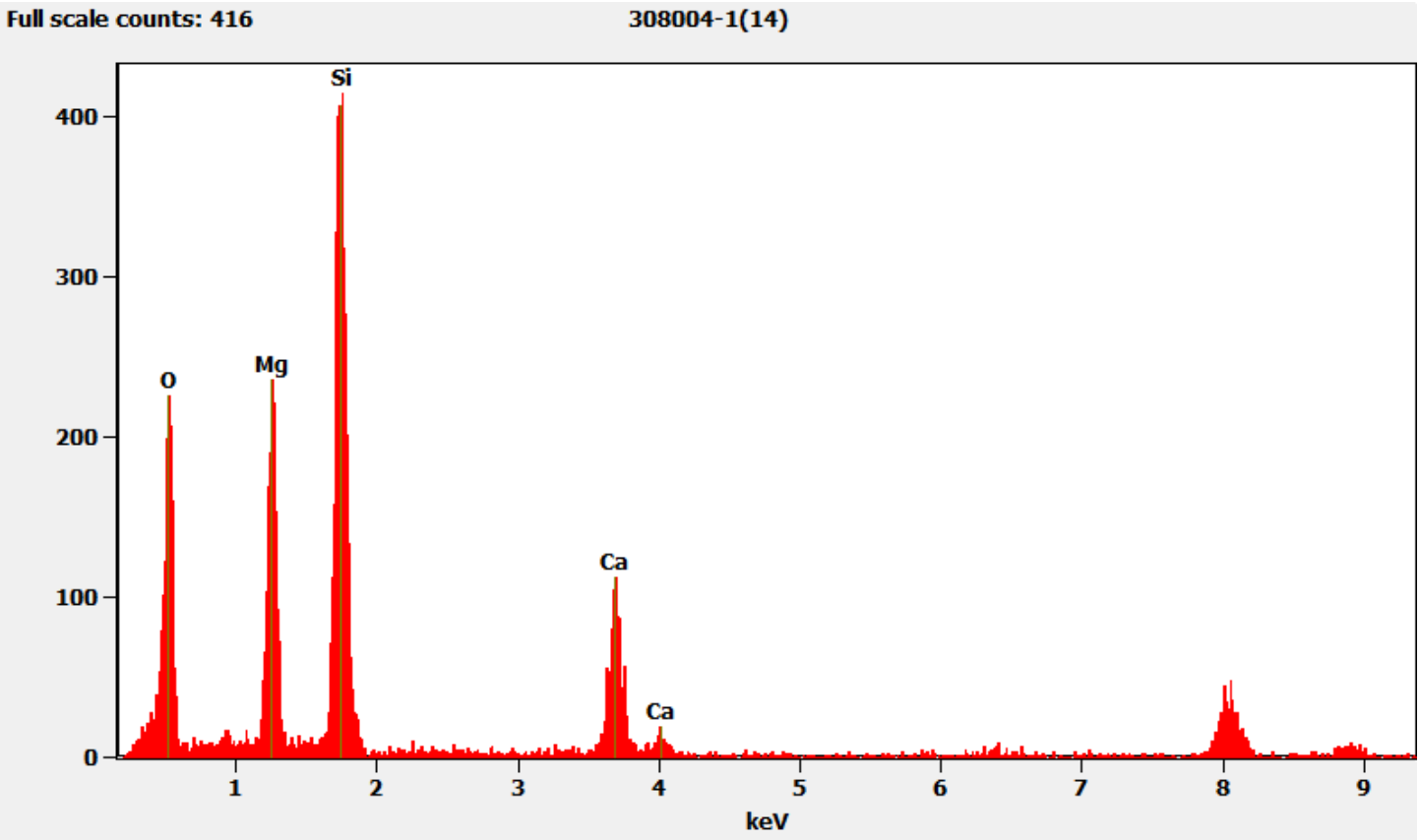
Diffraction Pattern from the Tremolite Particle pictured above

308004 FDA_025.jpg
Tremolite 3
17:08 7/29/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

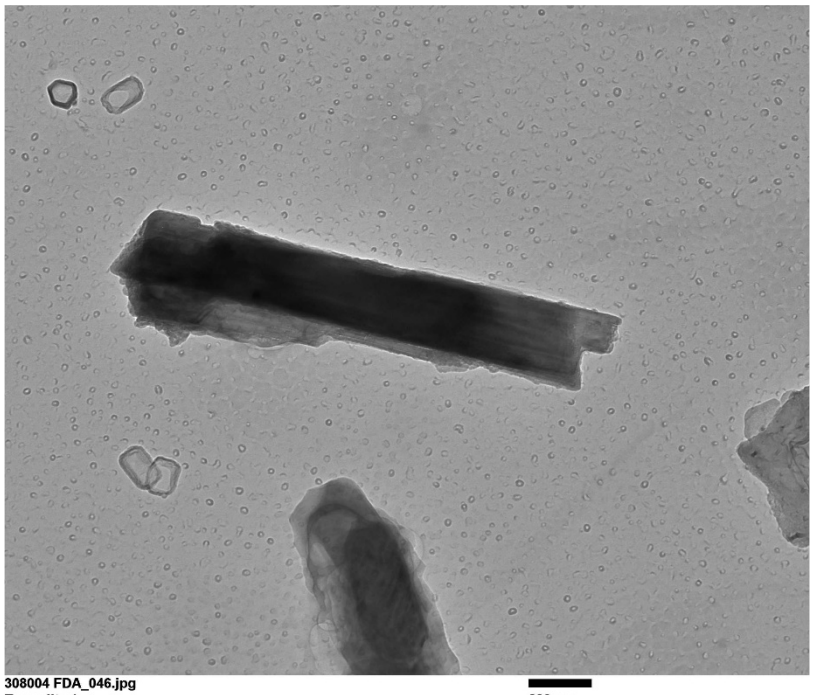
100 (1/Å)
HV=100kV
Cam Len: 0.2200 m
AMA Analytical Services, Inc

Diffraction Pattern from the Tremolite Particle pictured above

Chemistry from the Tremolite Particle pictured above



Tremolite Particle from 308004-1A



308004 FDA_046.jpg
Tremolite 1
Cal: 0.001029 µm/pix
18:11 7/30/2019
TEM Mode: Imaging
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

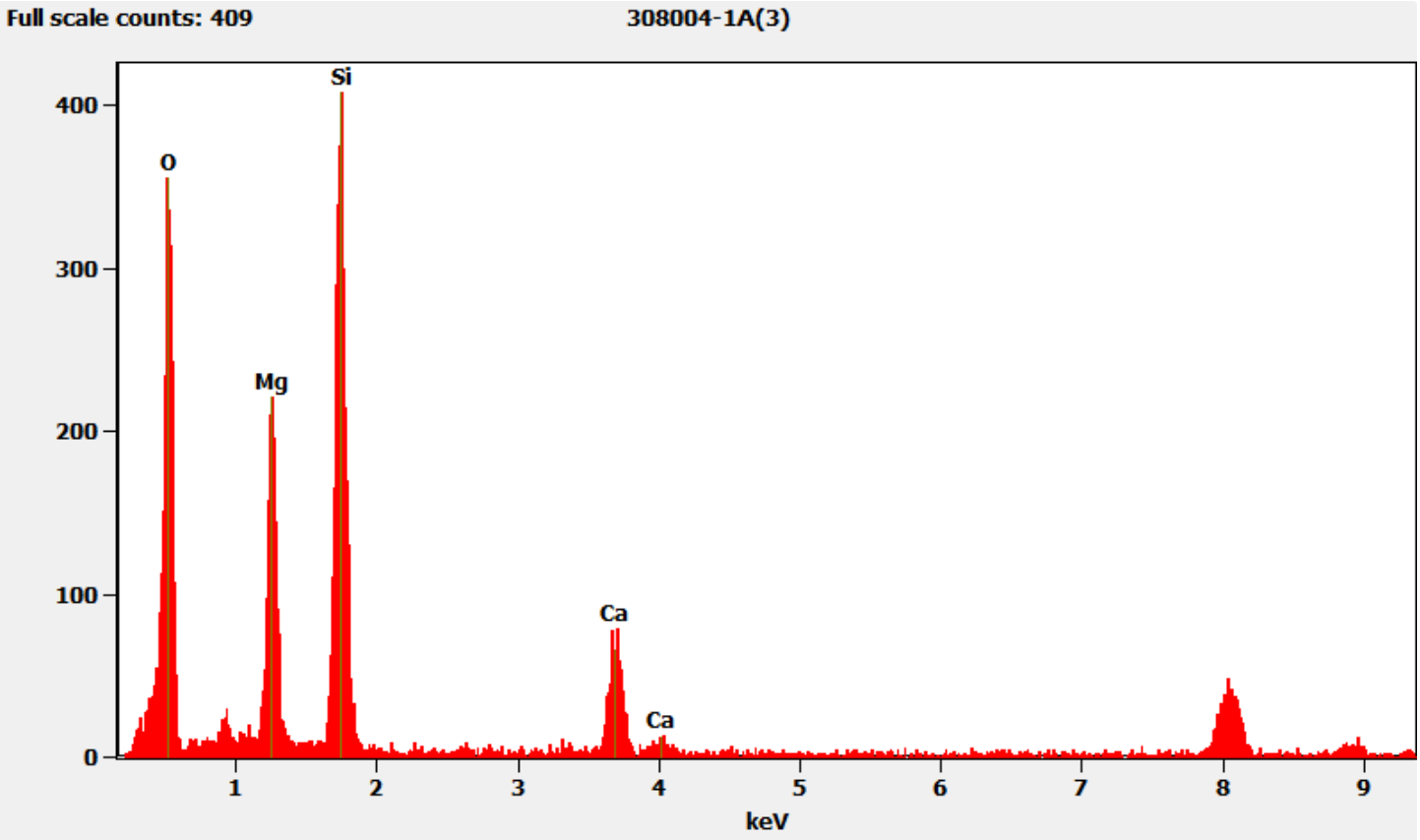
200 nm
HV=100kV
Direct Mag: 10000 x
AMA Analytical Services, Inc

Diffraction Pattern from the Tremolite Particle pictured above

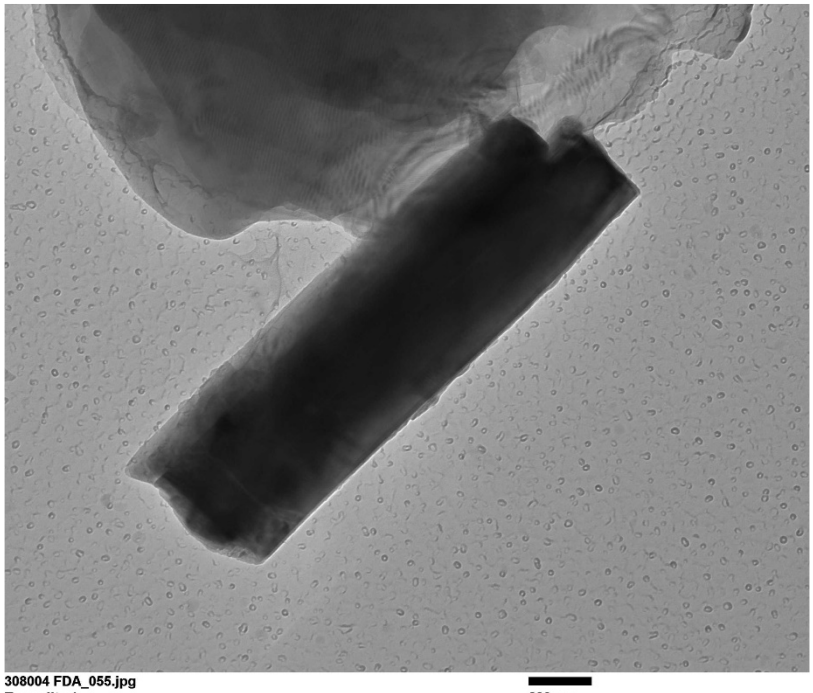
308004 FDA_047.jpg
Tremolite 1
18:14 7/30/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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



Diffraction Pattern from the Tremolite Particle pictured above

308004 FDA_056.jpg
Tremolite 1
14:31 8/1/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

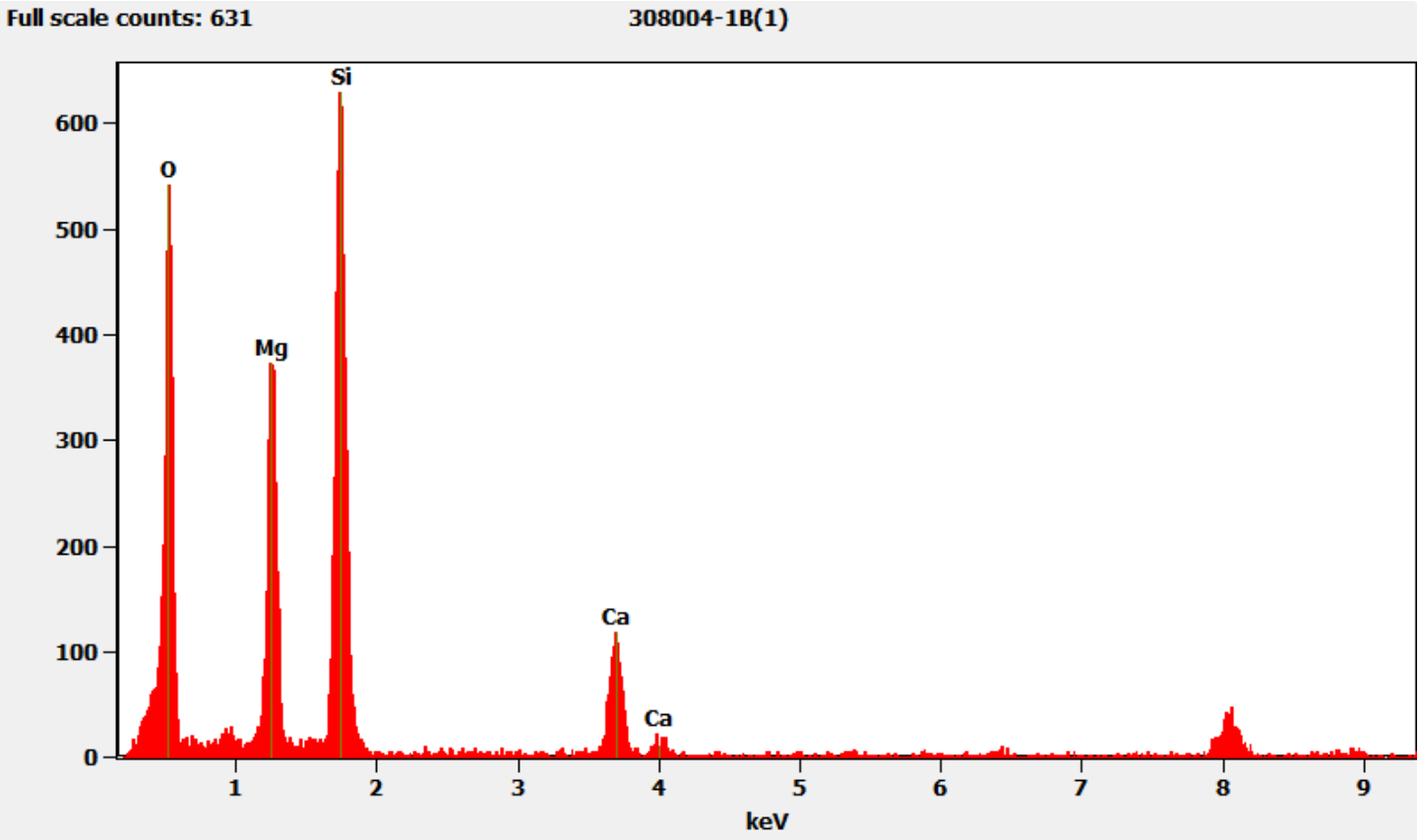
100 (1/Å)
HV=100kV
Cam Len: 0.2200 m
AMA Analytical Services, Inc

Diffraction Pattern from the Tremolite Particle pictured above

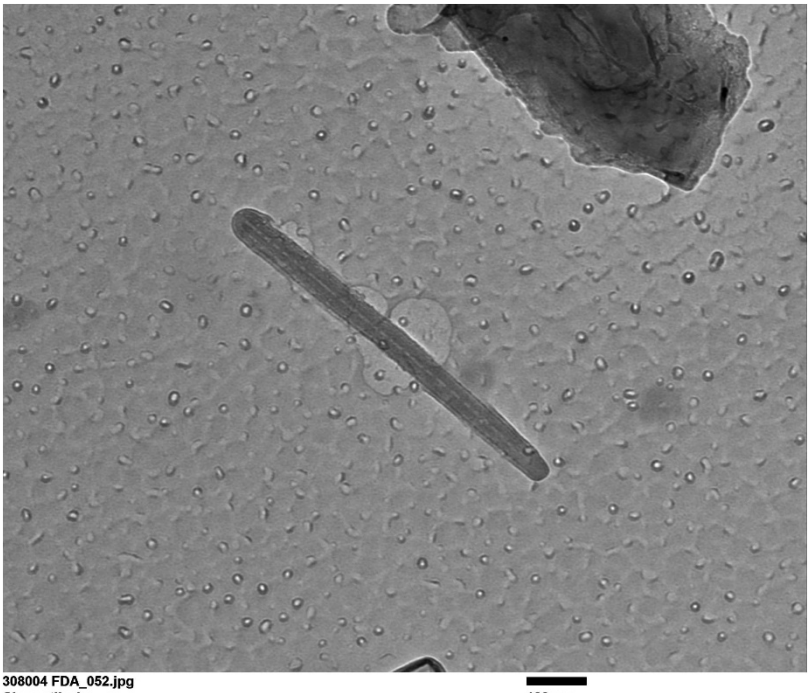
308004 FDA_057.jpg
Tremolite 1
14:32 8/1/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from the Tremolite Particle pictured above



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: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

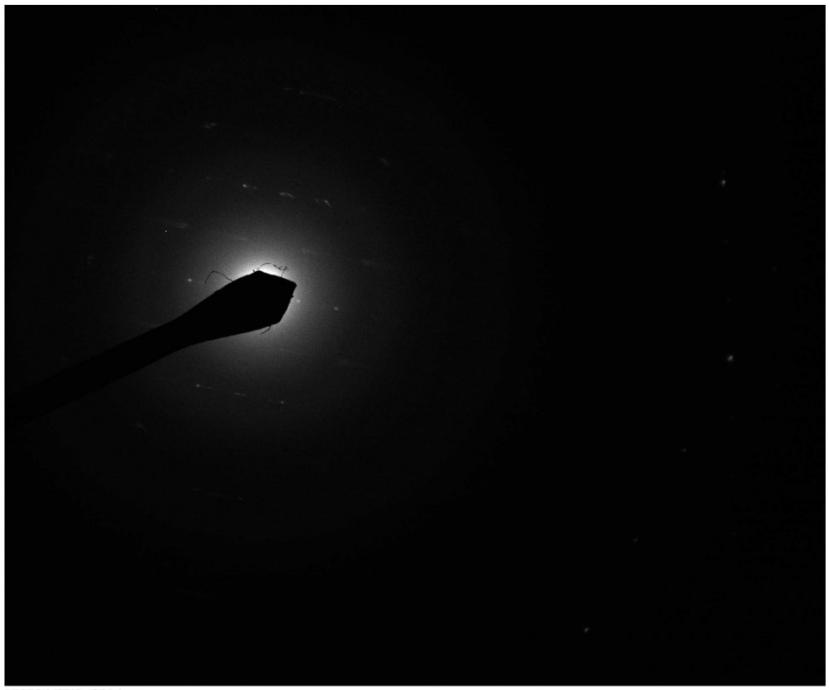
100 nm
HV=100kV
Direct Mag: 19000 x
AMA Analytical Services, Inc

Diffraction Pattern from the Chrysotile Fiber pictured above

308004 FDA_050.jpg
Chrysotile 4
18:33 7/30/2019
TEM Mode: Diffraction
Microscopist: (b)
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

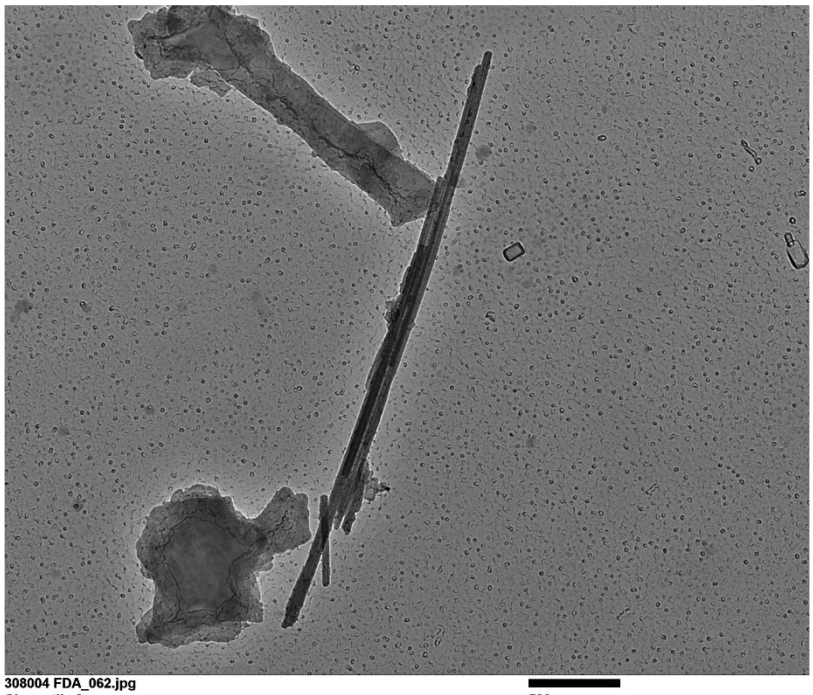
Diffraction Pattern from the Chrysotile Fiber pictured above



308004 FDA_051.jpg
Chrysotile 4
18:34 7/30/2019
TEM Mode: Diffraction
Microscopist: (b)
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

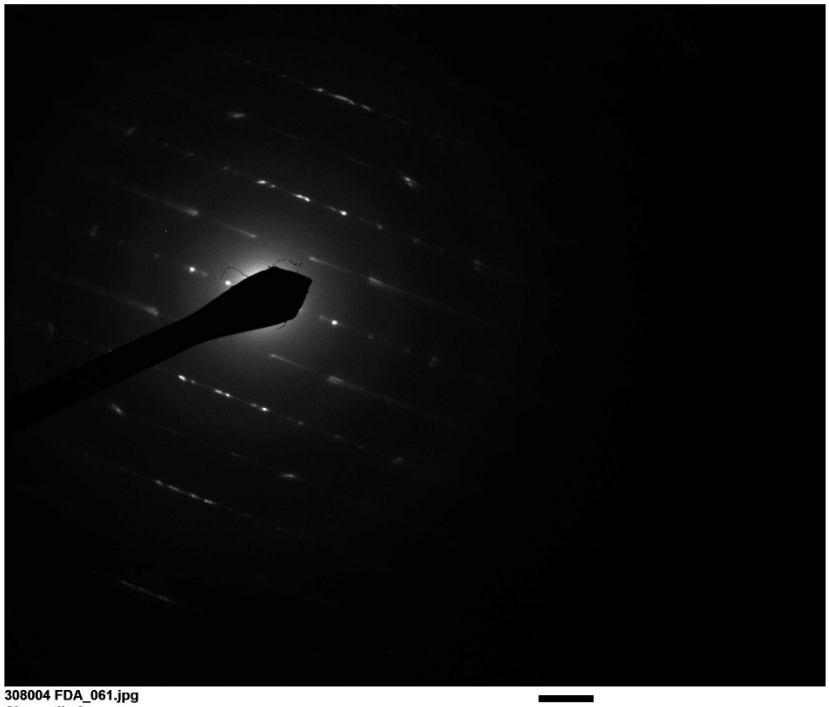
Chrysotile Structure from 308004-1B



308004 FDA_062.jpg
Chrysotile 3
Cal: 0.001774 µm/pix
14:54 8/1/2019
TEM Mode: Imaging
Microscopist: (b)
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

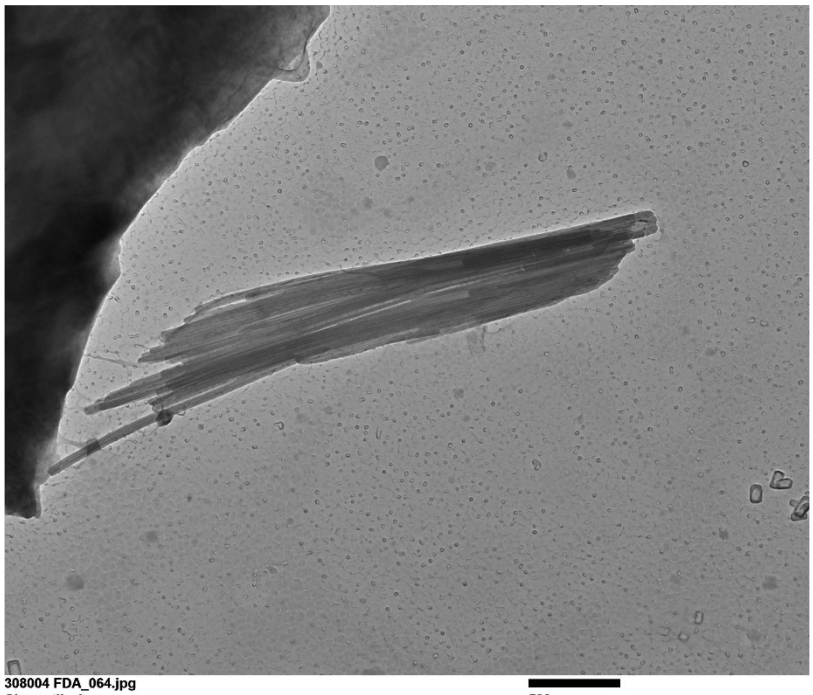
Diffraction Pattern from the Chrysotile Structure pictured above



308004 FDA_061.jpg
Chrysotile 3
14:53 8/1/2019
TEM Mode: Diffraction
Microscopist: (b)
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

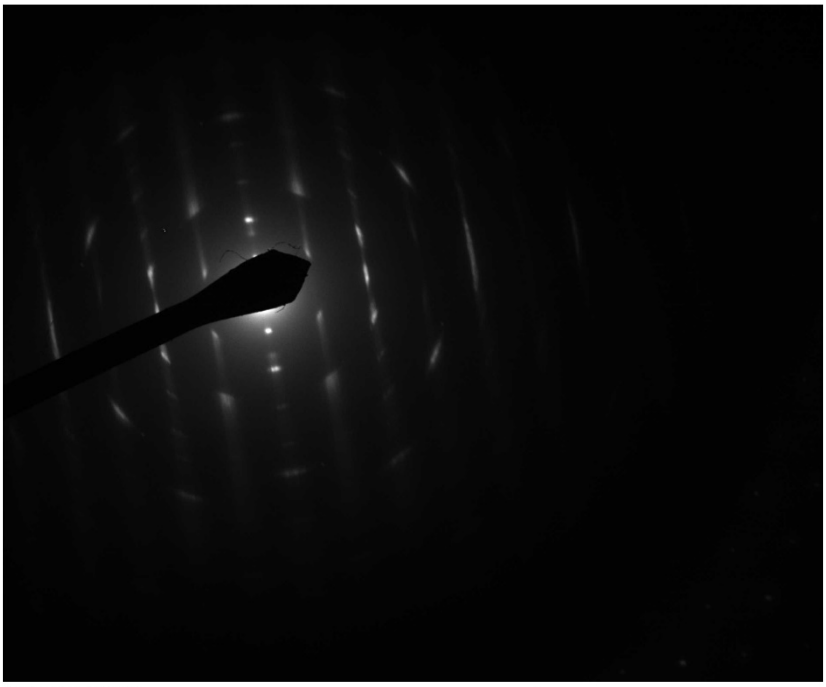
Chrysotile Structure from 308004-1B



308004 FDA_064.jpg
Chrysotile 4
Cal: 0.001774 µm/pix
14:59 8/1/2019
TEM Mode: Imaging
Microscopist: (b)
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: (b)
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

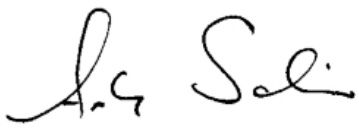
QC Discussion:
During preparation, one blank control sample and one reference control sample were prepared. These samples were prepared alongside the customer samples. The blank sample was prepared using Sigma-Aldrich Talc Powder, <10 micron. No asbestos was detected on the blank sample. The reference 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 (b) (6) for samples analyzed between 1/1/2019 & 8/8/2019
7) Replicate and Duplicate QC Charts for (b) (6) 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

AMA Analytical Services, Inc.
Focused On Results.

CERTIFICATE OF ANALYSIS

Chain of Custody: 308004
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: 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 - 8/8/2019
Report Date: 8/15/2019
Date Sampled: Not Provided
Person Submitting: Steve Wolfgang
Revised: 8/30/2019 2nd 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
308004-2	D-50	0.00000133%	0.00000533%	0.02722%	0.00003%	0.02725%	ND	14.5%	15.2%	70.2%	
308004-2A	D-50	0.00000112%	0.00000447%	0.00012%	0.00003%	0.00015%	ND	14.5%	14.0%	71.5%	
308004-2B	D-50	0.00000091%	0.00000363%	0.00351%	0.00016%	0.00367%	ND	14.6%	14.5%	70.9%	

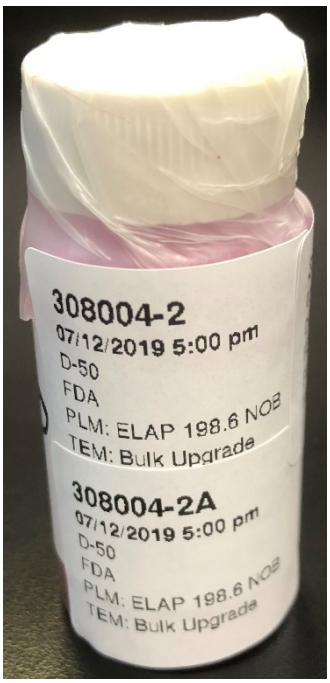
LOD = Limit of Detection **LOQ** = Limit of Quantification **ND** = Not Detected **PLM** = Polarized Light Microscopy **TEM** = Transmission Electron Microscopy

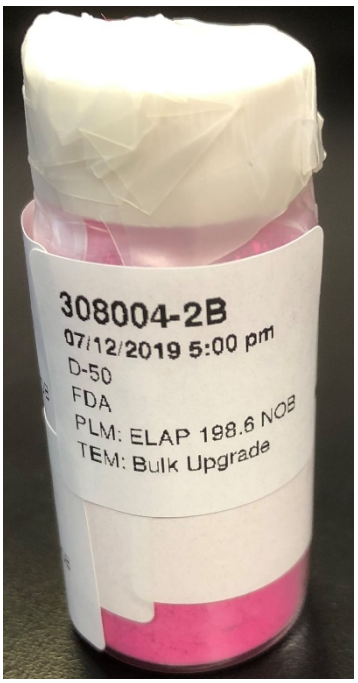
Analytical Method(s): PLM by Modified NY ELAP 198.6
TEM by Modified NY ELAP 198.4/ASTM D5756

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





Sample Preparation

Samples were prepared for PLM and TEM bulk analysis by (b) (6) 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 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.

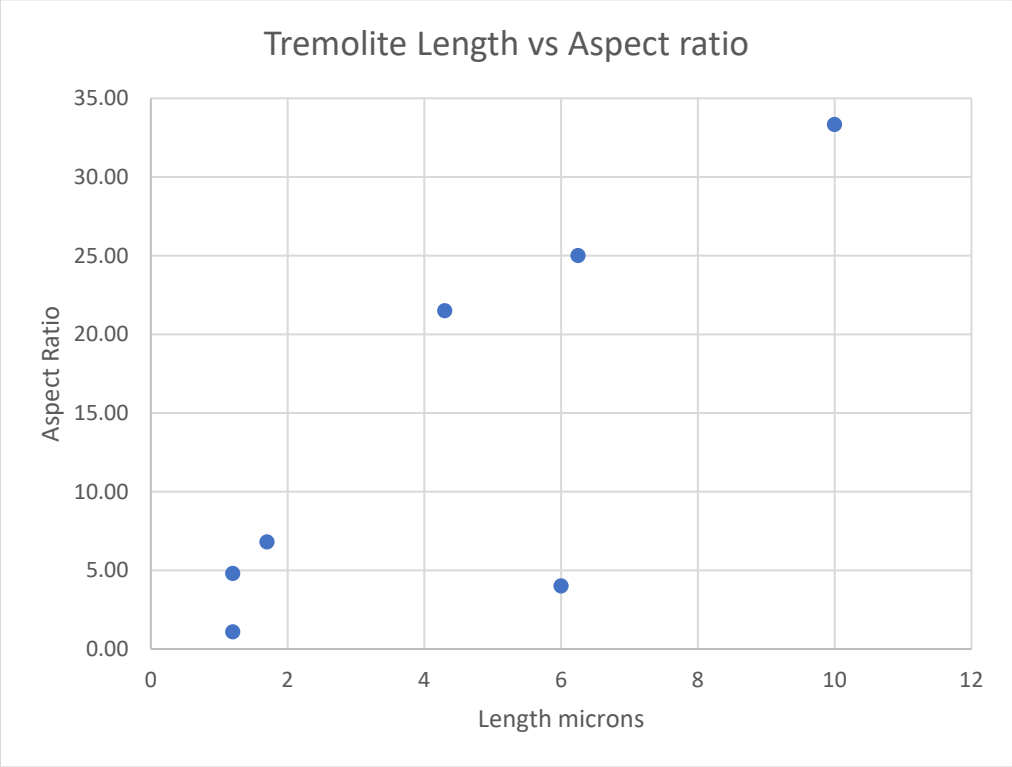
Discussion and Interpretation of Analytical Findings:
PLM
All three aliquots of sample D-50 were analyzed by (b) (6) on July 25, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

308004-2	NAD
308004-2A	NAD
308004-2B	NAD

TEM
(b) (6) analyzed sample 2 on July 29, 2019 and August 8, 2019, 2A on August 7, 2019 and 2B on August 8, 2019. The sample consisted of a mix of talc and mica particles, with a few talc fibers/ribbons, a few titanium fibers/particles and a few silica fibers/particles. Chrysotile and tremolite were observed on all three aliquots. The results were calculated using the equations detailed in the calculations section.

308004-2	0.02725%
308004-2A	0.00015%
308004-2B	0.00367%

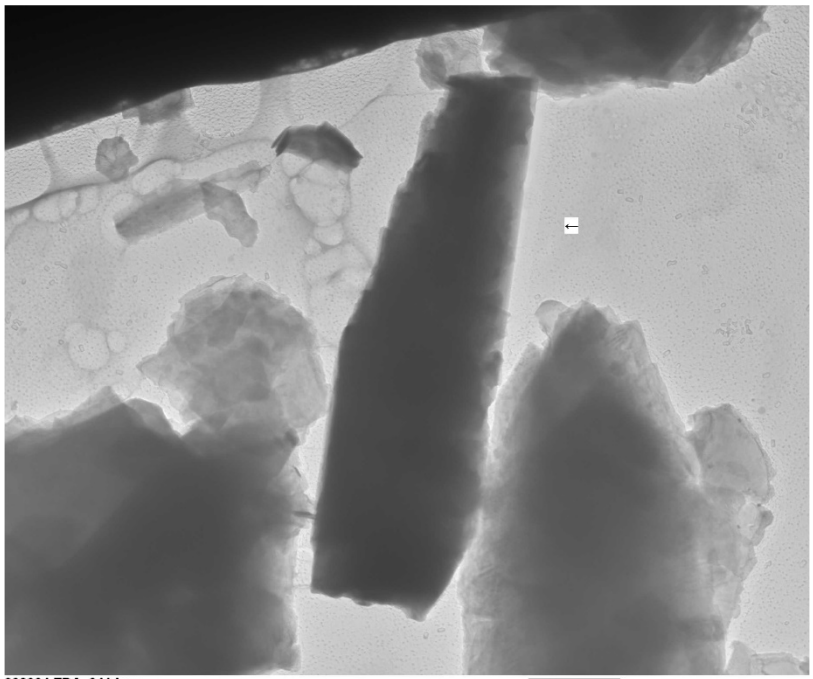
The following charts plot aspect ratio vs. length, aspect ratio vs. width, and length vs. width for all the tremolite particles counted over all three aliquots.


Tremolite Length vs Aspect ratio
Aspect Ratio
35.00
30.00
25.00
20.00
15.00
10.00
5.00
0.00
0
2
4
6
8
10
12
Length microns


Tremolite Width vs Aspect ratio
Aspect Ratio
35.00
30.00
25.00
20.00
15.00
10.00
5.00
0.00
0
0.2
0.4
0.6
0.8
1
1.2
1.4
1.6
Width microns

Below are representative 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.

Tremolite Particle from 308004-2



Diffraction Pattern from Tremolite Particle pictured above

308004 FDA_039.jpg
Tremolite 1
17:57 7/29/2019
TEM Mode: Diffraction
Microscopist:
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

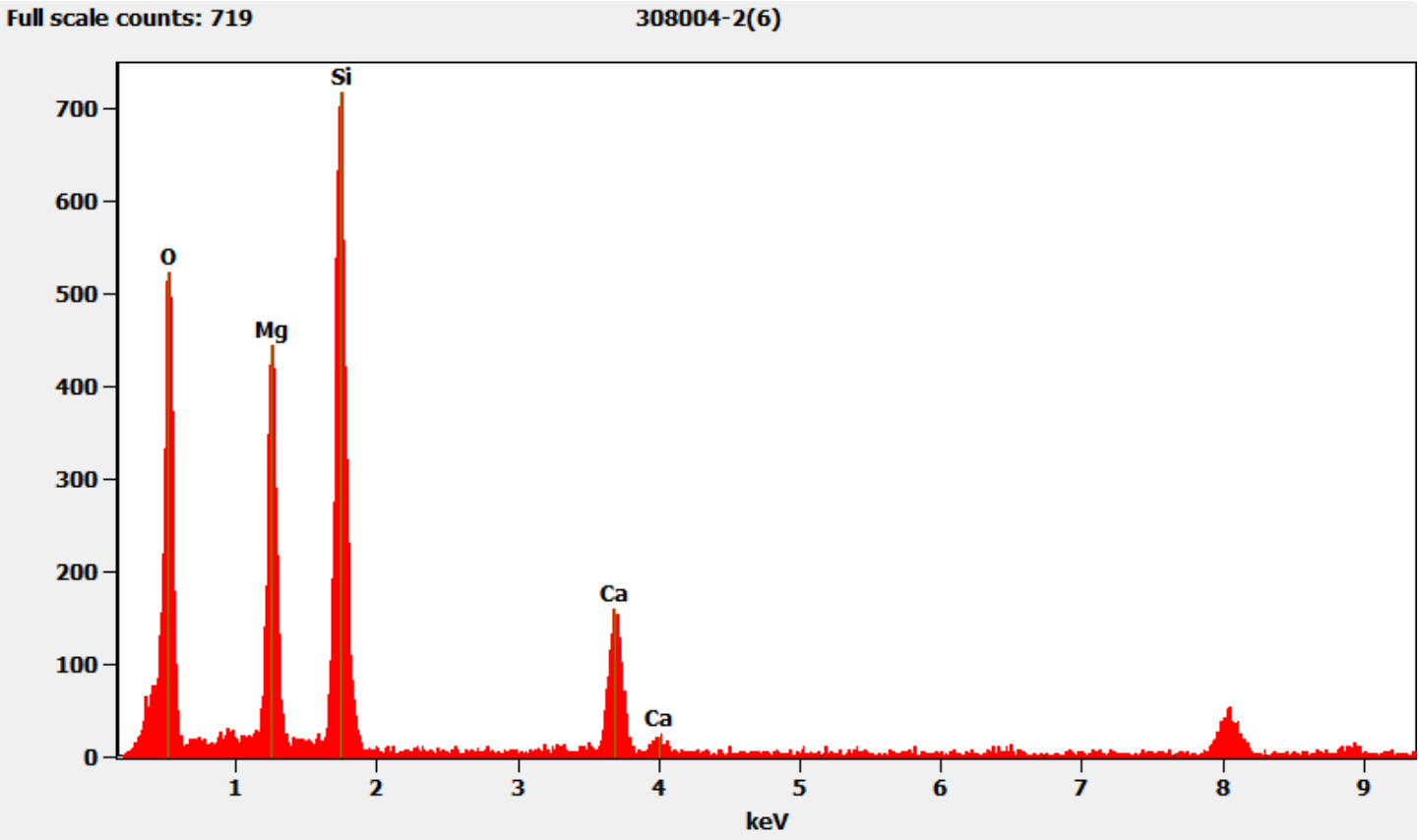
100 (1/Å)
HV=100kV
Cam Len: 0.2200 m
AMA Analytical Services, Inc

Zone-Axis Diffraction Pattern from the Tremolite Particle pictured above

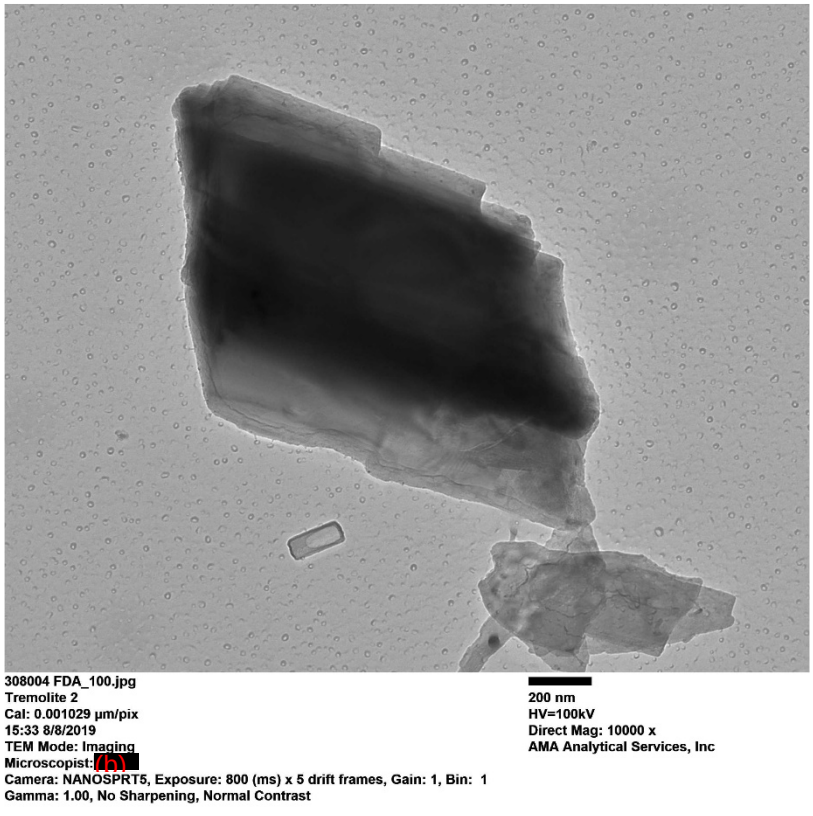
308004 FDA_040.jpg
Tremolite 2 Zone Axis
[-5 1 0]
18:00 7/29/2019
TEM Mode: Diffraction
Microscopist:
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from the Tremolite Particle pictured above



Tremolite Particle from 308004-2B

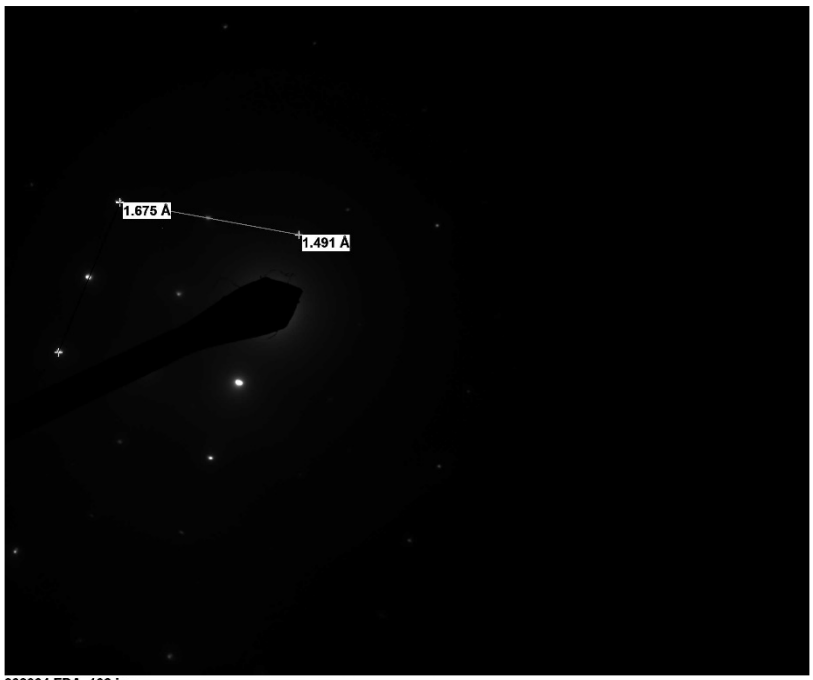


Diffraction Pattern from the Tremolite Particle pictured above

308004 FDA_101.jpg
Tremolite 2
15:36 8/8/2019
TEM Mode: Diffraction
Microscopist: (b)
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

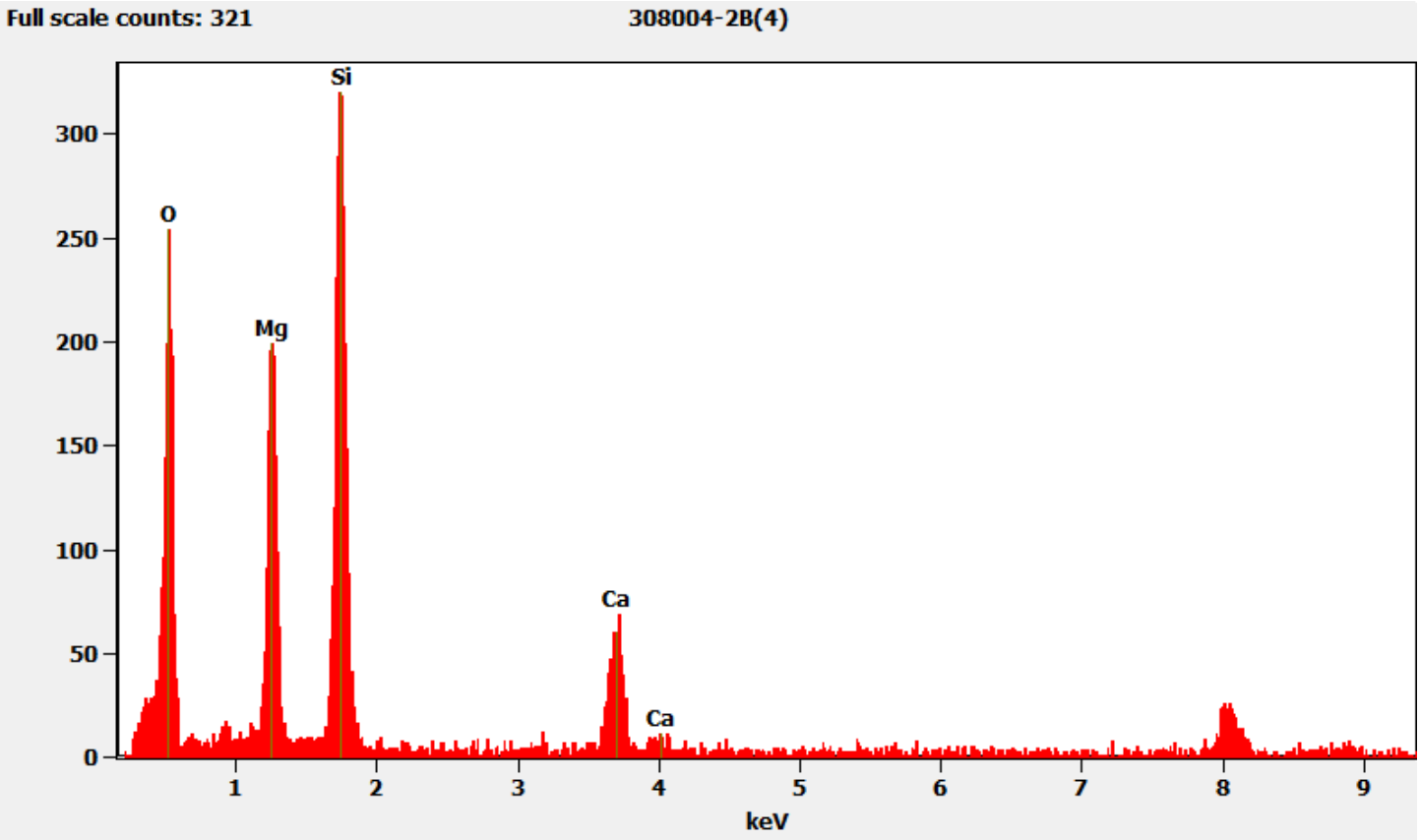
Diffraction Pattern from Tremolite Particle pictured above



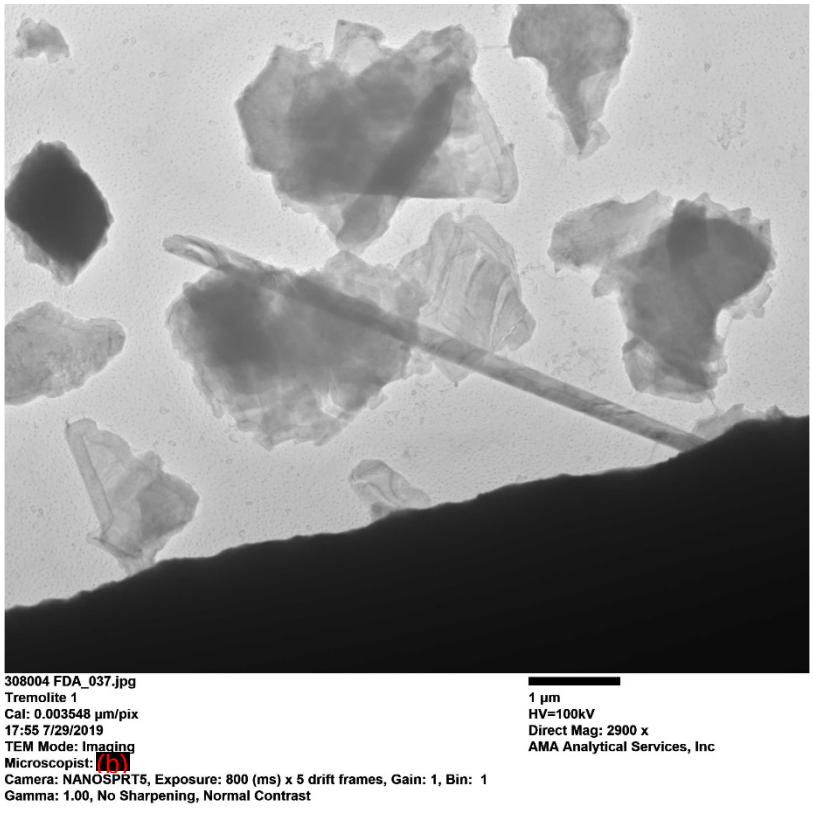
308004 FDA_102.jpg
Tremolite 2
[-8 2 -8]
15:37 8/8/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from Tremolite Particle pictured above



Tremolite Particle from 308004-2



Diffraction Pattern from the Tremolite Particle pictured above

308004 FDA_038.jpg
Tremolite 1
17:57 7/29/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

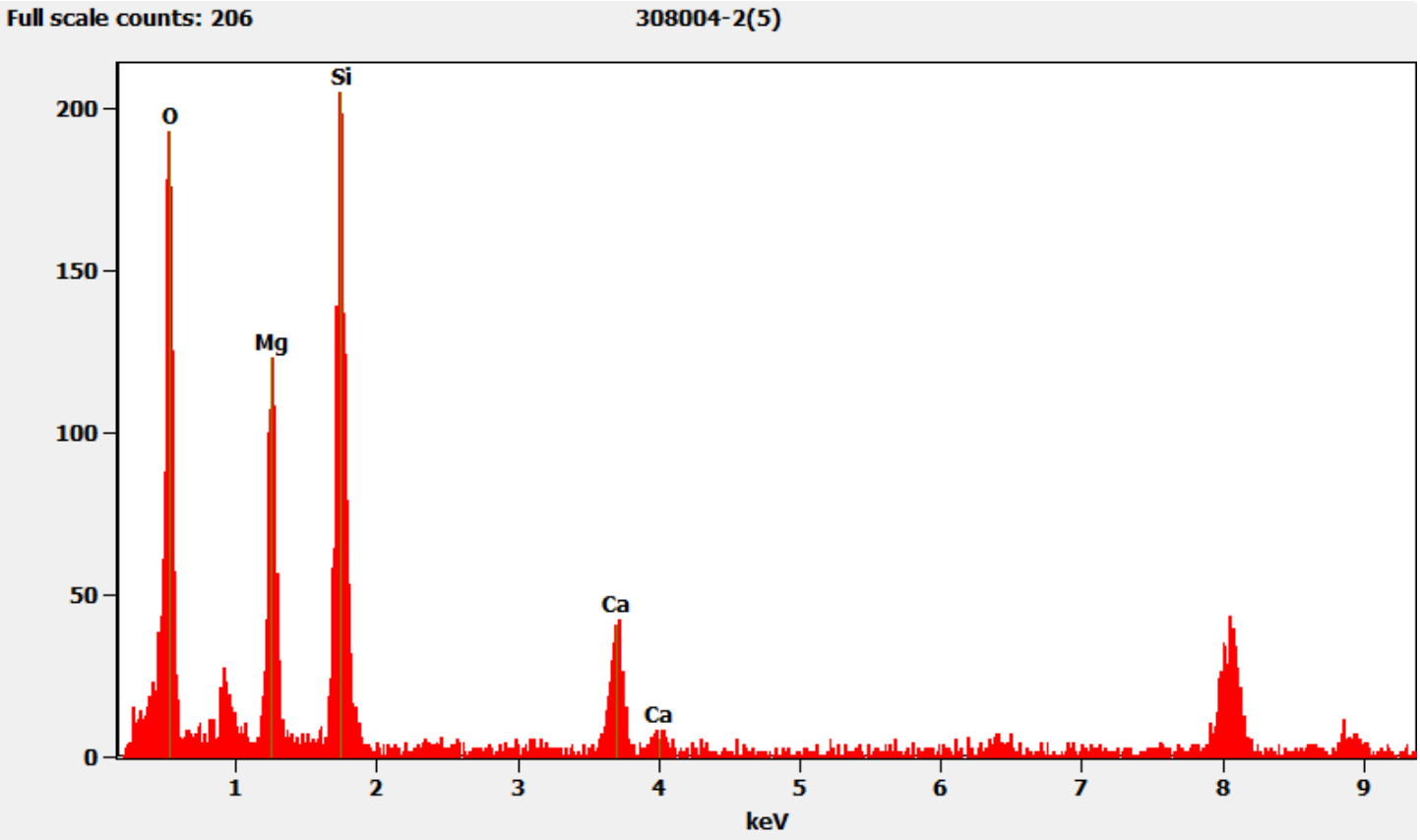
100 (1/Å)
HV=100kV
Cam Len: 0.2200 m
AMA Analytical Services, Inc

Diffraction Pattern from the Tremolite Particle pictured above

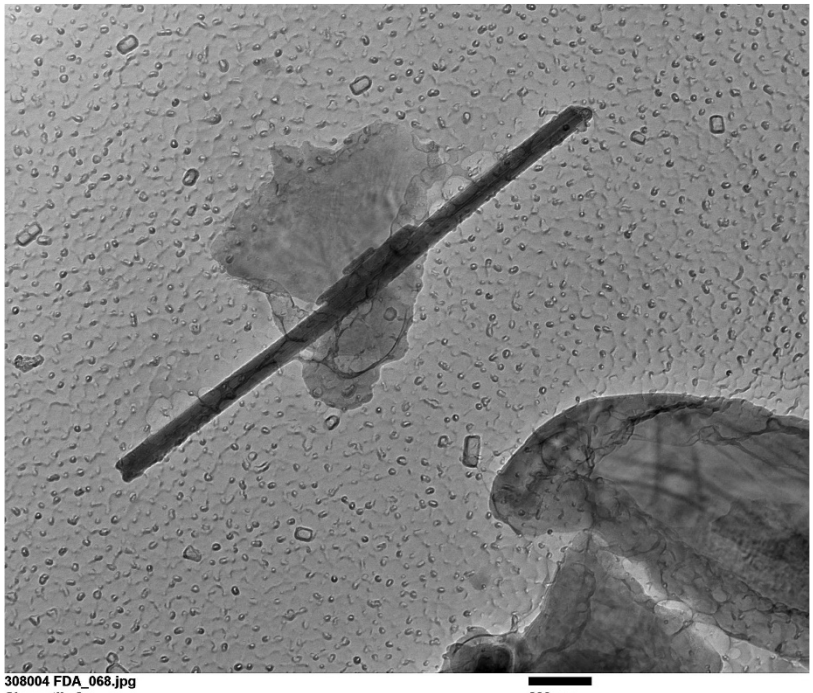
308004 FDA_039.jpg
Tremolite 1
17:57 7/29/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

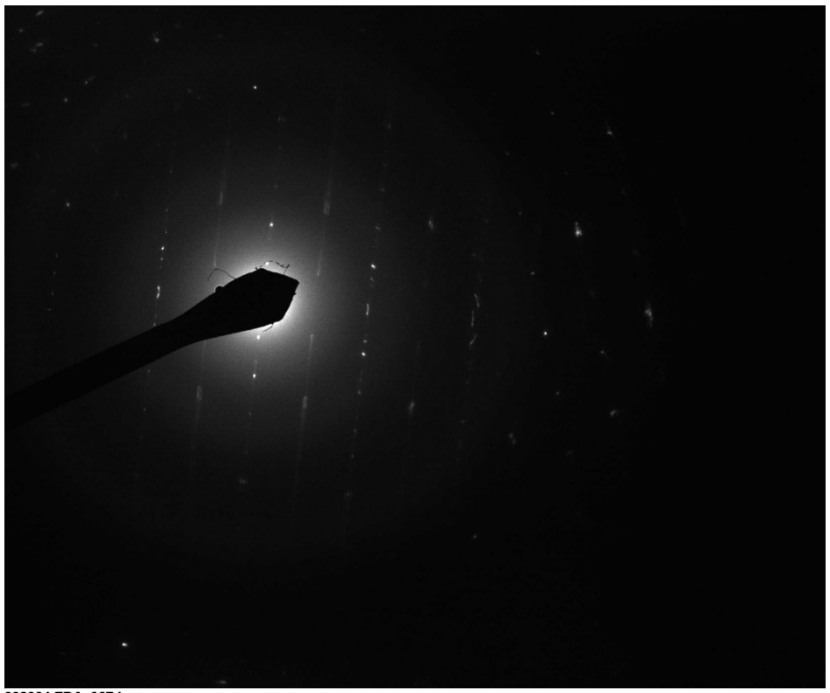
Chemistry from the Tremolite Particle pictured above



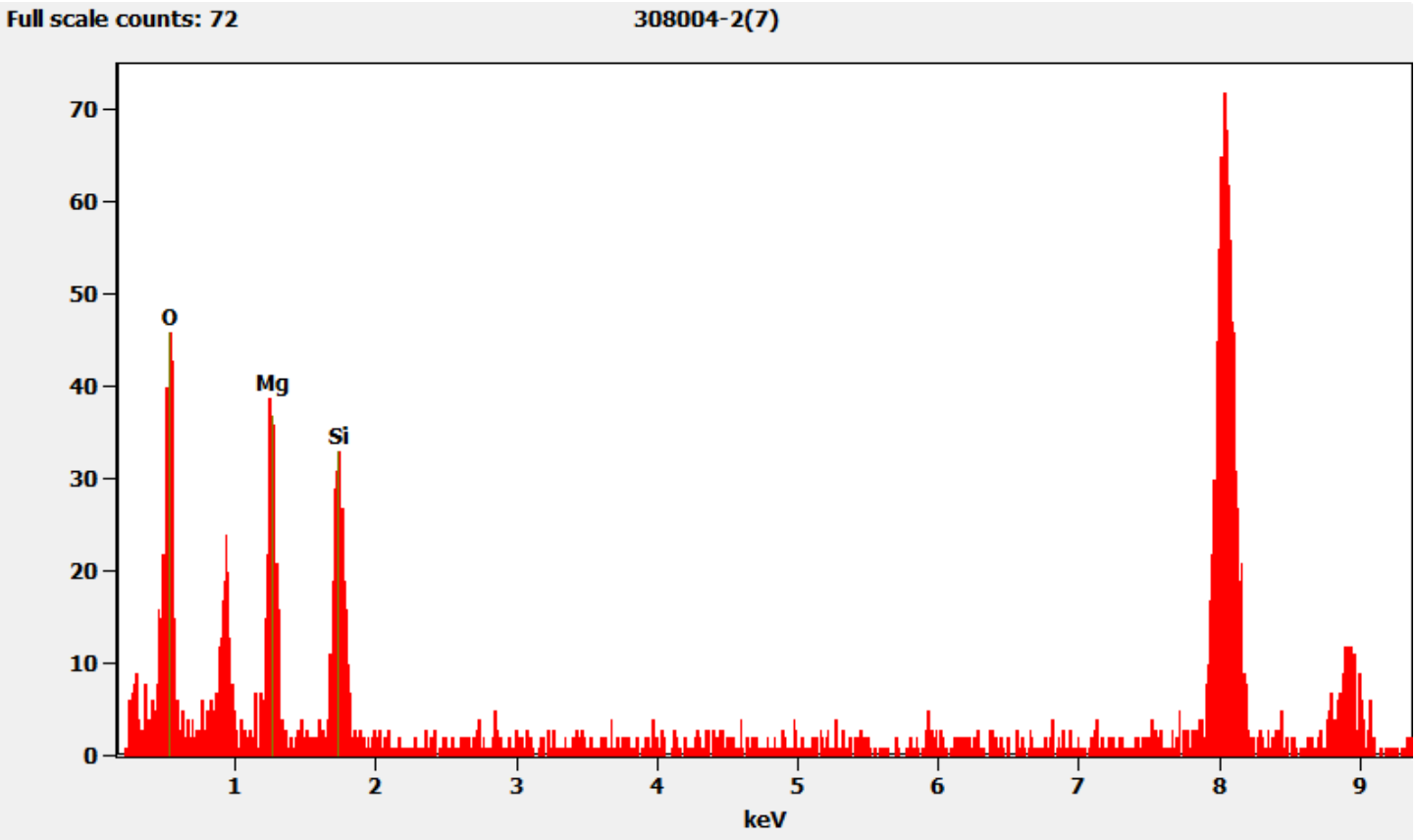
Chrysotile Fiber from 308004-2



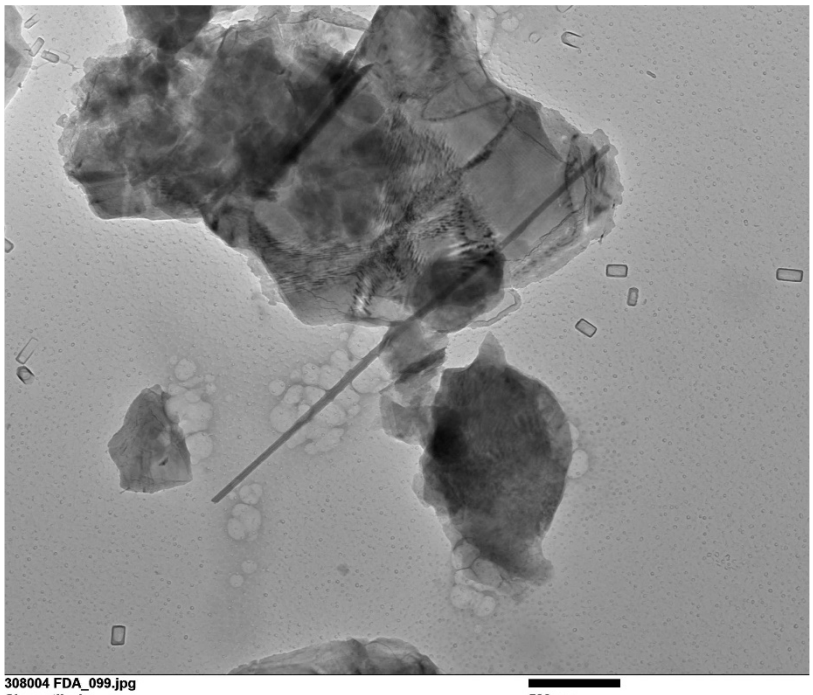
Diffraction Pattern from the Chrysotile Fiber pictured above



Chemistry from the Chrysotile Fiber pictured above



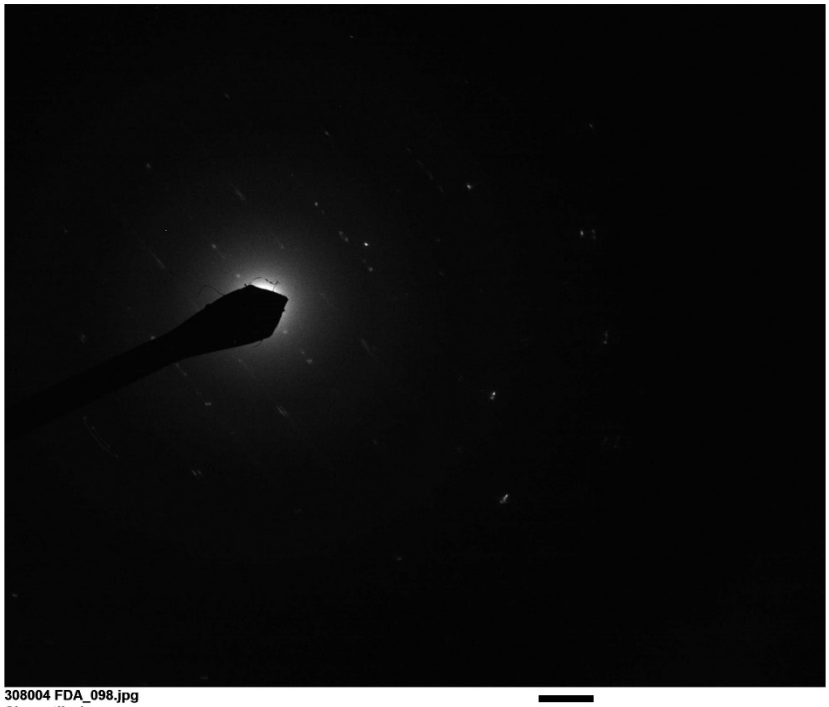
Chrysotile Fiber from 308004-2B



308004 FDA_099.jpg
Chrysotile 4
Cal: 0.001774 µm/pix
15:28 8/8/2019
TEM Mode: Imaging
Microscopist: (b)
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

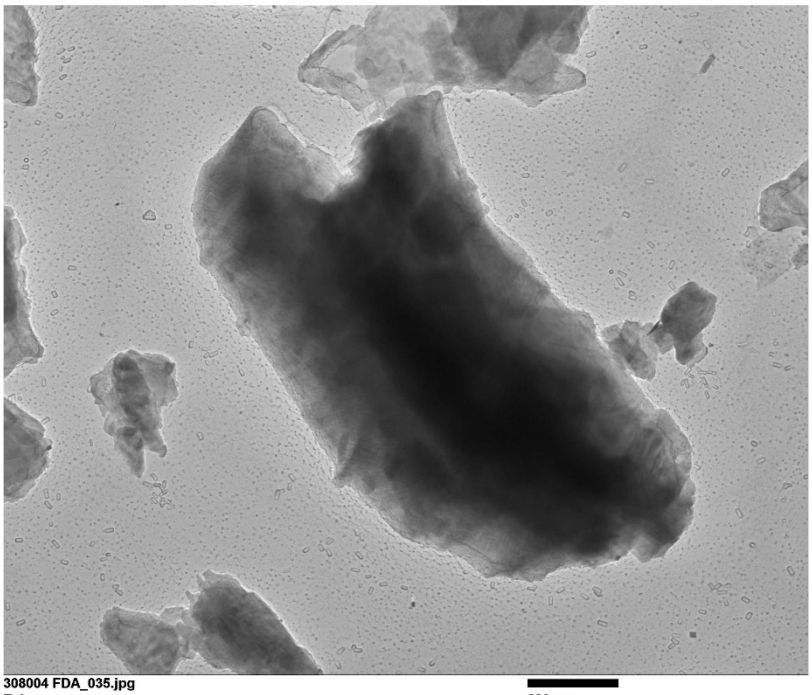
Diffraction Pattern from the Chrysotile Fiber pictured above



308004 FDA_098.jpg
Chrysotile 4
15:26 8/8/2019
TEM Mode: Diffraction
Microscopist: (b)
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

Talc Particle from 308004-2



308004 FDA_035.jpg
Talc
Cal: 0.002858 µm/pix
17:48 7/29/2019
TEM Mode: Imaging
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

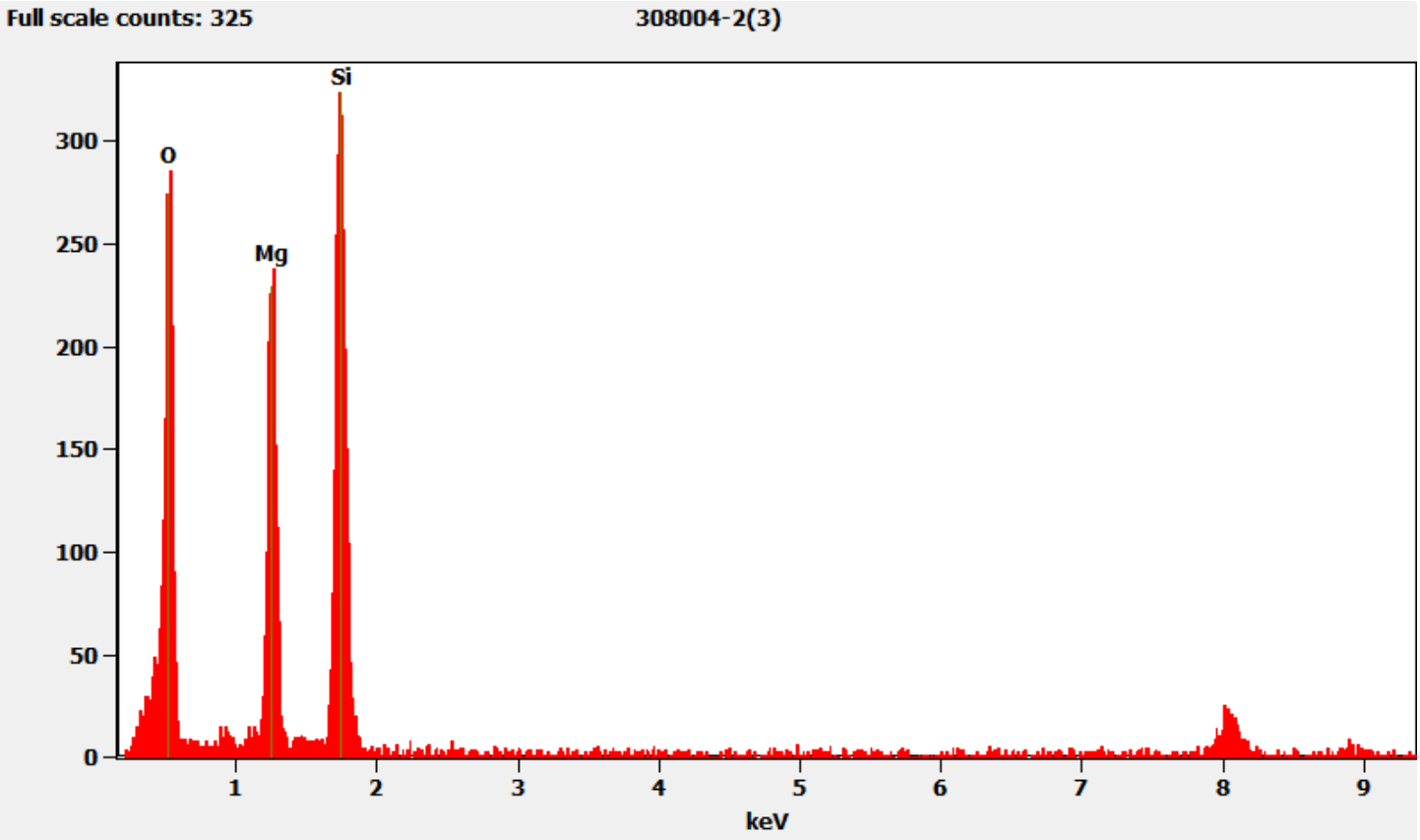
800 nm
HV=100kV
Direct Mag: 3600 x
AMA Analytical Services, Inc

Diffraction Pattern from the Talc Particle pictured above

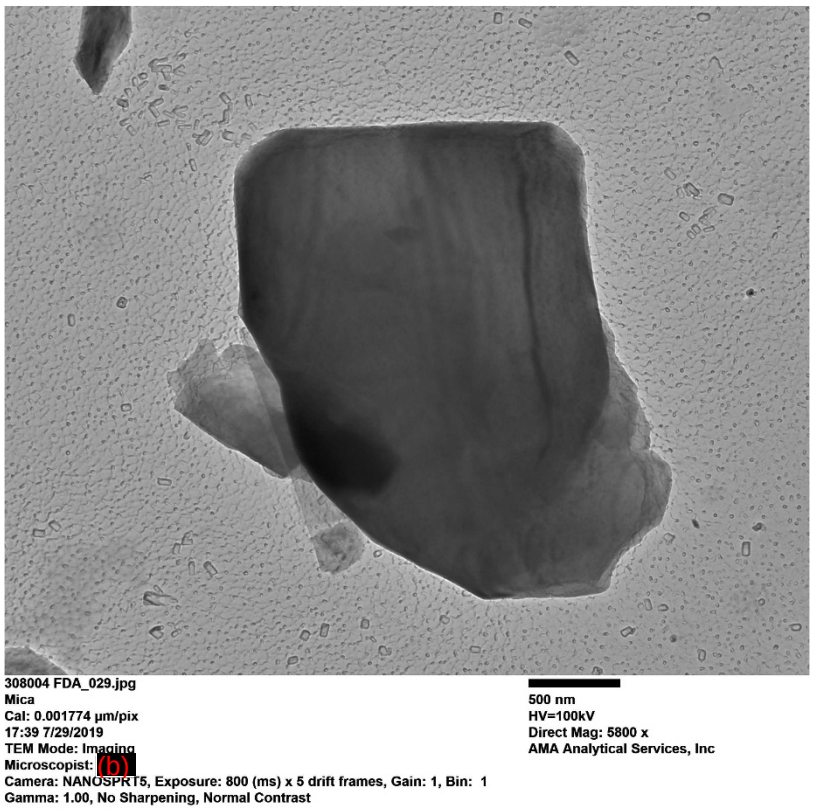
308004 FDA_036.jpg
Talc
17:50 7/29/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from the Talc Particle pictured above

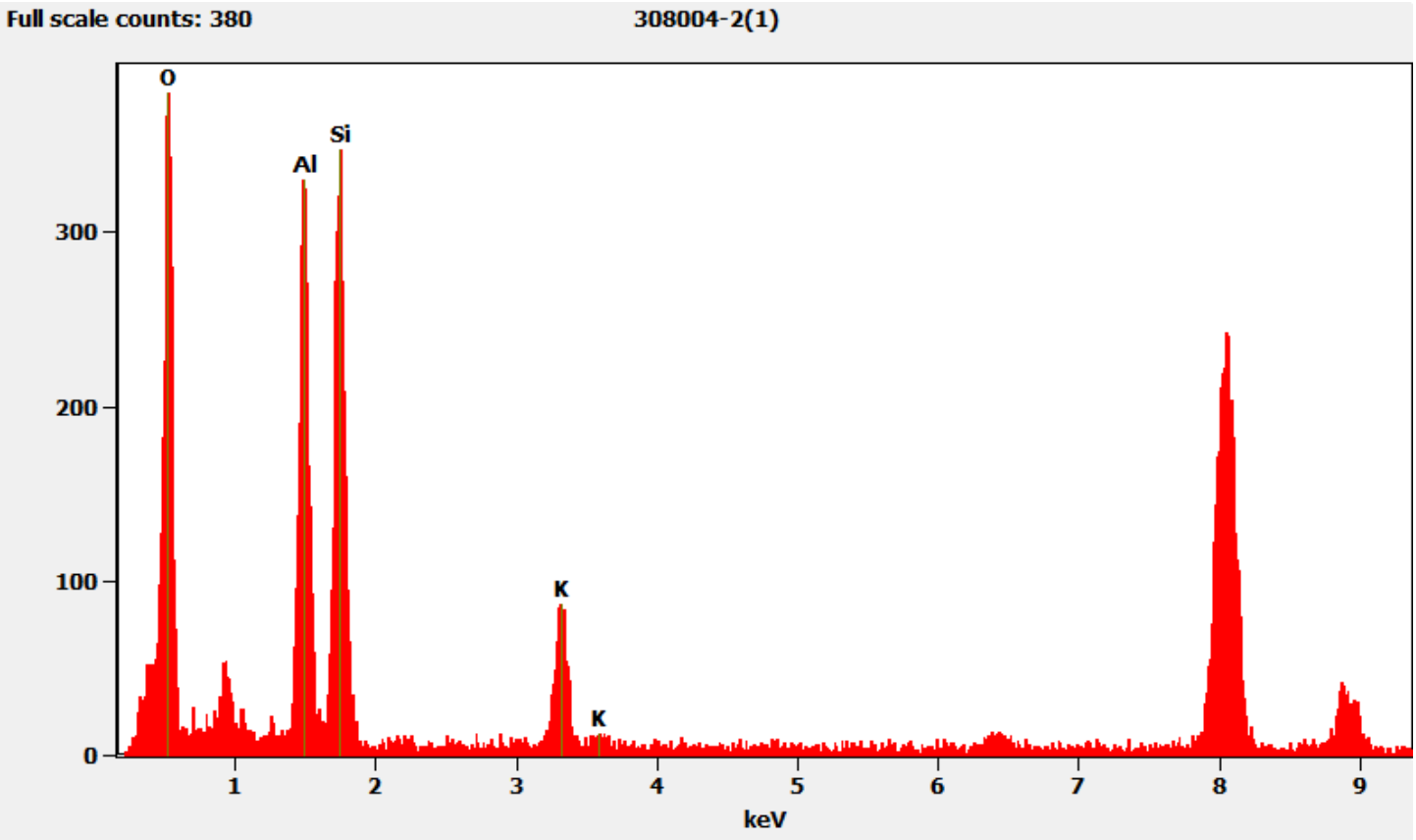


Mica Particle from 308004-2

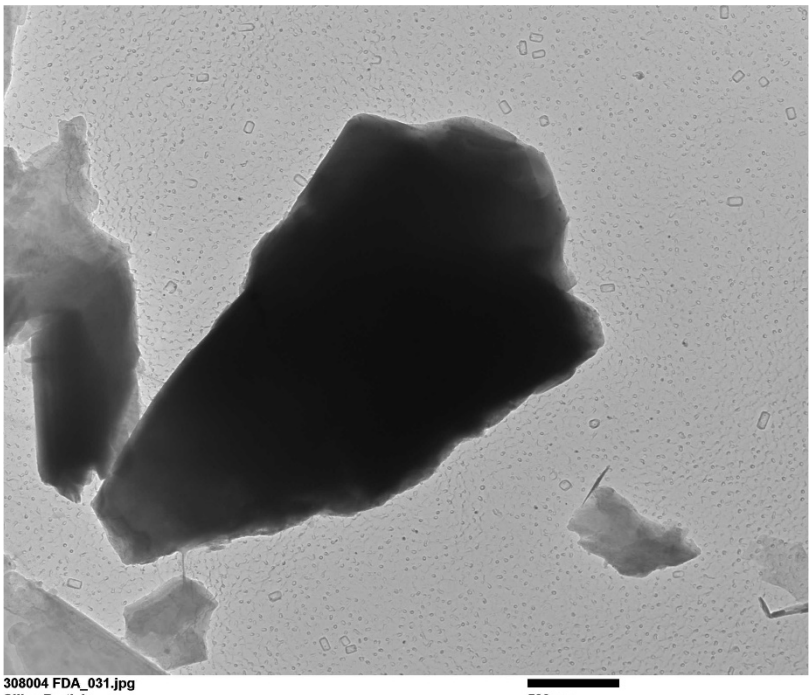


Diffraction Pattern from the Mica Particle pictured above

Chemistry from the Mica Particle pictured above



Silica Particle from 308004-1



308004 FDA_031.jpg
Silica Particle
Cal: 0.001774 µm/pix
17:42 7/29/2019
TEM Mode: Imaging
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

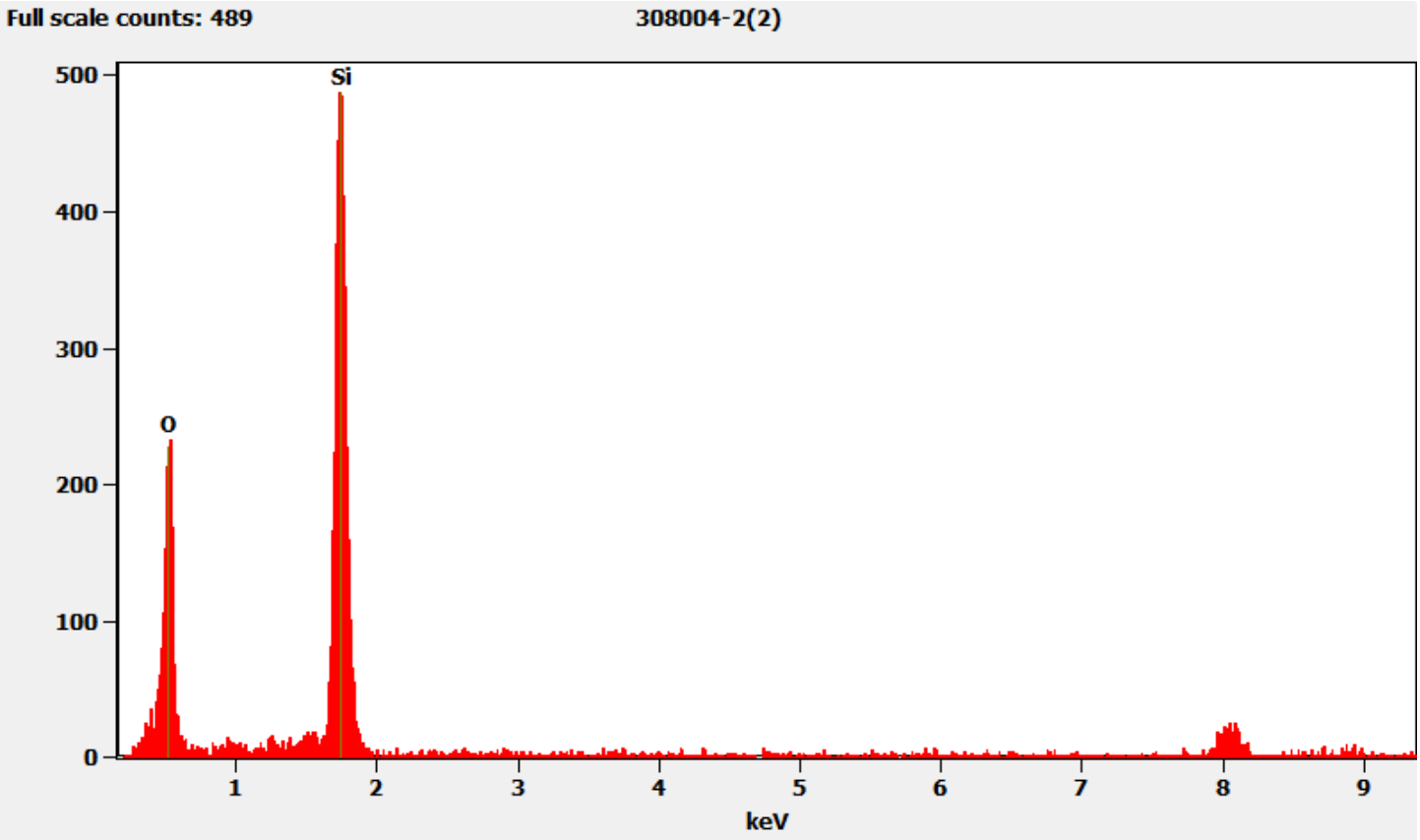
500 nm
HV=100kV
Direct Mag: 5800 x
AMA Analytical Services, Inc

Diffraction Pattern from the Silica Particle pictured above

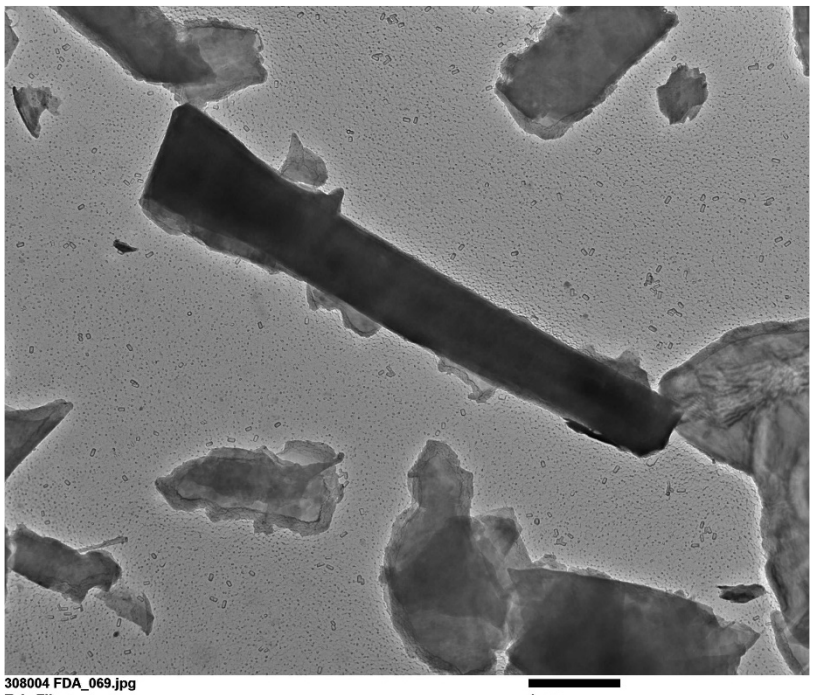
308004 FDA_032.jpg
Silica Particle
17:43 7/29/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from the Silica Particle pictured above



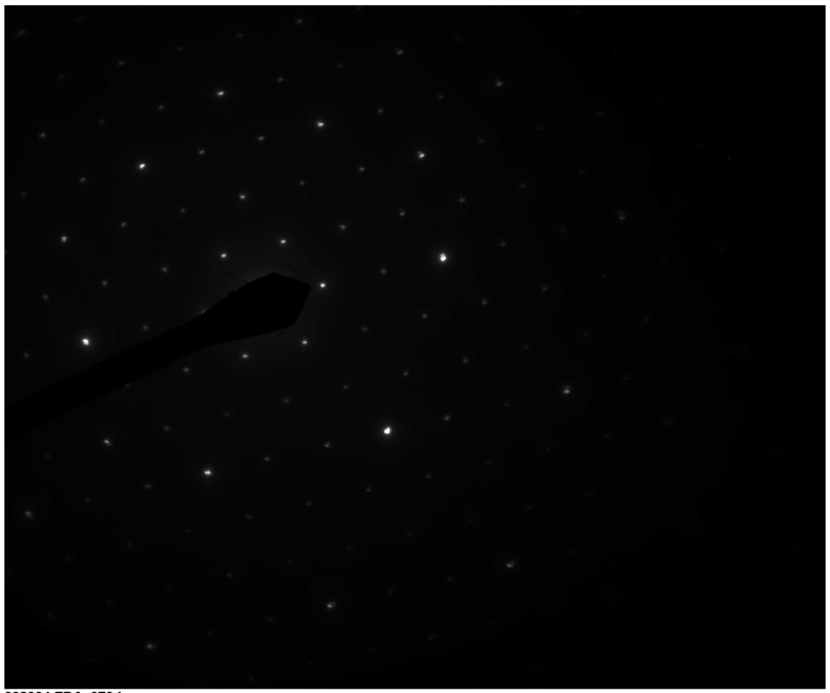
Talc Fiber from 308004-2



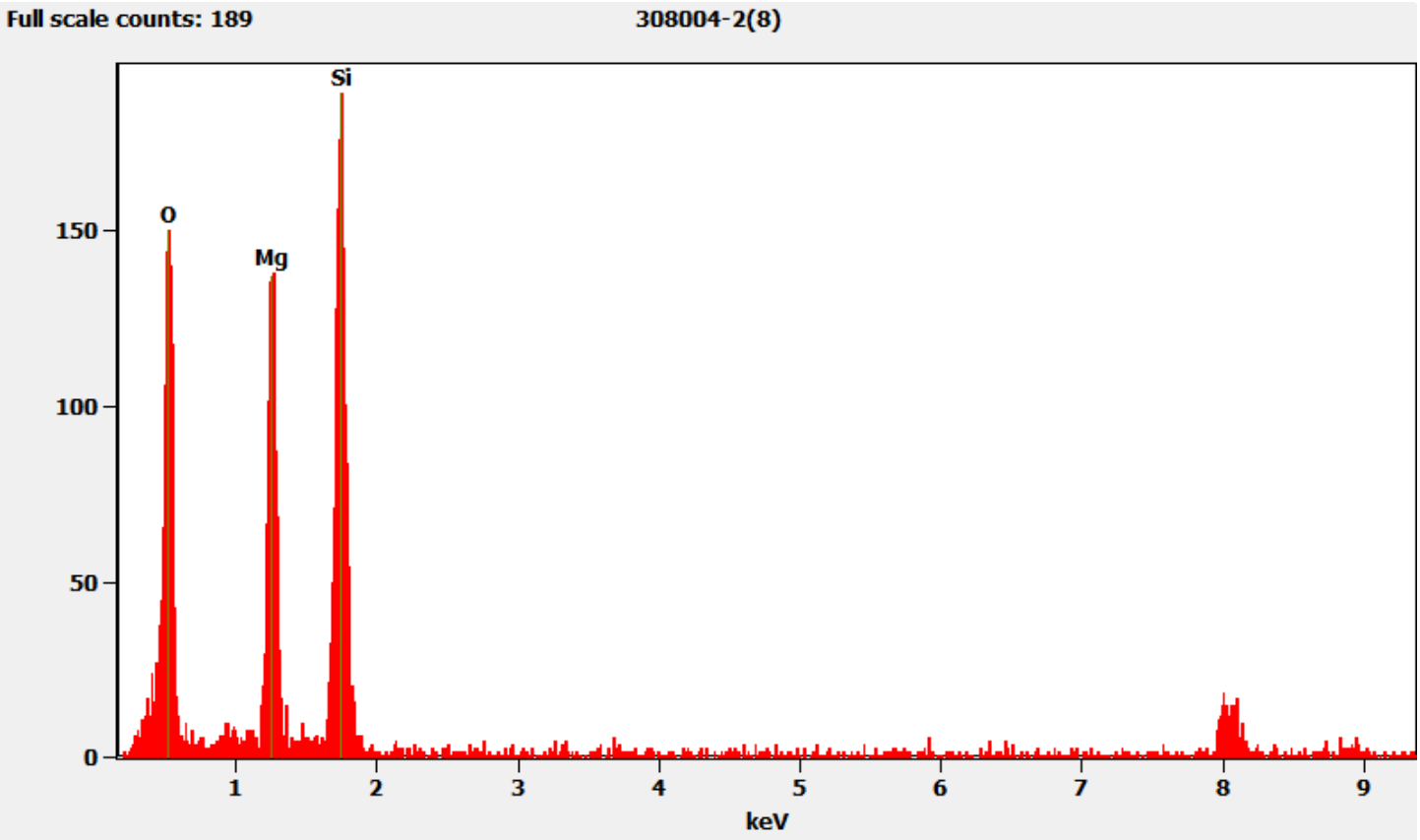
308004 FDA_069.jpg
Talc Fiber
Cal: 0.003548 µm/pix
16:14 8/6/2019
TEM Mode: Imaging
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

1 µm
HV=100kV
Direct Mag: 2900 x
AMA Analytical Services, Inc

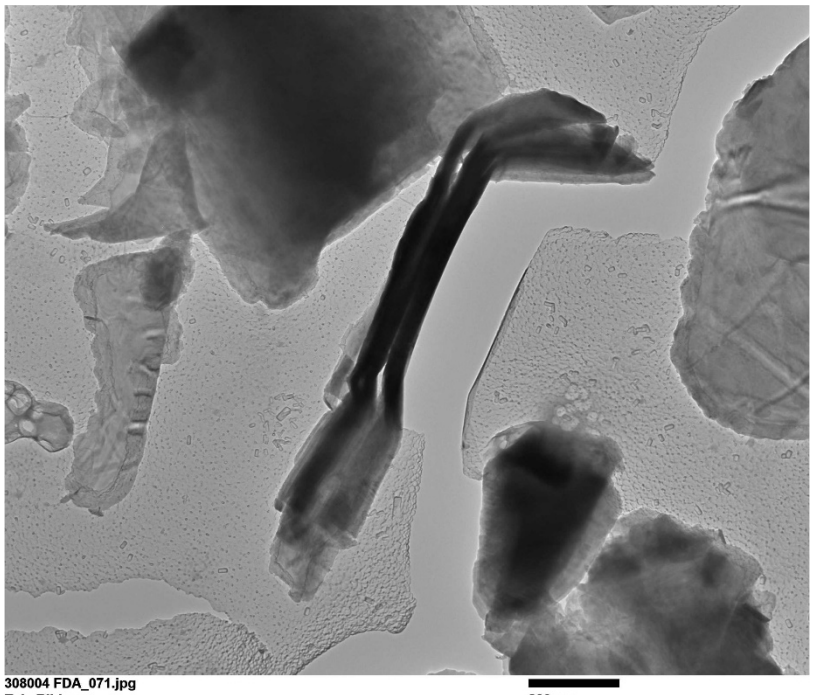
Diffraction Pattern from the Talc Fiber pictured above



Chemistry from the Talc Fiber pictured above



Talc Ribbon from 308004-2



308004 FDA_071.jpg
Talc Ribbon
Cal: 0.002858 µm/pix
16:18 8/6/2019
TEM Mode: Imaging
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

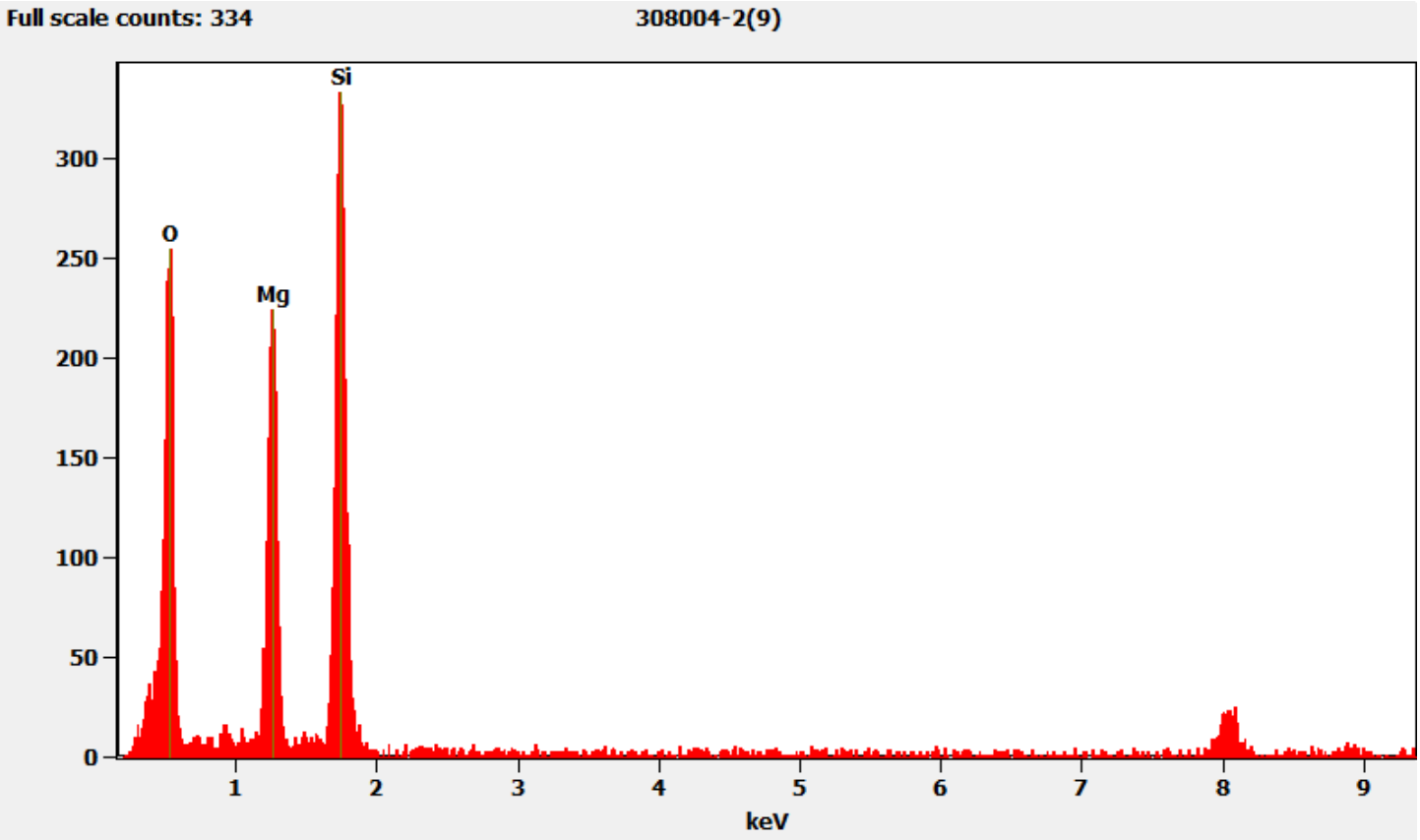
800 nm
HV=100kV
Direct Mag: 3600 x
AMA Analytical Services, Inc

Diffraction Pattern from 308004-2

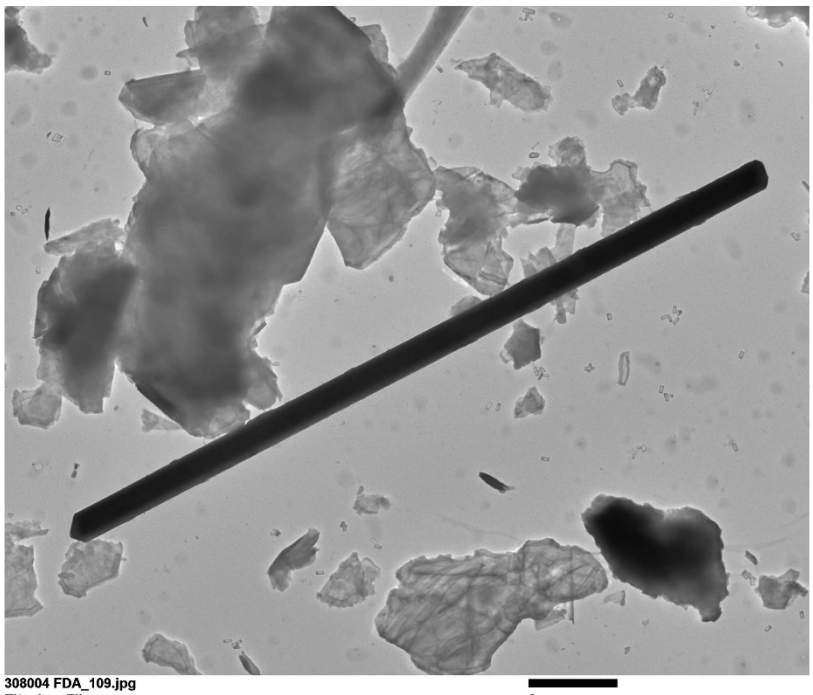
308004 FDA_072.jpg
Talc Ribbon
16:19 8/6/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

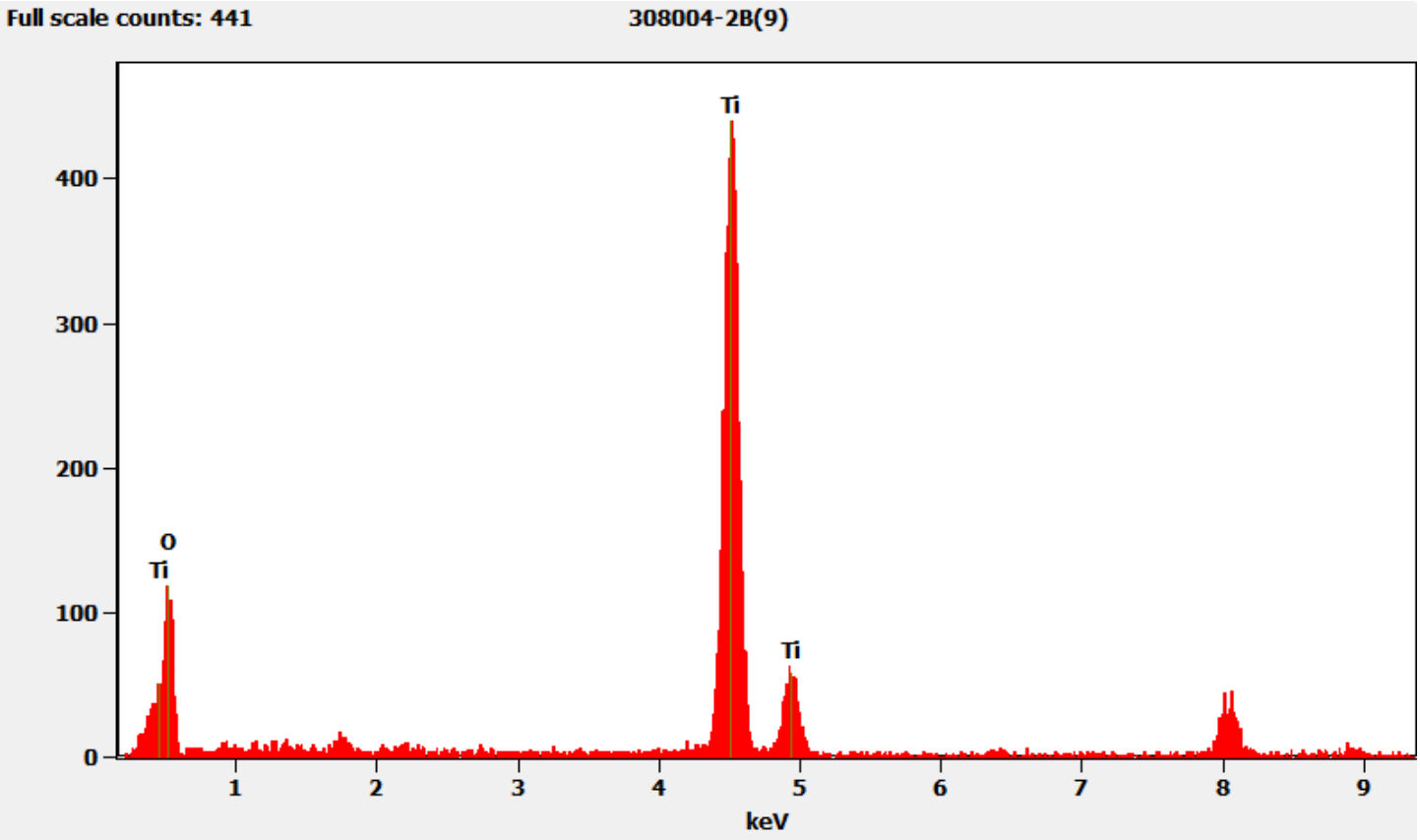
Chemistry from the Talc Ribbon pictured above



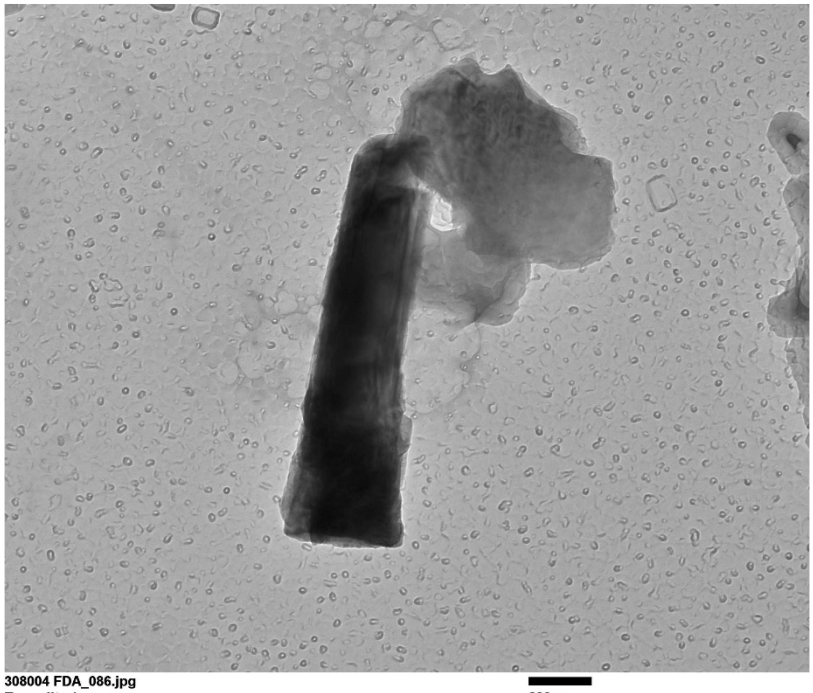
Titanium Fiber from 308004-2B



Chemistry from the Titanium Fiber pictured above

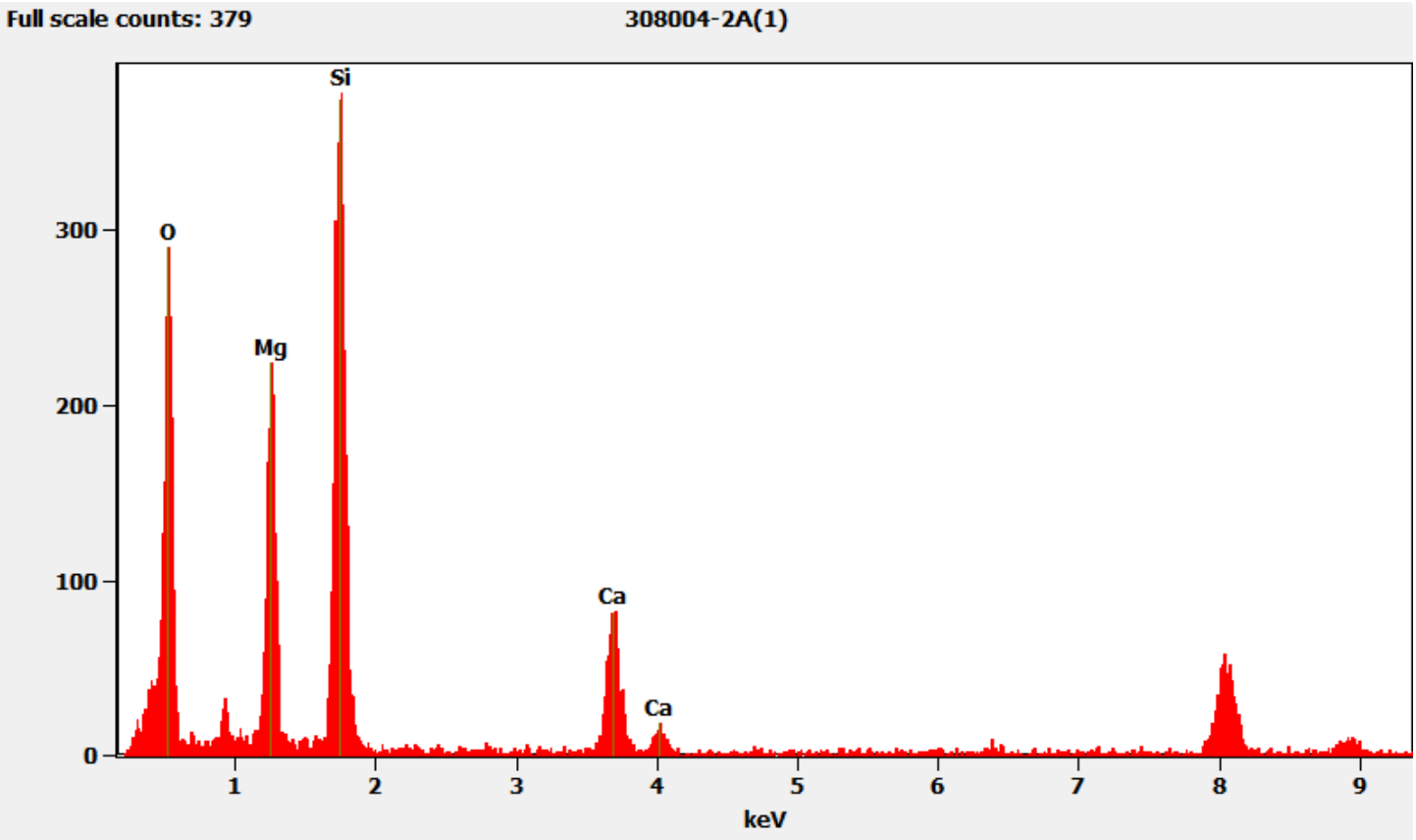


Tremolite Particle from 308004-2A

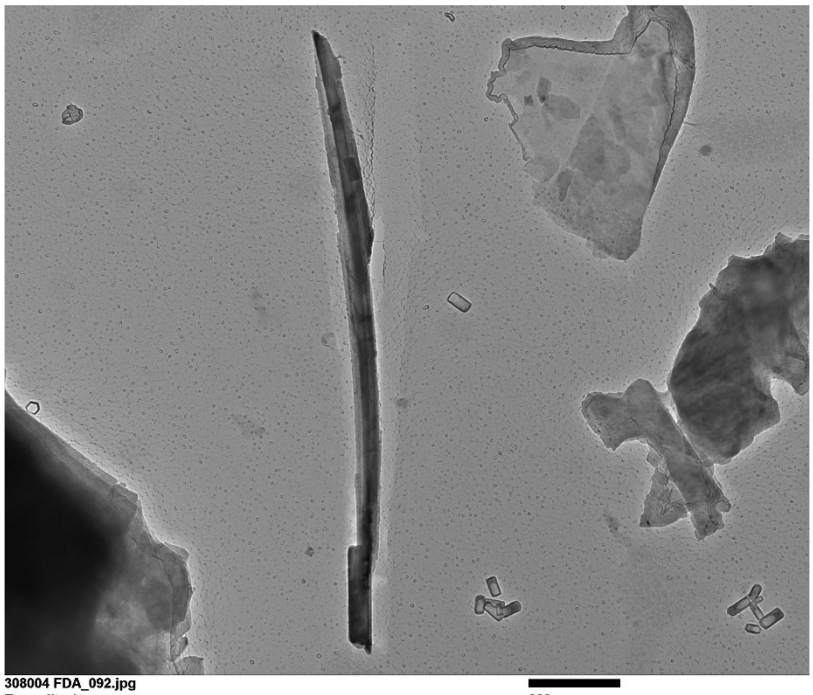


Diffraction Pattern from Tremolite Particle pictured above

Chemistry from the Tremolite Particle pictured above



Tremolite Particle from 308004-2B



308004 FDA_092.jpg
Tremolite 1
Cal: 0.002144 µm/pix
14:43 8/8/2019
TEM Mode: Imaging
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

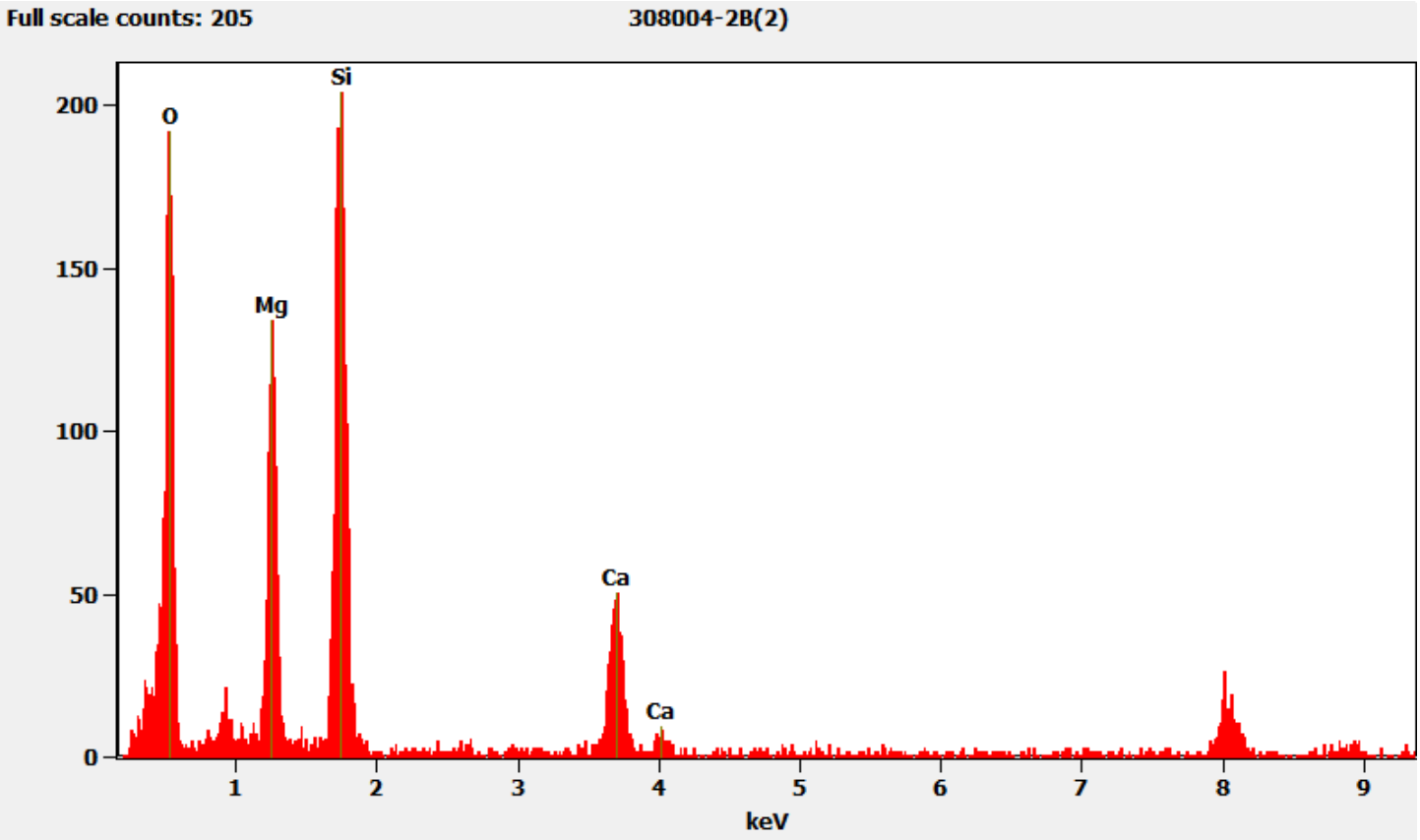
600 nm
HV=100kV
Direct Mag: 4800 x
AMA Analytical Services, Inc

Diffraction Pattern from the Tremolite Particle pictured above

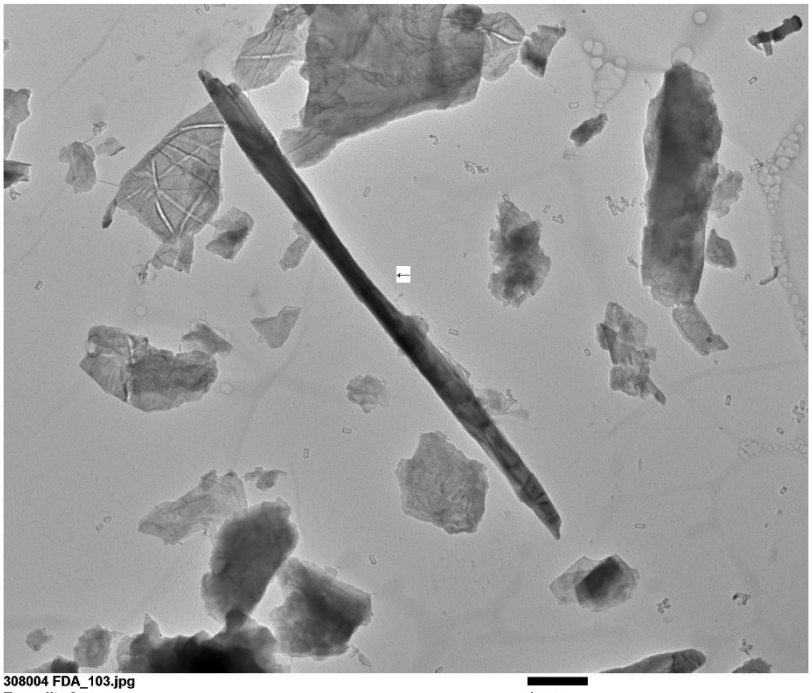
308004 FDA_093.jpg
Tremolite 1
14:45 8/8/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from the Tremolite Particle pictured above

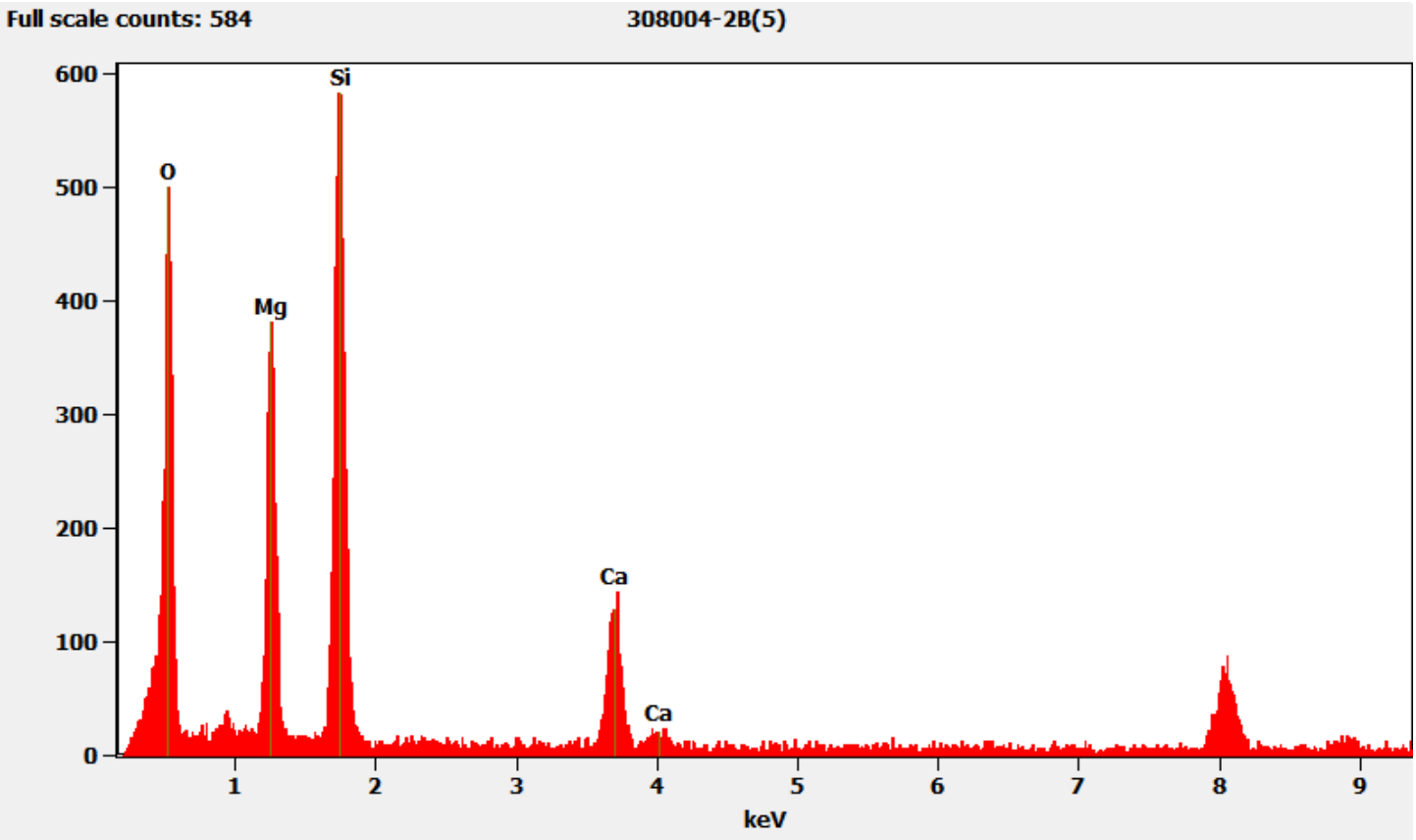


Tremolite Particle from 308004-2B

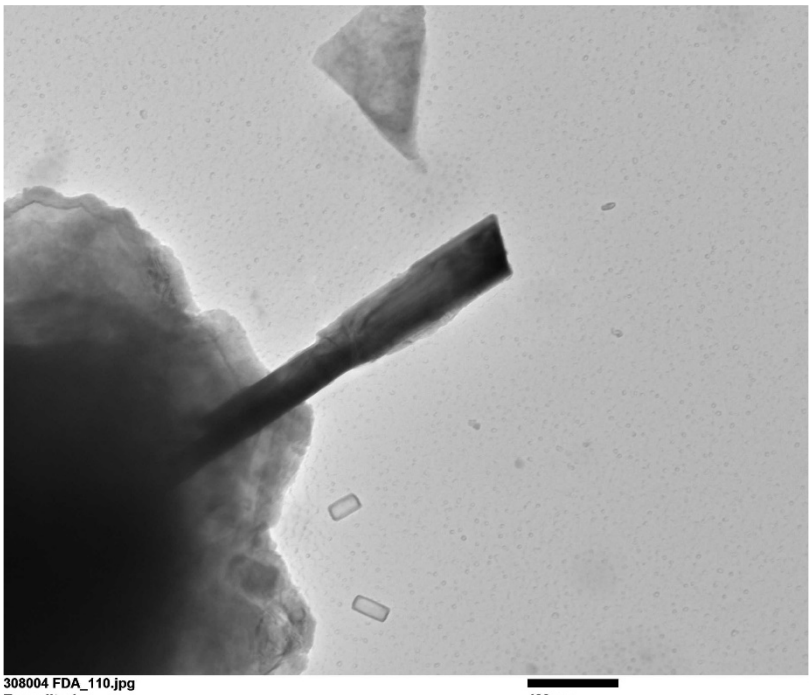


Diffraction Pattern from the Tremolite Particle pictured above

Chemistry from the Tremolite Particle pictured above



Tremolite Particle from 308004-2B



308004 FDA_110.jpg
Tremolite 4
Cal: 0.001429 µm/pix
17:04 8/8/2019
TEM Mode: Imaging
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

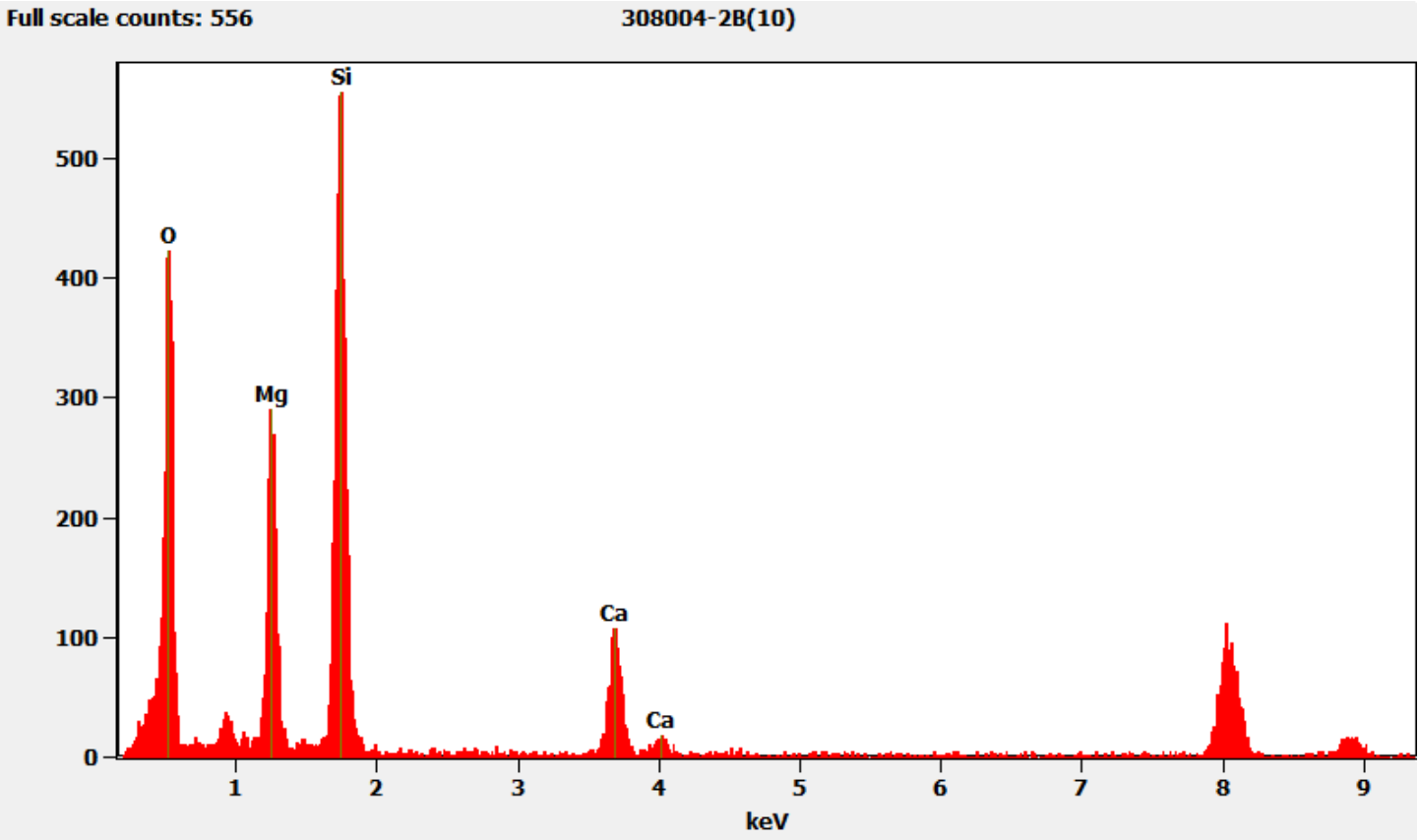
400 nm
HV=100kV
Direct Mag: 7200 x
AMA Analytical Services, Inc

Diffraction Pattern from the Tremolite Particle pictured above

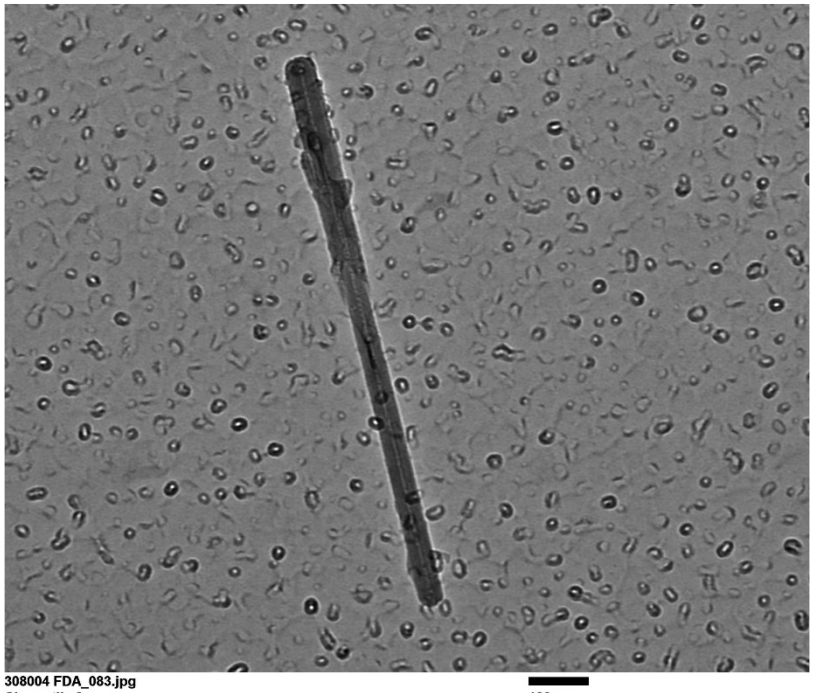
308004 FDA_111.jpg
Tremolite 4
17:06 8/8/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

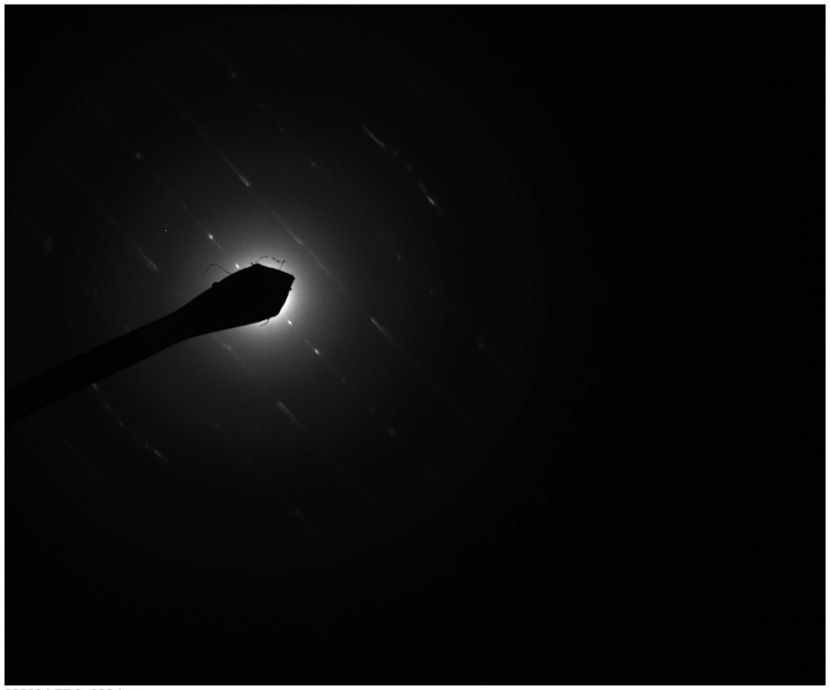
Chemistry from the Tremolite Particle pictured above



Chrysotile Fiber from 308004-2A



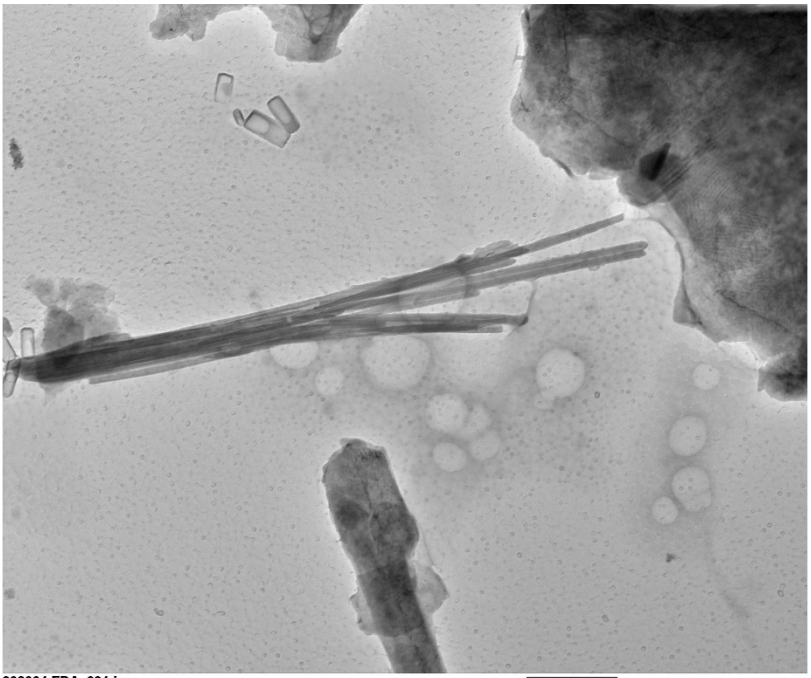
Diffraction Pattern from the Chrysotile Fiber pictured above



308004 FDA_082.jpg
Chrysotile 2
15:45 8/7/2019
TEM Mode: Diffraction
Microscopist: (b)
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

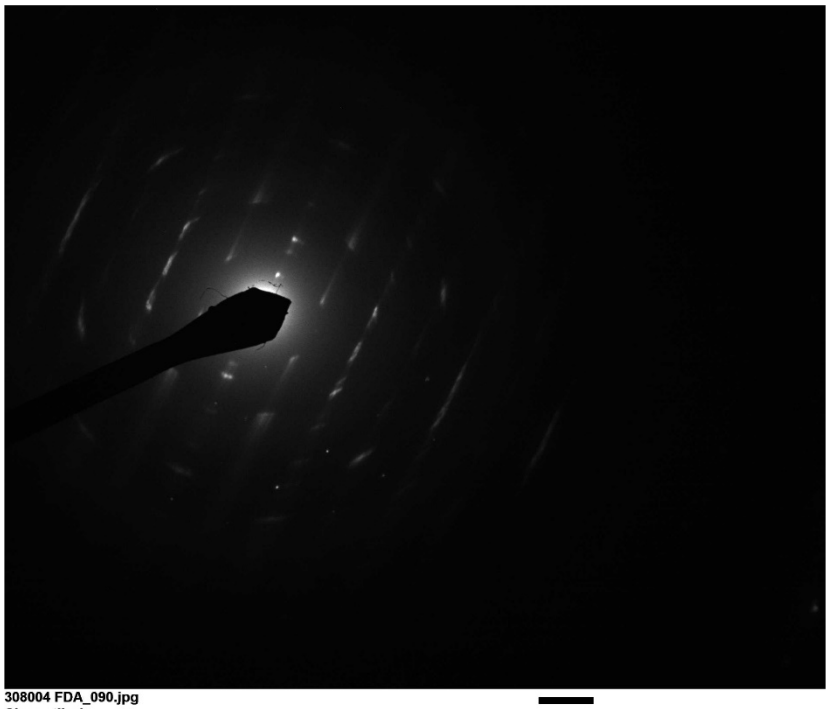
Chrysotile Structure from 308004-2B



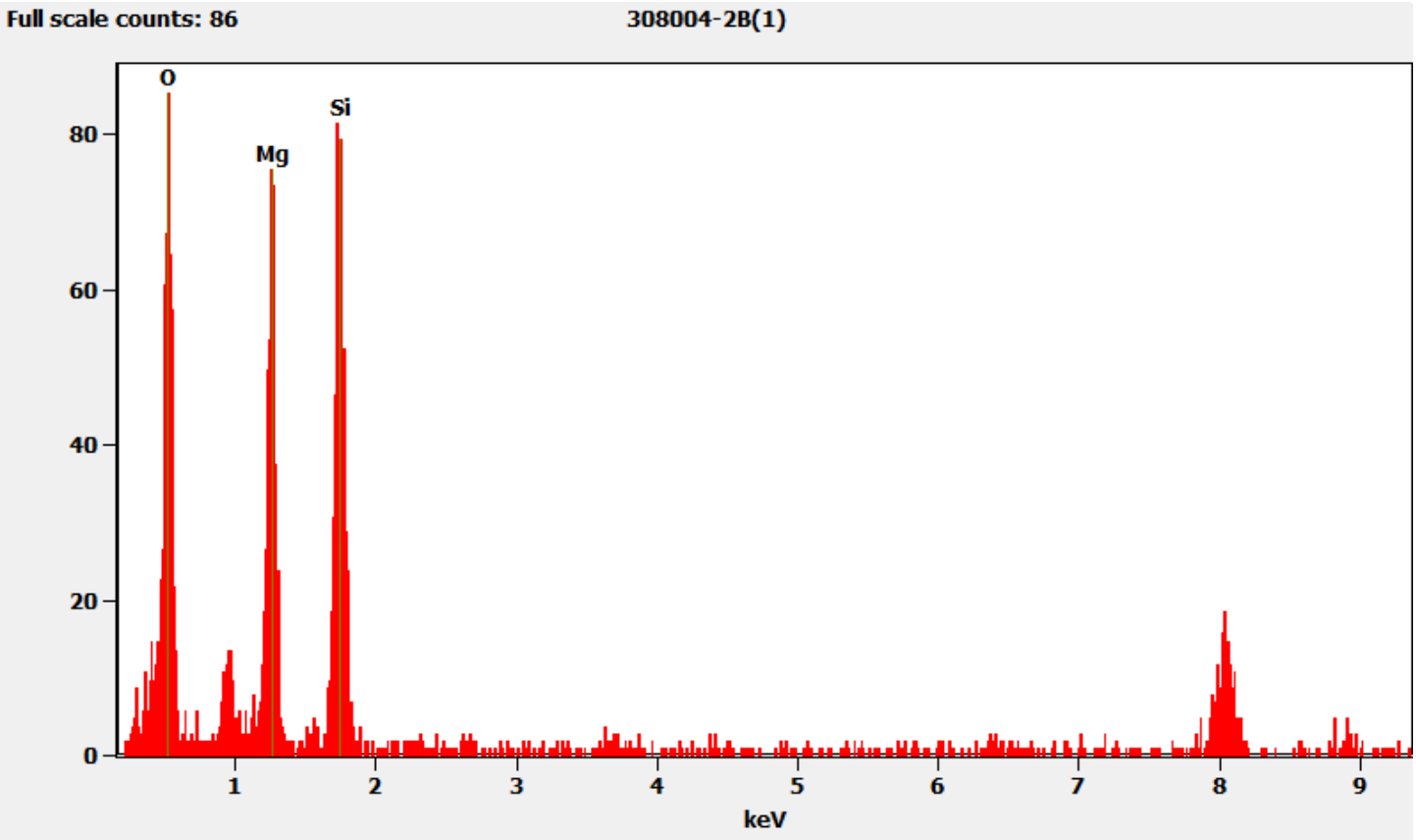
308004 FDA_091.jpg
Chrysotile 1
Cal: 0.001429 µm/pix
14:28 8/8/2019
TEM Mode: Imaging
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

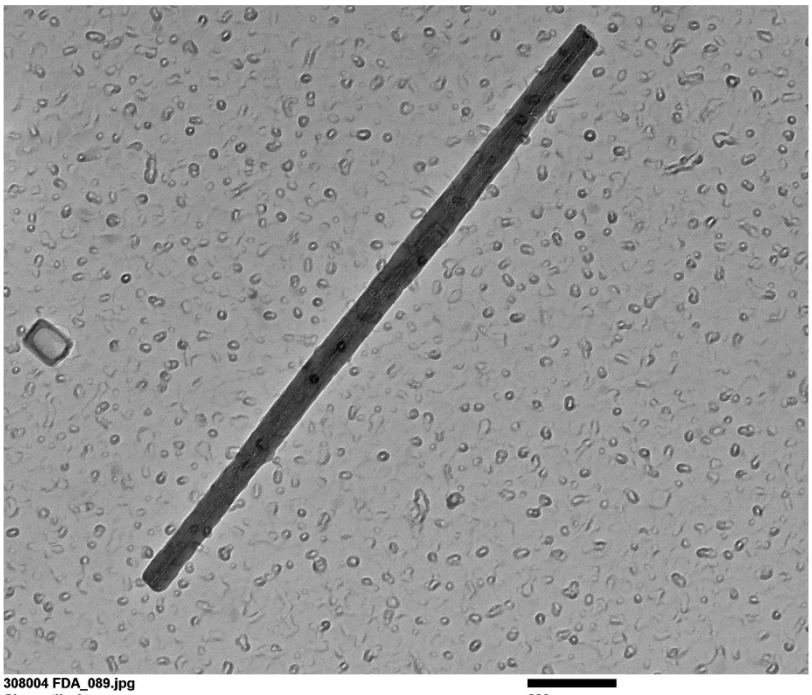
Diffraction Pattern from the Chrysotile Structure pictured above



Chemistry from the Chrysotile Structure pictured above



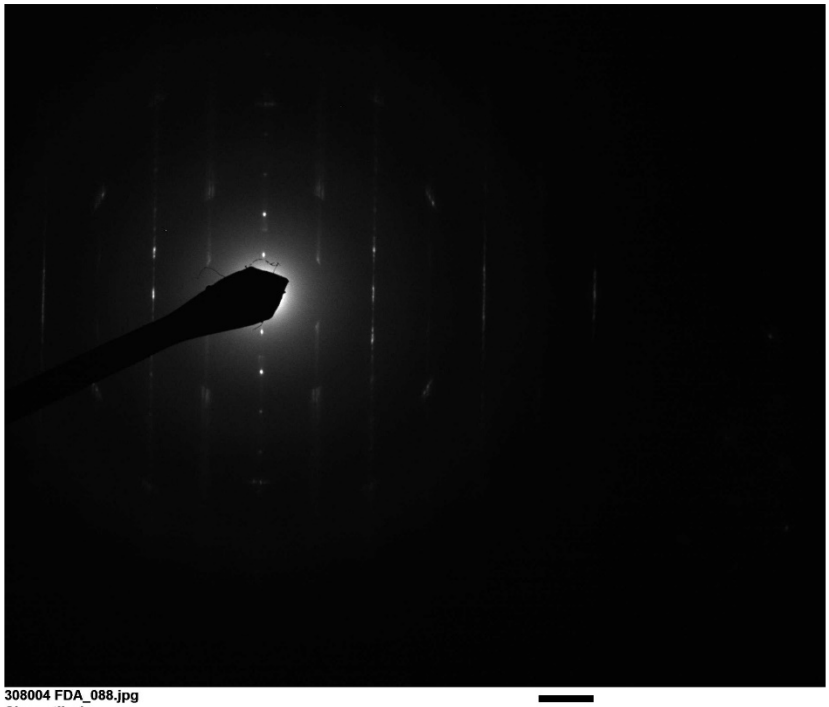
Chrysotile Fiber from 308004-2A



308004 FDA_089.jpg
Chrysotile 4
Cal: 0.734921 nm/pix
16:29 8/7/2019
TEM Mode: Imaging
Microscopist: [redacted]
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: 14000 x
AMA Analytical Services, Inc

Diffraction Pattern from the Chrysotile Fiber pictured above



308004 FDA_088.jpg
Chrysotile 4
16:29 8/7/2019
TEM Mode: Diffraction
Microscopist: [redacted]
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

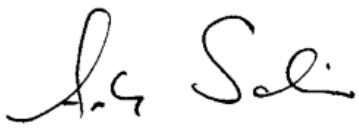
QC Discussion:
During preparation, one blank control sample and one reference control sample were prepared. These samples were prepared alongside the customer samples. The blank sample was prepared using Sigma-Aldrich Talc Powder, <10 micron and was analyzed by (b) (6) on August 8, 2019. No asbestos 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 (b) (6) on August 8, 2019 and found to be within acceptable limits.

Our LIMS randomly selects samples for additional replicate and duplicate QC. 308004-2, 2A, and 2B/D-50 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 (b) (6) for samples analyzed between 1/1/2019 & 8/8/2019
7) Replicate and Duplicate QC Charts for (b) (6) 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.



8/15/2019
Andreas Saldivar — Date
Laboratory Director