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Focused On Results.

CERTIFICATE OF ANALYSIS

Chain of Custody: 307491
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: 2nd Group - 10 Samples
Job Number: CLIN 1 - Task 3 (10 Samples)
PO Number: HHSF223201810337P

Date Submitted: 5/23/2019
Date Analyzed: 6/27/2019 - 7/17/2019
Report Date: 7/24/2019
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
307491-12	D-52	0 00000105%	0.00000418%	0.109%	<0.000008%	0.109%	ND	19.0%	17.6%	63.3%	
307491-12A	D-52	0.00000131%	0.00000526%	0.674%	ND	0.674%	ND	19.4%	17.2%	63.3%	
307491-12B	D-52	0.00000107%	0.00000427%	0 226%	ND	0 226%	ND	19.0%	16.5%	64.5%	

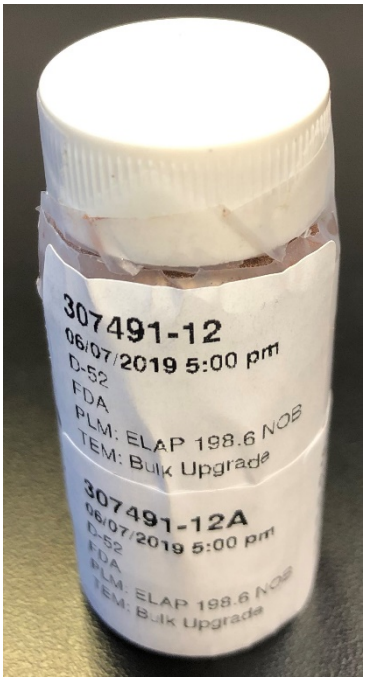
LOD = Limit of Detection **LOQ** = Limit of Quantification **ND** = Not Detected **PLM** = Polarized Light Microscopy **TEM** = Transmission Electron Microscopy

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

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 Chon Simpha on May 24, 2019 through May 31, 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 per sample were examined.

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 fiber as the basis for our calculations. Limit of detection was defined as 1 fiber and limit of quantification was defined as 4 fibers.

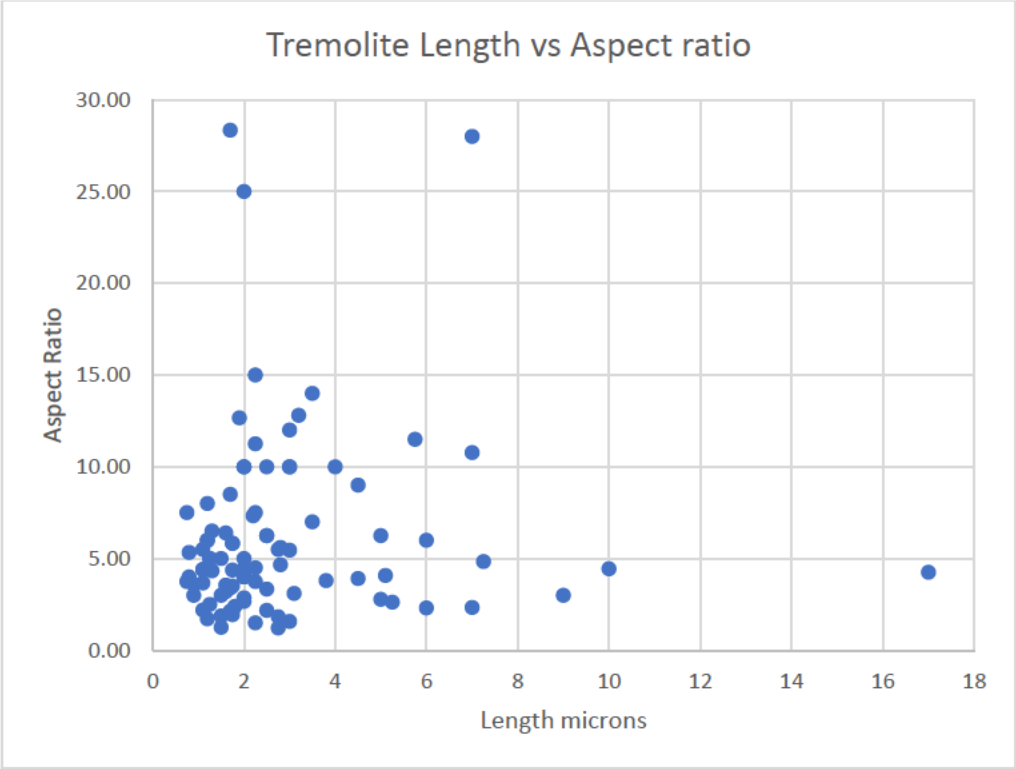
Discussion and Interpretation of Analytical Findings:
PLM
All three aliquots of sample D-52 were analyzed by Peerawut Chaikeenee on June 27, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

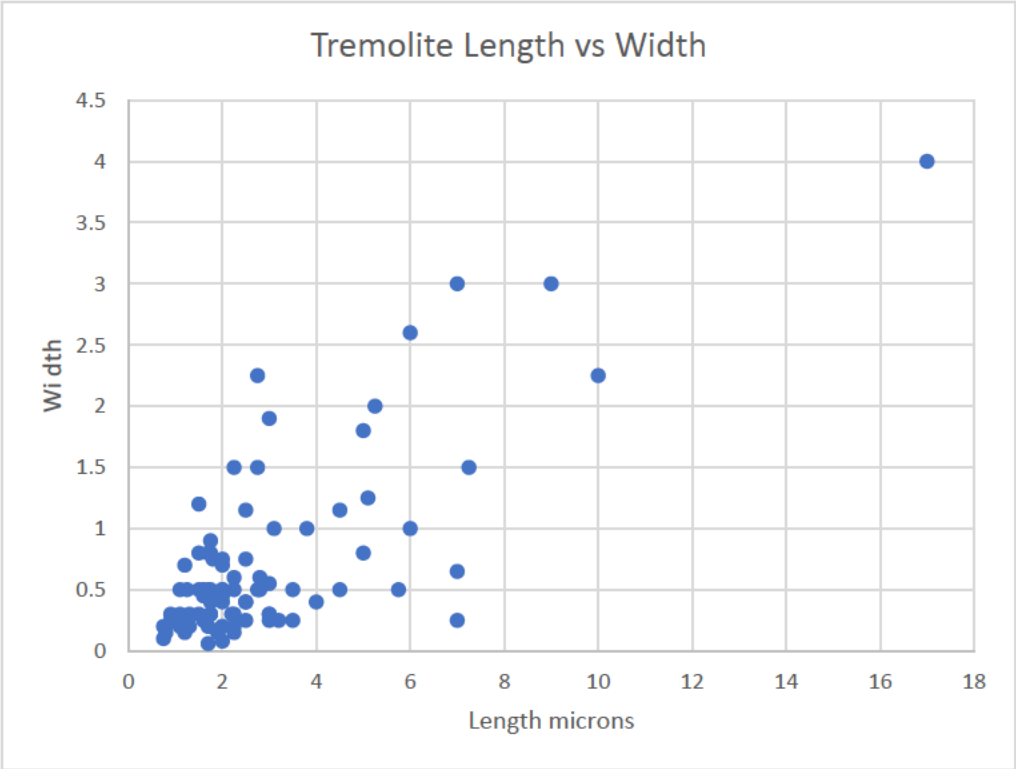
307491-12	NAD
307491-12A	NAD
307491-12B	NAD

TEM
Michael Greenberg analyzed sample 12 on July 2, 2019, 12A on July 2 & July 7, 2019, and 12B on July 7 & 17, 2019. The sample consisted of talc particles and mica particles. Some talc fibers and talc ribbons were also observed also. Tremolite was observed on all three aliquots. One chrysotile structure was observed on aliquot 12. No chrysotile was observed on aliquots 12A and 12B. The results were calculated using the equations detailed in the calculations section.

307491-12	0.109%
307491-12A	0.674%
307491-12B	0.226%

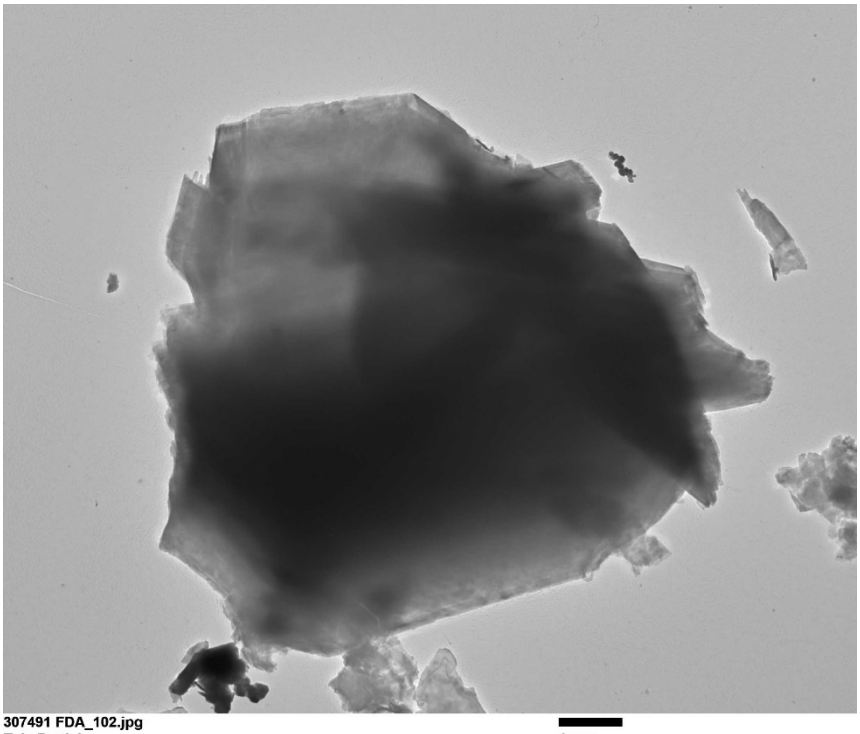
The following charts plot aspect ratio vs. length, aspect ratio vs. width, and length vs. width for all the particles counted over all three aliquots.





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

Talc particle from 307491-12



307491 FDA_102.jpg
Talc Particle
Cal: 0.005415 µm/pix
13:36 6/16/2019
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 µm
HV=100kV
Direct Mag: 1900 x
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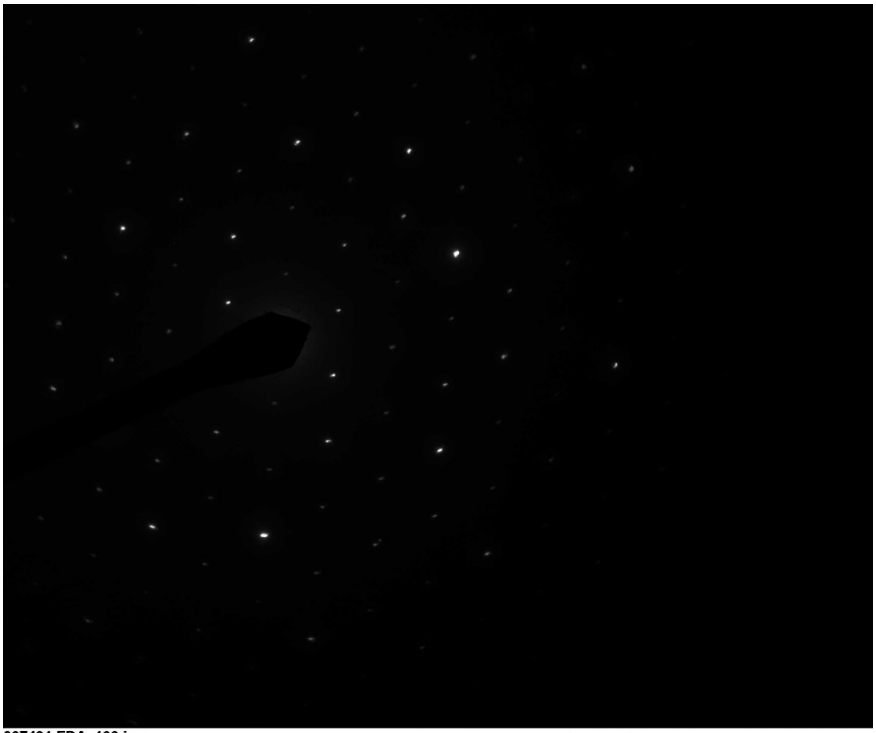
Diffraction pattern for the talc particle pictured above



307491 FDA_103.jpg
Talc Particle Diffraction
13:37 6/16/2019
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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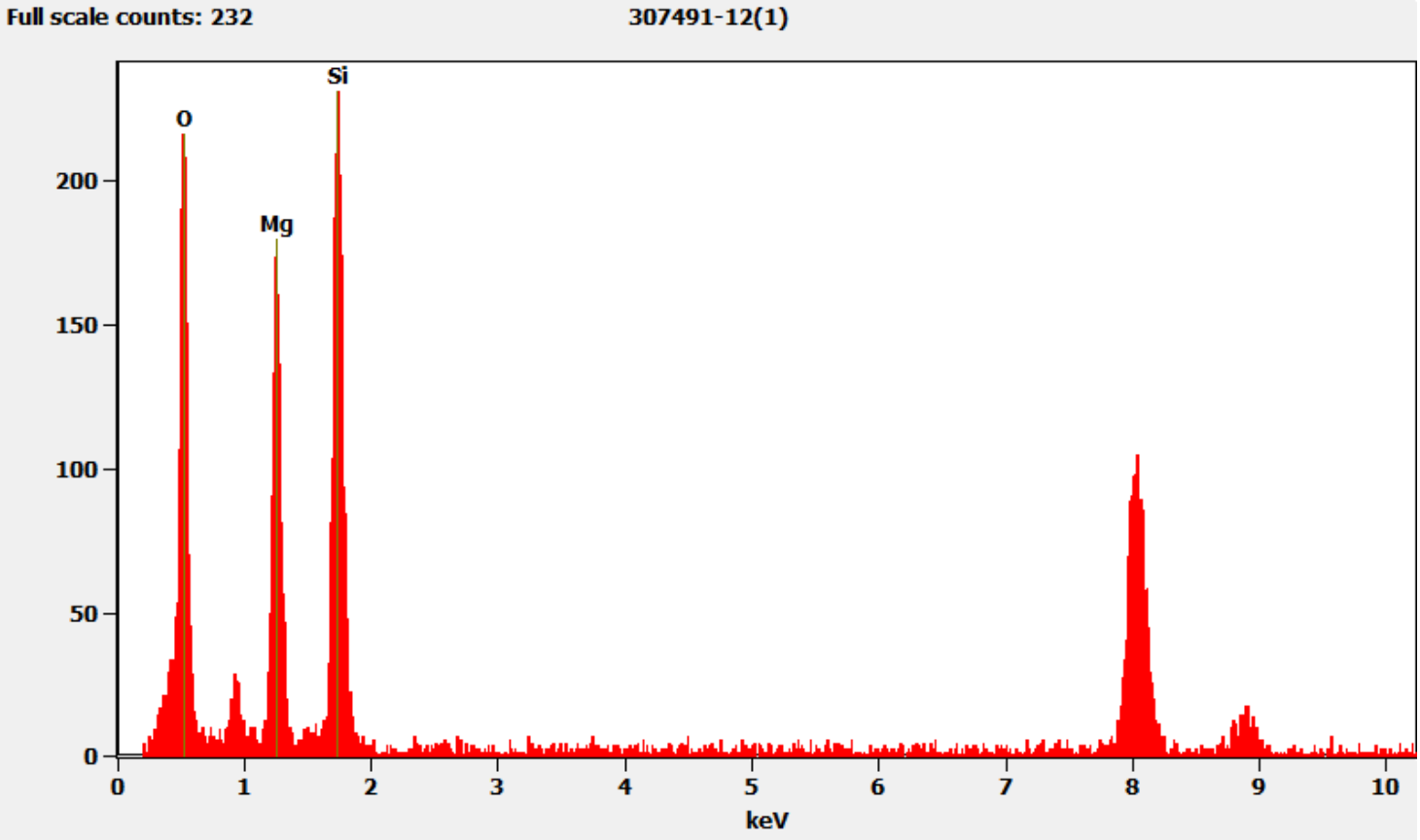
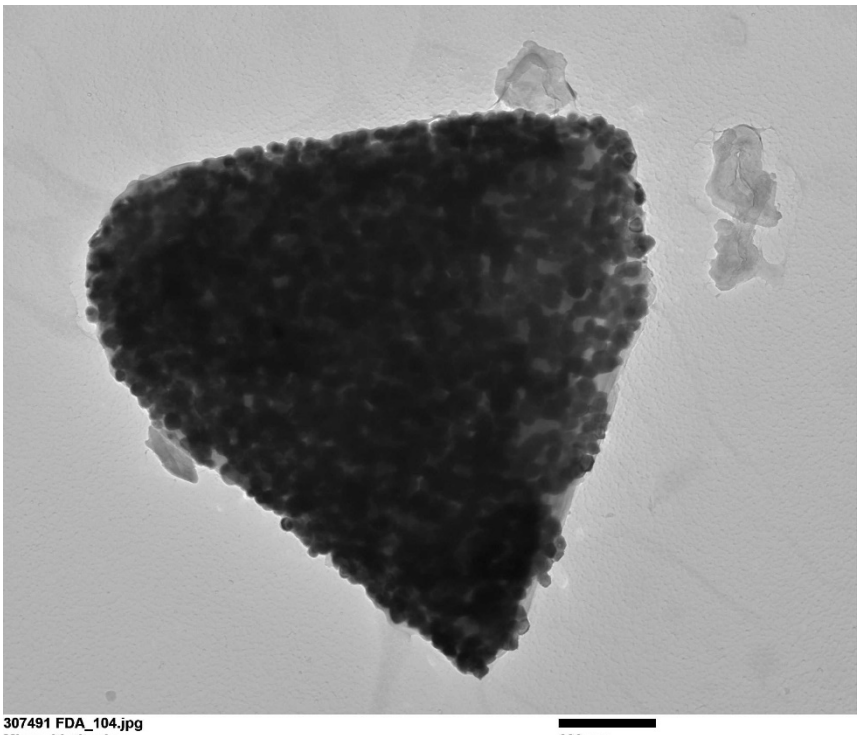
Chemistry from the talc particle pictured above



Mica particle with titanium particles adhered to it from 307491-12

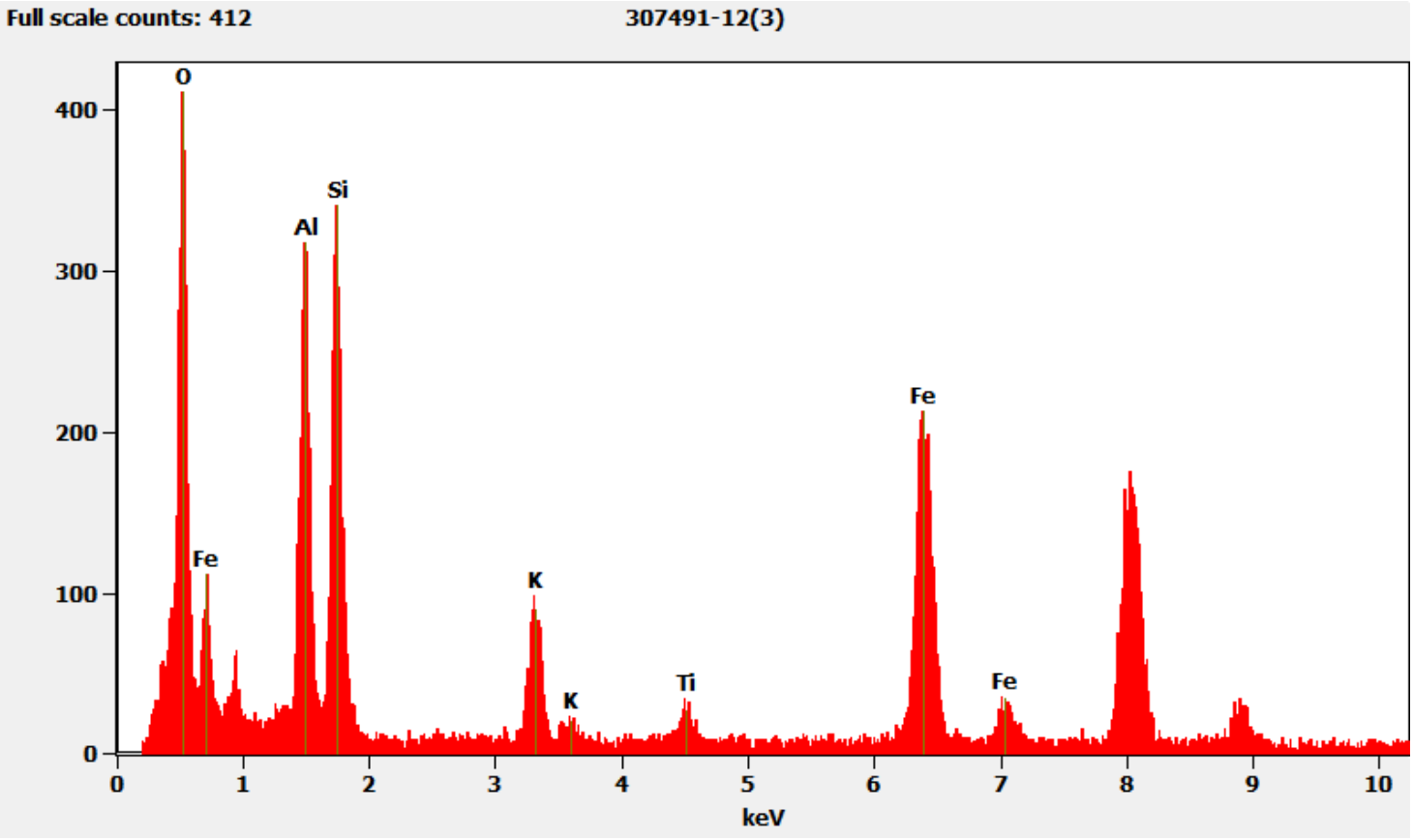


Diffraction pattern for the mica with titanium particle pictured above

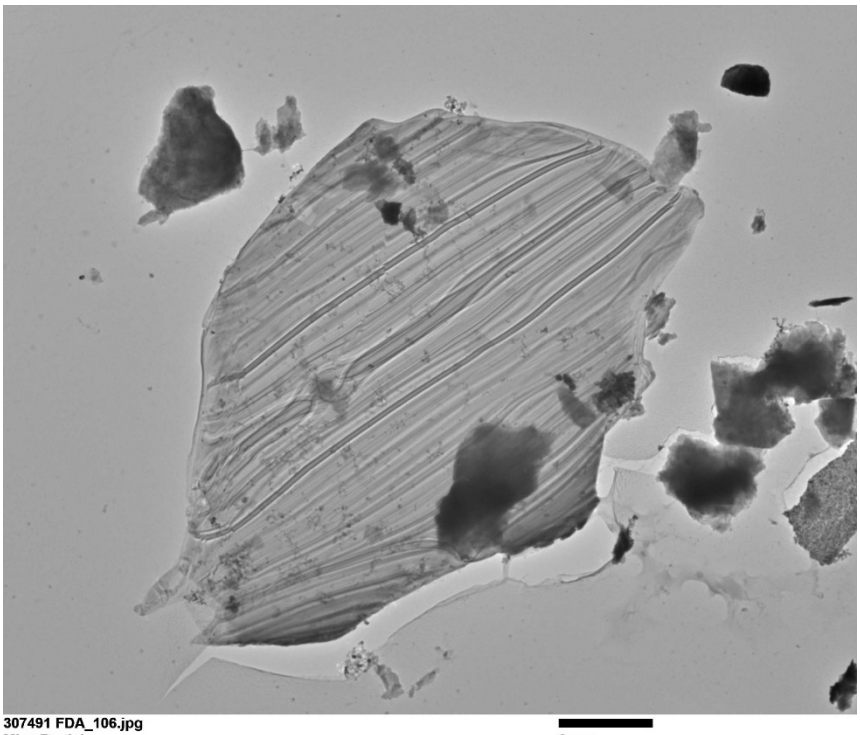
307491 FDA_105.jpg
Mica with titanium Diffraction
13:41 6/16/2019
TEM Mode: Diffraction
Microscopist: MG
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry for the mica with titanium particle pictured above



Mica particle from 307491-12



307491 FDA_106.jpg
Mica Particle
Cal: 0.007349 µm/pix
13:45 6/16/2019
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

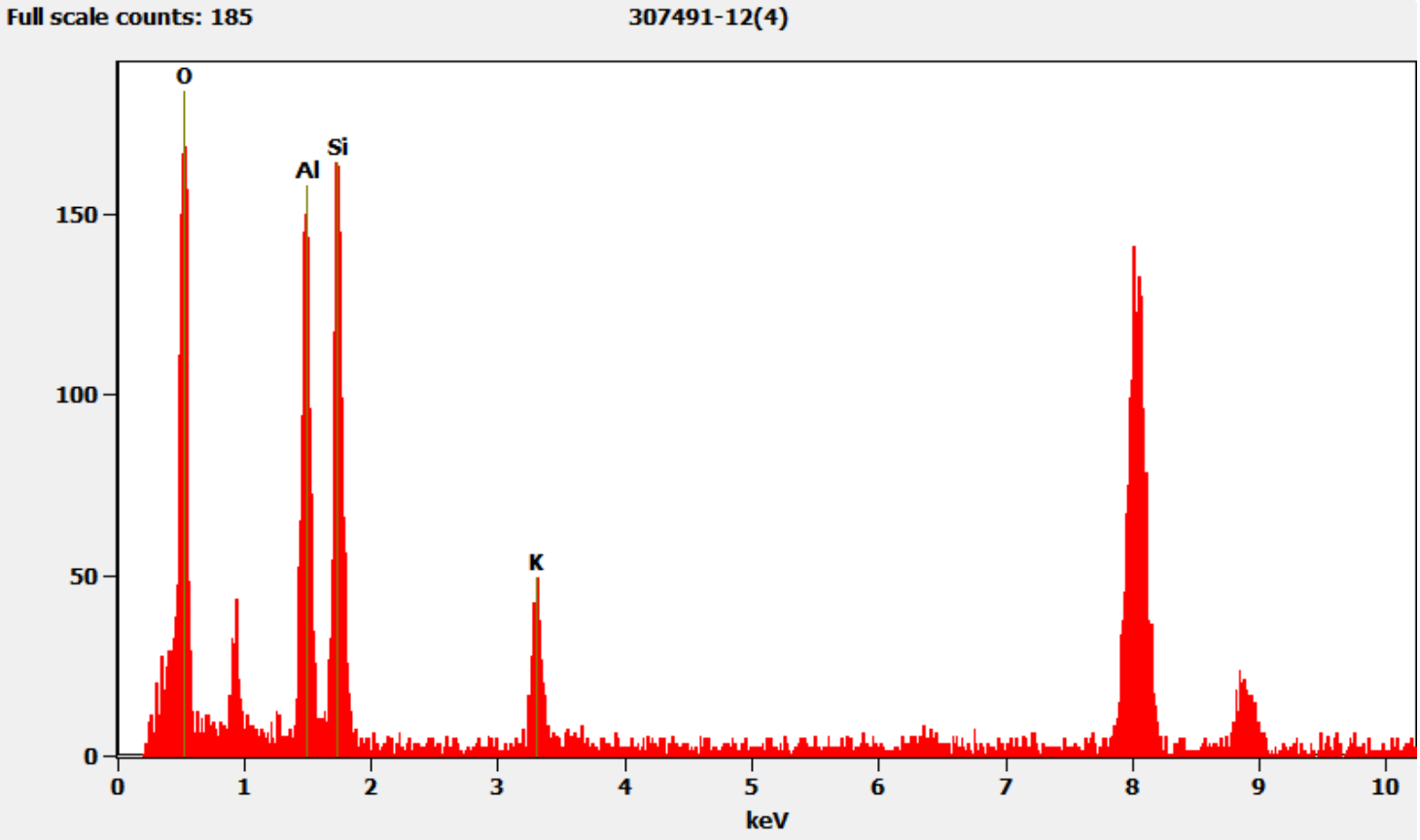
2 µm
HV=100kV
Direct Mag: 1400 x
AMA Analytical Services, Inc

Diffraction pattern for the mica particle pictured above

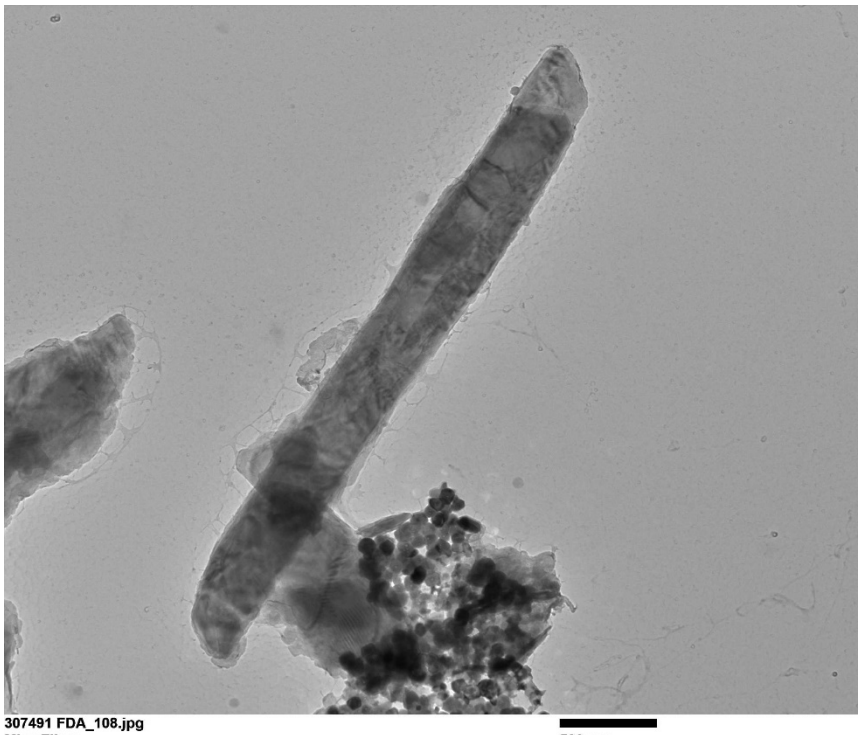
307491 FDA_107.jpg
Mica Particle Diffraction
13:46 6/16/2019
TEM Mode: Diffraction
Microscopist: MG
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry for the mica particle pictured above



Mica fiber from 307491-12



Diffraction pattern for the mica fiber picture above

307491 FDA_109.jpg
Mica Fiber Diff
14:08 6/16/2019
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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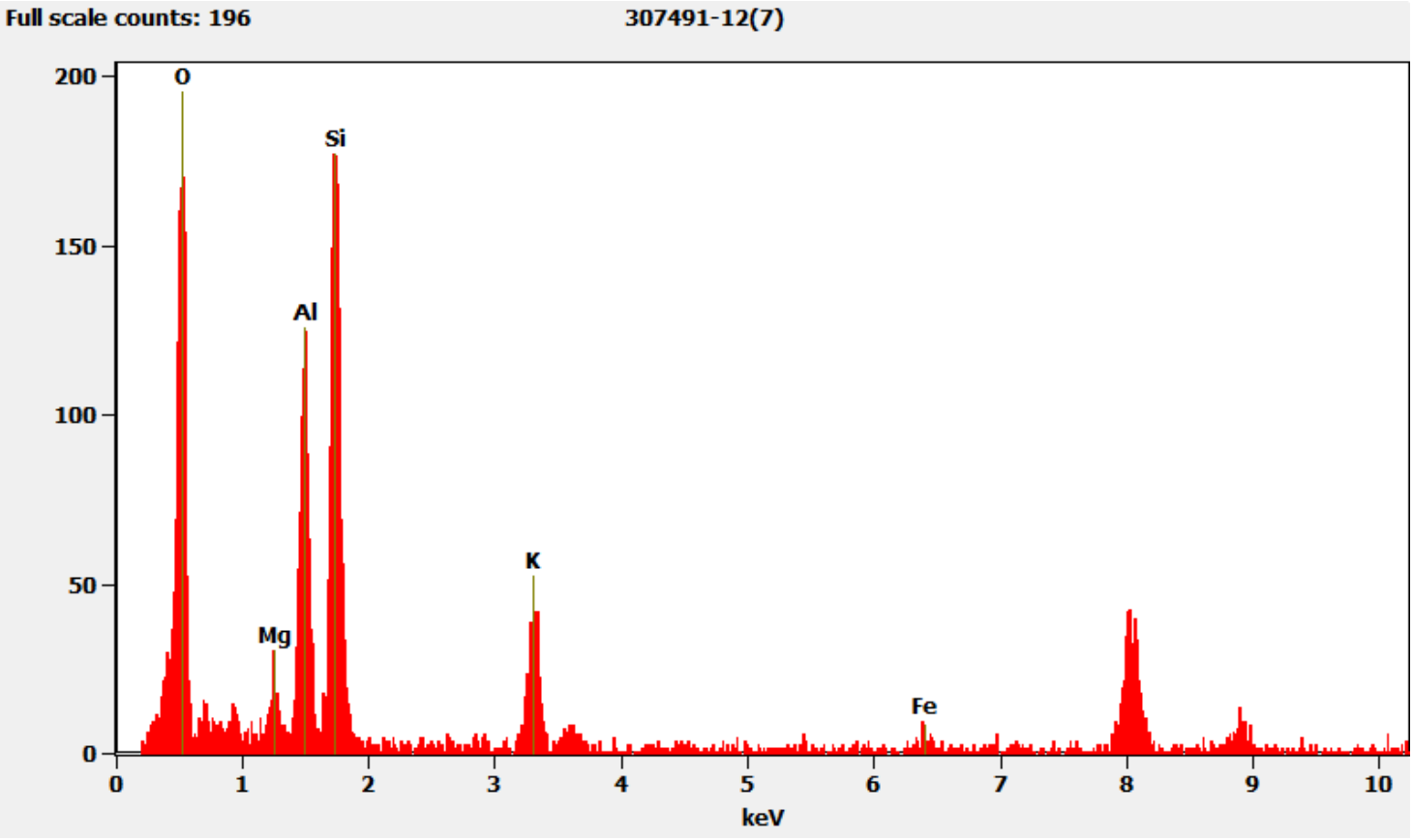
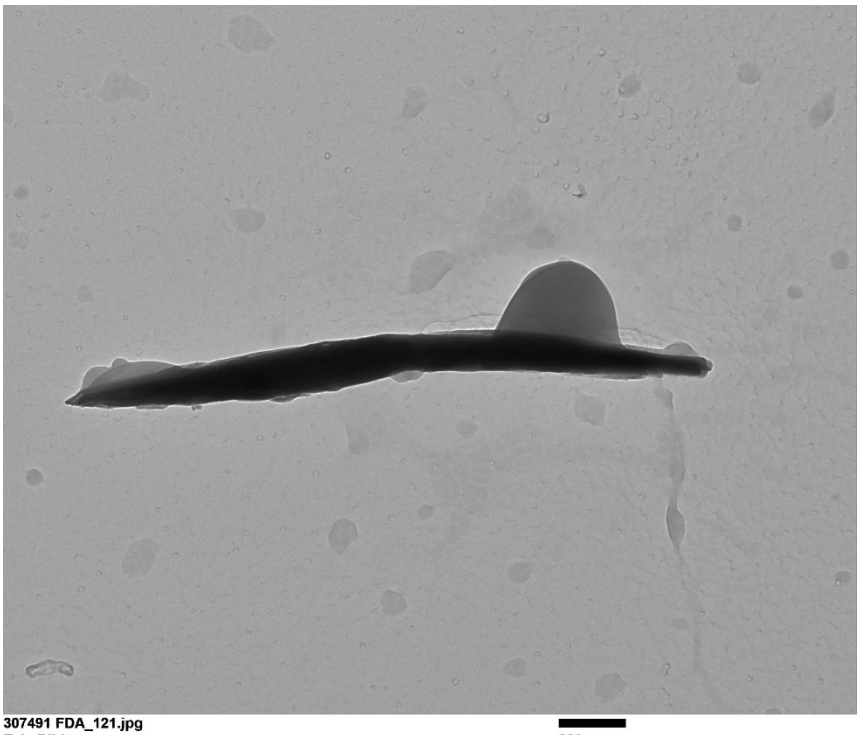
Chemistry for the mica fiber pictured above



Talc ribbon from 307491-12



307491 FDA_121.jpg
Talc Ribbon
Cal: 0.001029 µm/pix
15:27 6/16/2019
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 talc ribbon pictured above

307491 FDA_122.jpg
Talc Ribbon Diff
15:28 6/16/2019
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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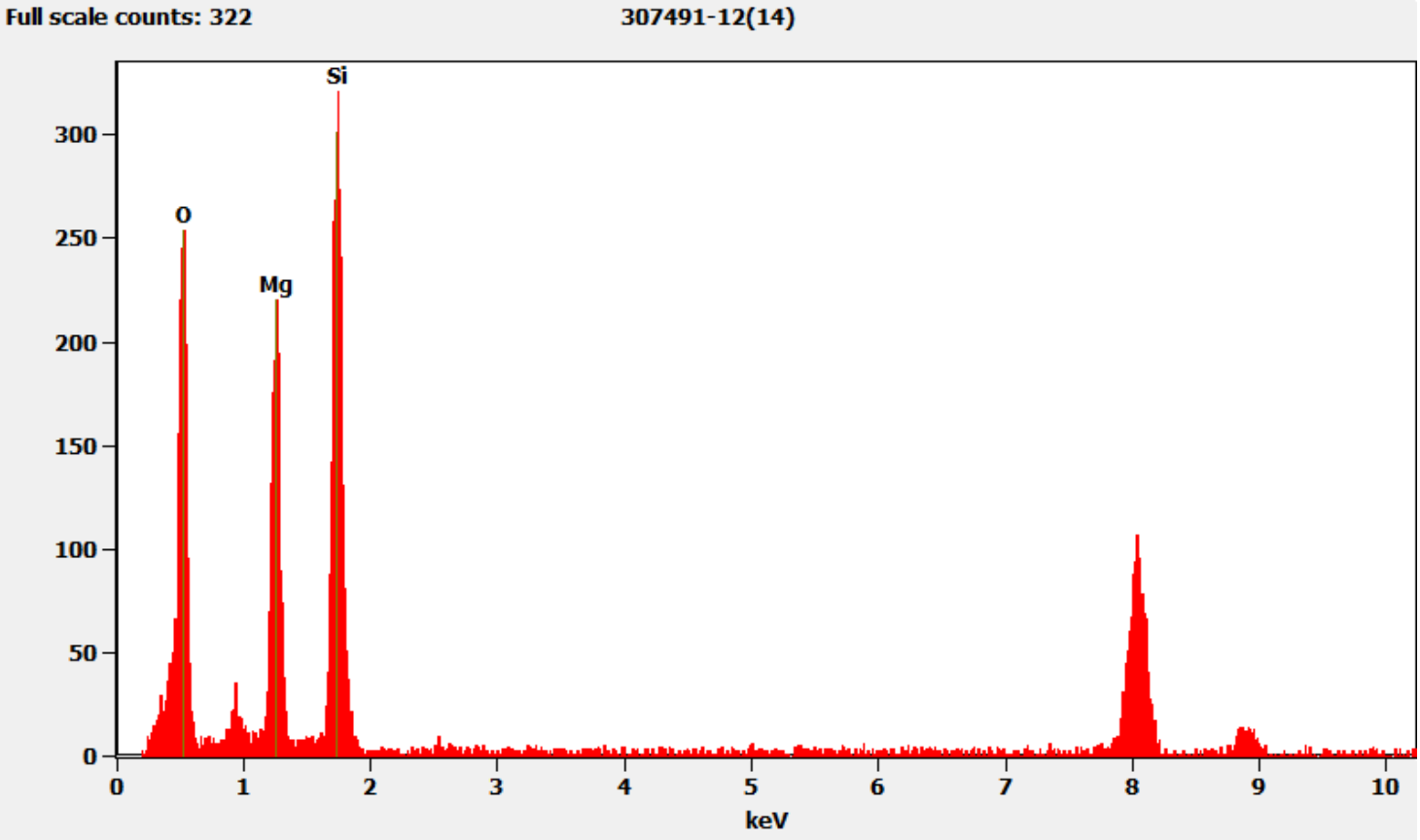
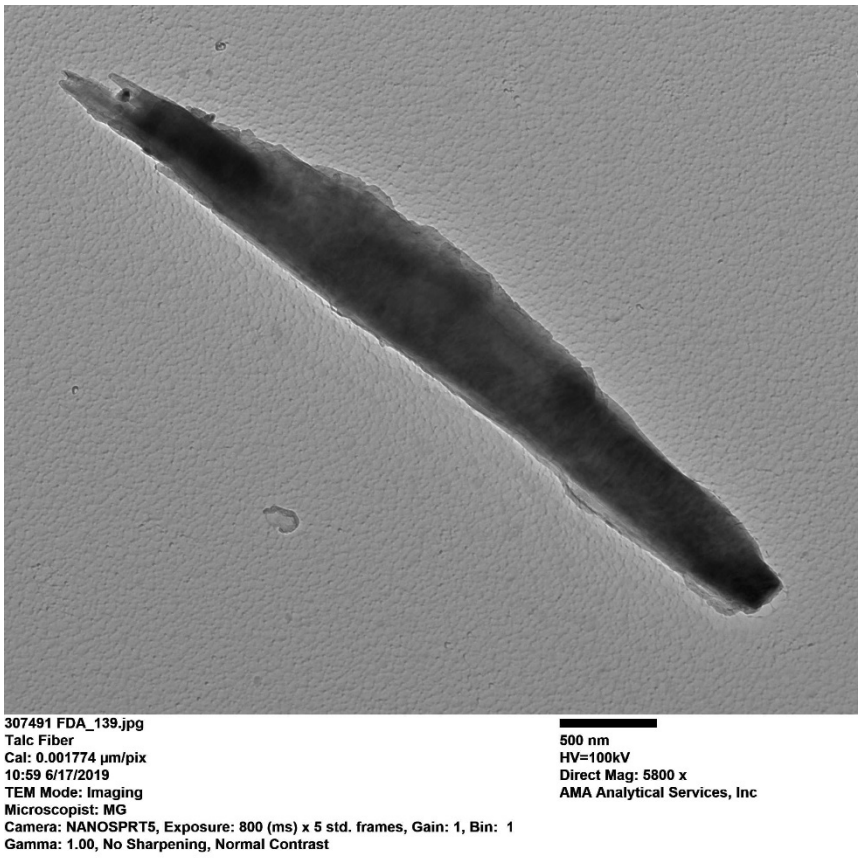
Chemistry from the talc ribbon pictured above



Talc fiber from 307491-12



Diffraction pattern from the talc fiber pictured above

307491 FDA_140.jpg
Talc Fiber Diff
10:59 6/17/2019
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/Å)
HV=100kV
Cam Len: 0.2200 m
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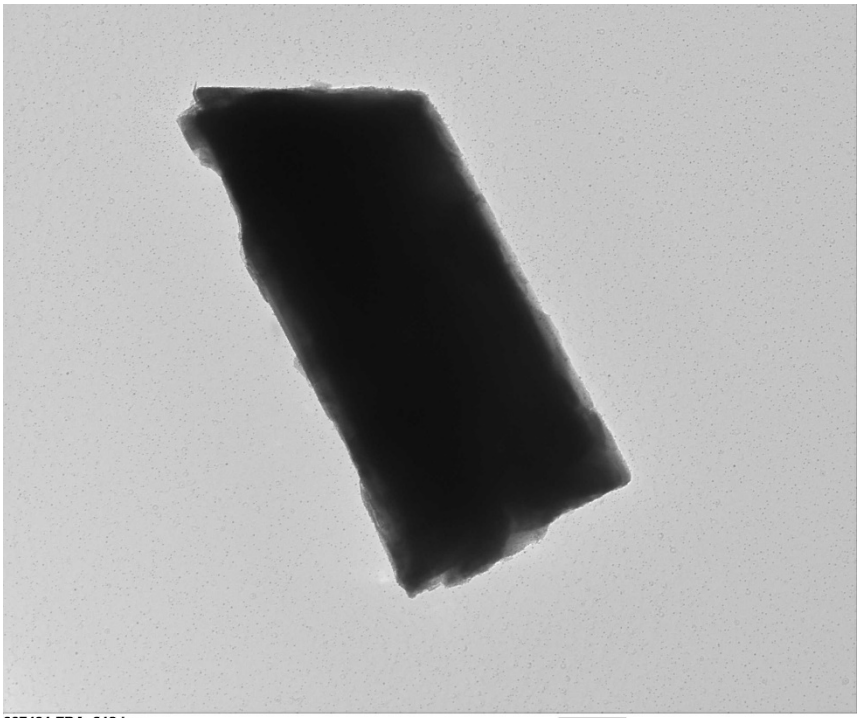
Tremolite particle from 307491-12.

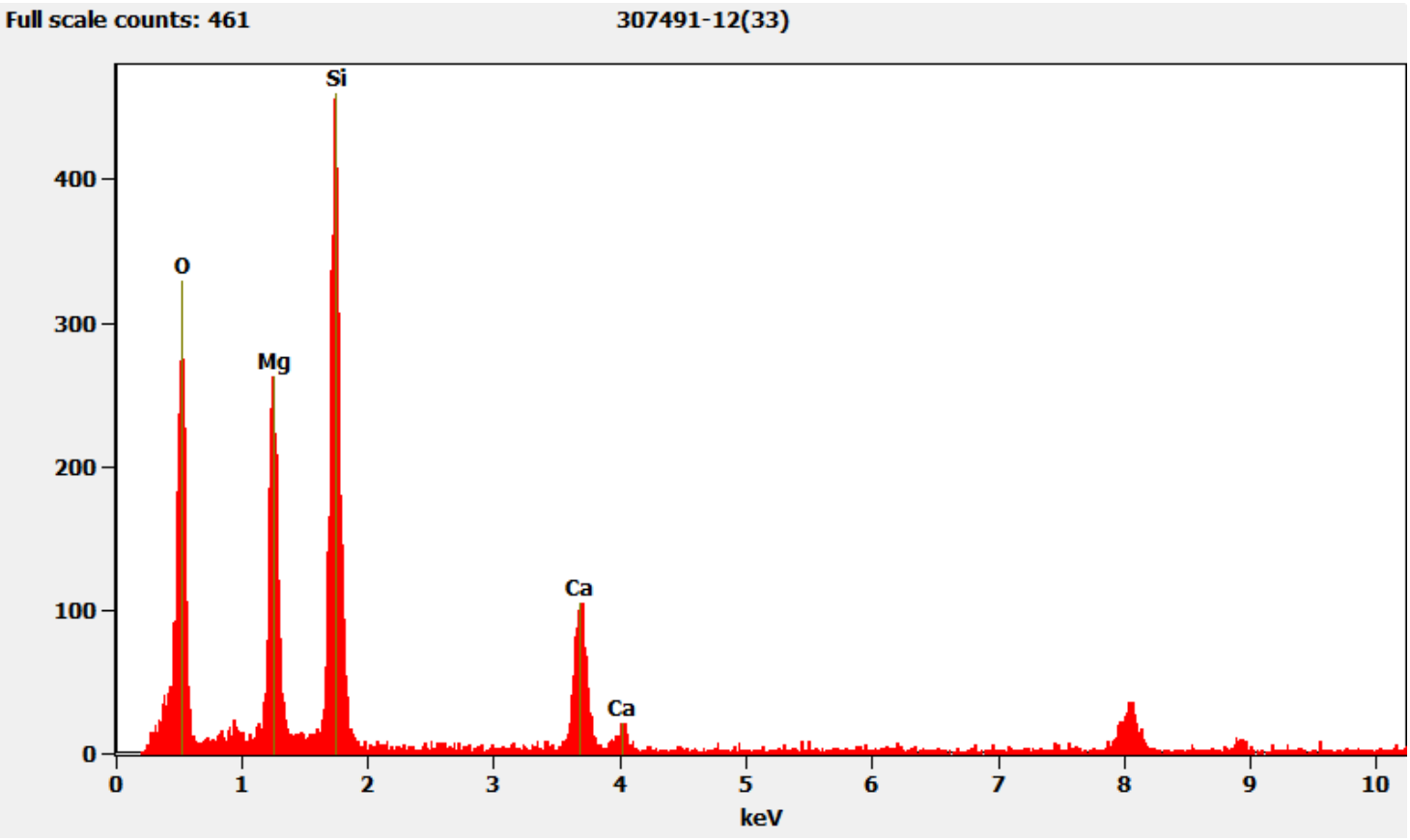


307491 FDA_218.jpg
Tremolite 19
Cal: 0.001029 µm/pix
11:25 7/2/2019
TEM Mode: Imaging
Microscopist: MG
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

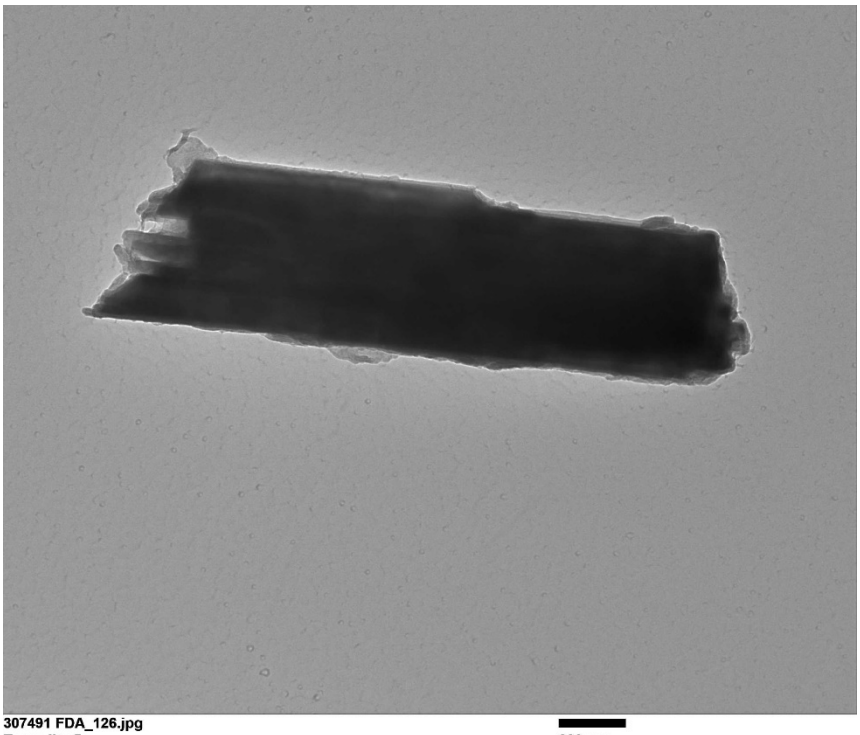
200 nm
HV=100kV
Direct Mag: 10000 x
AMA Analytical Services, Inc

Diffraction pattern from the tremolite particle pictured above

Chemistry from the tremolite particle pictured above



Tremolite particle from 307491-12



307491 FDA_126.jpg
Tremolite 5
Cal: 0.001029 µm/pix
15:49 6/16/2019
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
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Diffraction pattern from the tremolite particle pictured above

307491 FDA_125.jpg
Tremolite 5 Diffraction
15:48 6/16/2019
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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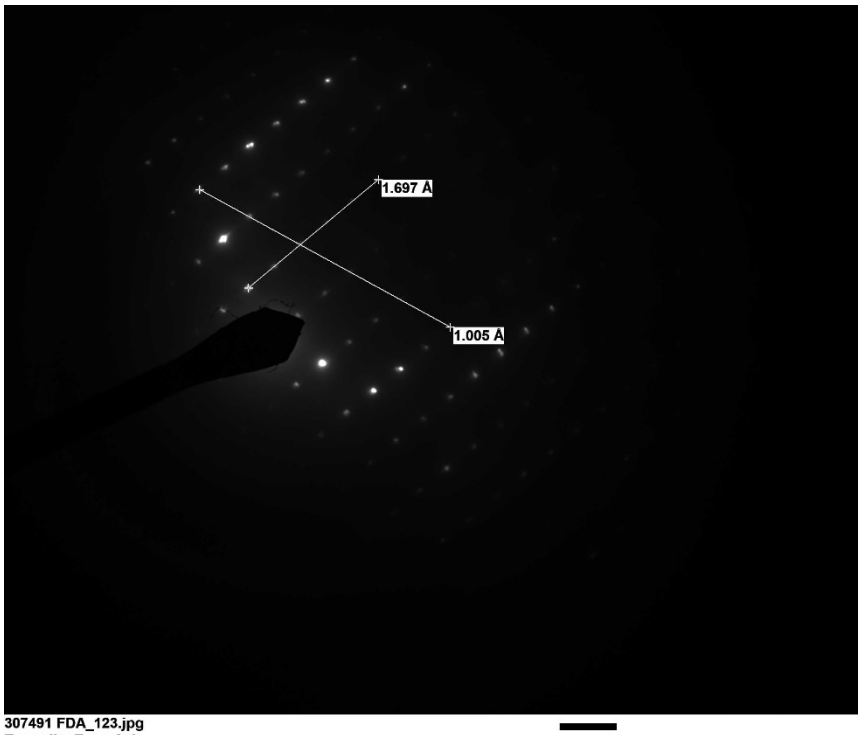
Zone access diffraction from the tremolite particle pictured above



307491 FDA_123.jpg
Tremolite Zone Axis
[1 -1 0]
15:35 6/16/2019
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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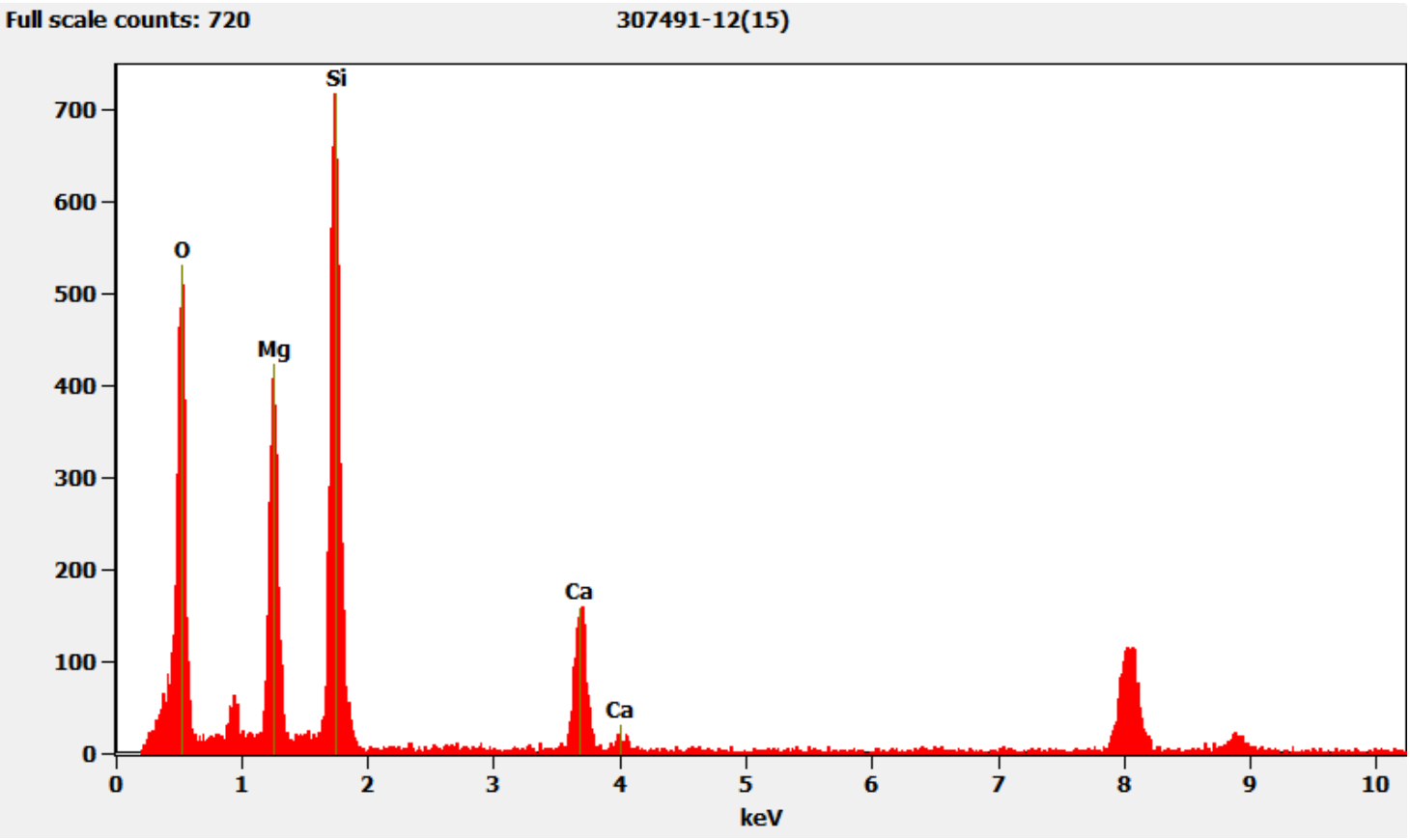
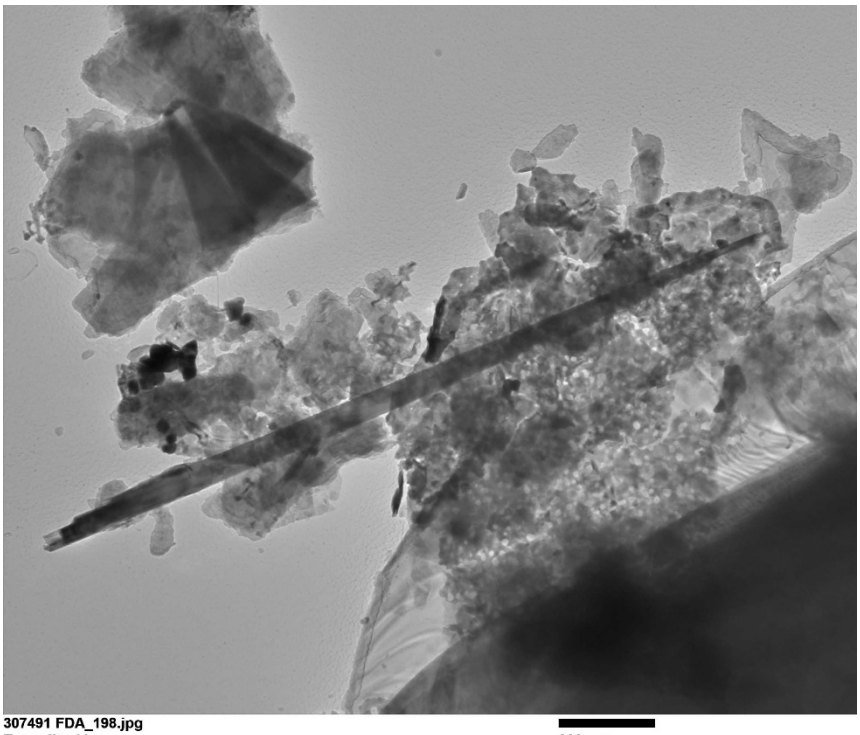
Chemistry from the tremolite particle pictured above



Tremolite particle from 307491-12



307491 FDA_198.jpg
Tremolite 11
Cal: 0.002858 µm/pix
09:52 7/2/2019
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

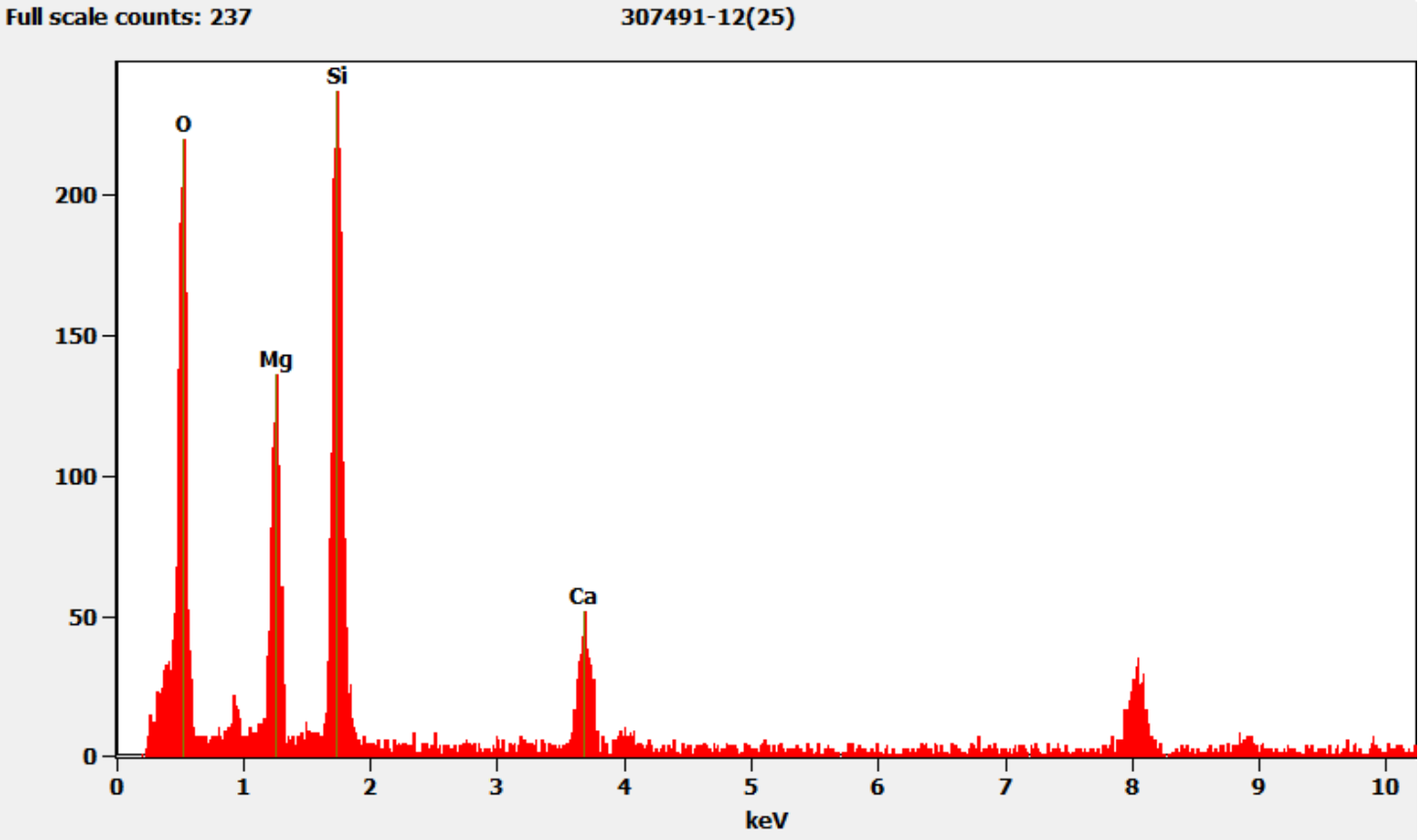
800 nm
HV=100kV
Direct Mag: 3600 x
AMA Analytical Services, Inc

Diffraction pattern from the tremolite particle pictured above

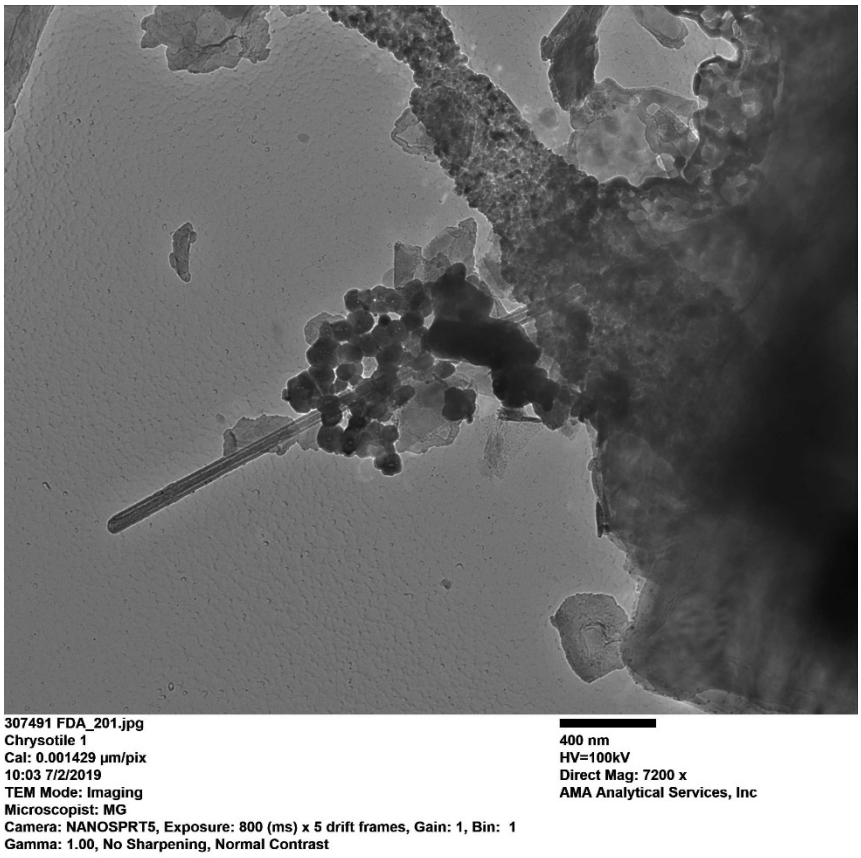
307491 FDA_199.jpg
Tremolite 11
09:54 7/2/2019
TEM Mode: Diffraction
Microscopist: MG
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from the tremolite particle pictured above



Chrysotile fiber from 307491-12



Diffraction pattern from the chrysotile fiber pictured above

307491 FDA_200.jpg
Chrysotile 1
10:01 7/2/2019
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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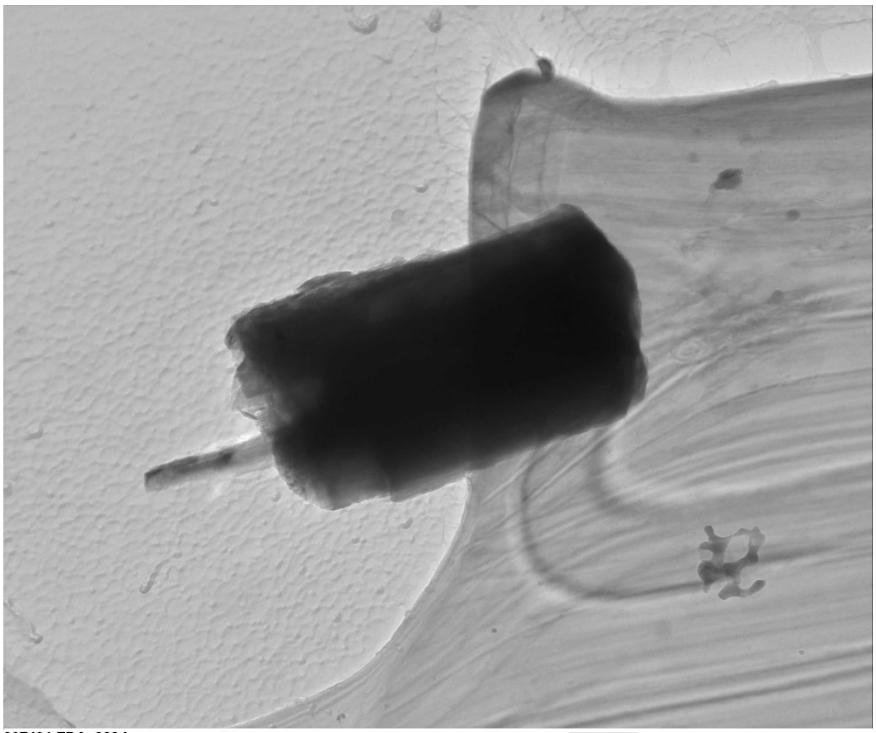
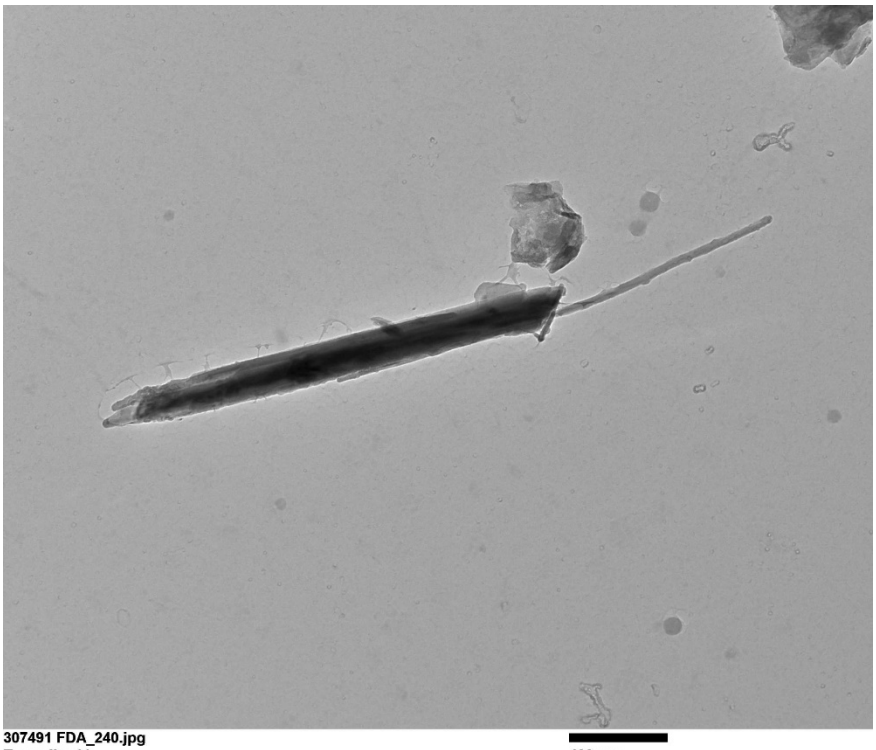
Below are additional photos of the tremolite particles counted from aliquots 12A and 12B



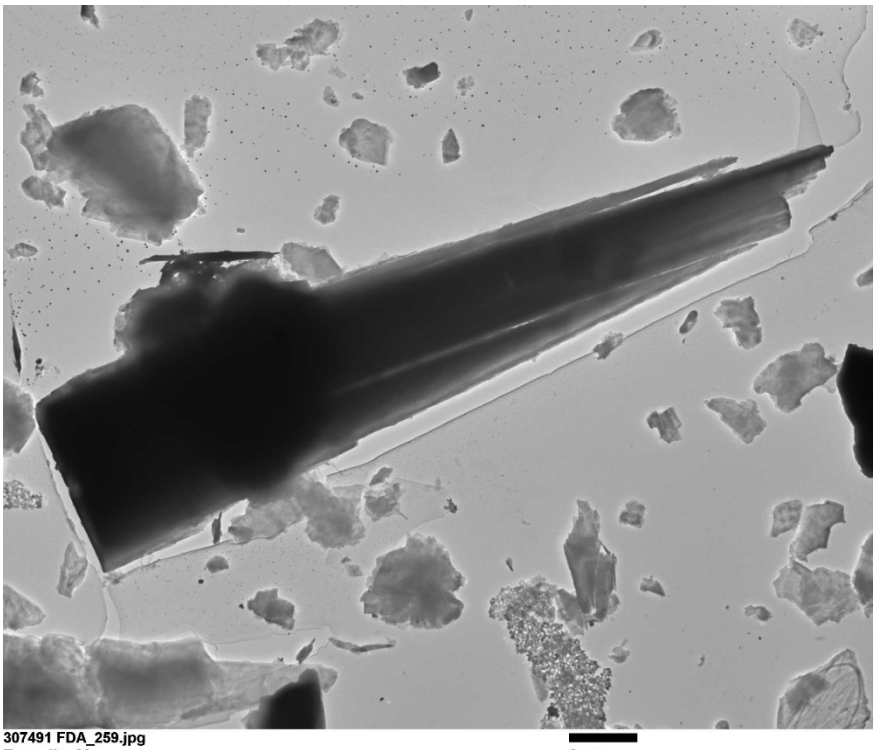
307491 FDA_228.jpg
Tremolite 5
Cal: 0.001029 µm/pix
16:14 7/2/2019
TEM Mode: Imaging
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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



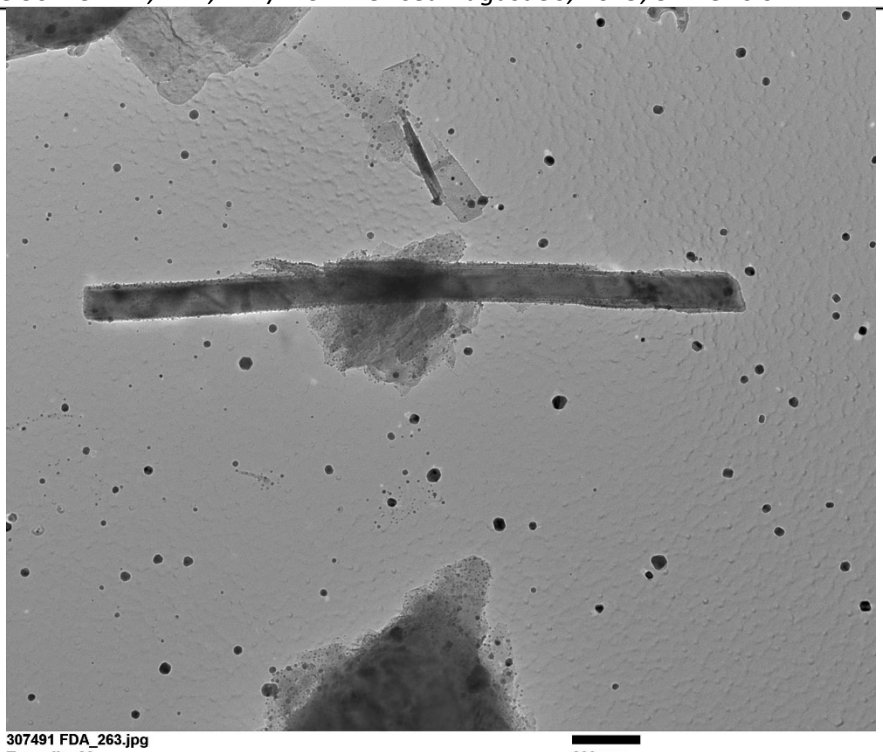
307491 FDA_240.jpg
Tremolite 11
Cal: 0.001429 µm/pix
09:51 7/3/2019
TEM Mode: Imaging
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 std. frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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



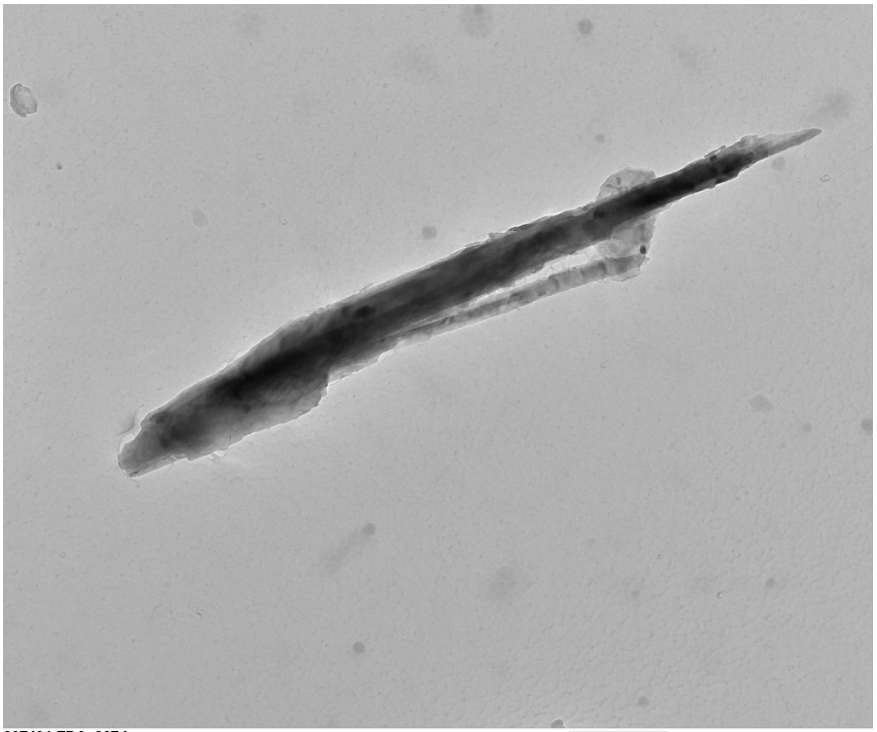
307491 FDA_259.jpg
Tremolite 20
Cal: 0.010289 µm/pix
09:34 7/7/2019
TEM Mode: Imaging
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

2 µm
HV=100kV
Direct Mag: 1000 x
AMA Analytical Services, Inc



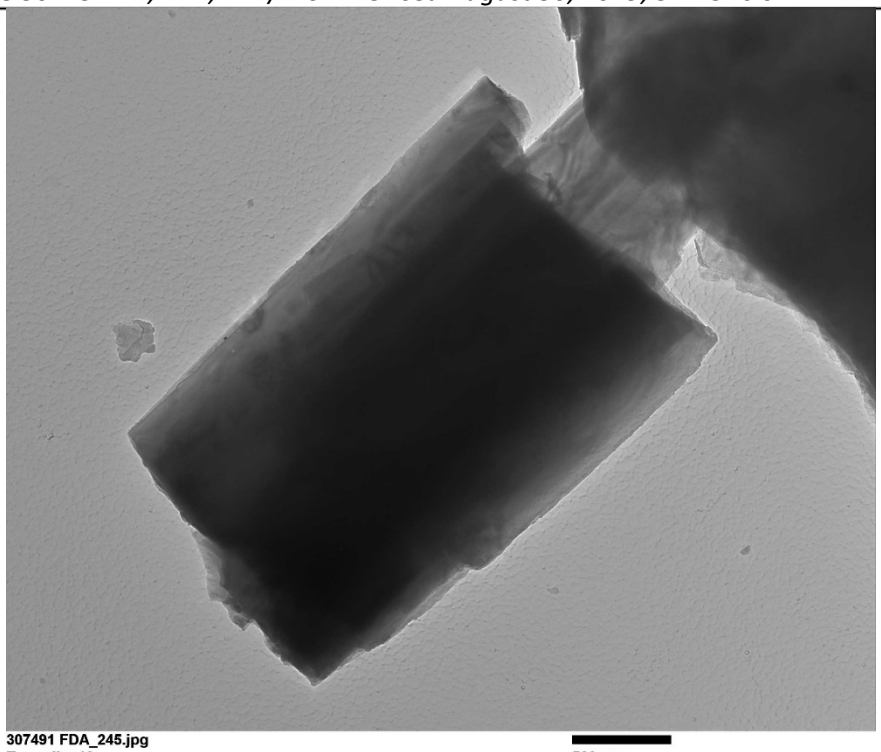
307491 FDA_263.jpg
Tremolite 22
Cal: 0.001029 µm/pix
09:44 7/7/2019
TEM Mode: Imaging
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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



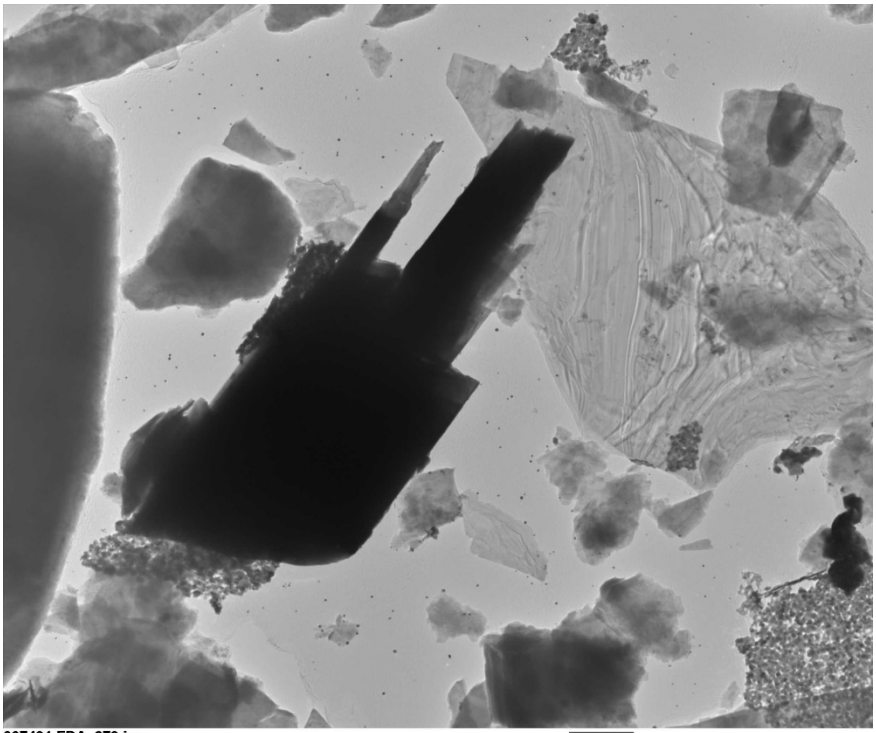
307491 FDA_267.jpg
Tremolite 24
Cal: 0.001429 µm/pix
09:58 7/7/2019
TEM Mode: Imaging
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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



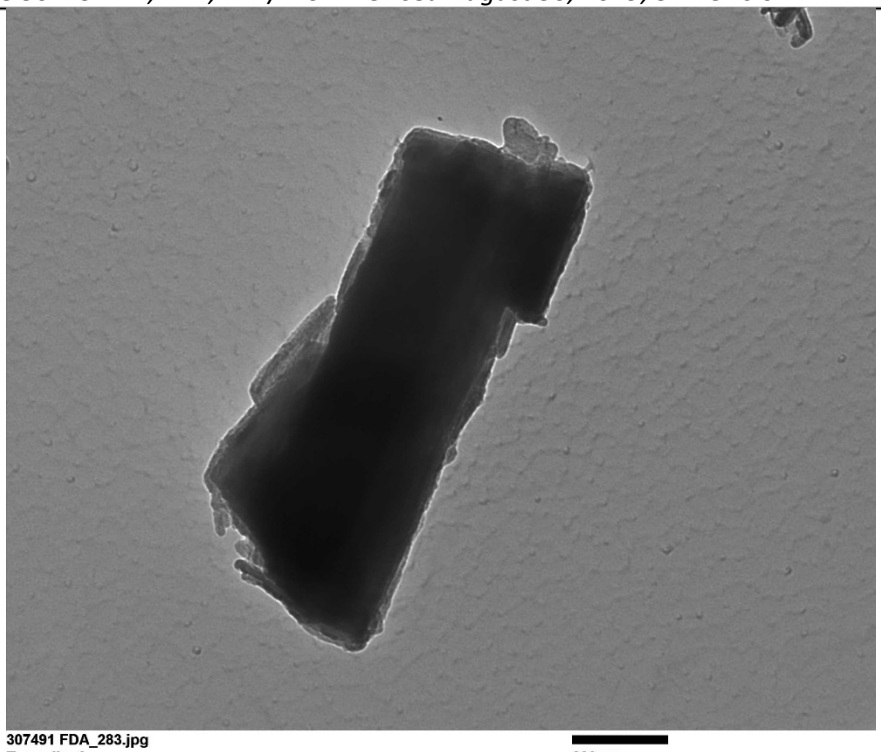
307491 FDA_245.jpg
Tremolite 13
Cal: 0.001774 µm/pix
10:16 7/3/2019
TEM Mode: Imaging
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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



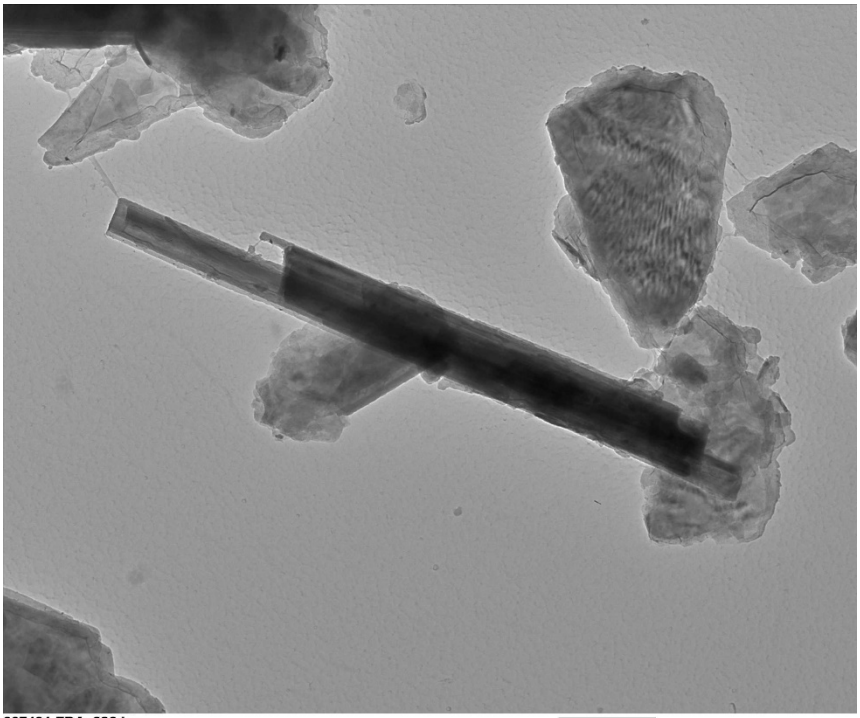
307491 FDA_279.jpg
Tremolite 4
Cal: 0.005415 µm/pix
11:56 7/7/2019
TEM Mode: Imaging
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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



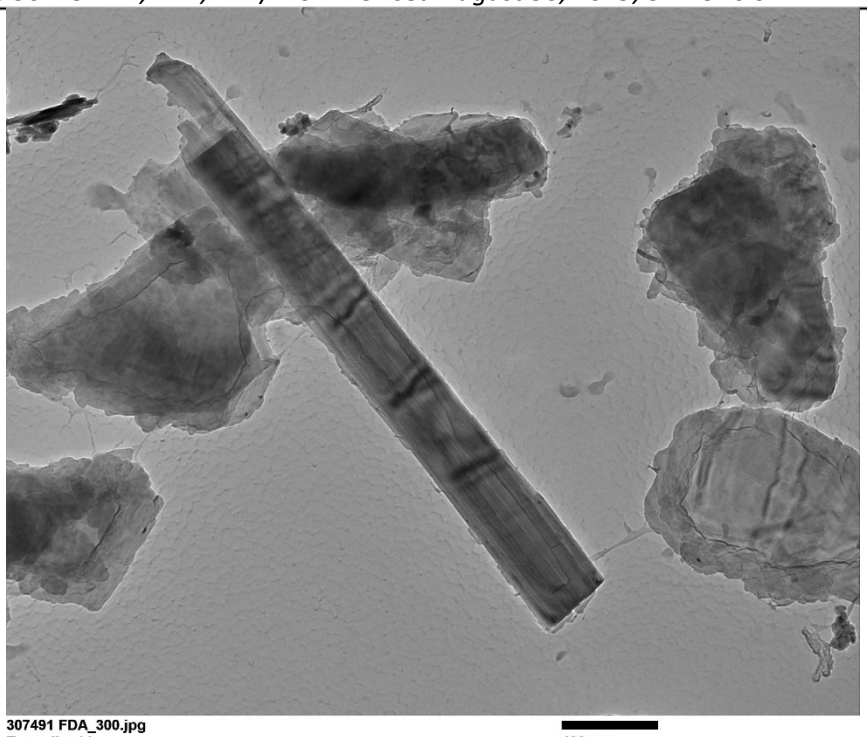
307491 FDA_283.jpg
Tremolite 6
Cal: 0.734921 nm/pix
12:06 7/7/2019
TEM Mode: Imaging
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

200 nm
HV=100kV
Direct Mag: 14000 x
AMA Analytical Services, Inc



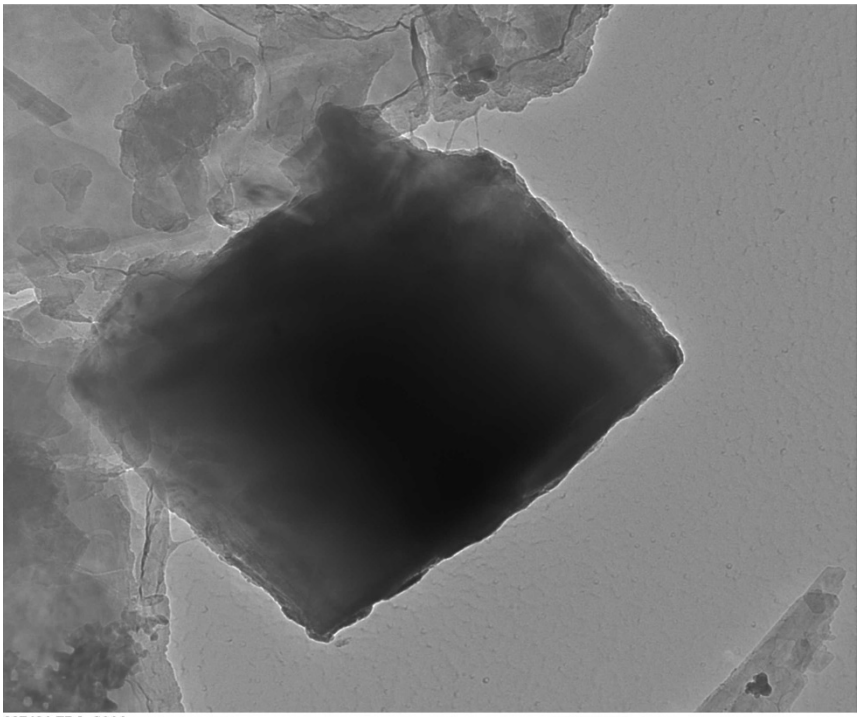
307491 FDA_286.jpg
Tremolite 7
Cal: 0.001774 µm/pix
12:14 7/7/2019
TEM Mode: Imaging
Microscopist: MG
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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



307491 FDA_300.jpg
Tremolite 14
Cal: 0.001429 µm/pix
13:33 7/7/2019
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



307491 FDA_314.jpg
Tremolite 21
Cal: 0.001029 µm/pix
15:44 7/17/2019
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

307491 FDA_316.jpg
Tremolite 22
Cal: 0.001429 µm/pix
15:50 7/17/2019
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

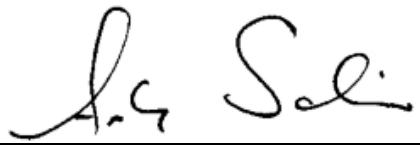
QC Discussion:
During preparation, one blank control sample and one reference control sample were prepared. These samples were prepared alongside the customer samples. The blank sample was prepared using Sigma-Aldrich Talc Powder, <10 micron. No asbestos was detected on the blank sample. The reference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

Our LIMS randomly selects samples for additional replicate and duplicate QC. 307491-12, 12A, and 12B/D-52 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 Chart for Peerawut Chaikeenee for samples analyzed 1/1/2019 through 6/30/2019.
7) Replicate and Duplicate QC Chart for Michael Greenberg for samples analyzed 1/1/2019 through 7/17/2019.
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.



7/24/2019

Andreas Saldivar — Date
Laboratory Director