AMA Analytical Services, Inc.
Focused On Results.

CERTIFICATE OF ANALYSIS

Chain of Custody: 308006
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: 4th Group - 15 Samples
Job Number: CLIN 1 - Task 3
PO Number: HHSF223201810337P

Date Submitted: 7/24/2019
Date Analyzed: 8/20/2019-9/18/2019
Report Date: 10/16/2019
Date Sampled: Not Provided
Person Submitting: Goran Periz
Revised: 11/8/2019, Revision #2

SUMMARY OF ANALYSIS

AMA Sample ID	Client Sample ID	TEM LOD Using ASTM D5756 Mass Calculation	TEM LOQ Using ASTM D5756 Mass Calculation	% Tremolite by TEM Using ASTM D5756 Mass Calculation	% Chrysotile by TEM Using ASTM D5756 Mass Calculation	% Total Tremolite & Chrysotile by TEM Using ASTM D5756 Mass Calculation	% Asbestos by PLM	% Organics	% Acid Soluable	% Other	Comments
308006-1	D-53	0.00000351%	0.00001405%	ND	ND	ND	ND	7.9%	10.6%	81.5%	
308006-1A	D-53	0.00000251%	0.00001006%	ND	ND	ND	ND	7.9%	6.2%	85.9%	
308006-1B	D-53	0.00000210%	0.00000840%	ND	ND	ND	ND	7.9%	9.0%	83.1%	
308006-2	D-54	0.00000150%	0.00000598%	ND	ND	ND	ND	12.3%	4.6%	83.1%	
308006-2A	D-54	0.00000215%	0.00004409%	ND	< 0.00004%	< 0.00004%	ND	12.2%	3.7%	84.1%	
308006-2B	D-54	0.00000245%	0.00000981%	ND	ND	ND	ND	12.6%	6.6%	80.8%	
308006-3	D-55	0.00000170%	0.00000679%	ND	ND	ND	ND	14.1%	6.3%	79.6%	
308006-3A	D-55	0.00000157%	0.00000627%	ND	ND	ND	ND	15.7%	10.2%	74.1%	
308006-3B	D-55	0.00000148%	0.00000593%	ND	ND	ND	ND	16.2%	11.4%	72.4%	
308006-4	D-56	0.00000170%	0.00000680%	ND	ND	ND	ND	16.2%	6.4%	77.4%	
308006-4A	D-56	0.00000163%	0.00012695%	ND	< 0.00013%	< 0.00013%	ND	16.3%	8.7%	75.1%	
308006-4B	D-56	0.00000211%	0.00000843%	ND	ND	ND	ND	15.8%	6.3%	77.9%	
308006-5	D-57	0.00000287%	0.00001146%	ND	ND	ND	ND	2.6%	7.5%	89.9%	
308006-5A	D-57	0.00000221%	0.00000884%	ND	ND	ND	ND	2.6%	4.8%	92.6%	
308006-5B	D-57	0.00000173%	0.00000692%	ND	ND	ND	ND	2.5%	3.9%	92.5%	
308006-7	D-59	0.00000150%	0.00000599%	ND	ND	ND	ND	15.2%	6.6%	78.2%	
308006-7A	D-59	0.00000141%	0.00000562%	ND	ND	ND	ND	15.2%	3.9%	80.9%	
308006-7B	D-59	0.00000164%	0.00000655%	ND	ND	ND	ND	12.2%	6.6%	78.3%	
308006-8	D-60	0.00000208%	0.00000832%	ND	ND	ND	ND	1.3%	4.5%	94.1%	
308006-8A	D-60	0.00000161%	0.00000645%	ND	ND	ND	ND	1.4%	2.1%	96.6%	
308006-8B	D-60	0.00000177%	0.00000706%	ND	ND	ND	ND	1.4%	7.9%	90.7%	
308006-9	D-61	0.00000258%	0.00001032%	ND	ND	ND	ND	3.7%	10.8%	85.5%	
308006-9A	D-61	0.00000220%	0.00000881%	ND	ND	ND	ND	3.6%	10.0%	86.3%	
308006-9B	D-61	0.00000214%	0.00002277%	ND	< 0.00002%	< 0.00002%	ND	3.7%	16.2%	80.1%	
308006-10	D-62	0.00000165%	0.00000661%	ND	ND	ND	ND	22.8%	9.3%	67.9%	
308006-10A	D-62	0.00000154%	0.00000615%	ND	ND	ND	ND	22.7%	8.0%	69.3%	
308006-10B	D-62	0.00000178%	0.00000710%	ND	ND	ND	ND	22.7%	4.7%	72.6%	
308006-11	D-63	0.00000152%	0.00000609%	ND	ND	ND	ND	23.8%	12.5%	63.7%	
308006-11A	D-63	0.00000147%	0.00000589%	ND	ND	ND	ND	23.8%	10.2%	66.0%	
308006-11B	D-63	0.00000181%	0.00000723%	ND	ND	ND	ND	23.8%	5.9%	70.4%	
308006-12	D-64	0.00000236%	0.00000945%	ND	ND	ND	ND	4.8%	9.1%	86.1%	
308006-12A	D-64	0.00000345%	0.00001380%	ND	ND	ND	ND	4.8%	6.5%	88.8%	
308006-12B	D-64	0.00000273%	0.00001092%	ND	ND	ND	ND	4.7%	7.8%	87.5%	
308006-13	D-65	0.00000159%	0.00000635%	ND	ND	ND	ND	28.5%	3.1%	68.3%	
308006-13A	D-65	0.00000167%	0.00000668%	ND	ND	ND	ND	28.7%	4.6%	66.7%	
308006-13B	D-65	0.00000121%	0.00000484%	ND	ND	ND	ND	28.7%	6.5%	64.8%	
308006-14	D-66	0.00000177%	0.00000707%	ND	ND	ND	ND	14.7%	6.3%	79.0%	
308006-14A	D-66	0.00000222%	0.00000889%	ND	ND	ND	ND	14.7%	8.2%	77.1%	
308006-14B	D-66	0.00000142%	0.00000569%	ND	ND	ND	ND	14.8%	9.4%	75.8%	
308006-15	D-67	0.00000145%	0.00000580%	ND	ND	ND	ND	16.6%	13.6%	69.8%	
308006-15A	D-67	0.00000124%	0.00000495%	ND	ND	ND	ND	16.6%	14.1%	69.3%	
308006-15B	D-67	0.00000111%	0.00000445%	ND	ND	ND	ND	16.7%	15.3%	68.1%	

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Date Sampled: Not Provided
Person Submitting: Goran Periz
Revised: 11/8/2019, Revision #2

SUMMARY OF ANALYSIS

AMA Sample ID	Client Sample ID	TEM LOD Using ASTM D5756 Mass Calculation	TEM LOQ Using ASTM D5756 Mass Calculation	% Tremolite by TEM Using ASTM D5756 Mass Calculation	% Chrysotile by TEM Using ASTM D5756 Mass Calculation	% Total Tremolite & Chrysotile by TEM Using ASTM D5756 Mass Calculation	% Asbestos by PLM	% Organics	% Acid Soluable	% Other	Comments

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

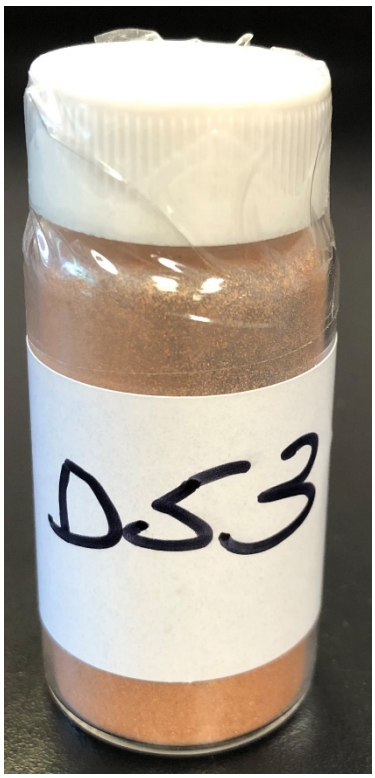
Analytical Method(s): PLM by Modified NY ELAP 198.6
TEM by Modified NY ELAP 198.4/ASTM D5756

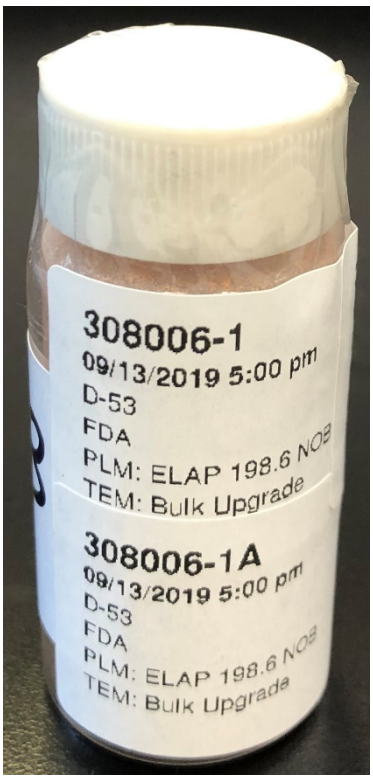
Analyst(s): **PLM** (b) (6)
TEM (b) (6)

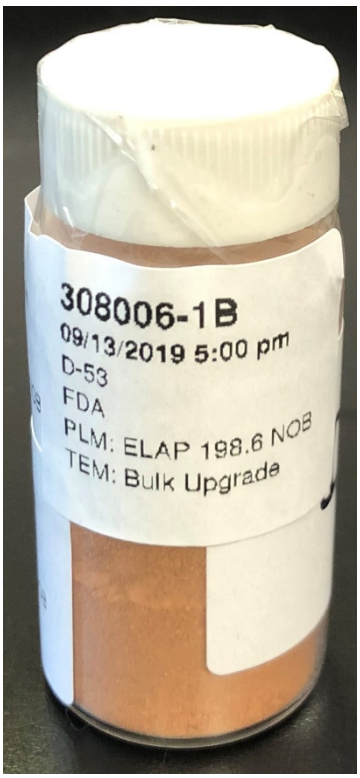
Technical Director: Andreas Saldivar

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

308006-1, 1A, 1B/D-53







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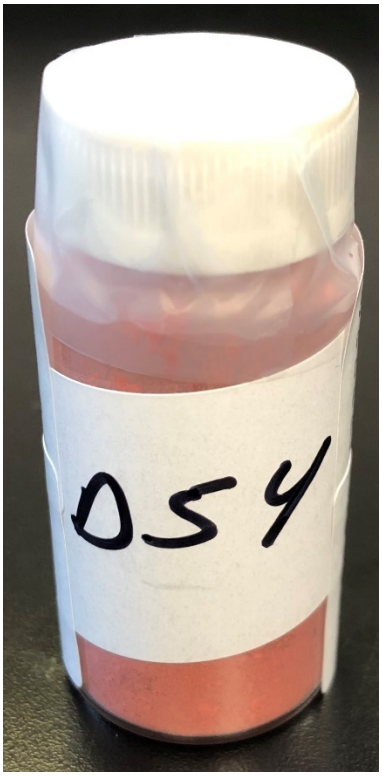

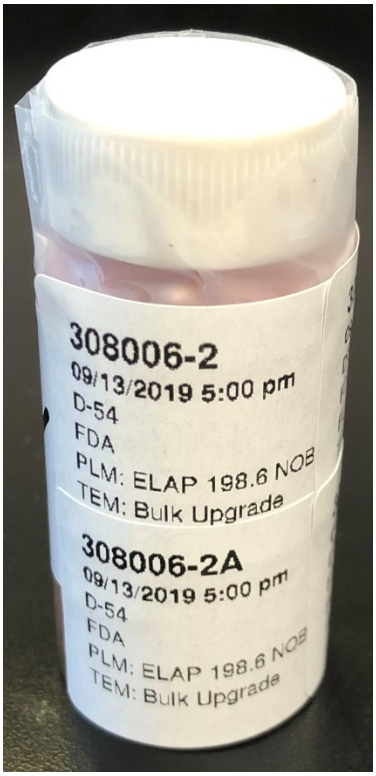

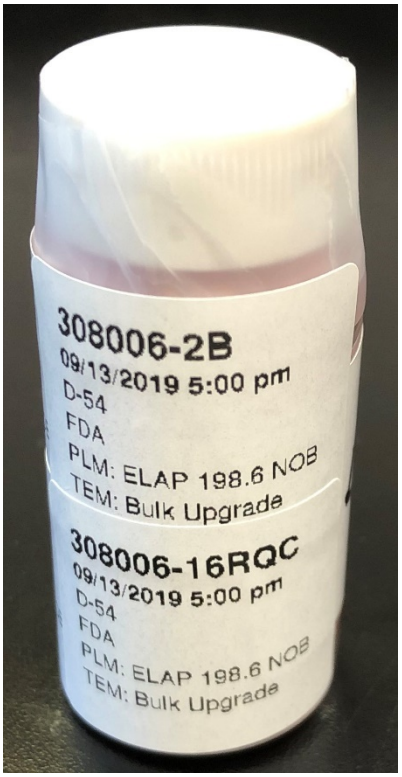

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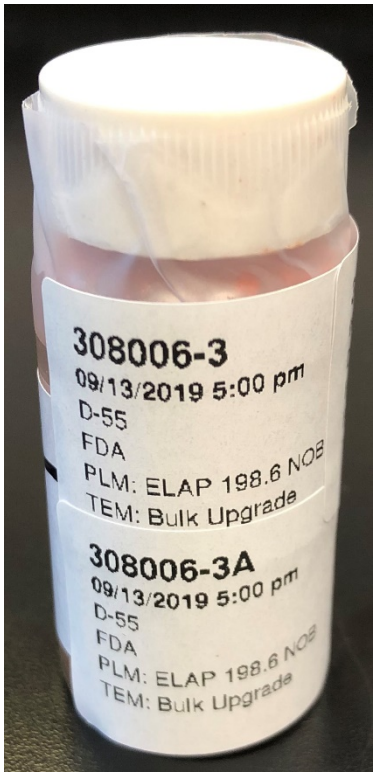

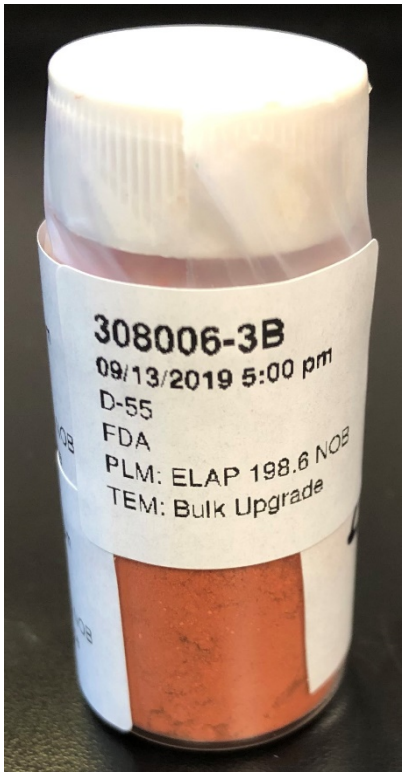

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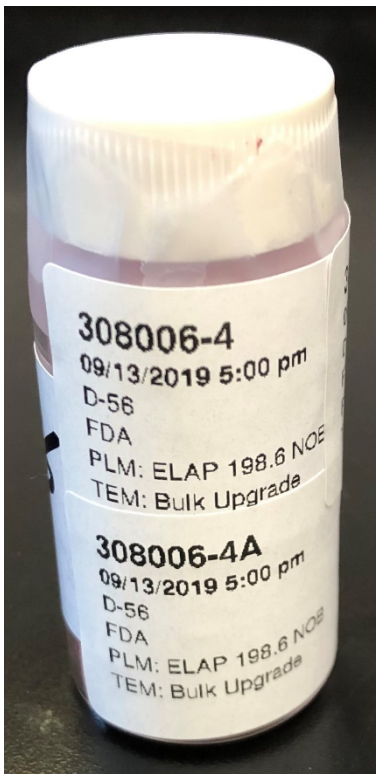

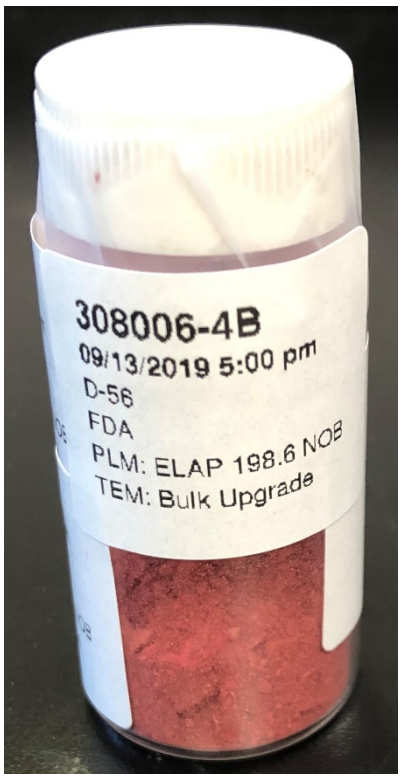

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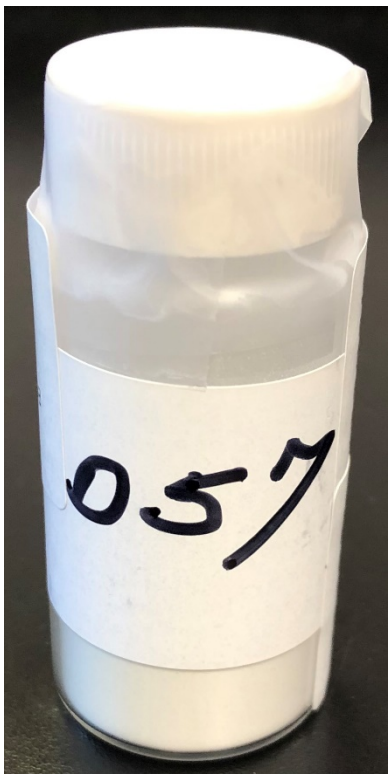
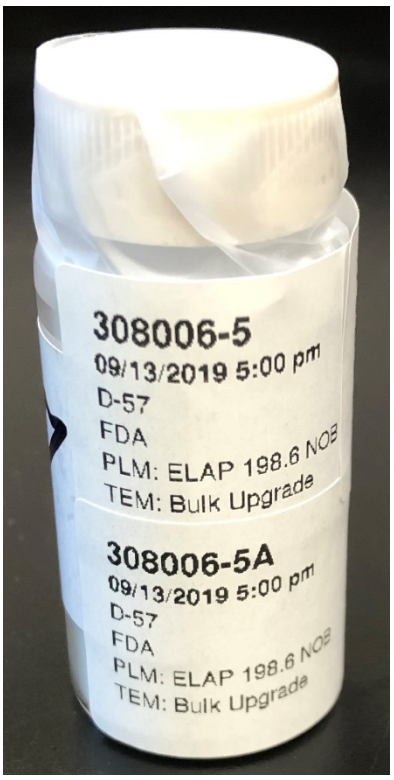
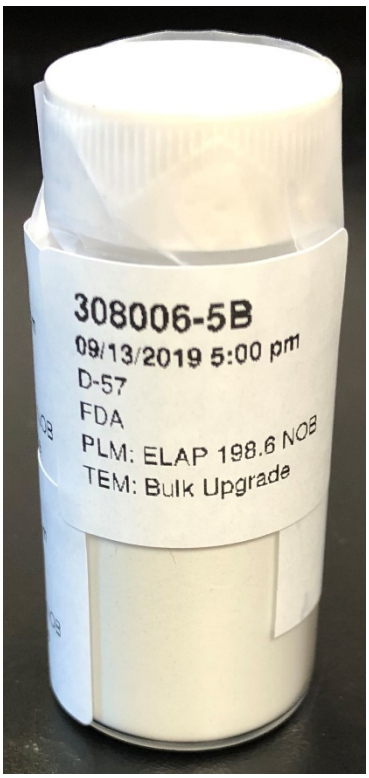
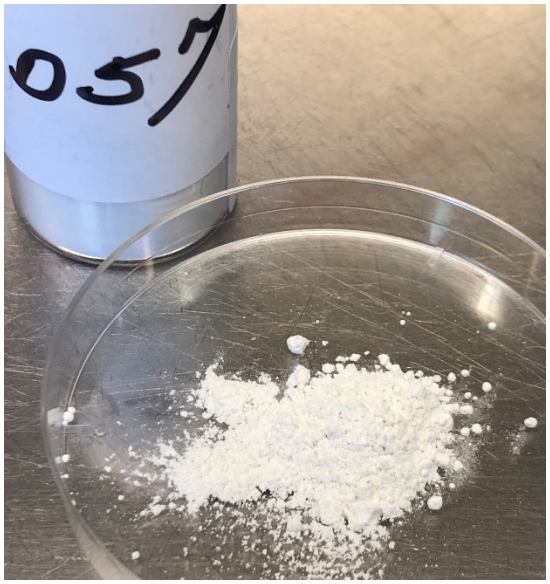
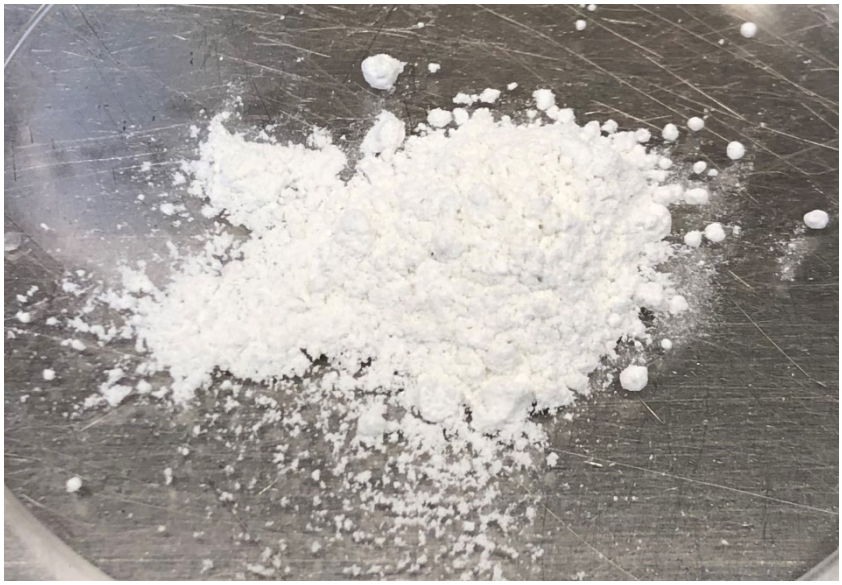


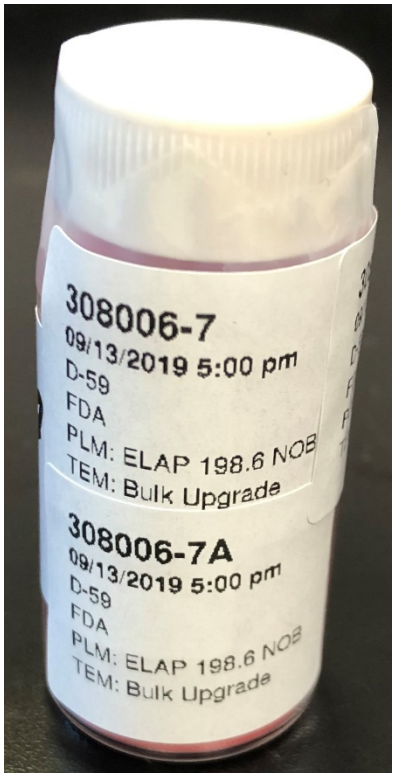


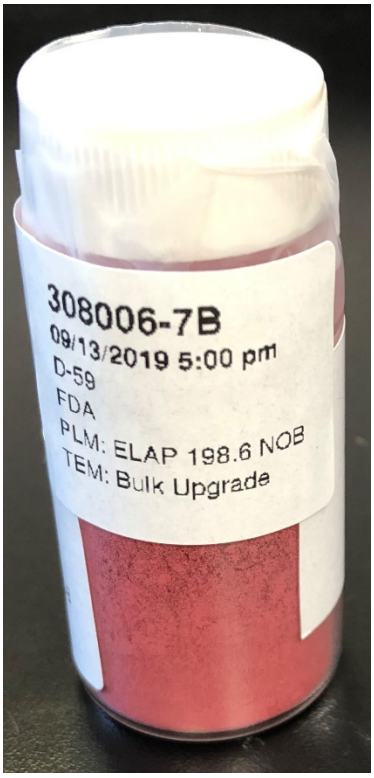




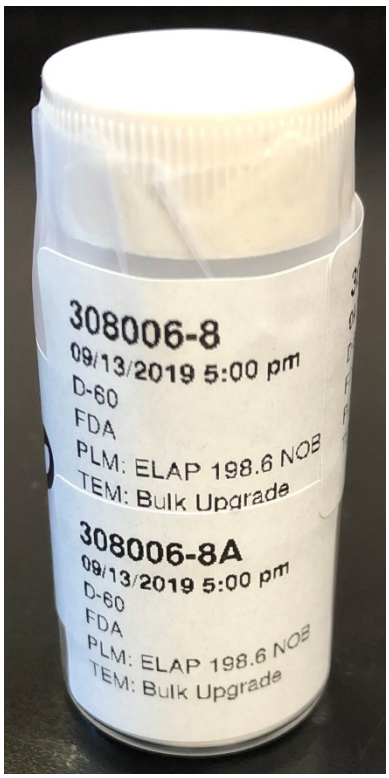


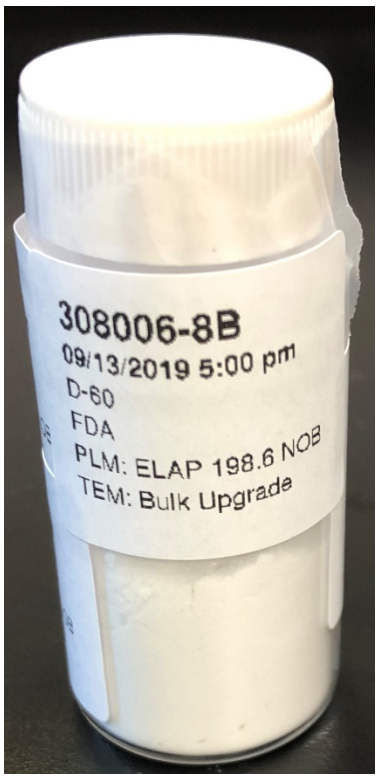
308006-7, 7A, 7B/D-59

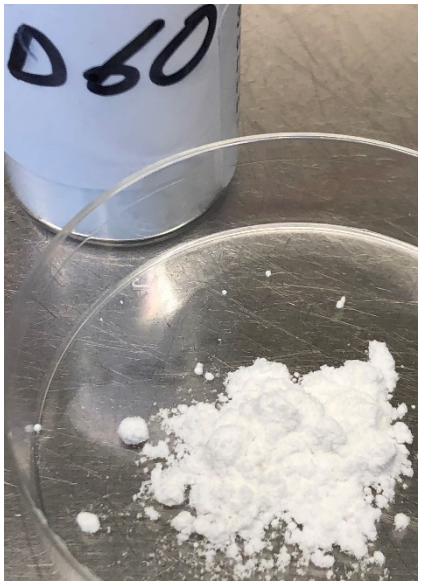


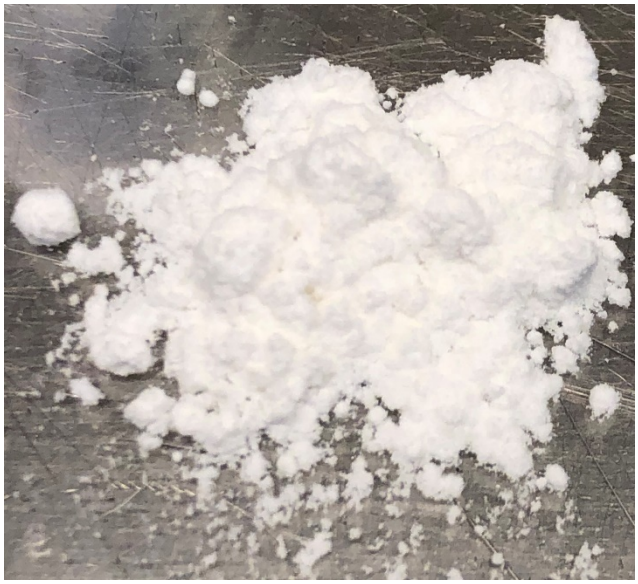


308006-8, 8A, 8B/D-60

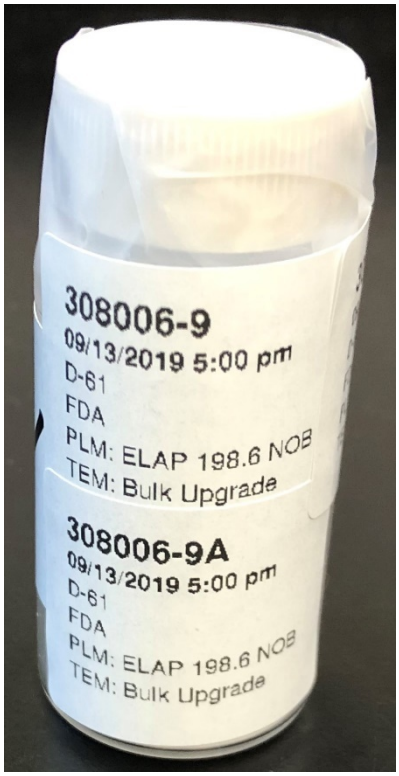


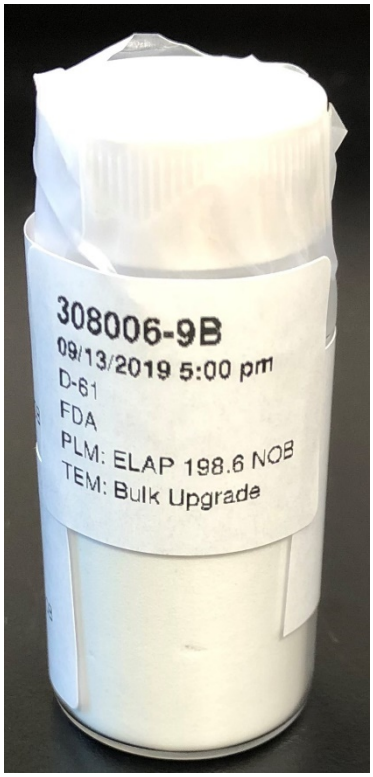


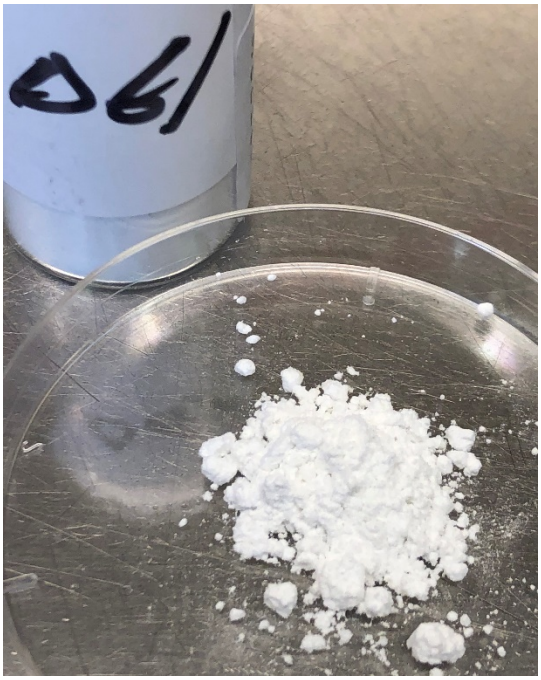


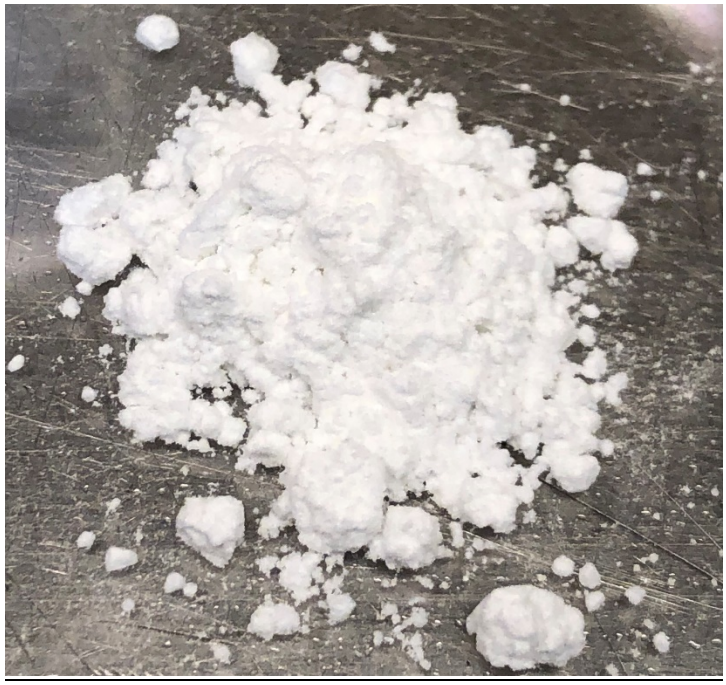


308006-9, 9A, 9B/D-61









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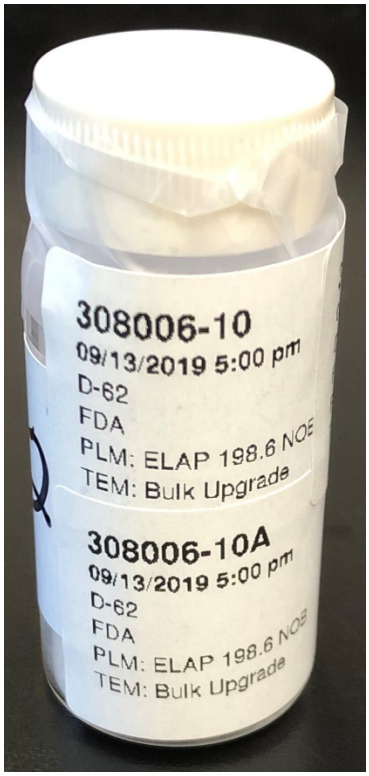

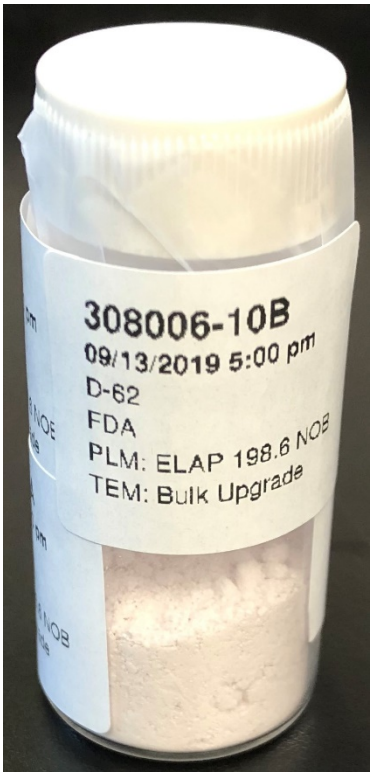

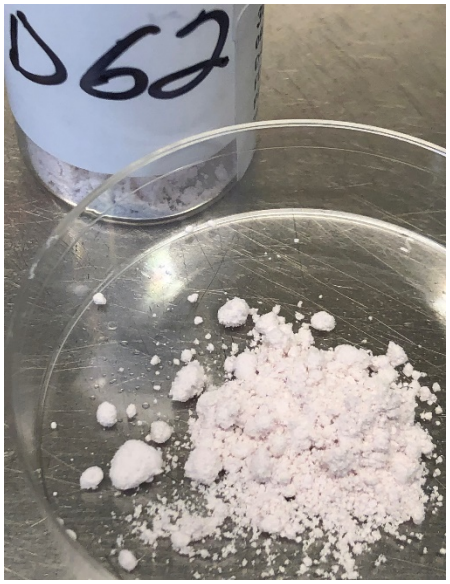

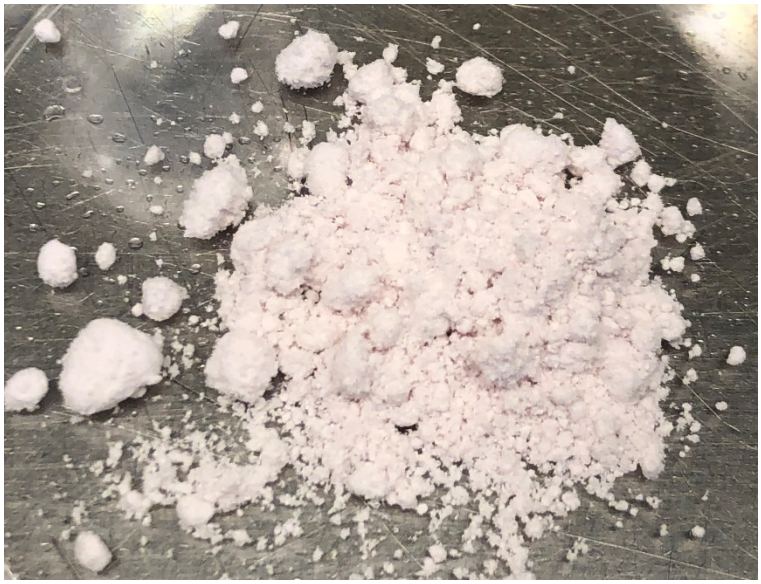

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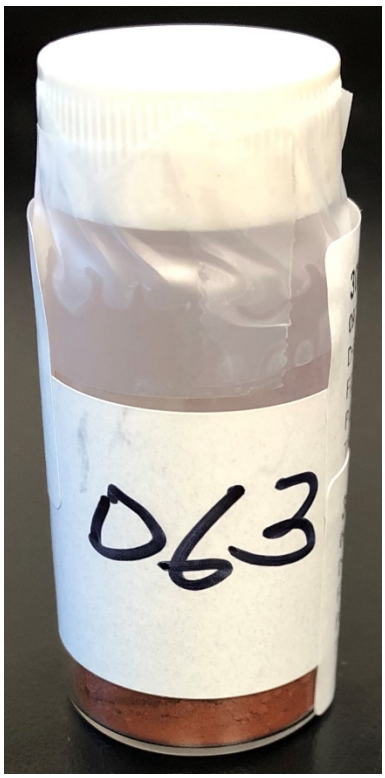

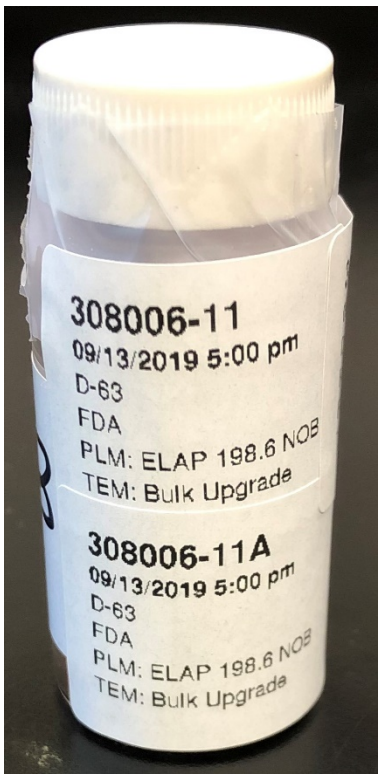

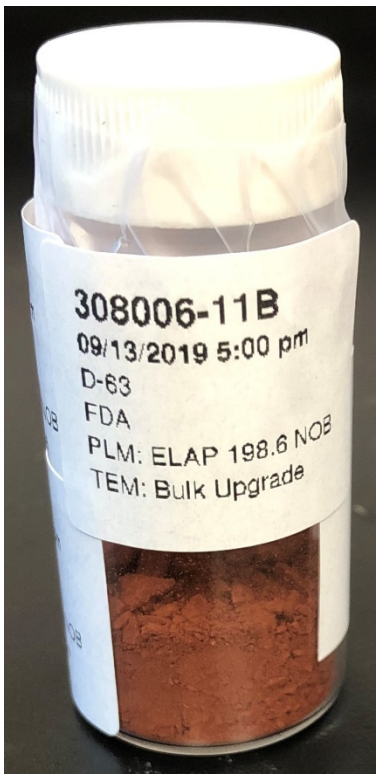

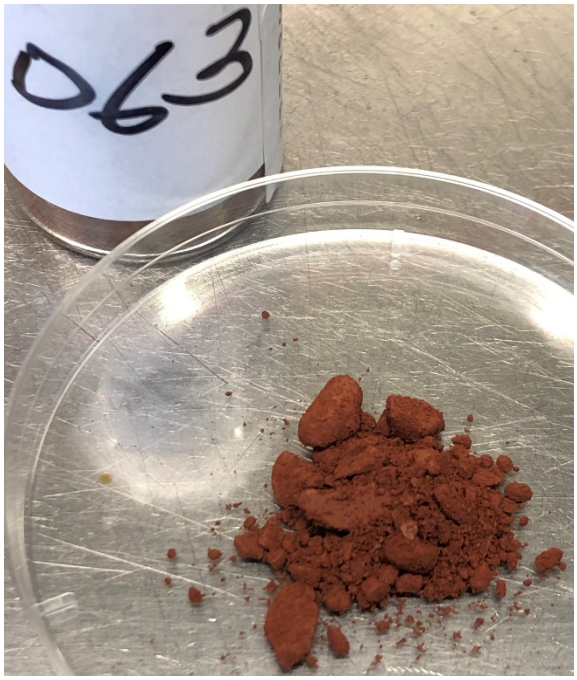

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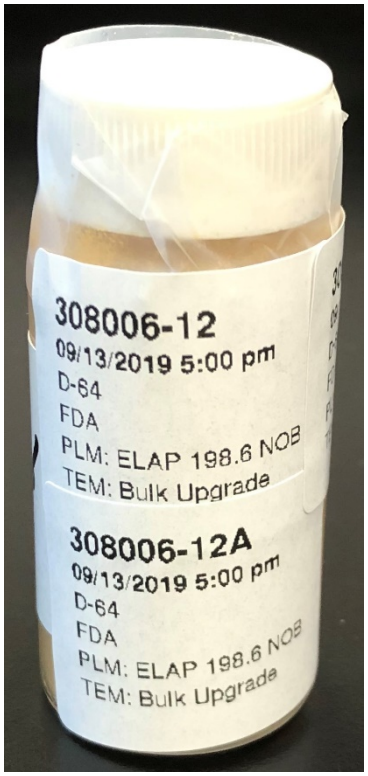

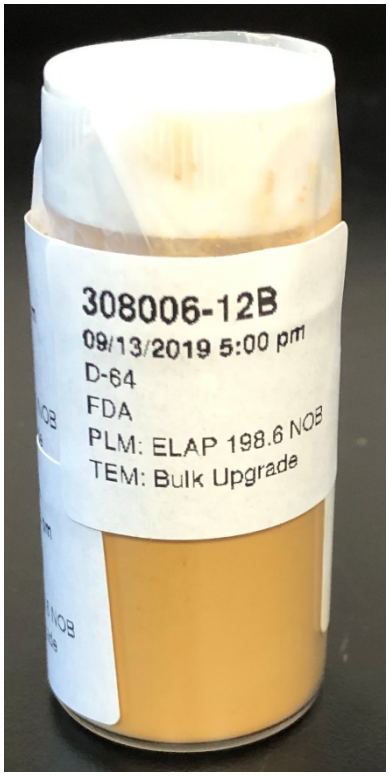

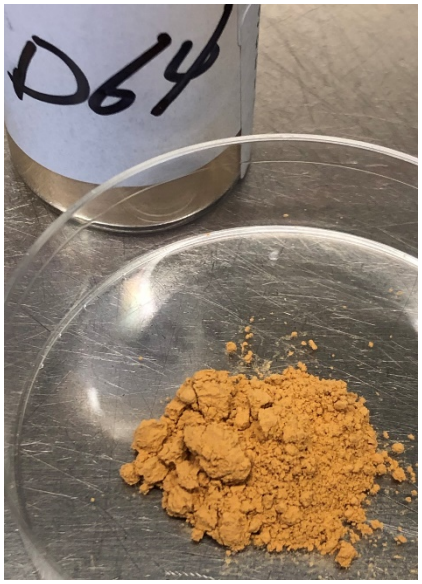

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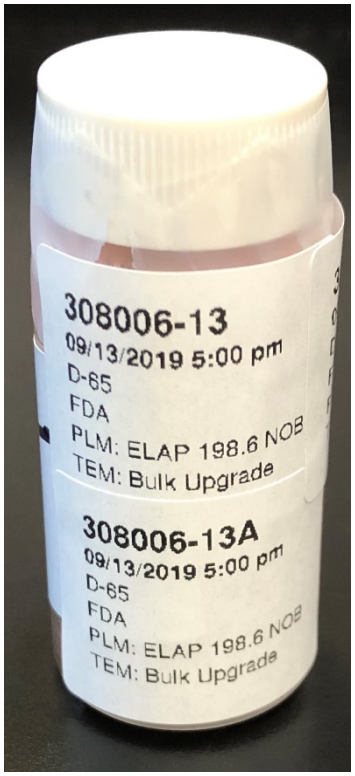
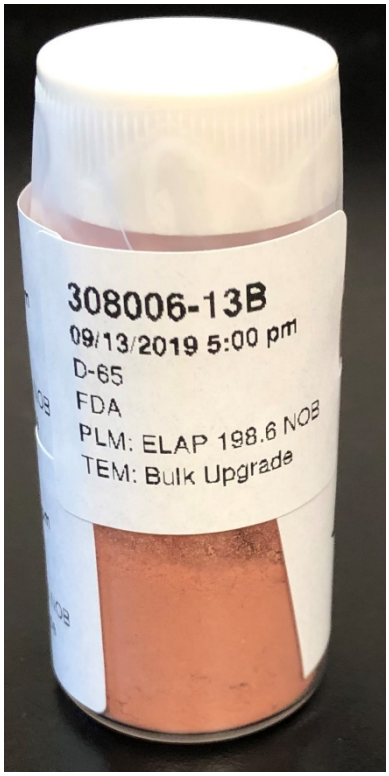
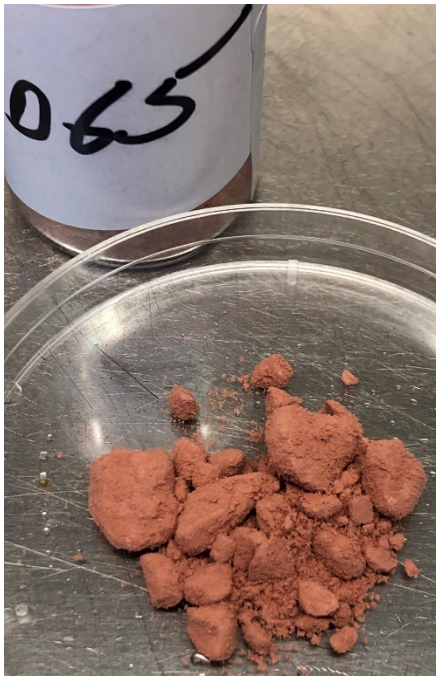
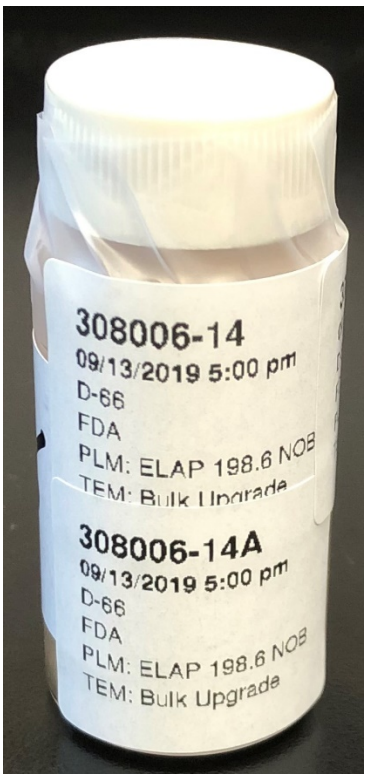
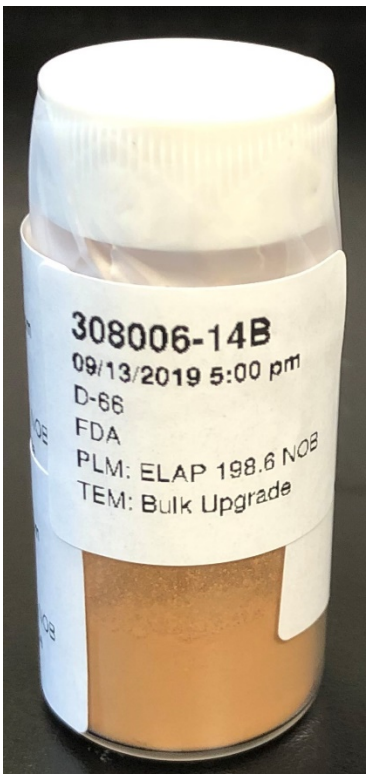




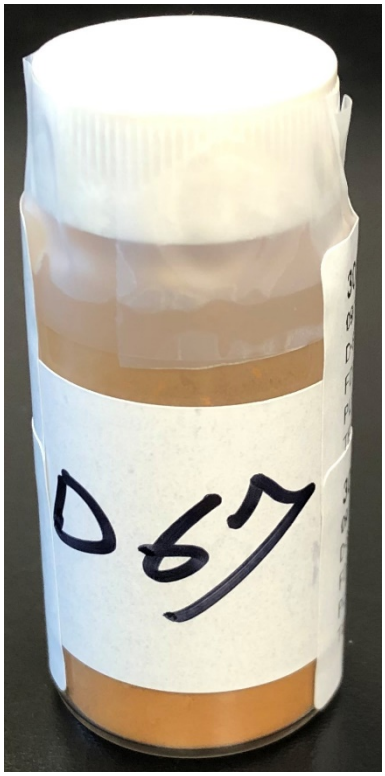


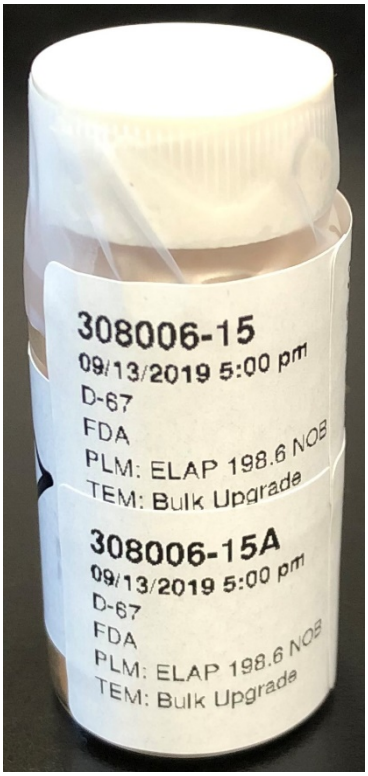
308006-14, 14A, 14B/D-66

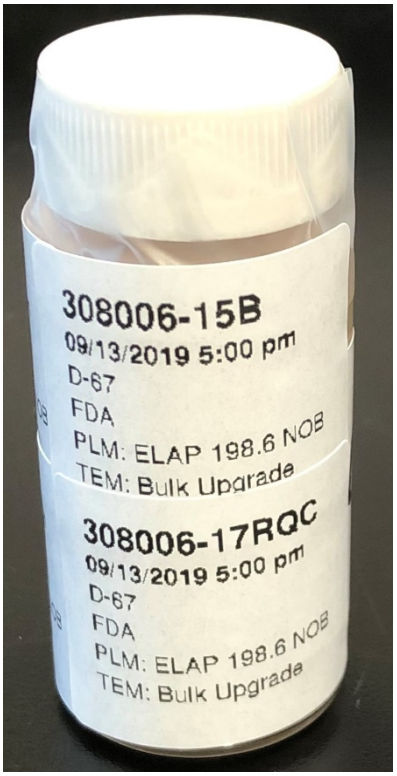


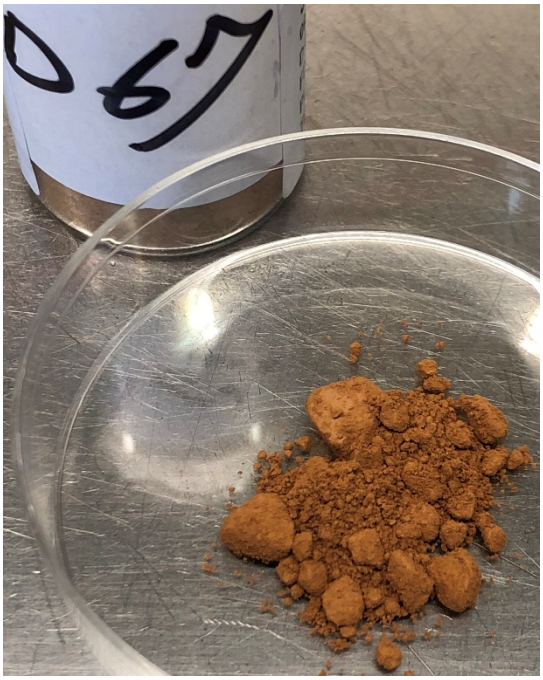


308006-15, 15A, 15B/D-67









Sample Preparation

Samples were prepared for PLM and TEM bulk analysis by (b) (6) on August 13, 2019 through September 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 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 and JEOL JEM-100CX transmission electron microscopes (TEM), equipped with a Thermo Fisher Quest Energy Dispersive X-Ray Analyzer (EDXA), at magnifications of 19,000-20,000x. Two grids for each aliquot were examined. Twenty (20) grid openings were examined per sample.

Modifications to the NY ELAP 198.4 Method were:

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

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

Calculations

ASTM D5756 Mass

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

M = mass

L = length
W = width
D = density

Percent Calculation

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

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

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

Limit of Detection and Quantification

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

Some aliquots of samples D54, D56 and D61 contained very small amounts of asbestos that were either at or below our 4-fiber limit of quantification. For these samples we defined our limit of quantification as follows:

308006-2A: mass of the single observed chrysotile structure plus the mass of three chrysotile fibers measuring 0.5 x 0.04 microns
308006-4A: mass of the single observed chrysotile structure plus the mass of three chrysotile fibers measuring 0.5 x 0.04 microns
308006-9B: mass of the single observed chrysotile structure plus the mass of three chrysotile fibers measuring 0.5 x 0.04 microns

Discussion and Interpretation of Analytical Findings:

308006-1, 1A, 1B, Client Sample D-53

PLM

All three aliquots of sample D-53 were analyzed by (b) (6) on September 13, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

308006-1	NAD
308006-1A	NAD
308006-1B	NAD

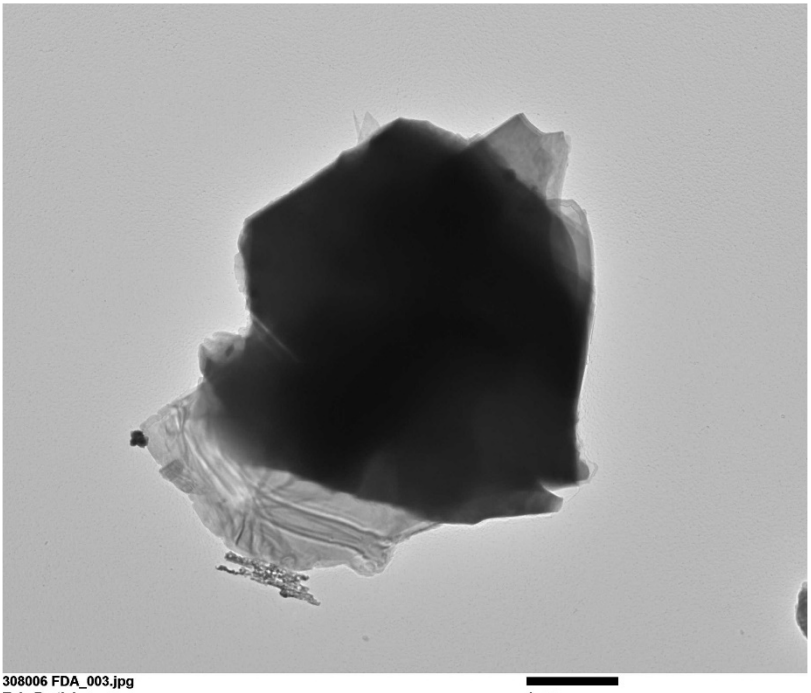
TEM

Sample 1 was analyzed by (b) (6) on August 20-21, 2019. (b) (6) analyzed samples 1A and 1B on August 28, 2019. The primary particles observed were mica and talc along with a few talc fibers, iron particles, titanium coated particles and very few silica spheres. No asbestos or non-asbestos amphibole variants were detected in the samples. The results were calculated using the equations detailed in the calculations section.

308006-1	NAD
308006-1A	NAD
308006-1B	NAD

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

Sample 308006-1, Talc Particle



308006 FDA_003.jpg
Talc Particle
Cal: 0.003548 µm/pix
19:58 8/20/2019
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

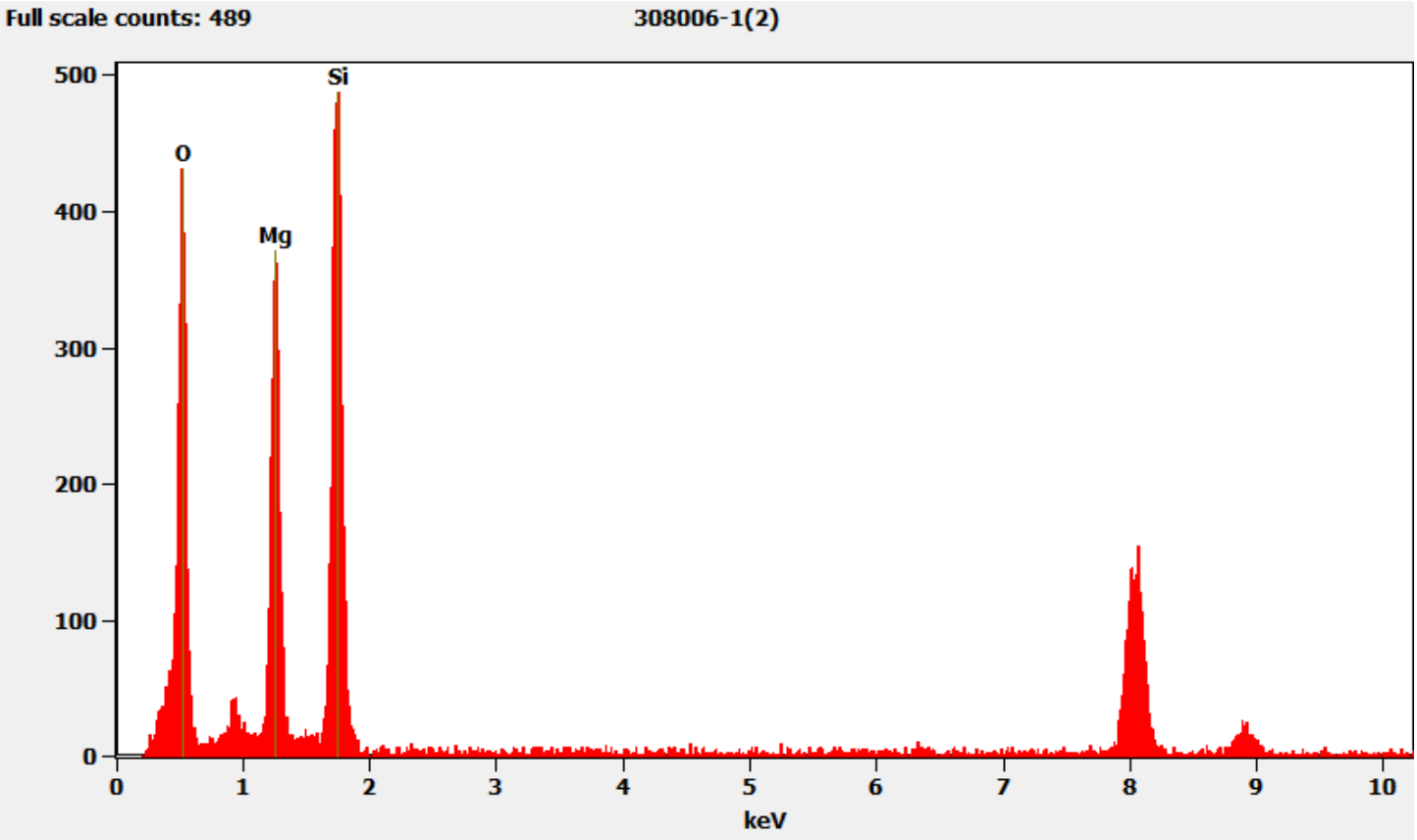
1 µm
HV=100kV
Direct Mag: 2900 x
AMA Analytical Services, Inc

Hexagonal diffraction from the Talc particle pictured above.

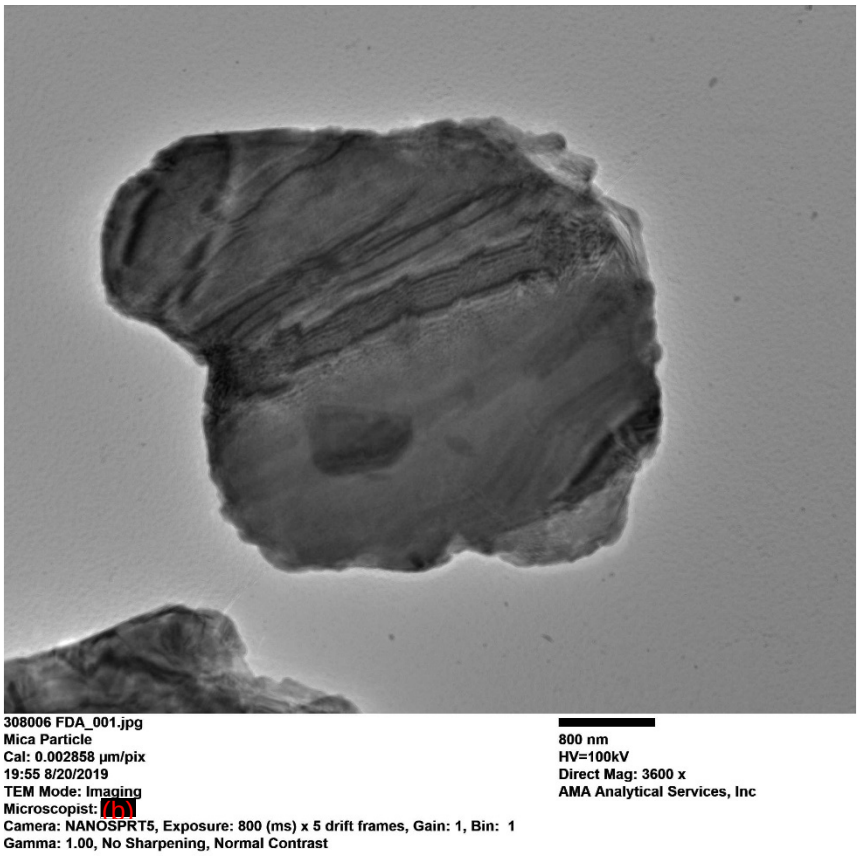
308006 FDA_004.jpg
Talc Particle
19:59 8/20/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from the Talc particle pictured above.

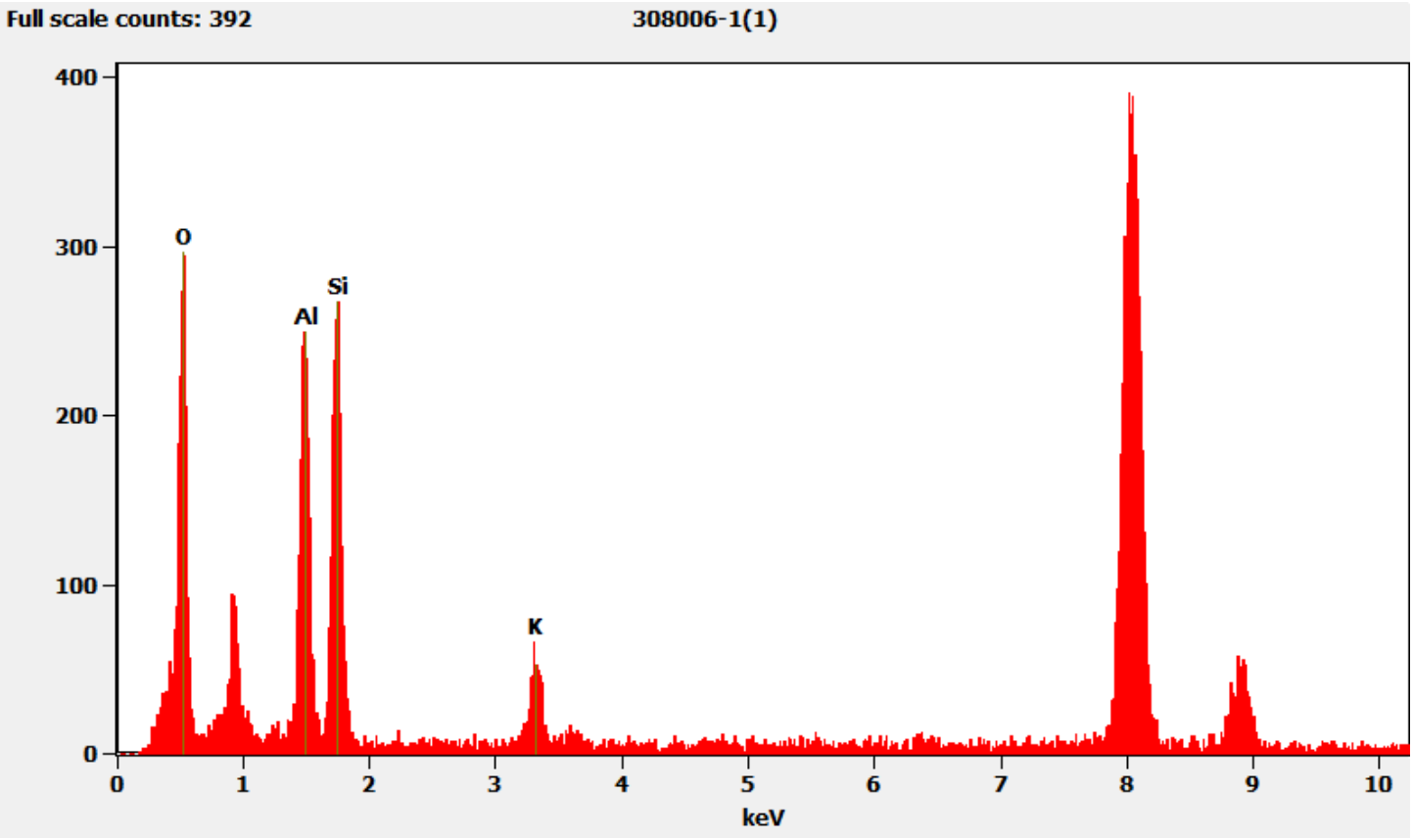


Sample 308006-1, Mica Particle

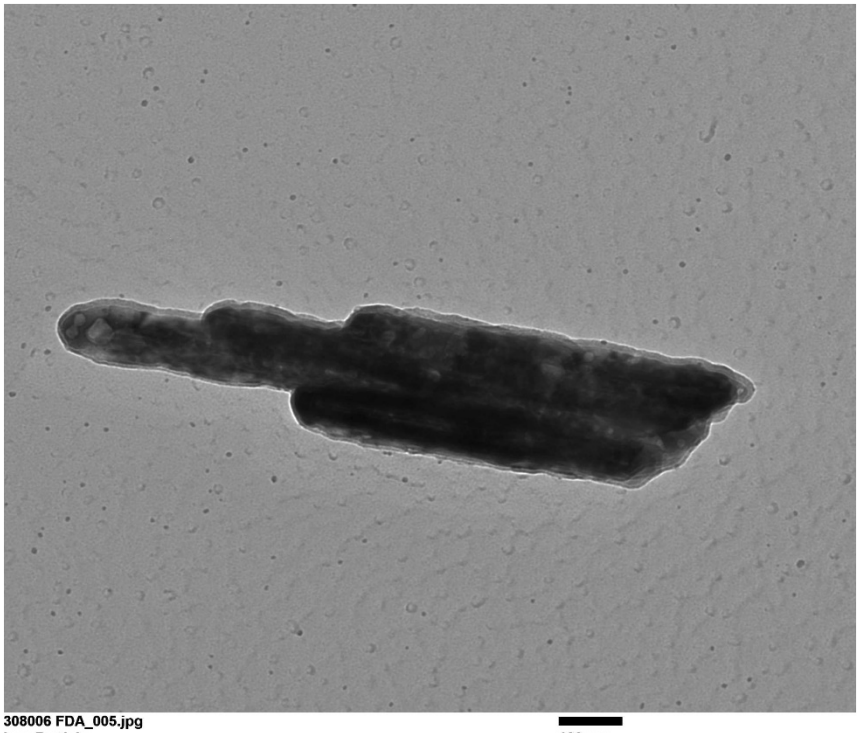


Diffraction pattern from the Mica particle pictured above.

Chemistry from the Mica particle pictured above



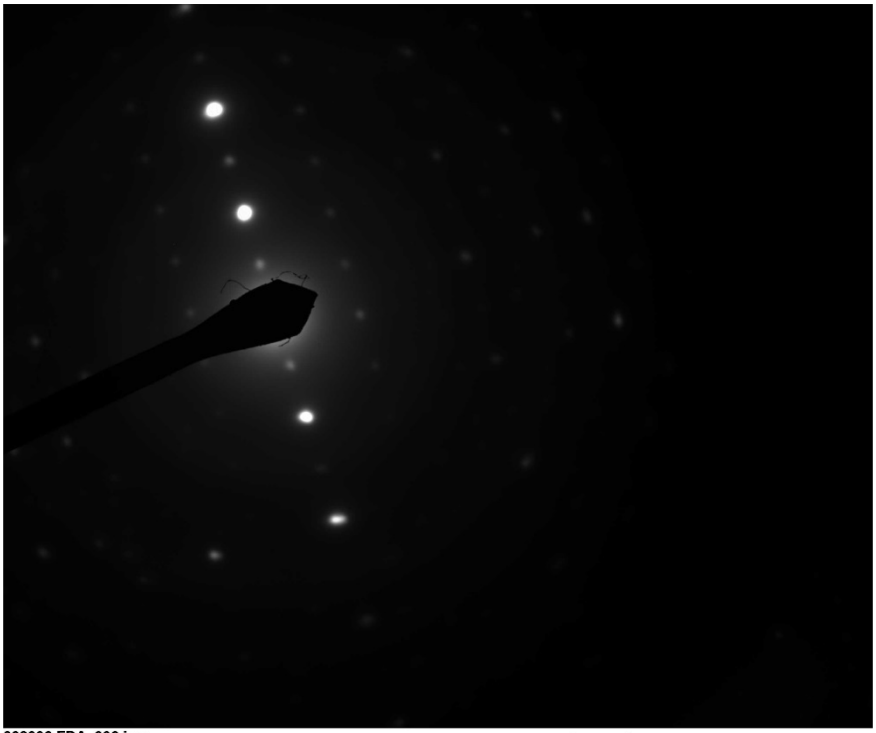
Sample 308006-1, Iron Particle



308006 FDA_005.jpg
Iron Particle
Cal: 0.541520 nm/pix
20:01 8/20/2019
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

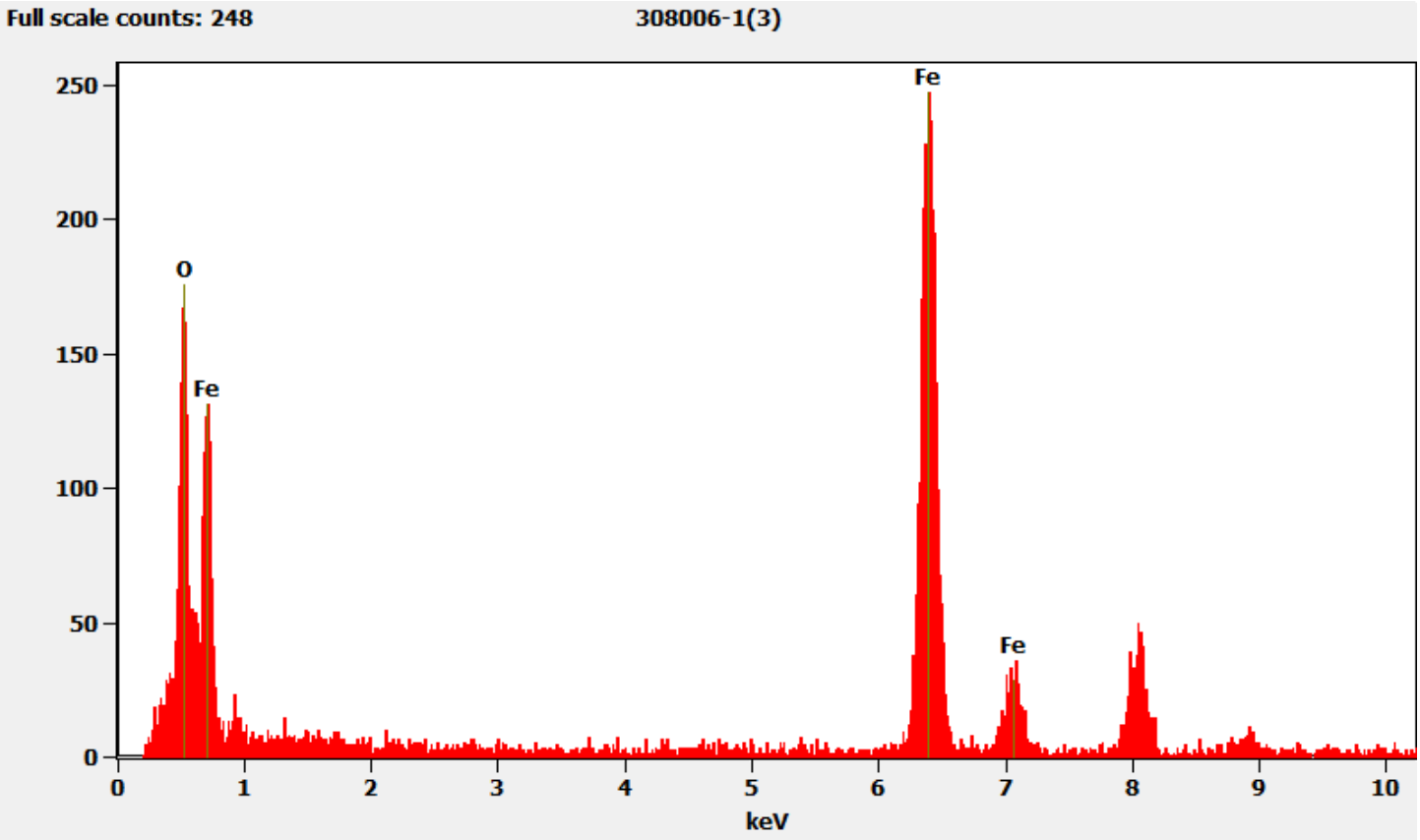
Diffraction pattern from the Iron particle pictured above.



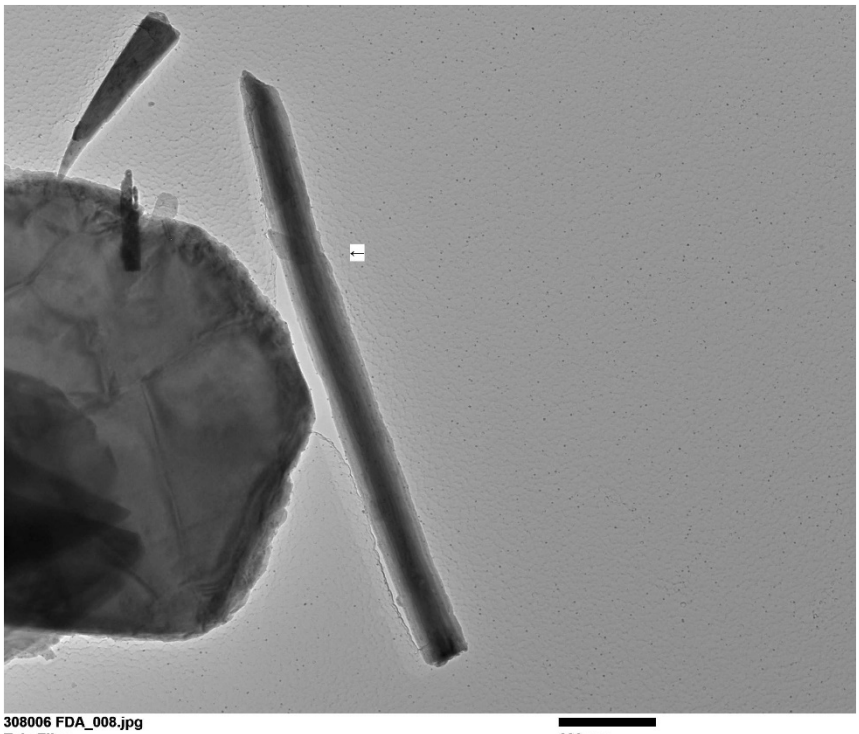
308006 FDA_006.jpg
Iron Particle
20:02 8/20/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from the Iron particle pictured above.



Sample 308006-1, Talc Fiber

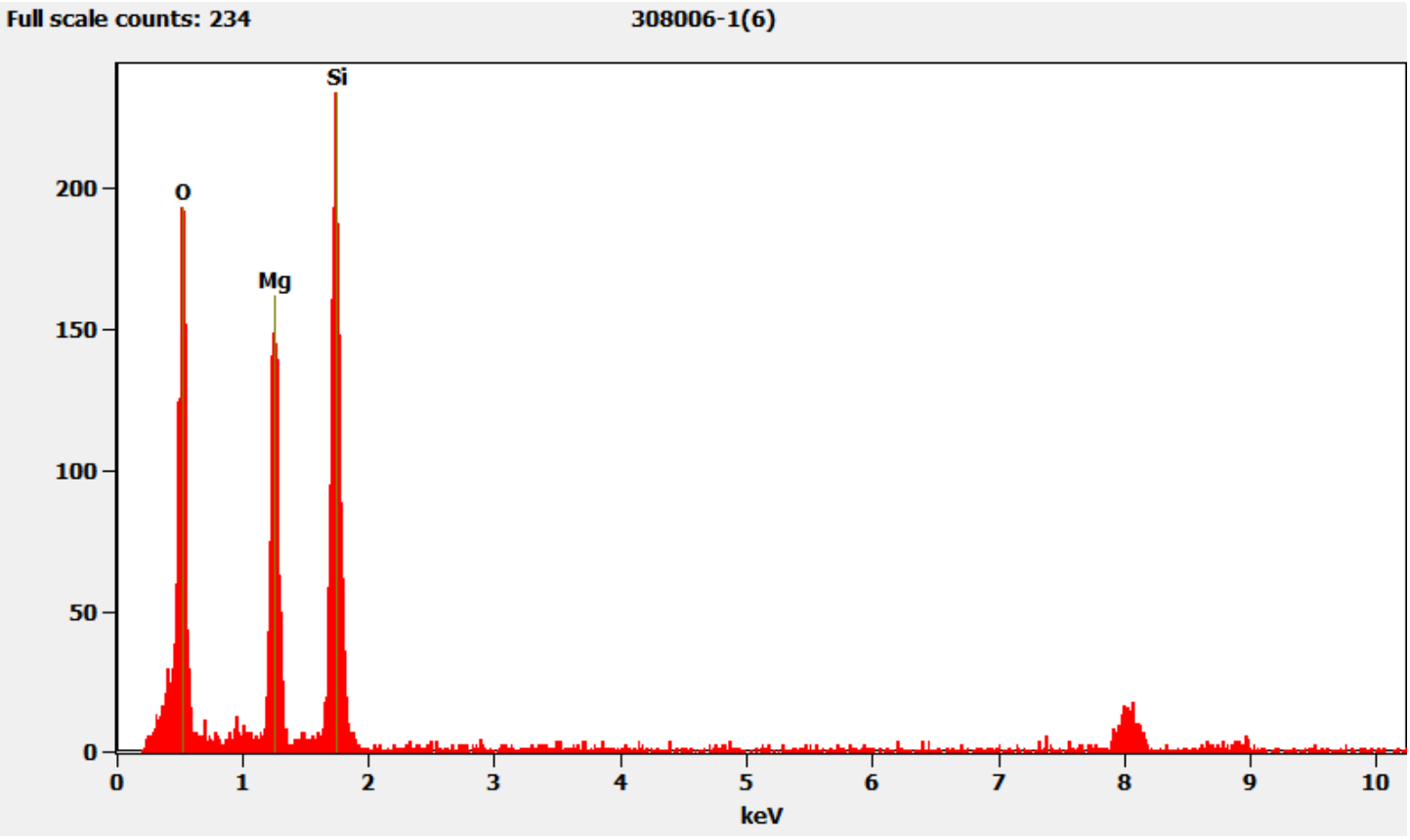


308006 FDA_008.jpg
Talc Fiber
Cal: 0.002144 µm/pix
20:12 8/20/2019
TEM Mode: Imaging
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

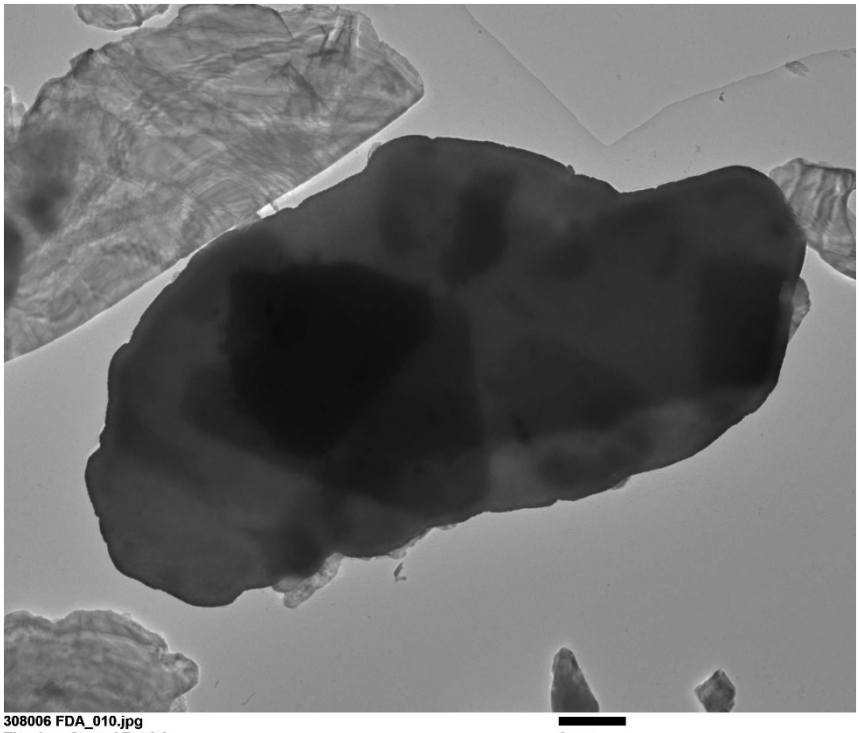
600 nm
HV=100kV
Direct Mag: 4800 x
AMA Analytical Services, Inc

Diffraction pattern from the Talc Fiber pictured above.

Chemistry from the Talc Fiber pictured above.



308006-1, Titanium Coated Particle



308006 FDA_010.jpg
Titanium Coated Particle
Cal: 0.010289 µm/pix
14:46 8/21/2019
TEM Mode: Imaging
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 std. frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

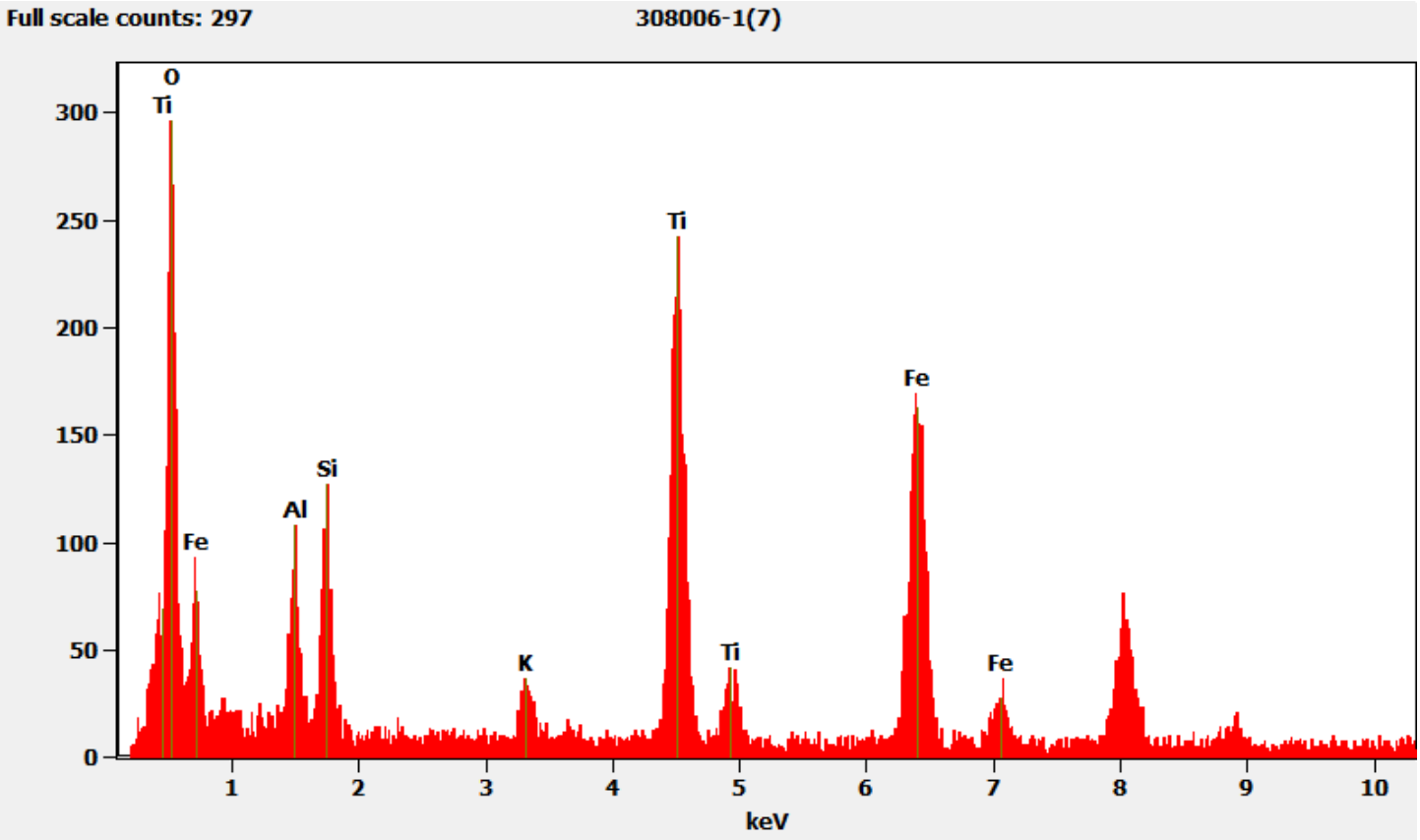
2 µm
HV=100kV
Direct Mag: 1000 x
AMA Analytical Services, Inc

Diffraction pattern from the Titanium Coated particle pictured above

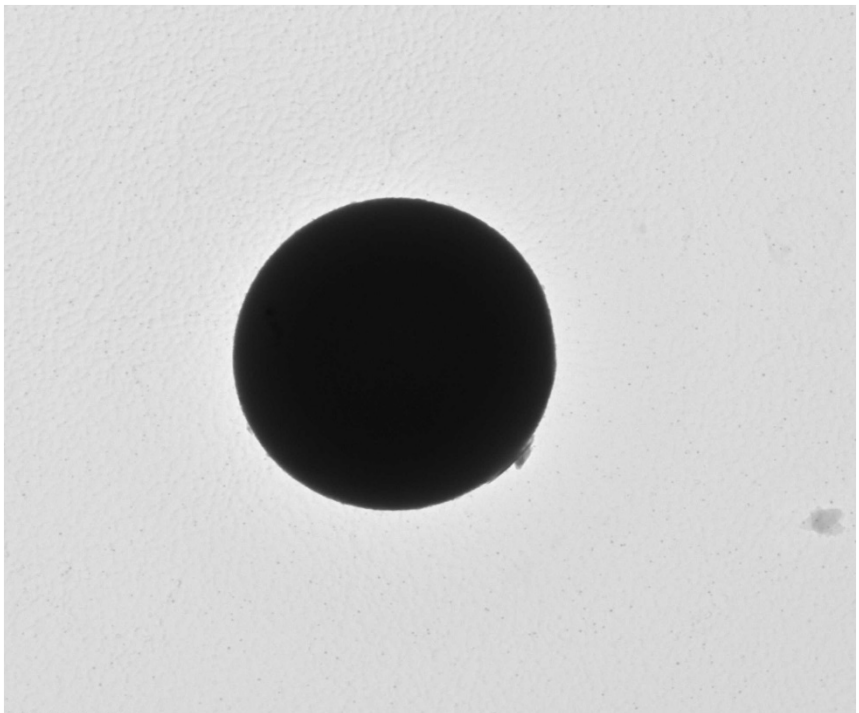
308006 FDA_011.jpg
Titanium Coated Particle
14:47 8/21/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 std. frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

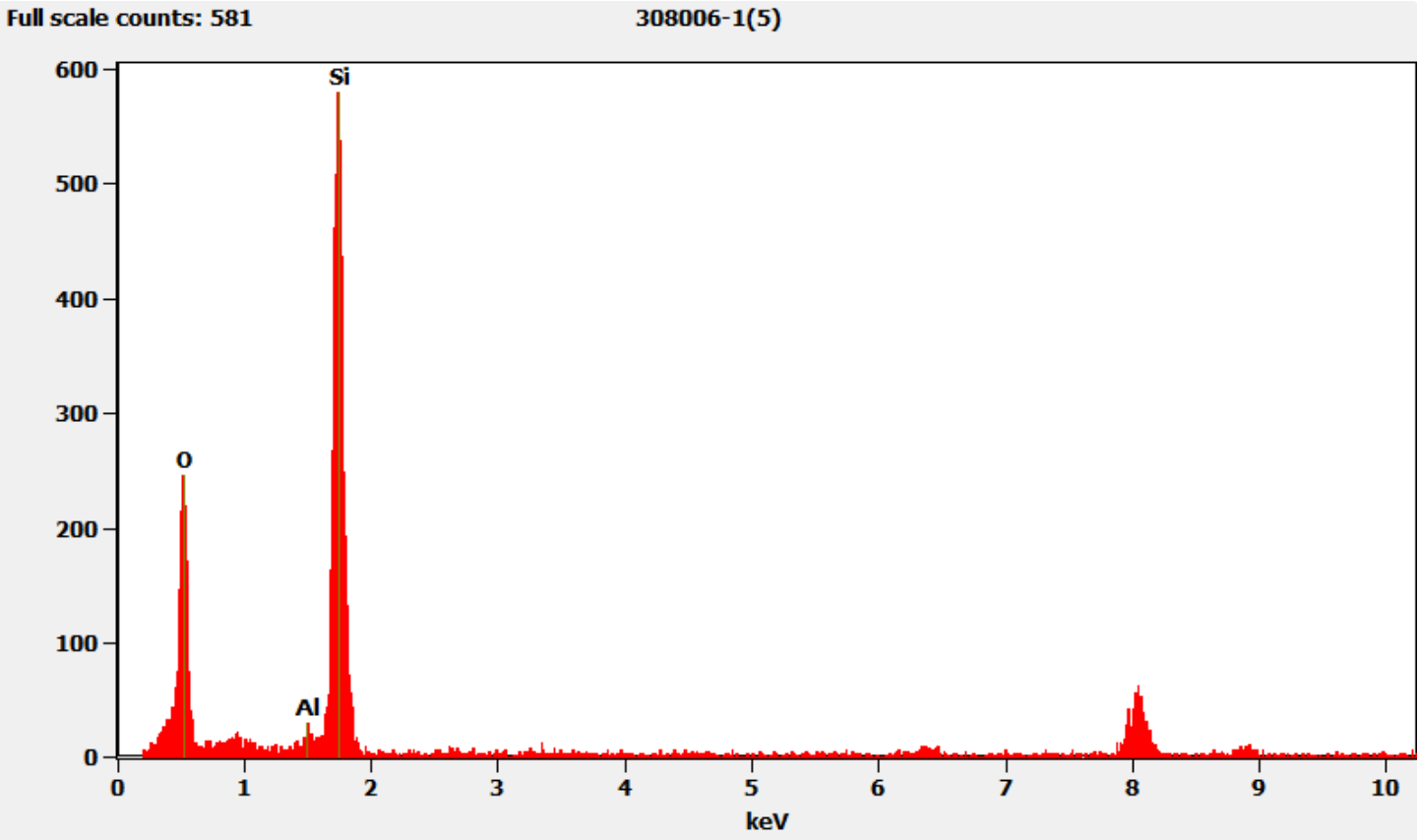
Chemistry from the Titanium Coated particle pictured above



308006-1, Silica Sphere



Chemistry from the Silica Sphere pictured above



308006-2, 2A, 2B, Client Sample D-54

PLM

All three aliquots of sample D-54 were analyzed by (b) (6) on September 13, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

308006-2	NAD
308006-2A	NAD
308006-2B	NAD

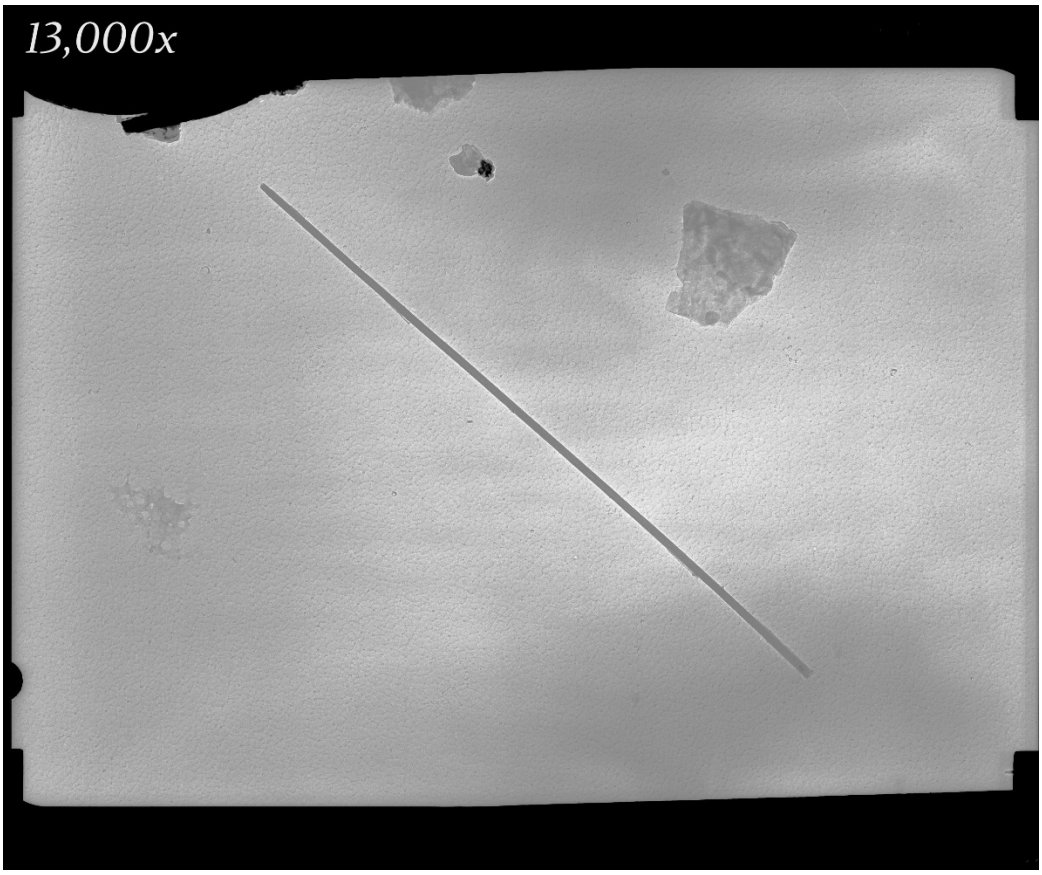
TEM

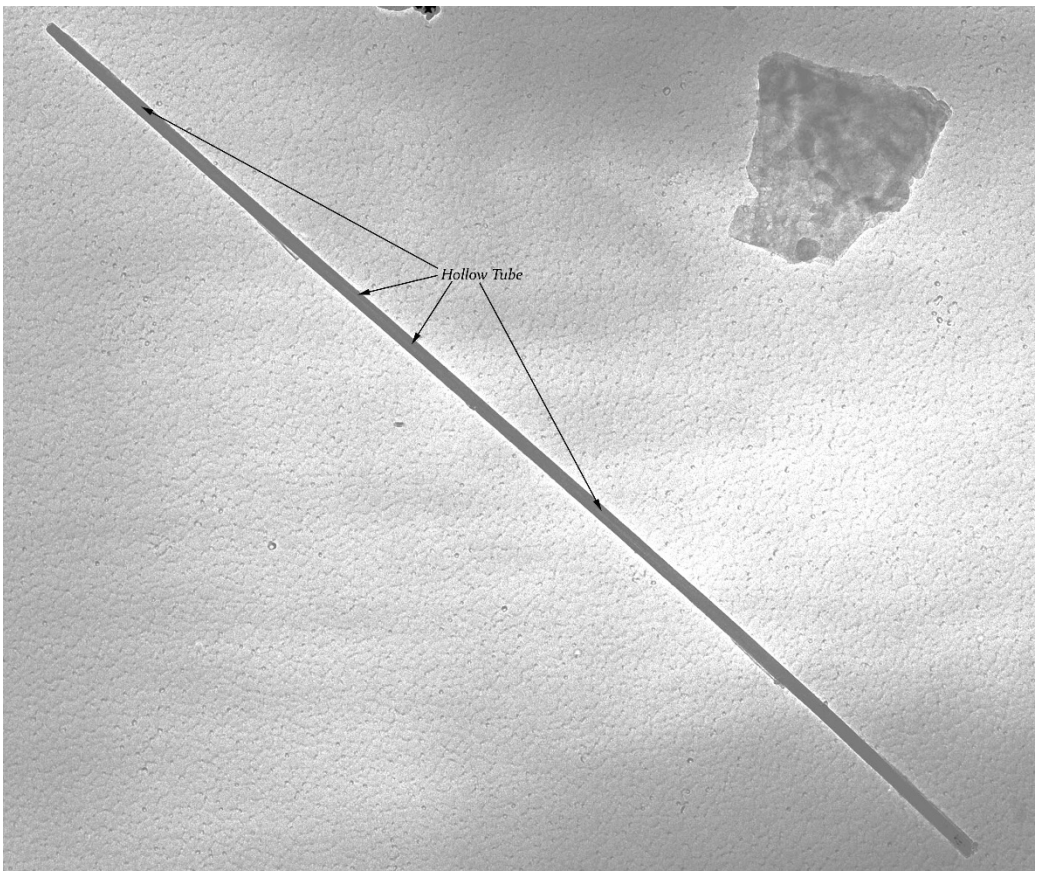
Sample 2 was analyzed by (b) (6) on August 21, 2019. (b) (6) analyzed sample 2A on August 28, 2019 and sample 2B on August 29, 2019. The primary particles observed were mica and talc along with a few titanium particles and silica particles/spheres. A chrysotile structure was observed on aliquot 2A. The results were calculated using the equations detailed in the calculations section.

308006-2	NAD
308006-2A	<0.00004
308006-2B	NAD

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

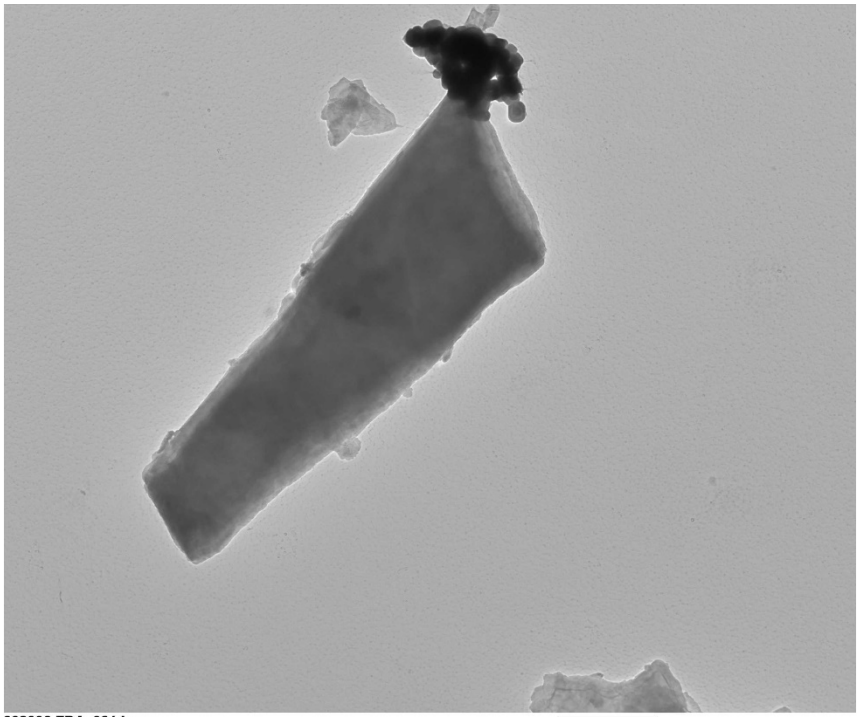
Sample 308006-2A Chrysotile Fiber *(the images below were taken on film; the digital versions were scanned from a negative)*





Diffraction Pattern from the chrysotile fiber pictured above. *(the image below was taken on film; the digital version was scanned from a negative)*

Sample 308006-2, Talc Particle

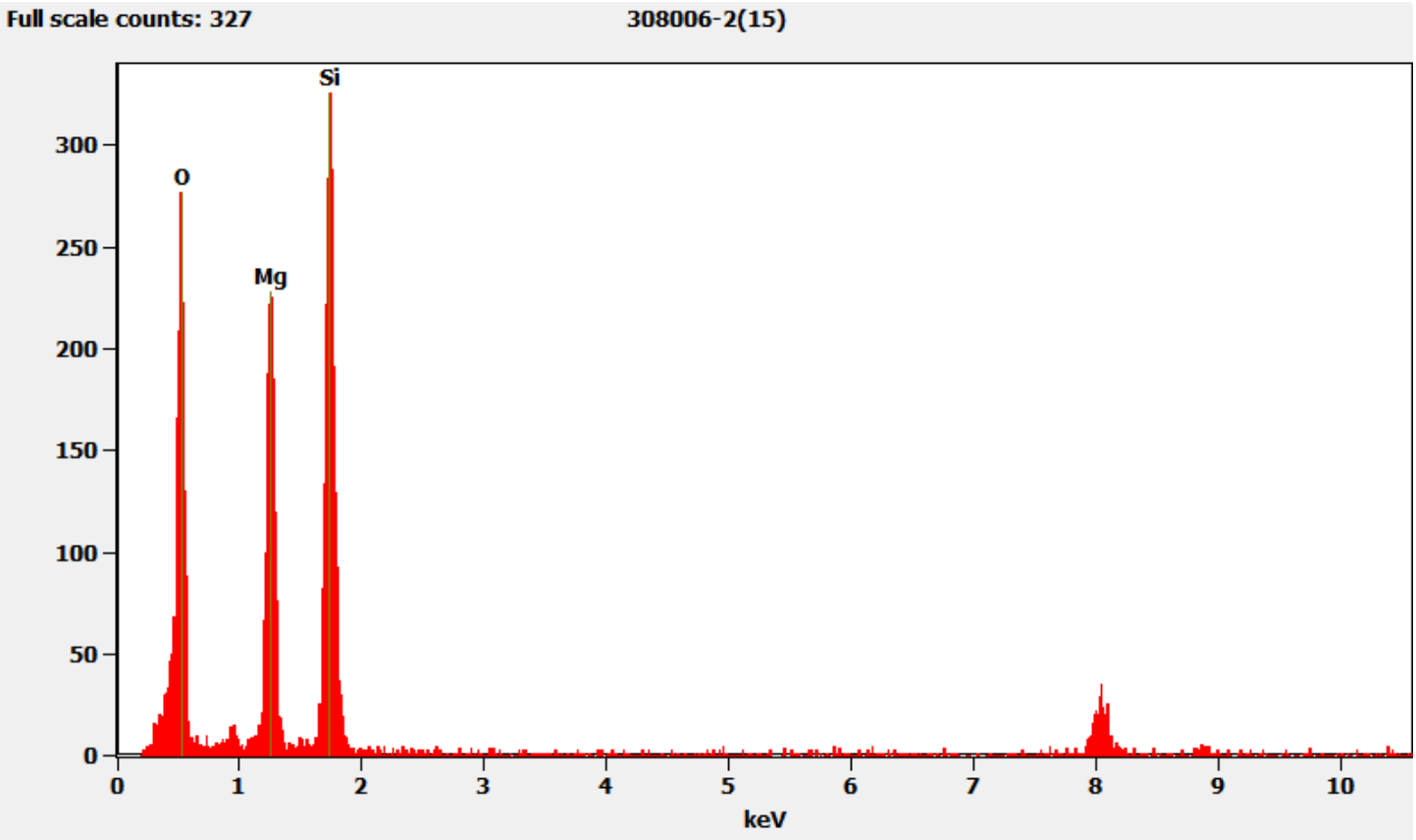


308006 FDA_021.jpg
Talc Particle
Cal: 0.002858 µm/pix
16:08 8/21/2019
TEM Mode: Imaging
Microscopist: [redacted]
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

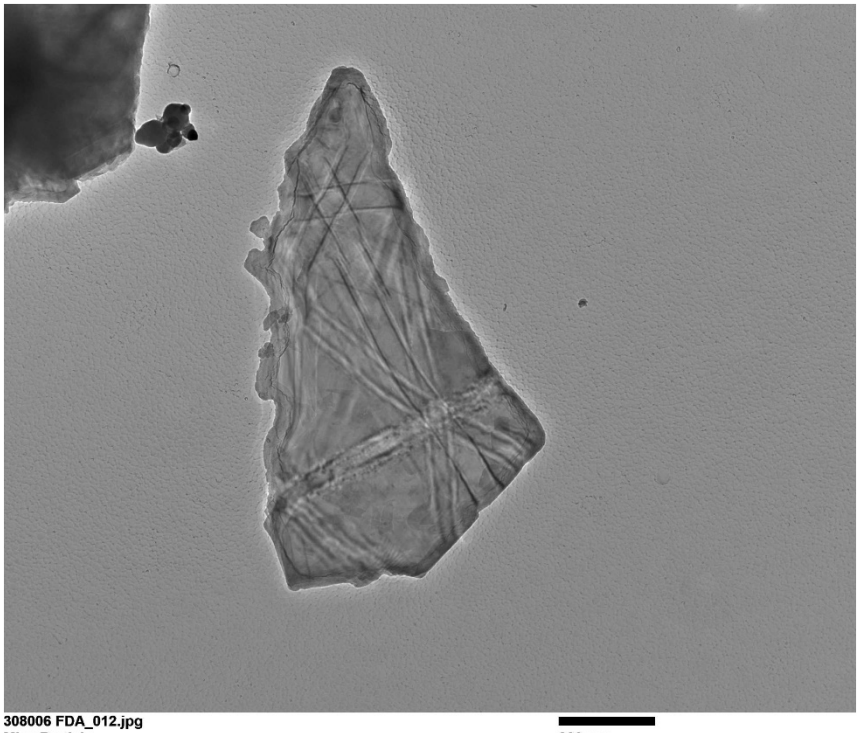
800 nm
HV=100kV
Direct Mag: 3600 x
AMA Analytical Services, Inc

Hexagonal Diffraction pattern from the Talc particle pictured above

Chemistry from the Talc particle pictured above



Sample 308006-2, Mica Particle



308006 FDA_012.jpg
Mica Particle
Cal: 0.002858 µm/pix
15:08 8/21/2019
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

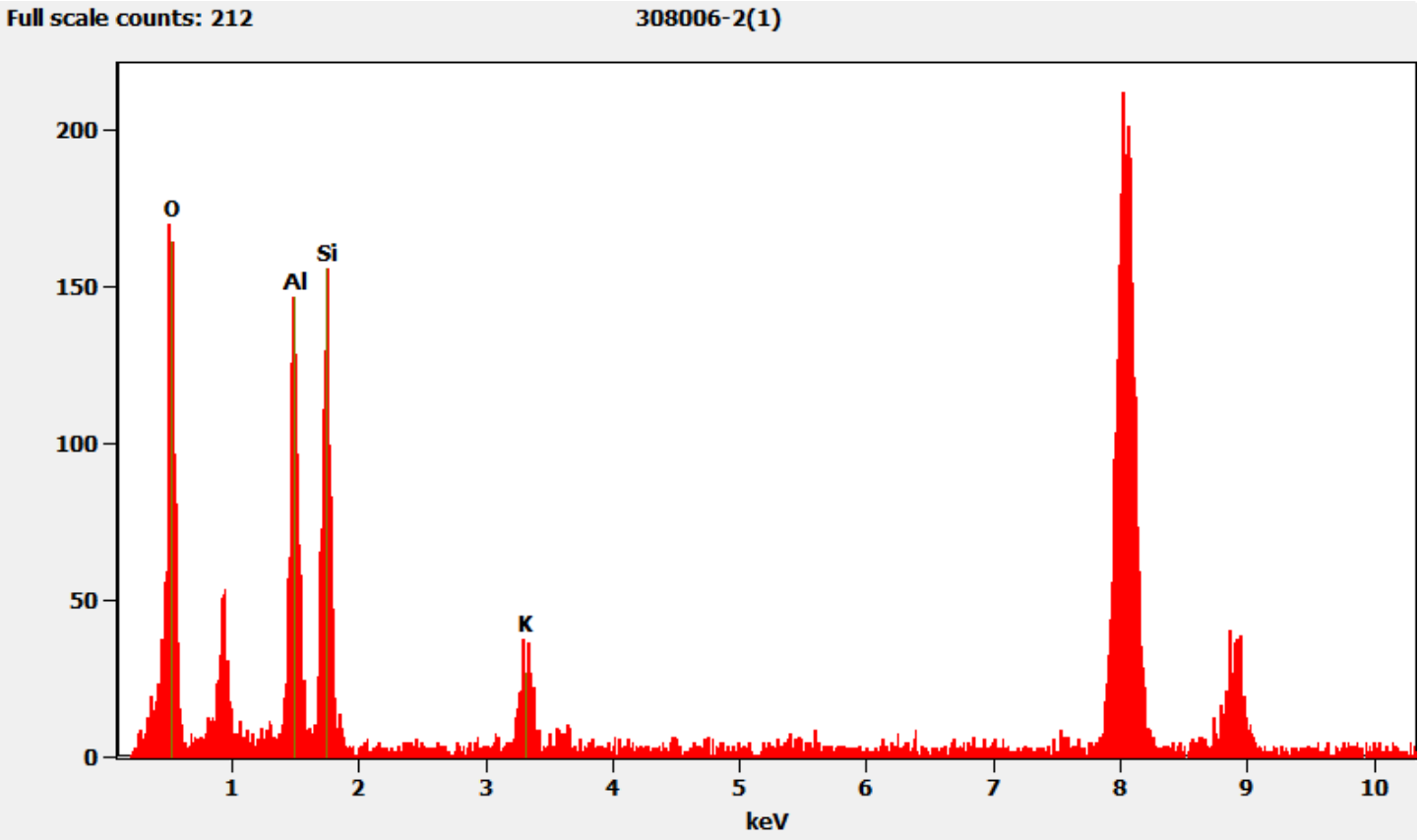
800 nm
HV=100kV
Direct Mag: 3600 x
AMA Analytical Services, Inc

Diffraction pattern from the Mica particle pictured above.

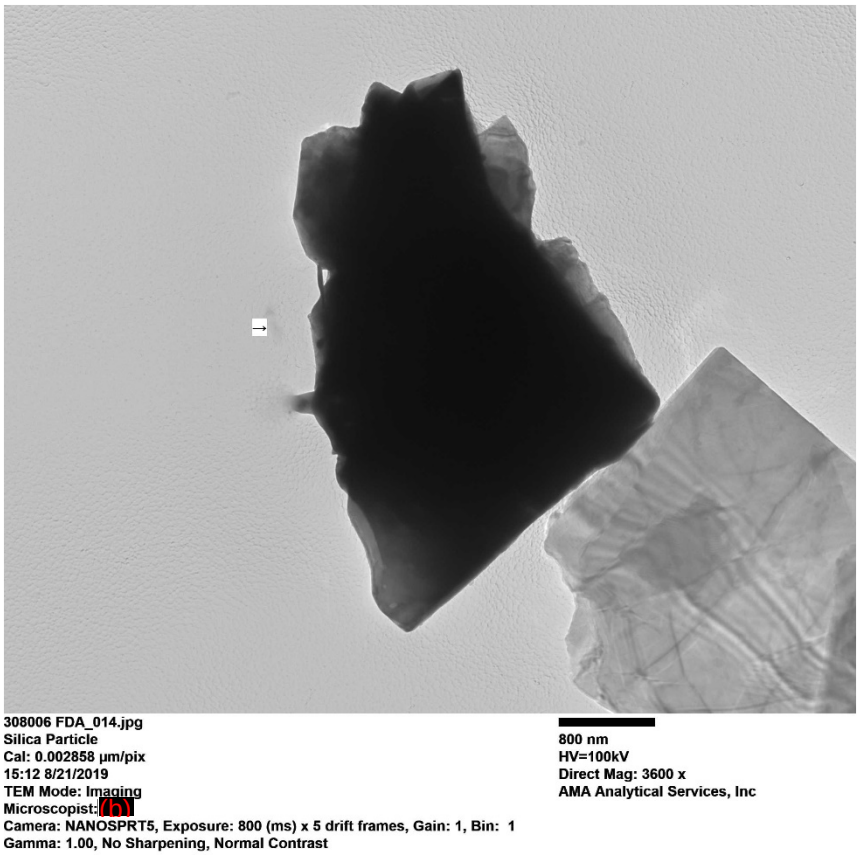
308006 FDA_013.jpg
Mica Particle
15:10 8/21/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

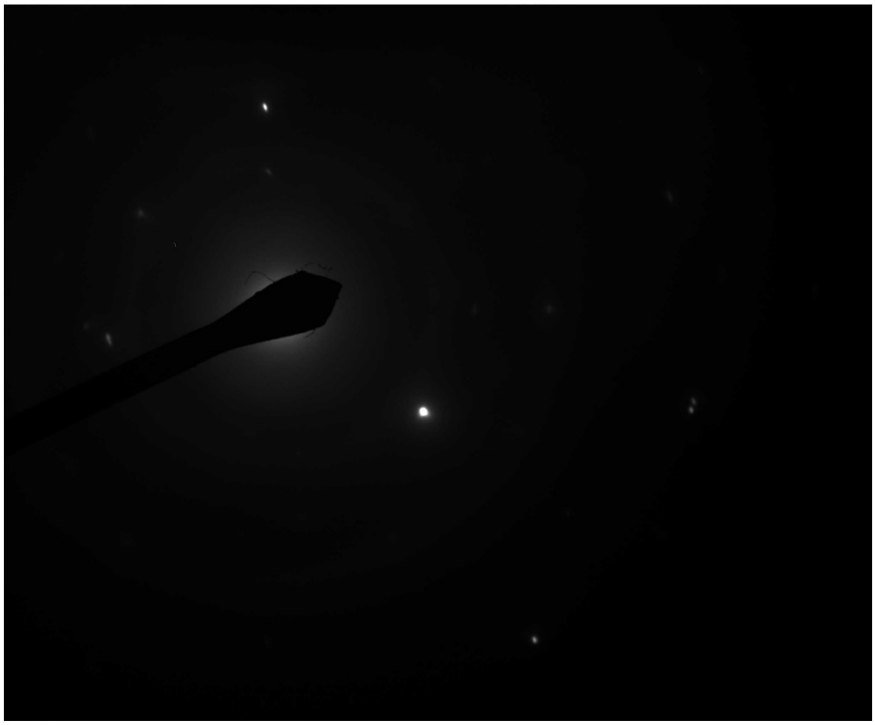
Chemistry from the Mica particle pictured above.



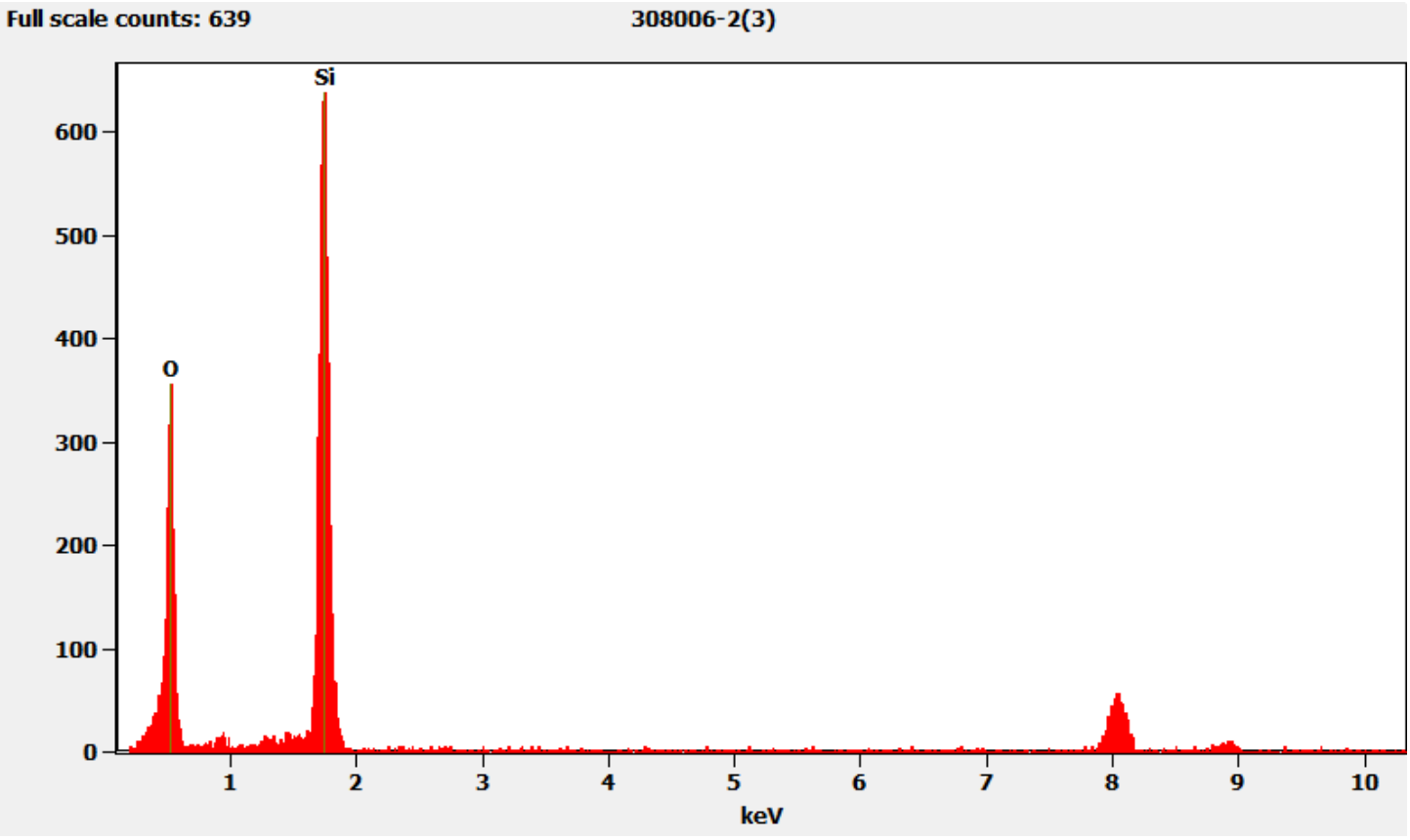
Sample 308006-2, Silica Particle



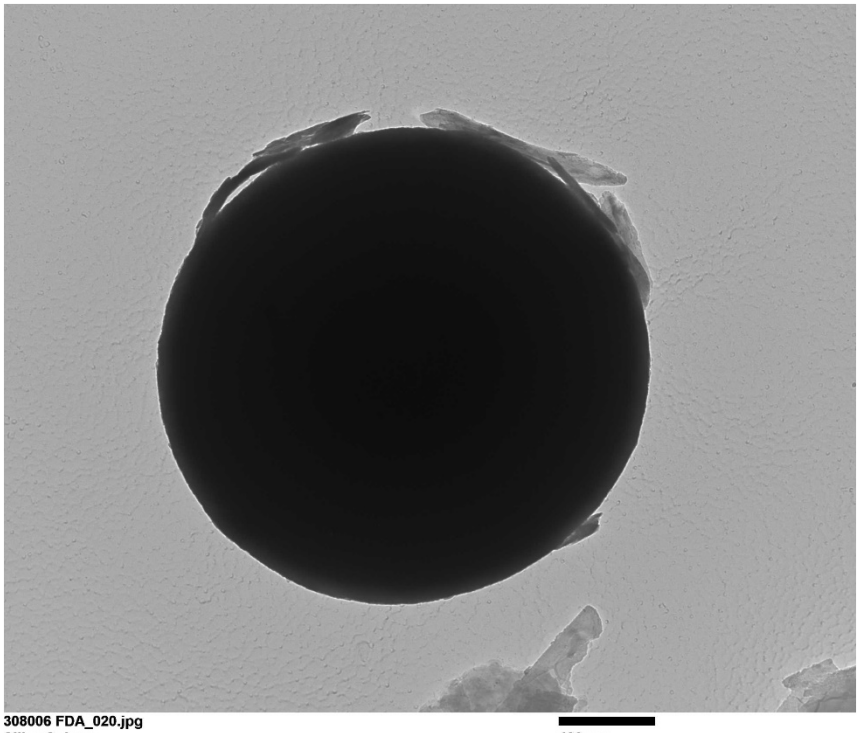
Diffraction pattern from the Silica particle pictured above



Chemistry from the Silica particle pictured above.



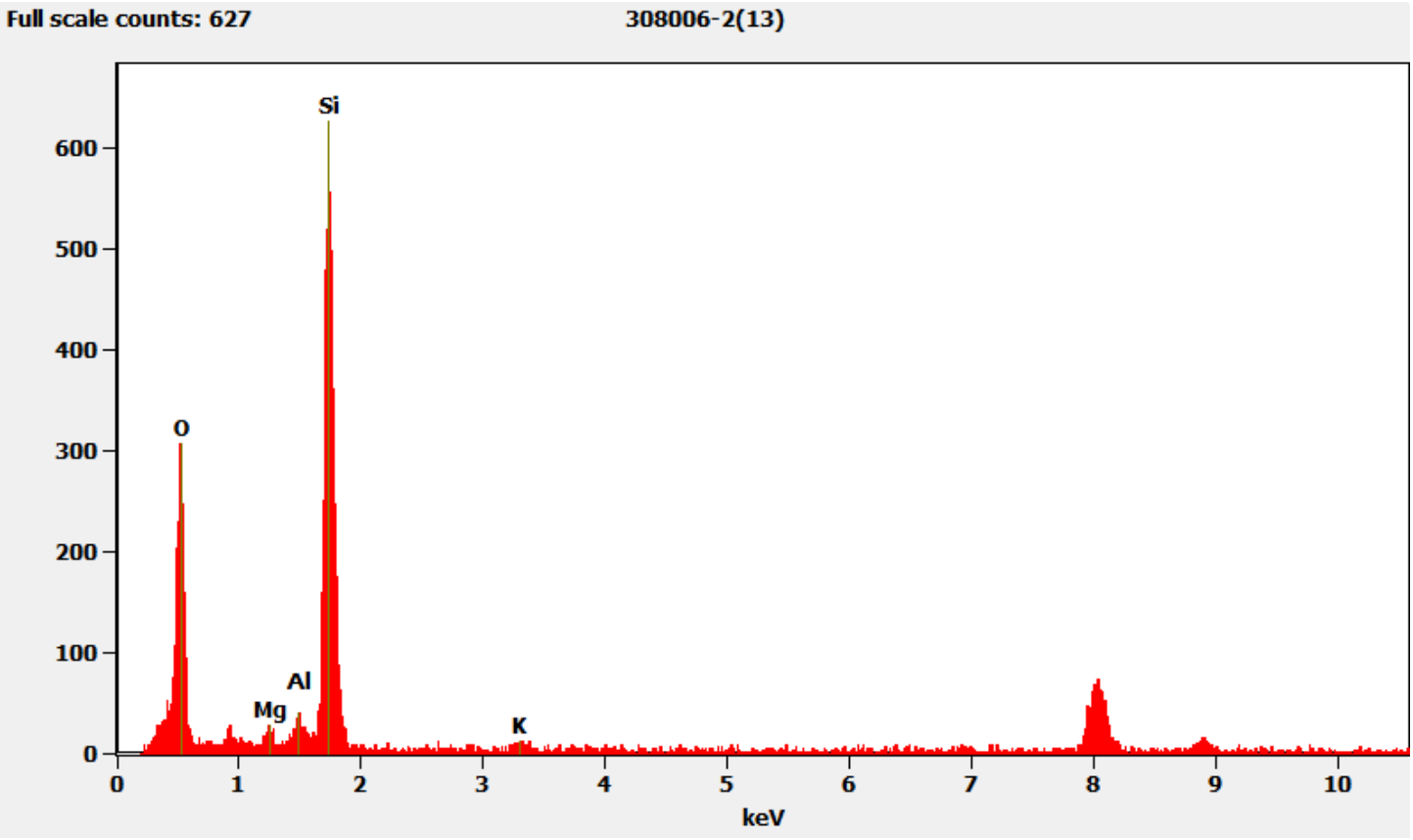
308006-2, Silica Sphere



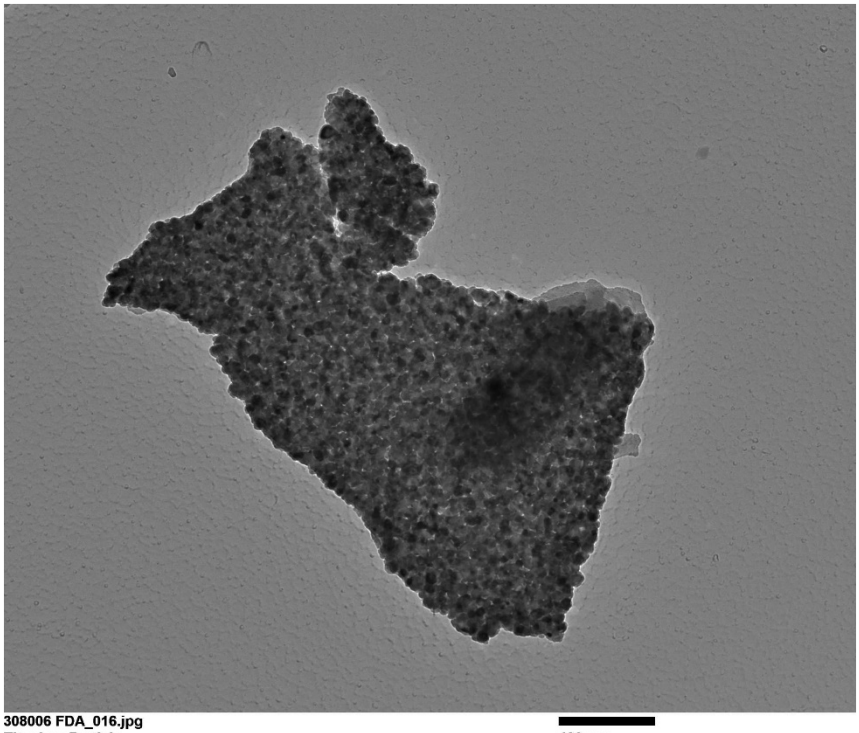
308006 FDA_020.jpg
Silica Sphere
Cal: 0.001429 µm/pix
15:47 8/21/2019
TEM Mode: Imaging
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

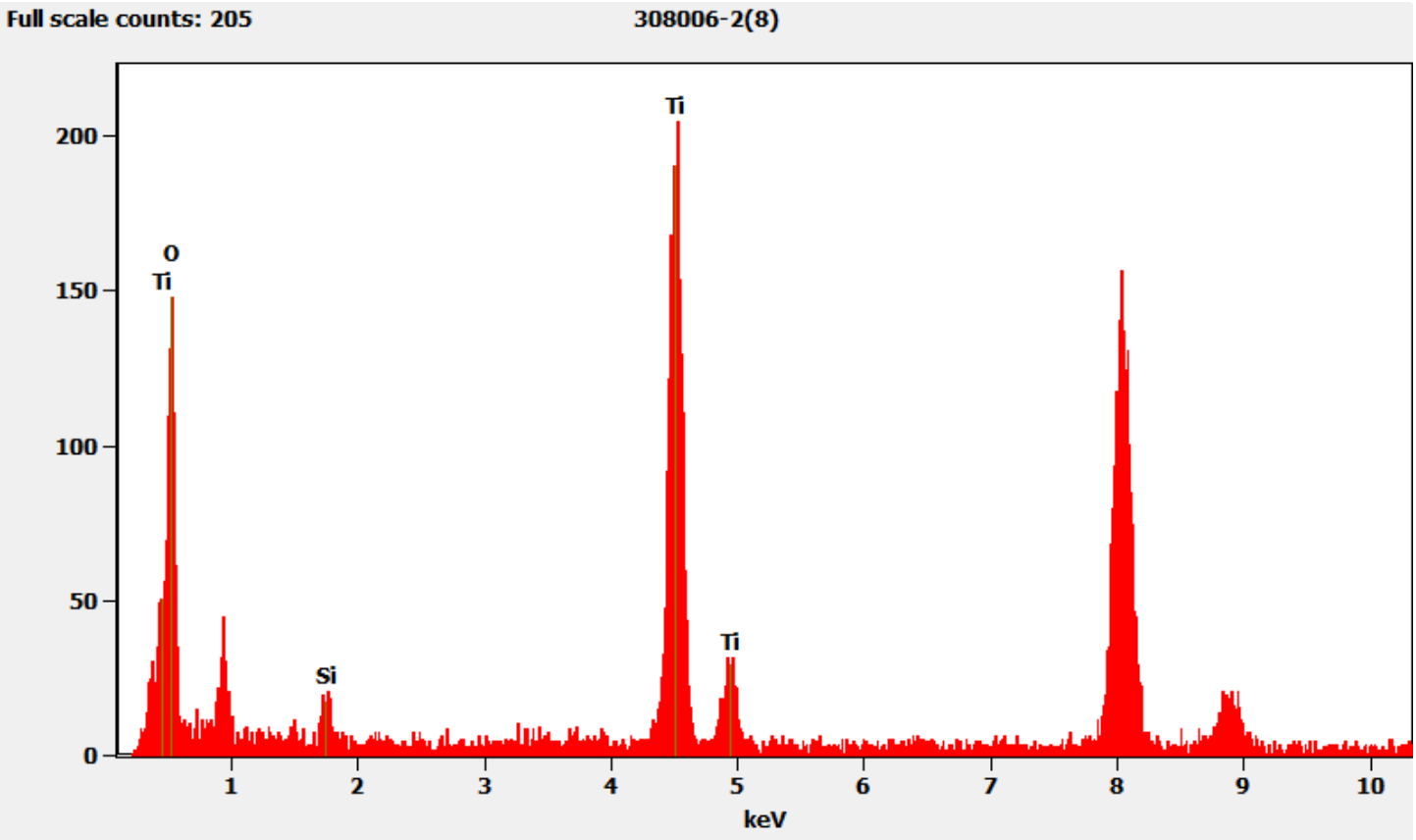
Chemistry from the Silica Sphere pictured above



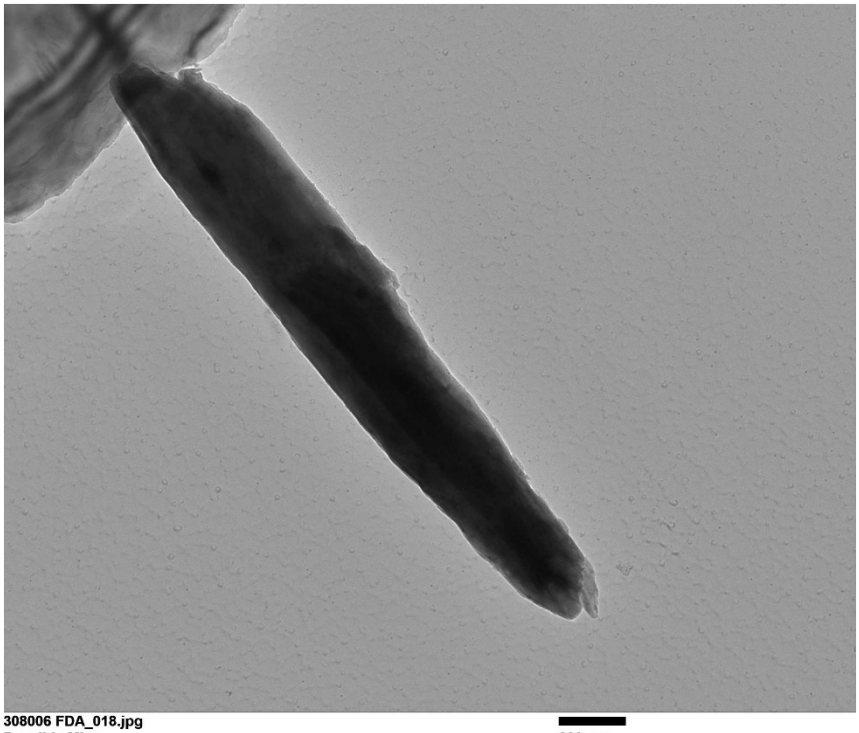
308006-2, Titanium Particle



Chemistry from the Titanium particle pictured above



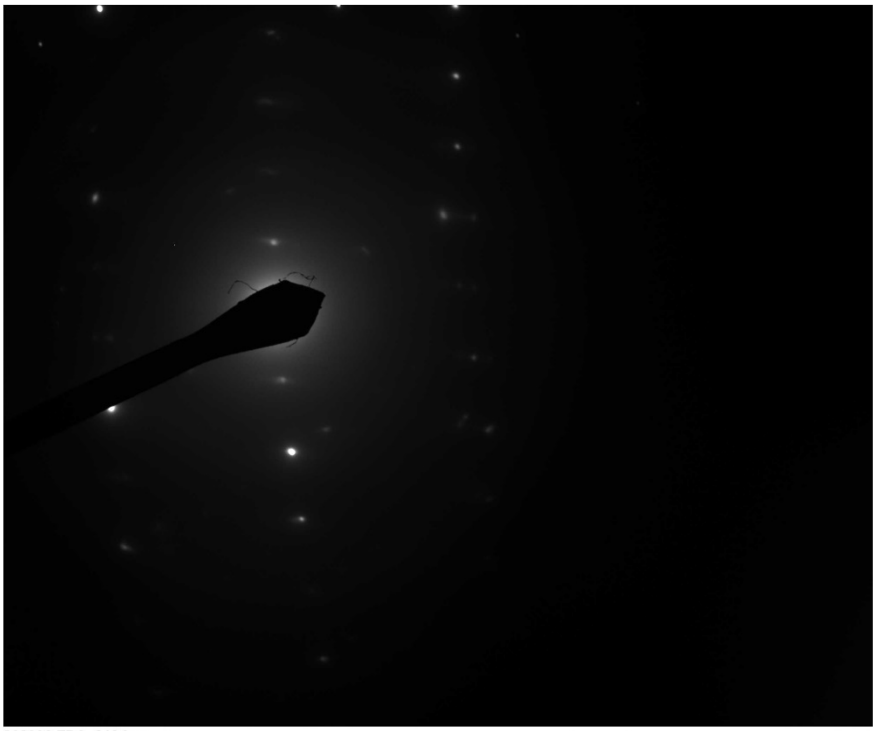
308006-2, Mica Particle



308006 FDA_018.jpg
Possible Mica
Cal: 0.001029 µm/pix
15:30 8/21/2019
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

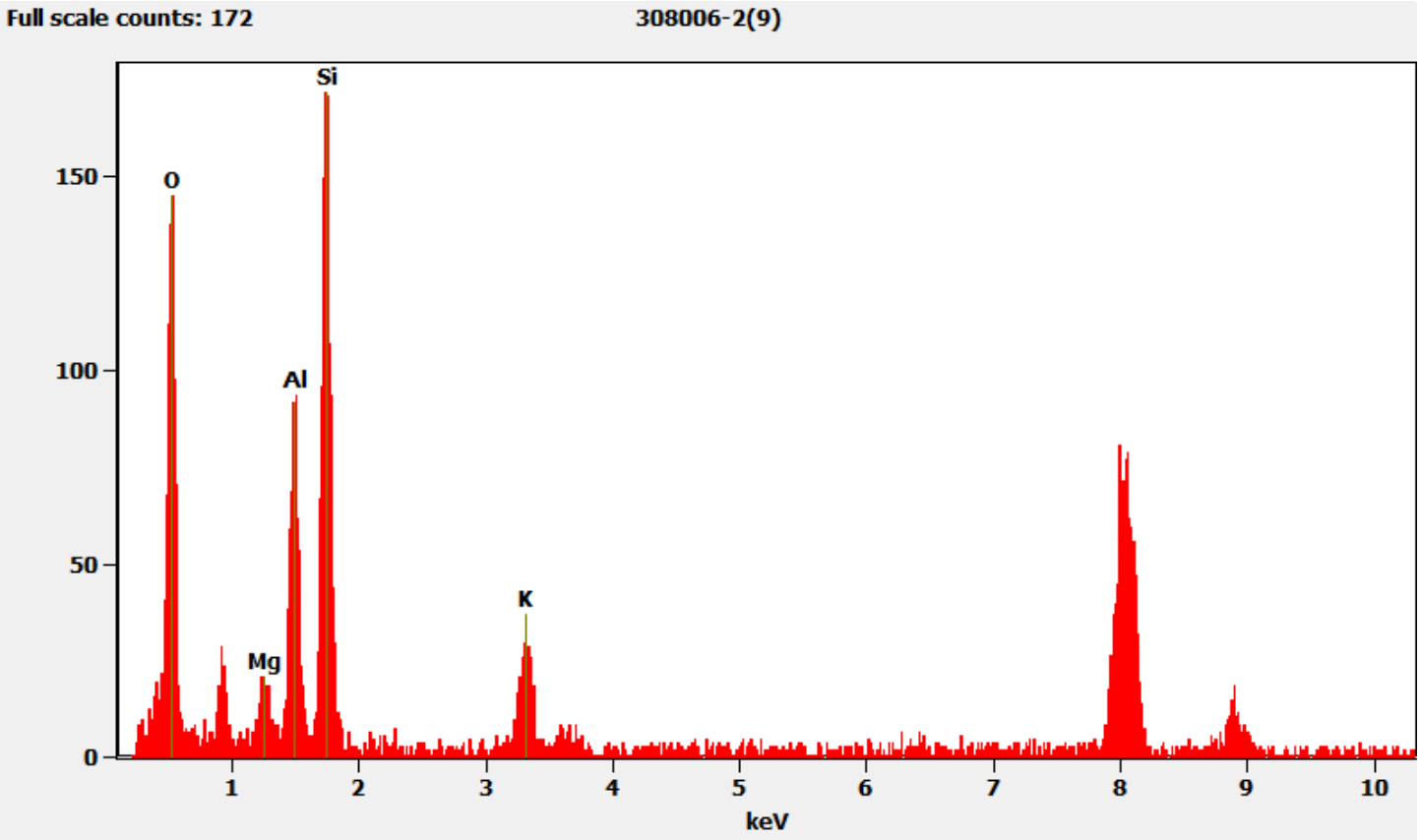
Diffraction pattern from the Mica particle pictured above



308006 FDA_019.jpg
Possible Mica
15:31 8/21/2019
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



<u>308006-3, 3A, 3B, Client Sample D-55</u>

PLM

All three aliquots of sample D-55 were analyzed by (b) (6) on September 13, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

308006-3	NAD
308006-3A	NAD
308006-3B	NAD

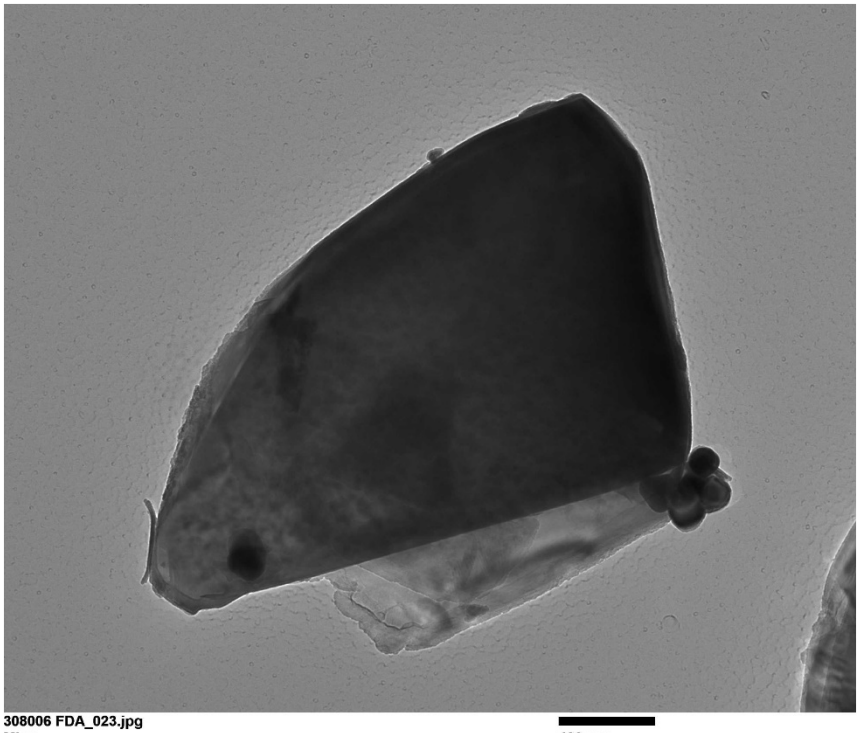
TEM

Sample 3 was analyzed by (b) (6) on August 21 and August 27, 2019. (b) (6) analyzed sample 3A on August 29, 2019 and sample 3B on September 5, 2019. The primary particles observed were mica and talc along with a few talc fibers, titanium particles, titanium coated particles, and silica particles. No asbestos or non-asbestos amphibole variants were detected in the samples. The results were calculated using the equations detailed in the calculations section.

308006-3	NAD
308006-3A	NAD
308006-3B	NAD

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

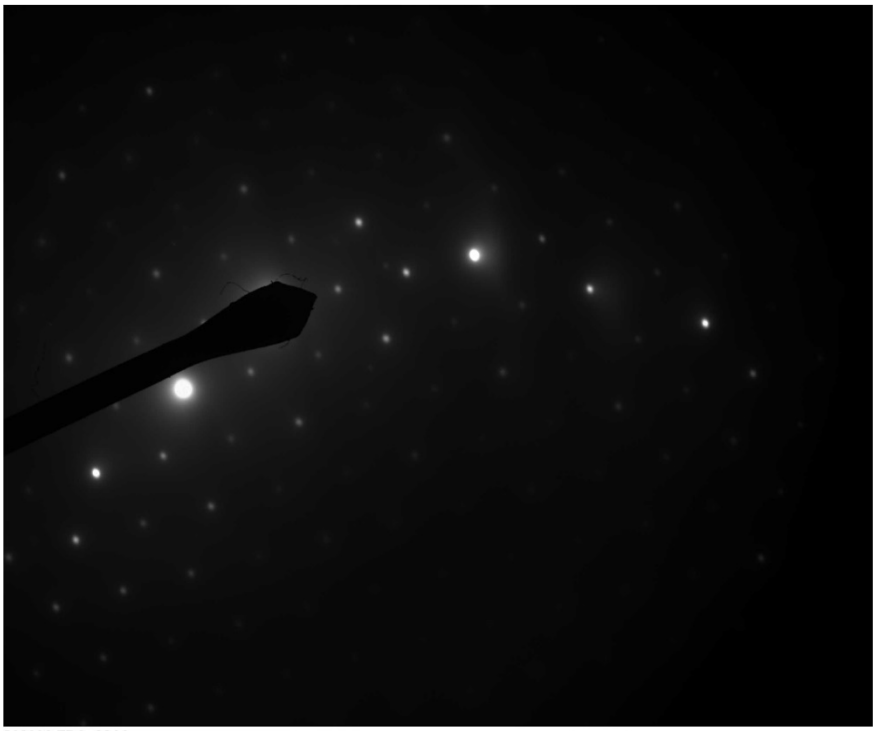
Sample 308006-3, Mica Particle



308006 FDA_023.jpg
Mica
Cal: 0.001429 µm/pix
16:50 8/21/2019
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

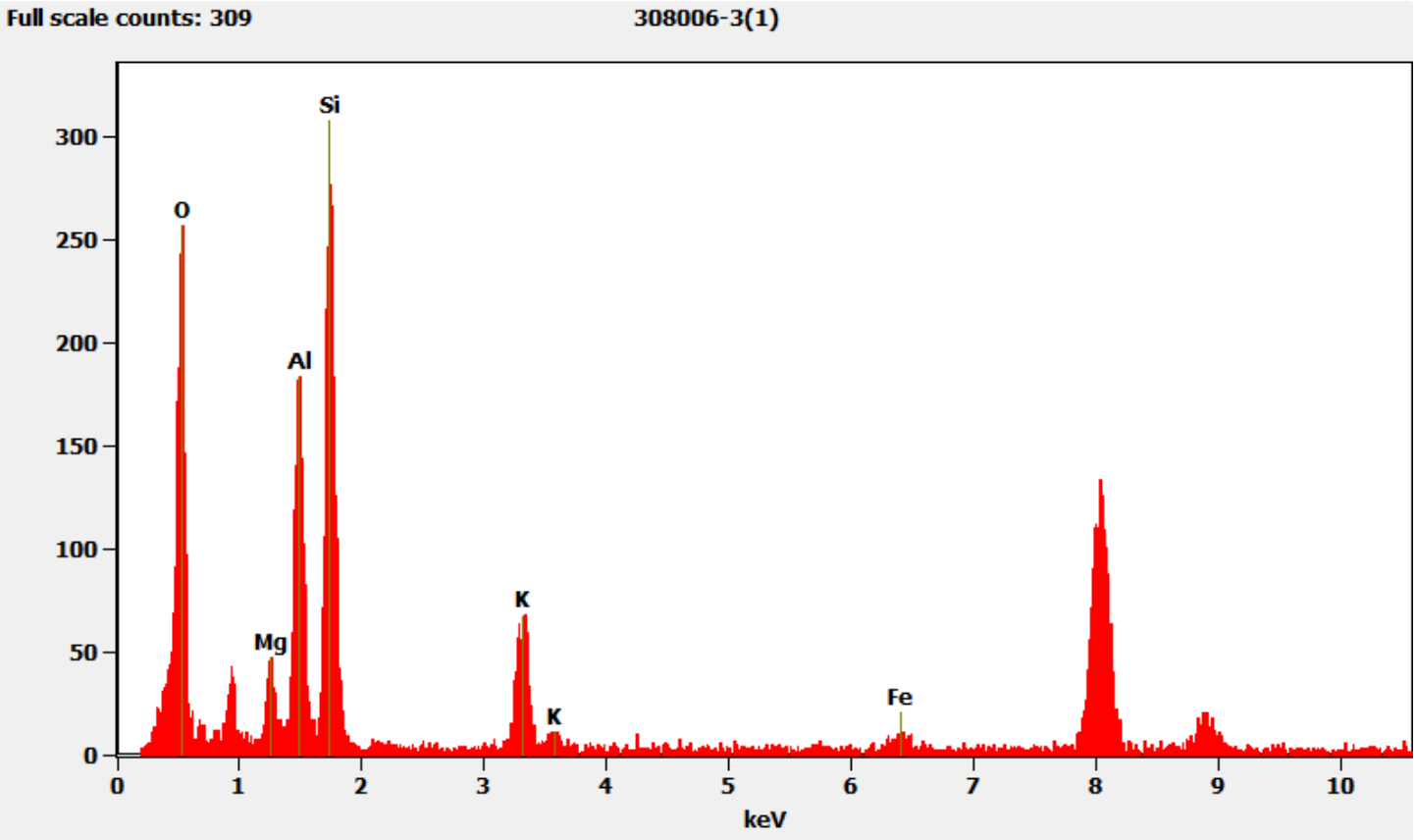
Diffraction pattern from the Mica particle pictured above.



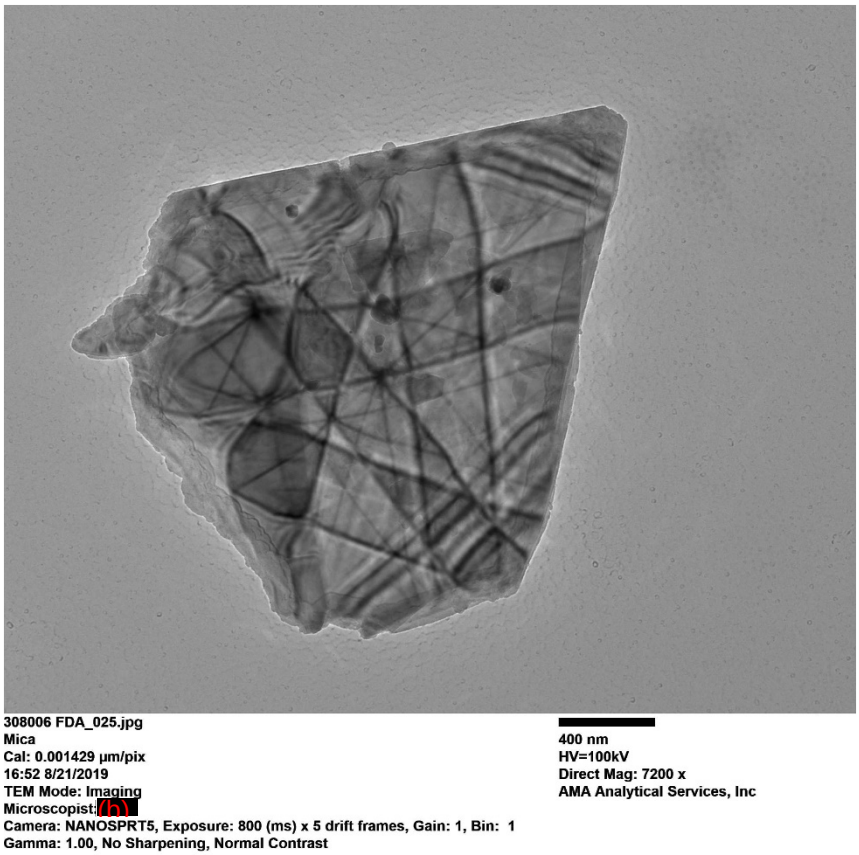
308006 FDA_024.jpg
Mica
16:50 8/21/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Sample 308006-3 Chemistry from the Mica particle pictured above

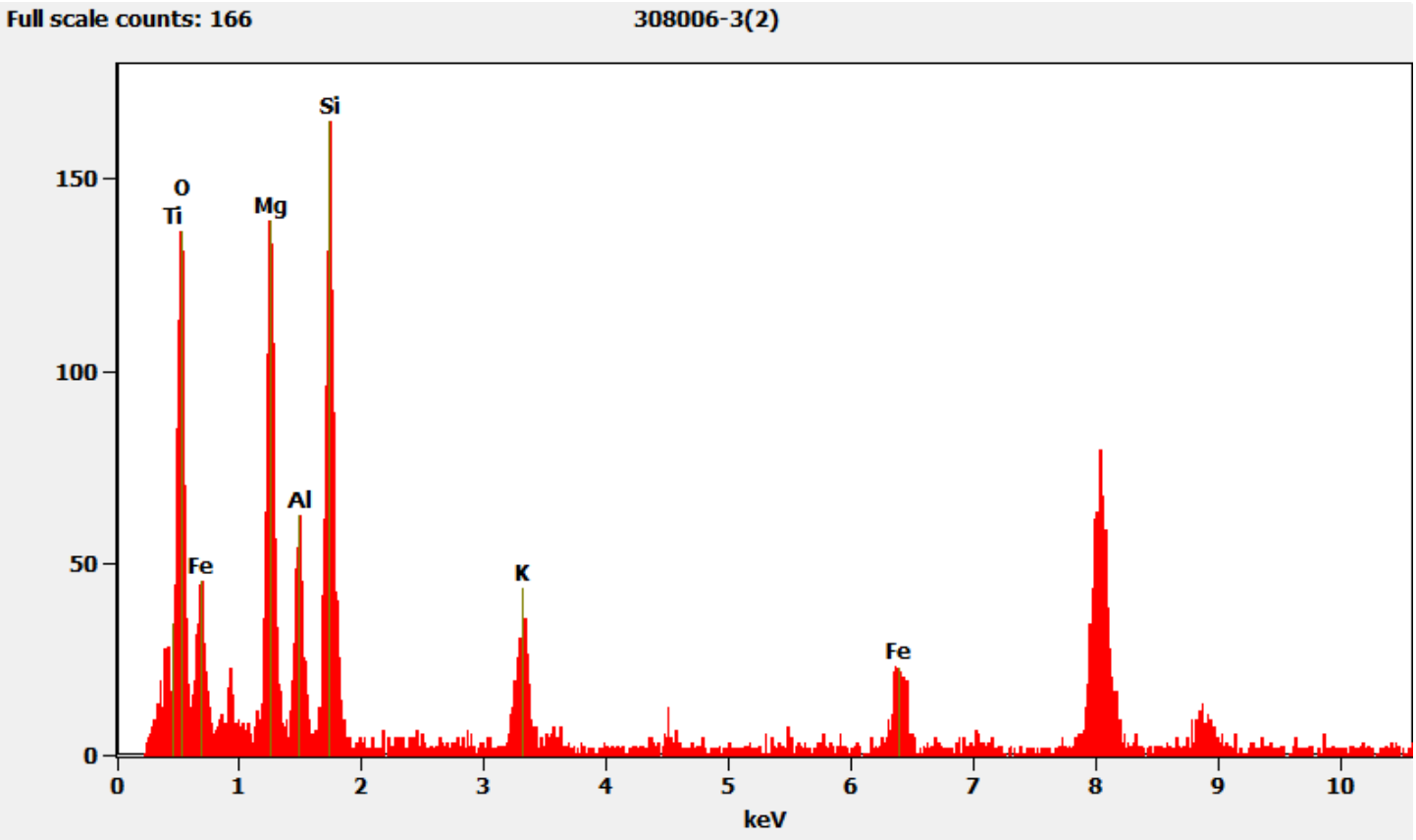


Sample 308006-3, Mica Particle

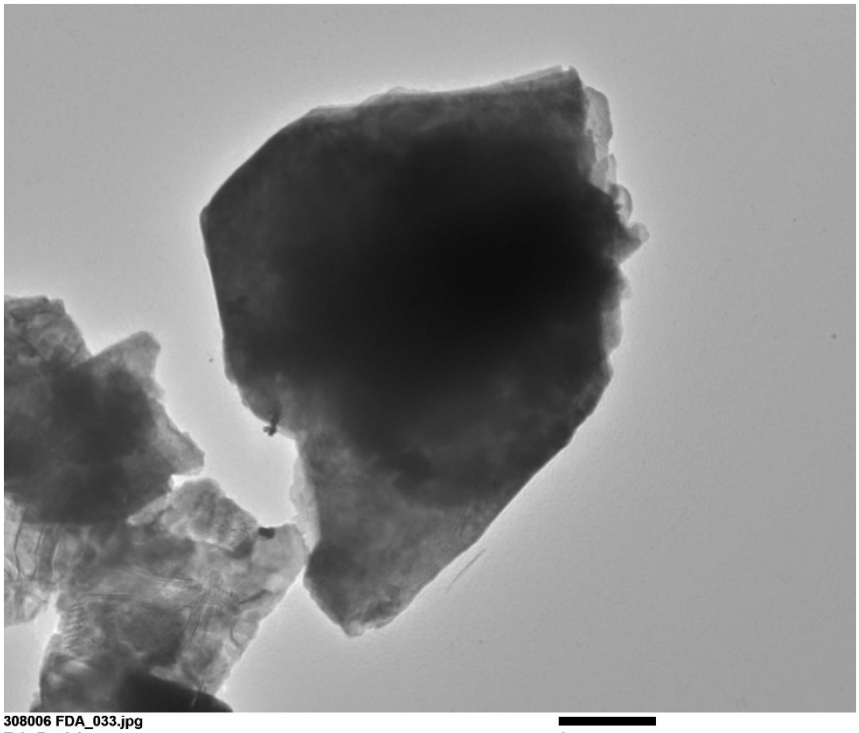


Diffraction pattern from the Mica particle pictured above

Chemistry from the Mica particle pictured above



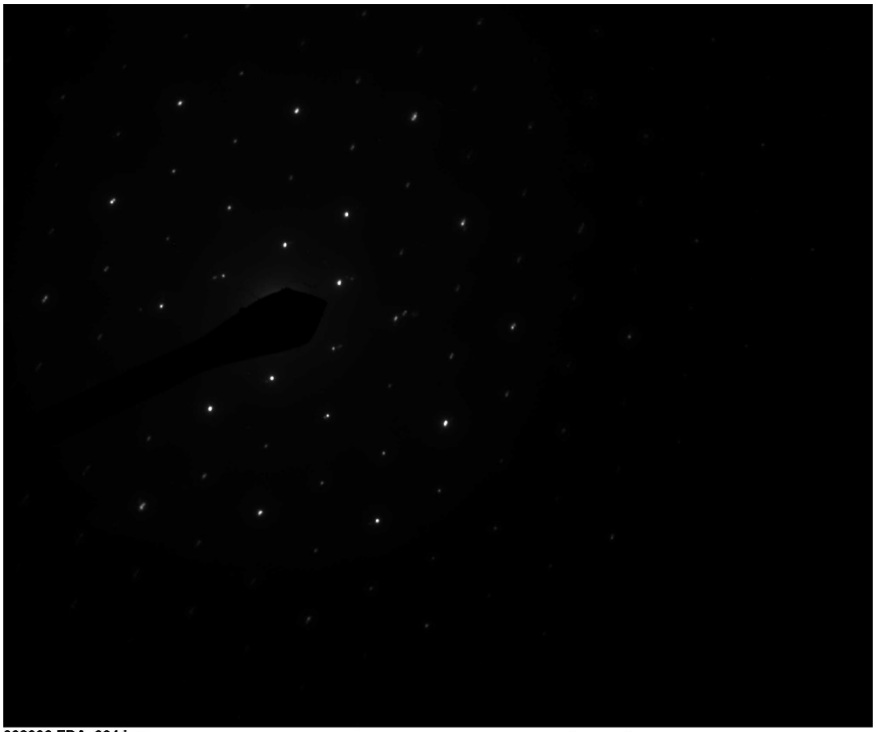
308006-3, Talc Particle



308006 FDA_033.jpg
Talc Particle
Cal: 0.003548 µm/pix
17:28 8/27/2019
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

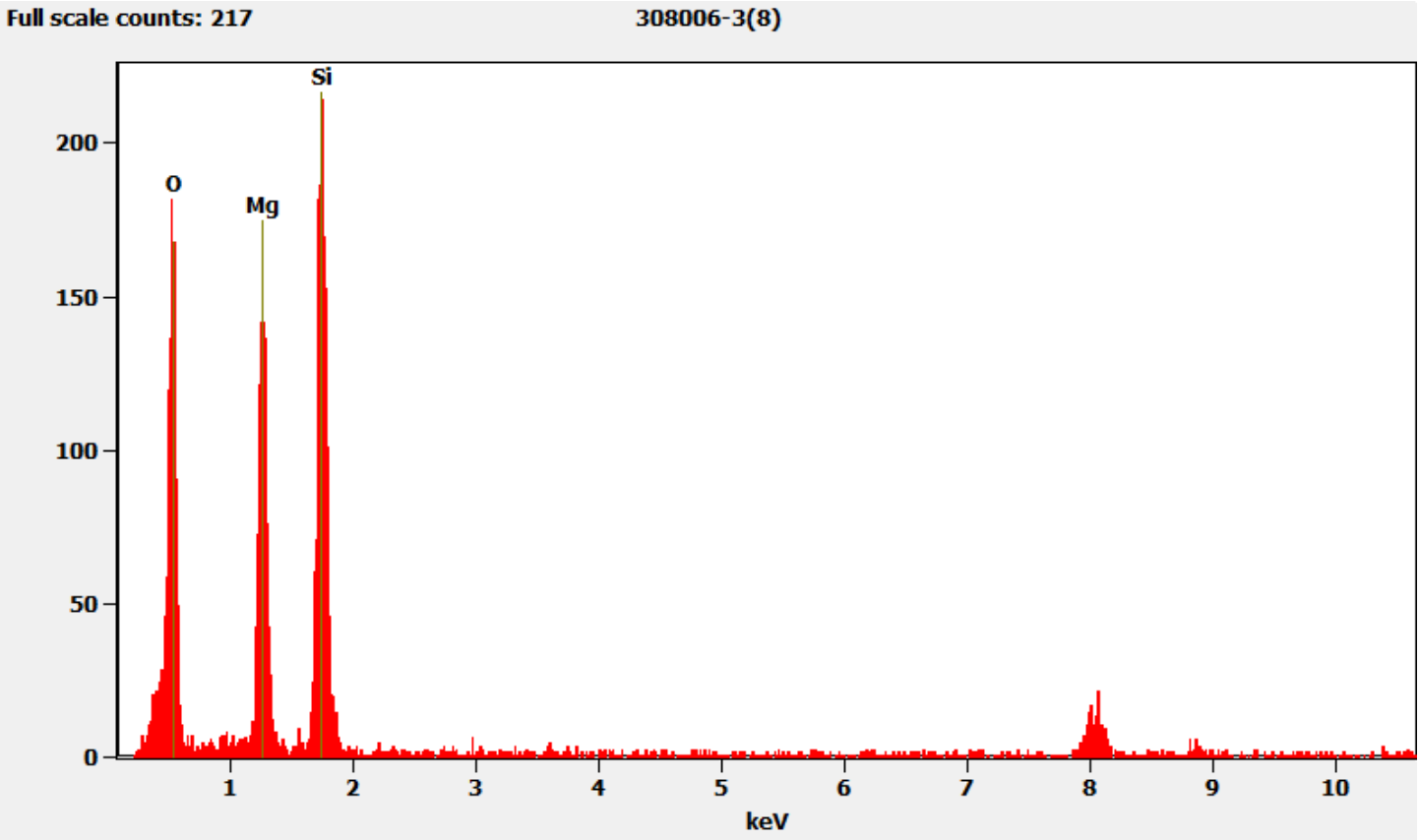
Diffraction Pattern from the Talc particle pictured above



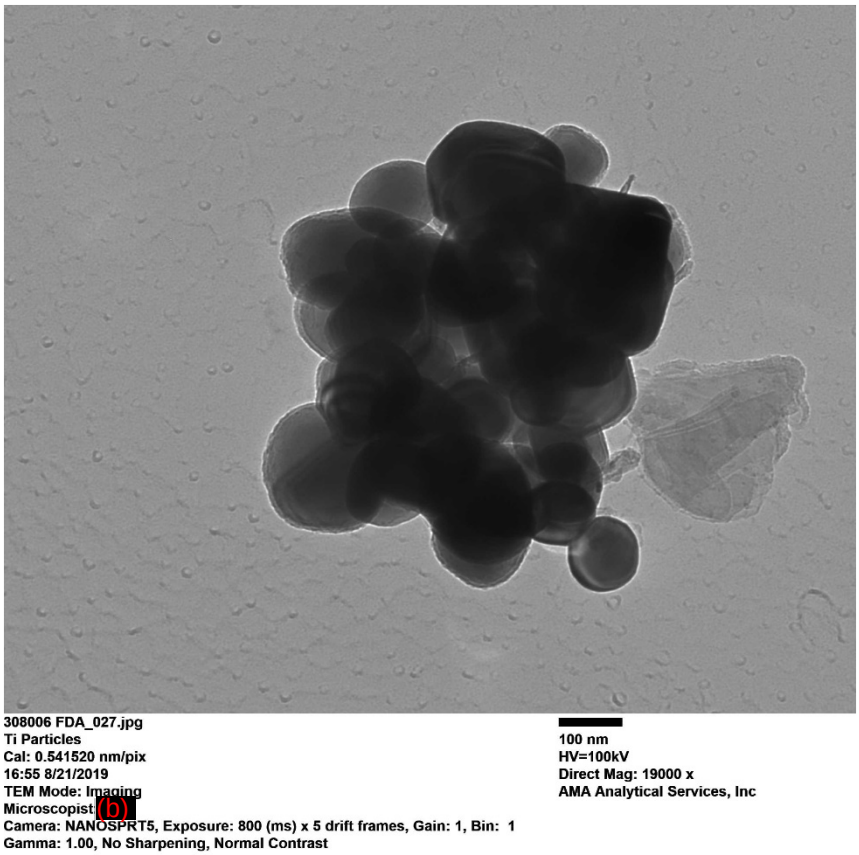
308006 FDA_034.jpg
Talc Particle
17:29 8/27/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

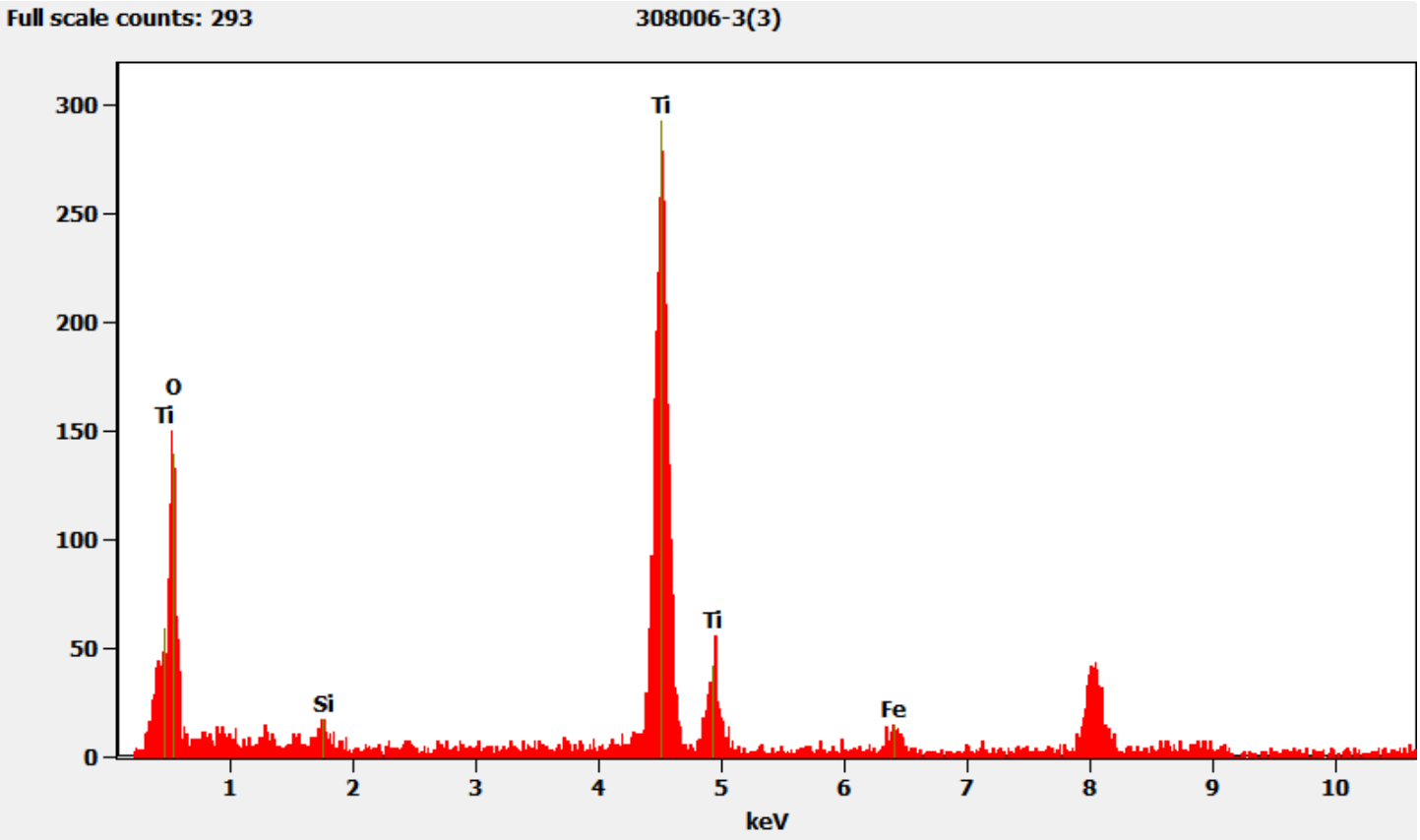
Chemistry from the Talc particle pictured above



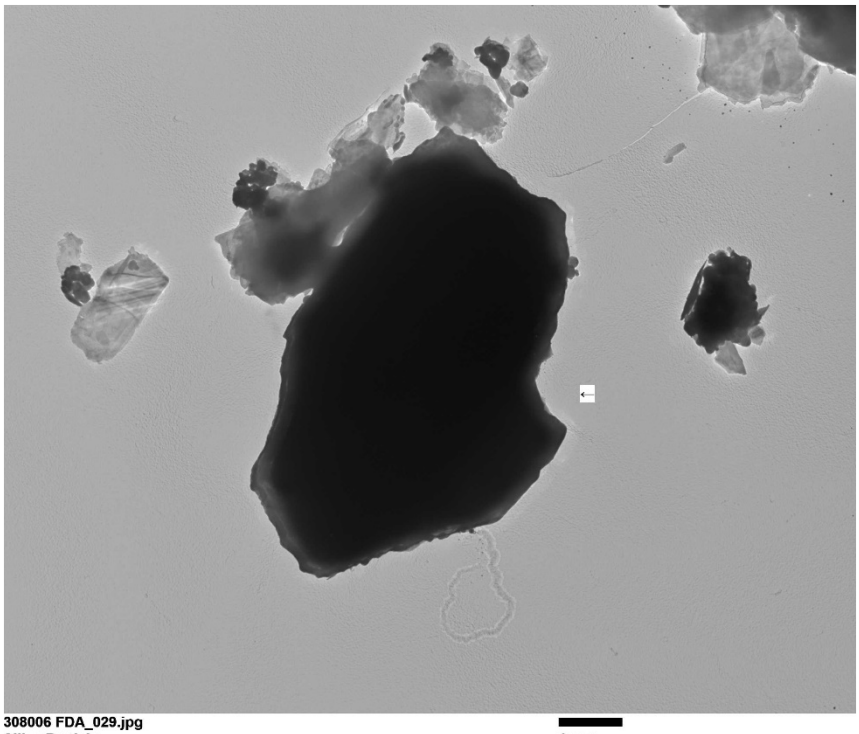
Sample 308006-3, Titanium Particles



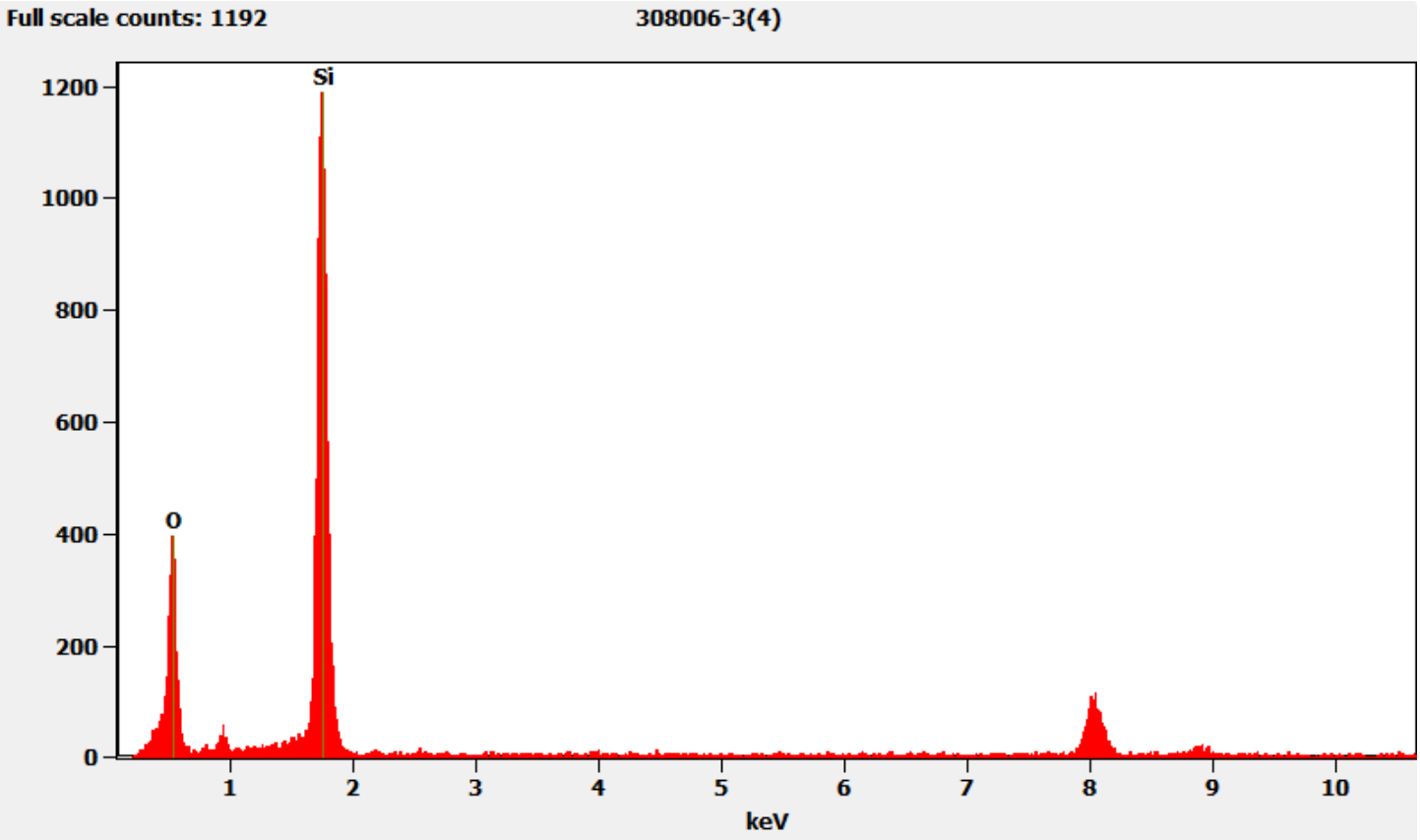
Chemistry from the Titanium particles pictured above



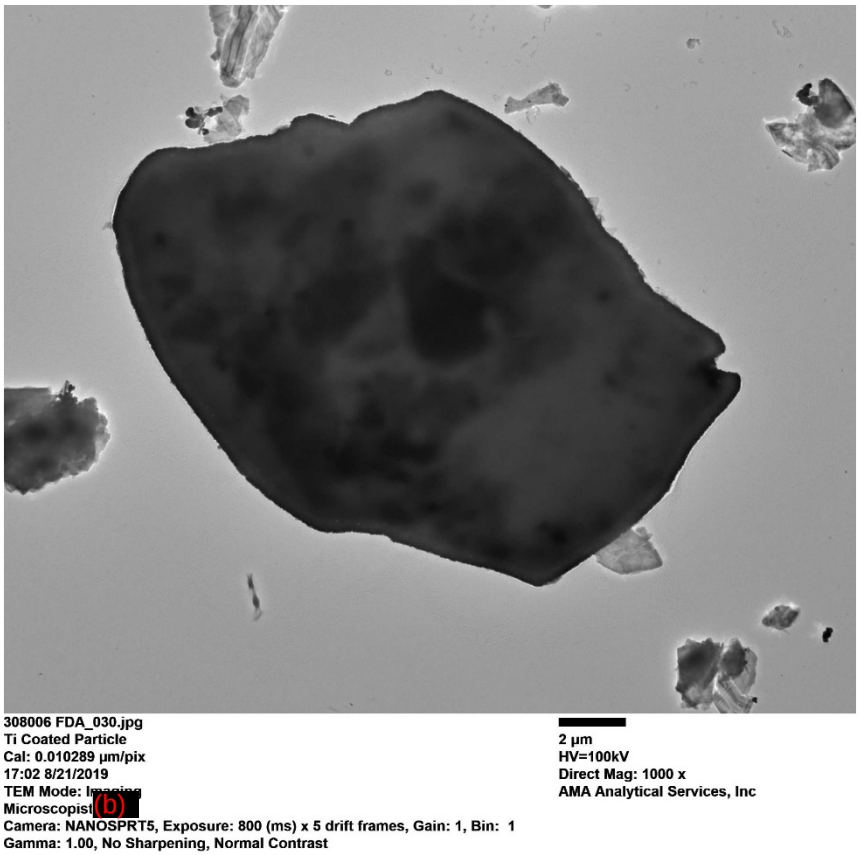
308006-3, Silica Particle



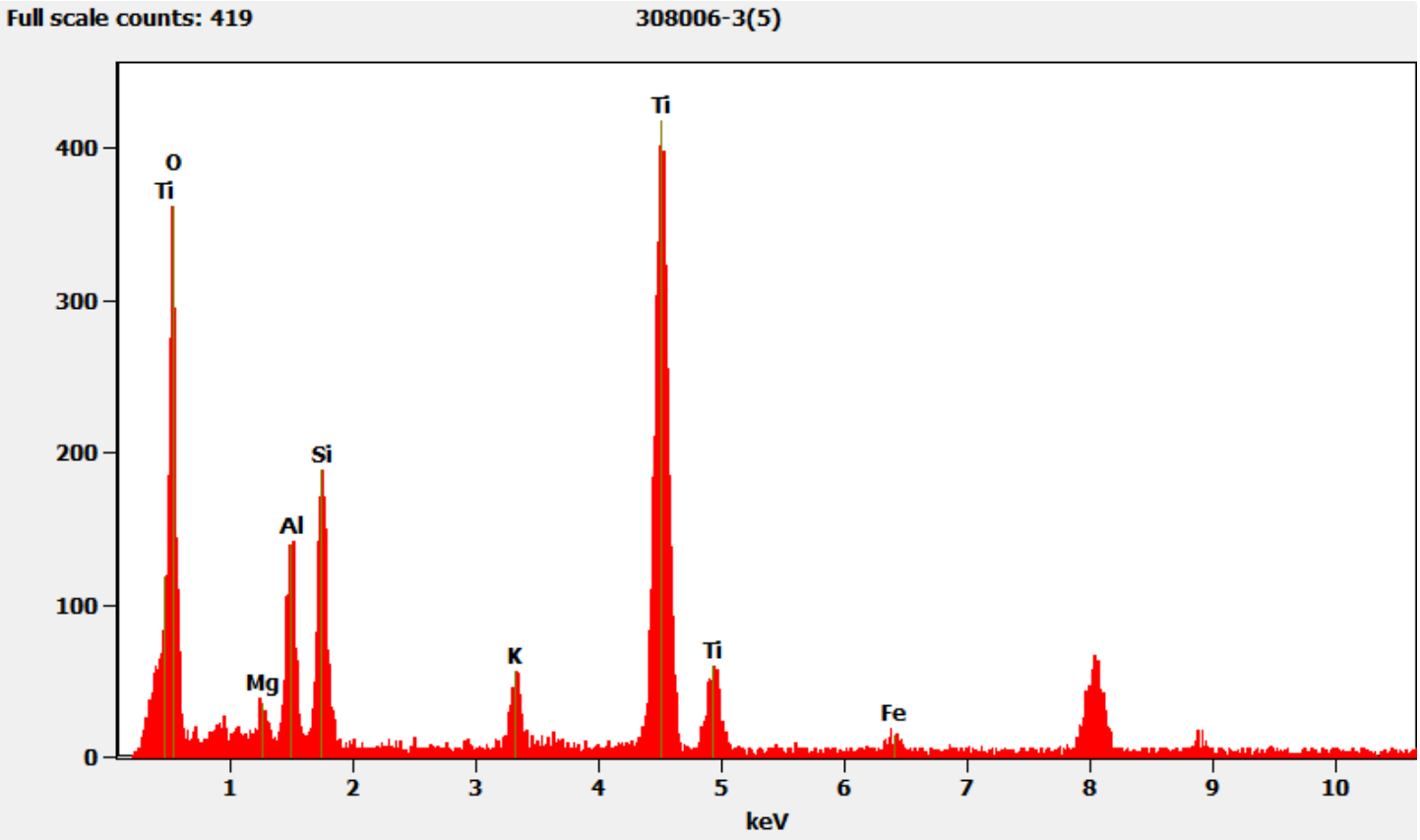
Chemistry from the Silica particle pictured above



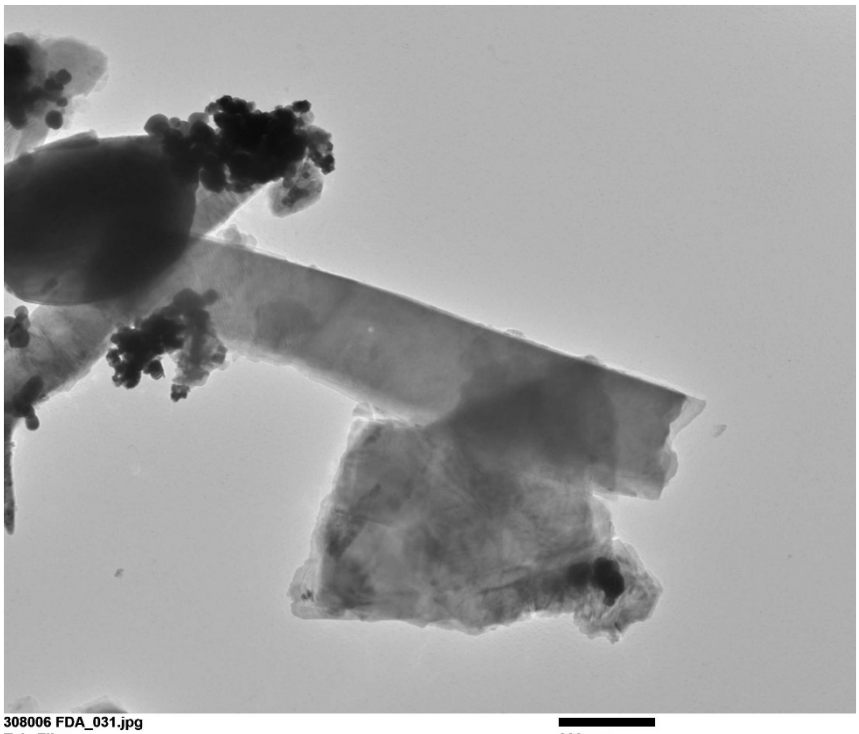
308006-3, Titanium Coated Particle



Chemistry from the Titanium coated particle pictured above



308006-3, Talc Fiber

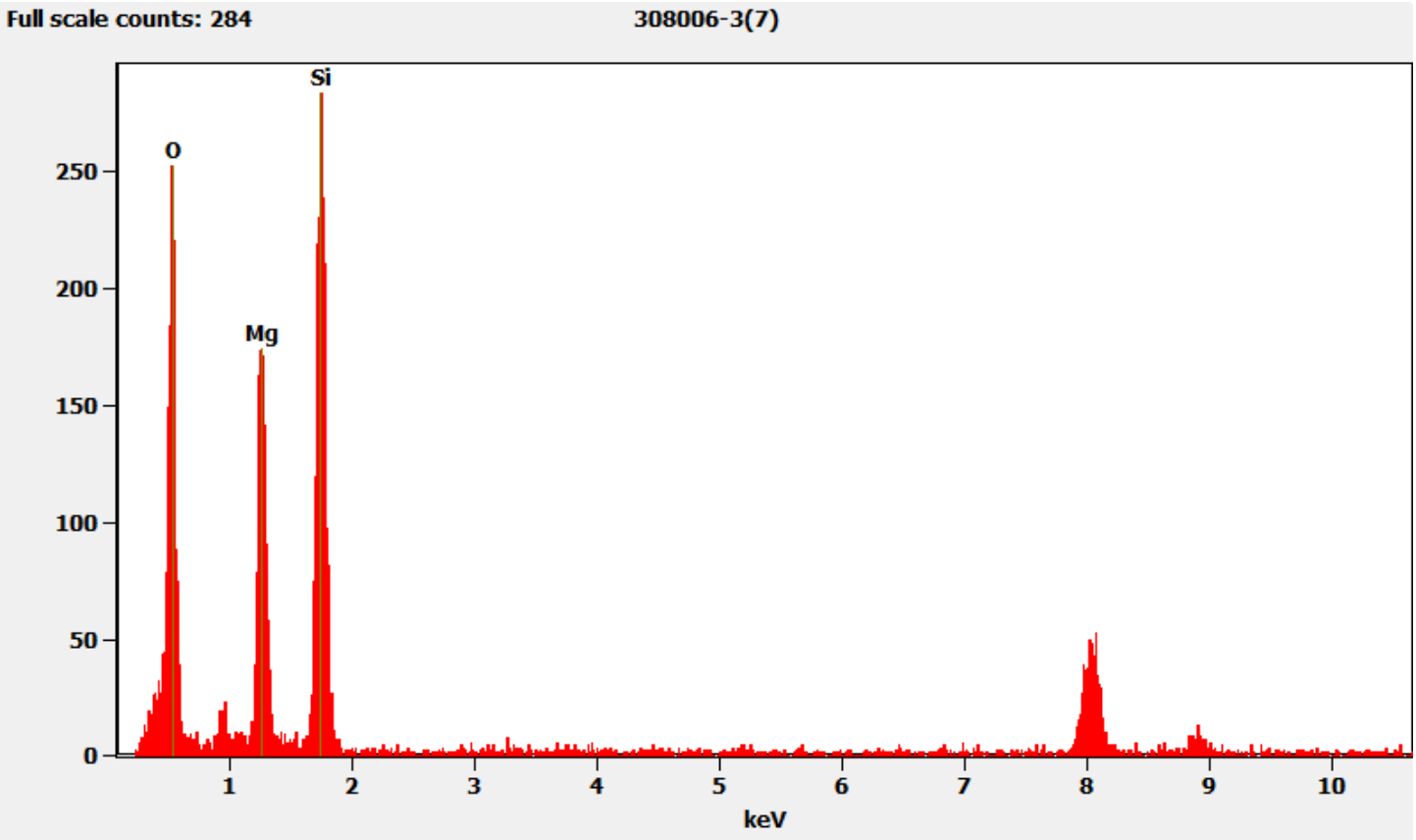


308006 FDA_031.jpg
Talc Fiber
Cal: 0.002858 µm/pix
17:25 8/27/2019
TEM Mode: Imaging
Microscopist: (b) (6)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Diffraction pattern from the Talc fiber pictured above

Chemistry from the Talc fiber pictured above



308006-4, 4A, 4B, Client Sample D56

PLM

All three aliquots of sample D56 were analyzed by (b) (6) on September 13, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

308006-4	NAD
308006-4A	NAD
308006-4B	NAD

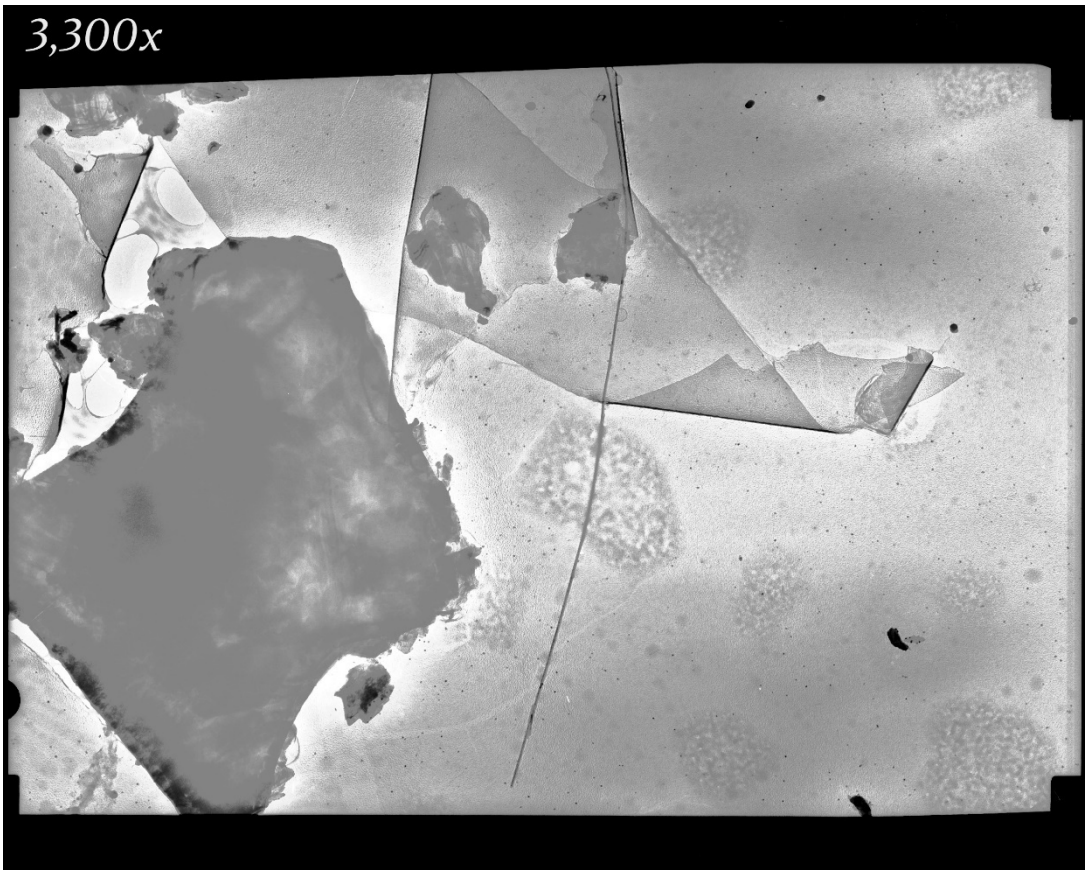
TEM

Sample 4 was analyzed by (b) (6) on August 28, 2019. (b) (6) analyzed samples 4A and 4B on September 5, 2019. The primary particles observed were mica and talc along with a few mica fibers, titanium particles, silica particles/spheres and other titanium coated particles. A chrysotile structure was observed on aliquot 4A. The results were calculated using the equations detailed in the calculations section.

308006-4	NAD
308006-4A	<0.00013%
308006-4B	NAD

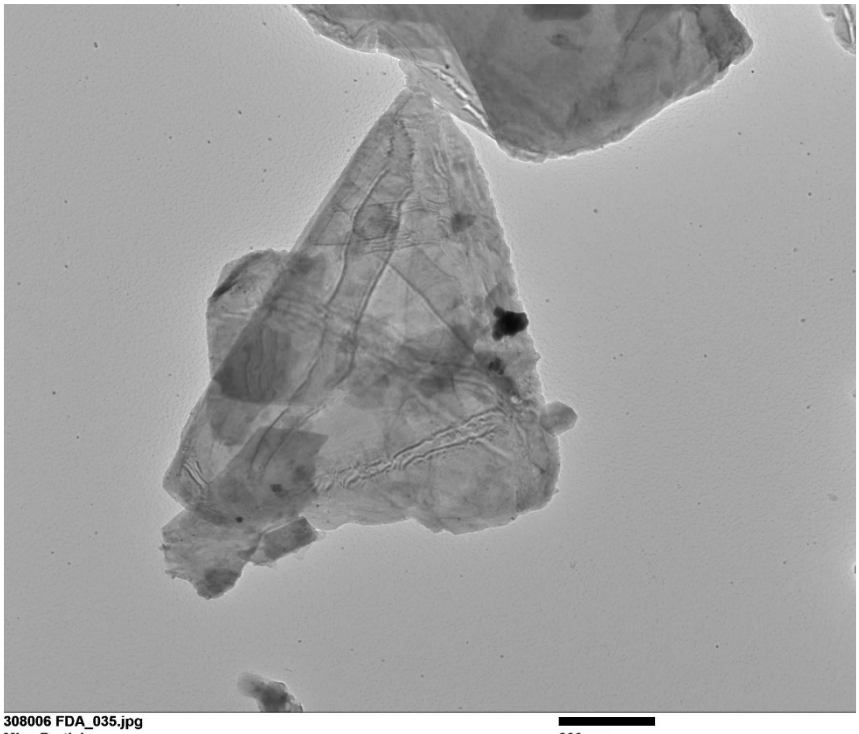
Below are pictures, diffraction patterns, and chemistry from some of the observed particles. The unidentified peaks in chemistry spectra are copper, zinc, and carbon. Those peaks are from the TEM specimen holder and specimen grid.

Sample 308006-4A Chrysotile Fiber *(the image below was taken on film; the digital version was scanned from a negative)*



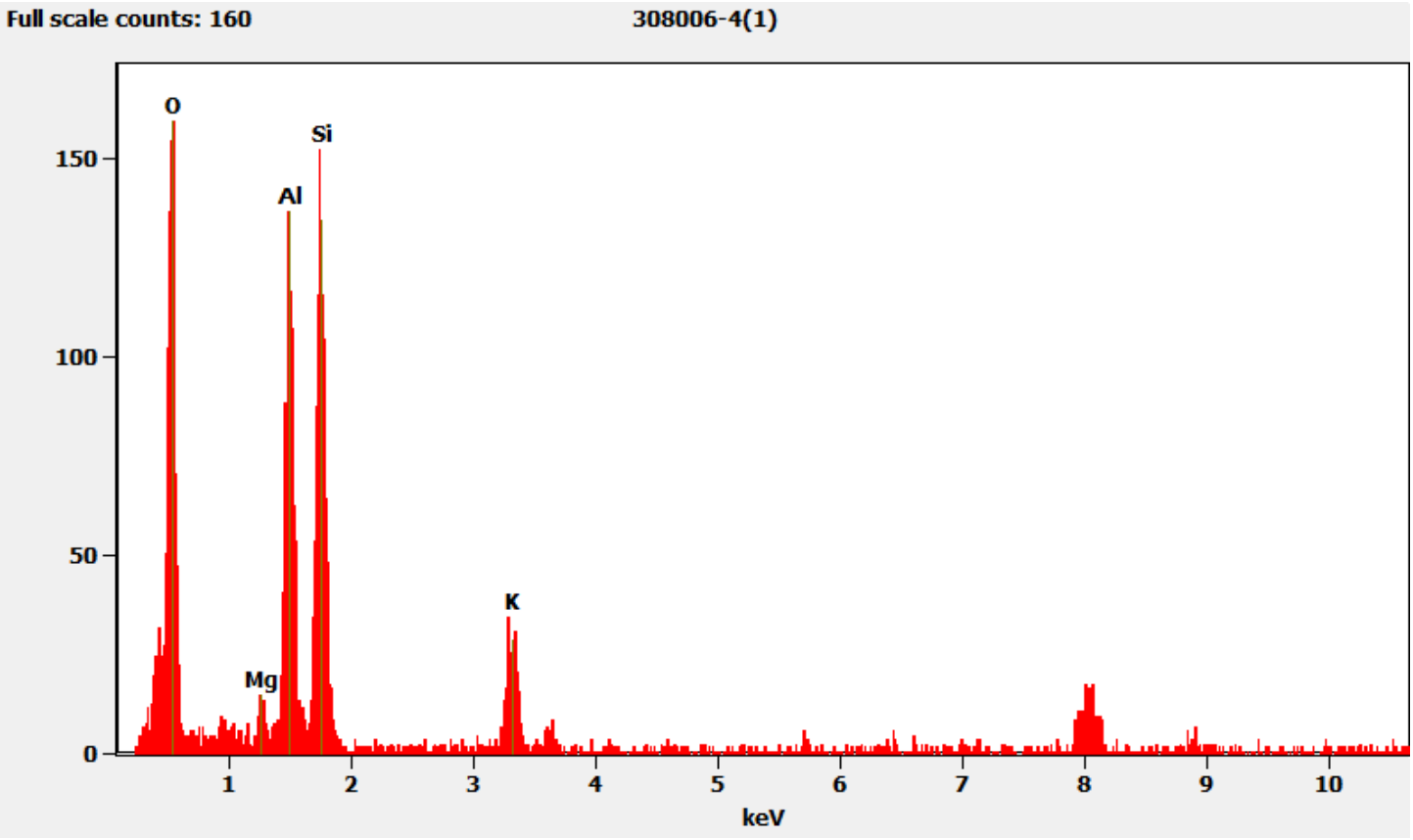
Diffraction Pattern from the chrysotile fiber pictured above. *(the image below was taken on film; the digital version was scanned from a negative)*

Sample 308006-4, Mica Particle

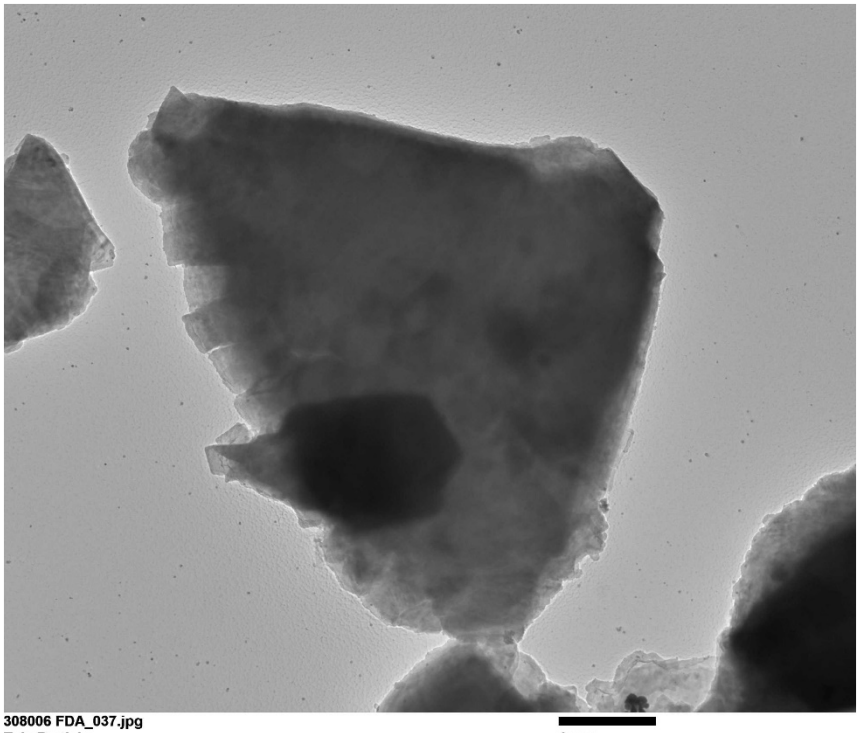


Diffraction pattern from the Mica particle pictured above.

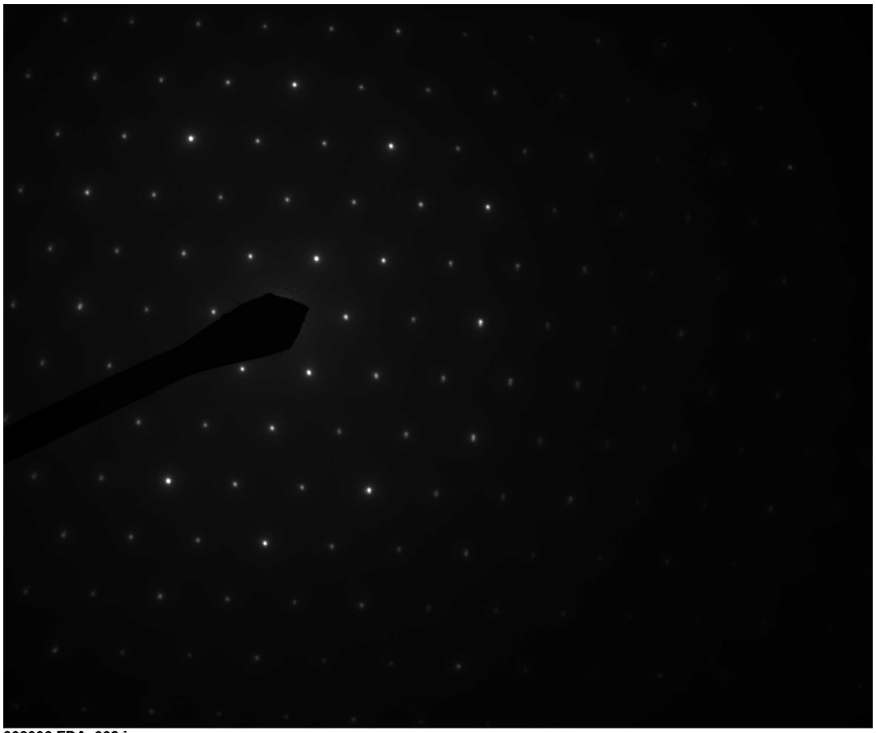
Chemistry from the Mica particle pictured above.



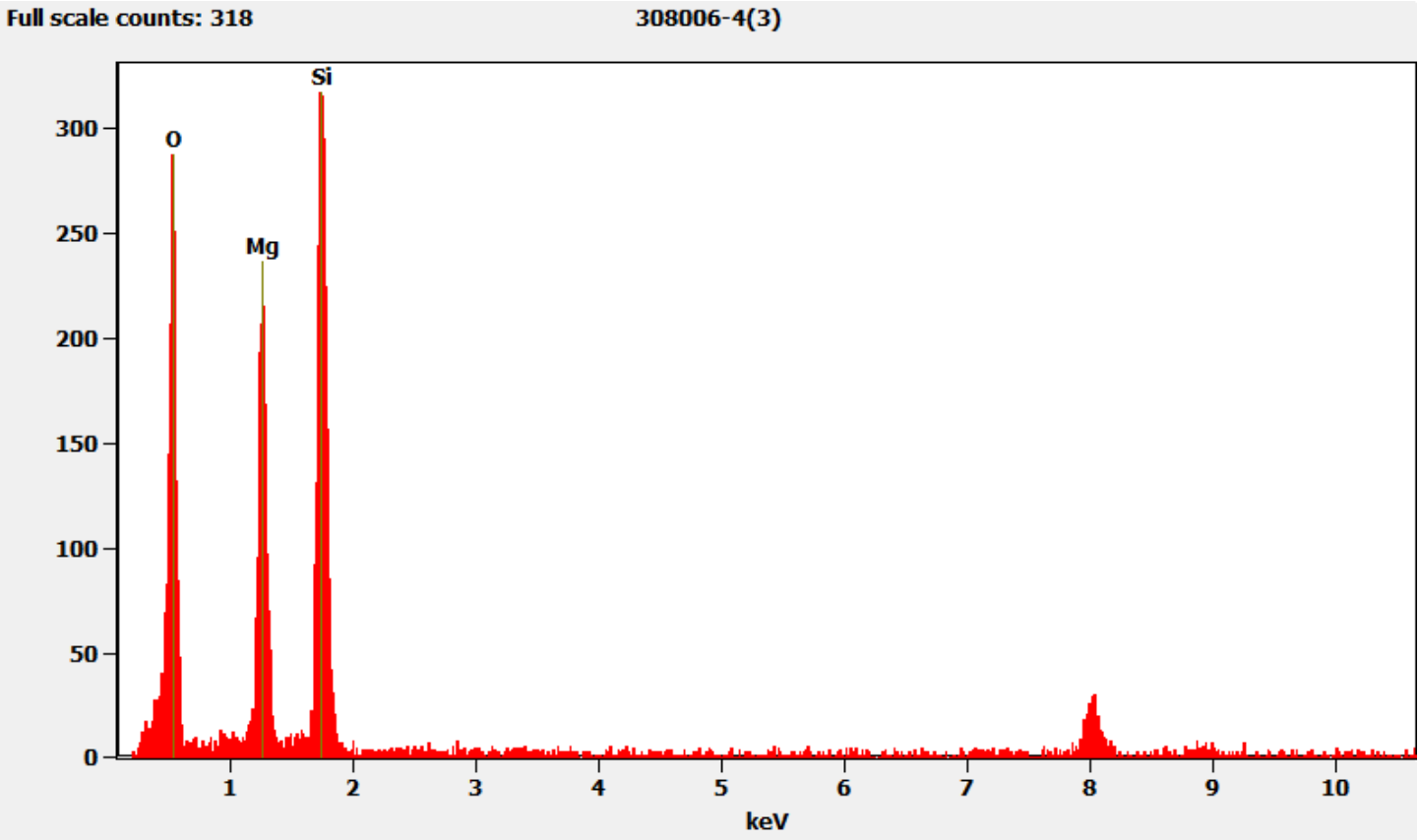
Sample 308006-4, Talc Particle



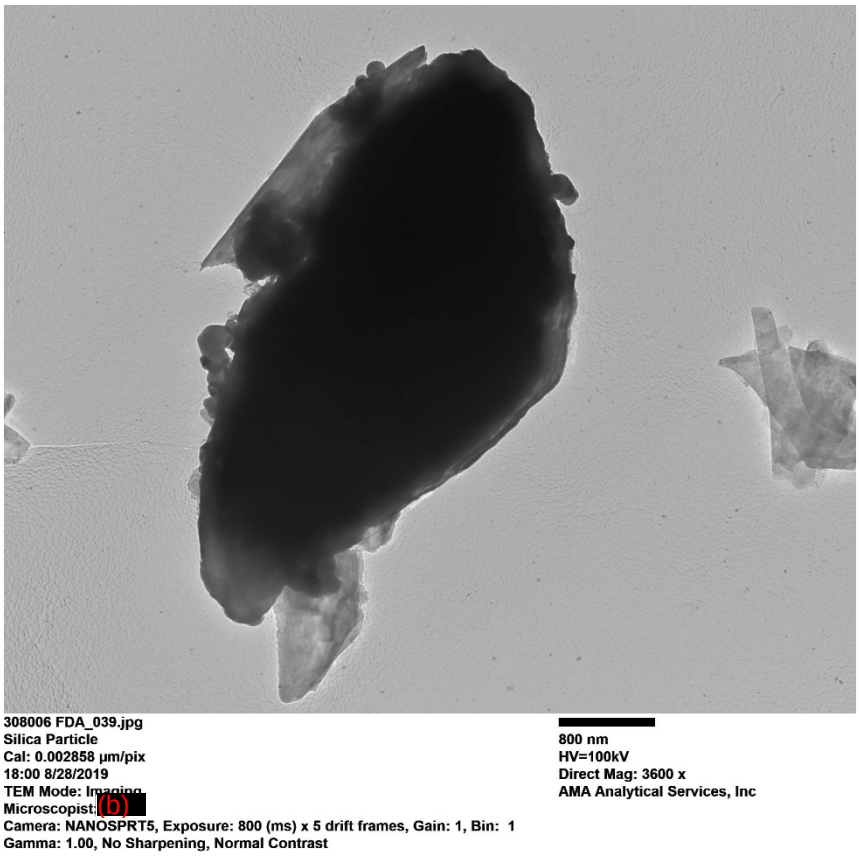
Hexagonal diffraction pattern from the Talc particle pictured above.



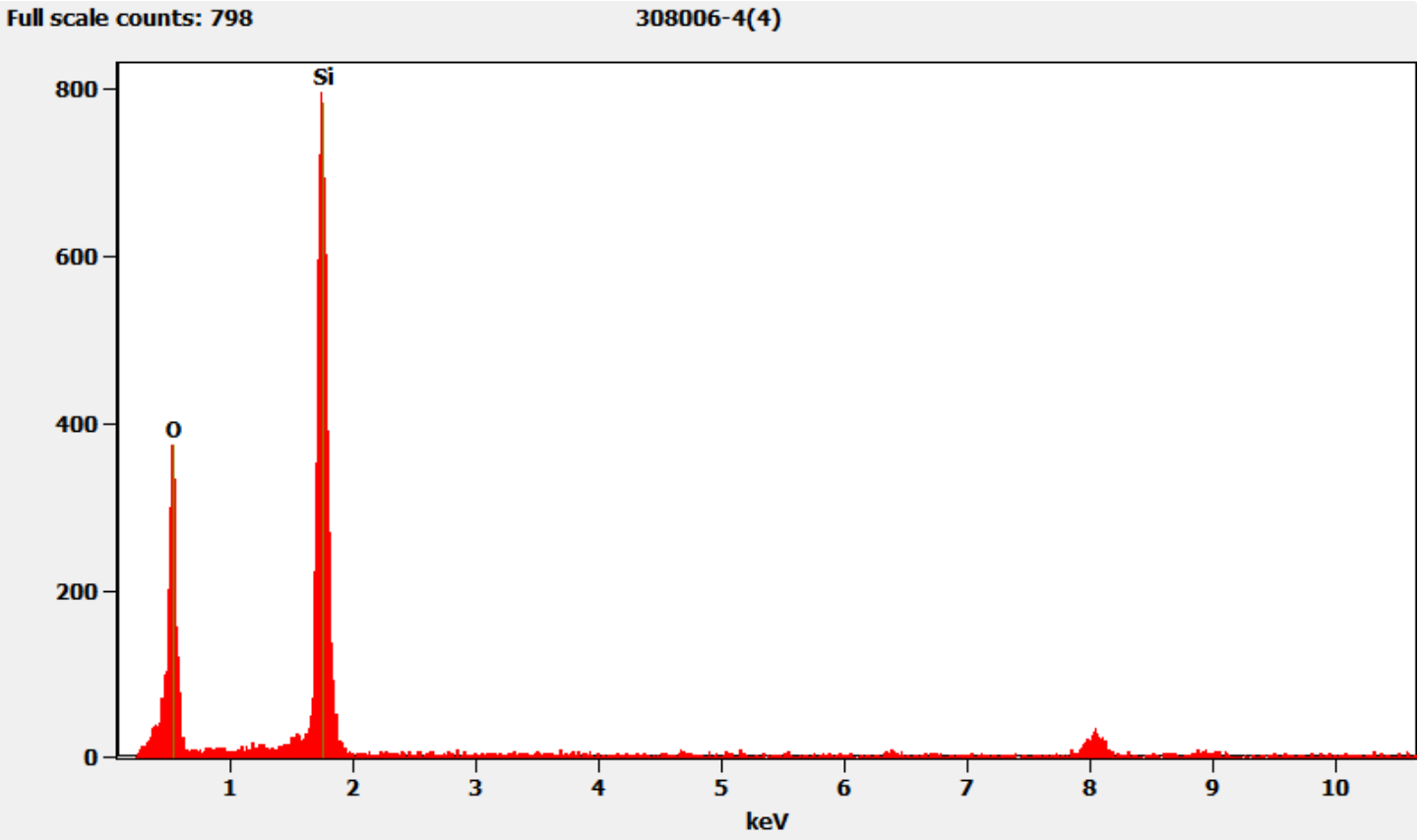
Chemistry from the Talc particle pictured above



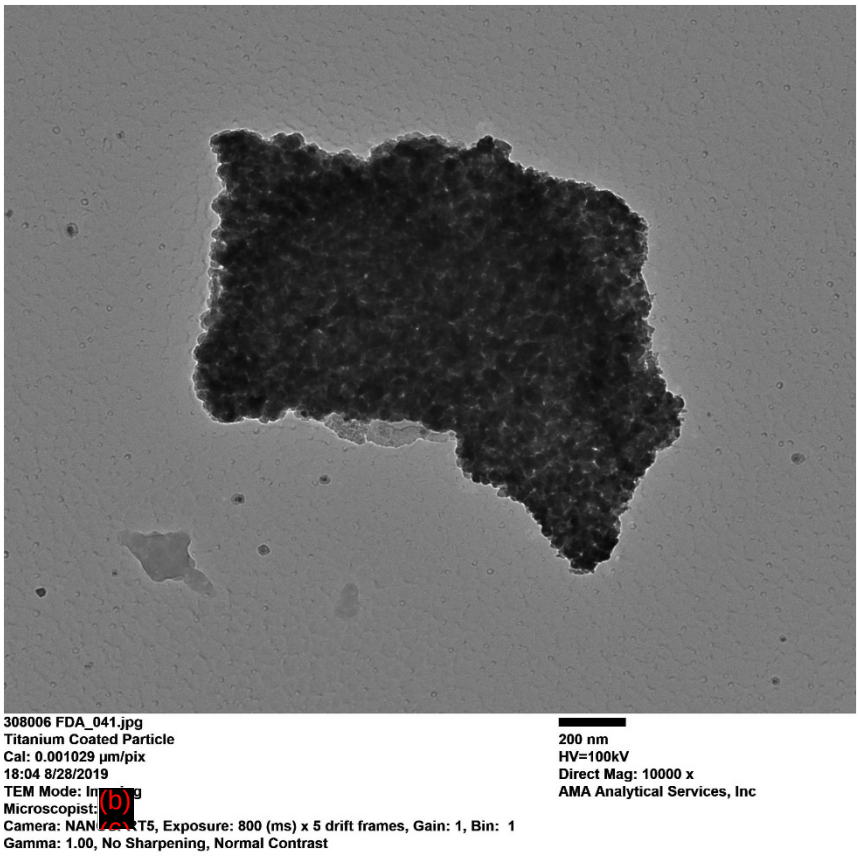
Sample 308006-4, Silica Particle



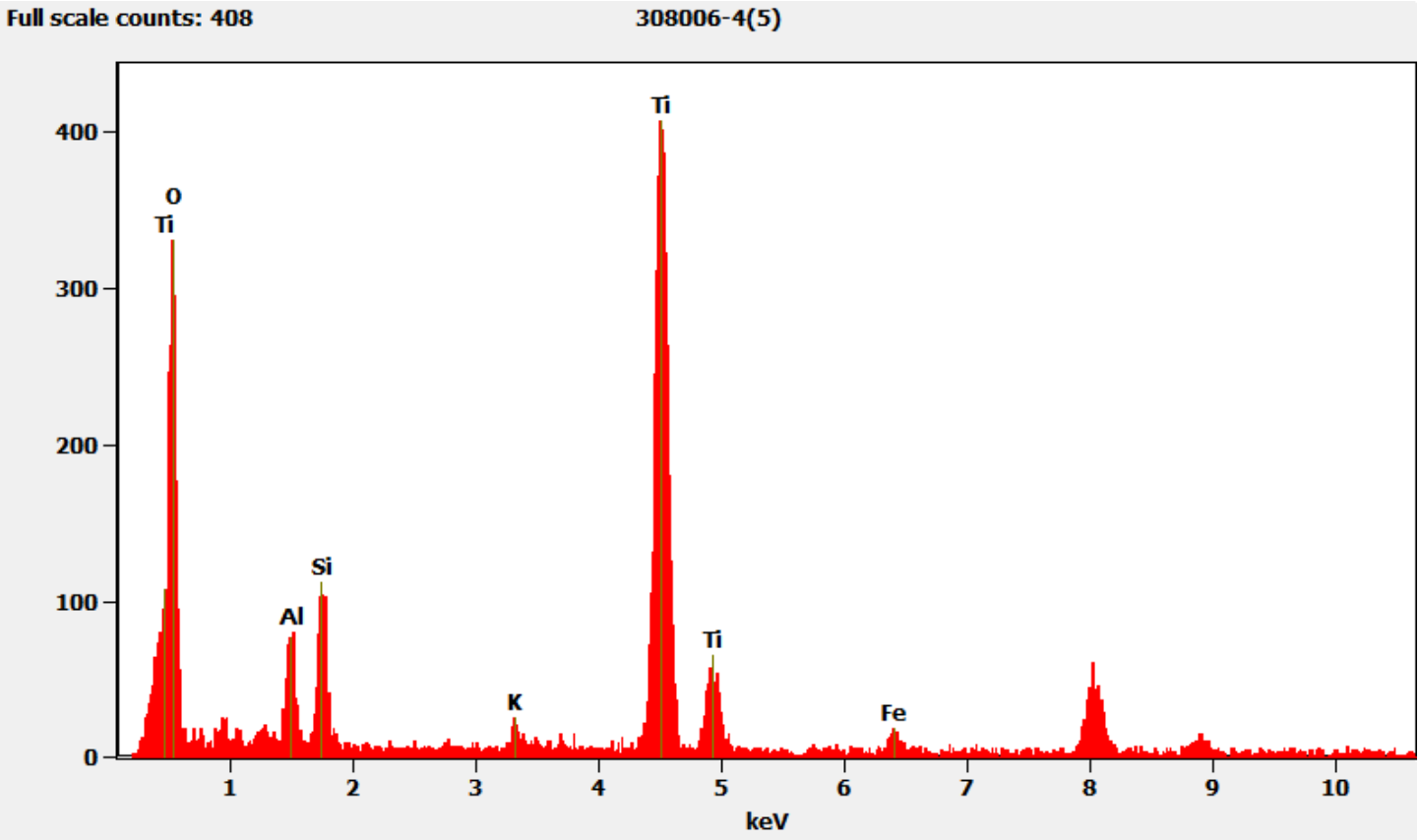
Chemistry from the Silica particle pictured above.



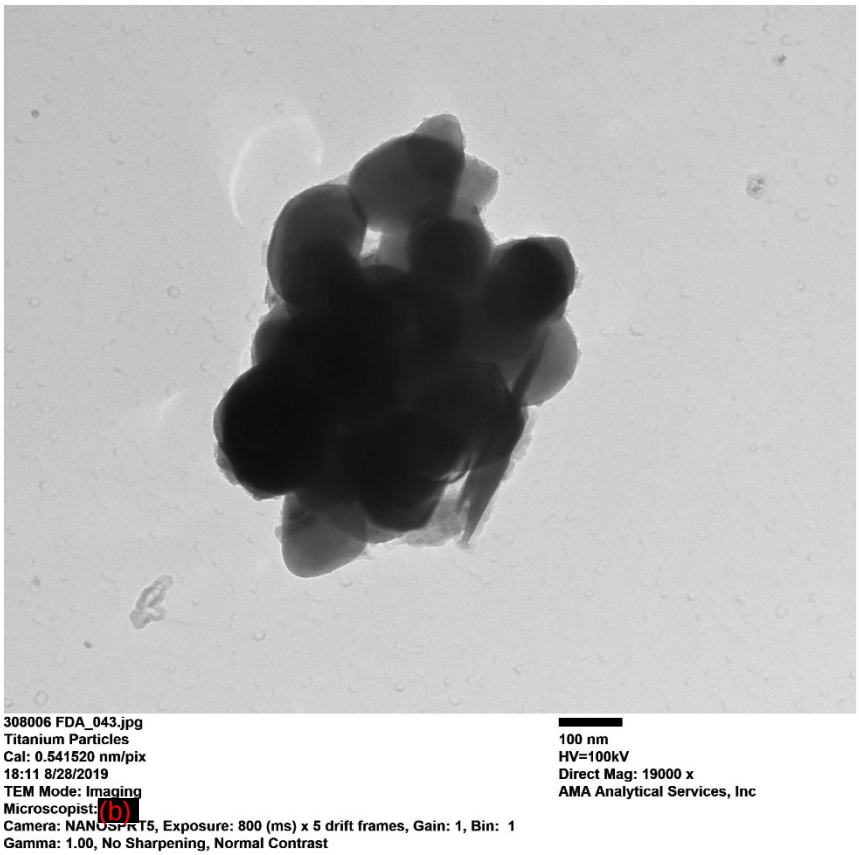
Sample 308006-4, Titanium Coated Particle



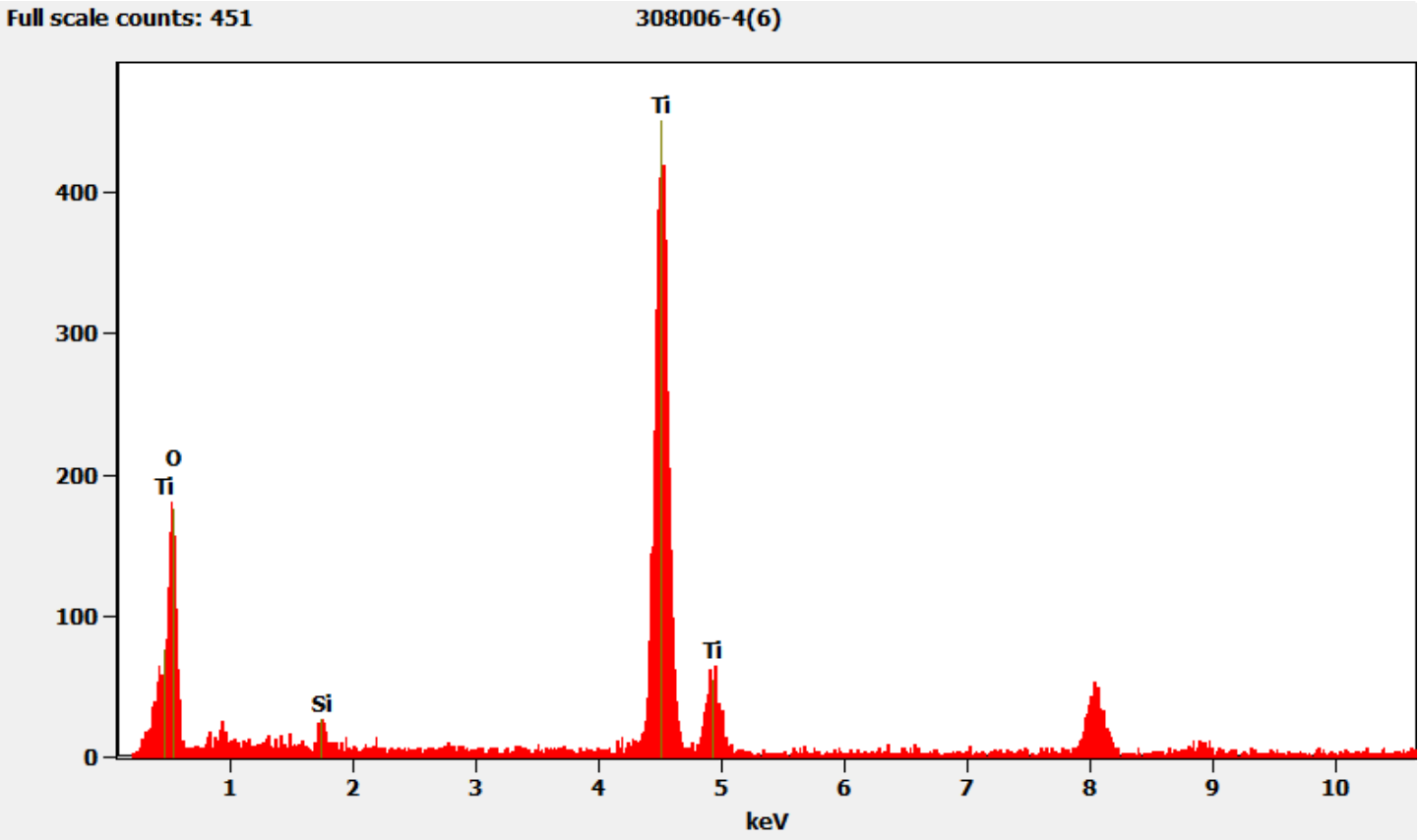
Chemistry from Titanium coated particle pictured above.



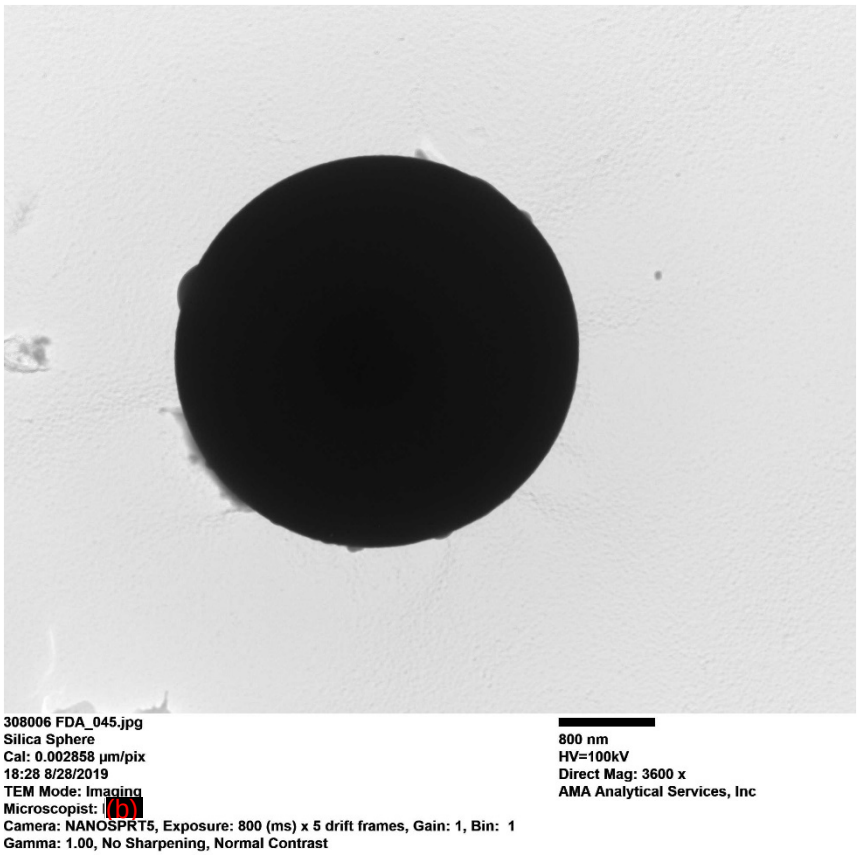
308006-4, Titanium Particles



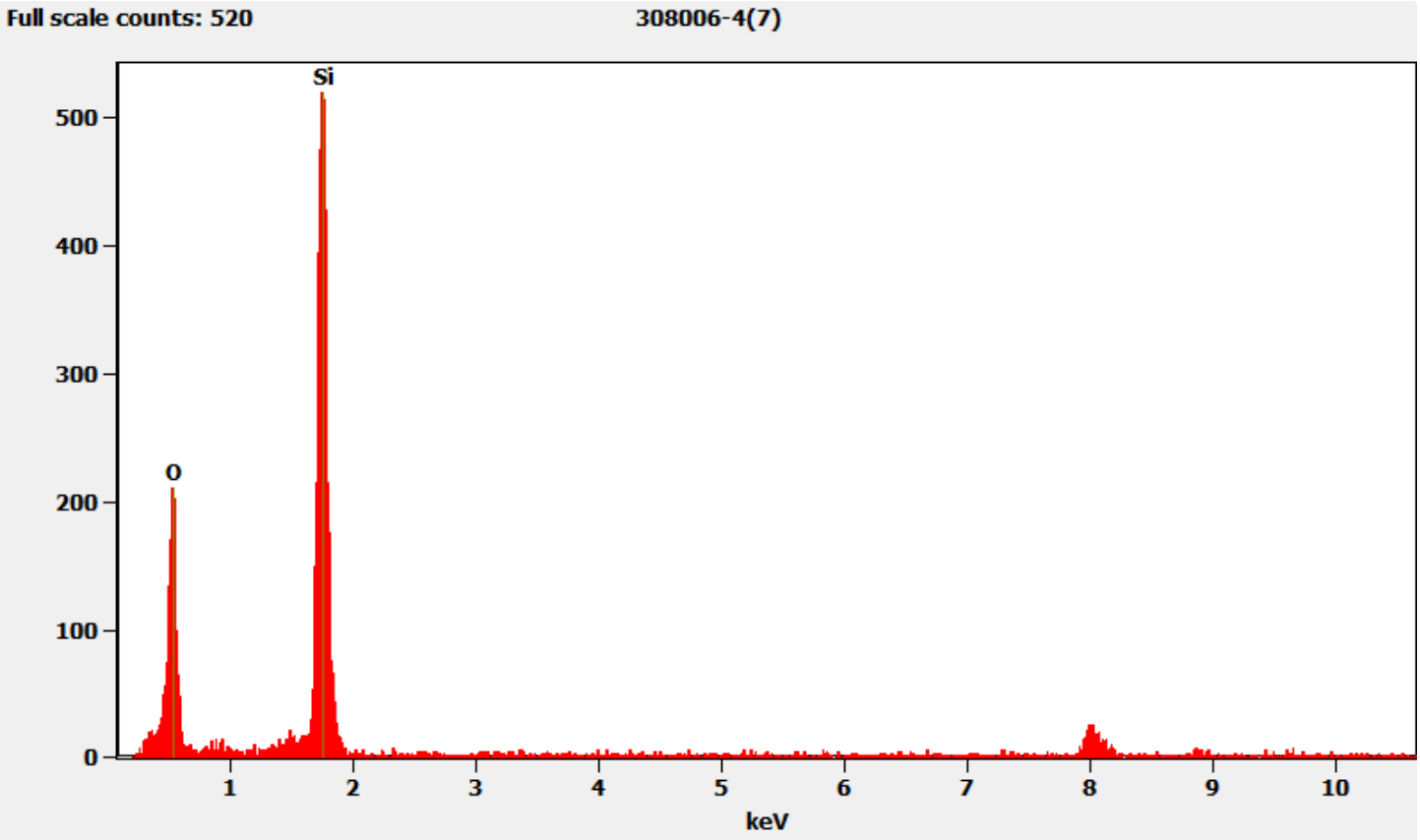
Chemistry from the Titanium particles pictured above



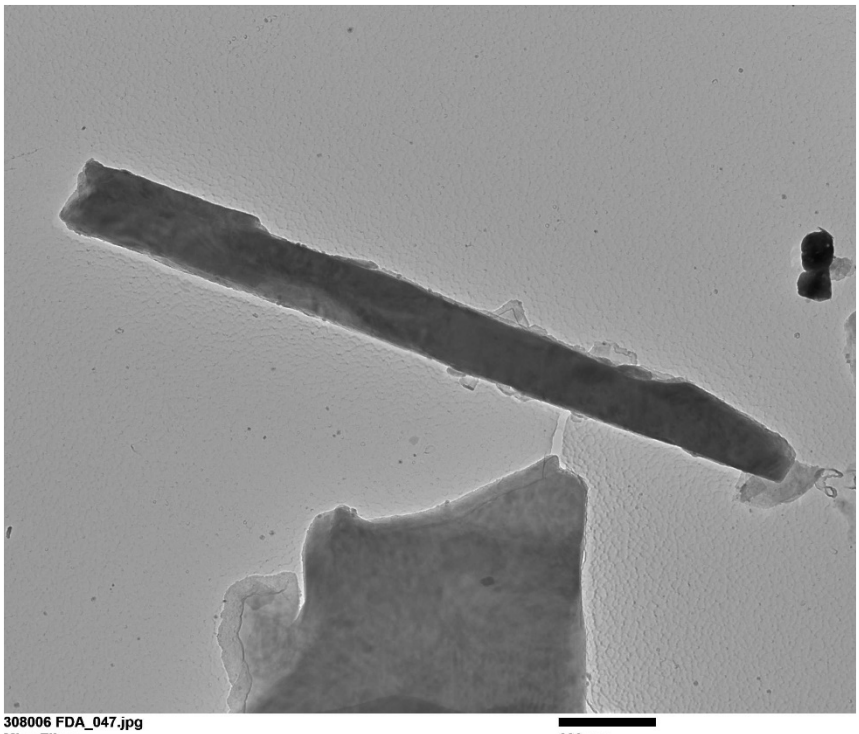
308006-4, Silica Sphere



Chemistry from the Silica sphere pictured above

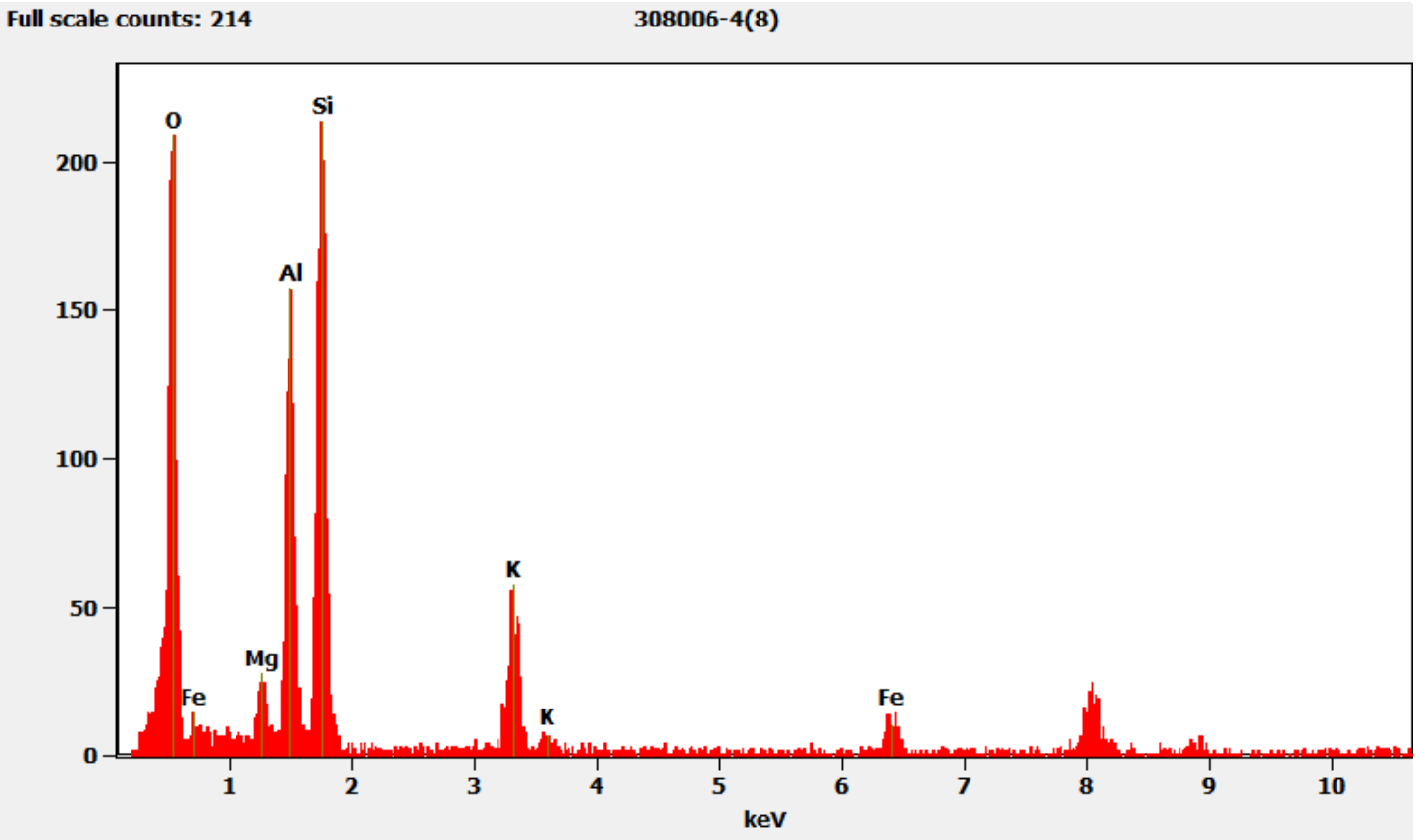


308006-4, Mica Fiber



Diffraction pattern from the Mica fiber pictured above

Chemistry from the Mica fiber pictured above



308006-5, 5A, 5B, Client Sample D57

PLM

All three aliquots of sample D-57 were analyzed by (b) (6) on September 13, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

308006-5	NAD
308006-5A	NAD
308006-5B	NAD

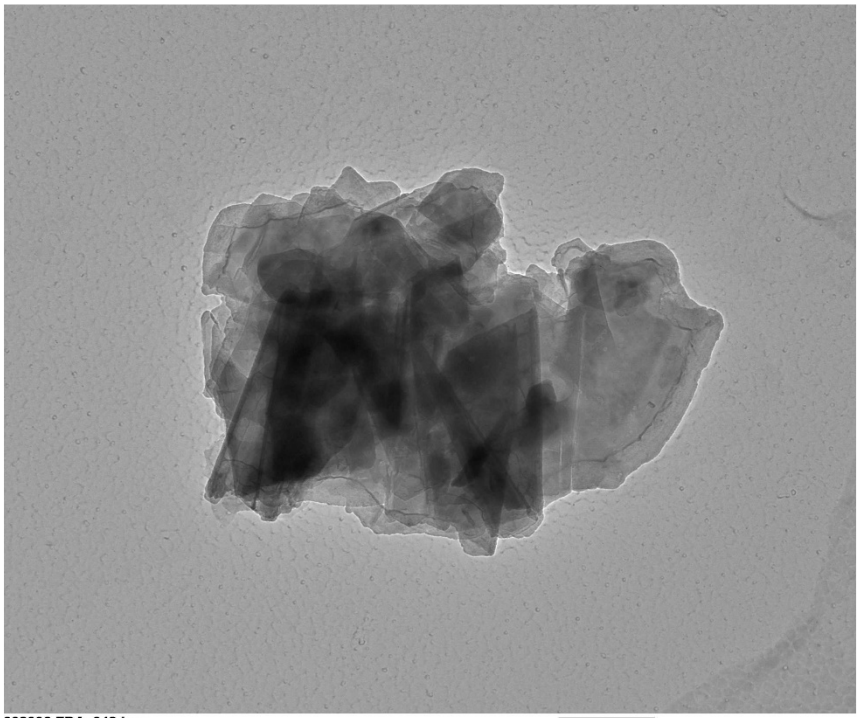
TEM

Samples 5, 5A and 5B were analyzed by (b) (6) September 3, 2019. The primary particle observed was talc along with a few talc fibers. No asbestos or non-asbestos amphibole variants were detected in the samples. The results were calculated using the equations detailed in the calculations section.

308006-5	NAD
308006-5A	NAD
308006-5B	NAD

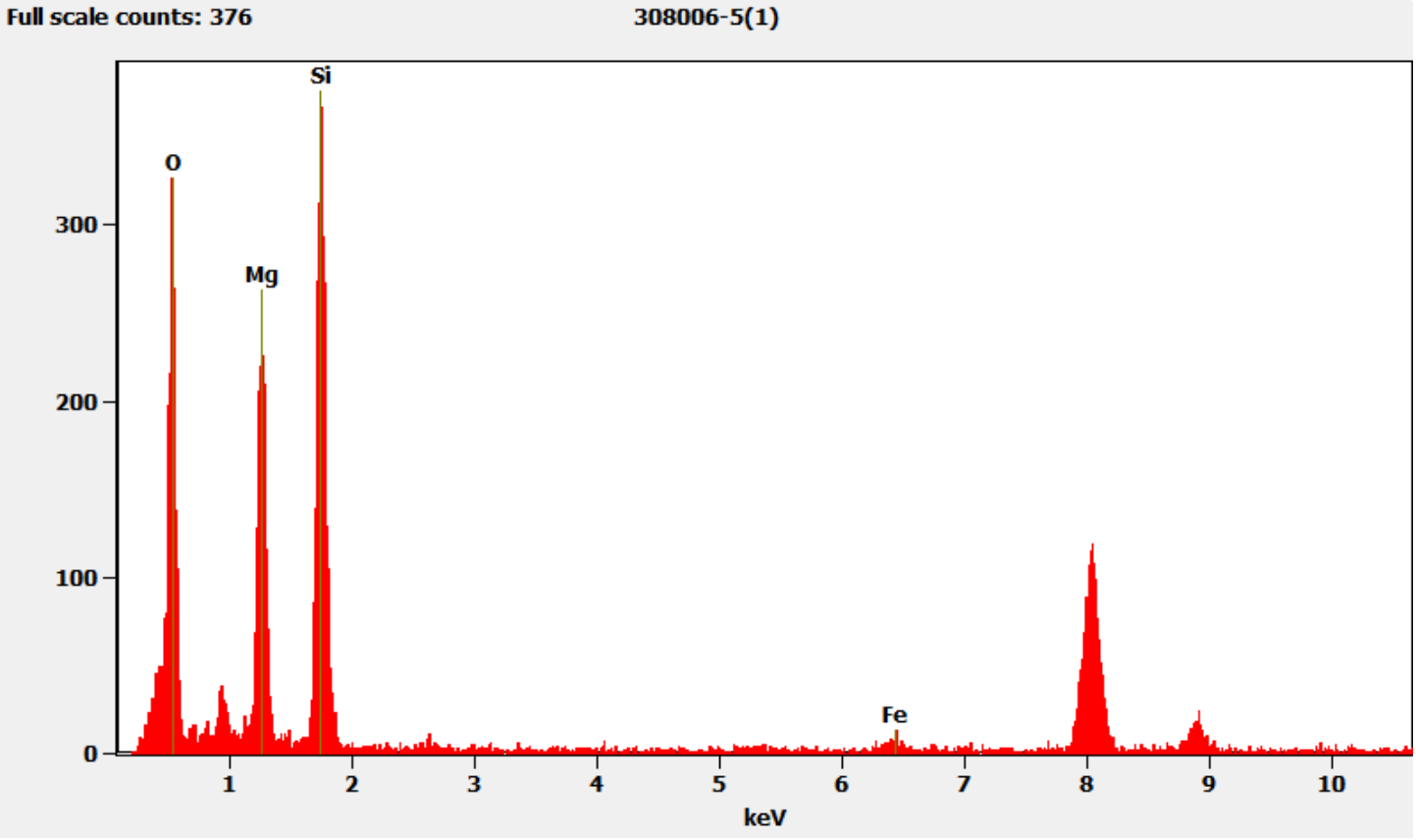
Below are pictures, diffraction patterns, and chemistry from some of the observed particles. The unidentified peaks in chemistry spectra are copper, zinc, and carbon. Those peaks are from the TEM specimen holder and specimen grid.

Sample 308006-5, Talc Particle

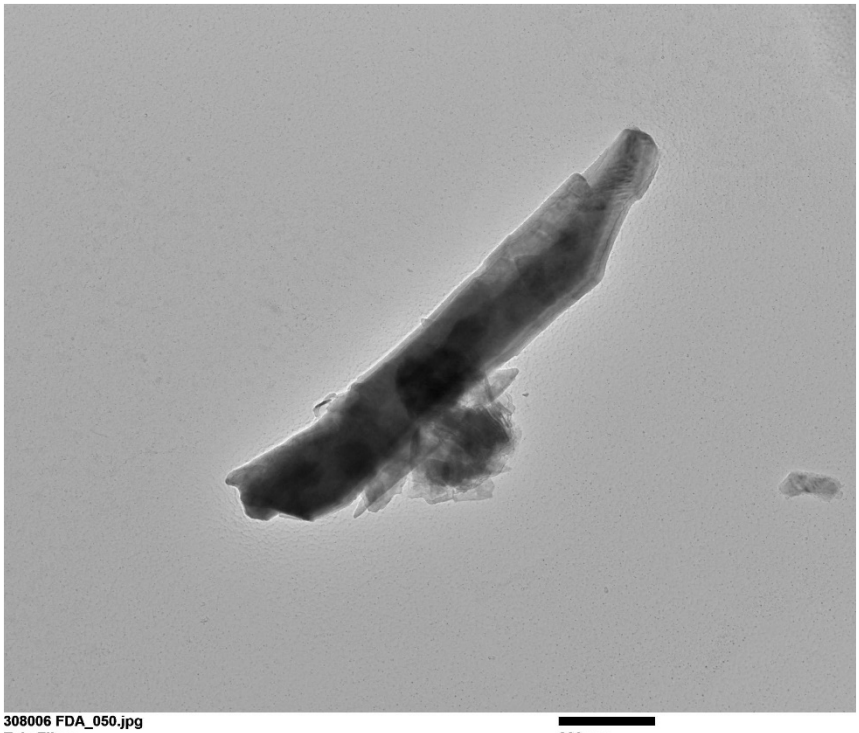


Hexagonal diffraction pattern from the talc particle pictured above.

Sample 308006-5 Chemistry from the talc particle pictured above



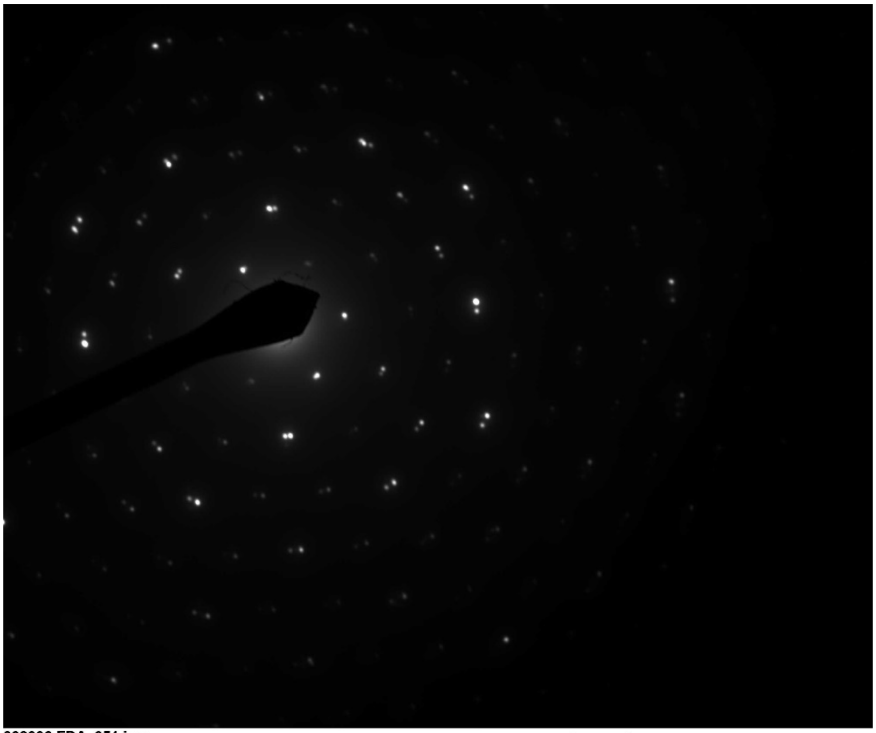
Sample 308006-5, Talc Fiber



308006 FDA_050.jpg
Talc Fibers
Cal: 0.002858 µm/pix
11:01 9/3/2019
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

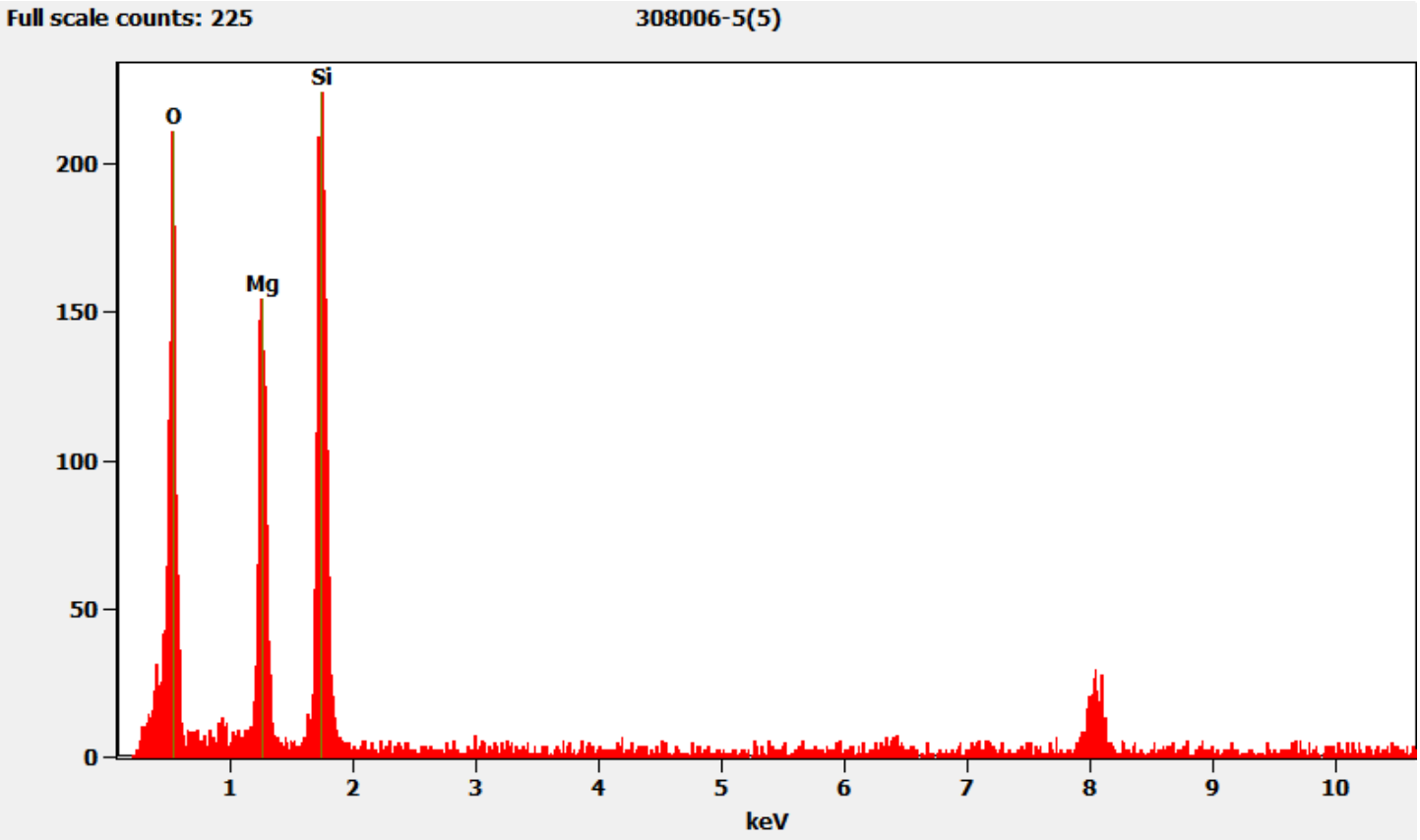
Diffraction pattern from the Talc fiber pictured above



308006 FDA_051.jpg
Talc Fibers
11:03 9/3/2019
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 fiber pictured above



<u>308006-7, 7A, 7B, Client Sample D-59</u>

PLM

All three aliquots of sample D-59 were analyzed by (b) (6) on September 13, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

308006-7	NAD
308006-7A	NAD
308006-7B	NAD

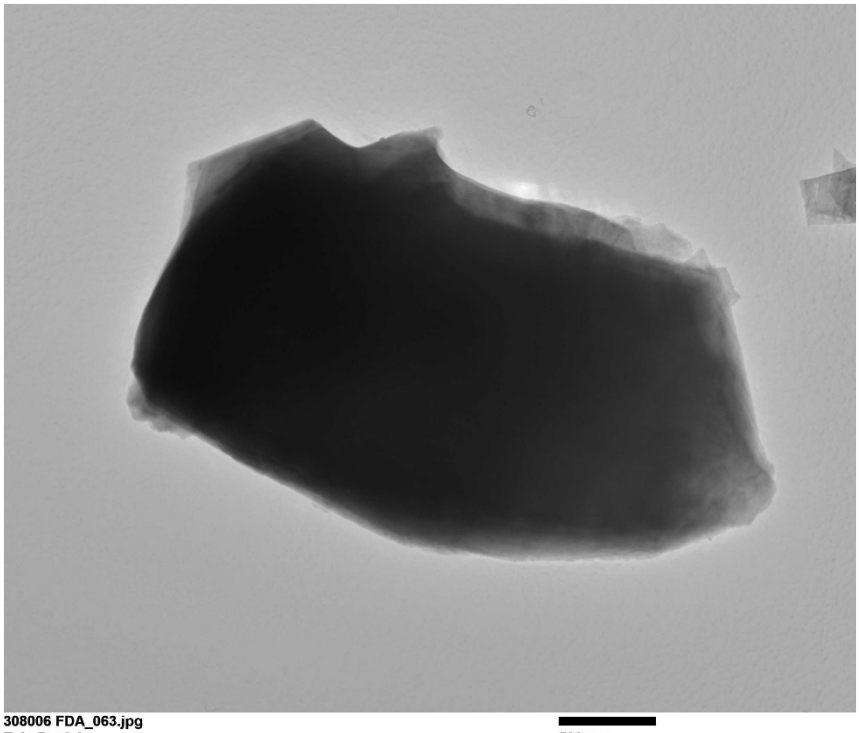
TEM

(b) (6) analyzed sample 7 on September 4, 2019 and sample 7B on September 12, 2019. (b) (6) analyzed sample 7A on September 10, 2019. The primary particles observed were talc and mica along with a few talc fibers, talc ribbons, titanium particles, iron particles, and titanium coated particles. No asbestos or non-asbestos amphibole variants were detected in the samples. The results were calculated using the equations detailed in the calculations section.

308006-7	NAD
308006-7A	NAD
308006-7B	NAD

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

Sample 308006-7, Talc Particle



308006 FDA_063.jpg
Talc Particle
Cal: 0.001774 µm/pix
15:01 9/4/2019
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

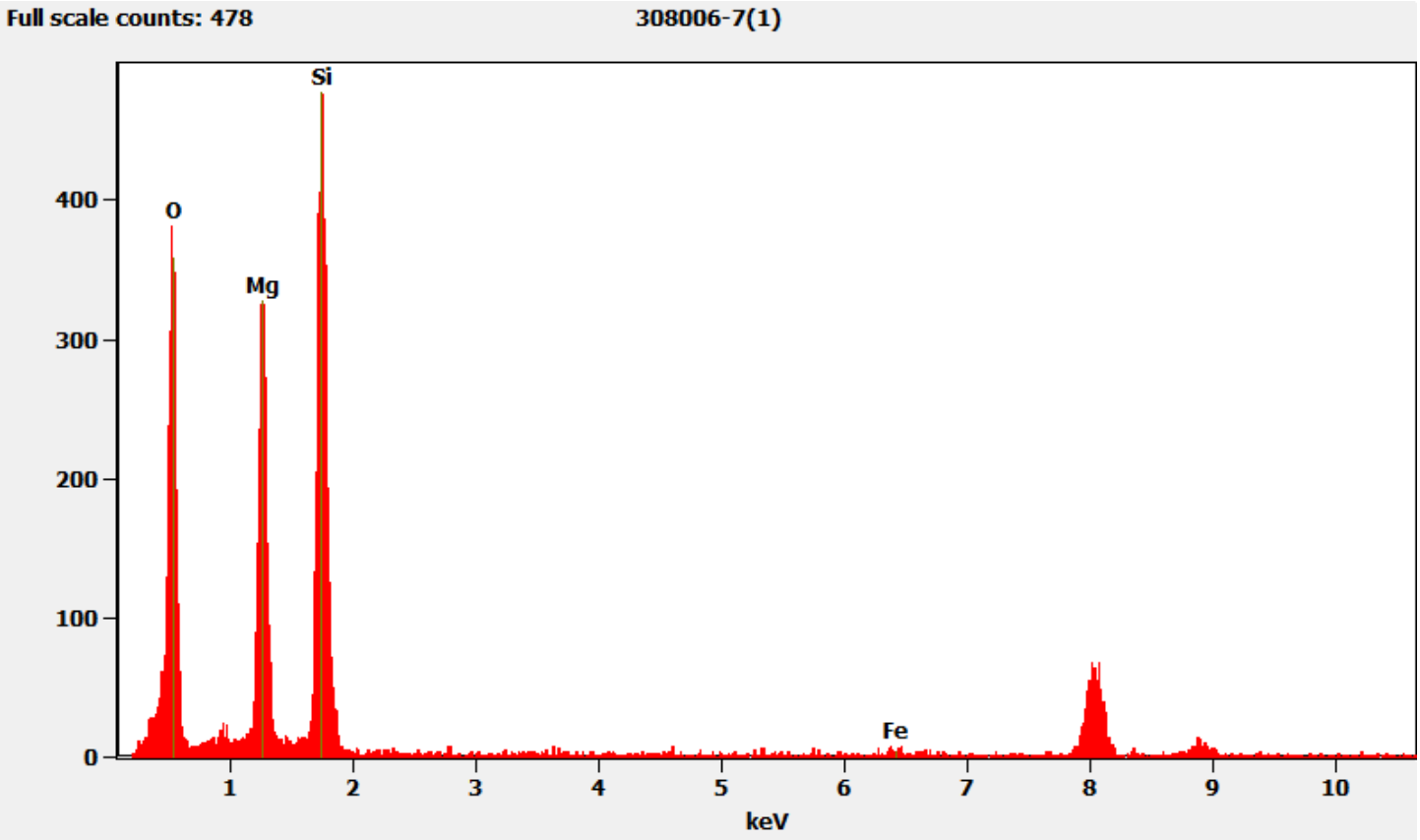
500 nm
HV=100kV
Direct Mag: 5800 x
AMA Analytical Services, Inc

Hexagonal diffraction from the Talc particle pictured above.

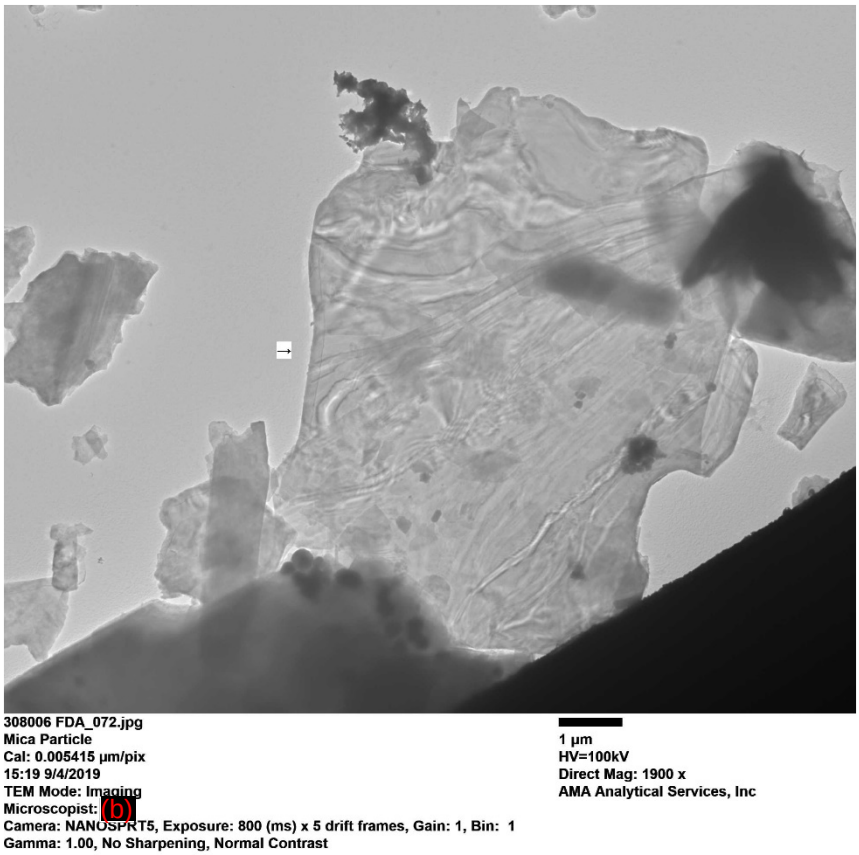
308006 FDA_064.jpg
Talc Particle
15:02 9/4/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from the Talc particle pictured above.



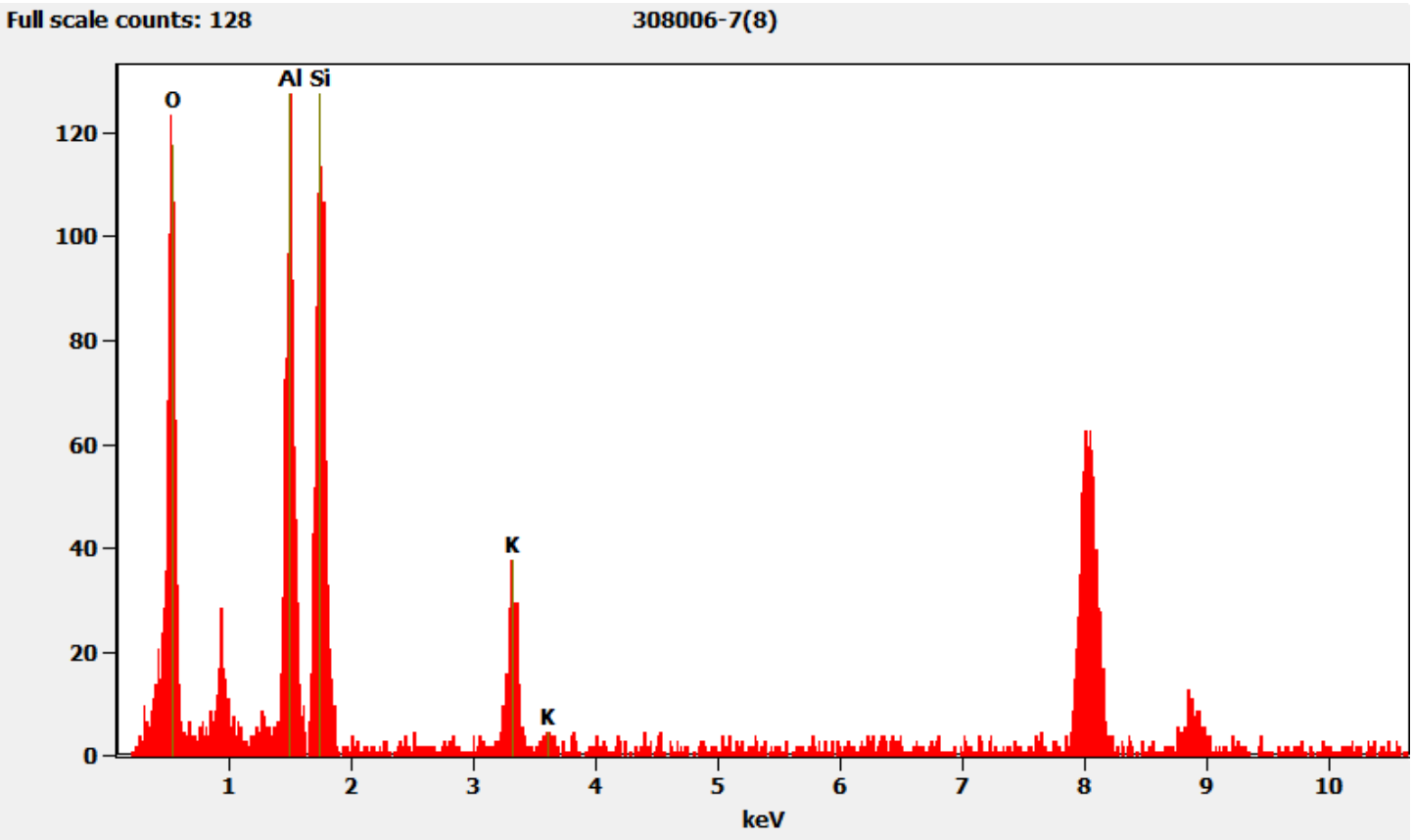
Sample 308006-7, Mica Particle



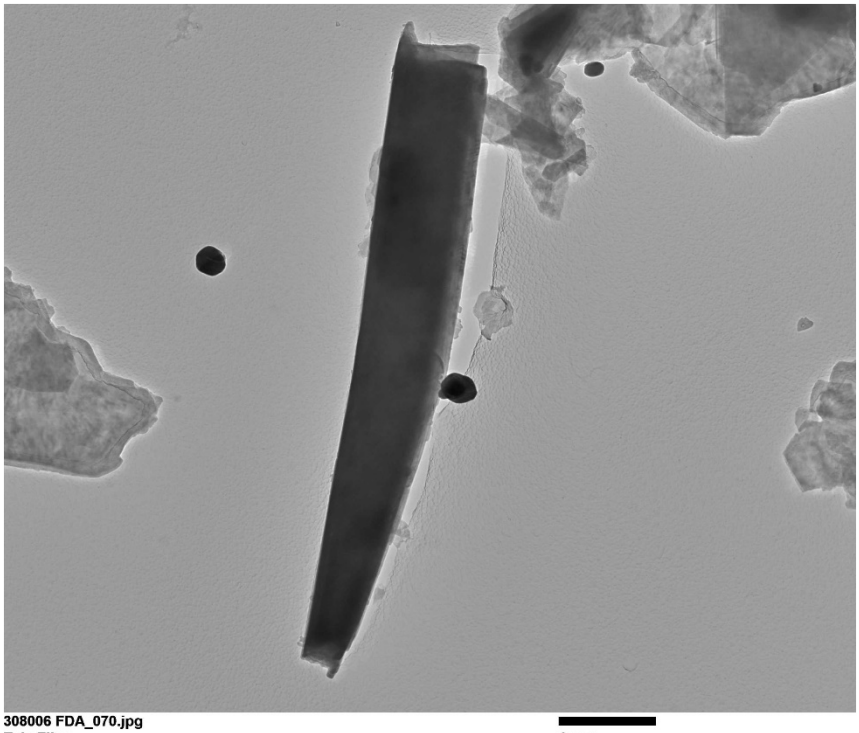
Diffraction pattern from the Mica particle pictured above.

1

Chemistry from the Mica particle pictured above



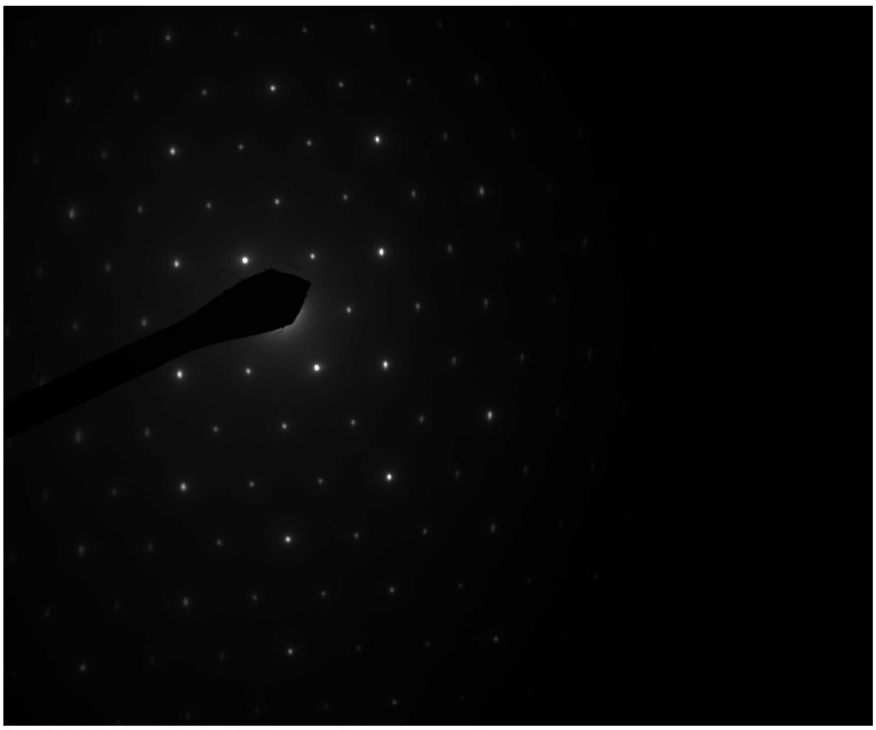
Sample 308006-7, Talc Fiber



308006 FDA_070.jpg
Talc Fiber
Cal: 0.003548 µm/pix
15:12 9/4/2019
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

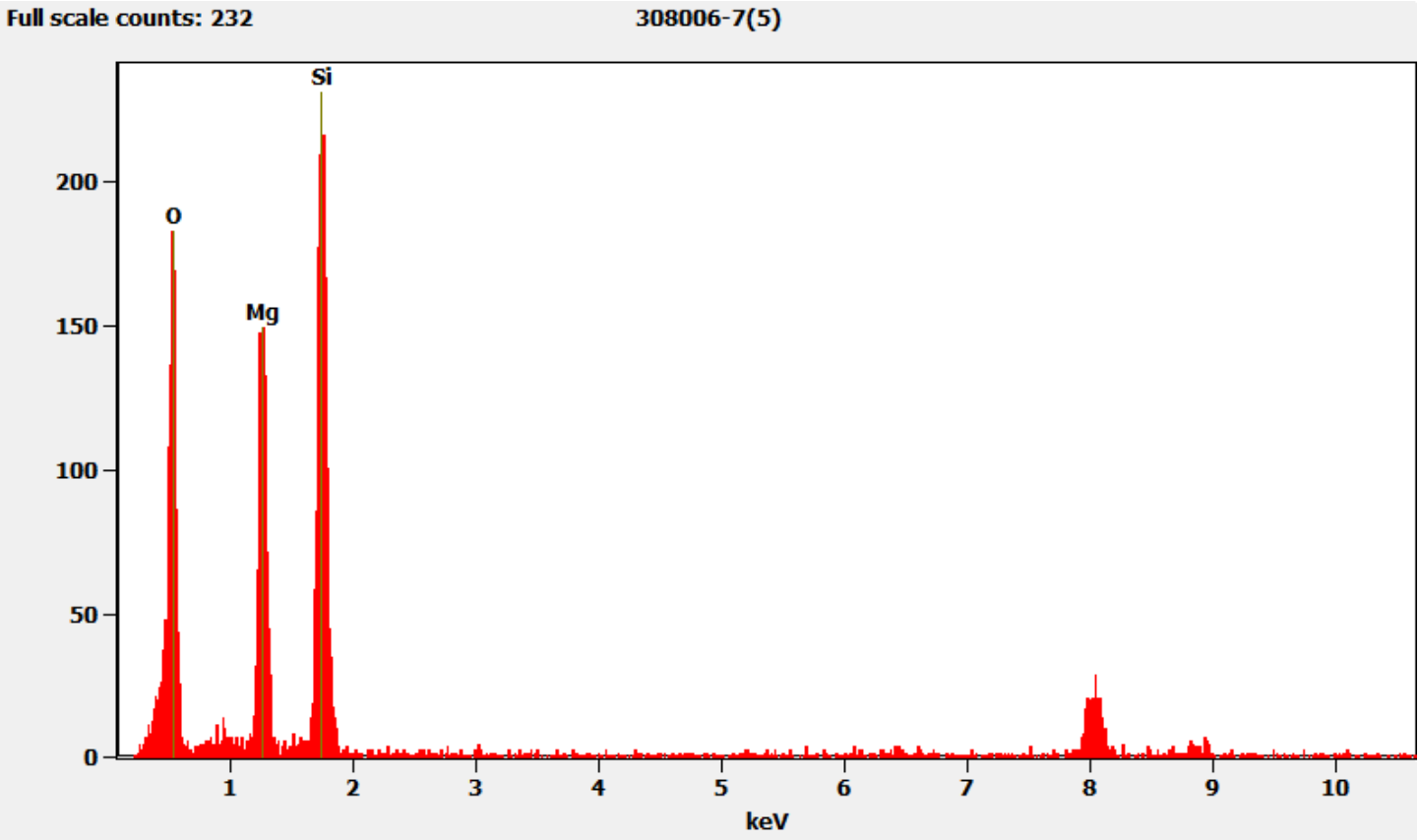
Diffraction pattern from the Talc fiber pictured above.



308006 FDA_071.jpg
Talc Fiber
15:13 9/4/2019
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

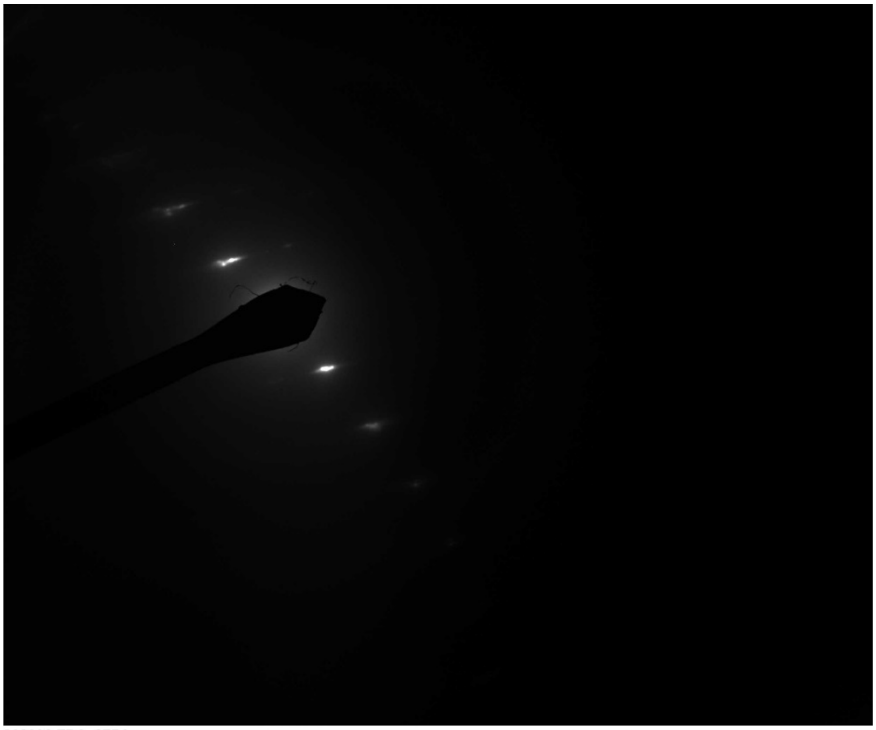
100 (1/Å)
HV=100kV
Cam Len: 0.2200 m
AMA Analytical Services, Inc

Chemistry from the Talc fiber pictured above.

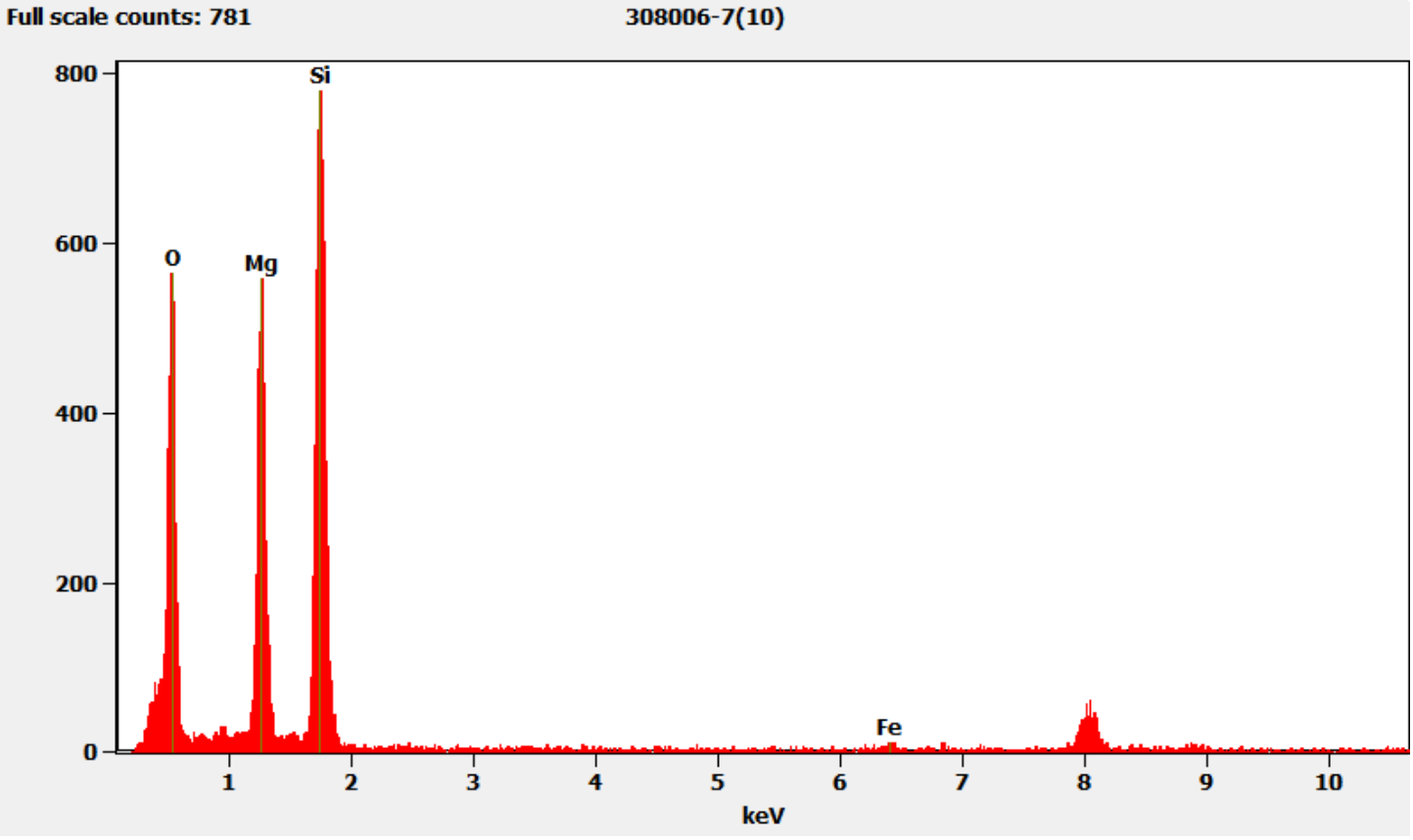


Sample 308006-7, Talc Ribbon

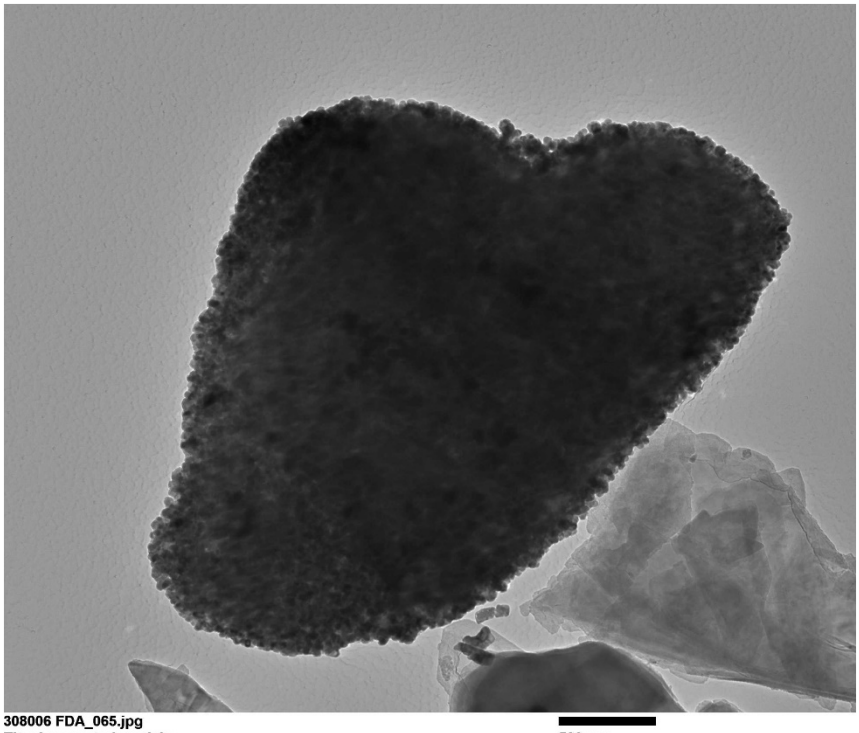
Diffraction pattern from the Talc ribbon pictured above.



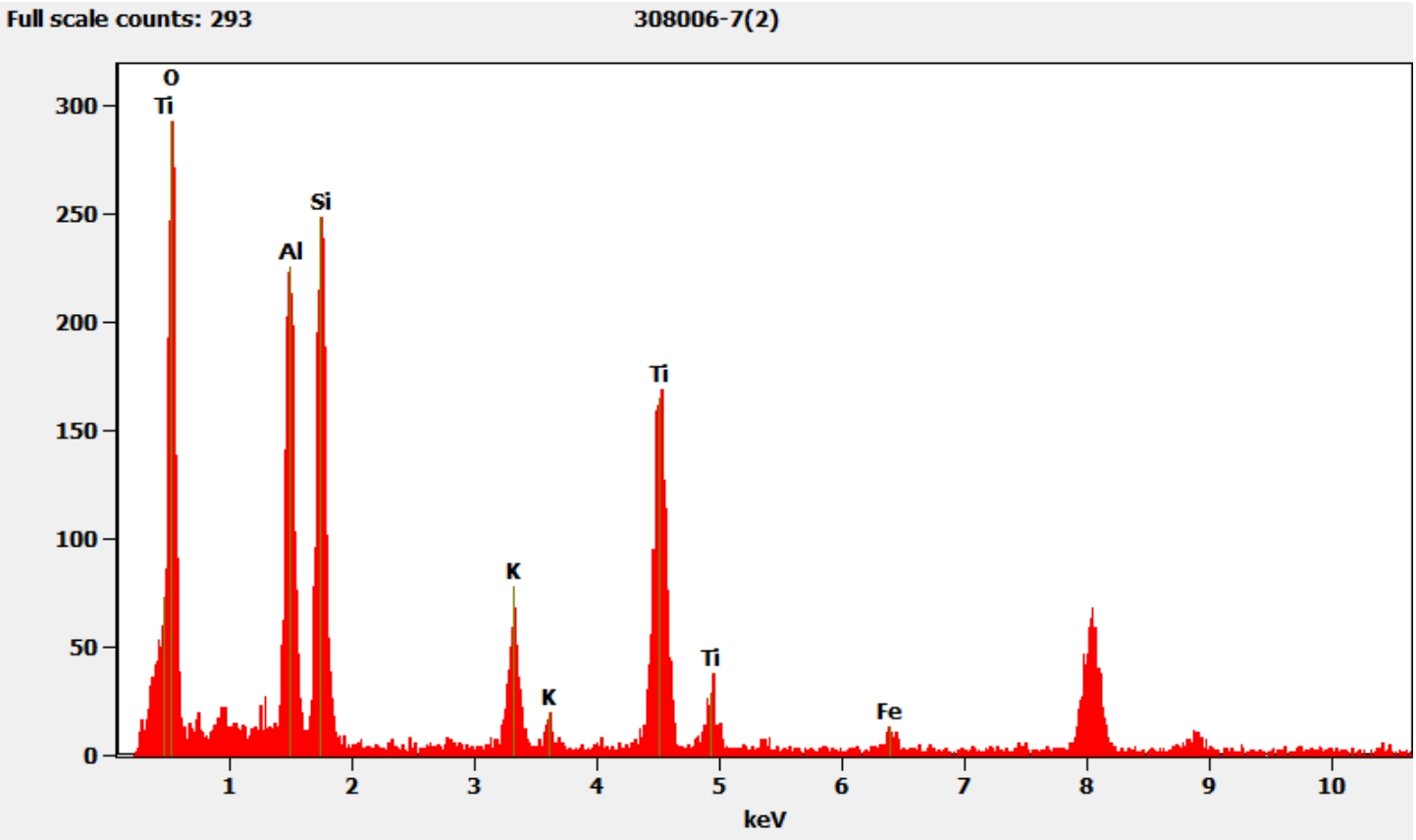
Chemistry from Talc ribbon pictured above.



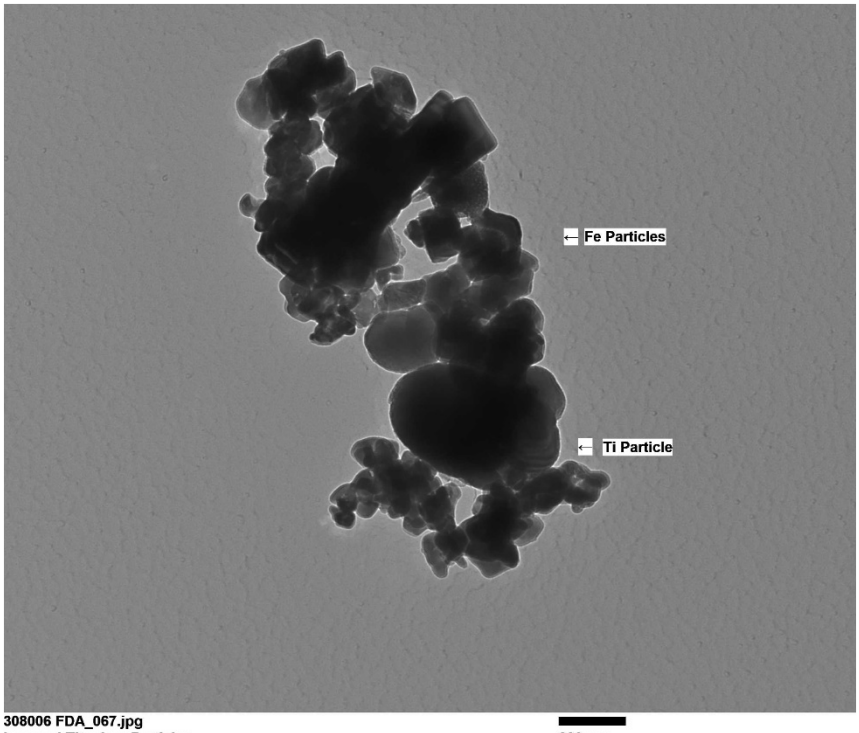
308006-7, Titanium Coated Particle



Chemistry from the Titanium coated particle pictured above



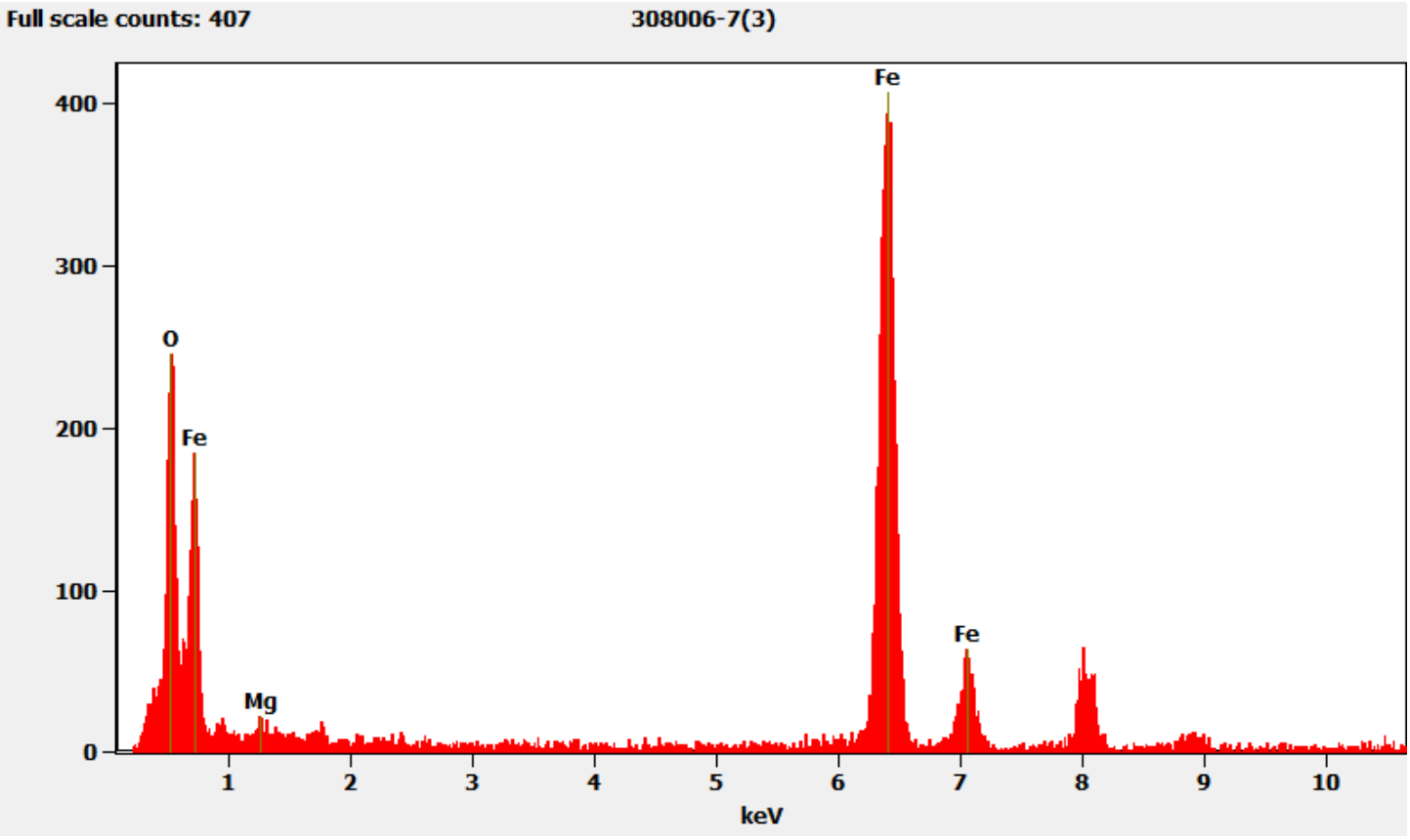
308006-7, Iron & Titanium Particles



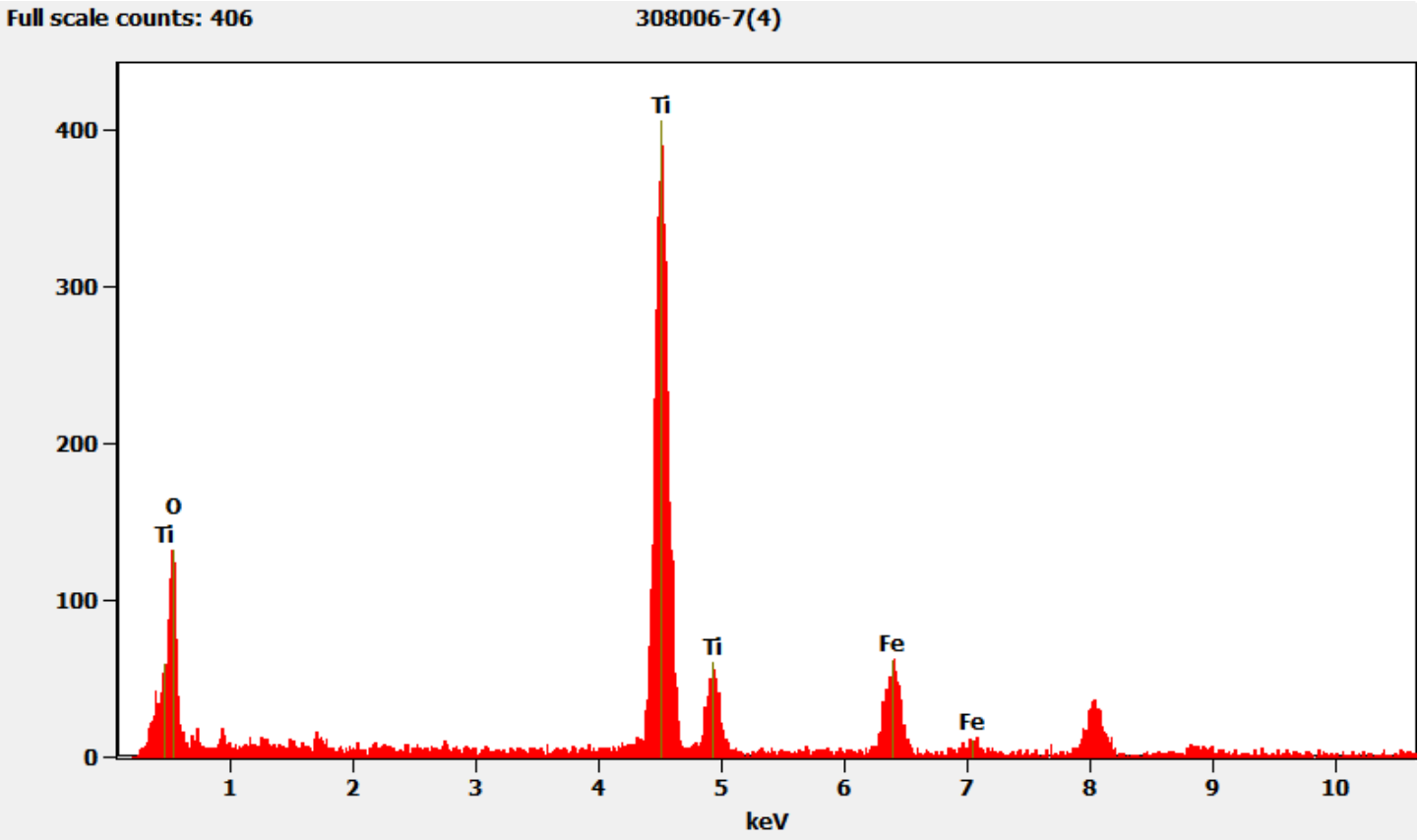
308006 FDA_067.jpg
Iron and Titanium Particles
Cal: 0.001029 µm/pix
15:06 9/4/2019
TEM Mode: Imaging
Microscopist: (h)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from the Iron particles pictured above



Chemistry from the Titanium particles pictured above



<u>308006-8, 8A, 8B, Client Sample D-60</u>

PLM

All three aliquots of sample D-60 were analyzed by (b) (6) on September 13, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

308006-8	NAD
308006-8A	NAD
308006-8B	NAD

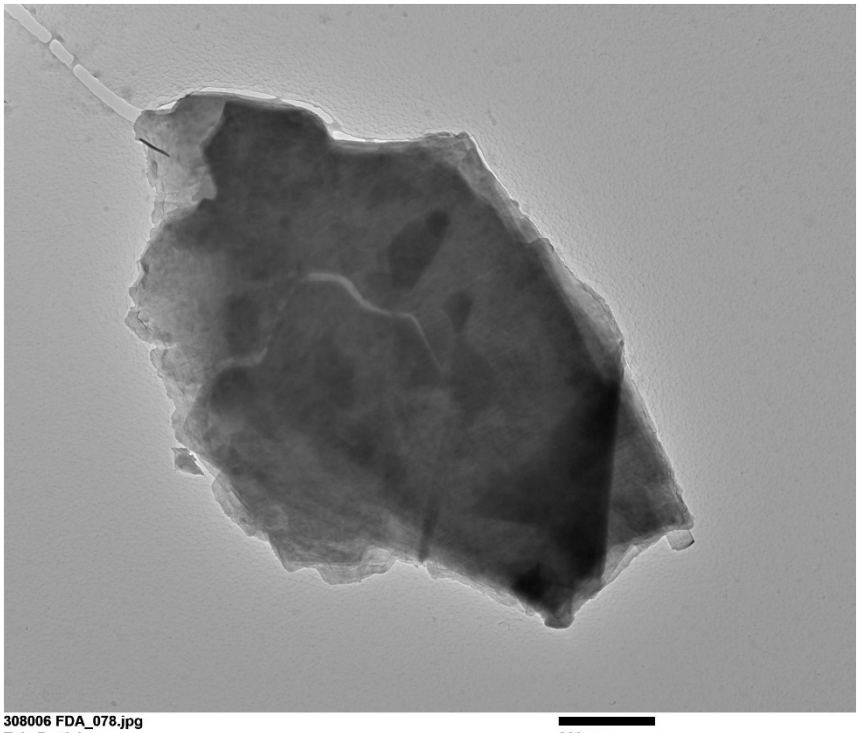
TEM

(b) (6) analyzed Sample 8 on September 5, 2019 and samples 8A and 8B on September 12, 2019. The primary particle observed was talc along with a few talc fibers and mica particles. No asbestos or non-asbestos amphibole variants were detected in the samples. The results were calculated using the equations detailed in the calculations section.

308006-8	NAD
308006-8A	NAD
308006-8B	NAD

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

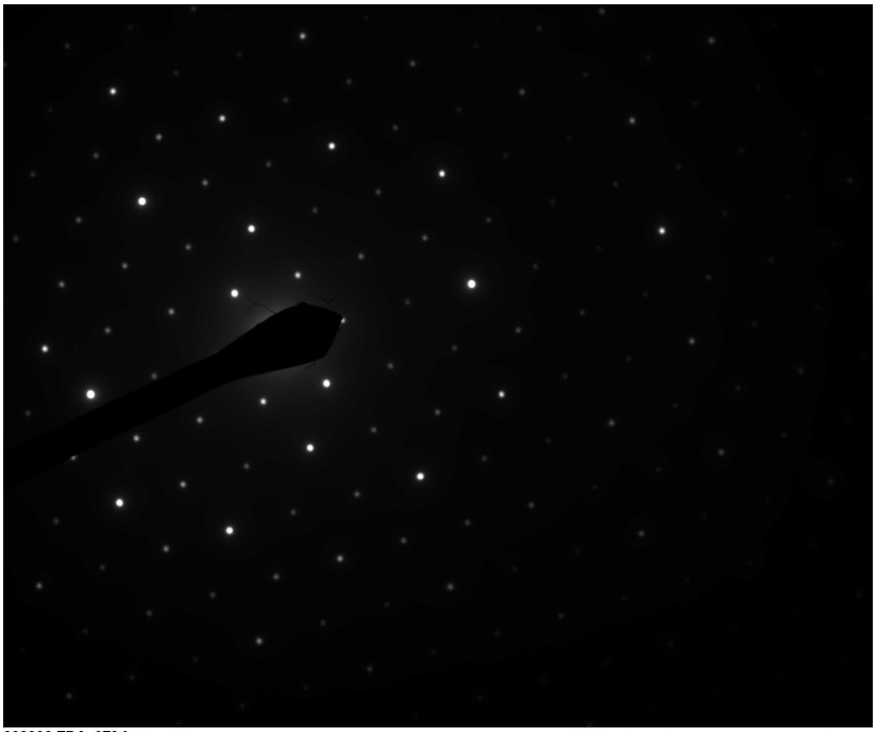
Sample 308006-8, Talc Particle



308006 FDA_078.jpg
Talc Particle
Cal: 0.002858 µm/pix
12:33 9/5/2019
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

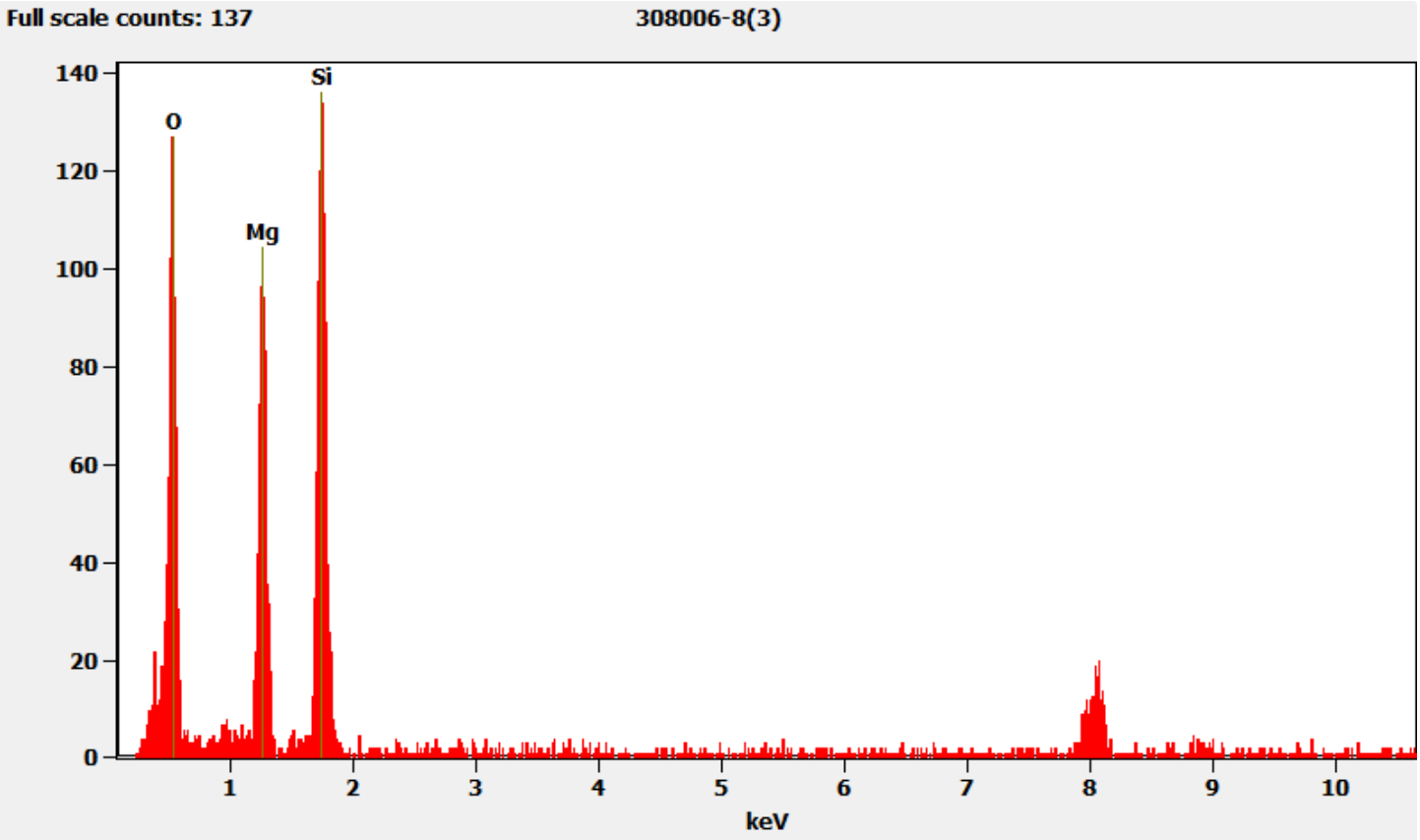
Hexagonal diffraction from the Talc particle pictured above.



308006 FDA_079.jpg
Talc Particle
12:34 9/5/2019
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

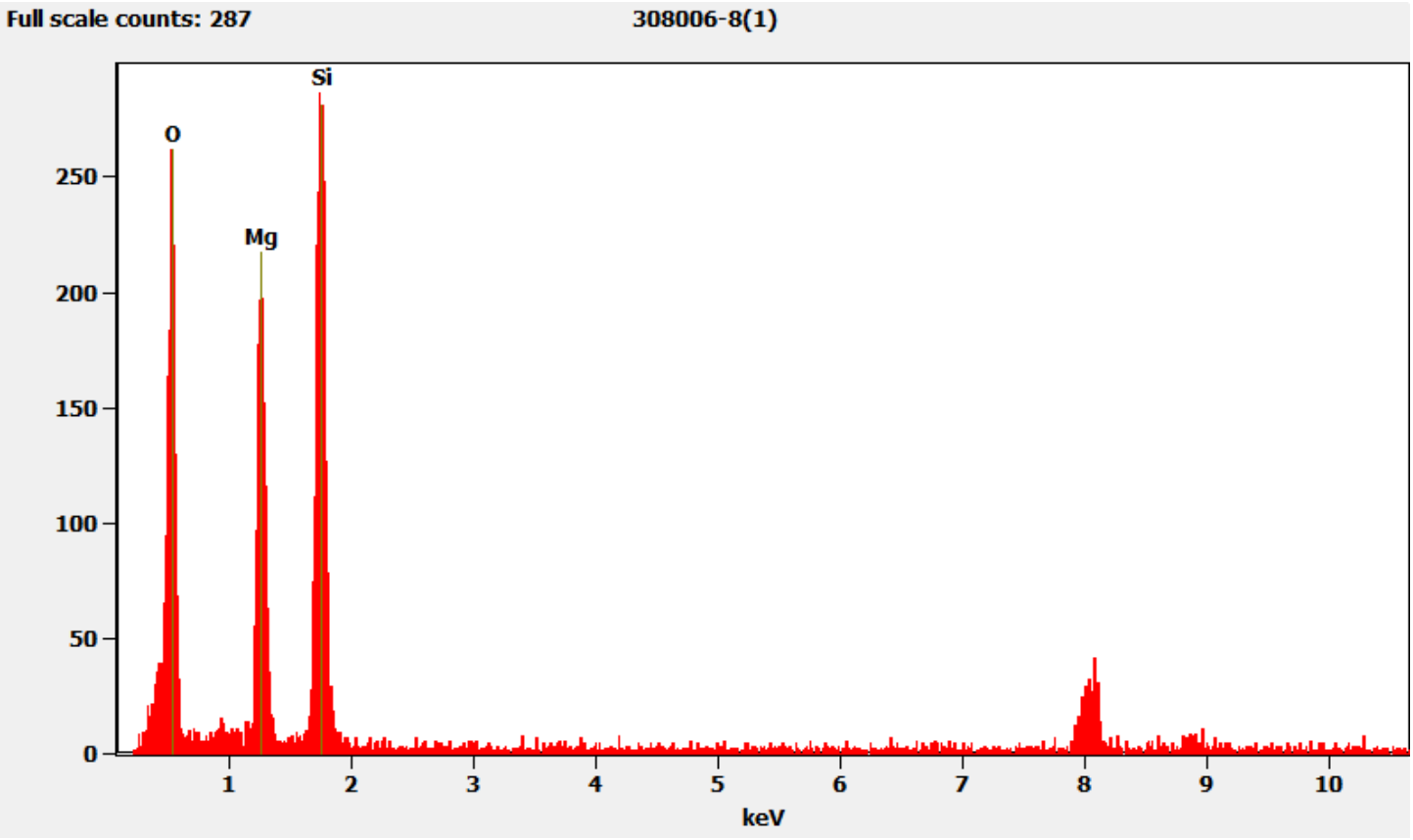
Chemistry from the Talc particle pictured above.



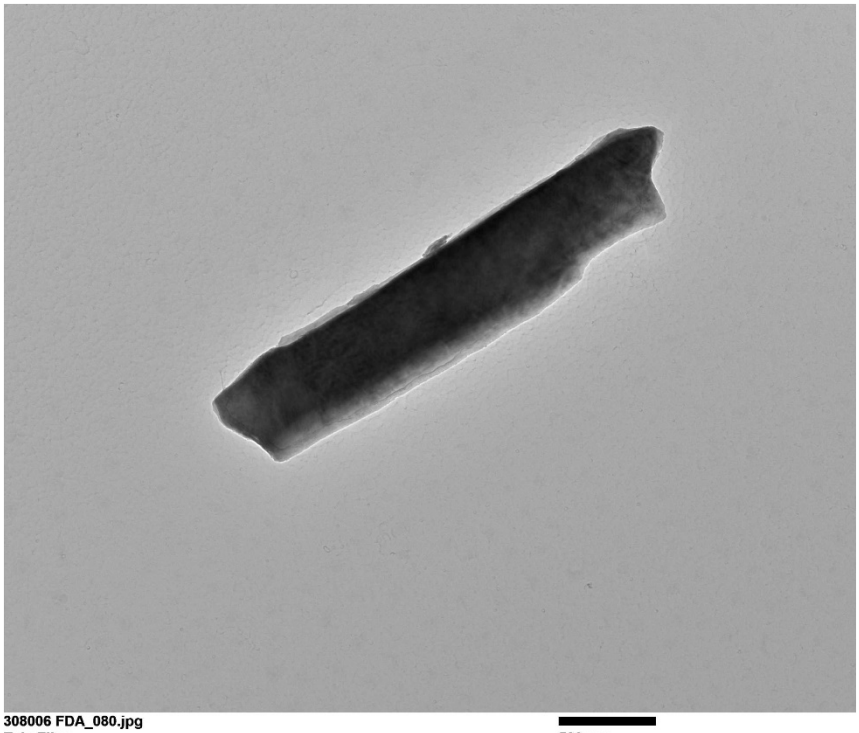
Sample 308006-8, Talc Fiber

Diffraction pattern from the Talc fiber pictured above.

Chemistry from the Talc fiber pictured above



Sample 308006-8, Talc Fiber



308006 FDA_080.jpg
Talc Fiber
Cal: 0.001774 µm/pix
12:47 9/5/2019
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

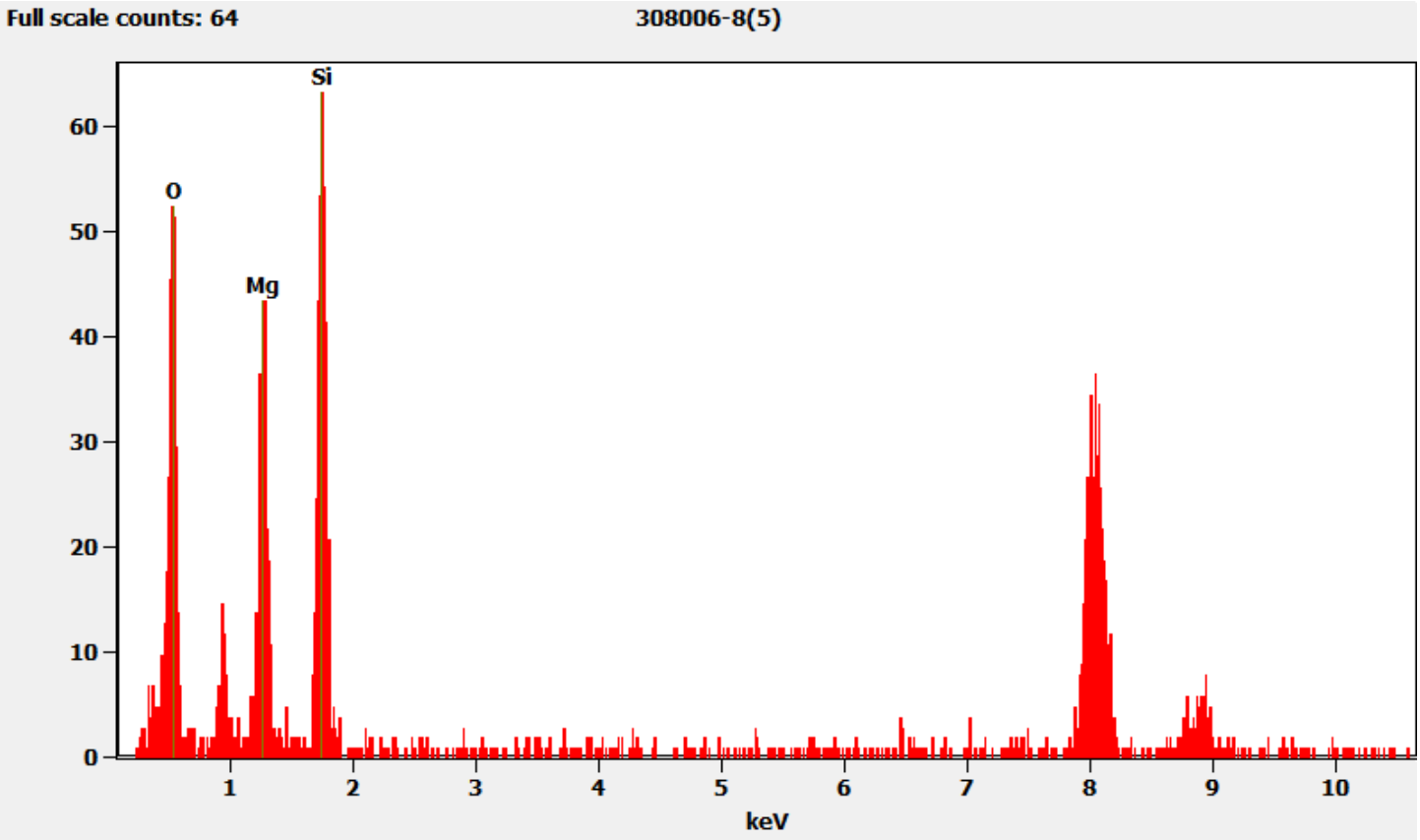
500 nm
HV=100kV
Direct Mag: 5800 x
AMA Analytical Services, Inc

Diffraction pattern from the Talc fiber pictured above.

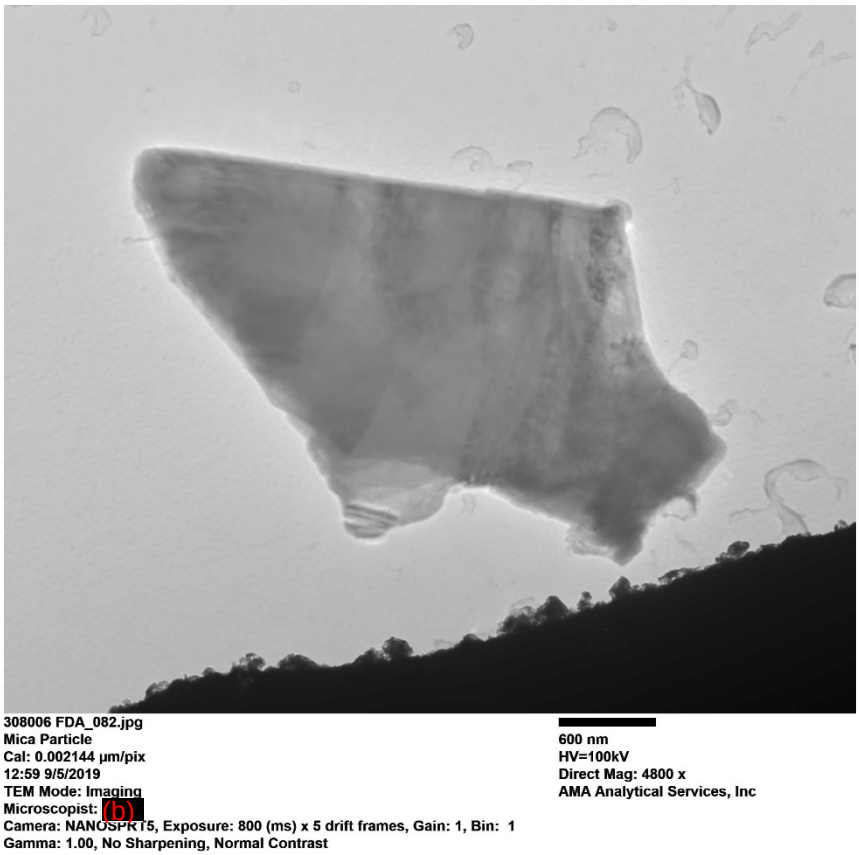
308006 FDA_081.jpg
Talc Fiber
12:48 9/5/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

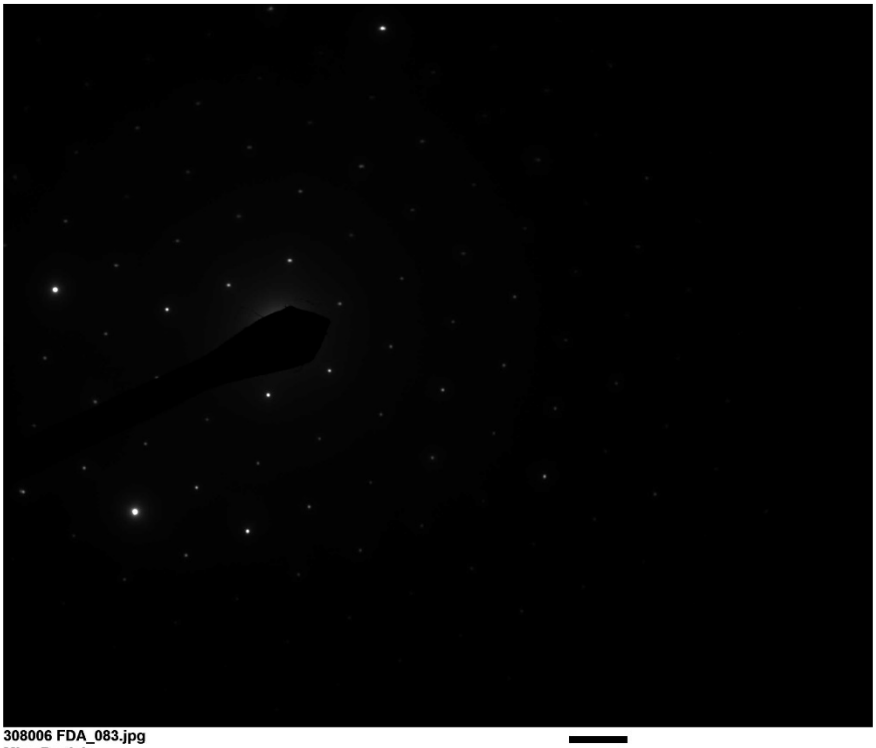
Chemistry from the Talc fiber pictured above.



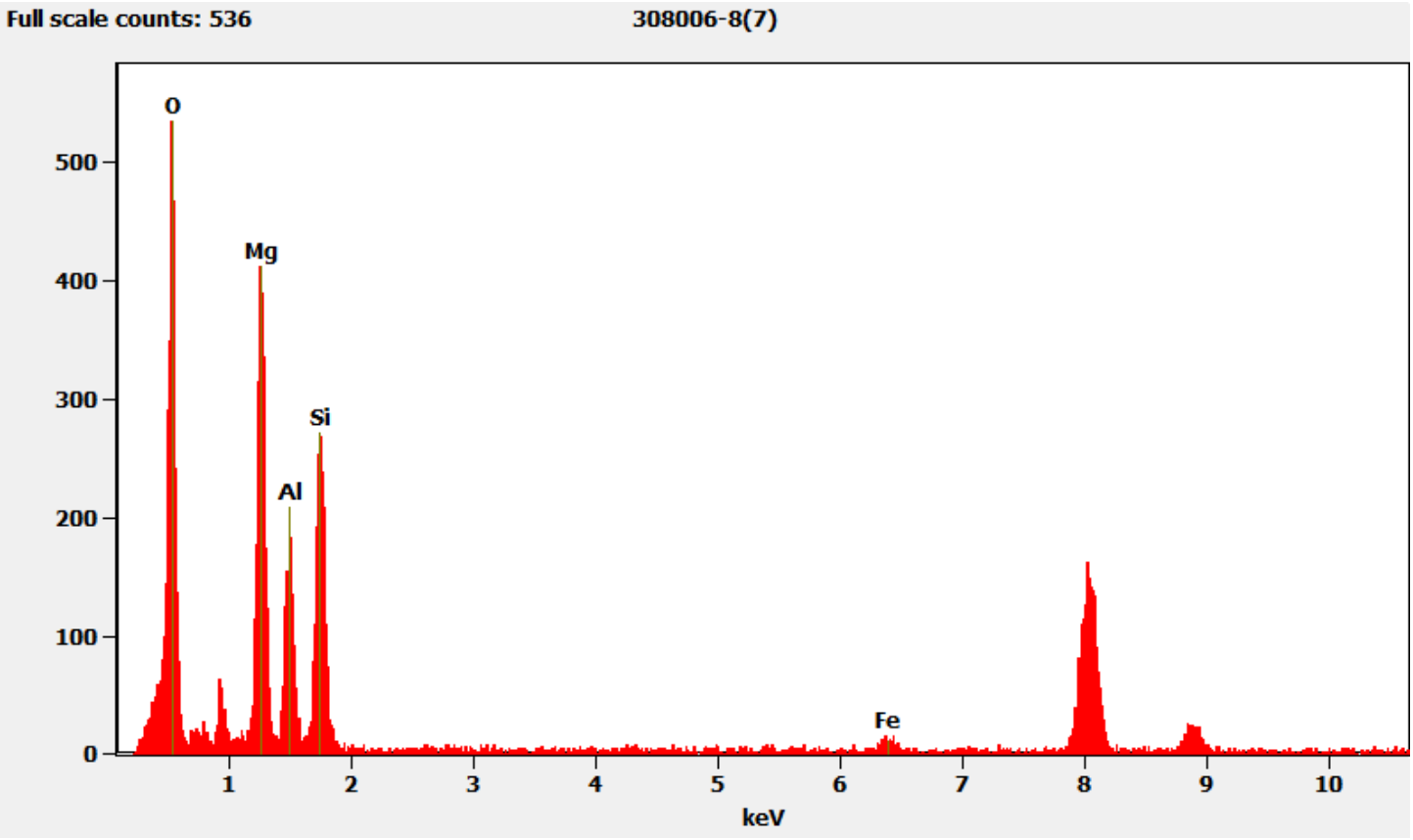
Sample 308006-8, Mica Particle



Diffraction pattern from the Mica particle pictured above.



Chemistry from the Mica particle pictured above.



308006-9, 9A, 9B, Client Sample D-61

PLM

All three aliquots of sample D-61 were analyzed by (b) (6) on September 13, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

308006-9	NAD
308006-9A	NAD
308006-9B	NAD

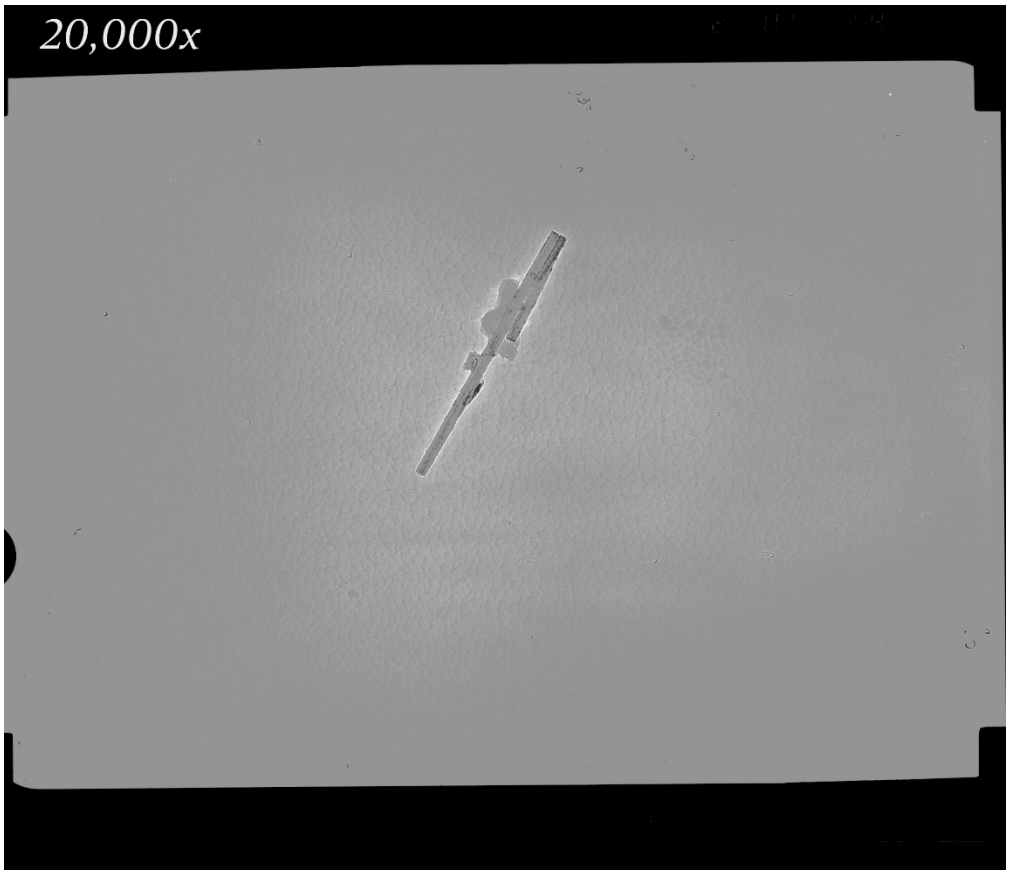
TEM

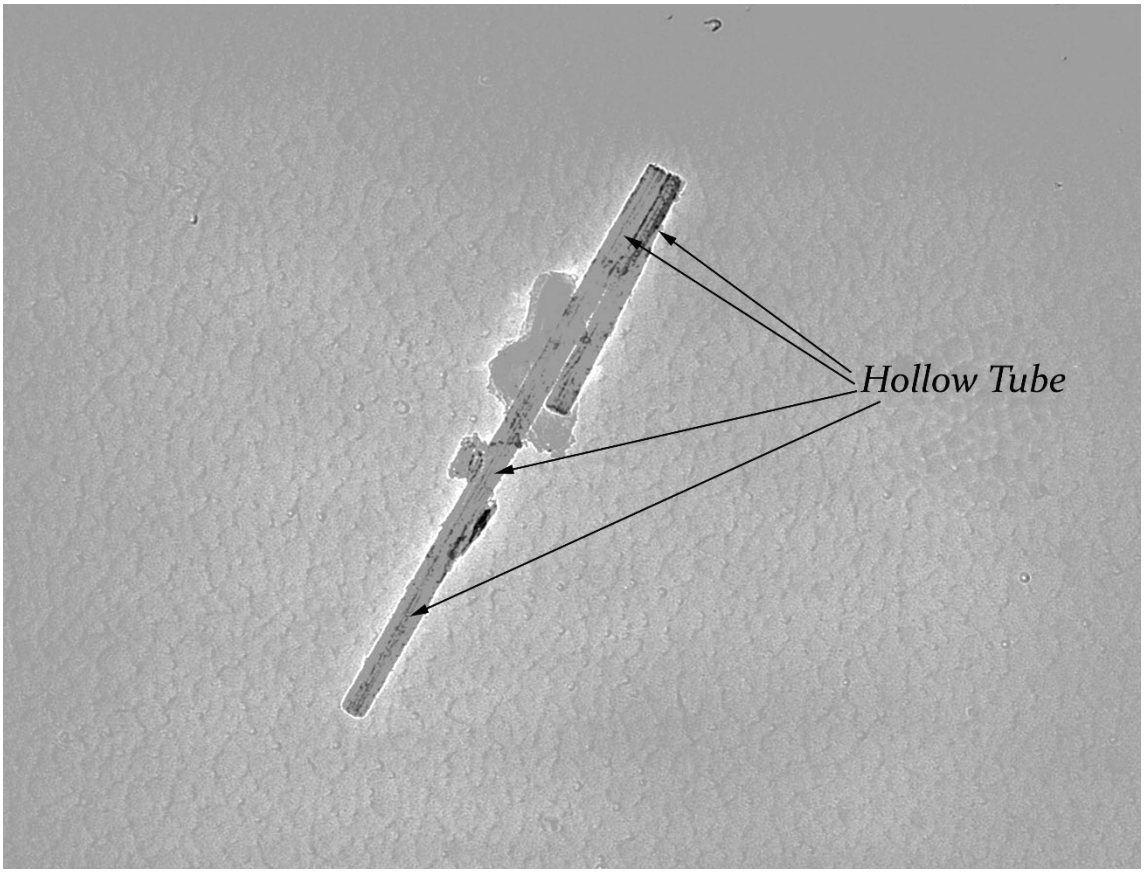
(b) (6) analyzed sample 9 on September 5, 2019 and sample 9A on September 12, 2019. (b) (6) analyzed sample 9B on September 12, 2019. The primary particle observed was talc along with a few talc fibers, talc ribbons and some silica particles. A chrysotile structure was observed on aliquot 9B. The results were calculated using the equations detailed in the calculations section.

308006-9	NAD
308006-9A	NAD
308006-9B	<0.00002%

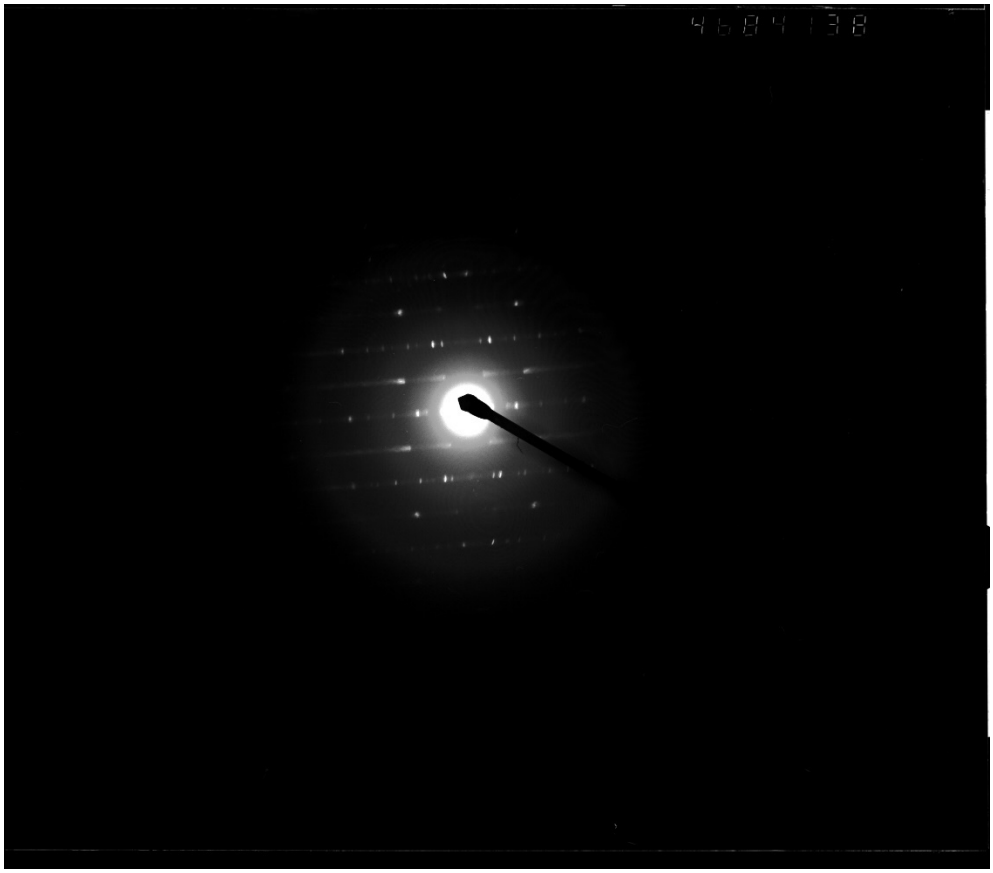
Below are pictures, diffraction patterns, and chemistry from some of the counted particles. The unidentified peaks in chemistry spectra are copper, zinc, and carbon. Those peaks are from the TEM specimen holder and specimen grid.

Sample 308006-9B Chrysotile *(the images below were taken on film; the digital versions were scanned from a negative)*

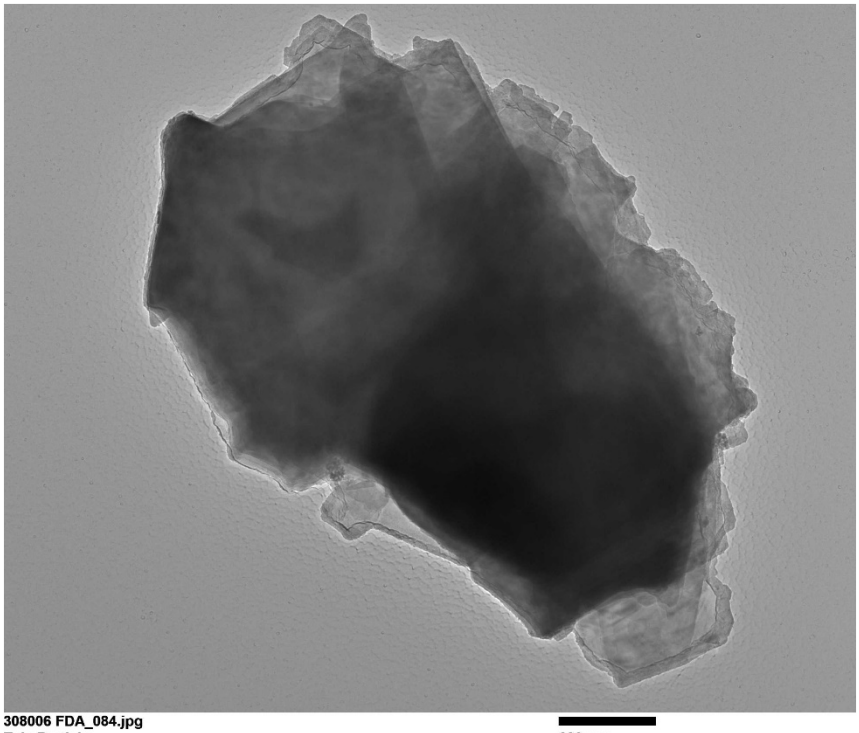




Diffraction pattern from the Chrysotile structure pictured above. *(the image below was taken on film; the digital version was scanned from a negative)*



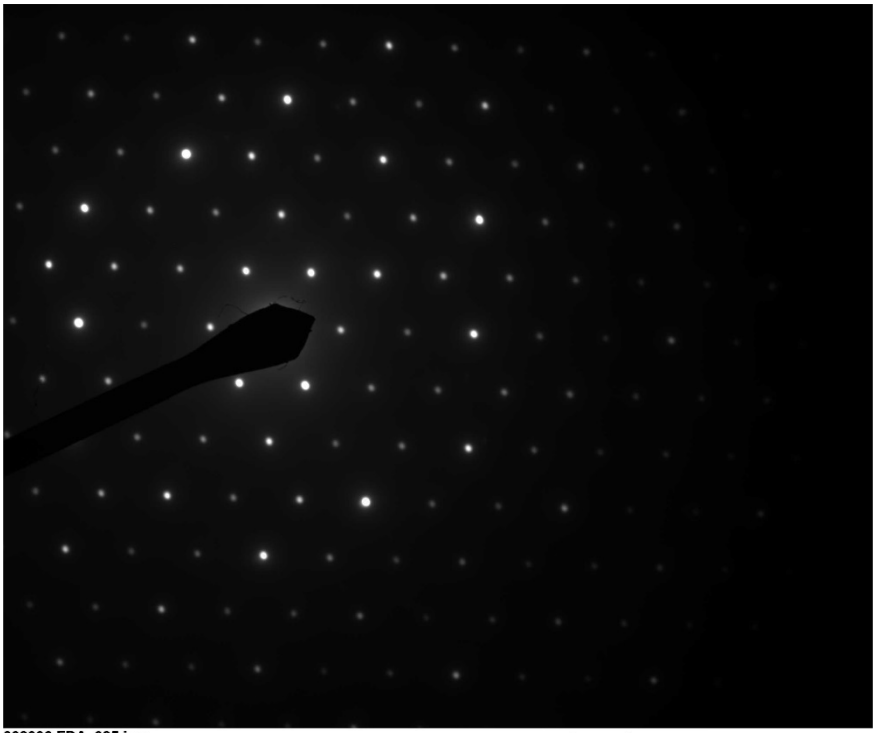
Sample 308006-9, Talc Particle



308006 FDA_084.jpg
Talc Particle
Cal: 0.002144 µm/pix
14:09 9/5/2019
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

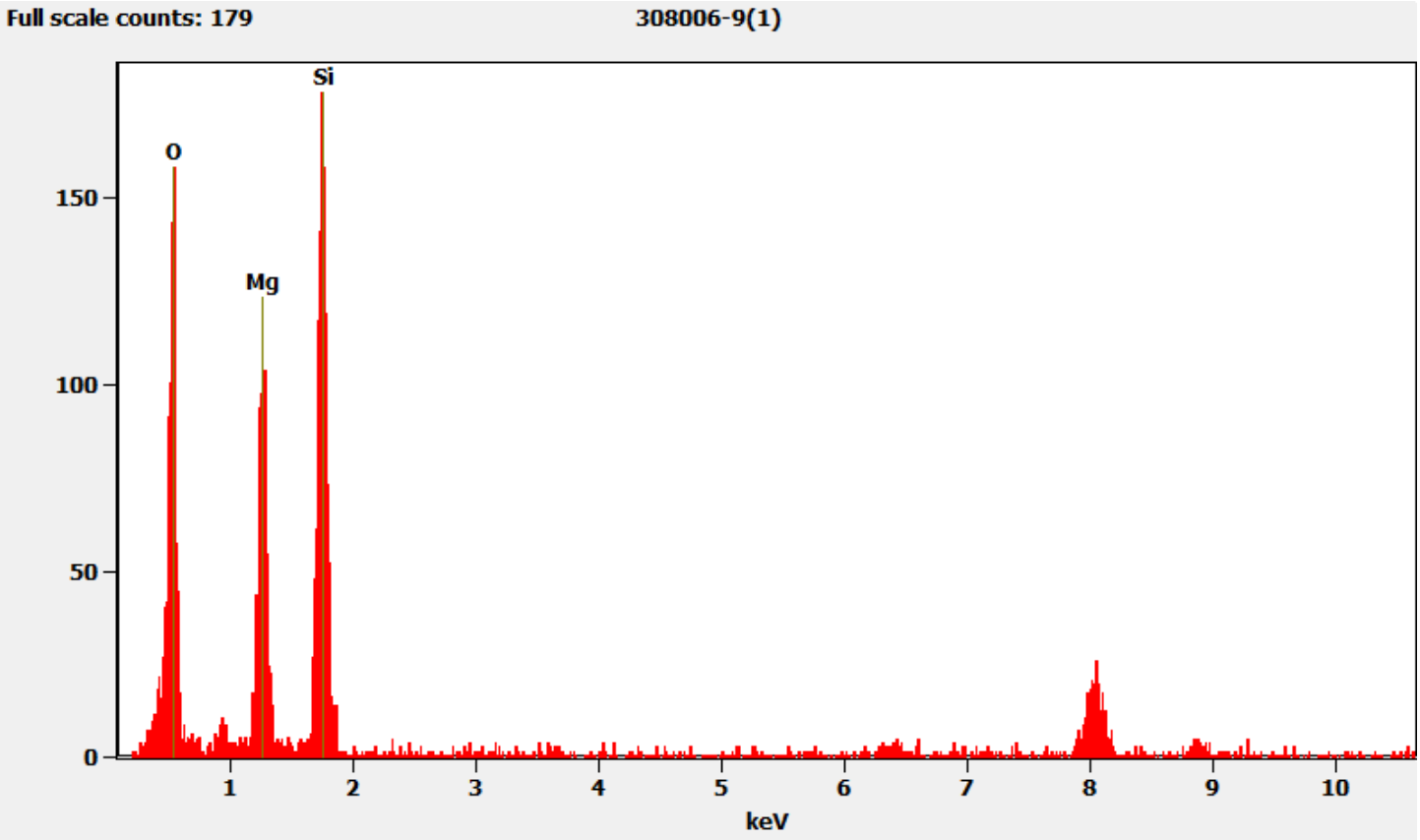
Hexagonal diffraction from the Talc particle pictured above.



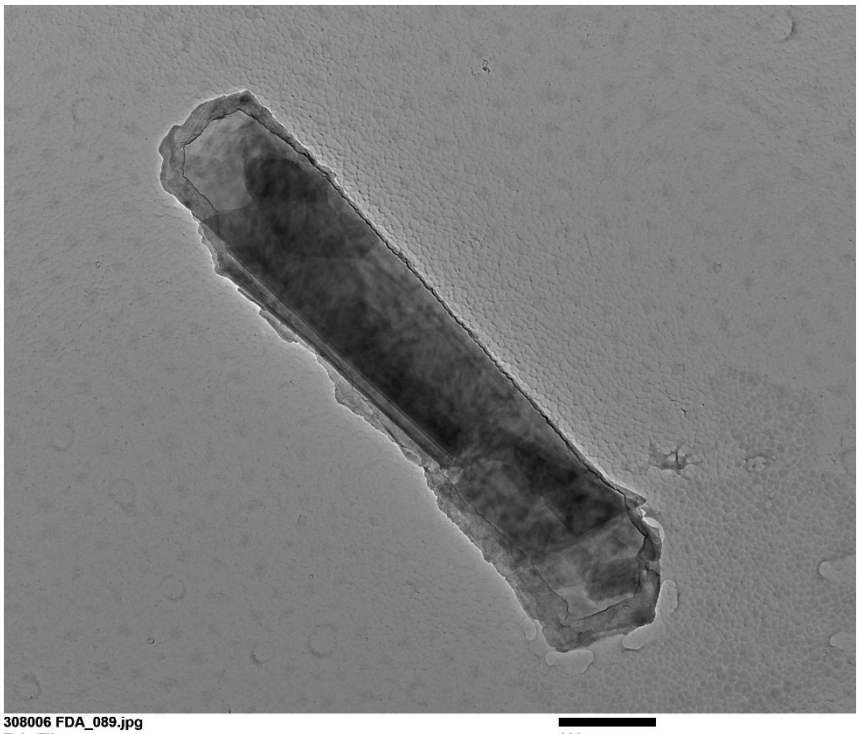
308006 FDA_085.jpg
Talc Particle
14:10 9/5/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from the Talc particle pictured above.

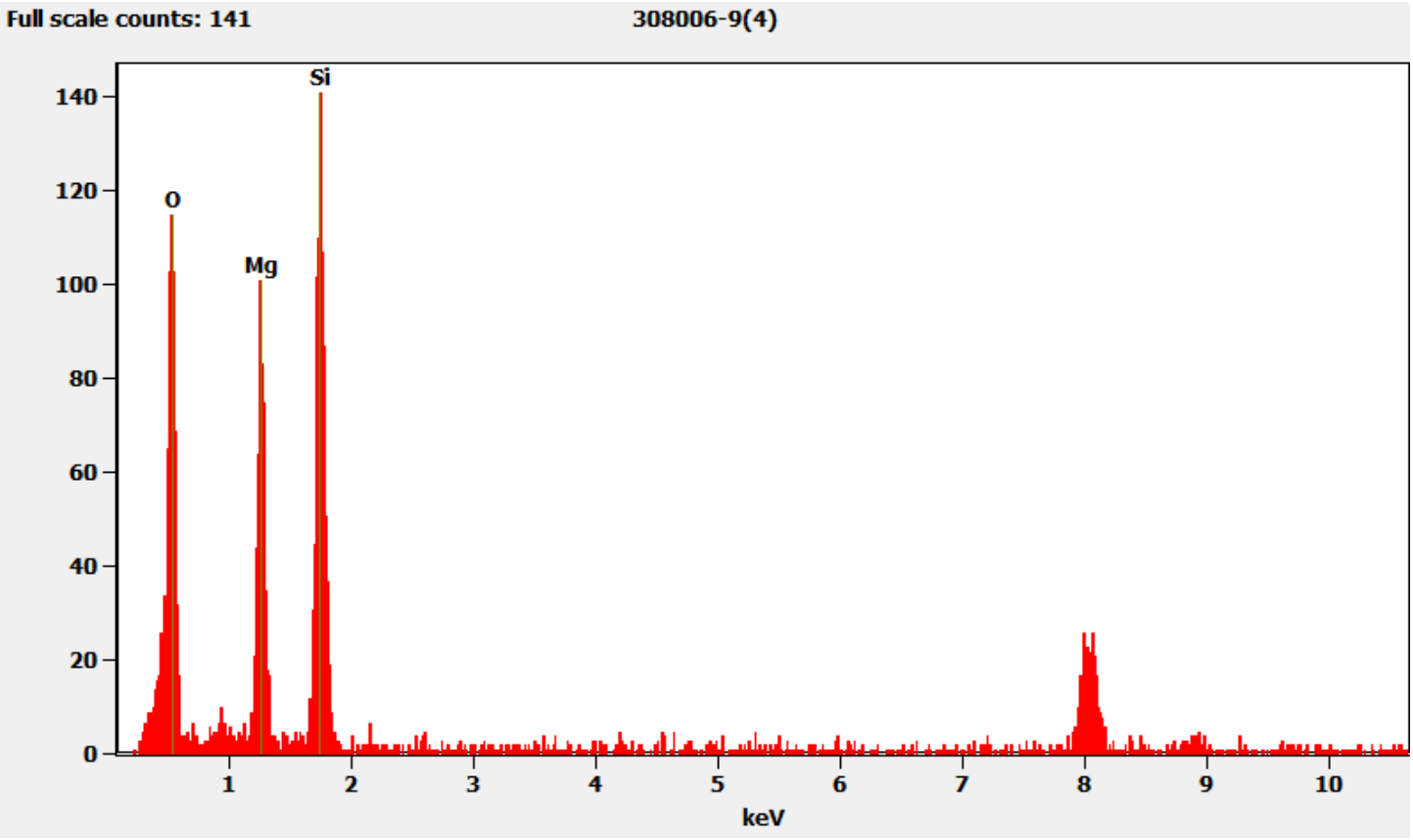


Sample 308006-9, Talc Fiber



Diffraction pattern from the Talc fiber pictured above.

Chemistry from the Talc fiber pictured above

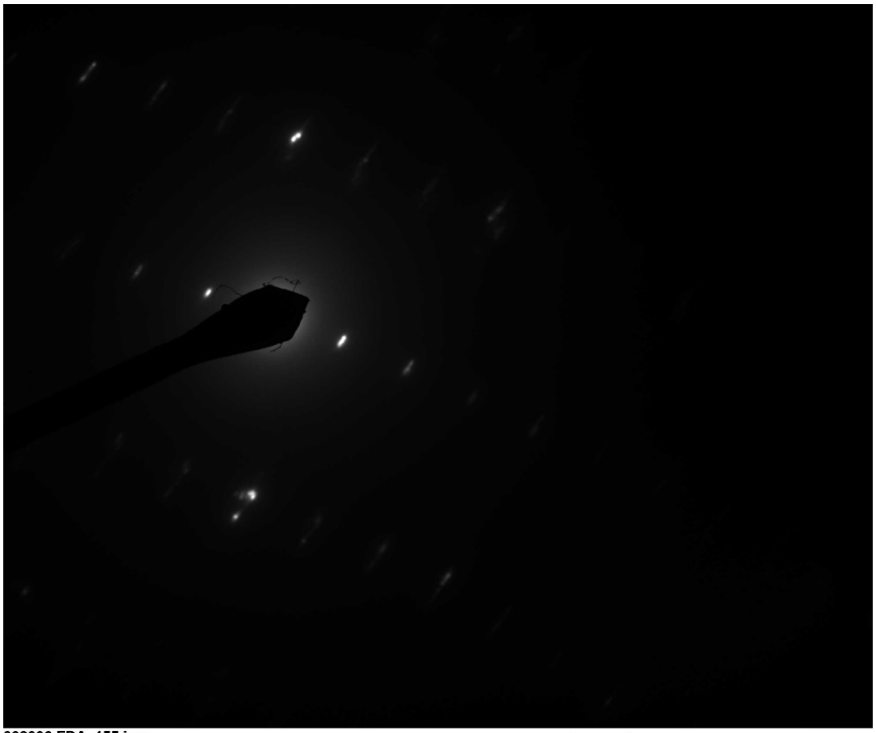


Sample 308006-9A, Talc Ribbon

308006 FDA_154.jpg
Talc Ribbon
Cal: 0.005415 µm/pix
15:05 9/12/2019
TEM Mode: Imaging
Microscopist: (b) (6)
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

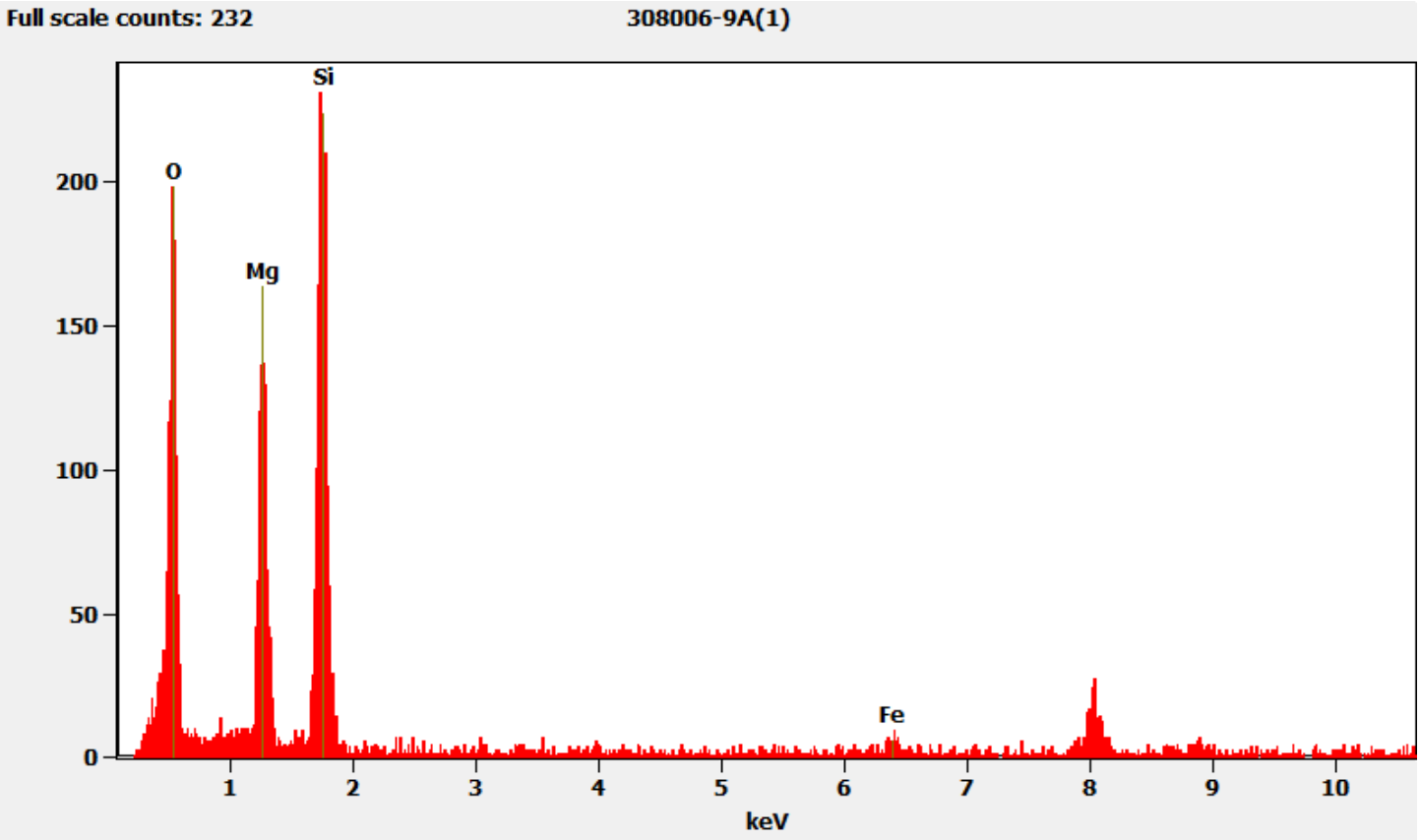
Diffraction pattern from the Talc ribbon pictured above.



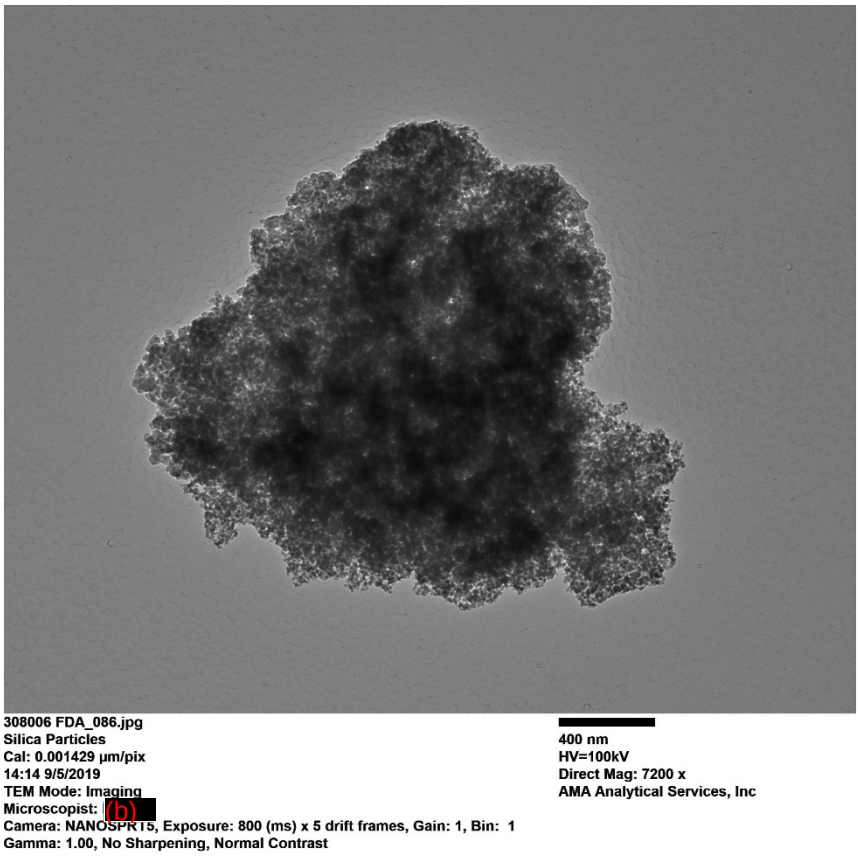
308006 FDA_155.jpg
Talc Ribbon
15:06 9/12/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

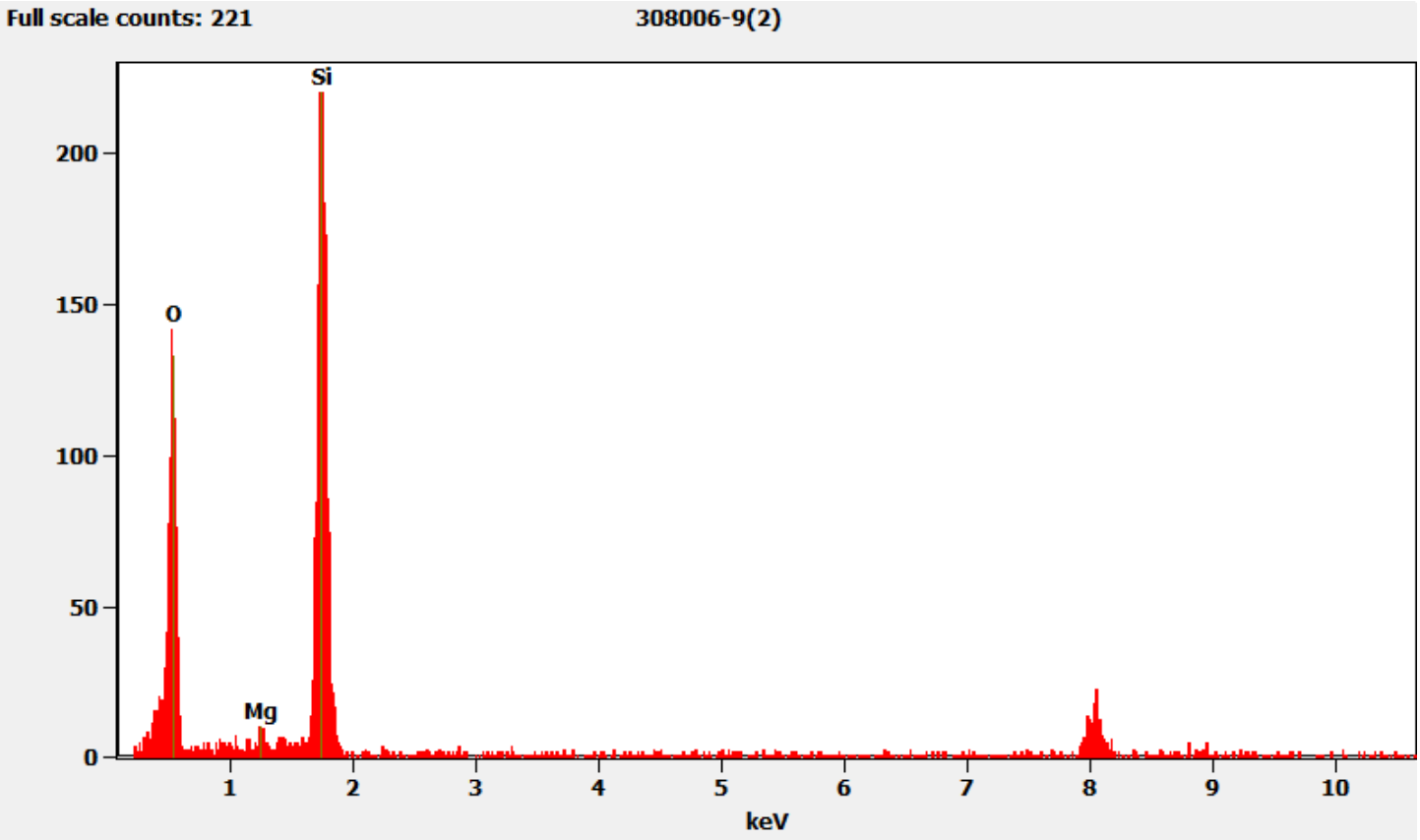
Chemistry from the Talc ribbon particle pictured above.



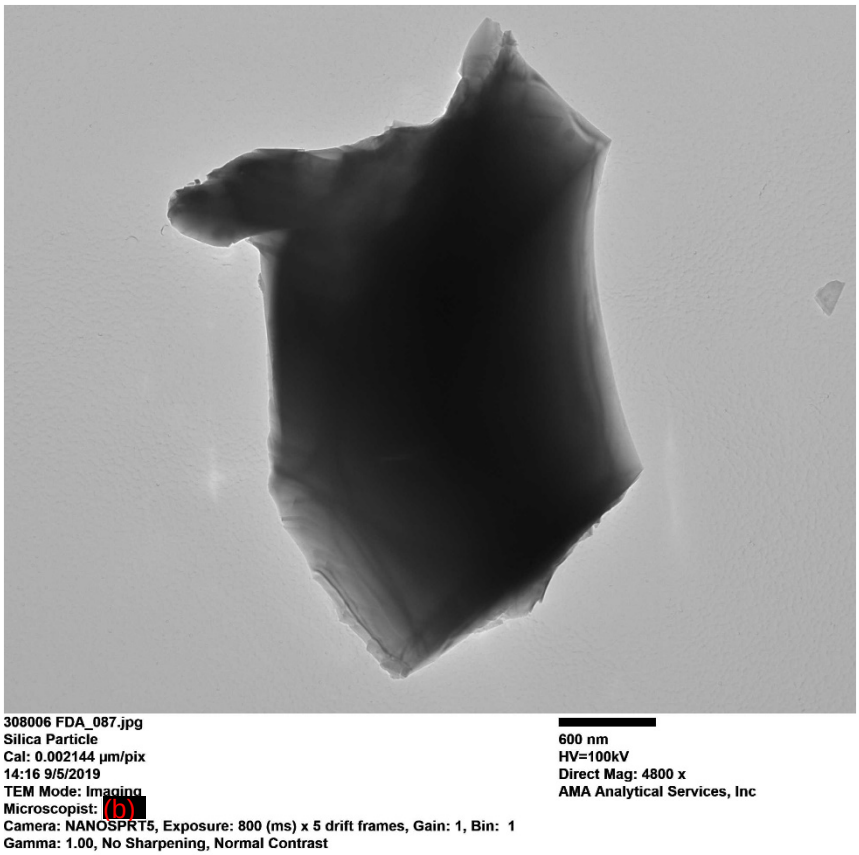
Sample 308006-9, Silica Particles



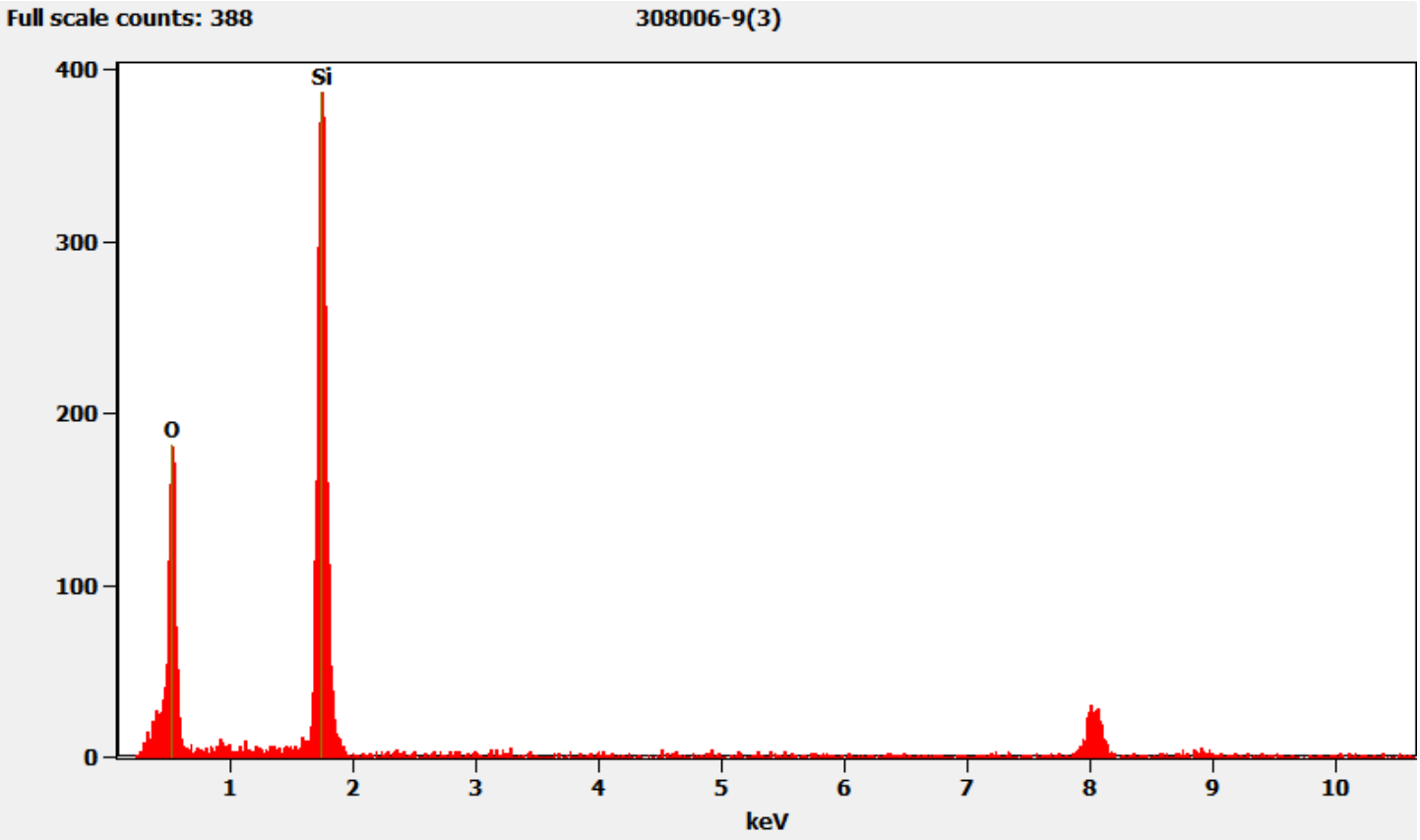
Chemistry from the Silica particles pictured above.



Sample 308006-9, Silica Particle



Chemistry from the Silica particle pictured above



<u>308006-10, 10A, 10B, Client Sample D-62</u>

PLM

All three aliquots of sample D-62 were analyzed by (b) (6) on September 13, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

308006-10	NAD
308006-10A	NAD
308006-10B	NAD

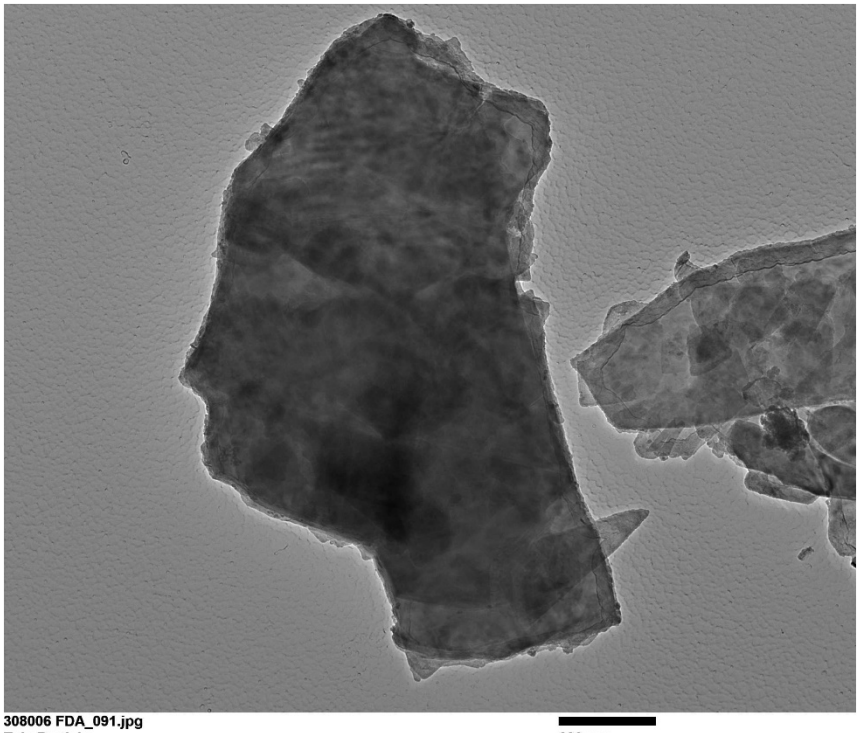
TEM

(b) (6) analyzed sample 10 on September 5, 2019 and sample 10B on September 15, 2019. (b) (6) analyzed sample 10A on September 12 & 18, 2019. The primary particle observed was talc along with a few talc fibers, talc ribbons, some silica particles, silica spheres and aluminum spheres. No asbestos or non-asbestos amphibole variants were detected in the samples. The results were calculated using the equations detailed in the calculations section.

308006-10	NAD
308006-10A	NAD
308006-10B	NAD

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

Sample 308006-10, Talc Particle



308006 FDA_091.jpg
Talc Particle
Cal: 0.002144 µm/pix
15:11 9/5/2019
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

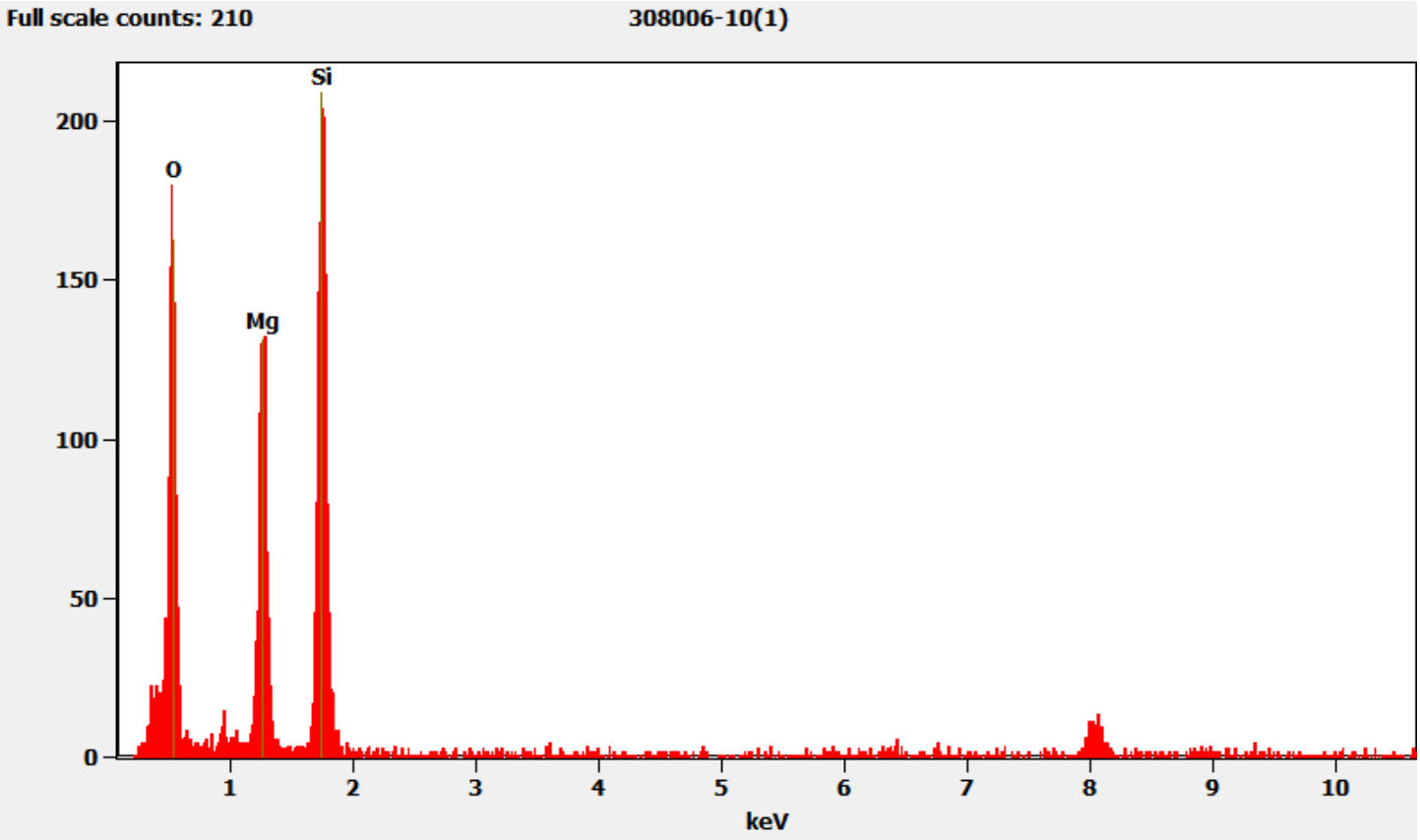
600 nm
HV=100kV
Direct Mag: 4800 x
AMA Analytical Services, Inc

Hexagonal diffraction pattern from the Talc particle pictured above.

308006 FDA_092.jpg
Talc Particle
15:12 9/5/2019
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

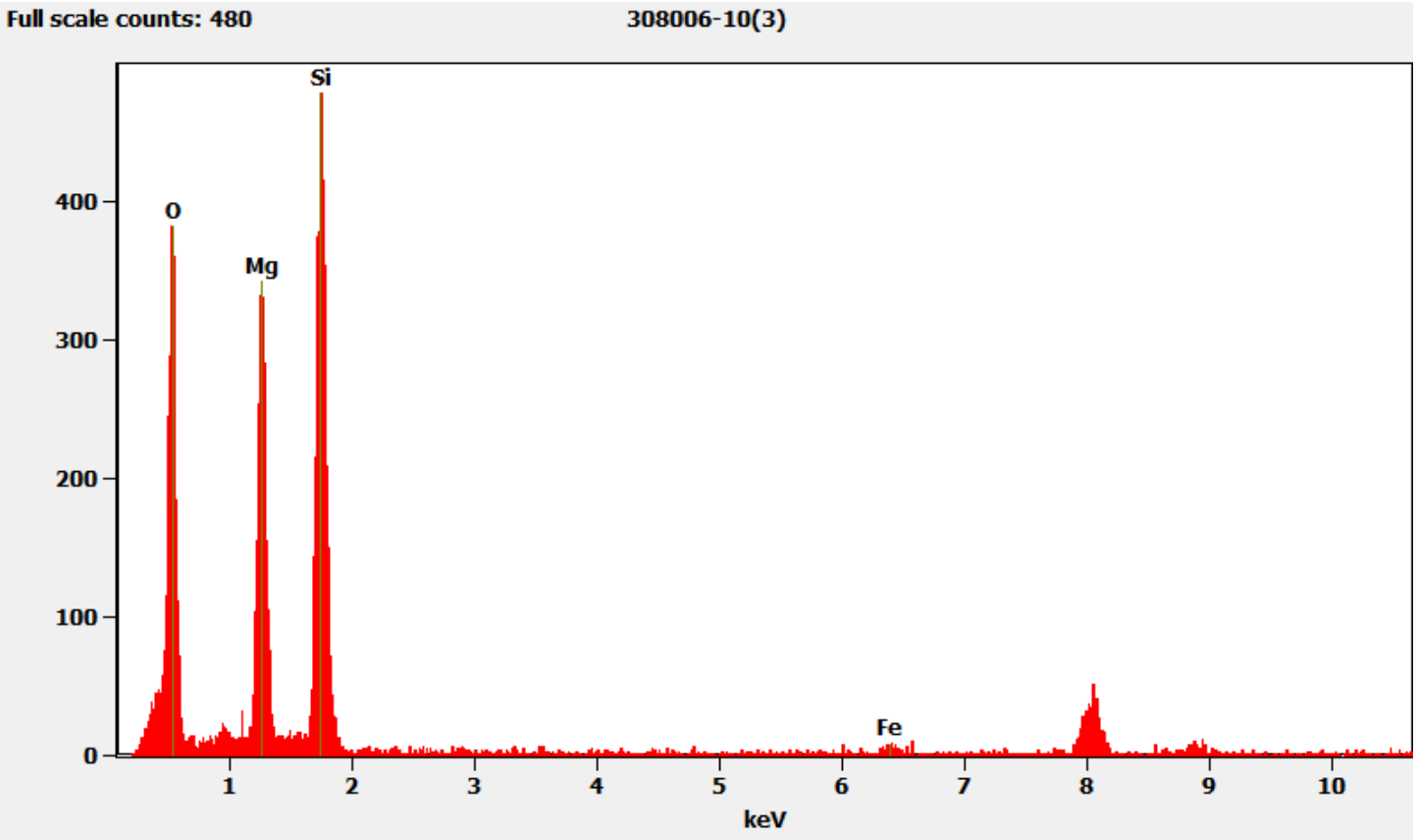
Chemistry from the Talc particle pictured above.



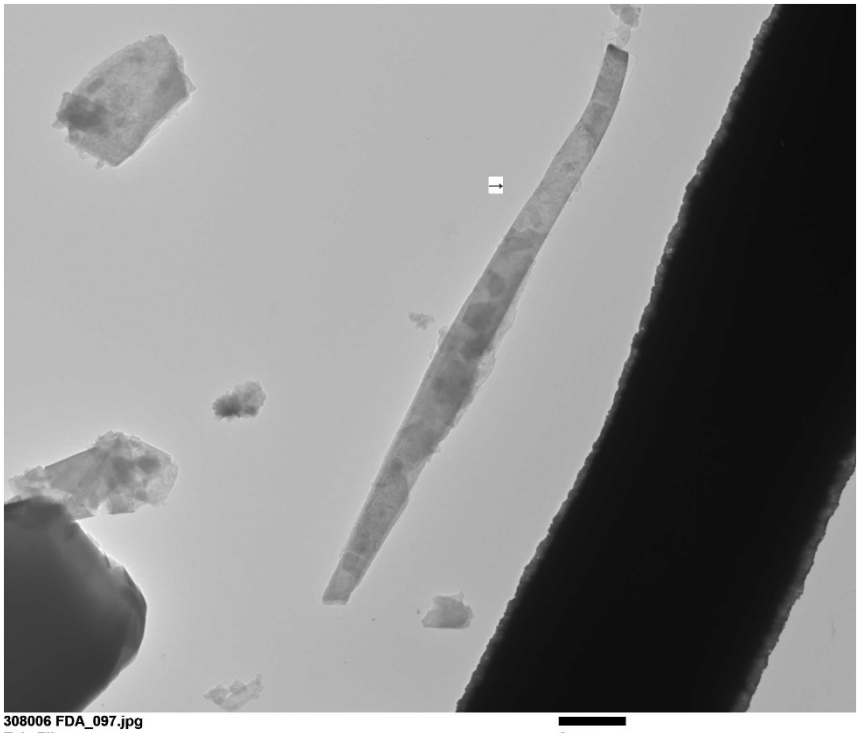
Sample 308006-10, Talc Ribbon

Diffraction pattern from the Talc ribbon pictured above.

Chemistry from the Talc ribbon pictured above



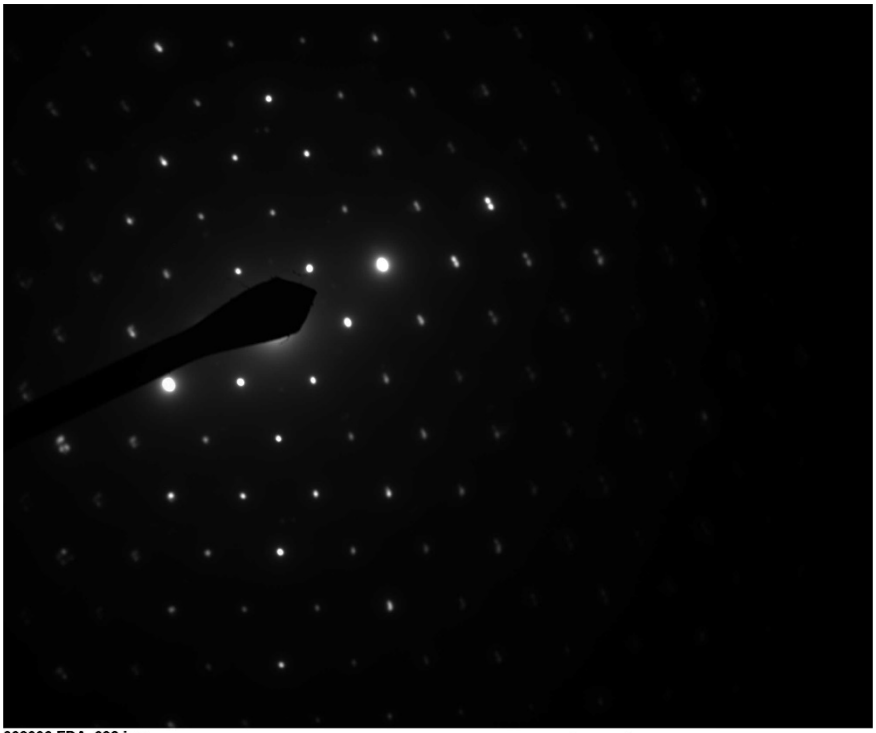
Sample 308006-10, Talc Fiber



308006 FDA_097.jpg
Talc Fiber
Cal: 0.010289 µm/pix
16:07 9/5/2019
TEM Mode: Imaging
Microscopist: (h)
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

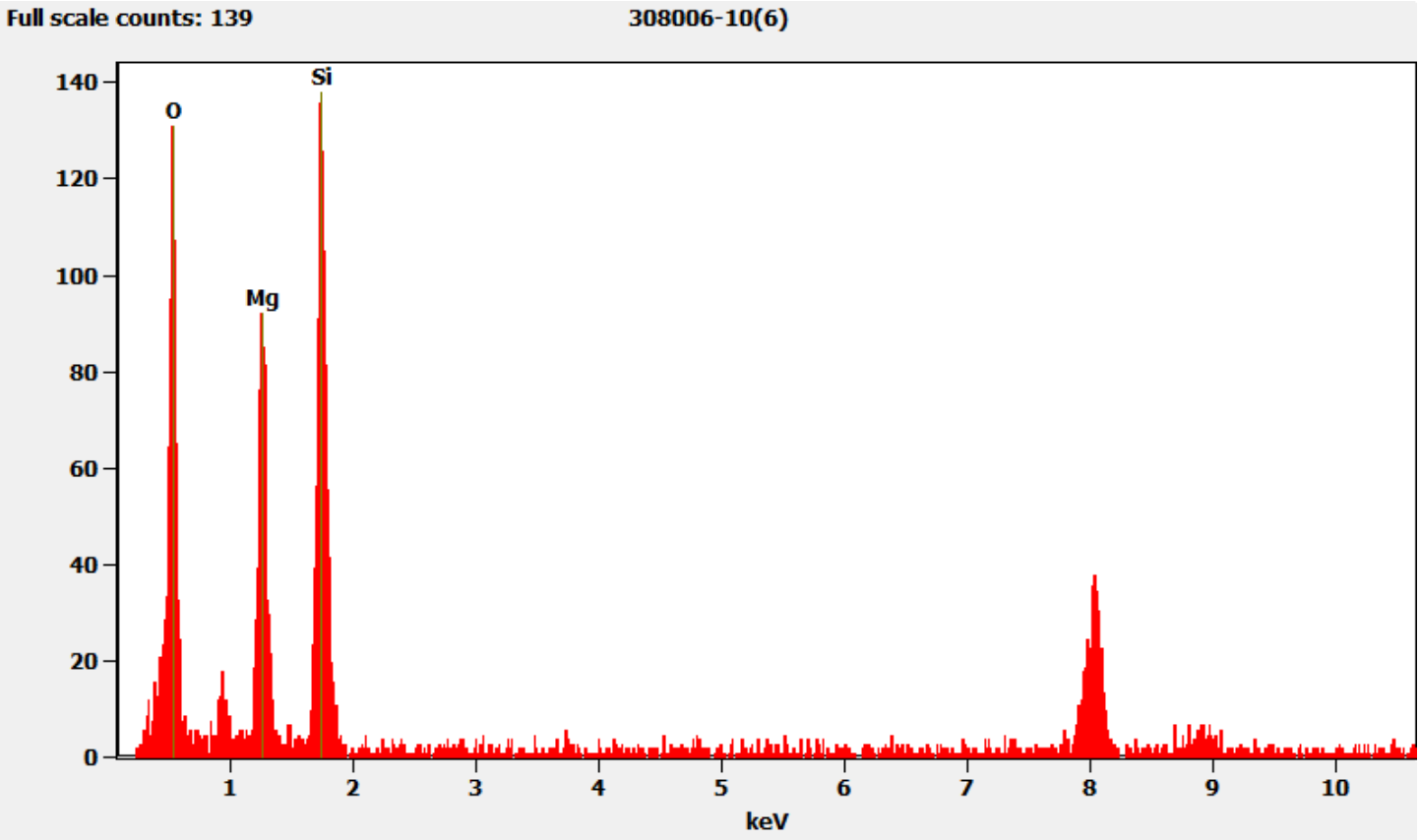
Hexagonal diffraction pattern from the Talc fiber pictured above.



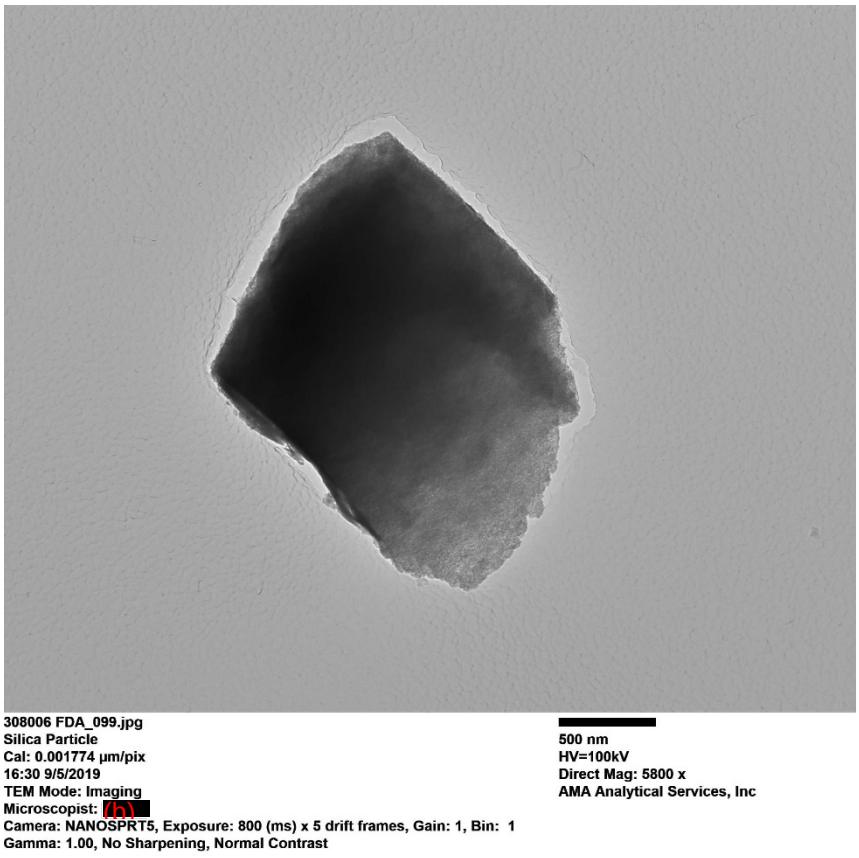
308006 FDA_098.jpg
Talc Fiber
16:08 9/5/2019
TEM Mode: Diffraction
Microscopist: (h)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

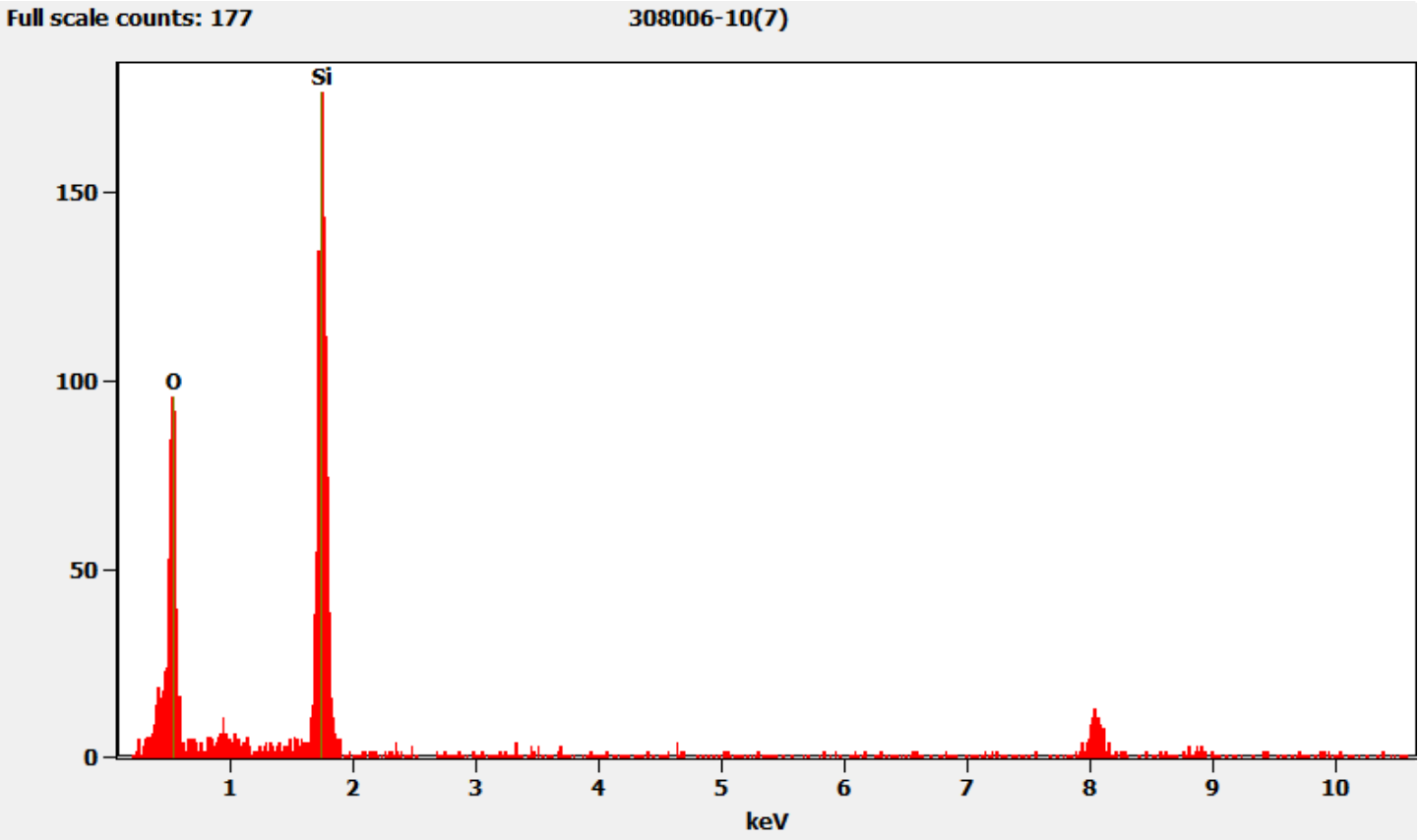
Chemistry from the Talc fiber pictured above.



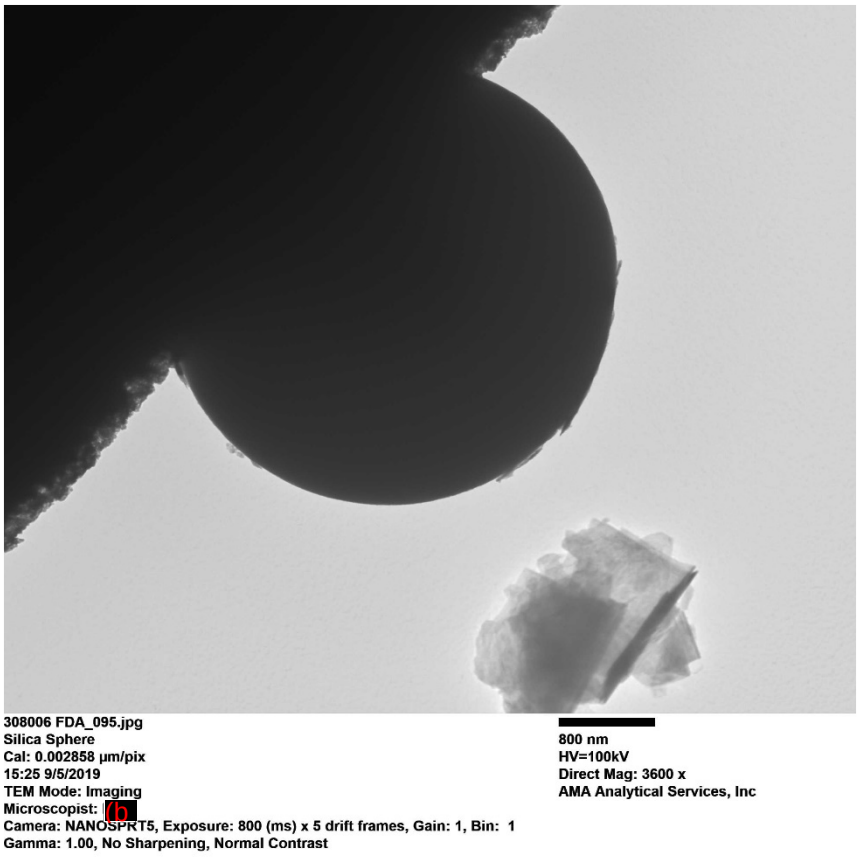
Sample 308006-10, Silica Particle



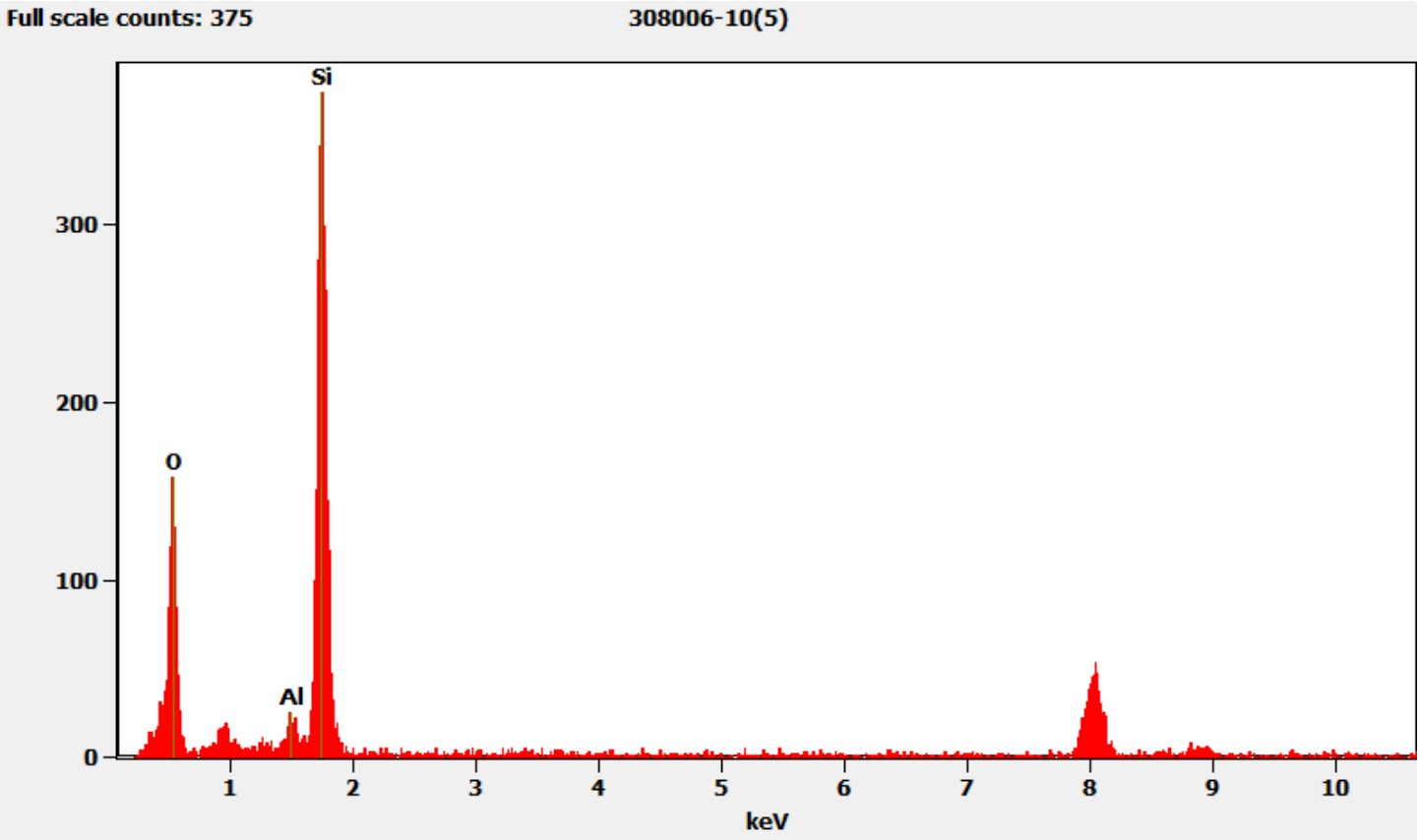
Chemistry pattern from the Silica particle pictured above.



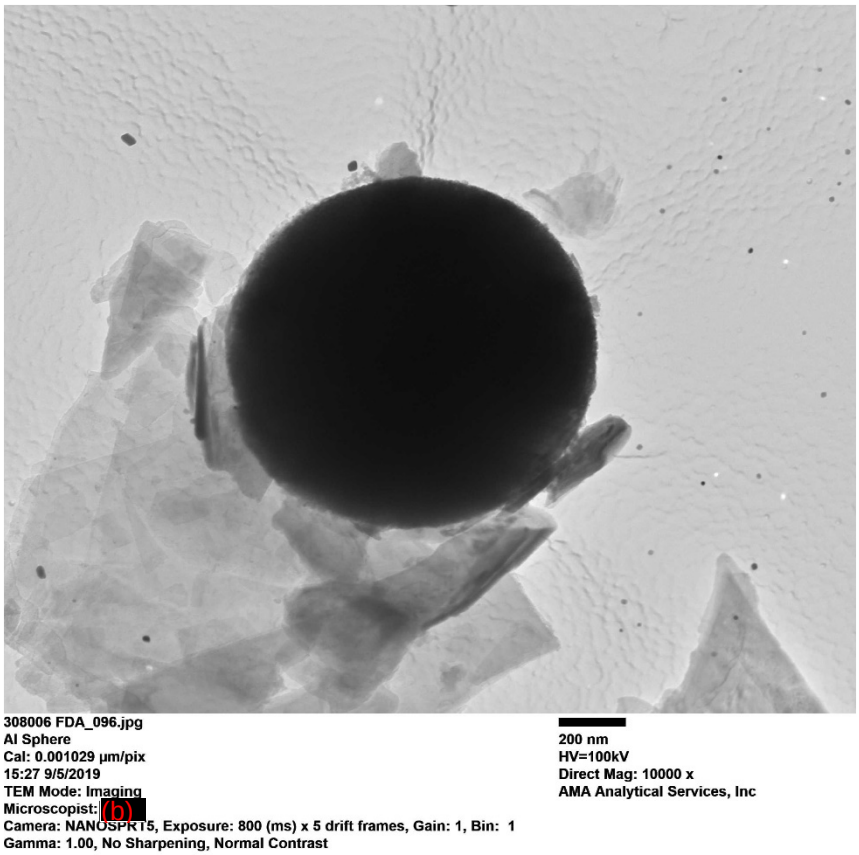
Sample 308006-10, Silica Sphere



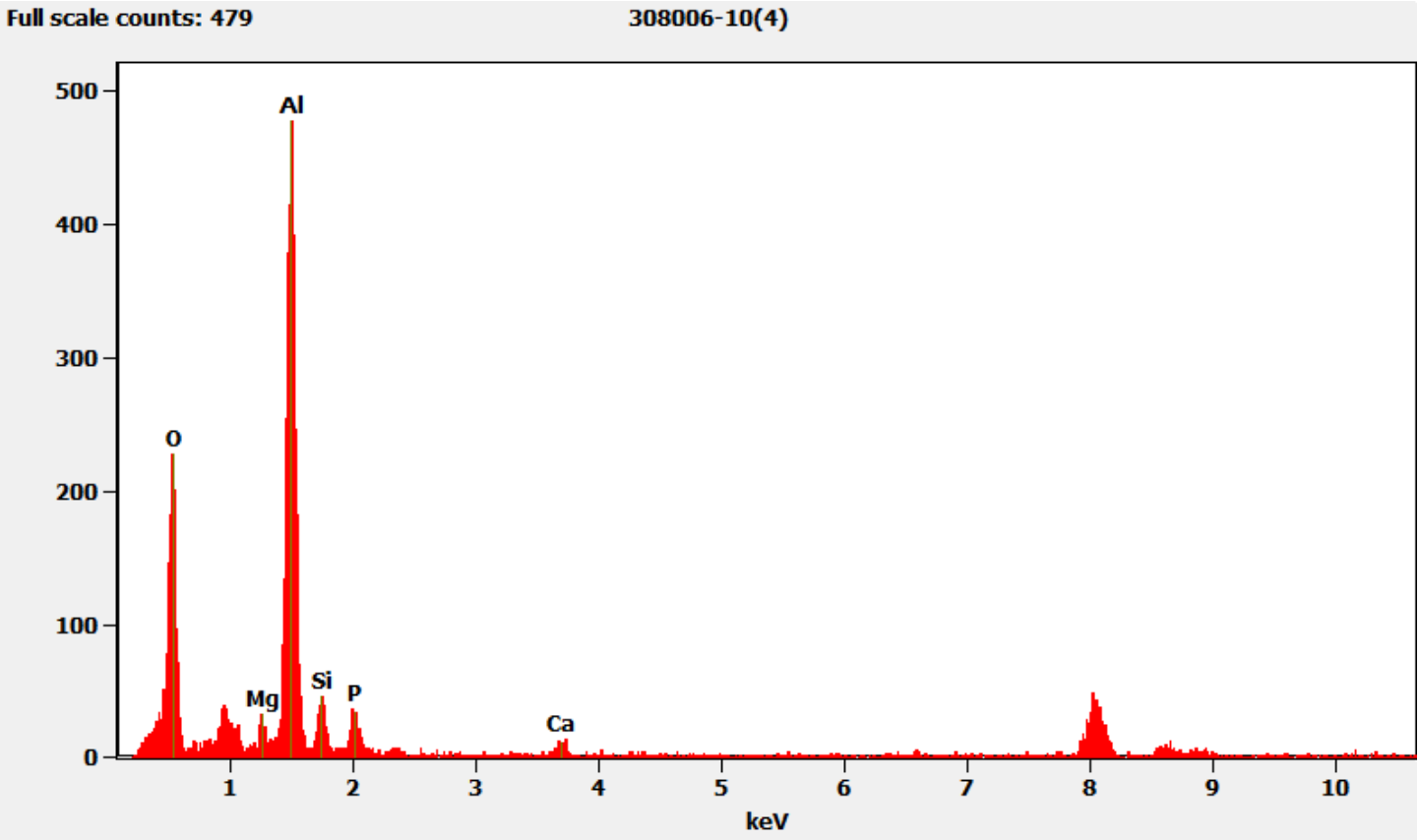
Chemistry from the Silica sphere pictured above



Sample 308006-10, Aluminum Sphere



Chemistry from the Aluminum sphere pictured above



<u>308006-11, 11A, 11B, Client Sample D-63</u>

PLM

All three aliquots of sample D-63 were analyzed by (b) (6) on September 13, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

308006-11	NAD
308006-11A	NAD
308006-11B	NAD

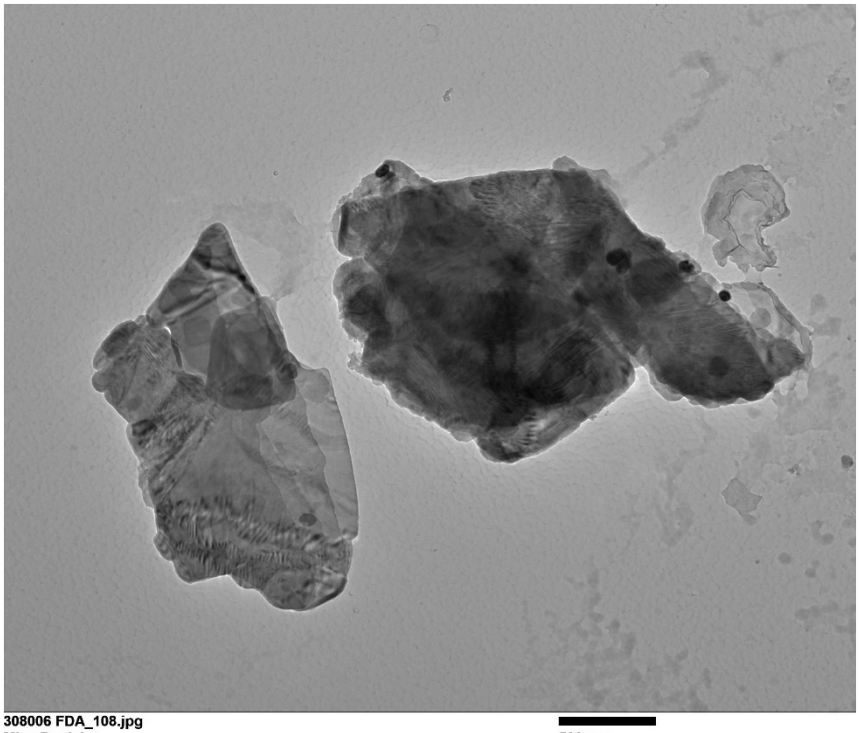
TEM

(b) (6) analyzed sample 11 on September 8, 2019 and samples 11A and 11B on September 15, 2019. The primary particle observed was mica along with a few talc particles and iron particles. No asbestos or non-asbestos amphibole variants were detected in the samples. The results were calculated using the equations detailed in the calculations section.

308006-11	NAD
308006-11A	NAD
308006-11B	NAD

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

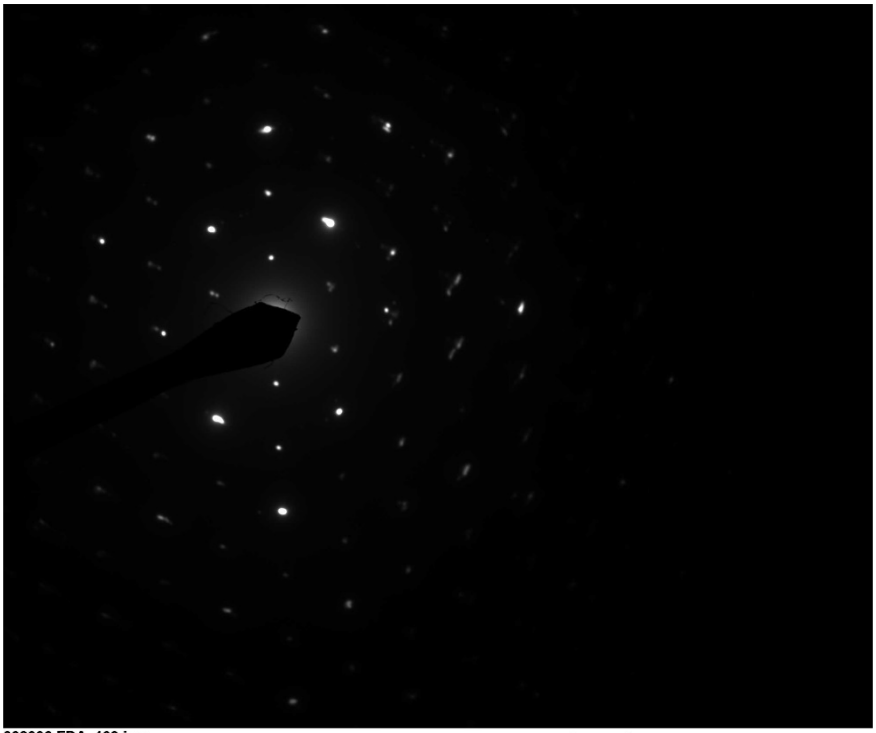
Sample 308006-11, Mica Particles



308006 FDA_108.jpg
Mica Particles
Cal: 0.001774 µm/pix
12:31 9/8/2019
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

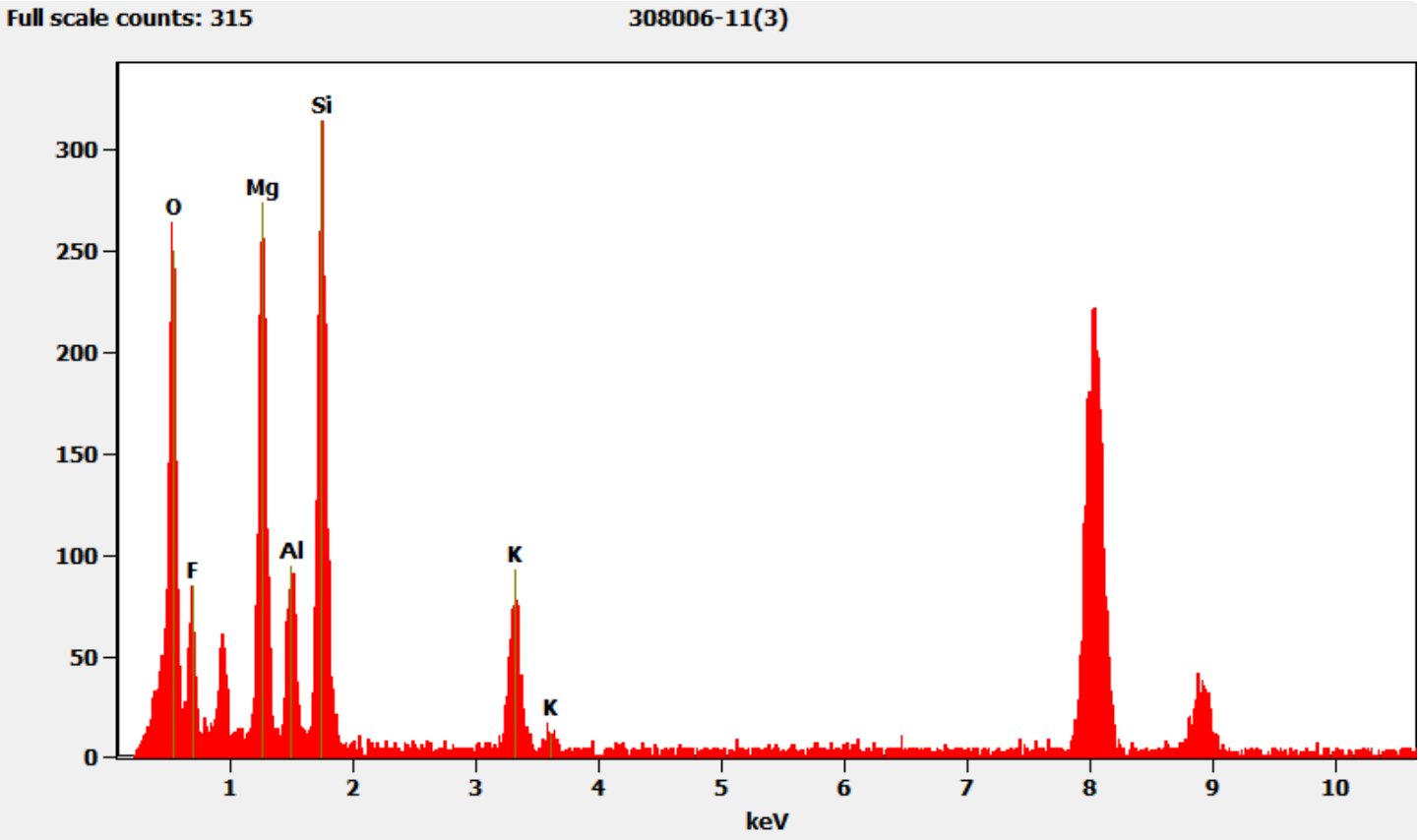
Diffraction patterns from the Mica particles pictured above.



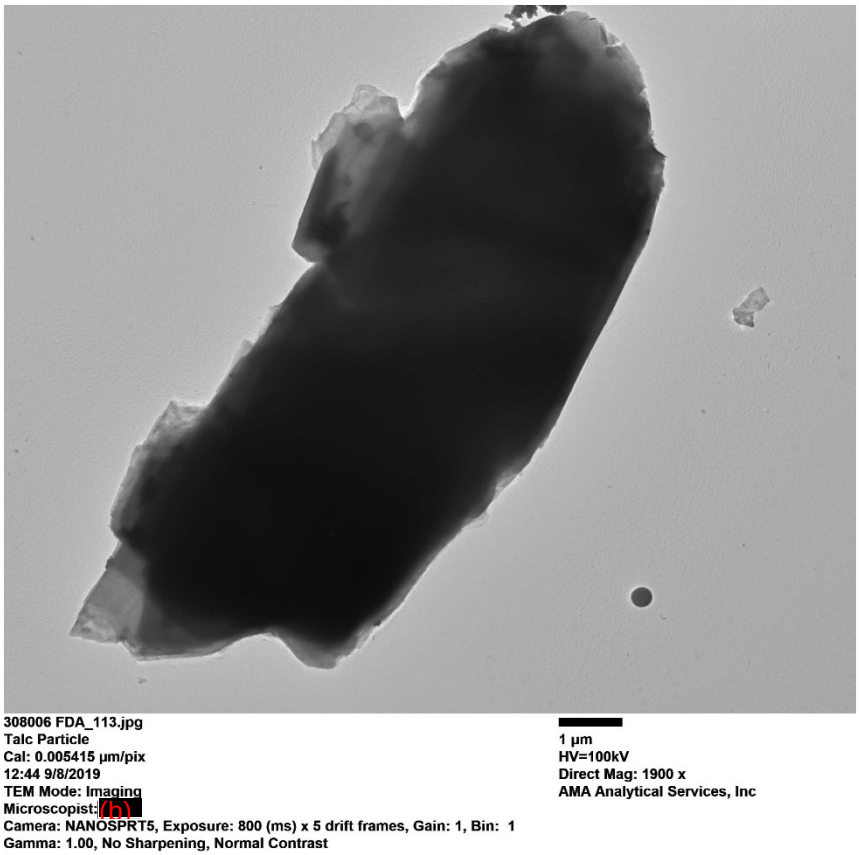
308006 FDA_109.jpg
Mica Particle
12:32 9/8/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from the Mica particles pictured above.

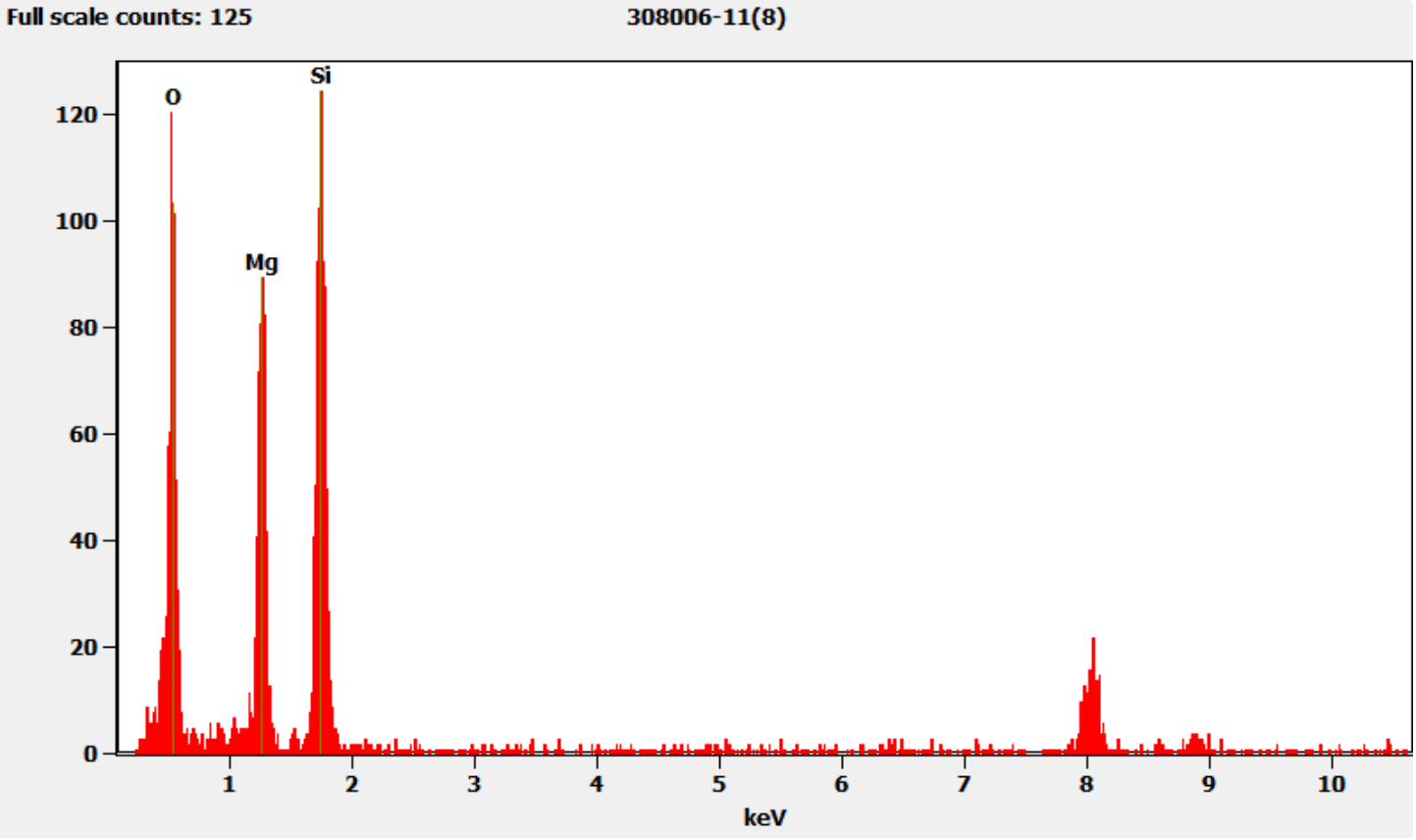


Sample 308006-11, Talc particle

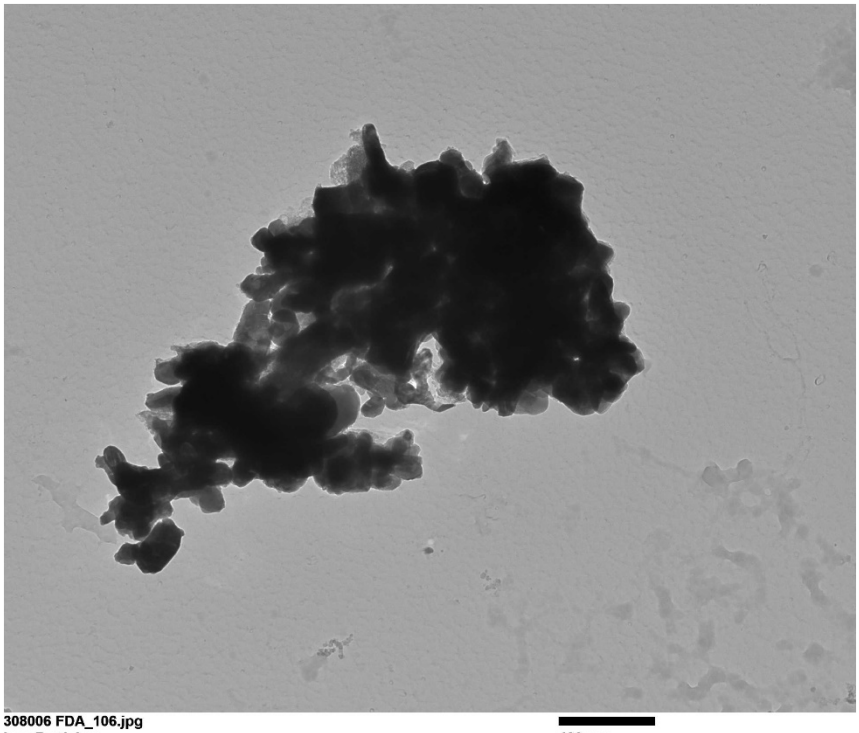


Hexagonal diffraction pattern from the talc particle pictured above.

Chemistry from the talc particle pictured above.



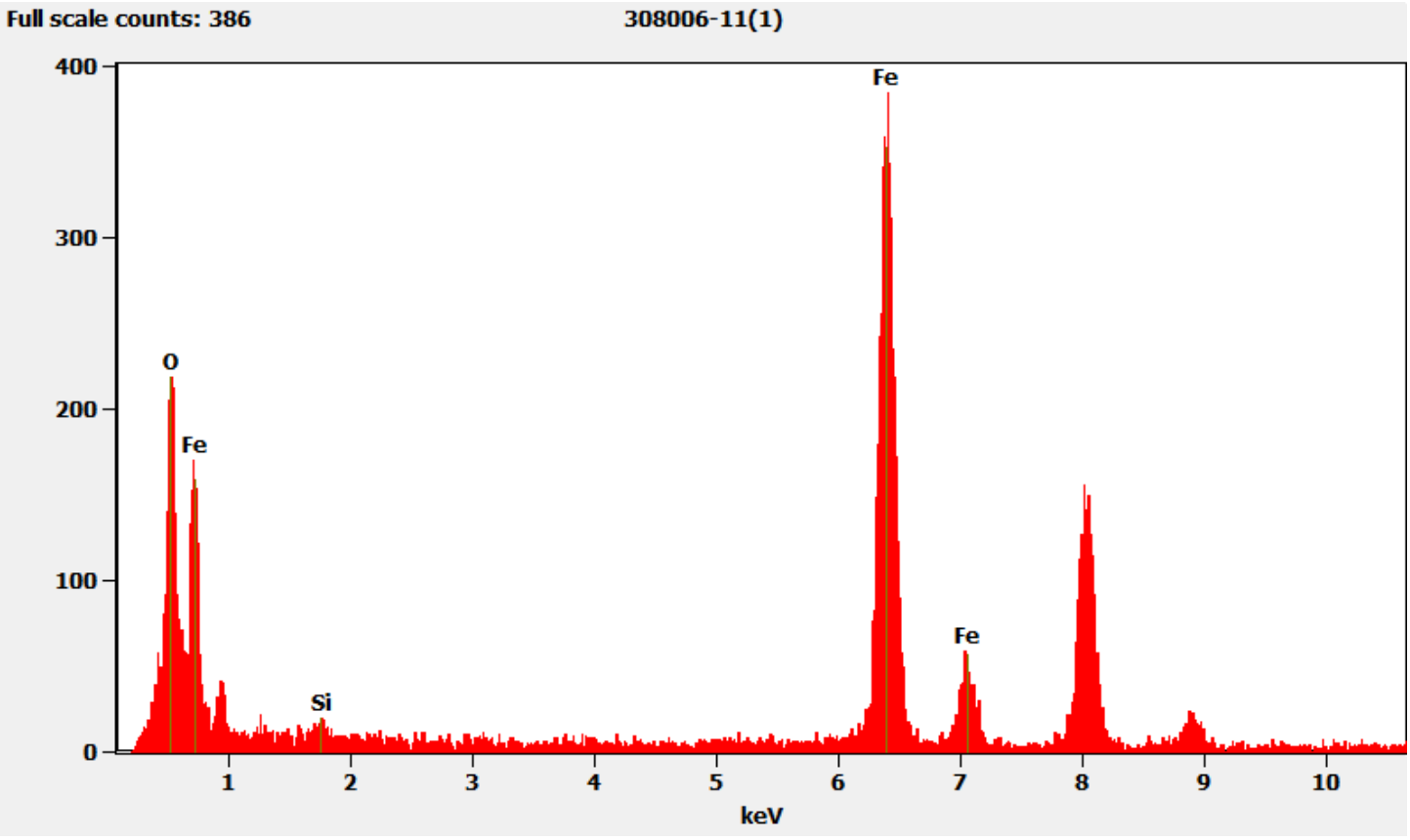
Sample 308006-11, Iron Particles



308006 FDA_106.jpg
Iron Particles
Cal: 0.001429 µm/pix
12:25 9/8/2019
TEM Mode: Imaging
Microscopist:
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

Chemistry from the Iron particles pictured above



308006-12, 12A, 12B, Client Sample D-64

PLM

All three aliquots of sample D-64 were analyzed by (b) (6) on September 13, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

308006-12	NAD
308006-12A	NAD
308006-12B	NAD

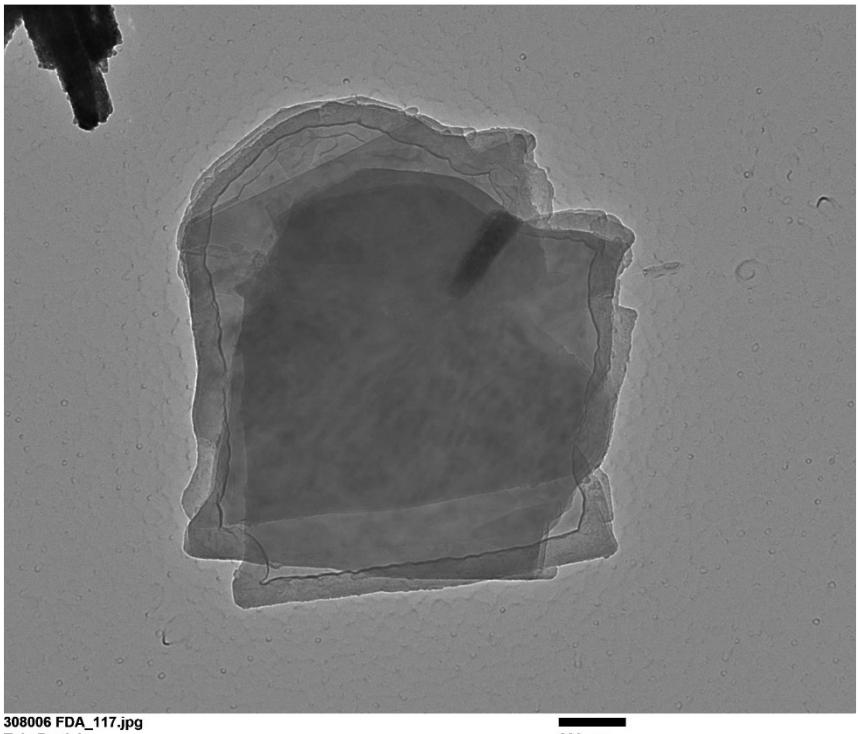
TEM

(b) (6) analyzed sample 12 on September 8, 2019 and sample 12A on September 15, 2019. (b) (6) analyzed sample 12B on September 18, 2019. The primary particle observed was talc along with lots of smaller iron particles/fibers along, a few talc fibers and some silica spheres. No asbestos or non-asbestos amphibole variants were detected in the samples. The results were calculated using the equations detailed in the calculations section.

308006-12	NAD
308006-12A	NAD
308006-12B	NAD

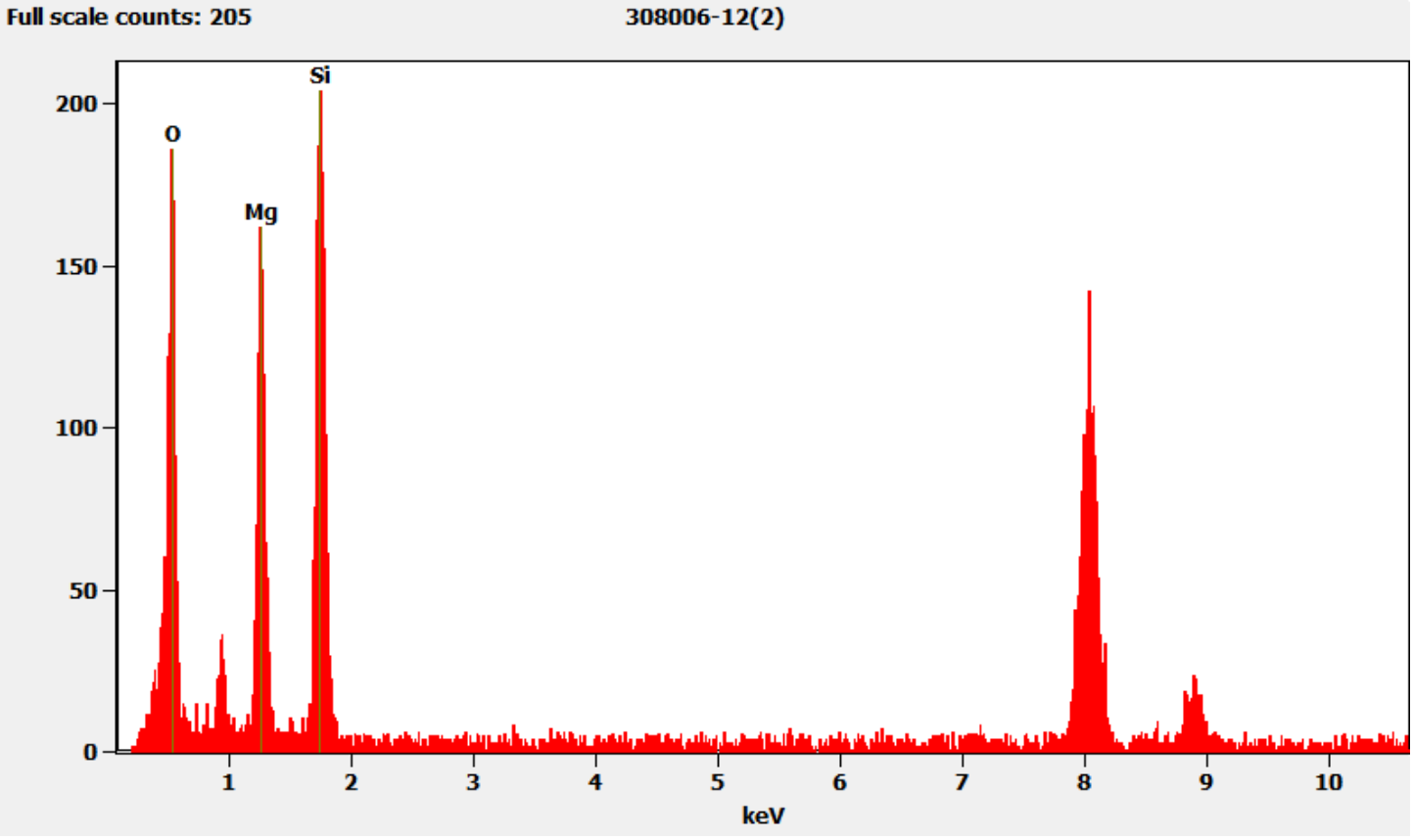
Below are pictures, diffraction patterns, and chemistry from some of the counted particles. The unidentified peaks in chemistry spectra are copper, zinc, and carbon. Those peaks are from the TEM specimen holder and specimen grid.

Sample 308006-12, Talc Particle

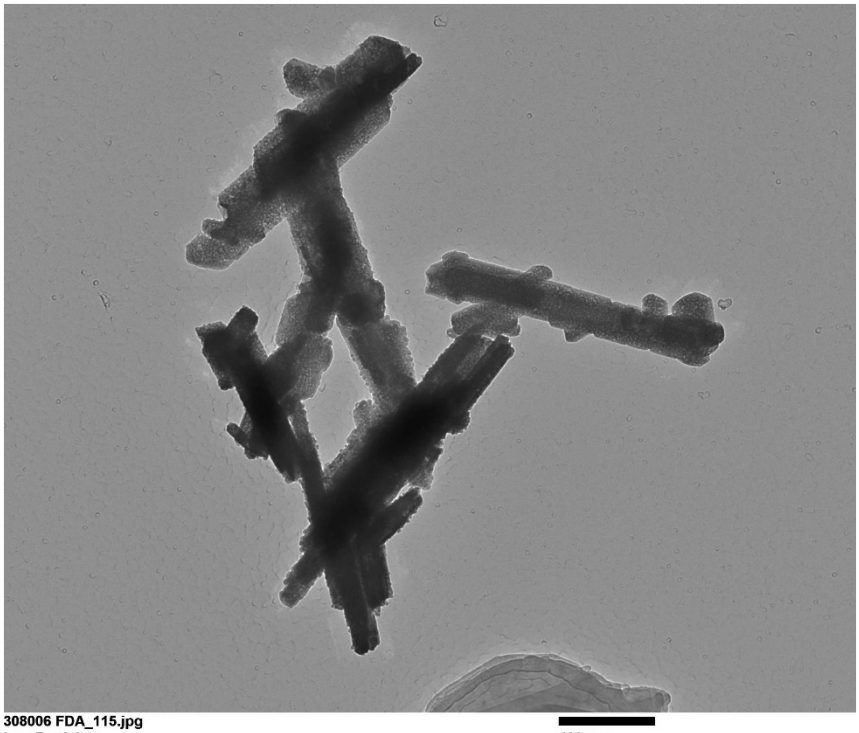


Hexagonal diffraction from the Talc particle pictured above.

Chemistry from the Talc particle pictured above.



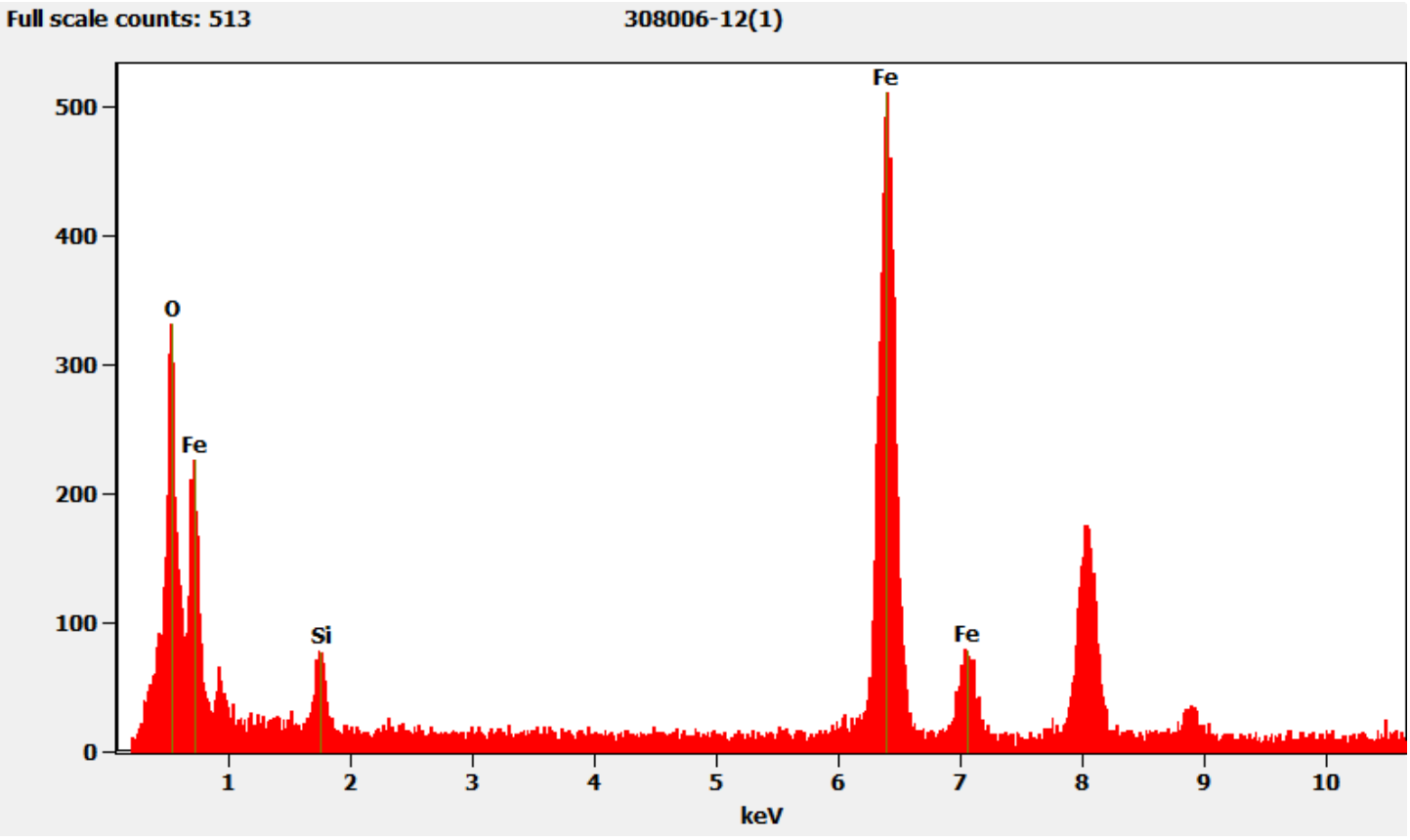
Sample 308006-12, Iron Particles/Fibers



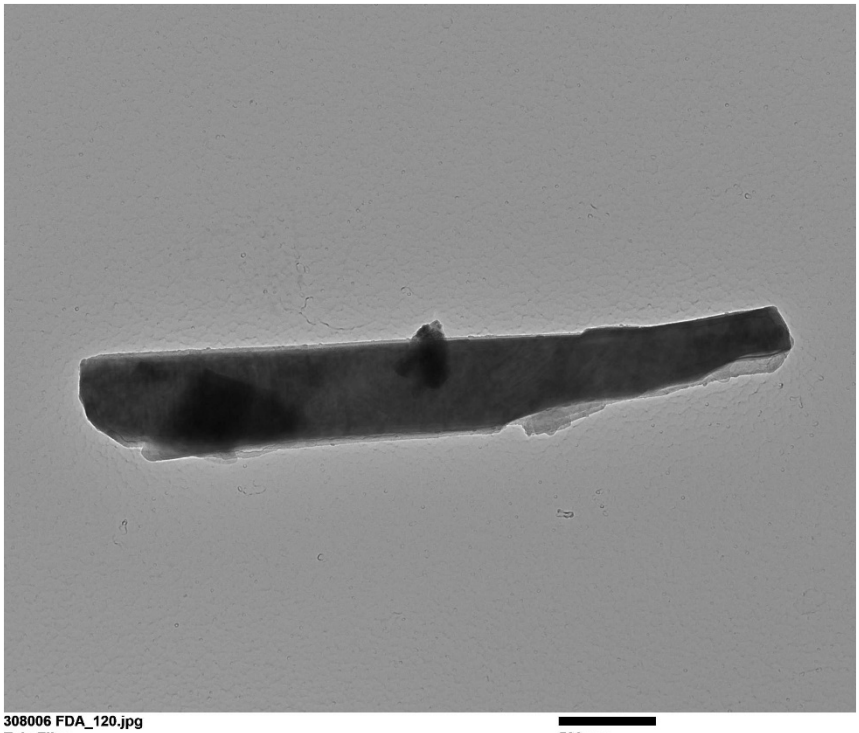
308006 FDA_115.jpg
Iron Particles
Cal: 0.001429 µm/pix
13:22 9/8/2019
TEM Mode: Imaging
Microscopist: (b)
Camera: NANO... XT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from the Iron particles/fibers pictured above



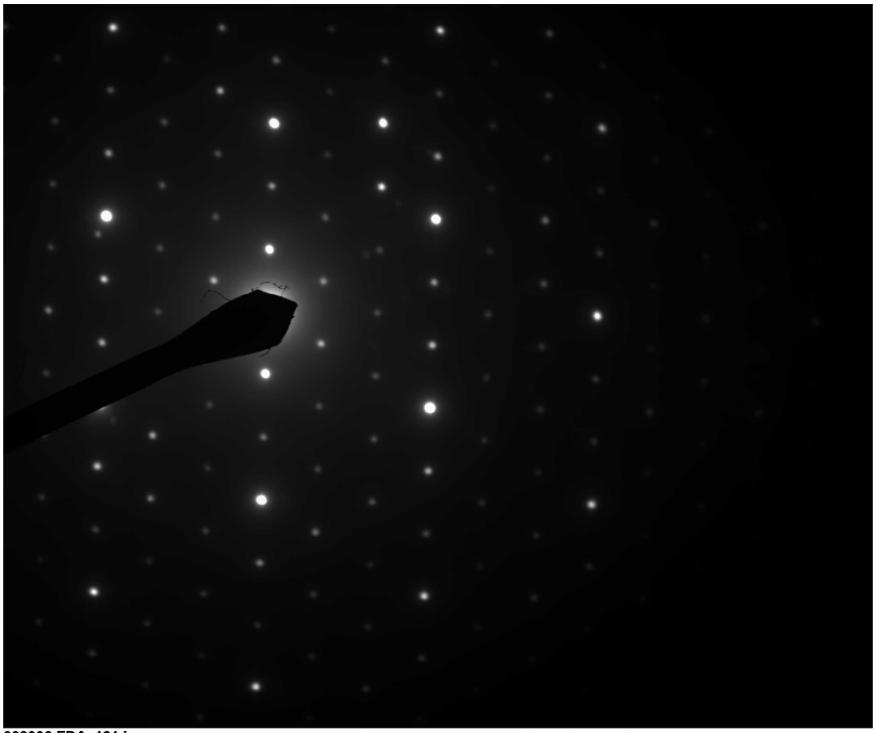
Sample 308006-12, Talc Fiber



308006 FDA_120.jpg
Talc Fiber
Cal: 0.001774 µm/pix
13:45 9/8/2019
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

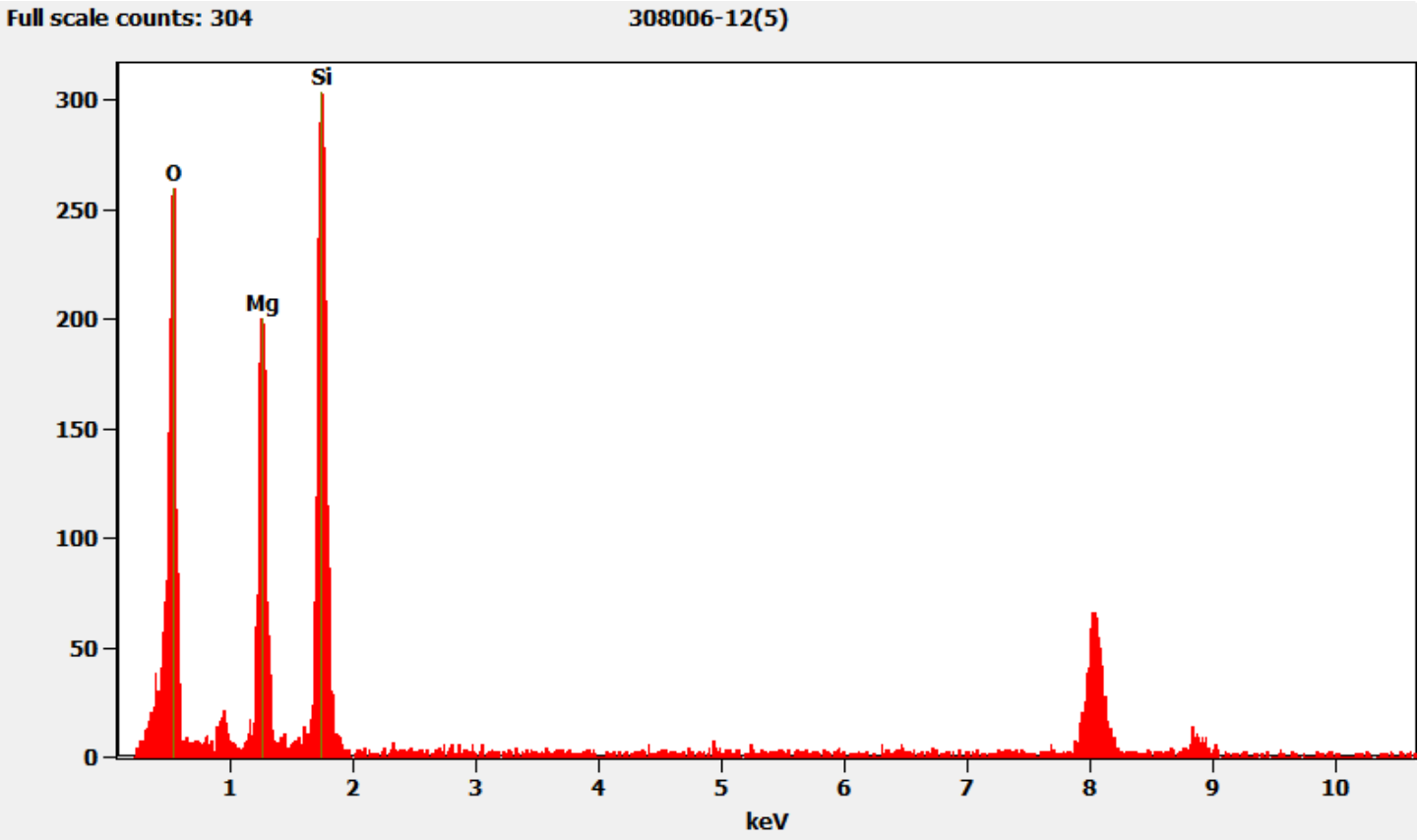
Hexagonal diffraction pattern from the Talc fiber pictured above.



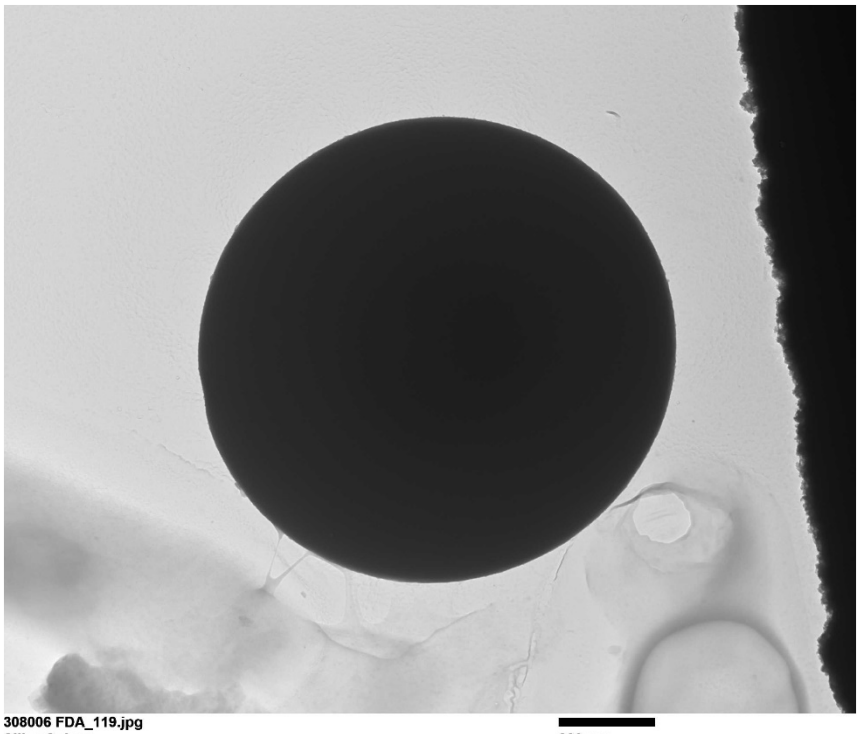
308006 FDA_121.jpg
Talc Fiber
13:46 9/8/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

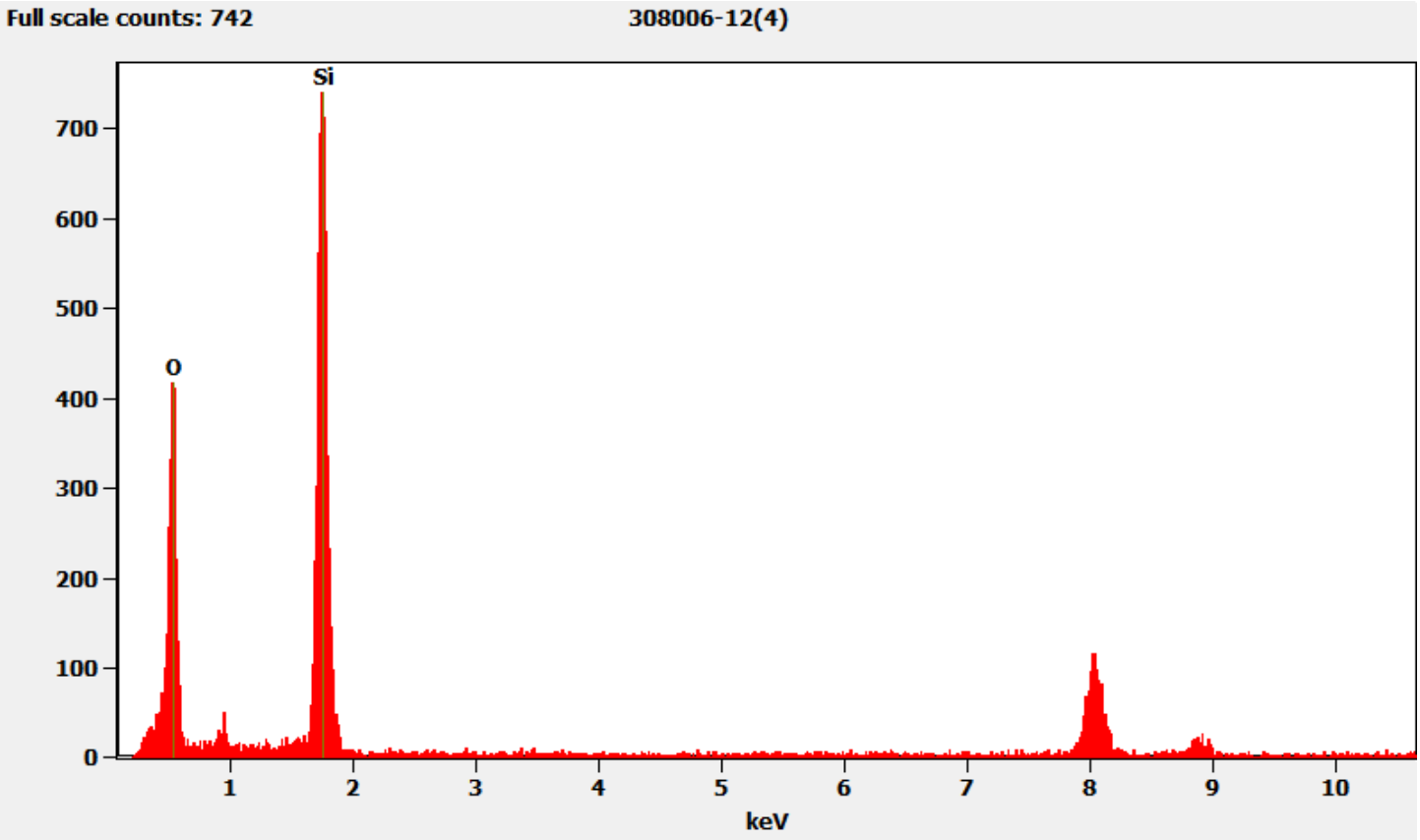
Chemistry from the Talc fiber pictured above.



Sample 308006-12, Silica Sphere



Chemistry from the Silica sphere pictured above



308006-13, 13A, 13B, Client Sample D-65

PLM

All three aliquots of sample D-65 were analyzed by (b) (6) e on September 13, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

308006-13	NAD
308006-13A	NAD
308006-13B	NAD

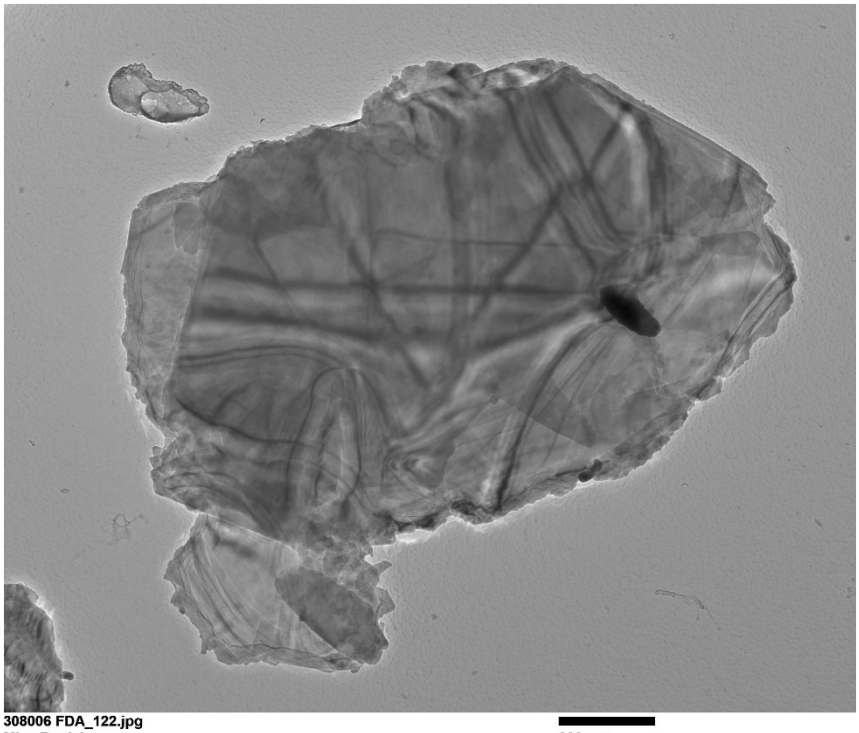
TEM

(b) (6) analyzed sample 13 on September 8, 2019 and sample 13A on September 18, 2019. (b) (6) analyzed sample 13B on September 18, 2019. The primary particle observed was mica along with some talc particles and fibers/ribbons, some mica fibers, iron particles and a few silica particles. No asbestos or non-asbestos amphibole variants were detected in the samples. The results were calculated using the equations detailed in the calculations section.

308006-13	NAD
308006-13A	NAD
308006-13B	NAD

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

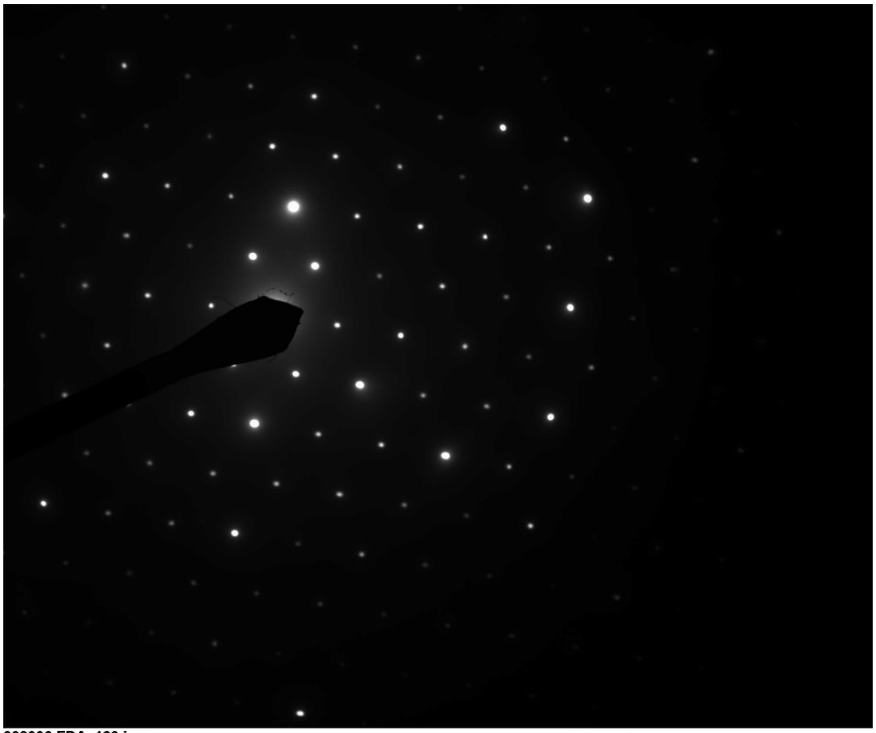
Sample 308006-13, Mica Particle



308006 FDA_122.jpg
Mica Particle
Cal: 0.002858 µm/pix
14:28 9/8/2019
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

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

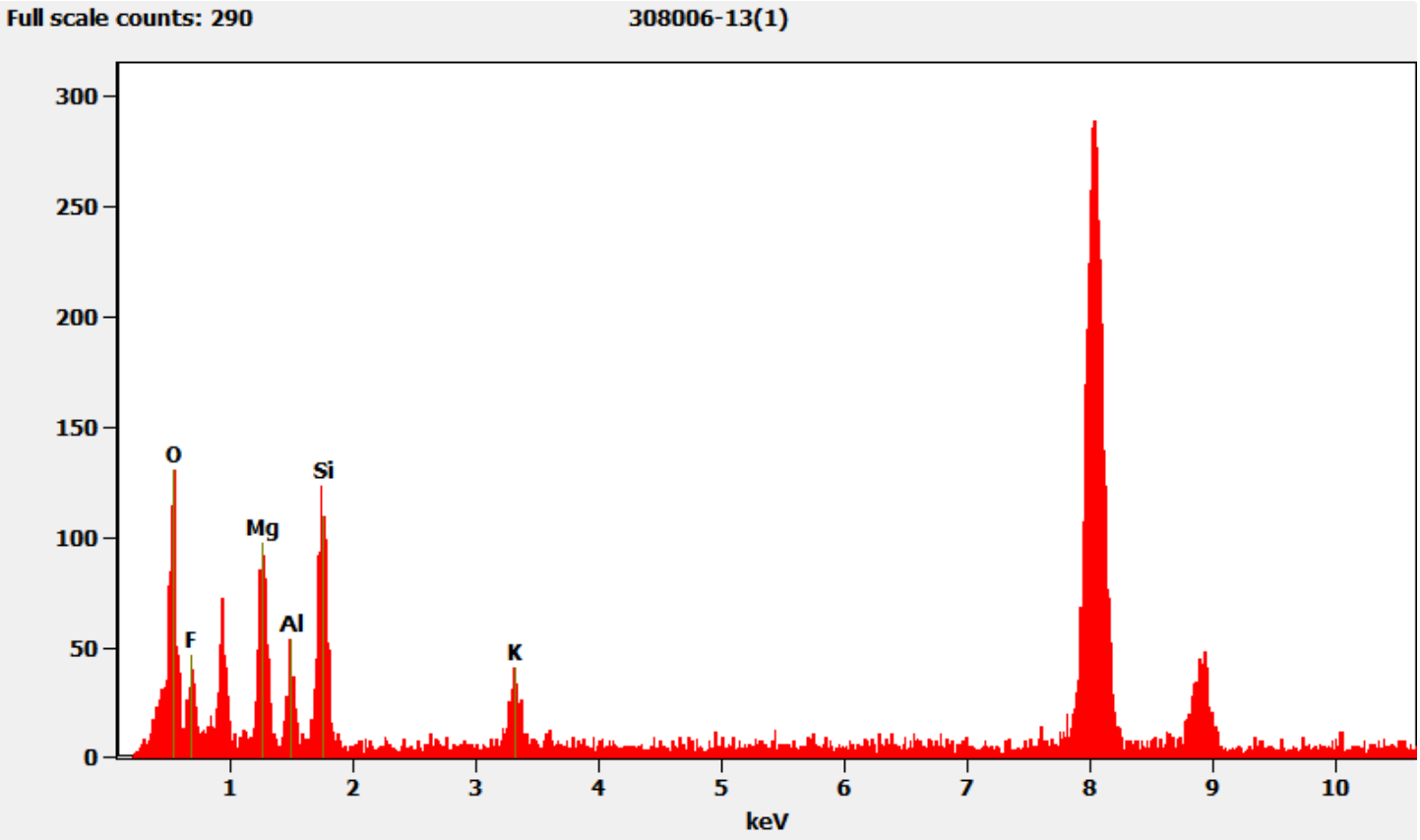
Diffraction pattern from the Mica particle pictured above.



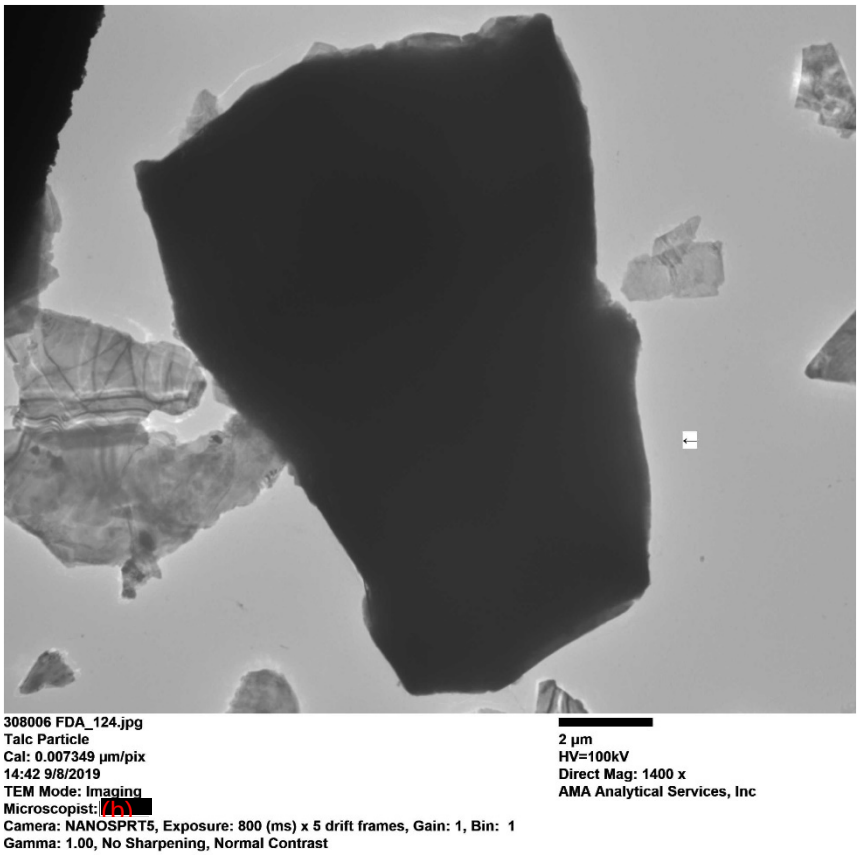
308006 FDA_123.jpg
Mica Particle
14:29 9/8/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from Mica particle pictured above.

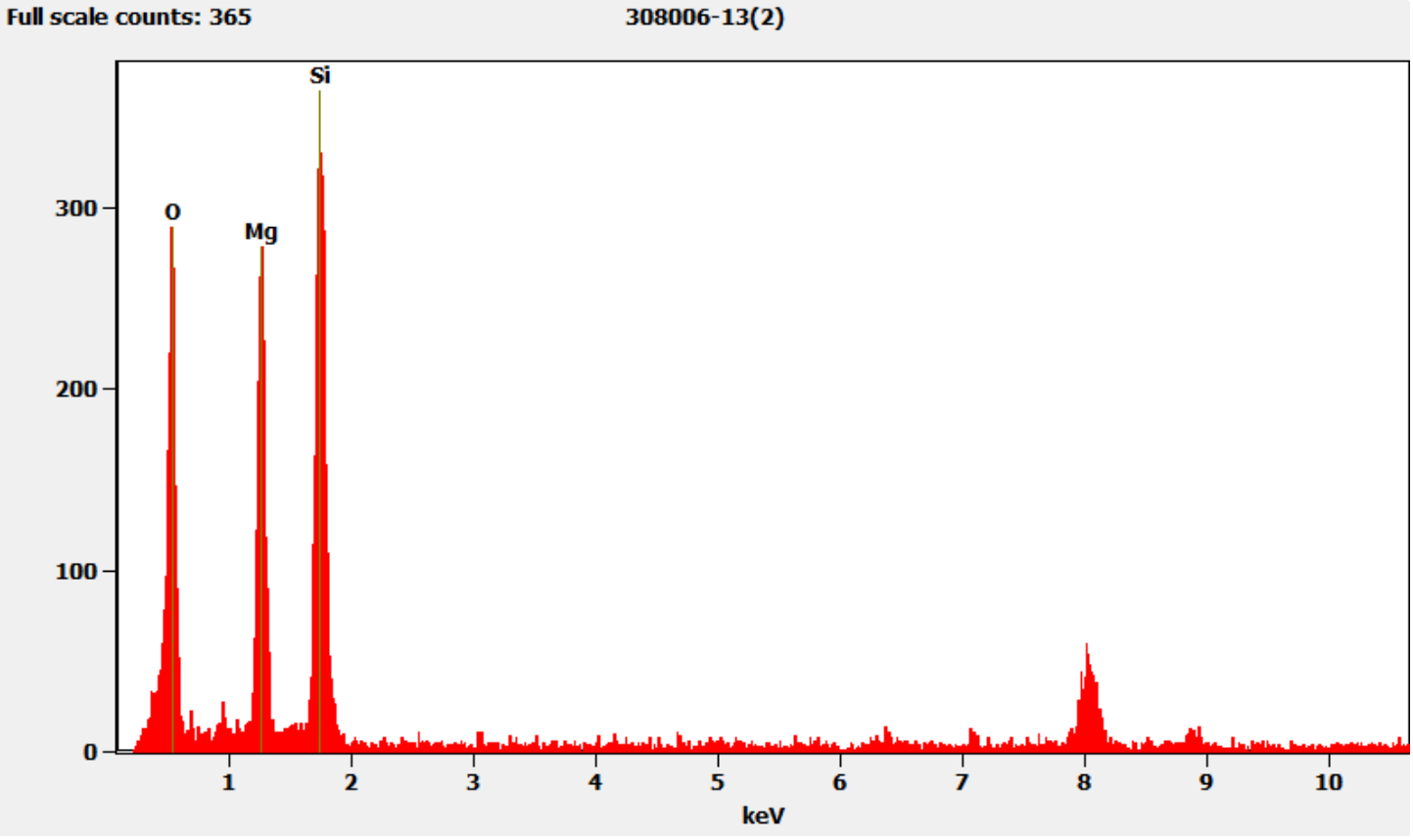


Sample 308006-13, Talc particle

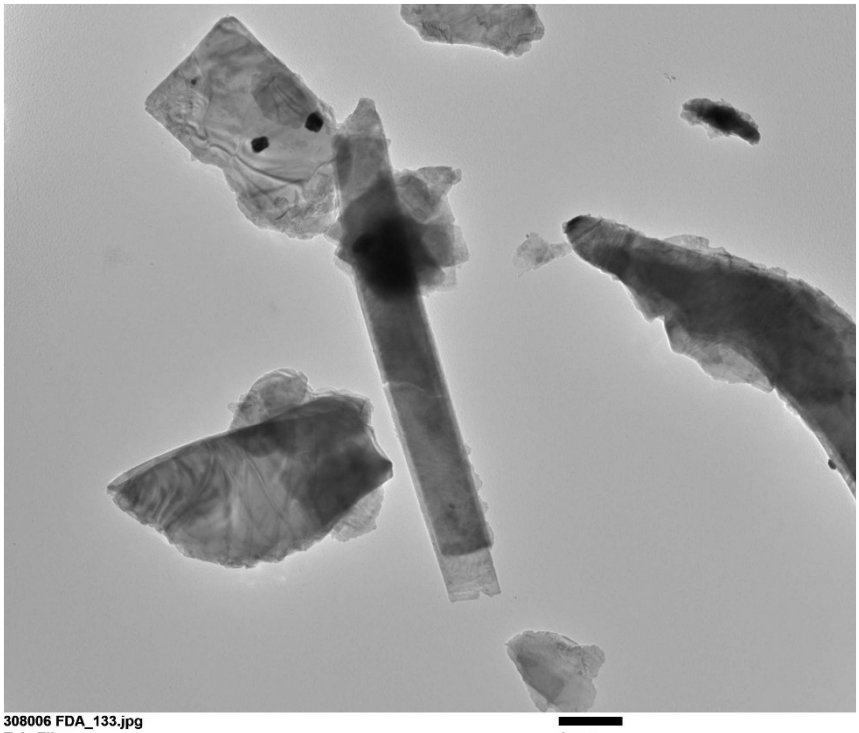


Hexagonal diffraction pattern from the Talc particle pictured above.

Chemistry from the Talc particle pictured above



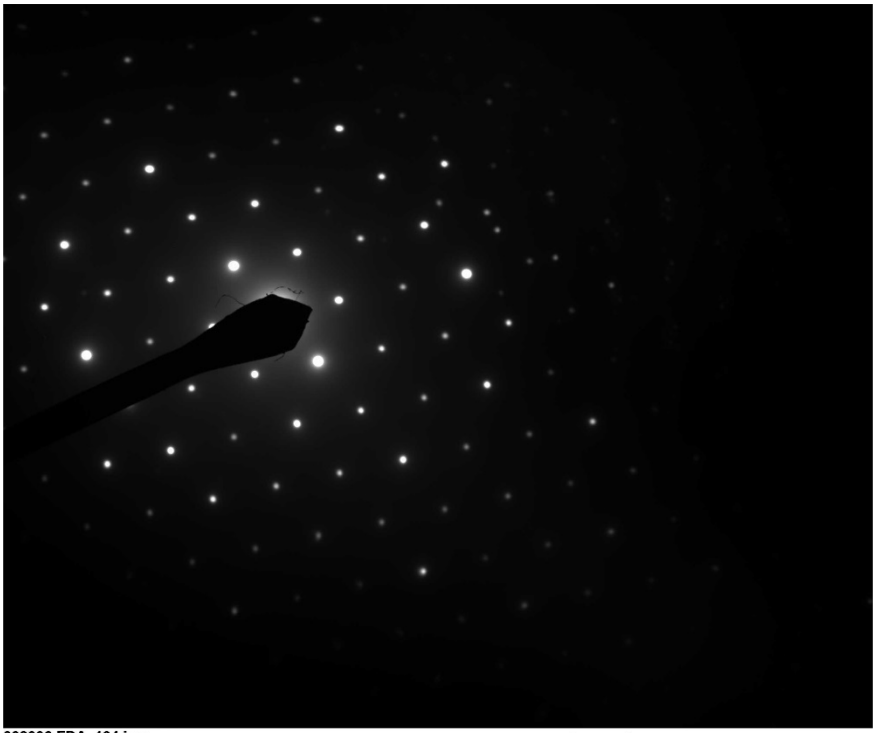
Sample 308006-13, Talc Fiber



308006 FDA_133.jpg
Talc Fiber
Cal: 0.005415 µm/pix
15:36 9/8/2019
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

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

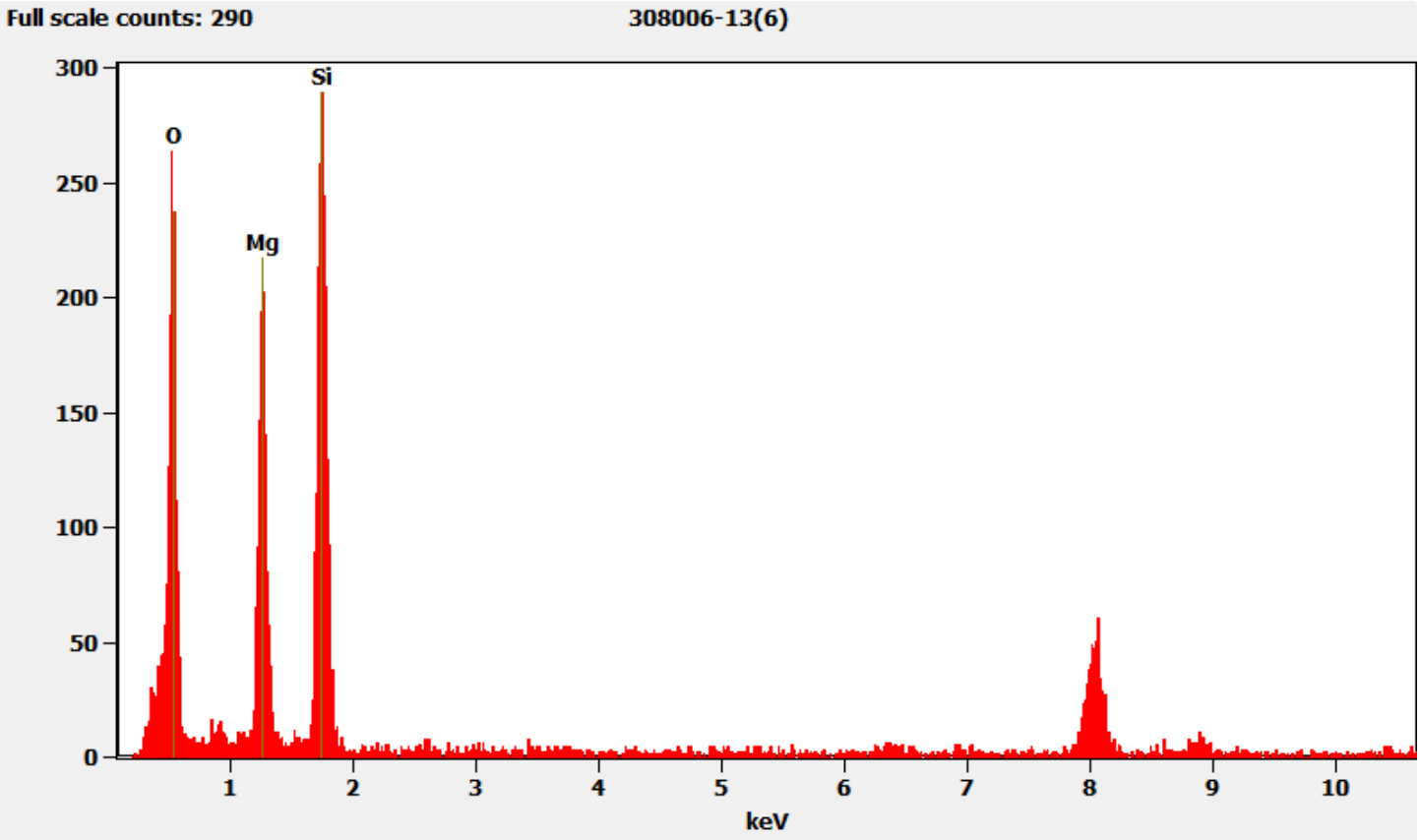
Hexagonal diffraction pattern from the Talc fiber pictured above.



308006 FDA_134.jpg
Talc Fiber
15:37 9/8/2019
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

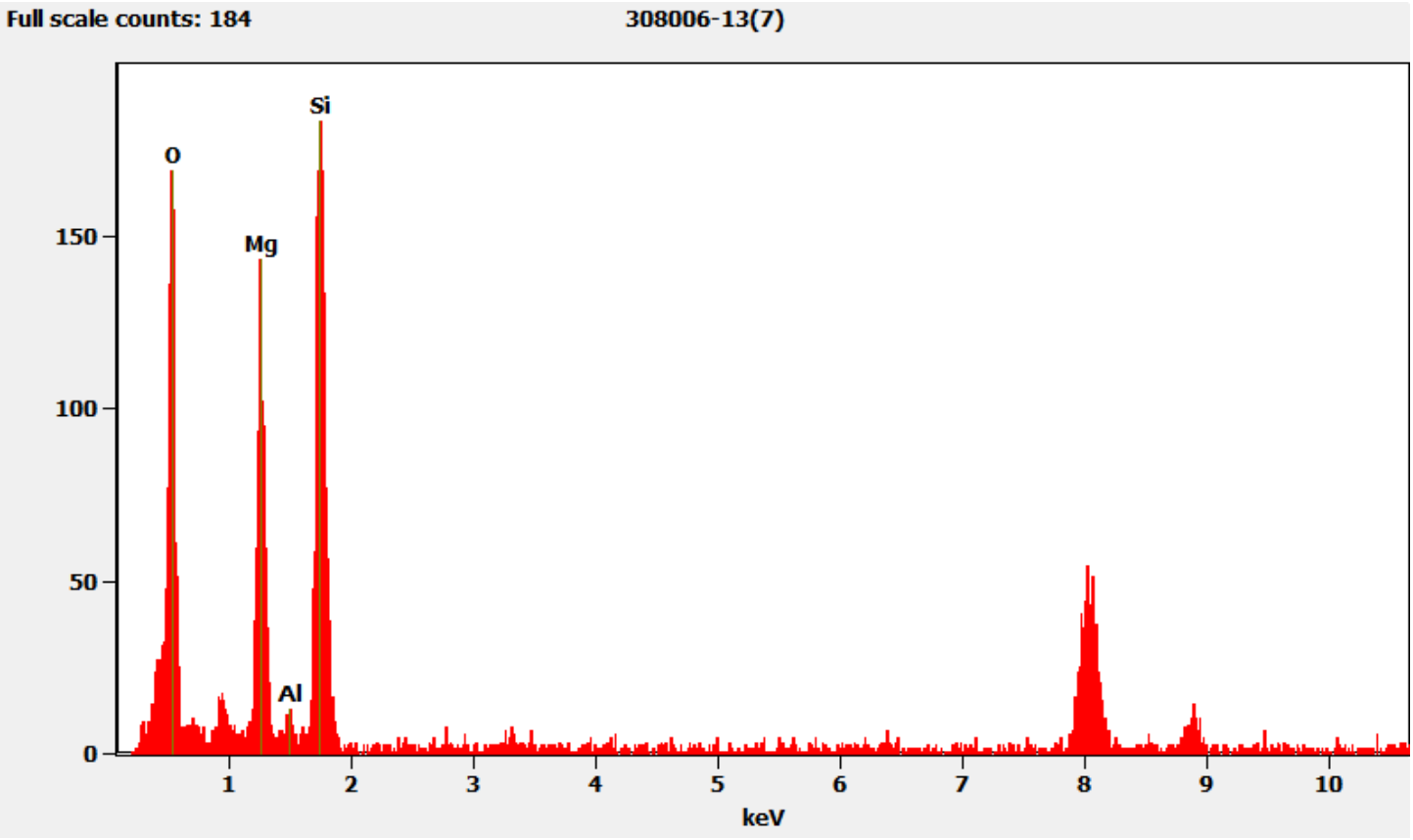
Chemistry from the Talc fiber pictured above.



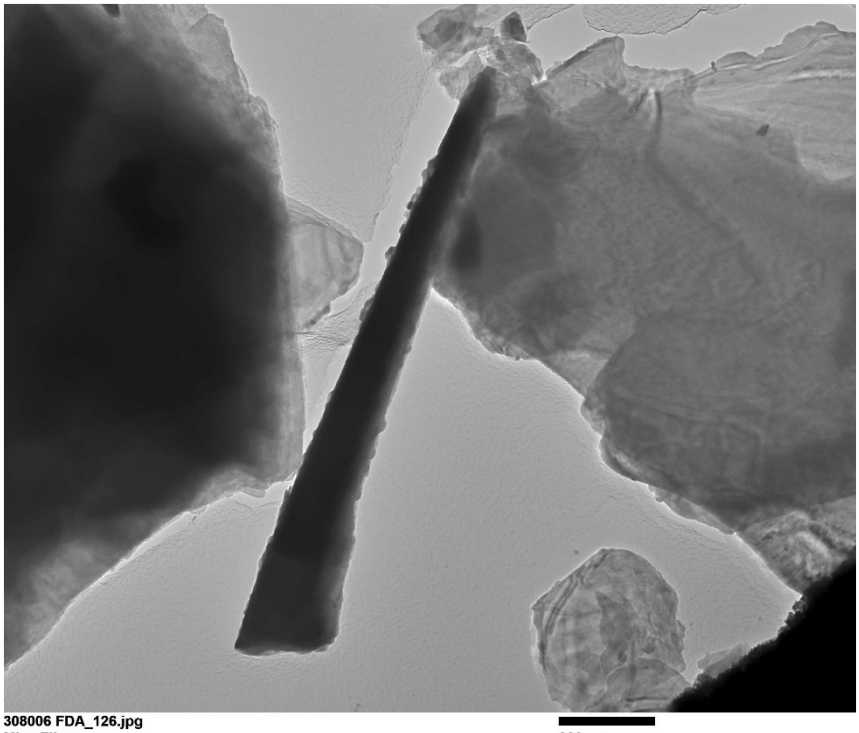
Sample 308006-13, Talc Ribbon

Diffraction pattern from the Talc ribbon pictured above

Chemistry from the Talc ribbon pictured above



308006-13, Mica Fiber



308006 FDA_126.jpg
Mica Fiber
Cal: 0.002858 µm/pix
14:49 9/8/2019
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

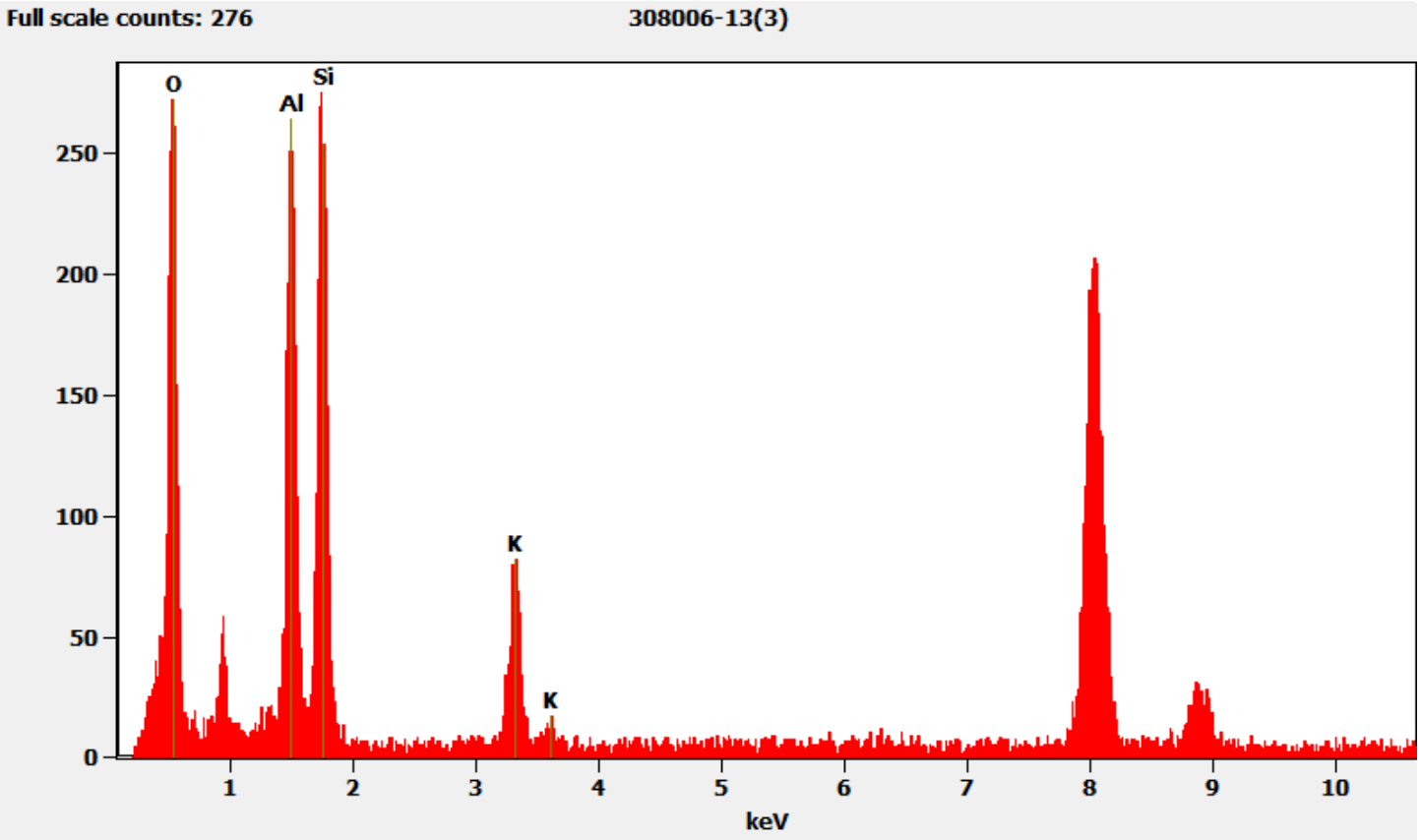
800 nm
HV=100kV
Direct Mag: 3600 x
AMA Analytical Services, Inc

Diffraction pattern from the Mica fiber pictured above

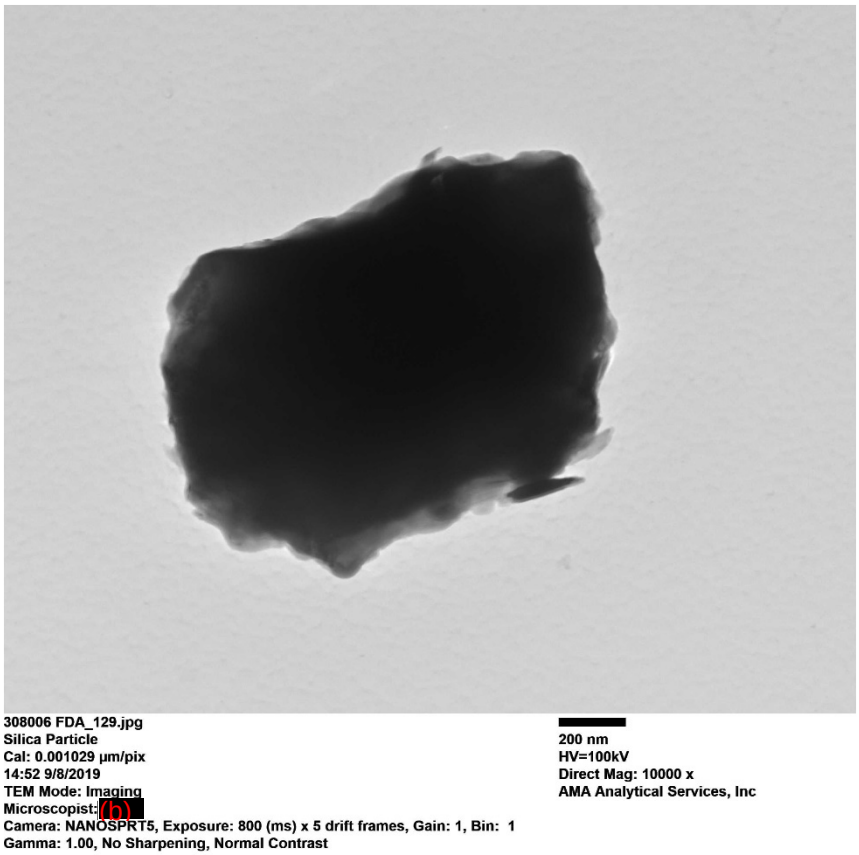
308006 FDA_128.jpg
Mica Fiber
14:50 9/8/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

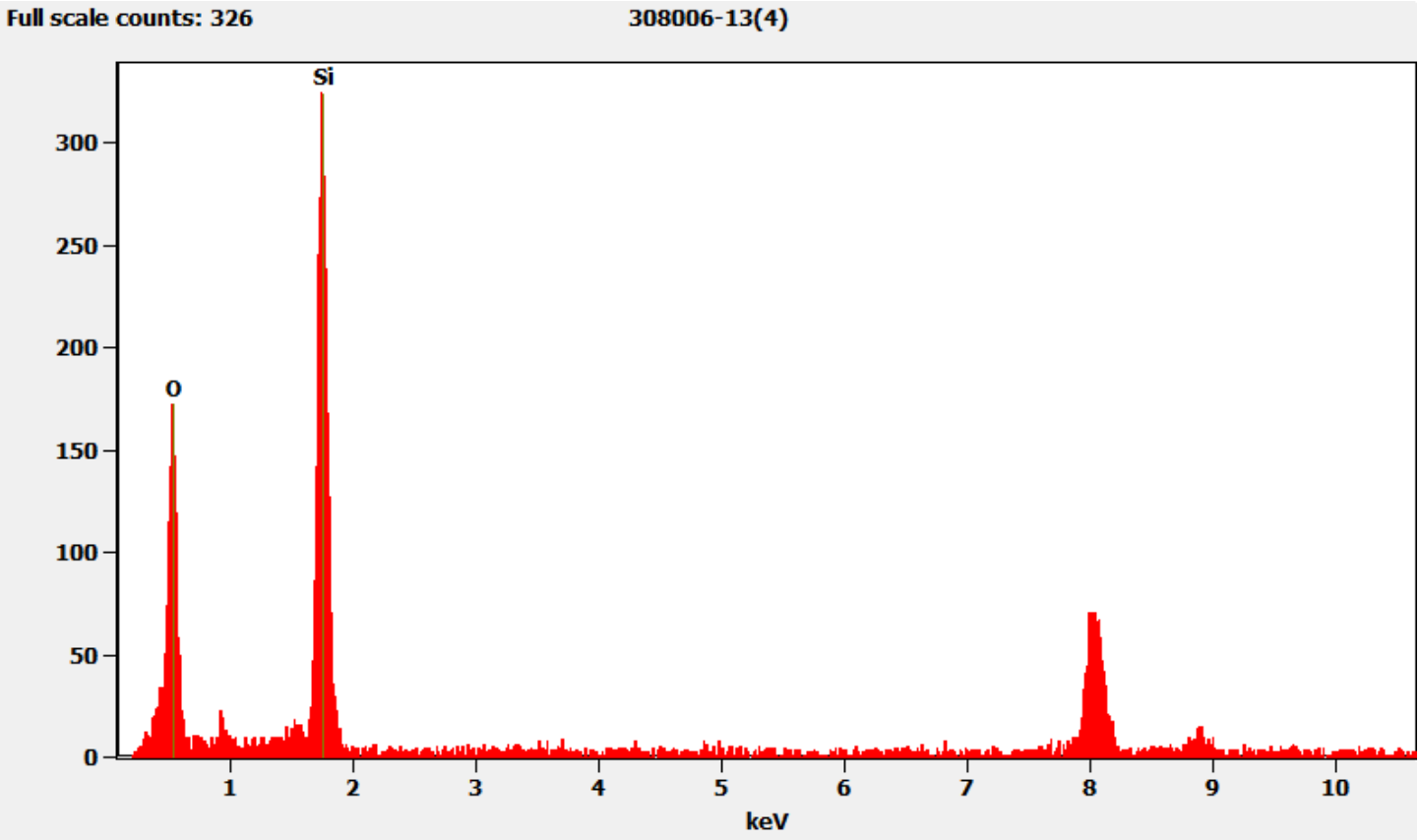
Chemistry from the Mica fiber pictured above



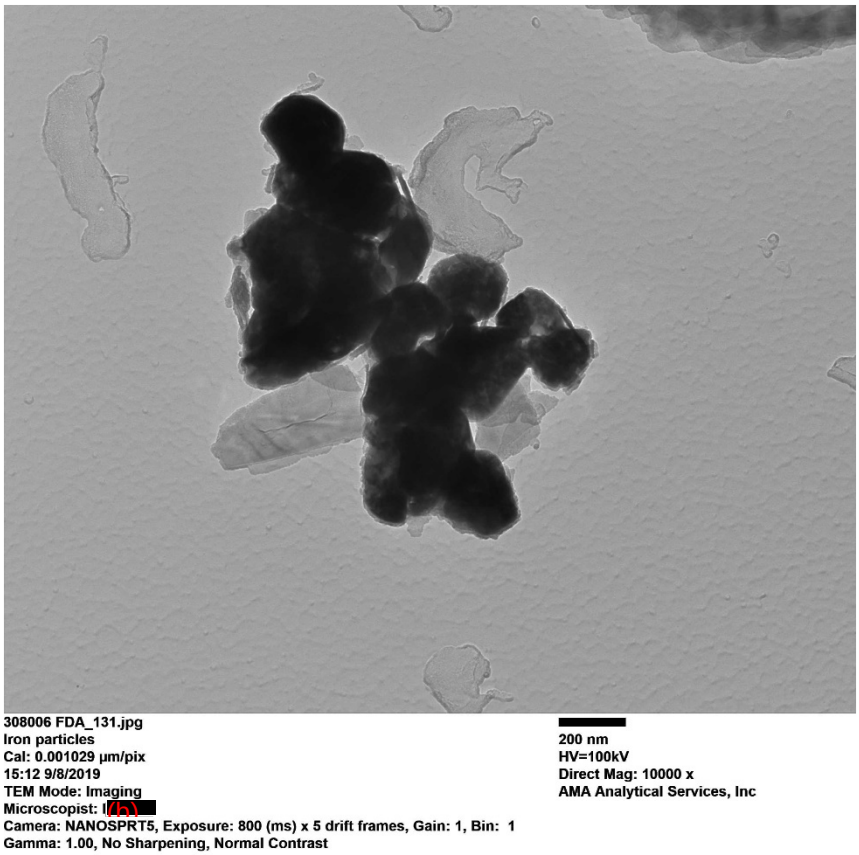
308006-13, Silica Particle



Chemistry from the Silica particle pictured above

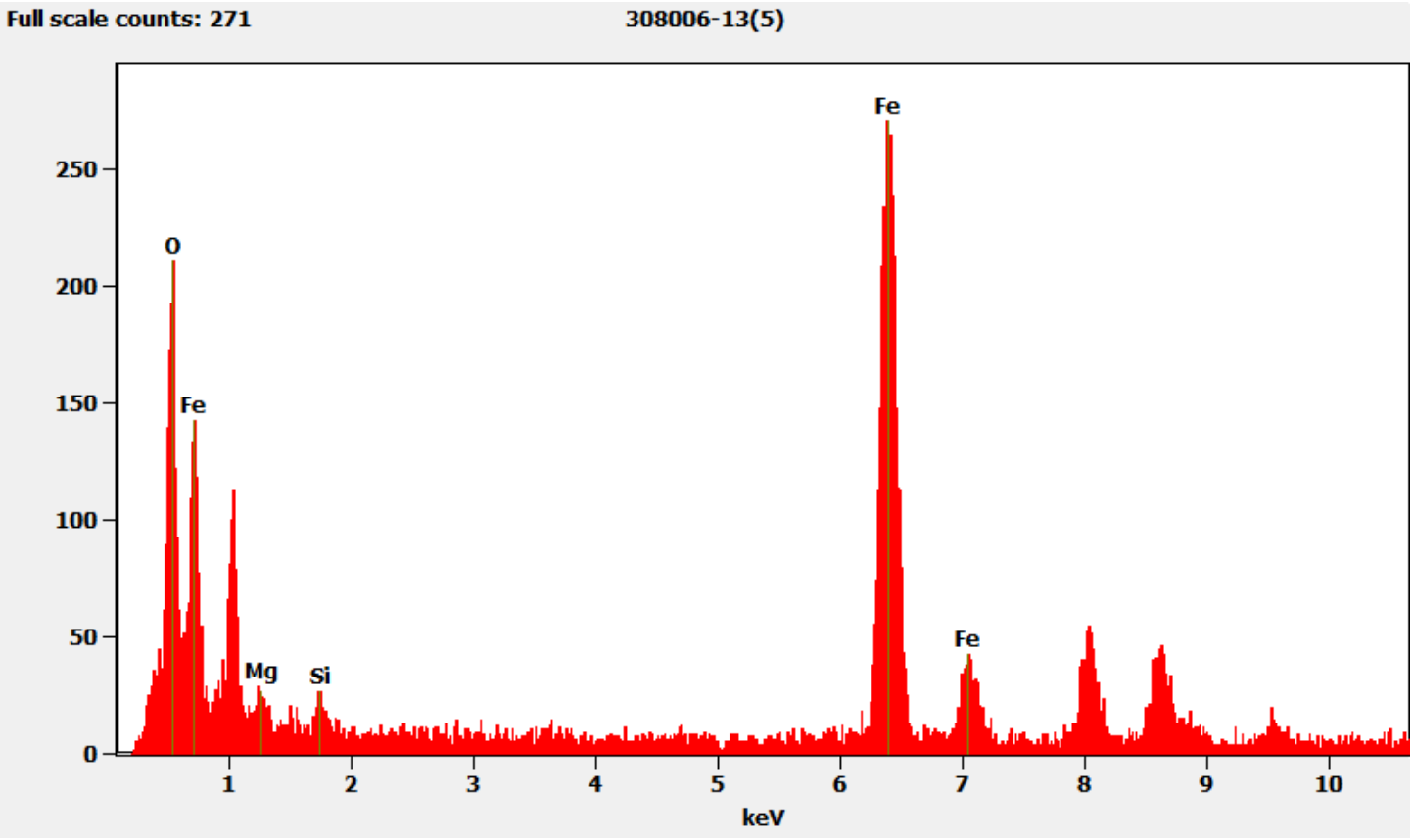


308006-13, Iron Particles



Diffraction pattern from the Iron particles pictured above

Chemistry from the Iron particles pictured above



308006-14, 14A, 14B, Client Sample D-66

PLM

All three aliquots of sample D-66 were analyzed by (b) (6) on September 13, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

308006-14	NAD
308006-14A	NAD
308006-14B	NAD

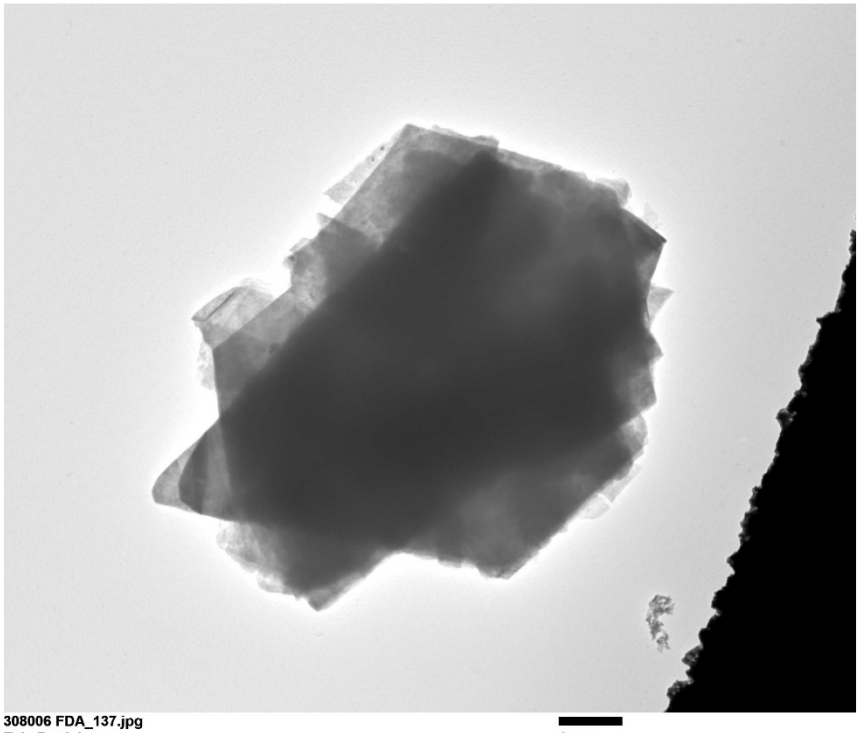
TEM

(b) (6) analyzed sample 14 on September 8 & 9, 2019 and sample 14A on September 18, 2019. (b) (6) analyzed sample 14B on September 18, 2019. The primary particle observed was talc along with a few talc fibers and ribbons and very few mica particles. No asbestos or non-asbestos amphibole variants were detected in the samples. The results were calculated using the equations detailed in the calculations section.

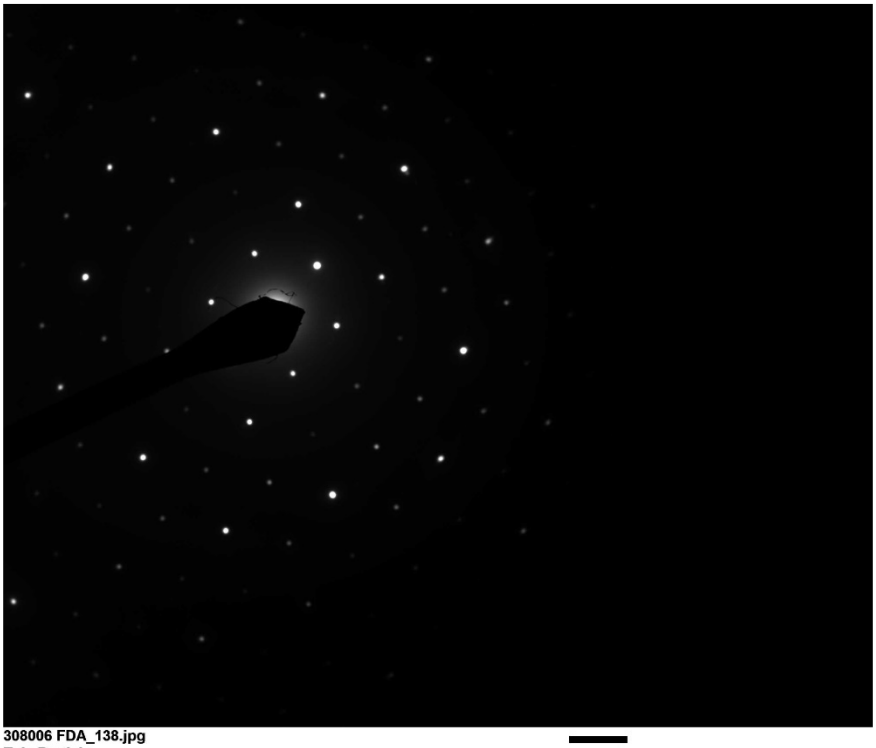
308006-14	NAD
308006-14A	NAD
308006-14B	NAD

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

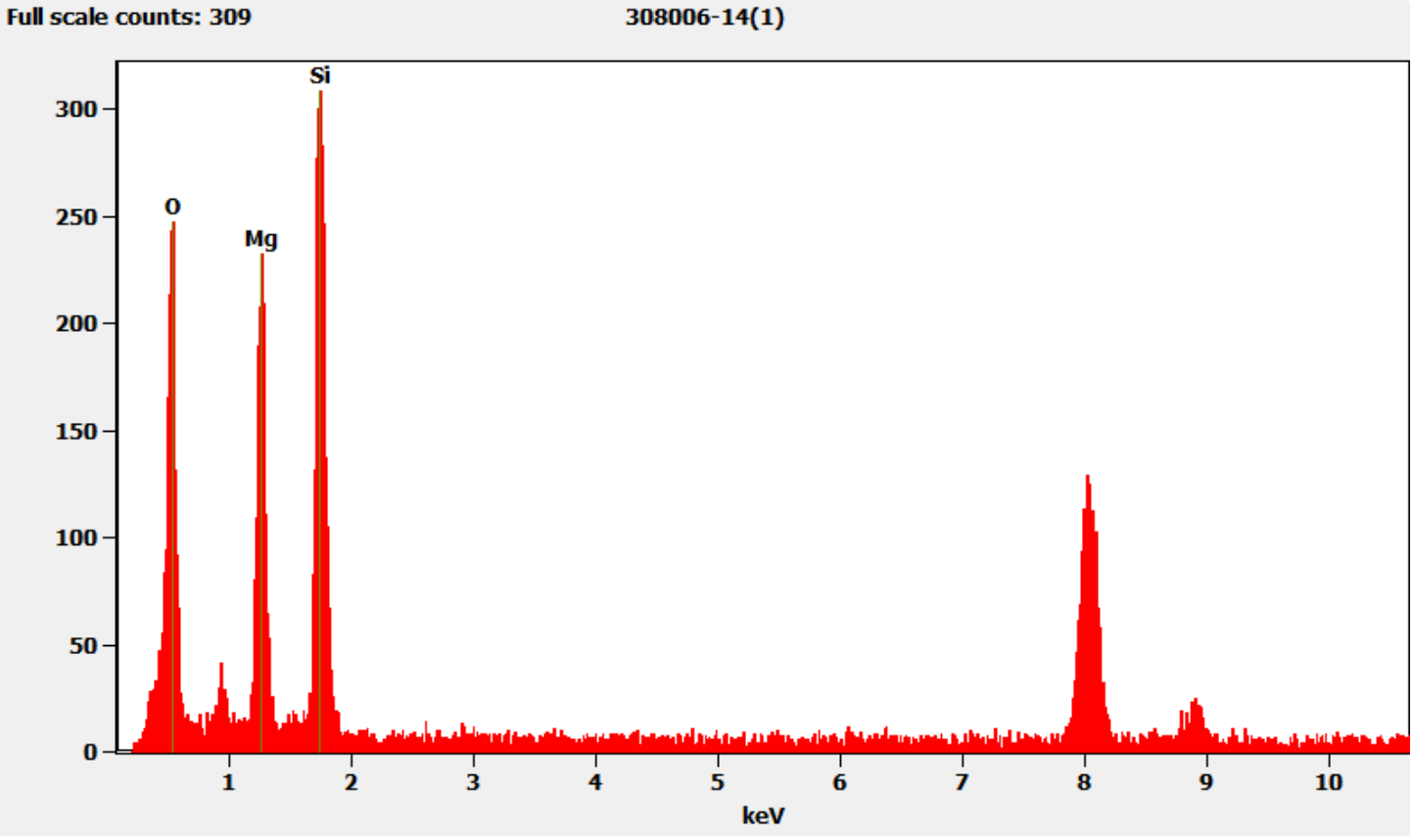
Sample 308006-14, Talc Particle



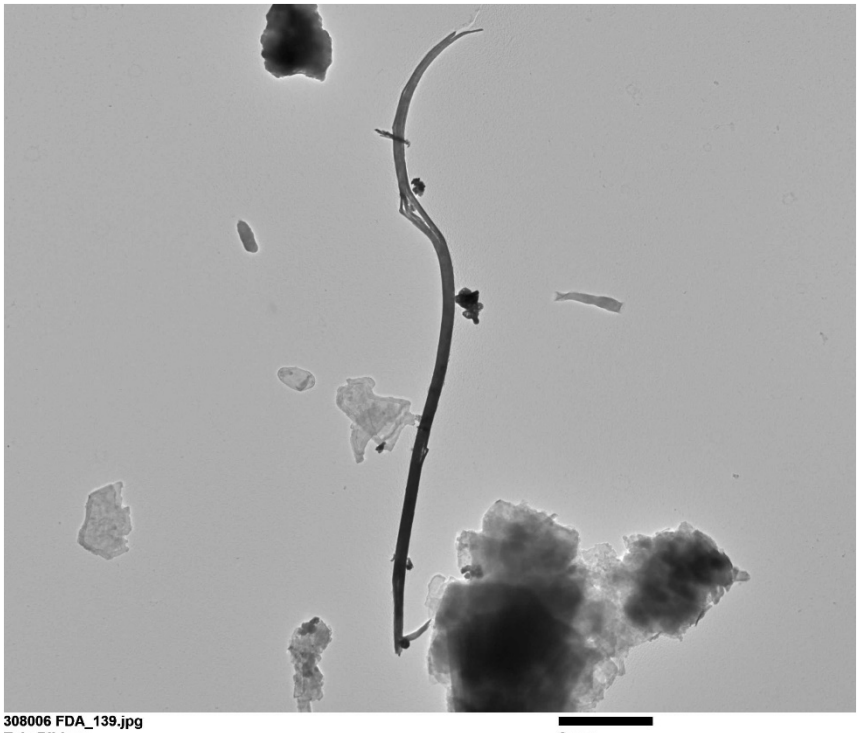
Hexagonal diffraction from the Talc particle pictured above.



Chemistry from the Talc particle pictured above.



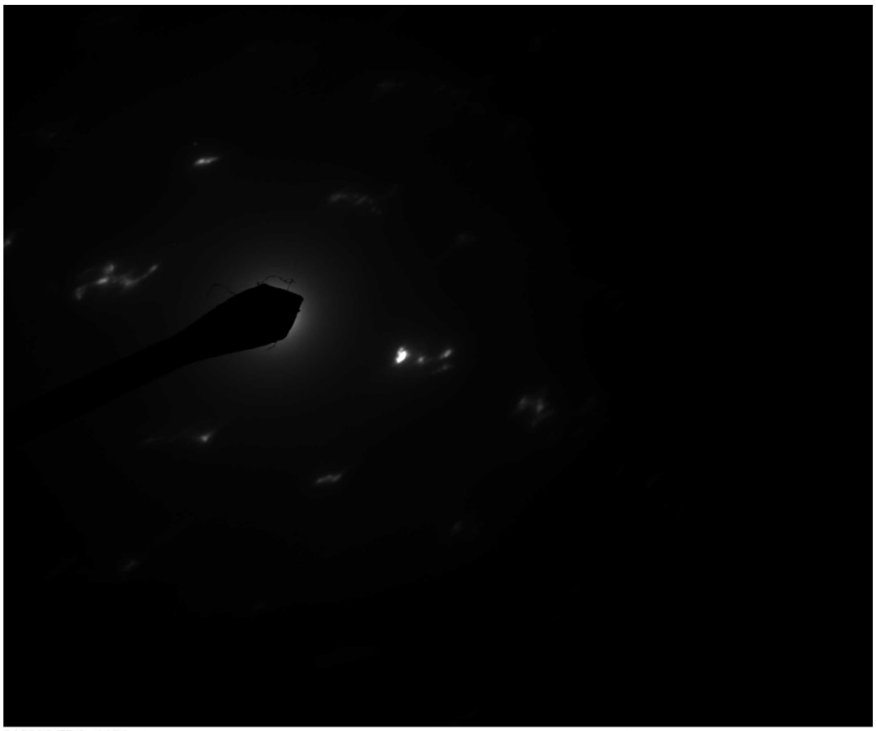
Sample 308006-14, Talc Ribbon



308006 FDA_139.jpg
Talc Ribbon
Cal: 0.007349 µm/pix
16:23 9/8/2019
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

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

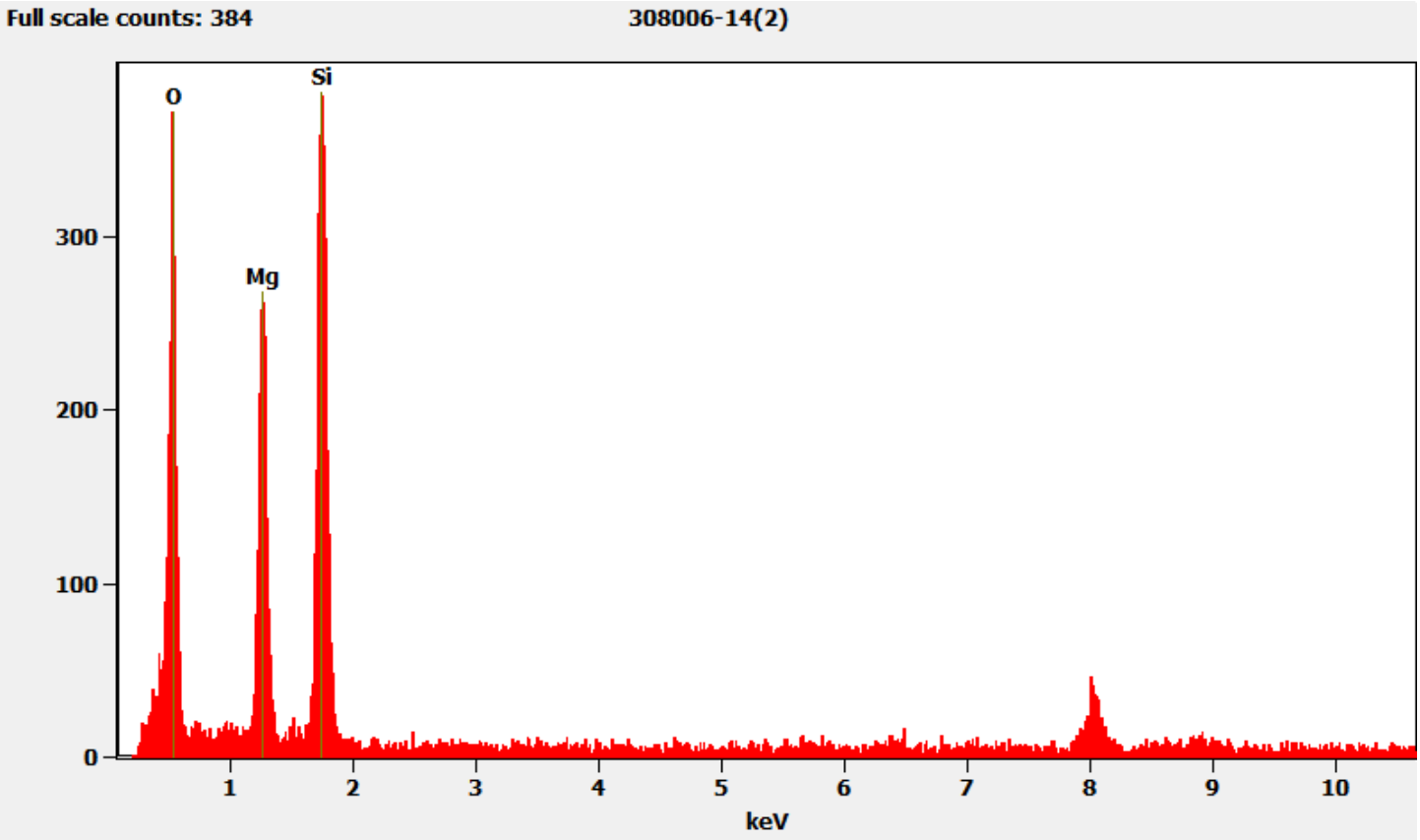
Diffraction pattern from the Talc ribbon pictured above.



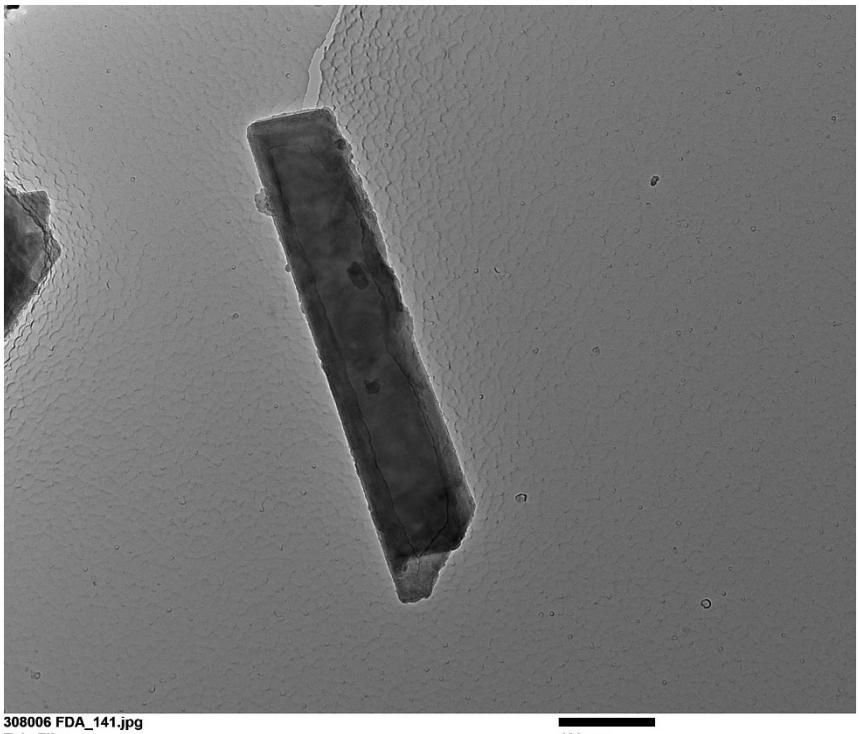
308006 FDA_140.jpg
Talc Ribbon
16:24 9/8/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from the Talc ribbon pictured above

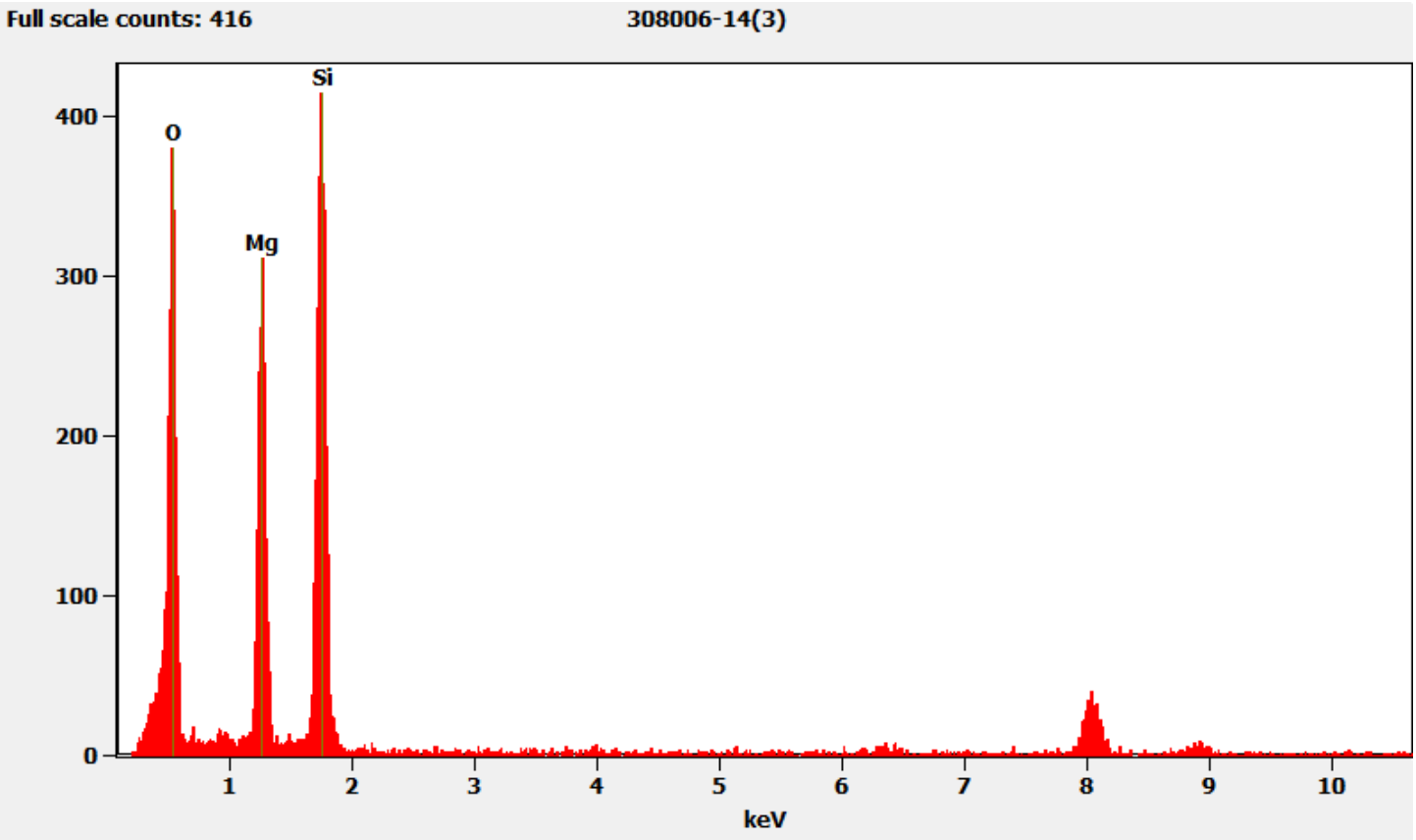


Sample 308006-14, Talc Fiber

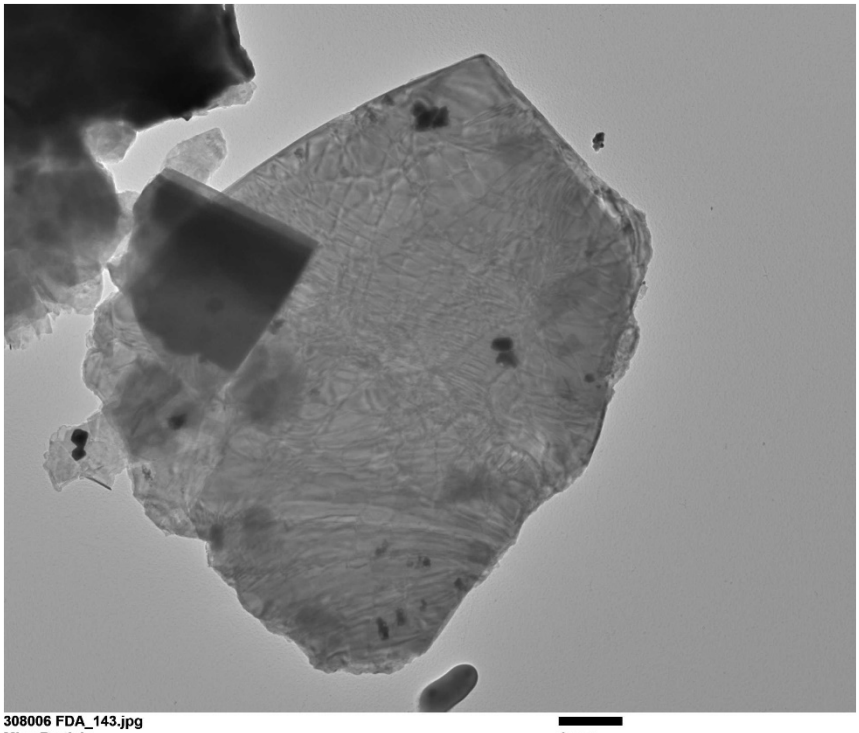


Hexagonal diffraction pattern from the Talc fiber pictured above.

Chemistry from the Talc fiber particle pictured above.



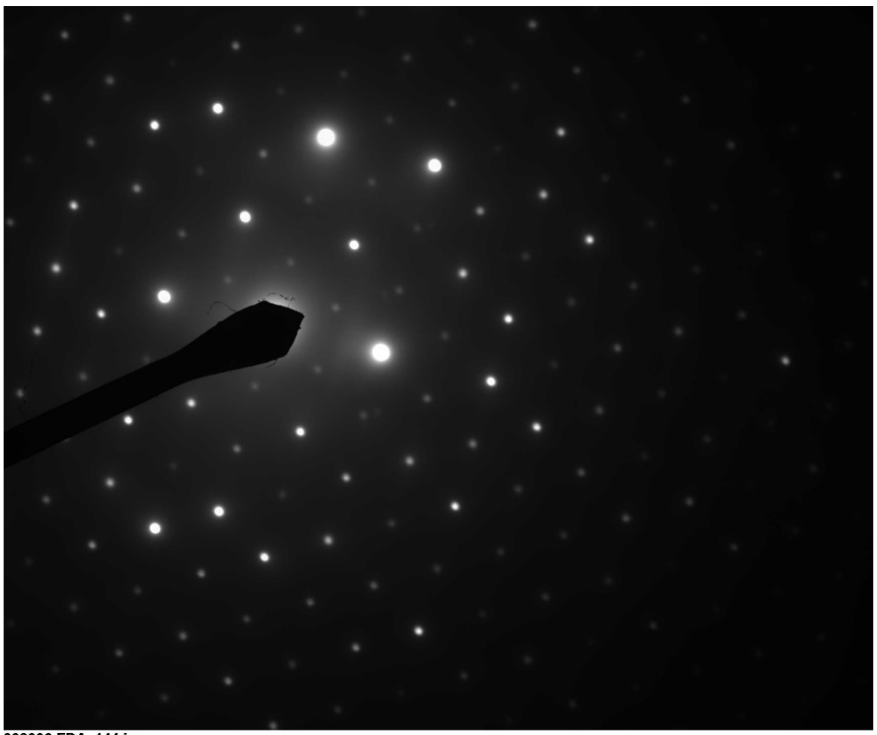
308006-14, Mica Particle



308006 FDA_143.jpg
Mica Particle
Cal: 0.005415 µm/pix
10:01 9/9/2019
TEM Mode: Imaging
Microscopist:
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

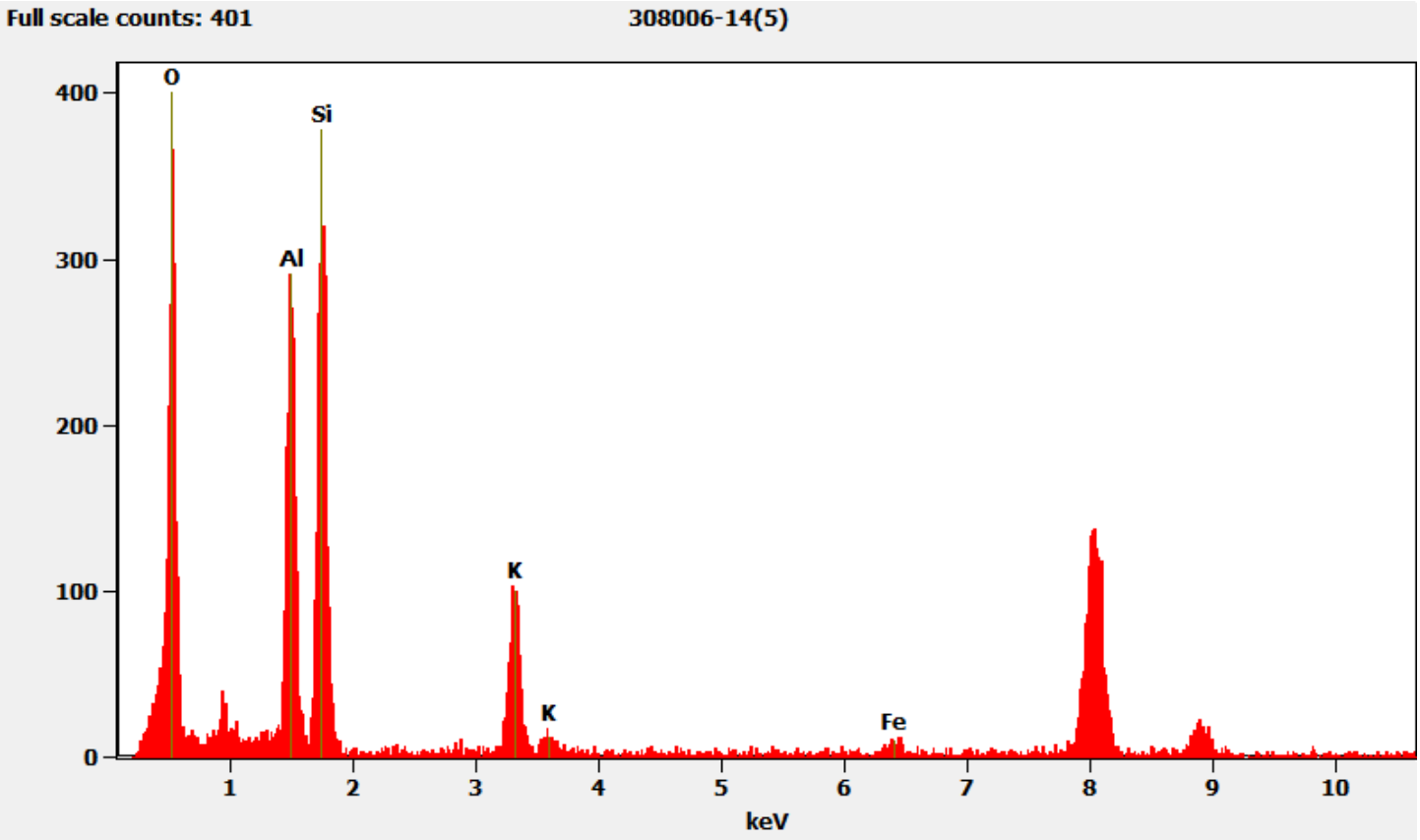
Diffraction pattern from the Mica particle pictured above



308006 FDA_144.jpg
Mica Particle
10:03 9/9/2019
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 Mica particle pictured above



<u>308006-15, 15A, 15B, Client Sample D-67</u>

PLM

All three aliquots of sample D-67 were analyzed by (b) (6) on September 13, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

308006-15	NAD
308006-15A	NAD
308006-15B	NAD

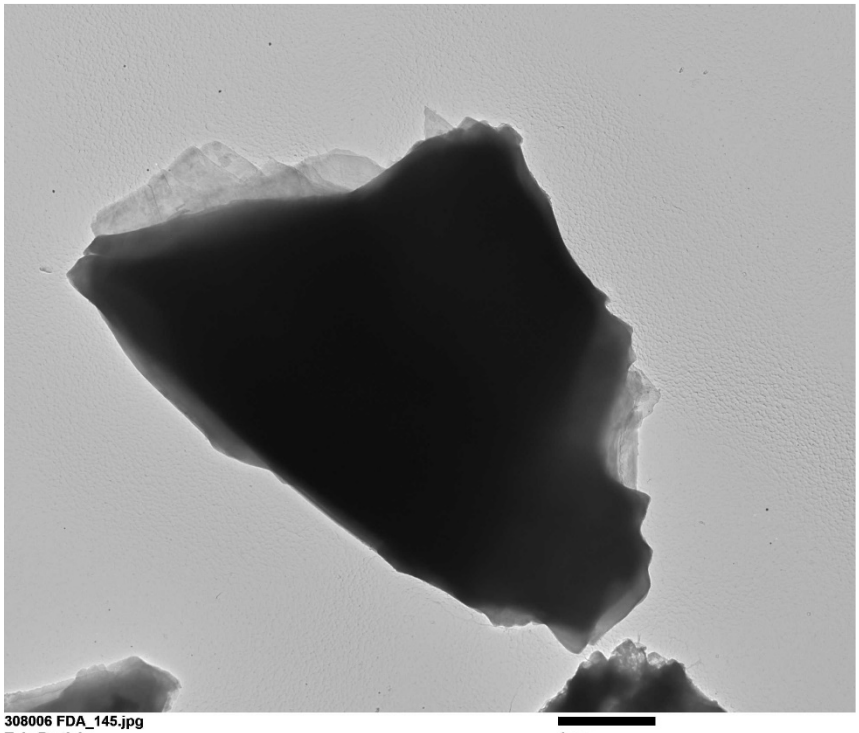
TEM

(b) (6) analyzed samples 15 and 15A on September 10, 2019 and sample 15B on September 11, 2019. The primary particle observed was talc along with a few talc fibers and very few talc ribbons and titanium particles. No asbestos or non-asbestos amphibole variants were detected in the samples. The results were calculated using the equations detailed in the calculations section.

308006-15	NAD
308006-15A	NAD
308006-15B	NAD

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

Sample 308006-15, Talc Particle



308006 FDA_145.jpg
Talc Particle
Cal: 0.003548 µm/pix
15:39 9/10/2019
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

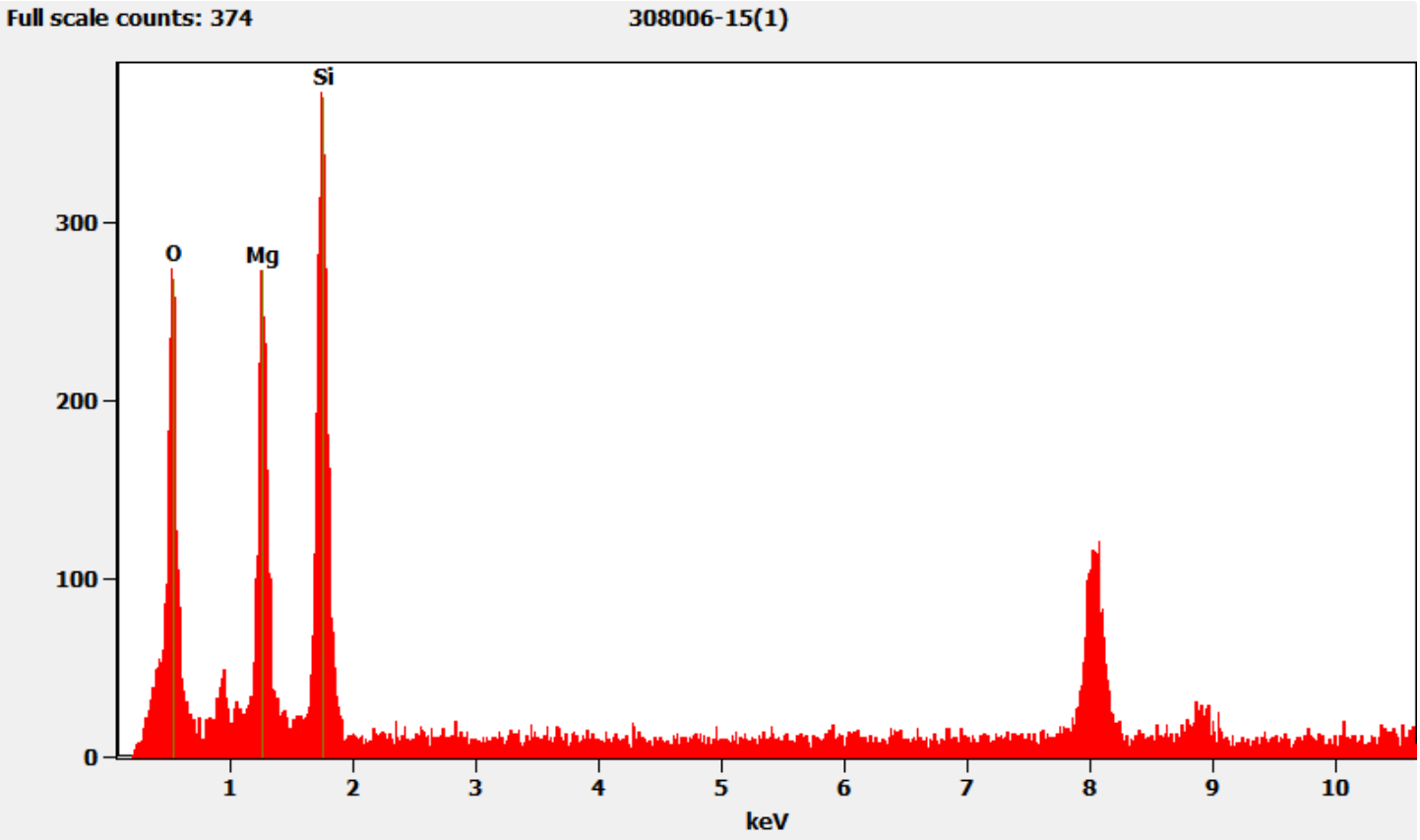
1 µm
HV=100kV
Direct Mag: 2900 x
AMA Analytical Services, Inc

Hexagonal diffraction from the Talc particle pictured above.

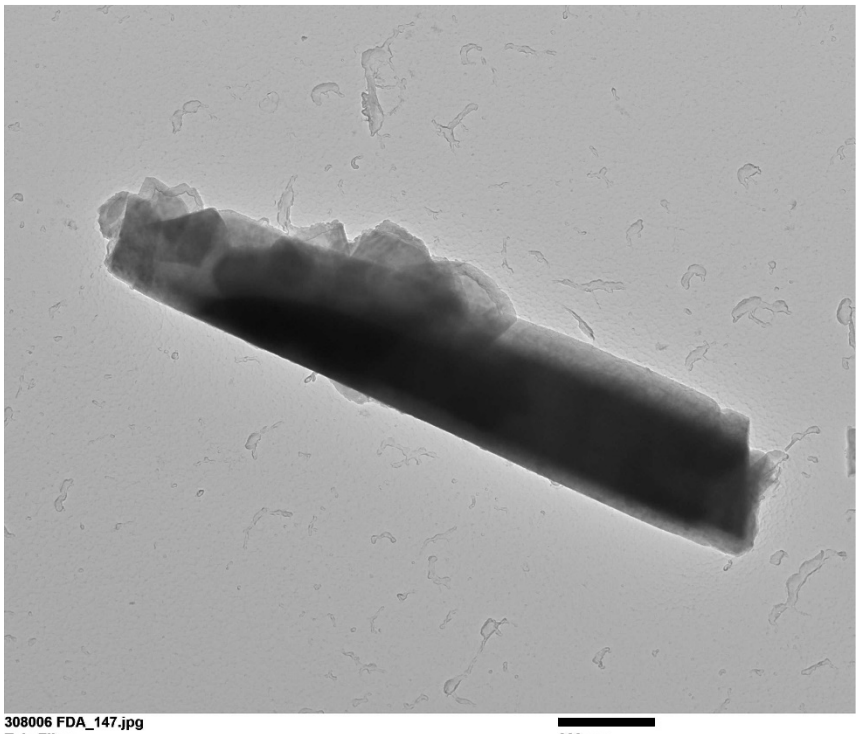
308006 FDA_146.jpg
Talc Particle
15:40 9/10/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from the Talc particle pictured above.

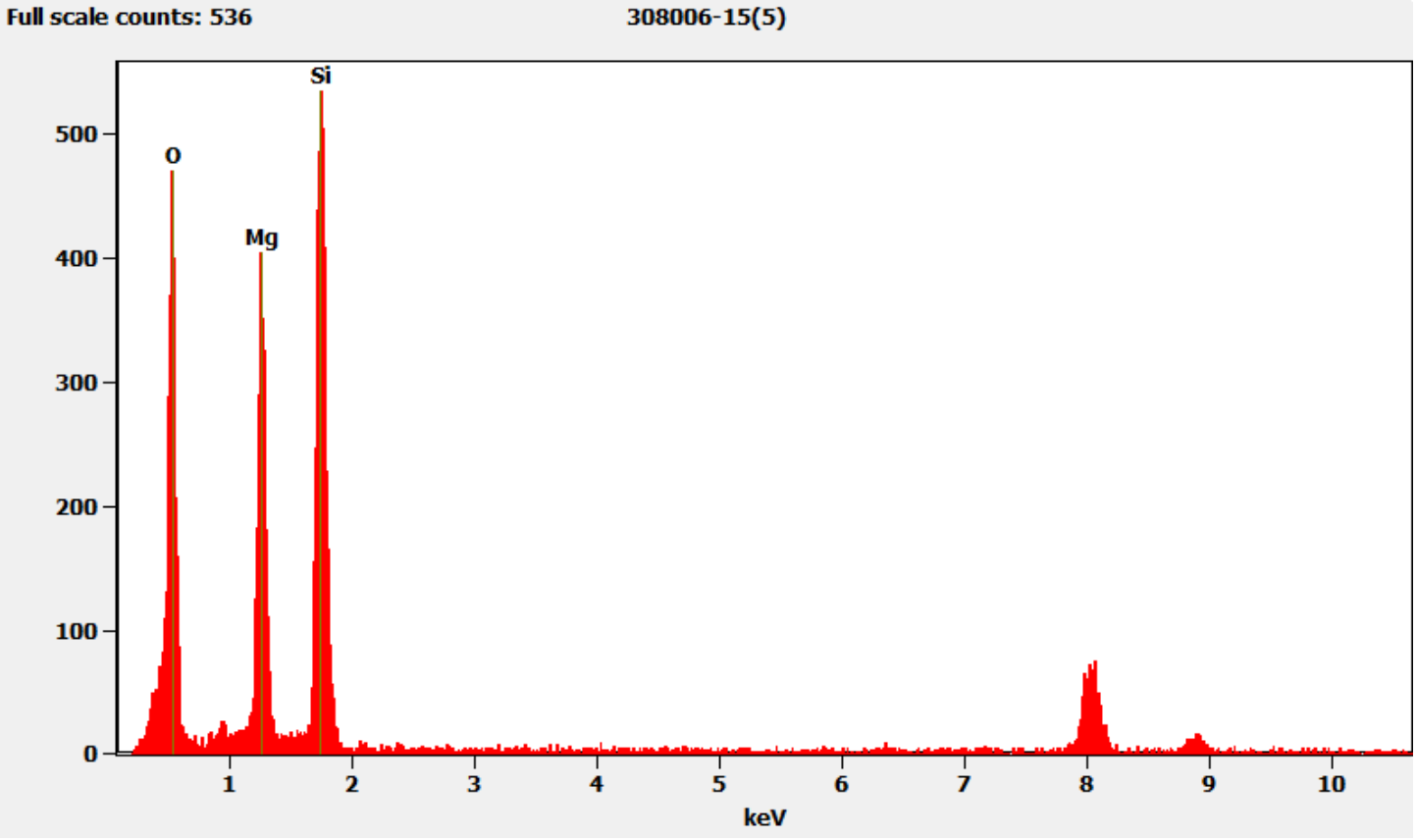


Sample 308006-15, Talc Fiber

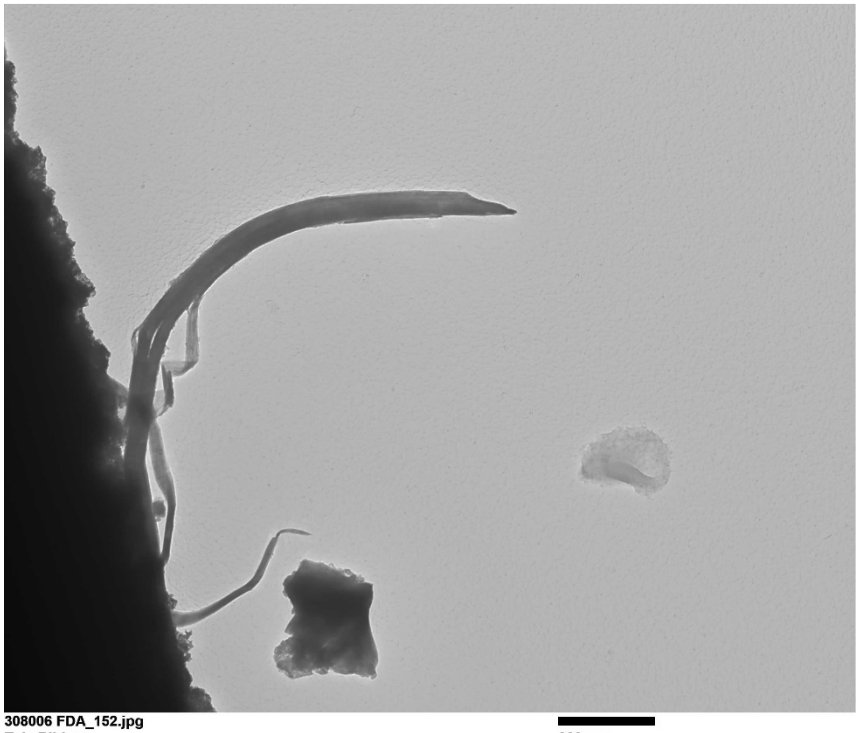


Hexagonal diffraction pattern from the Talc fiber pictured above.

Chemistry from the Talc fiber pictured above



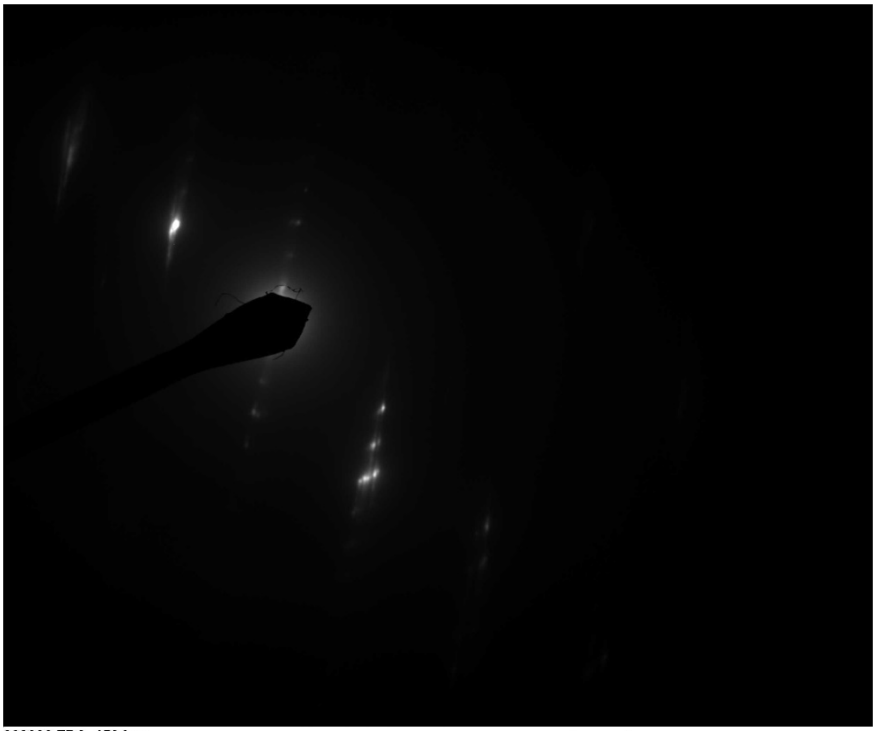
Sample 308006-15B, Talc Ribbon



308006 FDA_152.jpg
Talc Ribbon
Cal: 0.002858 µm/pix
11:40 9/11/2019
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

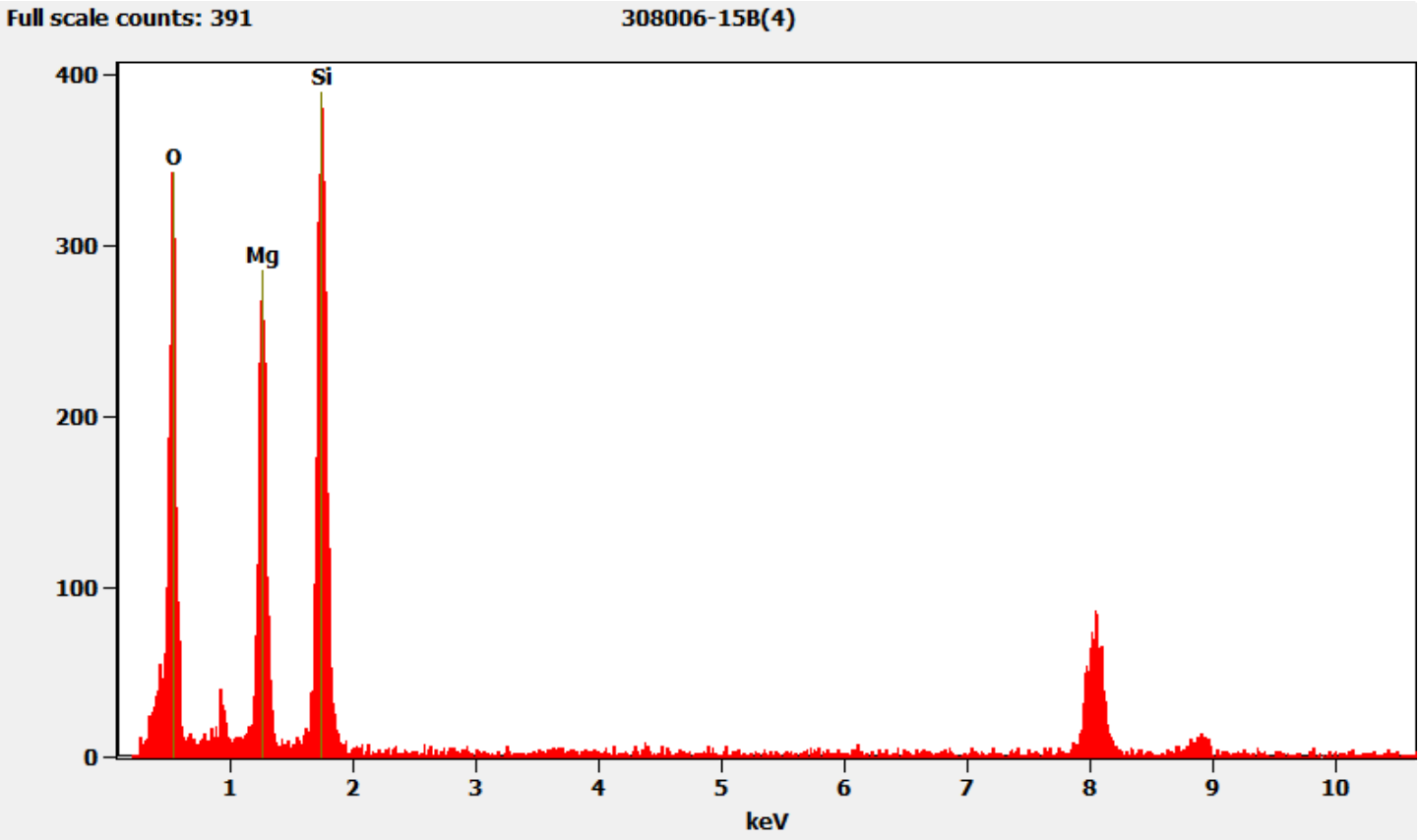
Diffraction pattern from the Talc ribbon pictured above.



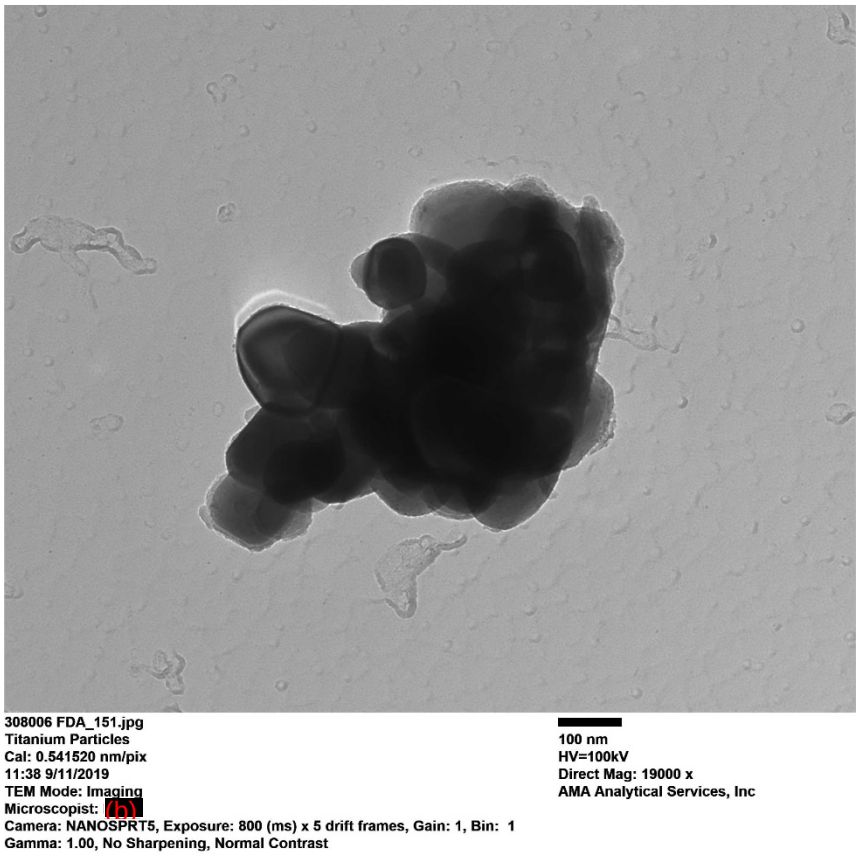
308006 FDA_153.jpg
Talc Ribbon
11:41 9/11/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

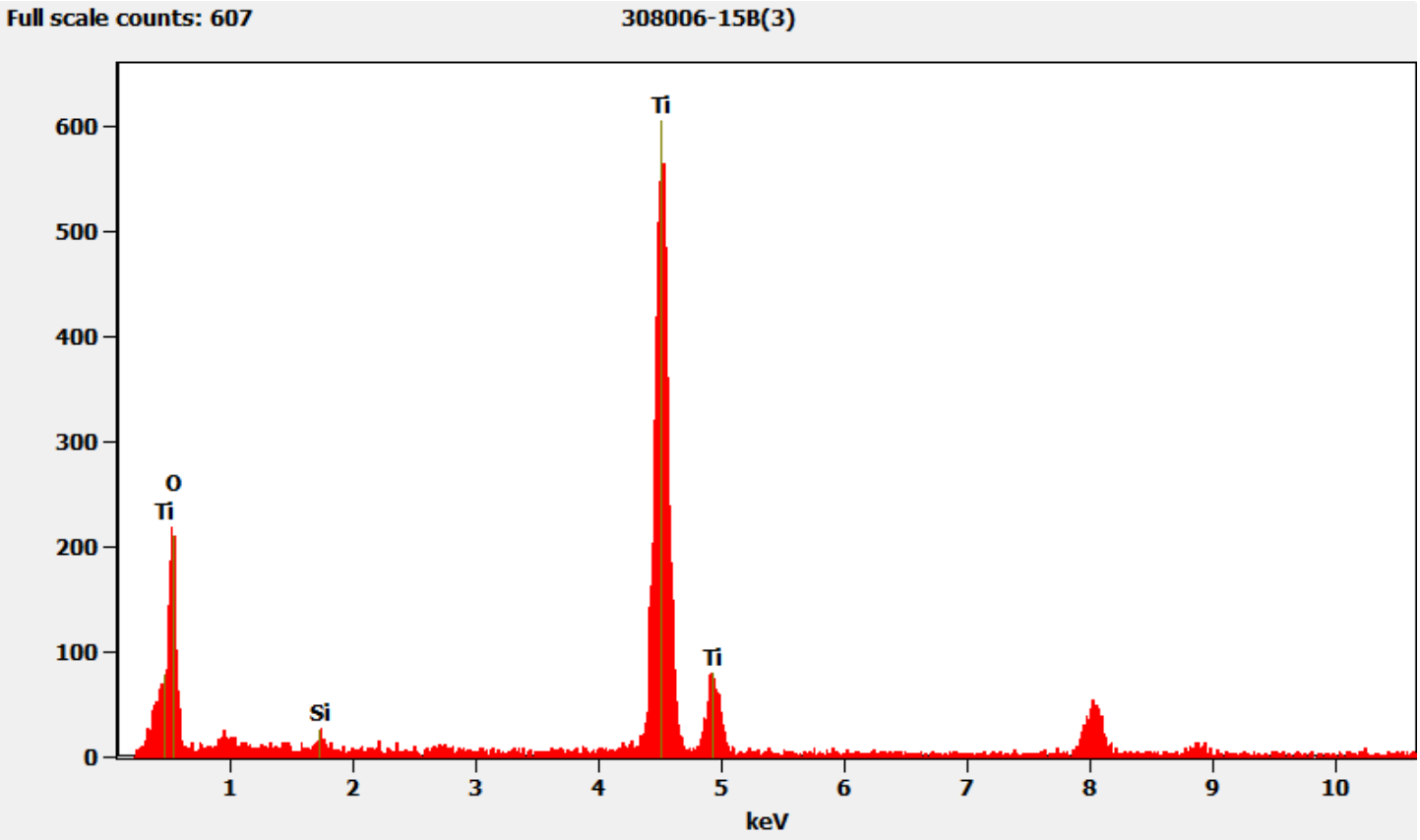
Chemistry from the talc ribbon pictured above.



Sample 308006-15B, Titanium Particles



Chemistry from the Titanium particles pictured above



QC Discussion:
During preparation, three blank control samples and one reference control sample were prepared. These samples were prepared alongside the customer samples. The blank samples were prepared using Sigma-Aldrich Talc Powder, <10 micron (Product No. 643604-500G; Batch No. 10830AJ) and was analyzed by (b) (6) on September 18, 2019. No asbestos was detected on the blank samples. The reference sample was made from the same Sigma-Aldrich talc powder spiked with 10% Chrysotile. The reference sample was analyzed by (b) (6) on September 18, 2019 and found to be within acceptable limits. Additionally, filter blanks were prepared with each batch of carbon coated filters. Filter blank number EB-54047 was associated with the carbon coating for samples 308006-2, 2A/D-54. Filter blank number EB-54049 was associated with the carbon coating for samples 308006-4A, 4B/D-56. Filter blank number EB-54157 was associated with the carbon coating for samples 308006-9A, 9B/D-61. No asbestos was detected on the filter blank samples.

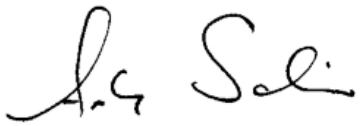
Our laboratory information management system (LIMS) randomly selected samples 308006-2/D-54 and 308006-15/D-67 for additional replicate QC analysis. Separate preparations were made for PLM and TEM analysis. The replicate QC analysis was performed by (b) (6) on September 13, 2019, 2019 for PLM analysis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

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

1) Sample Log-In Sheet
2) Daily PLM Scope Calibration Log
3) Refractive Index Oil Calibration Log
4) Daily TEM Scope Calibration Log
5) QC Results Summary

6) Replicate & Duplicate QC Chart for (b) (6) for samples analyzed between 1/1/2019 and 9/18/2019
7) Replicate & Duplicate QC Chart for (b) (6) for samples analyzed between 1/1/2019 and 9/18/2019
8) Replicate & Duplicate QC Chart for (b) (6) for samples analyzed between 1/1/2019 and 9/18/2019
9) Raw Data Sheets
    a. Gravimetric Data
    b. Filtration Worksheets
    c. PLM Analysis
    d. TEM Analysis
    e. QC Samples

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



10/16/2019

Andreas Saldivar — Date
Laboratory Director

AMA Analytical Services, Inc.
Focused On Results.

CERTIFICATE OF ANALYSIS

Chain of Custody: 308006
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: 4th Group - 15 Samples
Job Number: CLIN 1 - Task 3
PO Number: HHSF223201810337P

Date Submitted: 7/24/2019
Date Analyzed: 8/20/2019-9/18/2019
Report Date: 10/3/2019
Date Sampled: Not Provided
Person Submitting: Goran Periz
Revised: 12/19/2019 (Revision #4)

SUMMARY OF ANALYSIS

AMA Sample ID	Client Sample ID	TEM LOD Using ASTM D5756 Mass Calculation	TEM LOQ Using ASTM D5756 Mass Calculation	% Tremolite by TEM Using ASTM D5756 Mass Calculation	% Chrysotile by TEM Using ASTM D5756 Mass Calculation	% Total Tremolite & Chrysotile by TEM Using ASTM D5756 Mass Calculation	% Asbestos by PLM	% Organics	% Acid Soluable	% Other	Comments
308006-6	D-58	0.00000169%	0.00000675%	ND	ND	ND	ND	0.3%	6.7%	93.1%	Gravimetric Loss from PLM Prep: Organics = 0.3%; Acid Soluable = 7.1%; Other = 92.6%
308006-6A	D-58	0.00000133%	0.00001485%	ND	< 0.00001%	< 0.00001%	ND	0.2%	19.5%	80.2%	Gravimetric Loss from PLM Prep: Organics = 0.2%; Acid Soluable = 8.5%; Other = 91.3%
308006-6B	D-58	0.00000135%	0.00000540%	ND	0.00002%	0.00002%	ND	0.2%	11.2%	88.6%	Gravimetric Loss from PLM Prep: Organics = 0.3%; Acid Soluable = 5.5%; Other = 94.2%

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

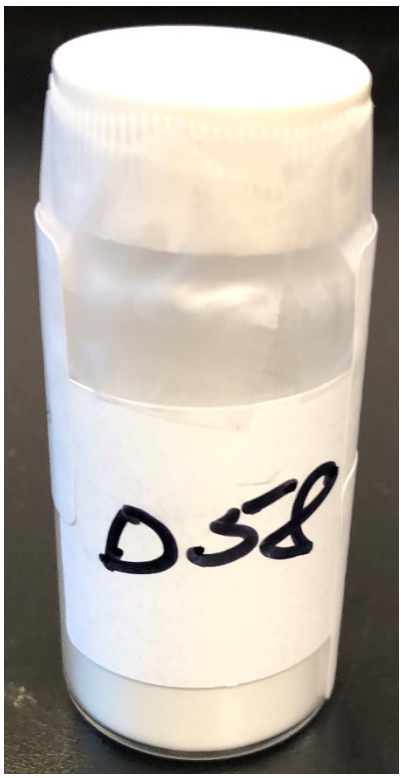
Analytical Method(s): PLM by Modified NY ELAP 198.6
TEM by Modified NY ELAP 198.4/ASTM D5756

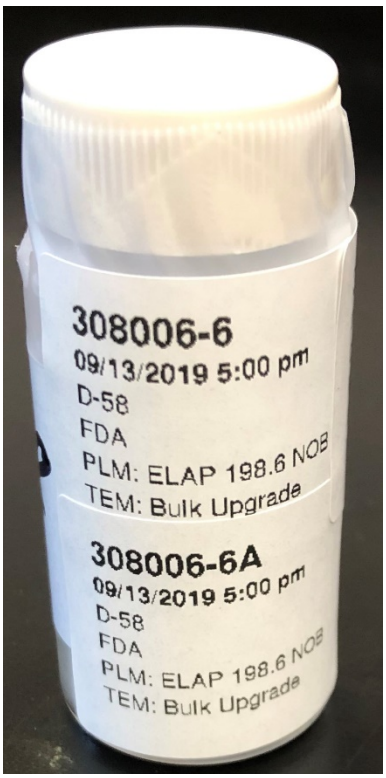
Analyst(s): **PLM** (b) (6)
TEM (b) (6)

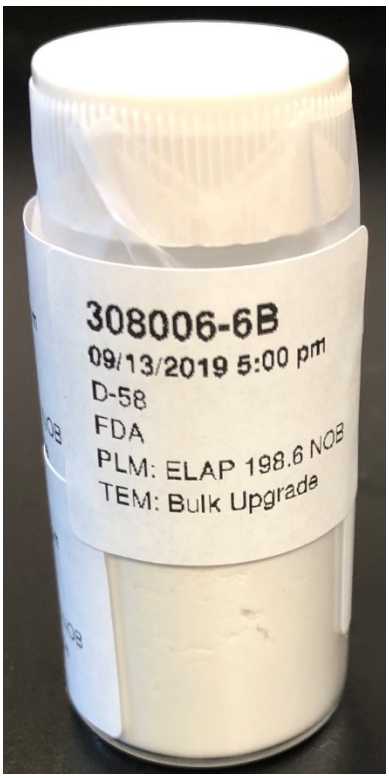
Technical Director: Andreas Saldivar

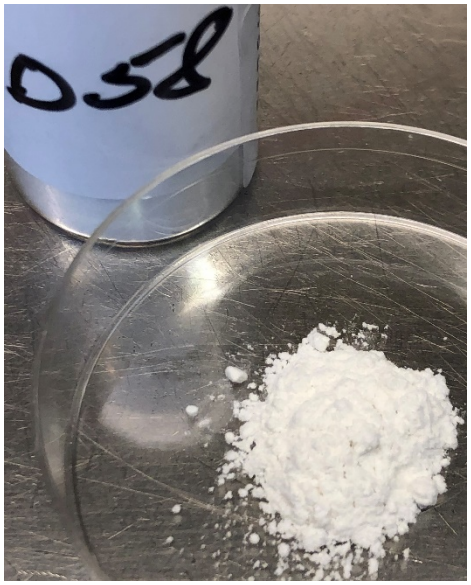
All results are to be considered preliminary and subject to change unless signed by the Technical Director or Deputy

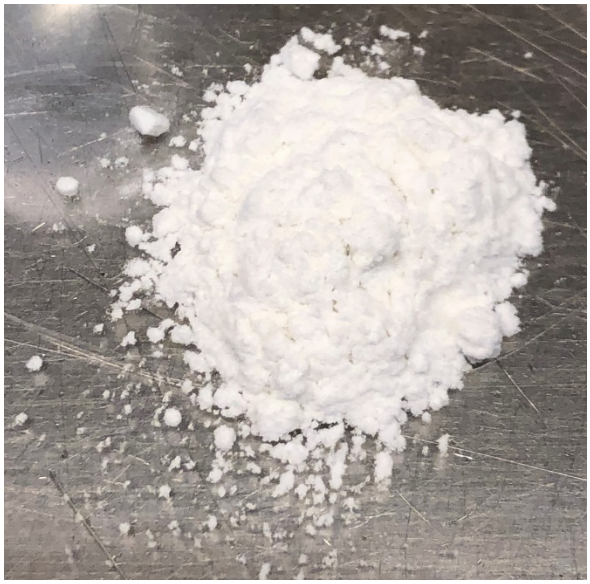
308006-6, 6A, 6B/D58











Sample Preparation

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

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

PLM Analysis

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

TEM Analysis

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

Modifications to the NY ELAP 198.4 Method were:

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

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

Calculations

ASTM D5756 Mass

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

M = mass
L = length

W = width
D = density
Percent Calculation

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

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

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

Limit of Detection and Quantification

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

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

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

Discussion and Interpretation of Analytical Findings:

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

PLM

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

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

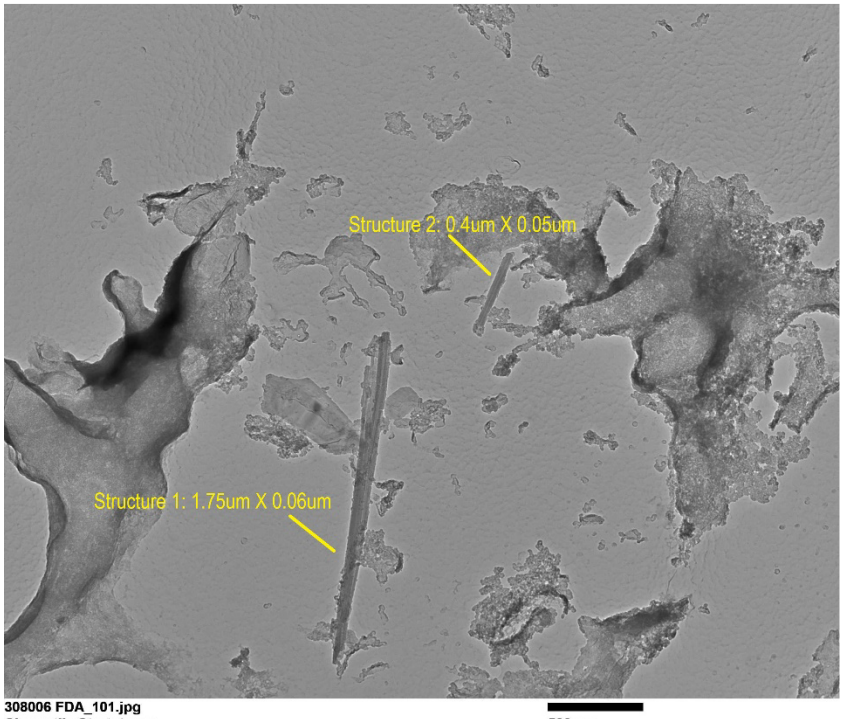
TEM

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

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

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

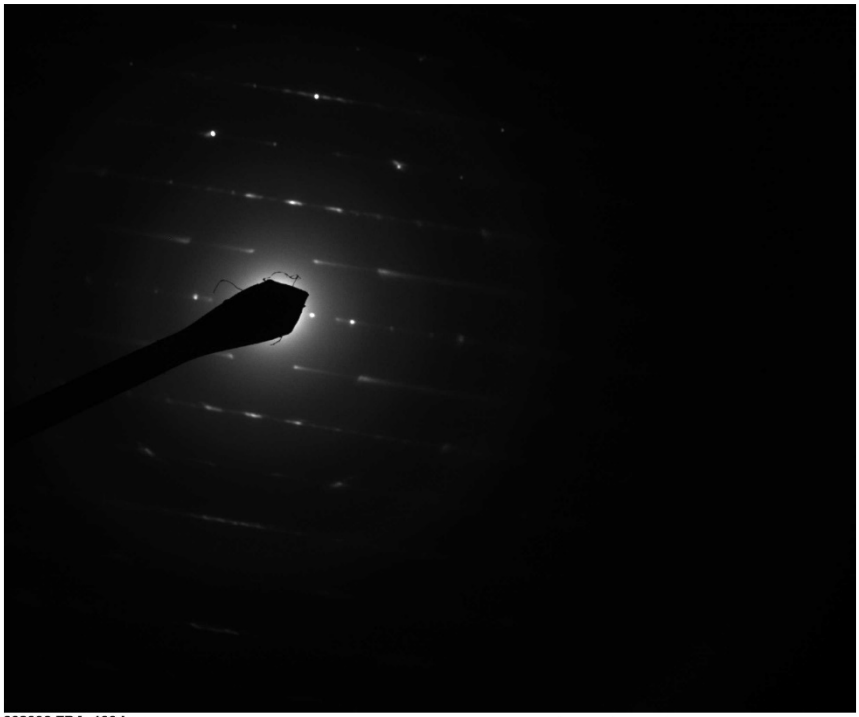
Sample 308006-6A, Chrysotile Structures



308006 FDA_101.jpg
Chrysotile Strutctures
308006-6a
Cal: 0.001774 µm/pix
14:06 9/7/2019
TEM Mode: Imaging
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 std. frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

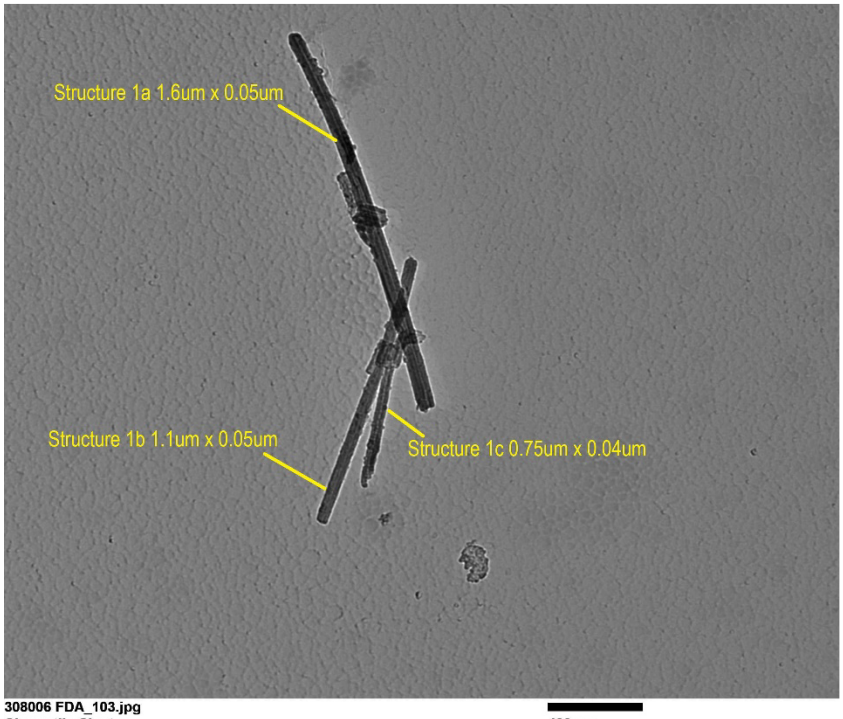
Diffraction Pattern from Chrysotile Structure 1 pictured above



308006 FDA_100.jpg
Chrysotile Dif
308006-6a
14:03 9/7/2019
TEM Mode: Diffraction
Microscopist: (b) (6)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 std. frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Sample 308006-6B, Chrysotile Structure 1



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

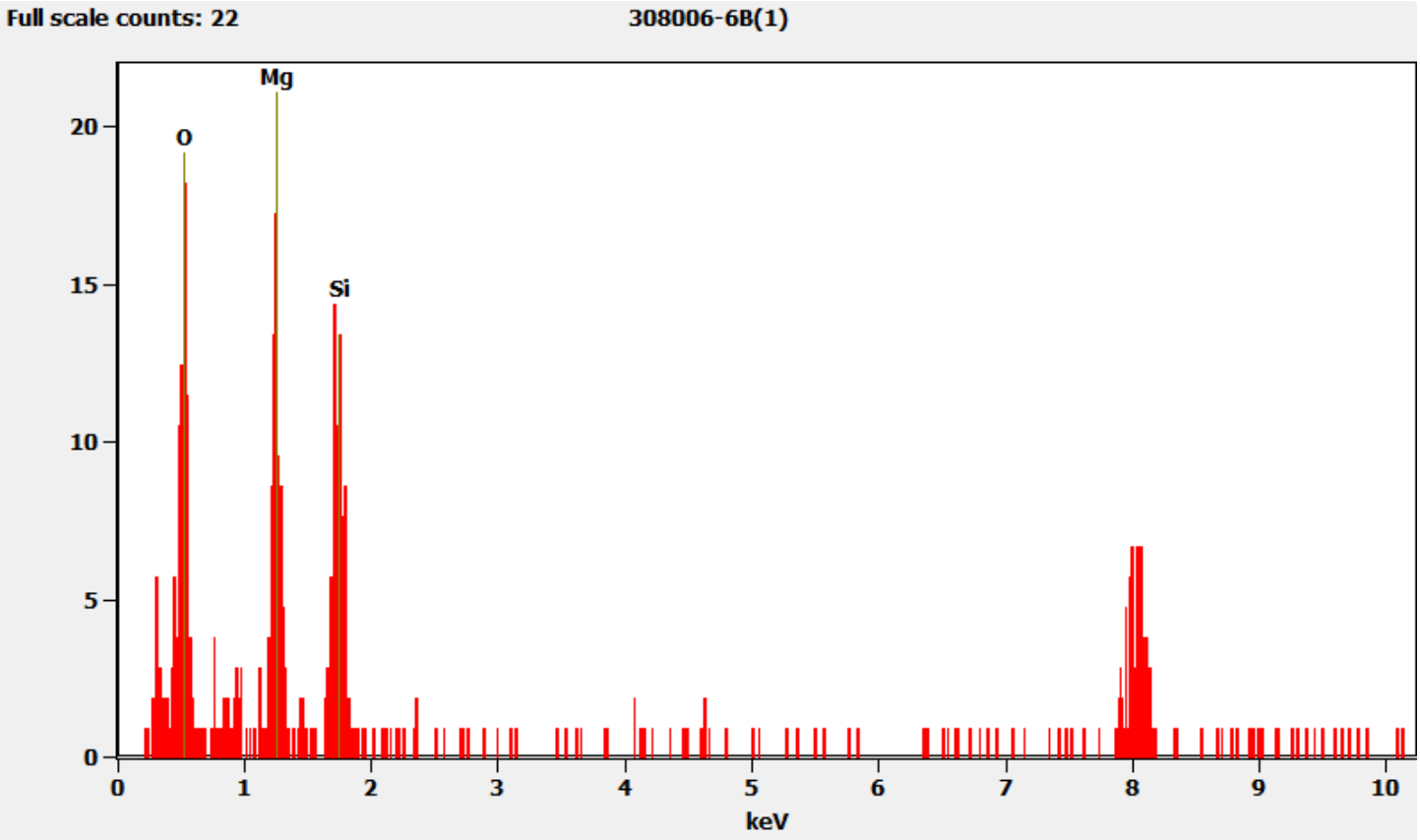
400 nm
HV=100kV
Direct Mag: 7200 x
AMA Analytical Services, Inc

Diffraction Pattern from Chrysotile Structure pictured above

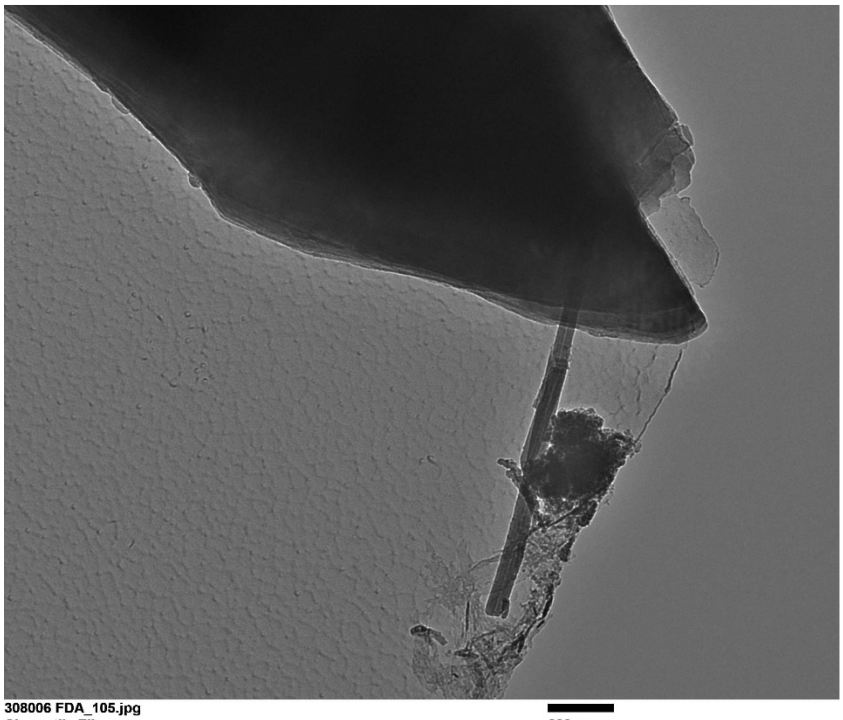
308006 FDA_102.jpg
Chrysotile Dif
308006-6b
15:32 9/7/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 std. frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from Chrysotile Structure pictured above



308006-6B, Chrysotile Structure 2

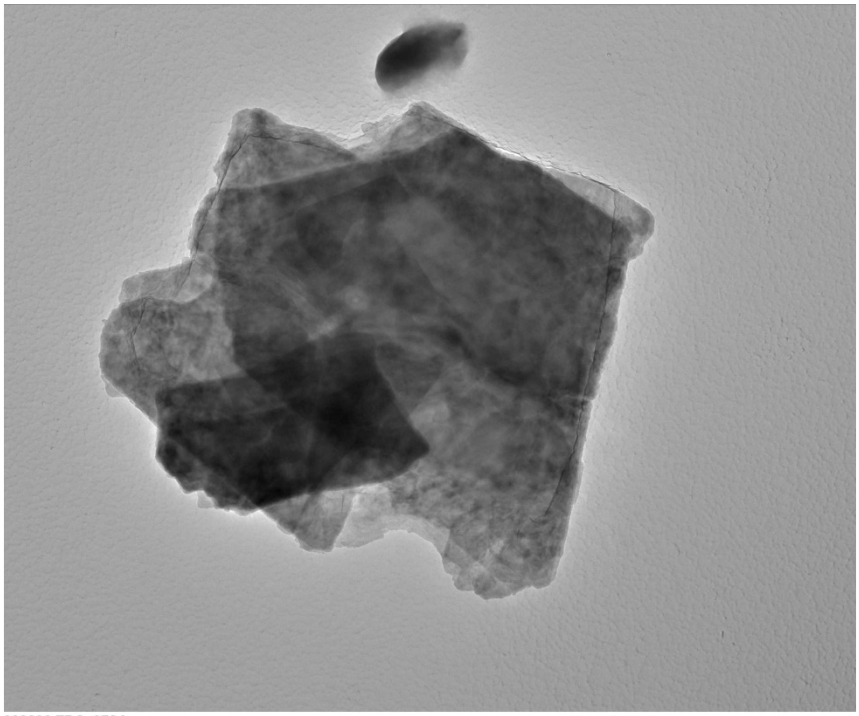


Diffraction Pattern from Chrysotile Structure pictured above

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

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

308006-6, Talc Particle

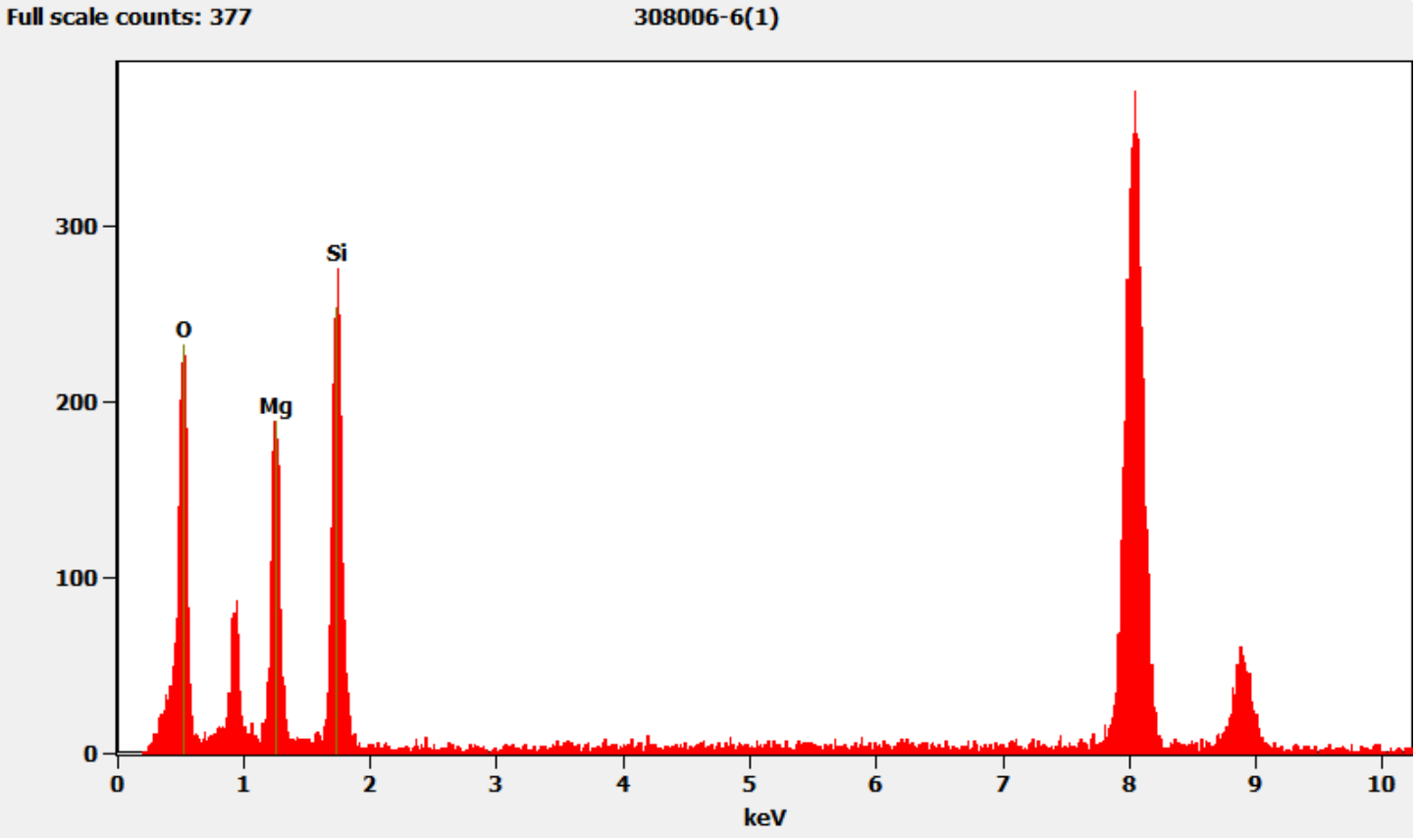


308006 FDA_052.jpg
Talc Particle
Cal: 0.001774 µm/pix
17:18 9/3/2019
TEM Mode: Imaging
Microscopist: (b) (6)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

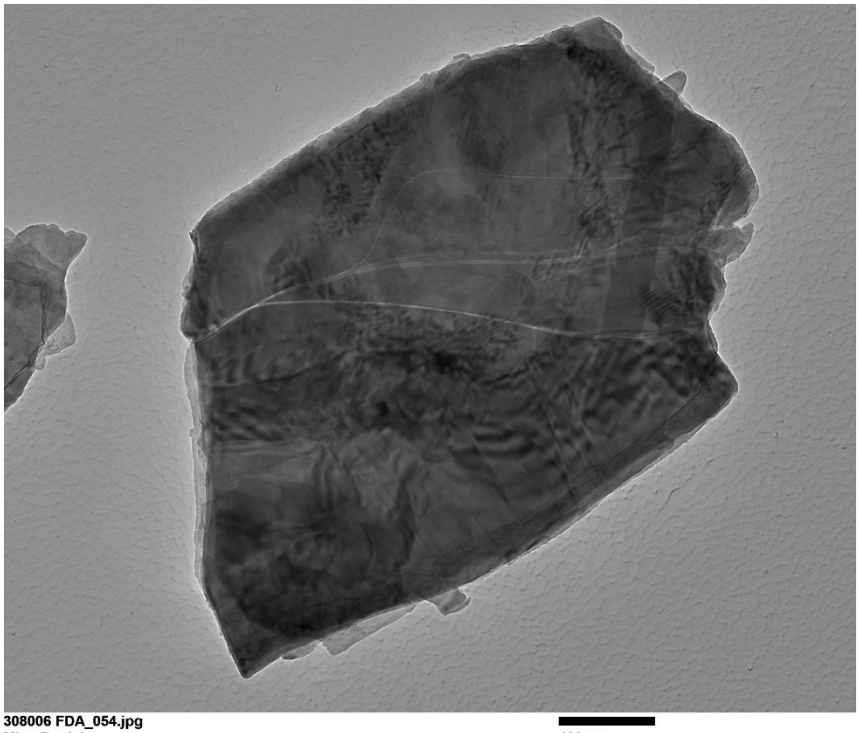
500 nm
HV=100kV
Direct Mag: 5800 x
AMA Analytical Services, Inc

Hexagonal Diffraction Pattern from Talc Particle pictured above

Chemistry from Talc Particle pictured above



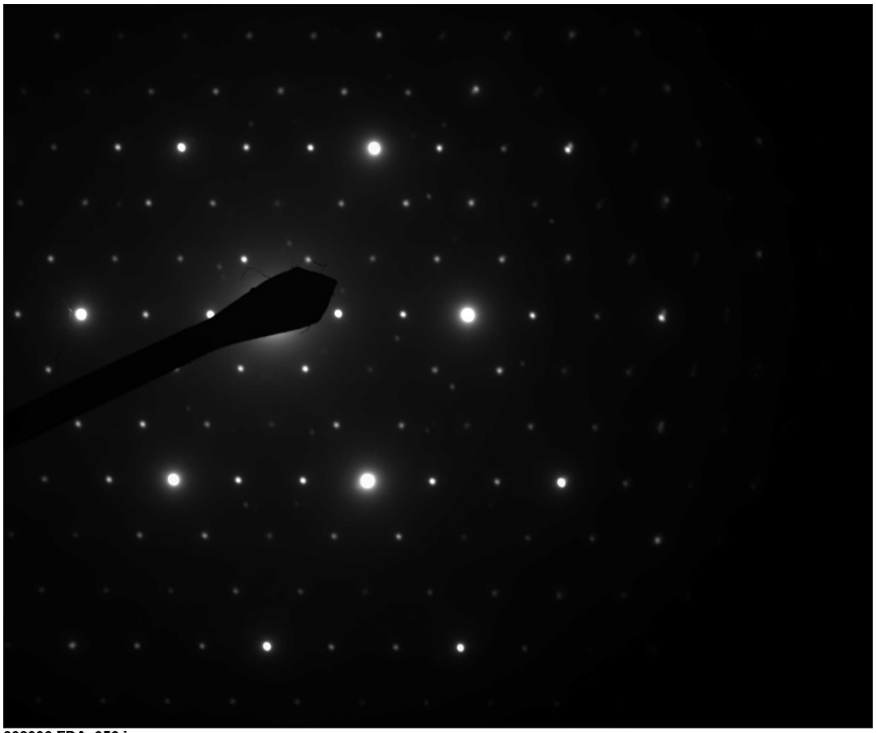
306008-6, Mica Particle



308006 FDA_054.jpg
Mica Particle
Cal: 0.001429 µm/pix
17:21 9/3/2019
TEM Mode: Imaging
Microscopist: (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

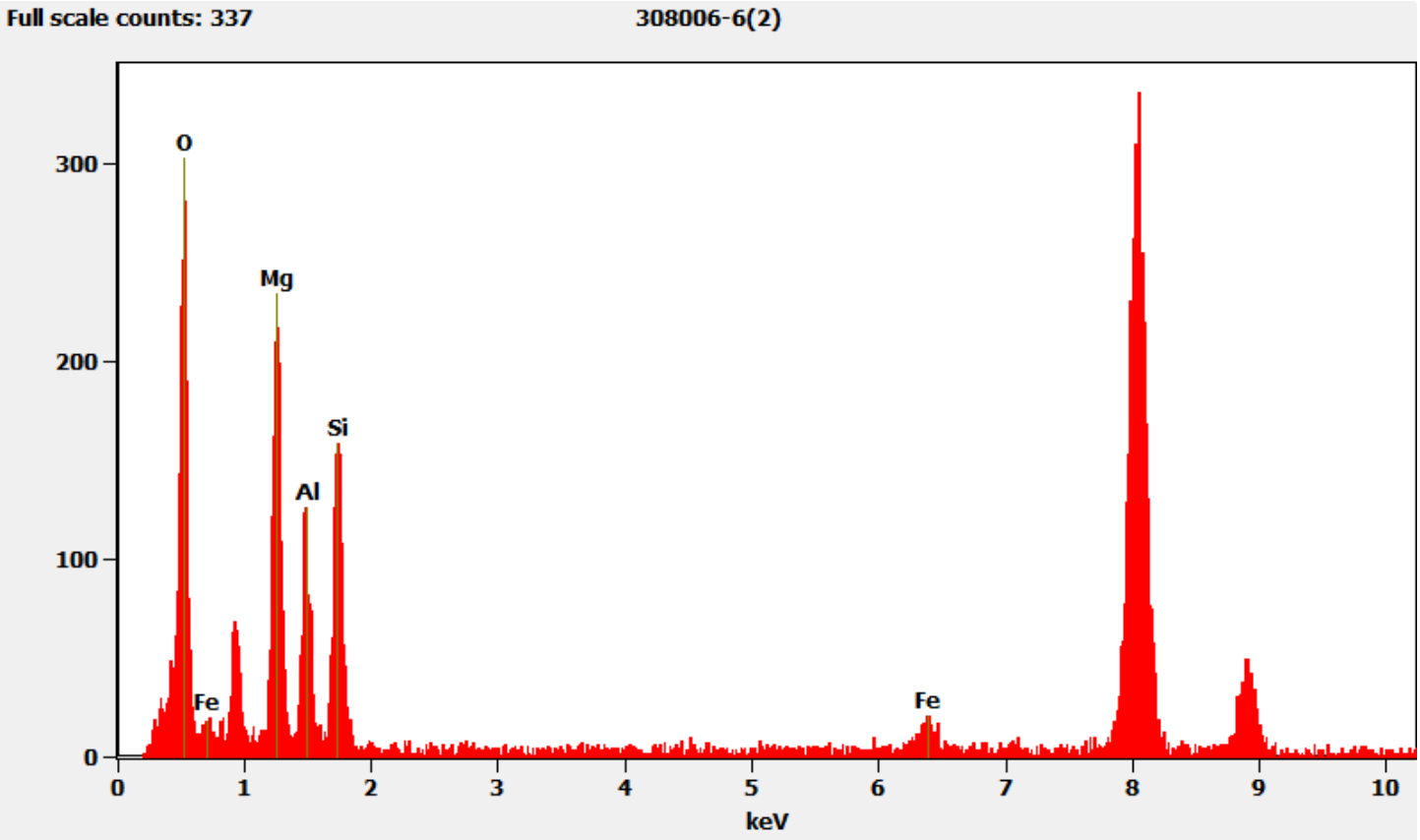
Diffraction Pattern from Mica Particle pictured above



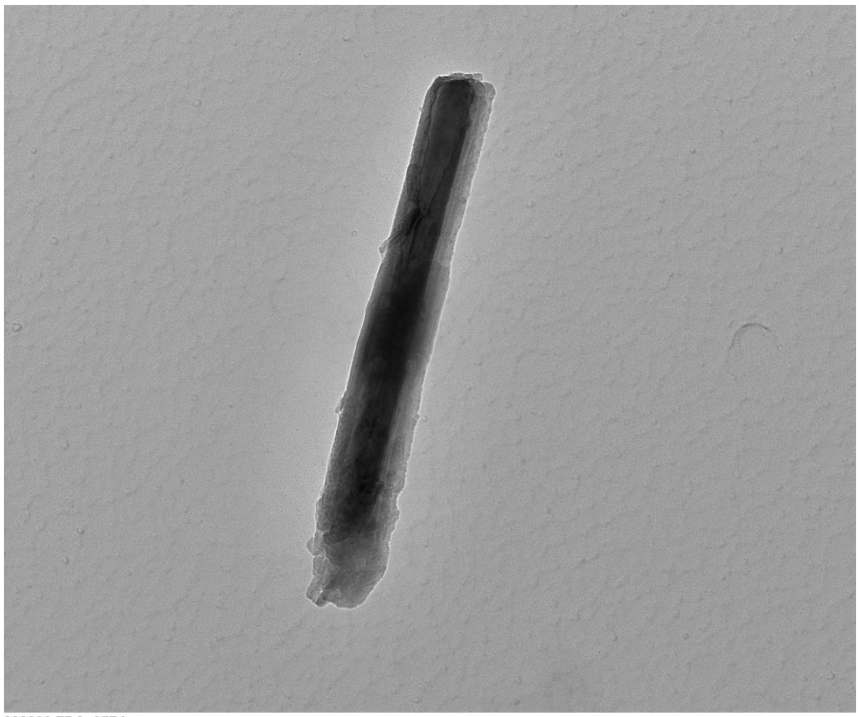
308006 FDA_056.jpg
Mica Particle
17:22 9/3/2019
TEM Mode: Diffraction
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

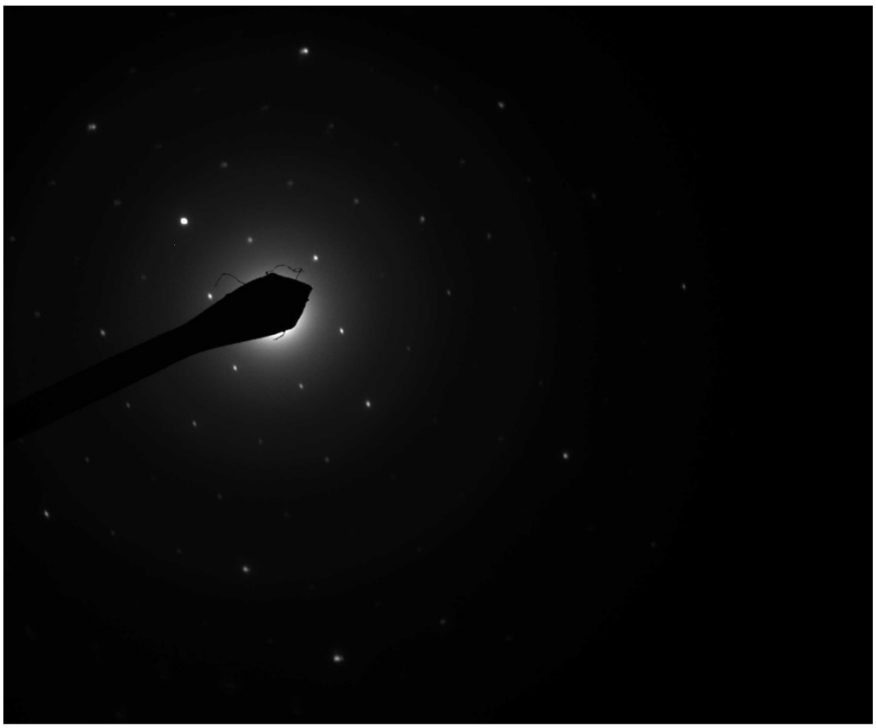
Chemistry from Mica Particle pictured above



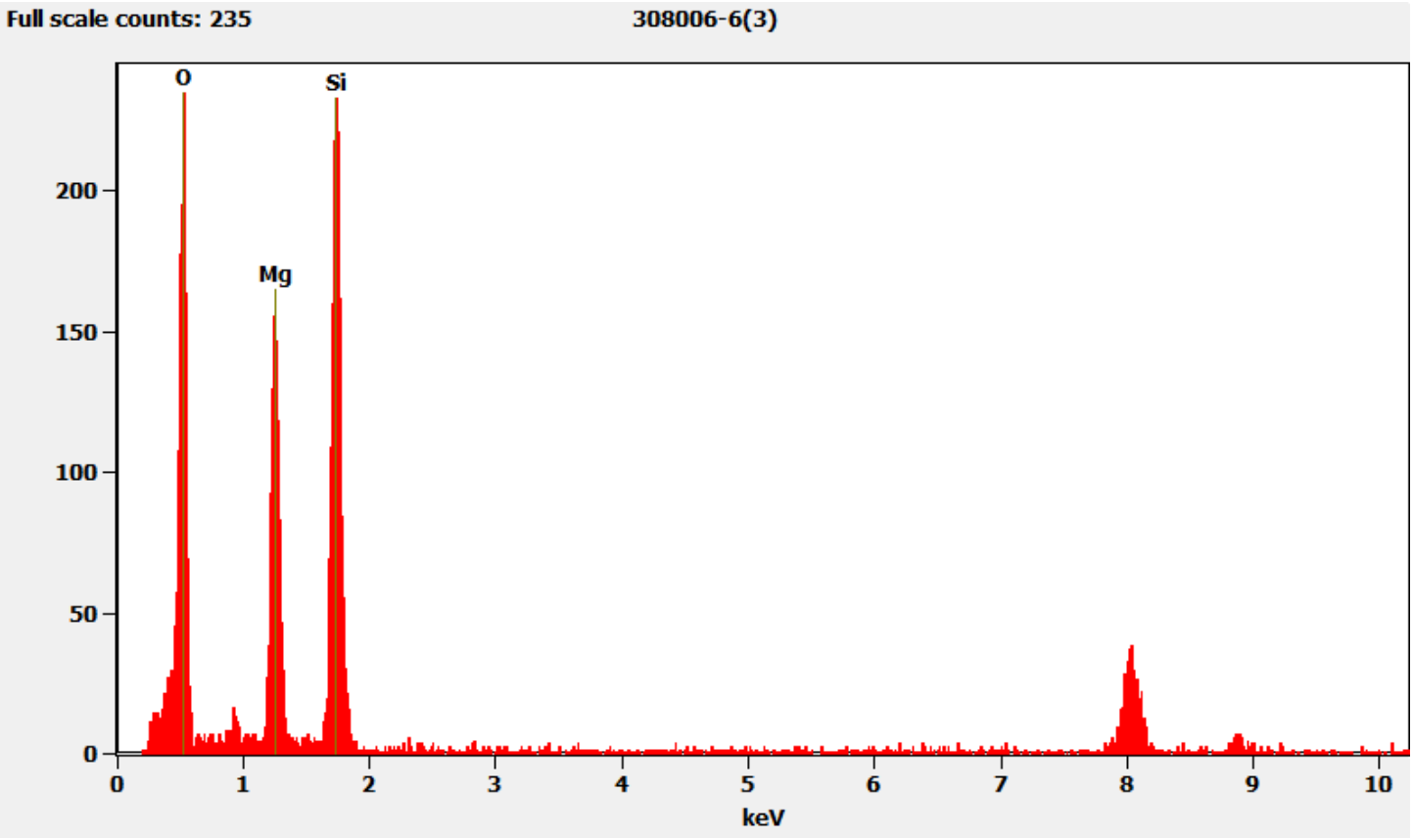
308006-6, Talc Fiber



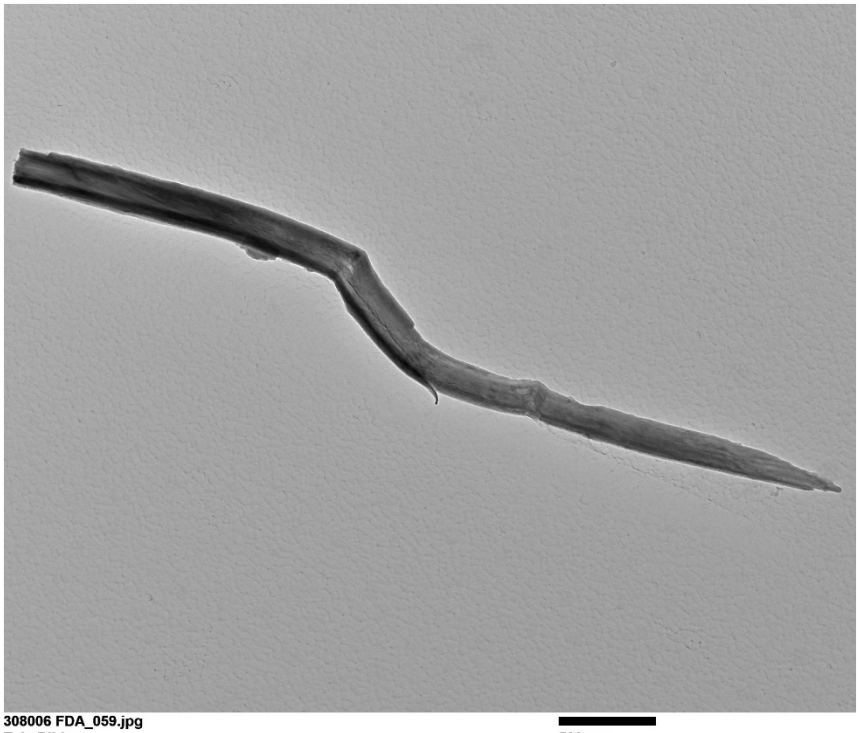
Diffraction Pattern from Talc Fiber pictured above



Chemistry from Talc Fiber pictured above



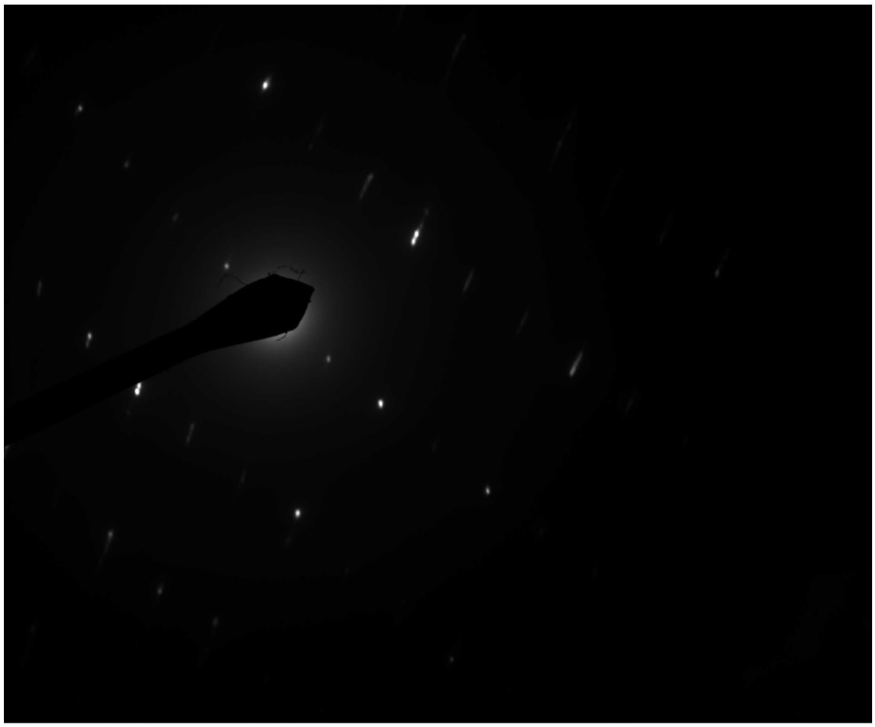
308006-6, Talc Ribbon



308006 FDA_059.jpg
Talc Ribbon
Cal: 0.001774 µm/pix
17:37 9/3/2019
TEM Mode: Imaging
Microscopist: (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

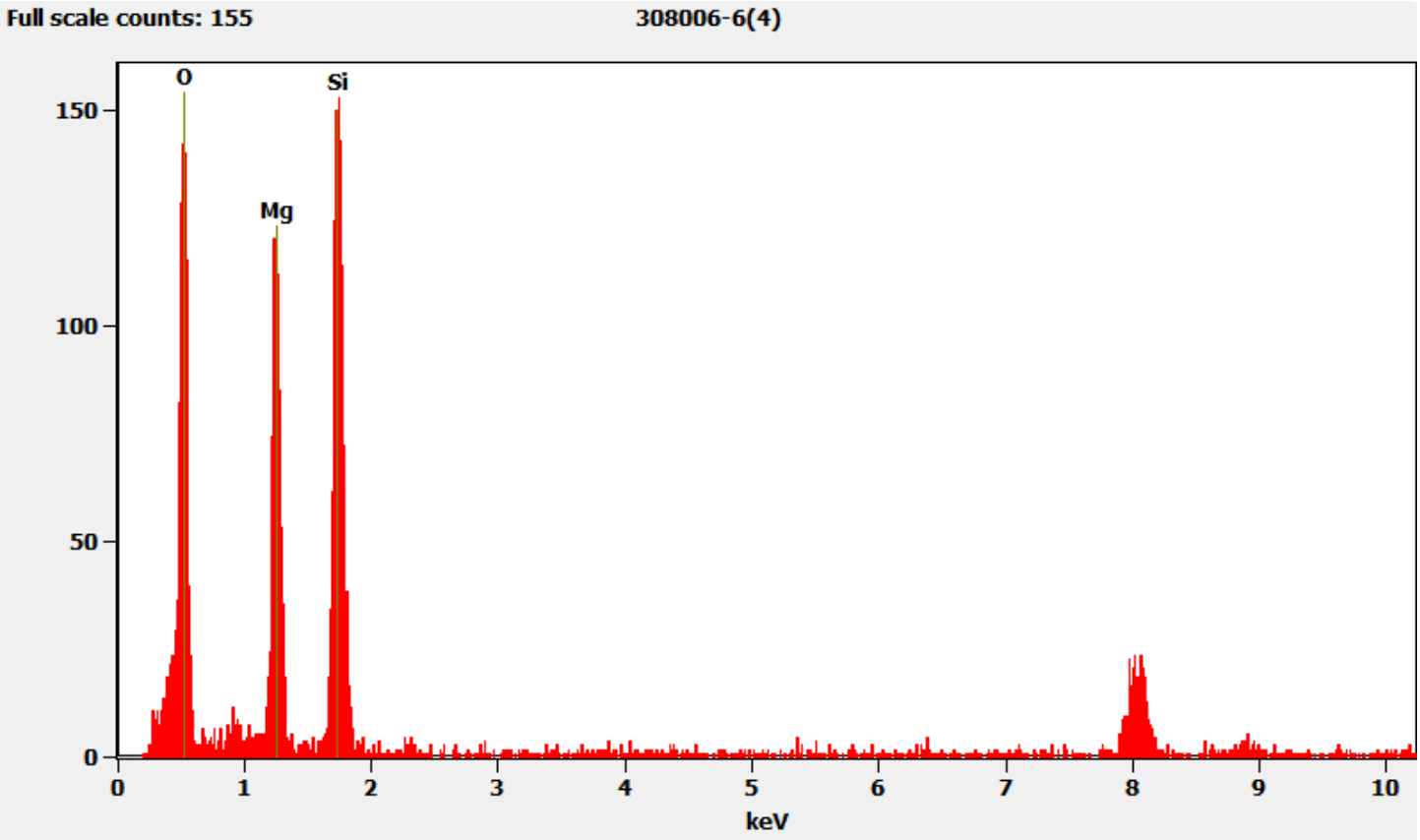
Diffraction Pattern from Talc Ribbon pictured above



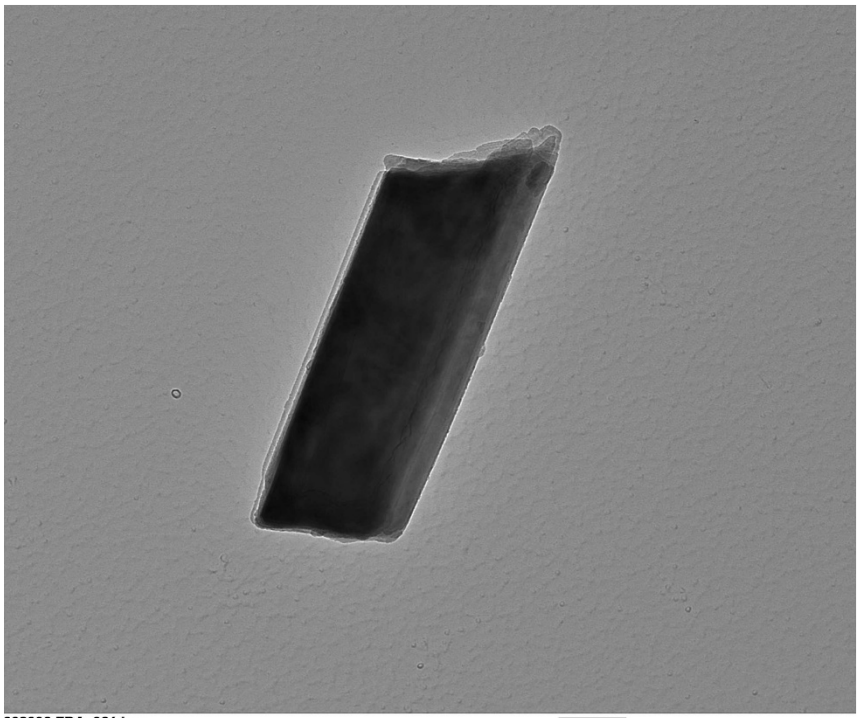
308006 FDA_060.jpg
Talc Ribbon
17:38 9/3/2019
TEM Mode: Diffraction
Microscopist: (b) (6)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Chemistry from Talc Ribbon pictured above



308006-6, Talc Fiber

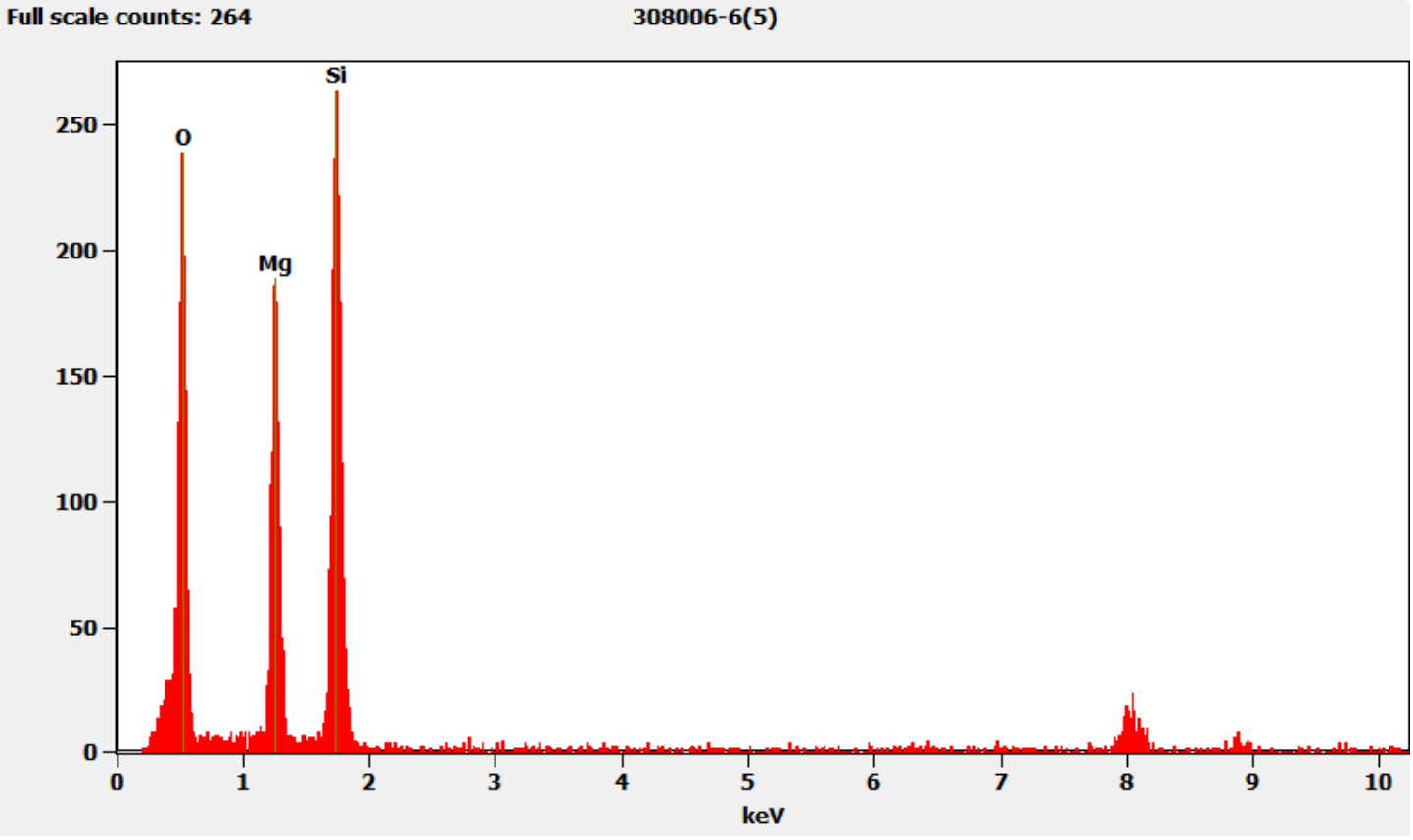


Diffraction Pattern from Talc Fiber pictured above

308006 FDA_062.jpg
Talc Fiber
17:51 9/3/2019
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 Talc Fiber pictured above



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

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

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

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

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

10/11/2019

Andreas Saldivar — Date
Laboratory Director