AMA Analytical Services, Inc.
Focused On Results.

CERTIFICATE OF ANALYSIS

Chain of Custody: 300396
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: 1st Group - 8 Samples
Job Number: CLIN 1 - Task 3 (8 Samples)
PO Number: HHSF223201810337P

Date Submitted: 3/14/2019
Date Analyzed: 3/29/2019 - 4/18/2019
Report Date: 4/25/2019
Date Sampled: Not Provided
Person Submitting: Steve Wolfgang
Revised: 1/23/2020 (2nd Revision)

Summary of Analysis

AMA Sample ID	Client Sample ID	TEM LOD Using ASTM D5756 Mass Calculation	TEM LOQ Using ASTM D5756 Mass Calculation	% Tremolite by TEM Using ASTM D5756 Mass Calculation	% Chrysotile by TEM Using ASTM D5756 Mass Calculation	% Total Tremolite and Chrysotile by TEM Using ASTM D5756 Mass Calculation	% Asbestos by PLM	% Organics	% Acid Soluable	% Other	Comments
300396-1	D-32	0.00000218%	0.00000872%	ND	ND	ND	ND	6.2%	5.0%	88.8%	
300396-1A	D-32	0.00000162%	0.00000648%	ND	ND	ND	ND	6.2%	1.4%	92.4%	
300396-1B	D-32	0.00000144%	0.00000574%	ND	ND	ND	ND	6.2%	4.0%	89.8%	
300396-2	D-33	0.00000192%	0.00000769%	ND	ND	ND	ND	28.7%	3.3%	68.0%	
300396-2A	D-33	0.00000205%	0.00000819%	ND	ND	ND	ND	29.4%	2.1%	68.5%	
300396-2B	D-33	0.00000193%	0.00000773%	ND	ND	ND	ND	29.1%	3.3%	67.6%	
300396-3	D-34	0.00000254%	0.00001016%	ND	ND	ND	ND	24.7%	4.7%	70.6%	
300396-3A	D-34	0.00000285%	0.00080274%	ND	< 0.00080%	< 0.00080%	ND	23.4%	4.5%	72.1%	
300396-3B	D-34	0.00000370%	0.00001479%	ND	0.00030%	0.00030%	ND	24.0%	3.9%	72.0%	
300396-4	D-35	0.00000134%	0.00000536%	0.00071%	0.00503%	0.00574%	ND	12.8%	12.4%	74.7%	
300396-4A	D-35	0.00000188%	0.00012905%	< 0.00013%	< 0.00013%	< 0.00013%	ND	13.7%	13.8%	72.5%	
300396-4B	D-35	0.00000168%	0.00000671%	0.00367%	0.00005%	0.00371%	ND	12.5%	15.5%	73.0%	
300396-5	D-36	0.00000188%	0.00000751%	ND	ND	ND	ND	24.0%	3.5%	72.5%	
300396-5A	D-36	0.00000114%	0.00000454%	ND	ND	ND	ND	24.1%	3.0%	72.8%	
300396-5B	D-36	0.00000150%	0.00000599%	ND	ND	ND	ND	24.1%	2.5%	73.4%	
300396-6	D-37	0.00000150%	0.00000599%	ND	ND	ND	ND	19.2%	6.7%	74.1%	
300396-6A	D-37	0.00000178%	0.00000714%	ND	ND	ND	ND	18.9%	5.3%	75.8%	
300396-6B	D-37	0.00000157%	0.00000629%	ND	ND	ND	ND	18.3%	6.0%	75.8%	
300396-7	D-38	0.00000134%	0.00000536%	ND	ND	ND	ND	0.0%	3.1%	96.8%	
300396-7A	D-38	0.00000173%	0.00000694%	ND	ND	ND	ND	0.1%	2.4%	97.5%	
300396-7B	D-38	0.00000135%	0.00000539%	ND	ND	ND	ND	0.0%	2.5%	97.4%	
300396-8	D-39	0.00000131%	0.00000524%	ND	ND	ND	ND	55.3%	11.0%	33.7%	
300396-8A	D-39	0.00000180%	0.00000721%	ND	ND	ND	ND	55.3%	8.3%	36.4%	
300396-8B	D-39	0.00000135%	0.00000540%	ND	ND	ND	ND	55.3%	15.4%	29.3%	

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

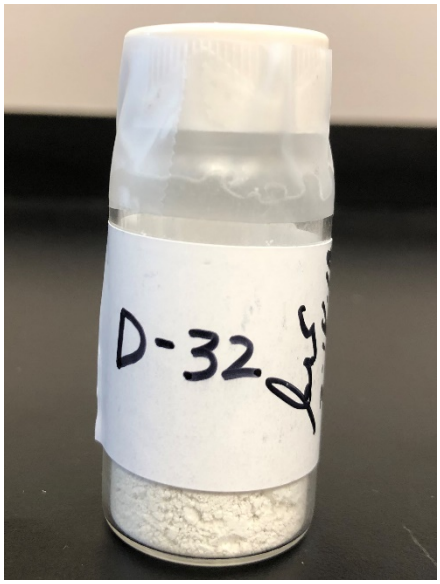
Analytical Method(s): PLM by Modified NY ELAP 198.6
TEM by Modified NY ELAP 198.4/ASTM D5756

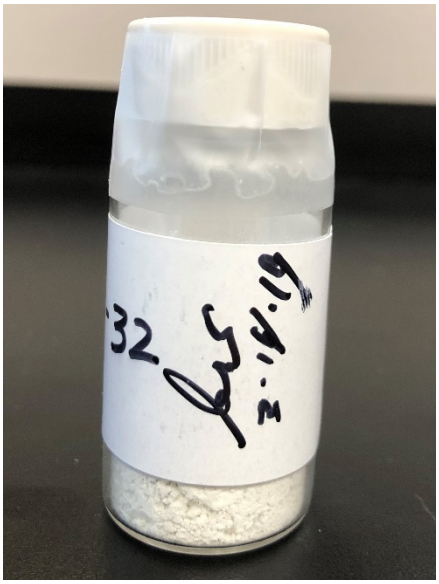
Analyst(s): **PLM** (b) (6)
TEM (b) (6)

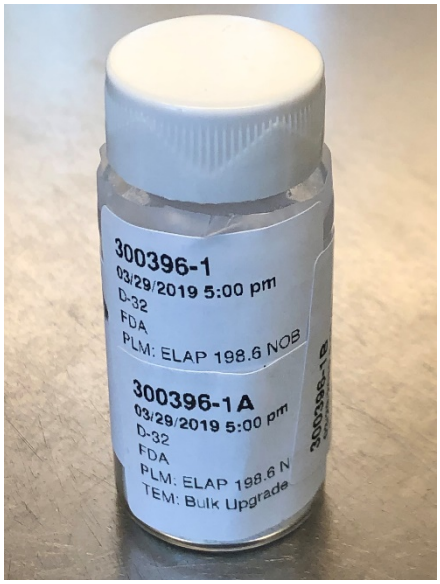
Technical Director: Andreas Saldivar

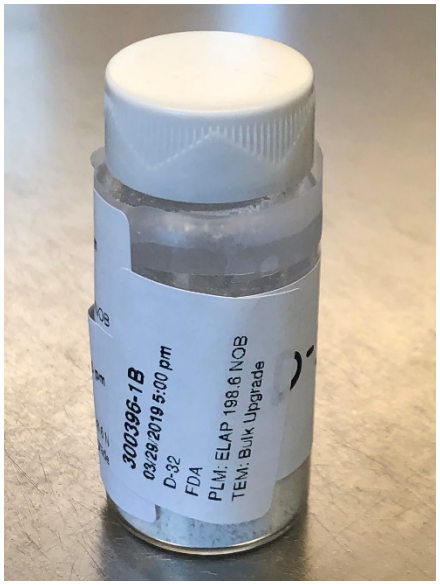
All results are to be considered preliminary and subject to change unless signed by the Technical Director or Deputy

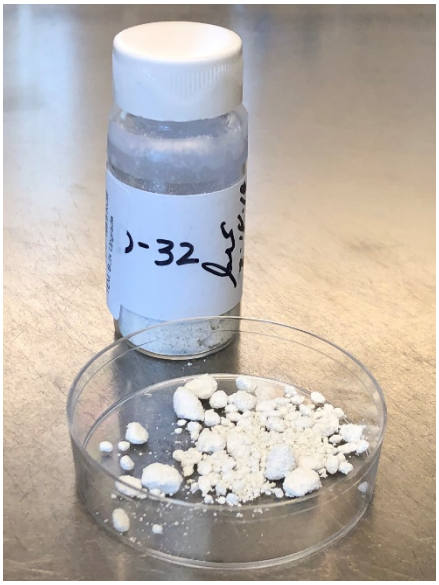
300396-1, 1A, 1B/D-32

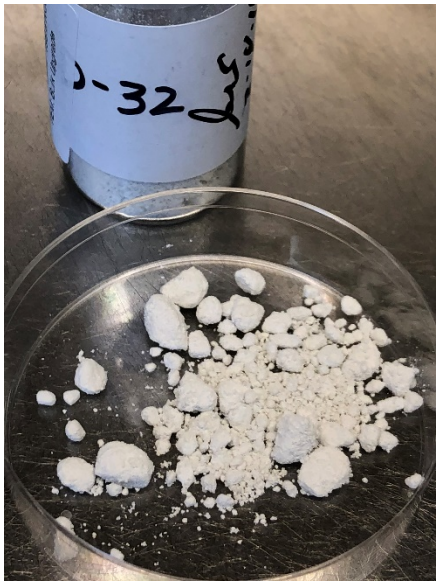




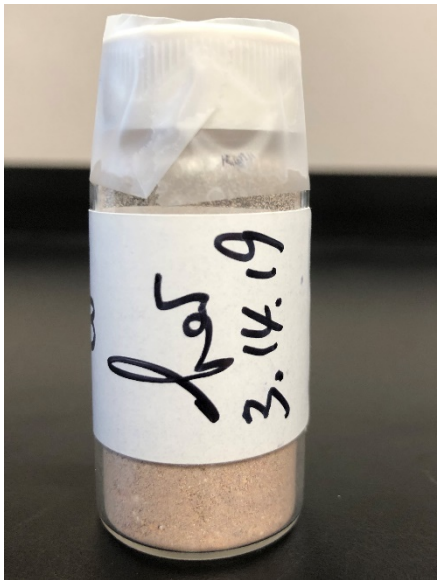


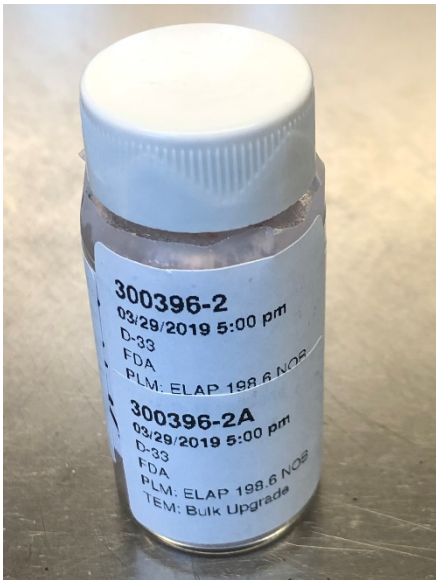


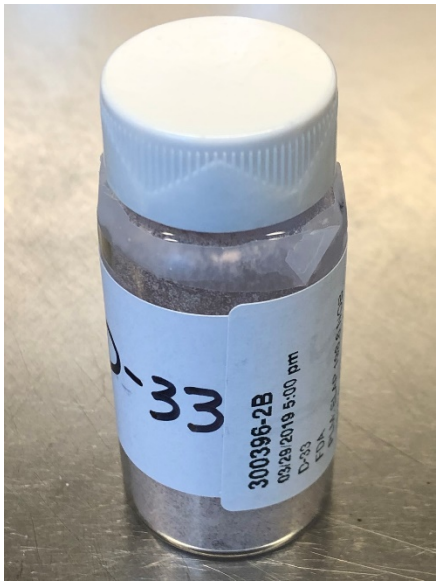


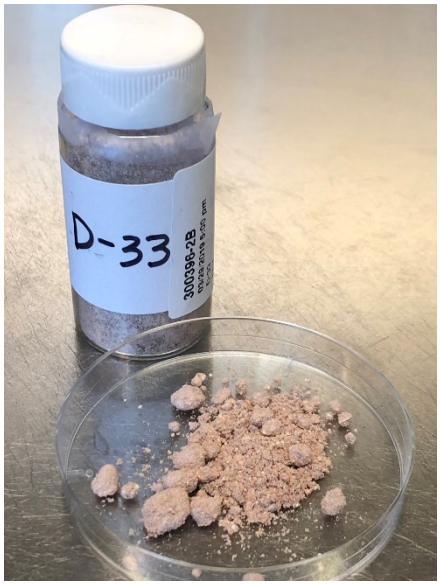


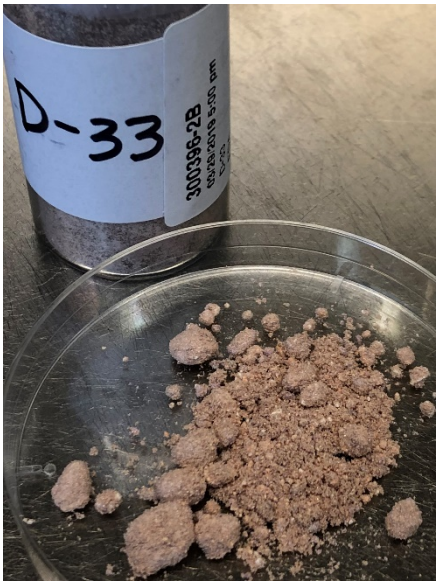
300396-2, 2A, 2B/D-33



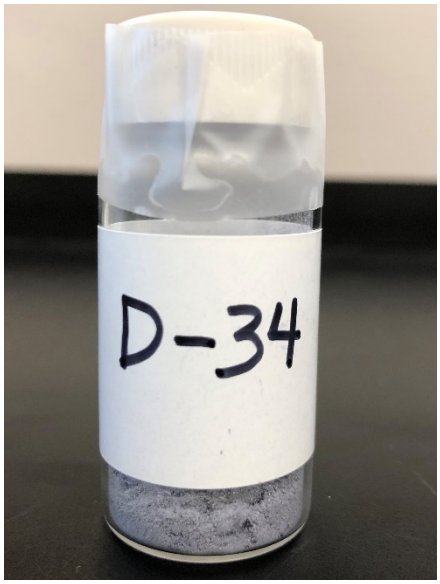


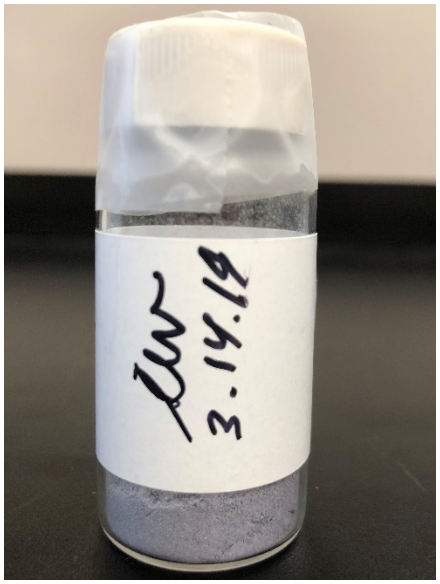


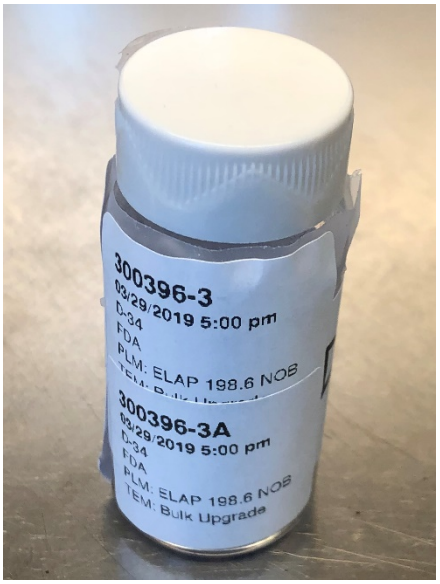


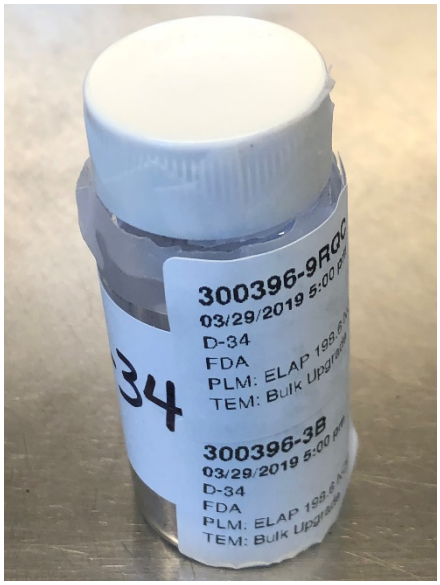


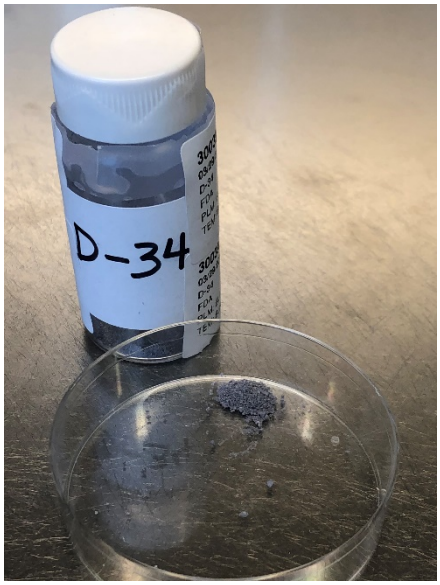
300396-3, 3A, 3B/D-34



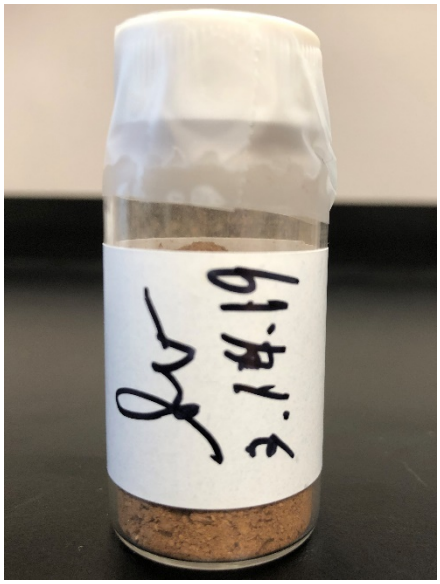


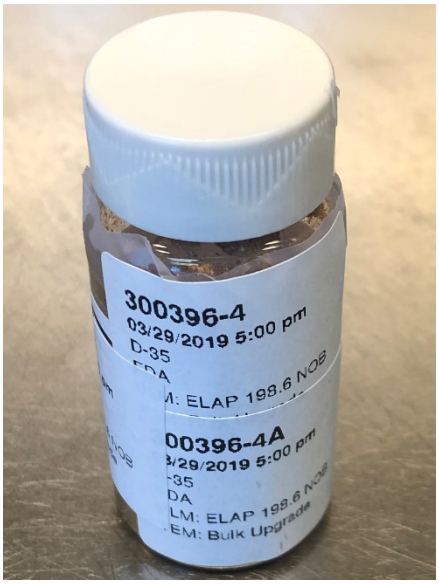


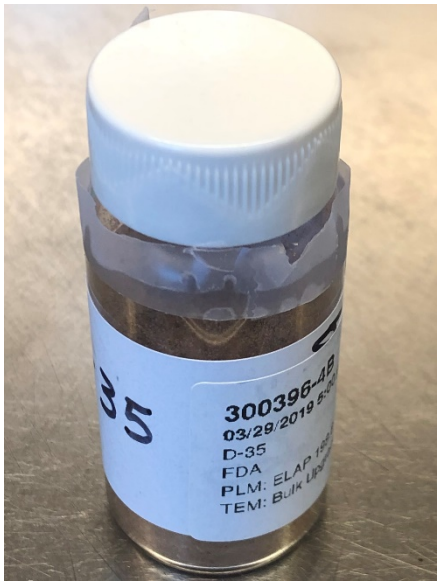


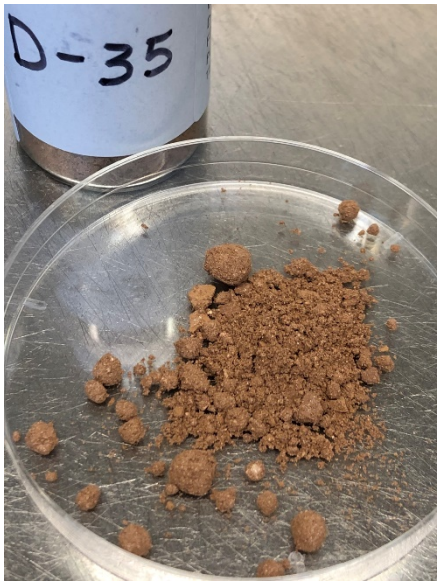


300396-4, 4A, 4B/D-35

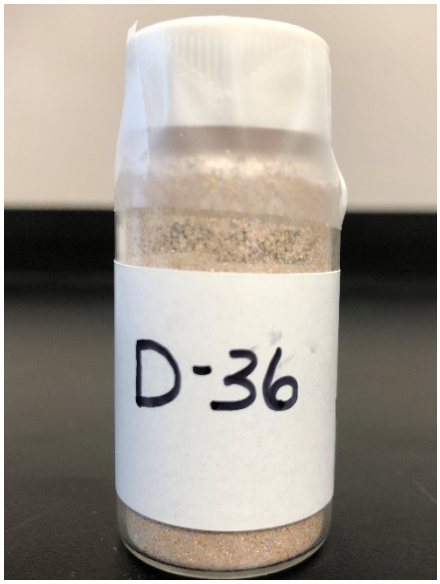


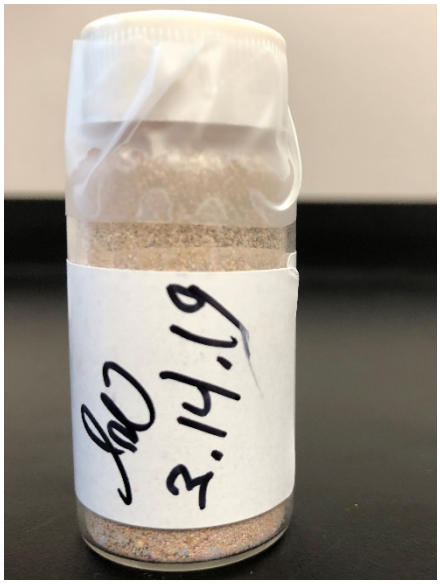


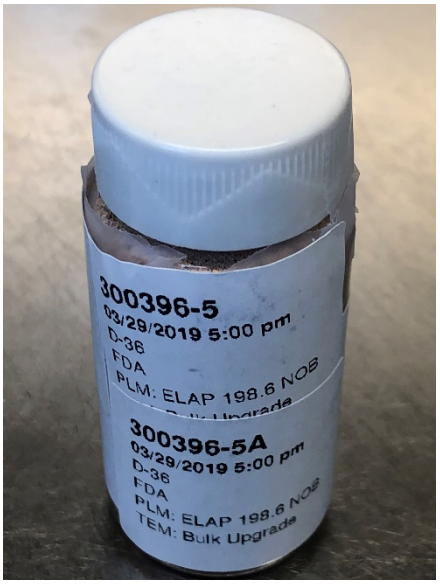


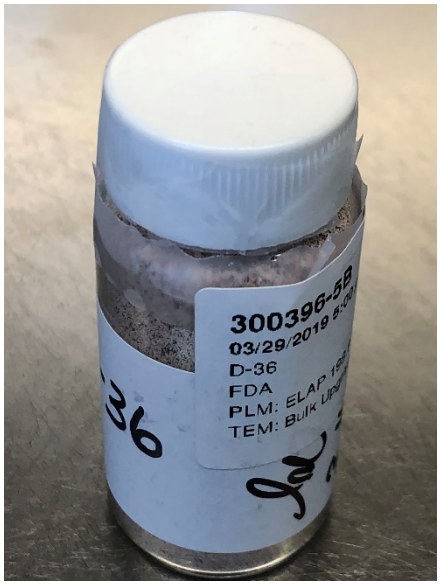


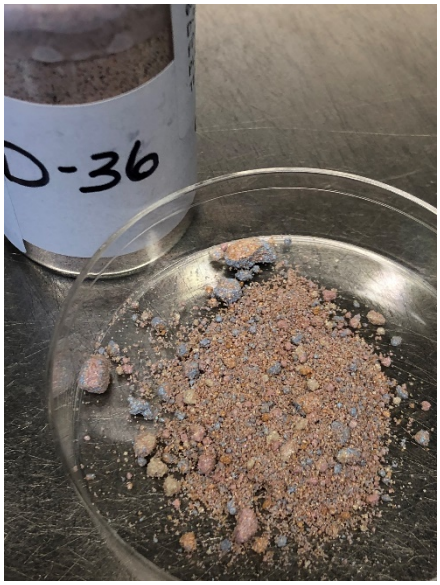
300396-5, 5A, 5B/D-36



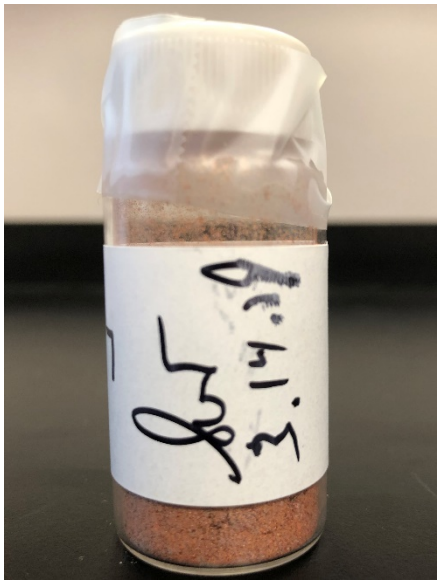


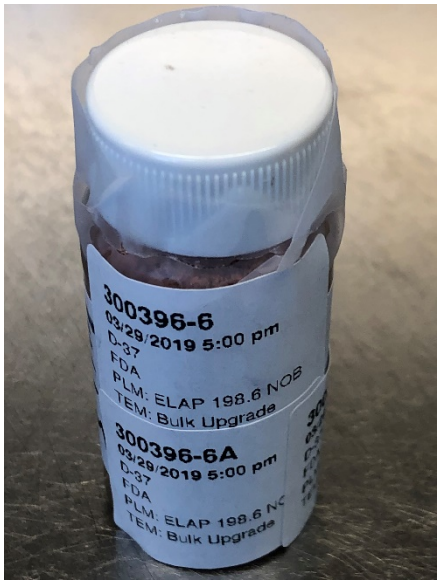


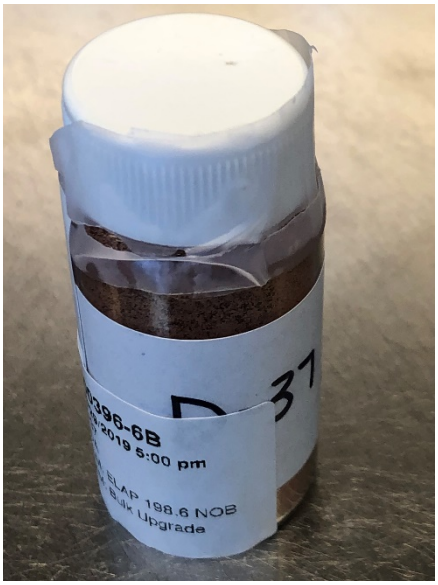




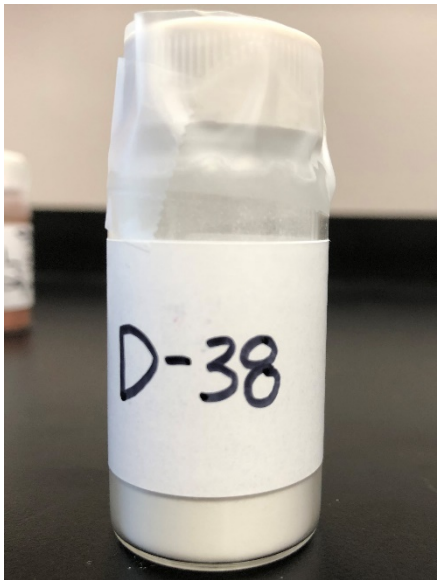
300396-6, 6A, 6B/D-37

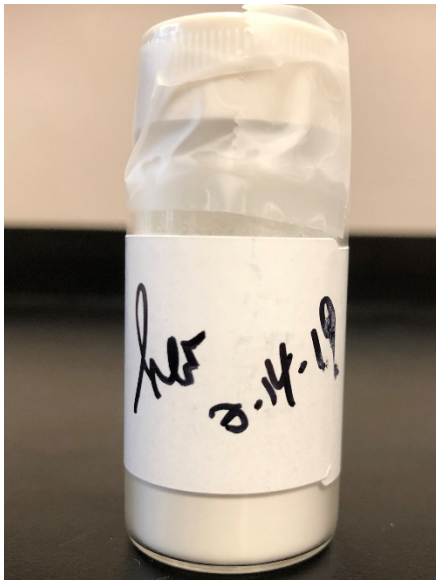


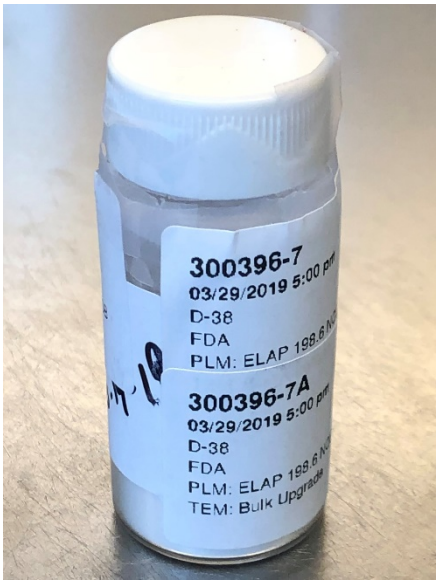




300396-7/D-38


D-38

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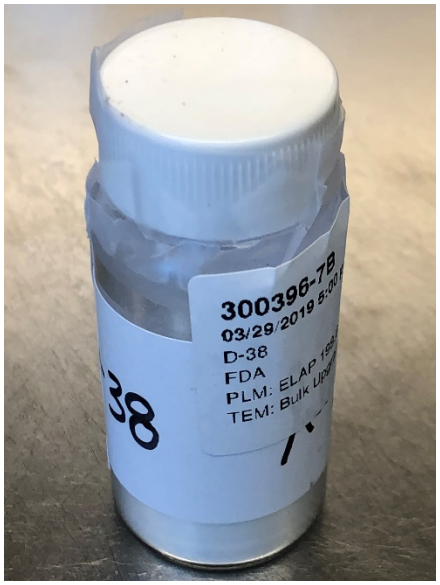
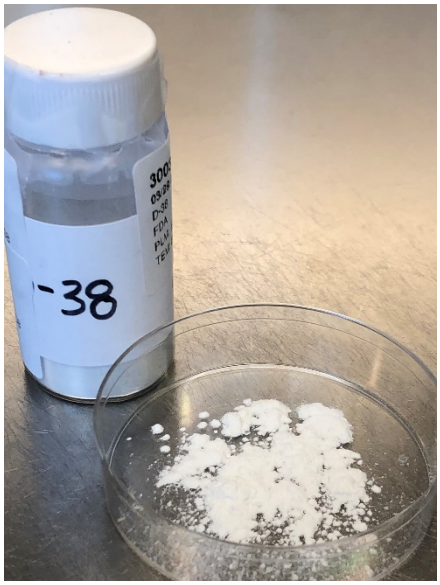
03/29/2019 5:00 pm
D-38
FDA
PLM: ELAP 198.6
300396-7A
03/29/2019 5:00 pm
D-38
FDA
PLM: ELAP 198.6
TEM: Bulk Upgrade


300396-7B
03/29/2019
D-38
FDA
PLM: ELAP
TEM: Bulk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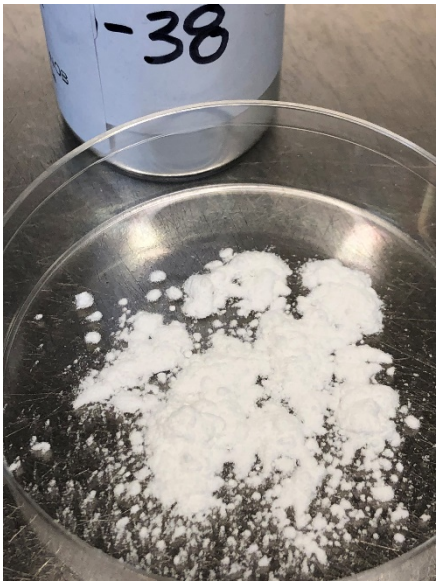

-38

300396-8/D39

Sample Preparation

Samples were prepared for PLM and TEM bulk analysis by [redacted] on March 15, 2019 through March 29, 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.

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 D34 and D35 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:

300396-3A: mass of the single observed chrysotile fiber plus the mass of three tremolite fibers measuring 0.5 x 0.04 microns

300396-4A: mass of the two observed chrysotile fibers, the single observed tremolite structure plus the mass of one 0.5 x 0.04 microns tremolite fiber.

Discussion and Interpretation of Analytical Findings:

<u>300396-1, 1A, 1B, Client Sample D-32</u>

PLM

All three aliquots of sample D-32 were analyzed by (b) (6) on March 29, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

300396-1	NAD
300396-1A	NAD
300396-1B	NAD

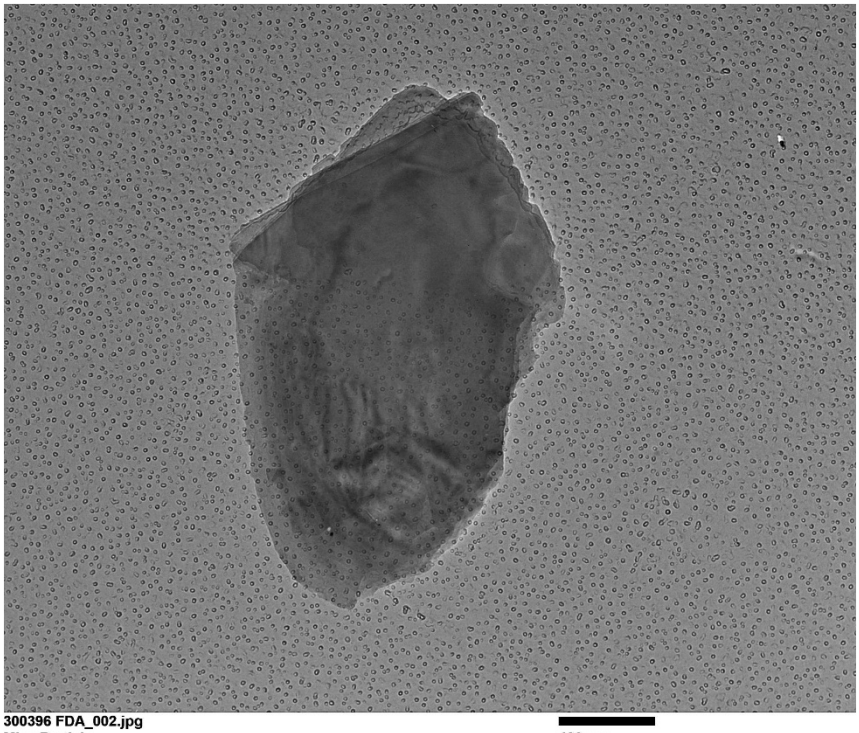
TEM

Sample 1 and 1A were analyzed by (b) (6) on April 2, 2019. Sample 1B was analyzed by (b) (6) on April 4, 2019. The primary particles observed were mica and titanium oxide particles. No talc and no asbestos or non-asbestos amphibole variants were detected in the samples. The results were calculated using the equations detailed in the calculations section.

300396-1	NAD
300396-1A	NAD
300396-1B	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.

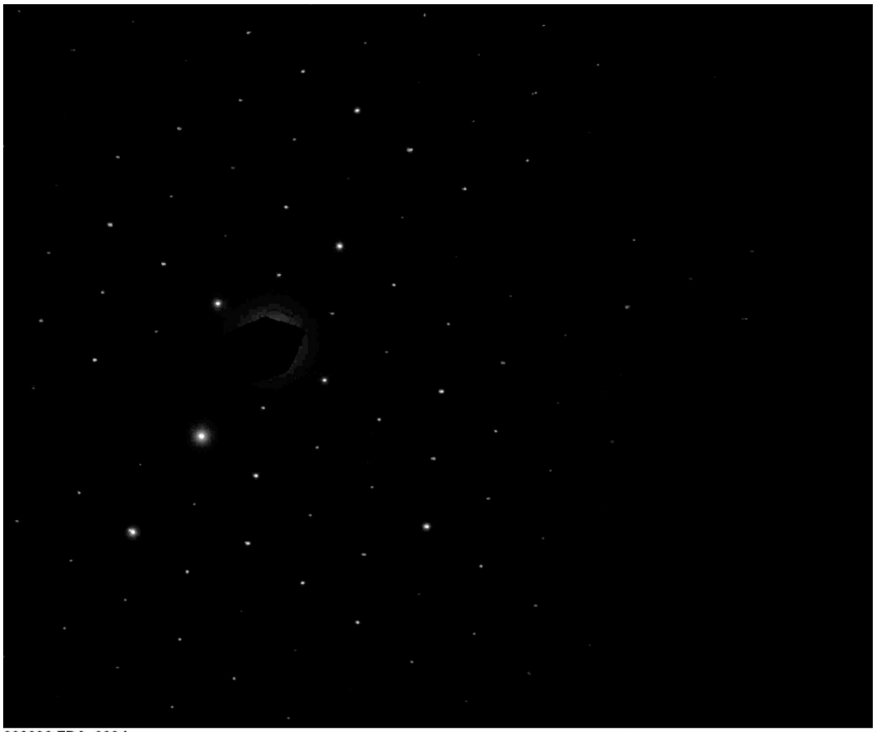
Sample 300396-1, Mica particle



300396 FDA_002.jpg
Mica Particle
Spectra 1
Cal: 0.001429 µm/pix
09:45 4/2/2019
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

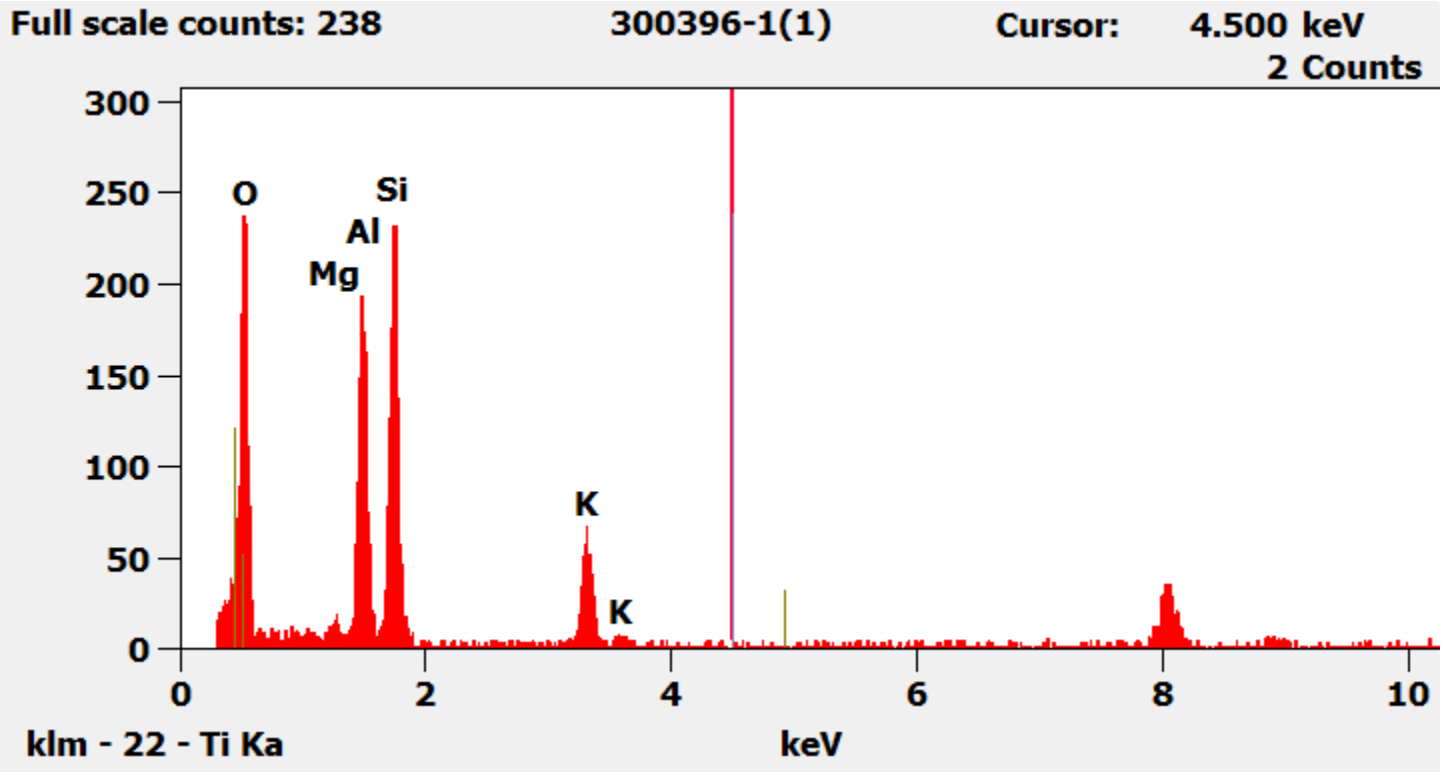
Sample 300396-1 Hexagonal diffraction from mica particle pictured above.



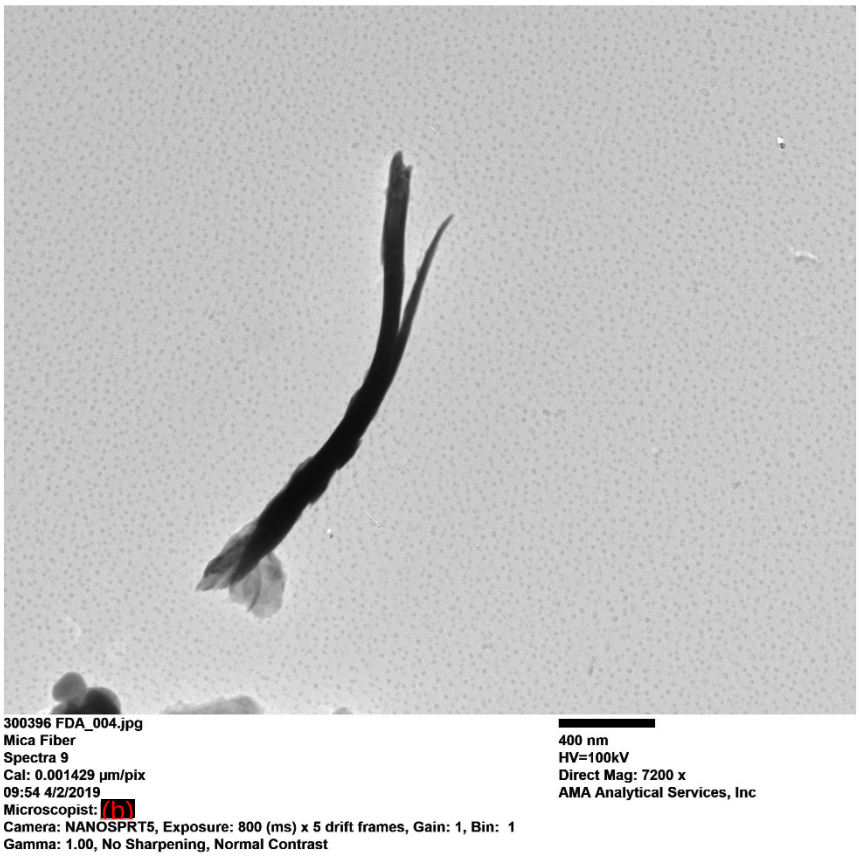
300396 FDA_003.jpg
Mica Particle Diffraction
Spectra 1
09:46 4/2/2019
Microscopist: (b)
Camera: (b) (4) Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

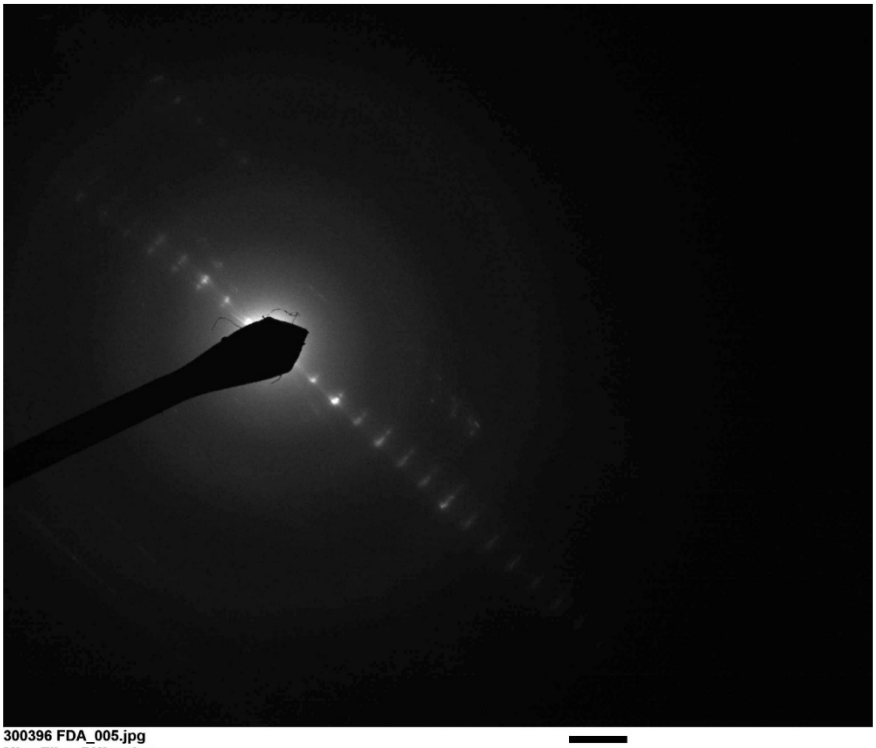
Sample 300396-1 Chemistry from mica particle pictured above.



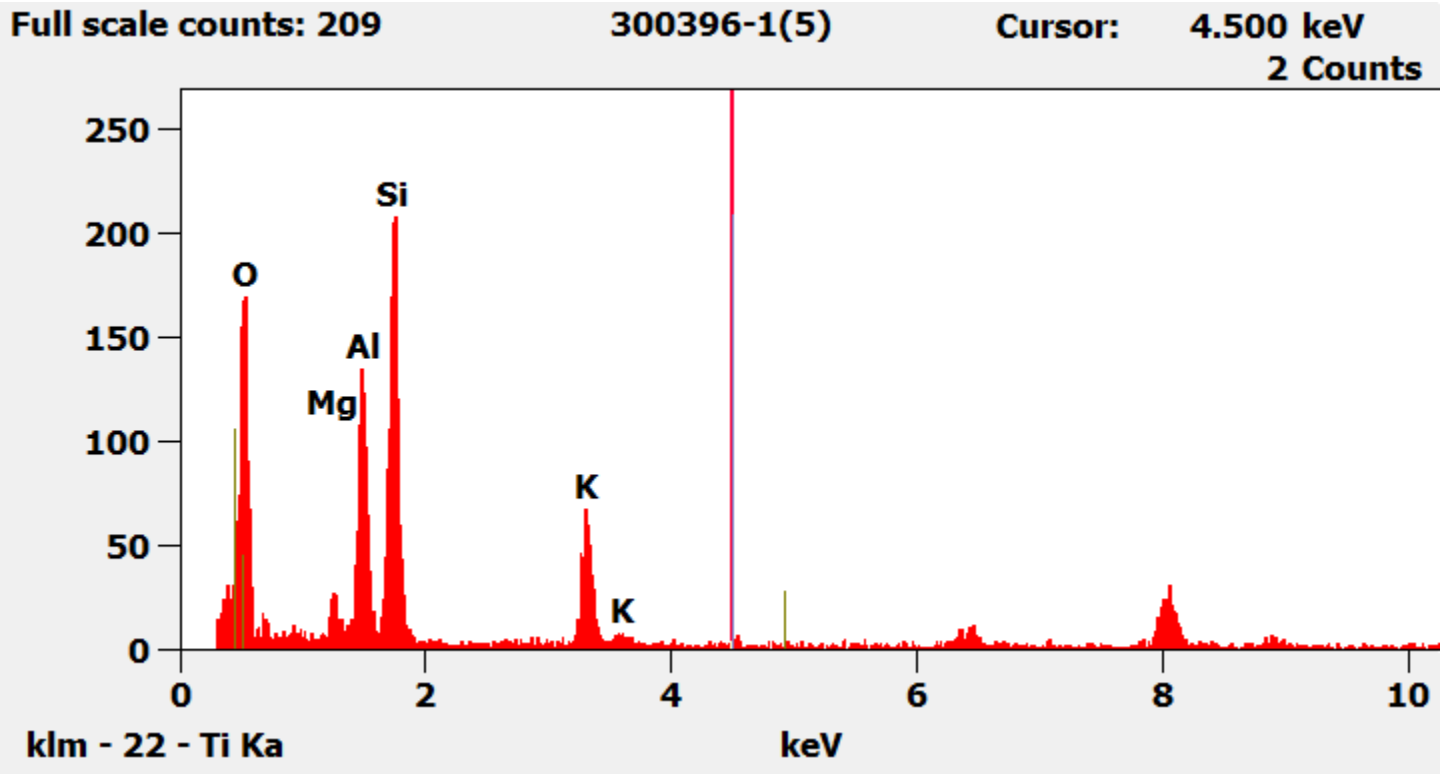
Sample 300396-1 Mica fiber



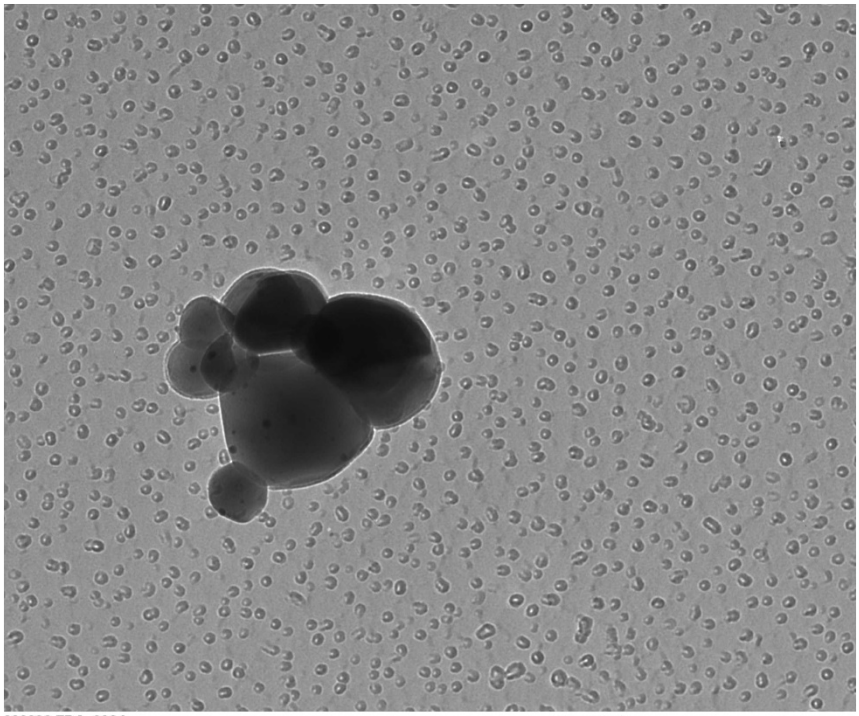
Sample 300396-1 Diffraction pattern from mica fiber pictured above.



Sample 300396-1 Chemistry from mica fiber pictured above



Sample 300396-1 Titanium particle



300396 FDA_006.jpg
Titaniaum Dioxide Particles
Spectra 10
Cal: 0.541520 nm/pix
09:59 4/2/2019
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

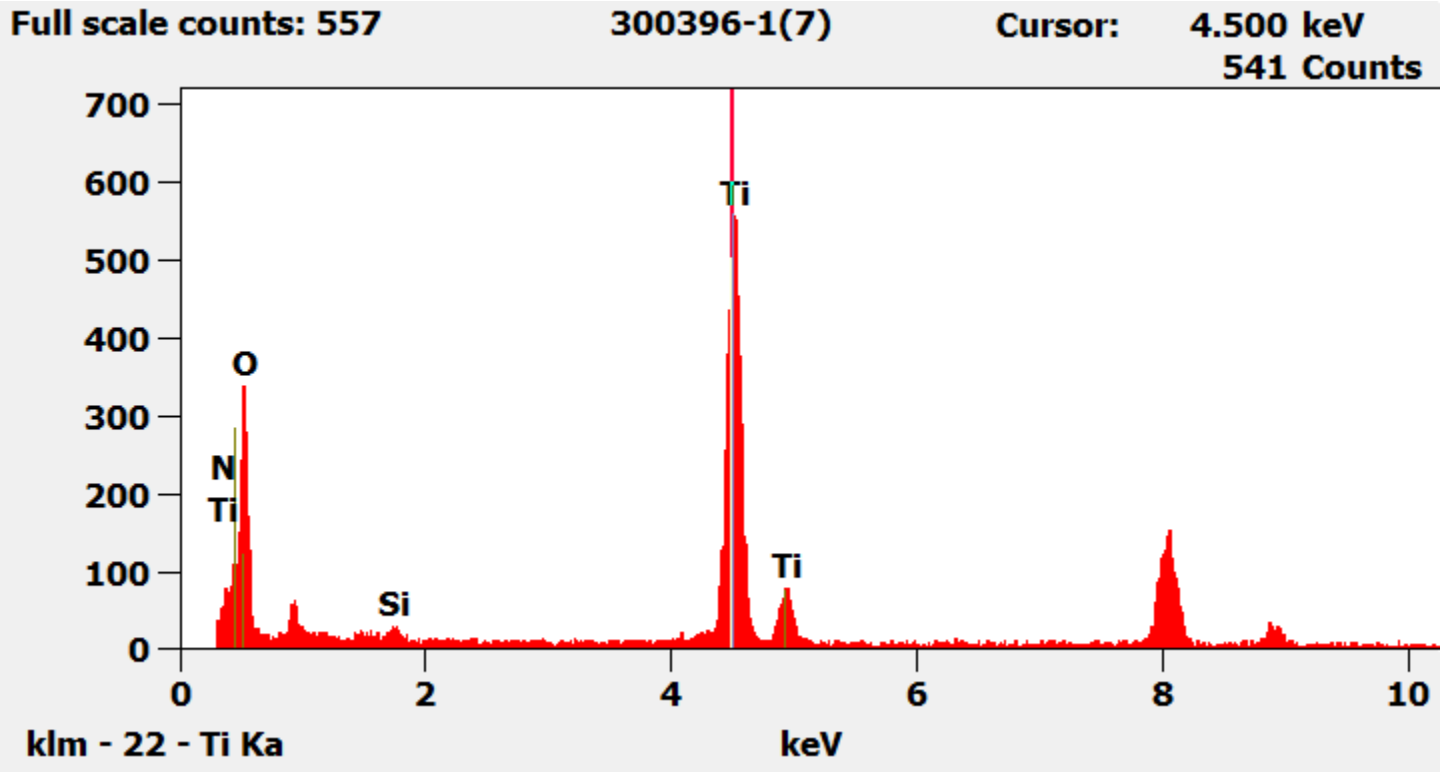
100 nm
HV=100kV
Direct Mag: 19000 x
AMA Analytical Services, Inc

Sample 300396-1 Diffraction pattern from titanium particle pictured above.

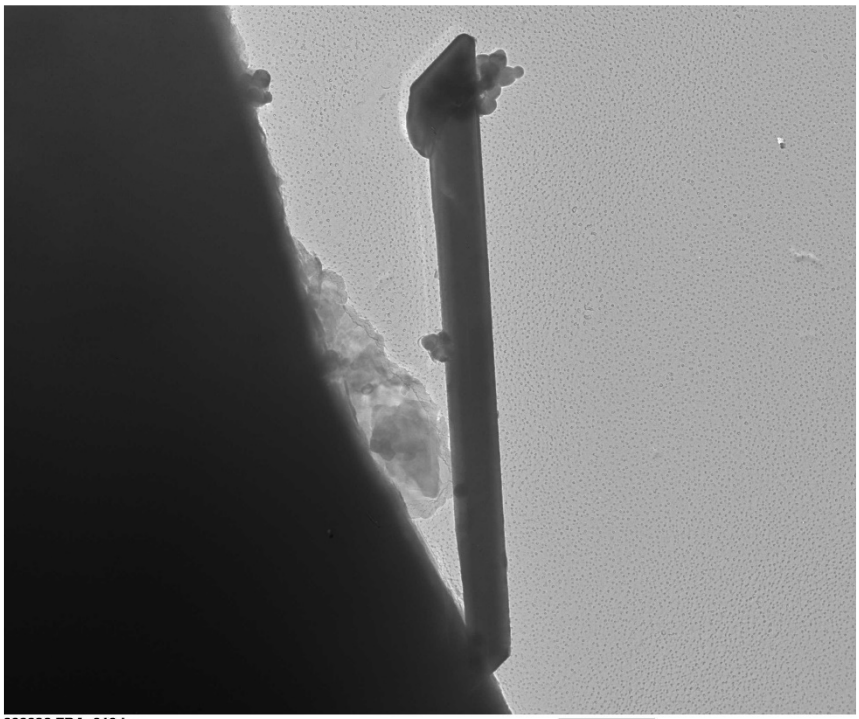
300396 FDA_007.jpg
Titaniaum Dioxide Diffraction
Spectra 10
10:02 4/2/2019
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Sample 300396-1 Chemistry from titanium particle pictured above.



Sample 300396-1 Titanium fiber

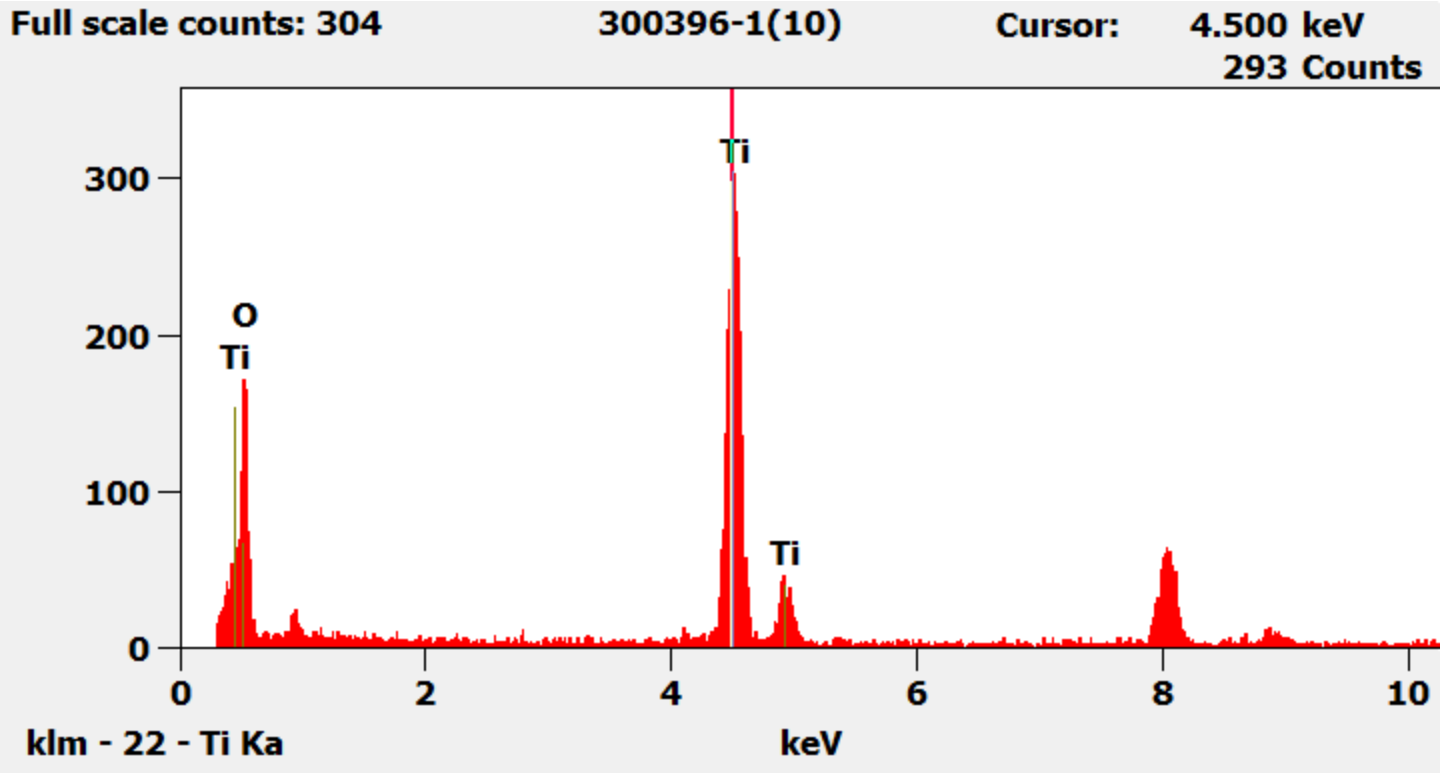


300396 FDA_010.jpg
Titanium Fiber
Spectra 17
Cal: 0.002858 µm/pix
10:16 4/2/2019
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Sample 300396-1 Diffraction pattern from titanium particle pictured above.

Sample 300396-1 Chemistry from titanium fiber pictured above.



300396-2, 2A, 2B, Client Sample D-33

PLM

All three aliquots of sample D-33 were analyzed by (b) (6) on March 29, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

300396-2	NAD
300396-2A	NAD
300396-2B	NAD

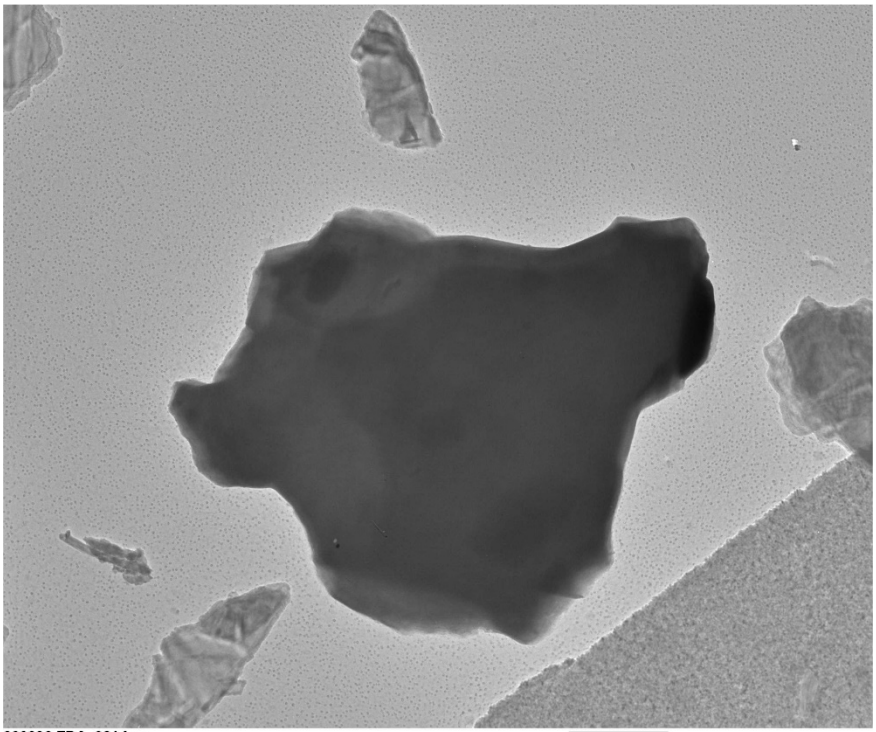
TEM

Sample 2 was analyzed by (b) (6) on April 4, 2019. (b) (6) analyzed sample 2A on April 12, 2019 and sample 2B on April 15, 2019. The primary particles observed were mica and titanium. Some talc flakes and ribbons were observed. No asbestos or non-asbestos amphibole variants were detected in the samples. The results were calculated using the equations detailed in the calculations section.

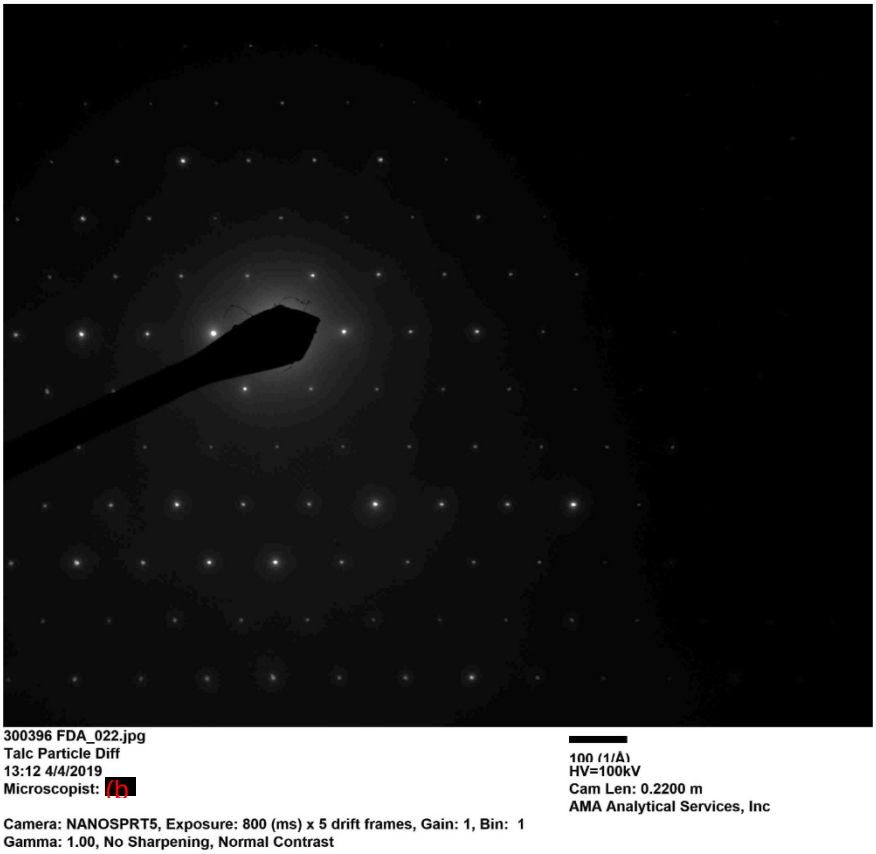
300396-2	NAD
300396-2A	NAD
300396-2B	NAD

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.

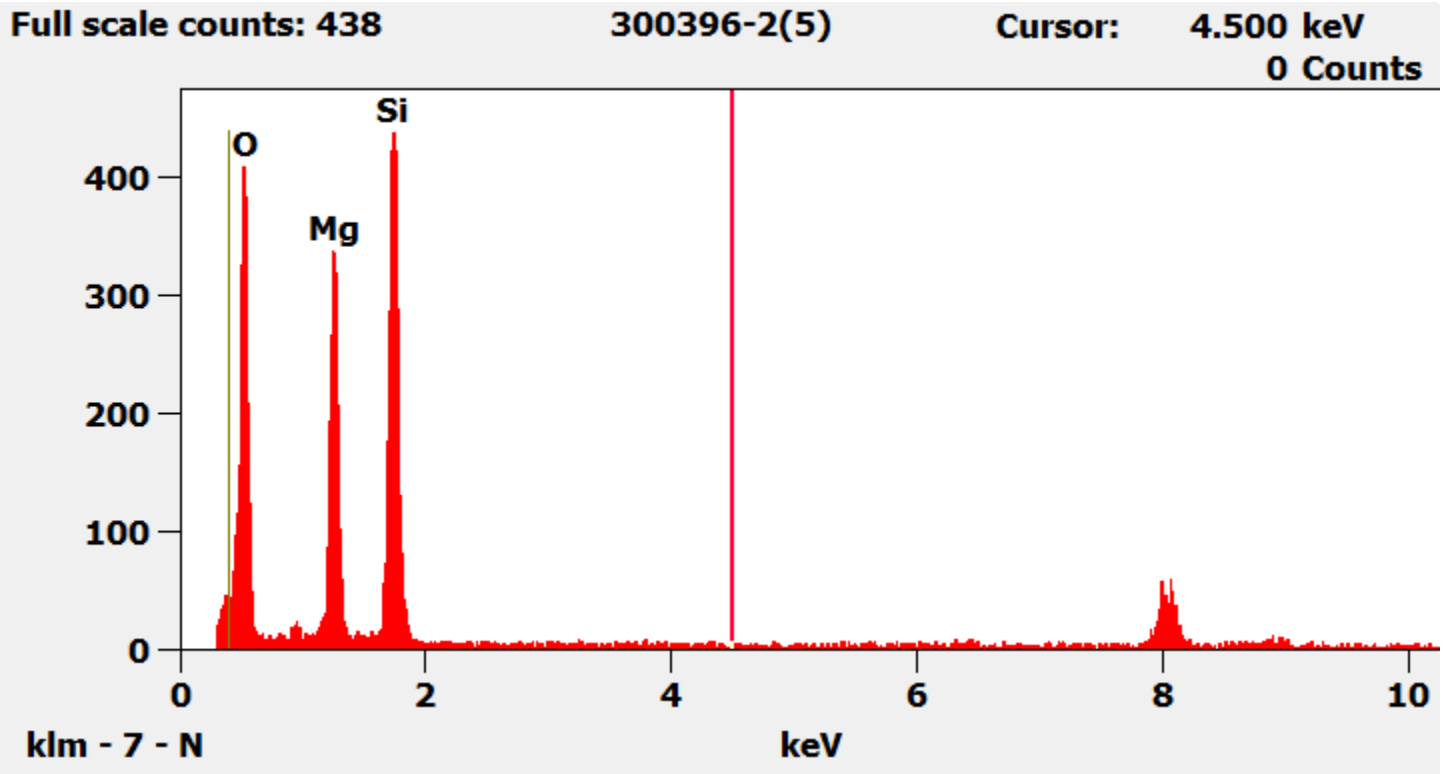
Sample 300396-2 Talc flake



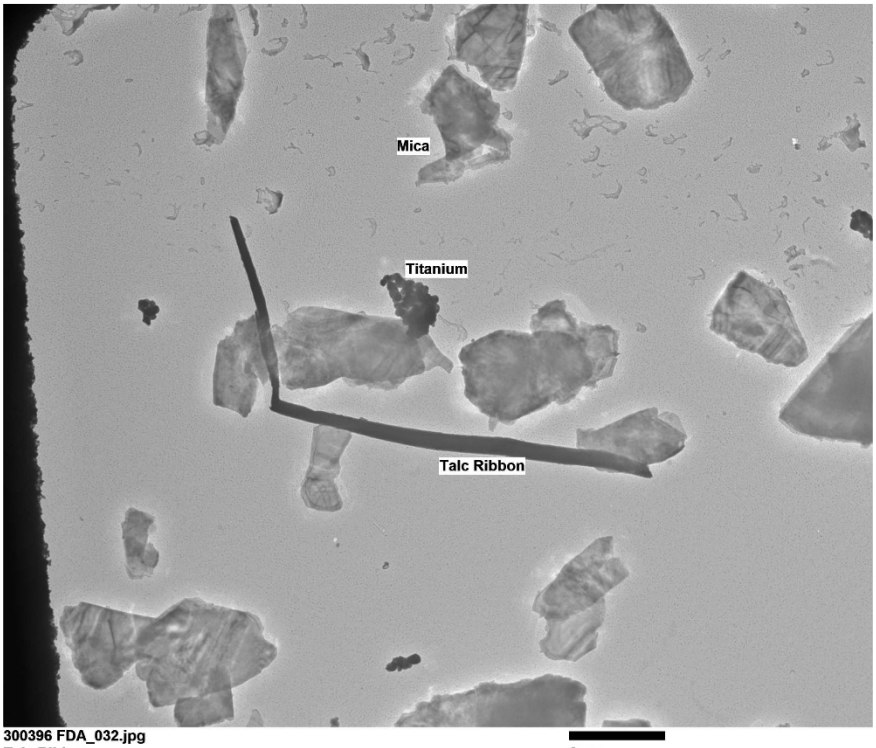
Sample 300396-2 Hexagonal diffraction from talc particle pictured above.



Sample 300396-2 Chemistry from talc particle pictured above



Sample 300396-2 Talc Ribbon



300396 FDA_032.jpg
Talc Ribbon
Cal: 0.007349 µm/pix
14:33 4/4/2019
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

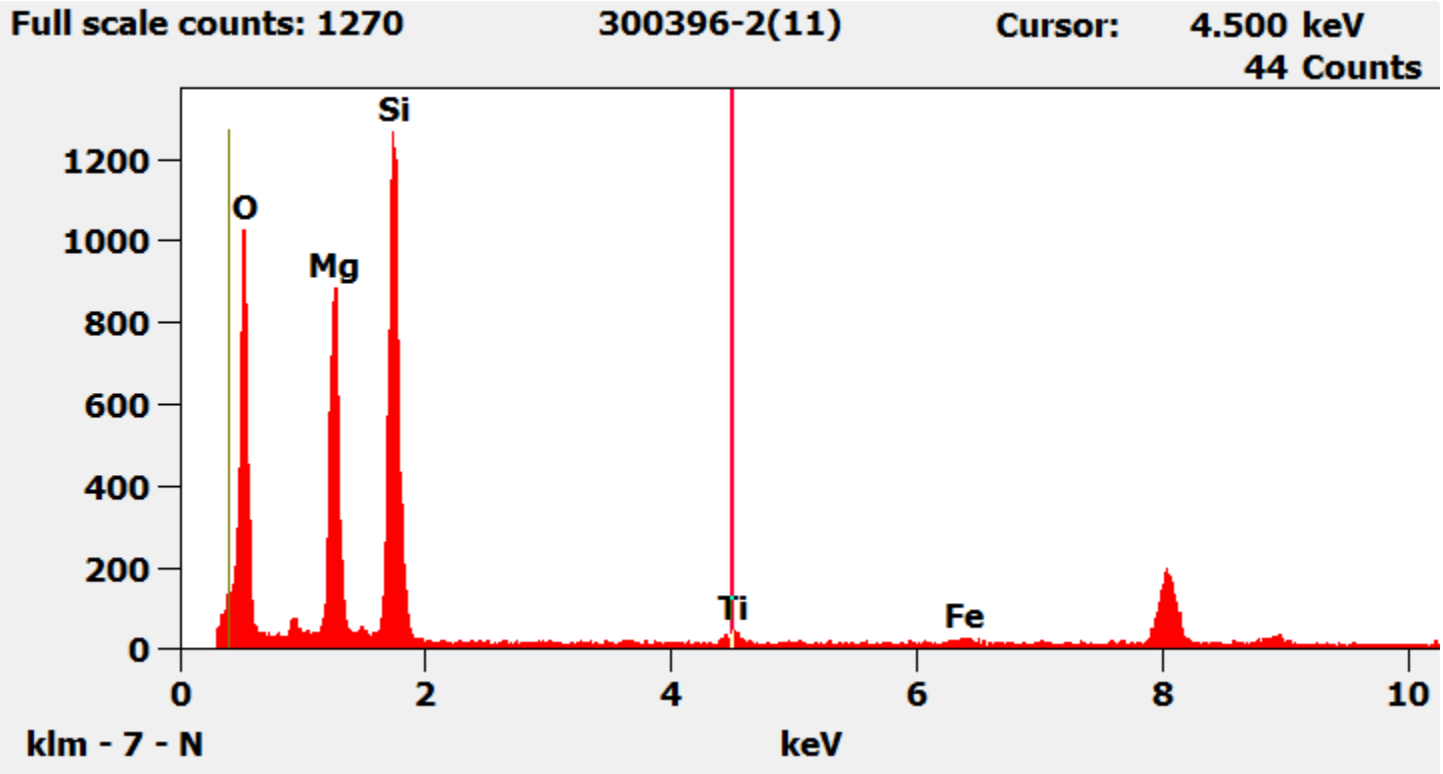
Sample 300396-2 Diffraction pattern from talc ribbon pictured above

300396 FDA_033.jpg
Talc Ribbon Diff
14:37 4/4/2019
Microscopist: (b)

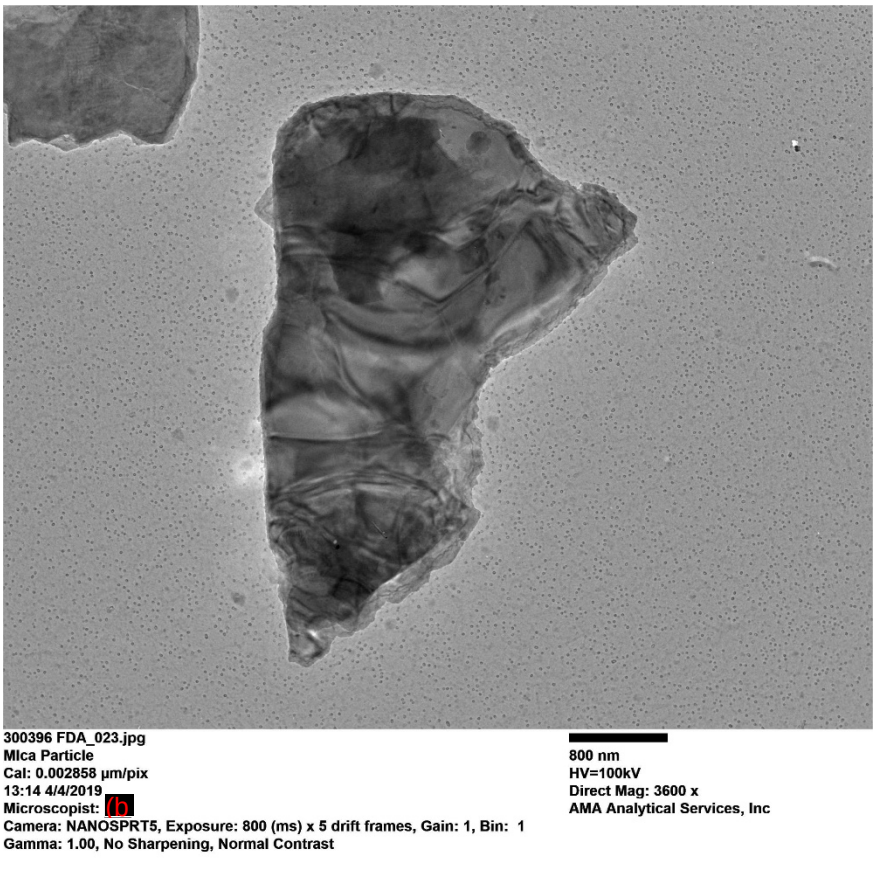
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

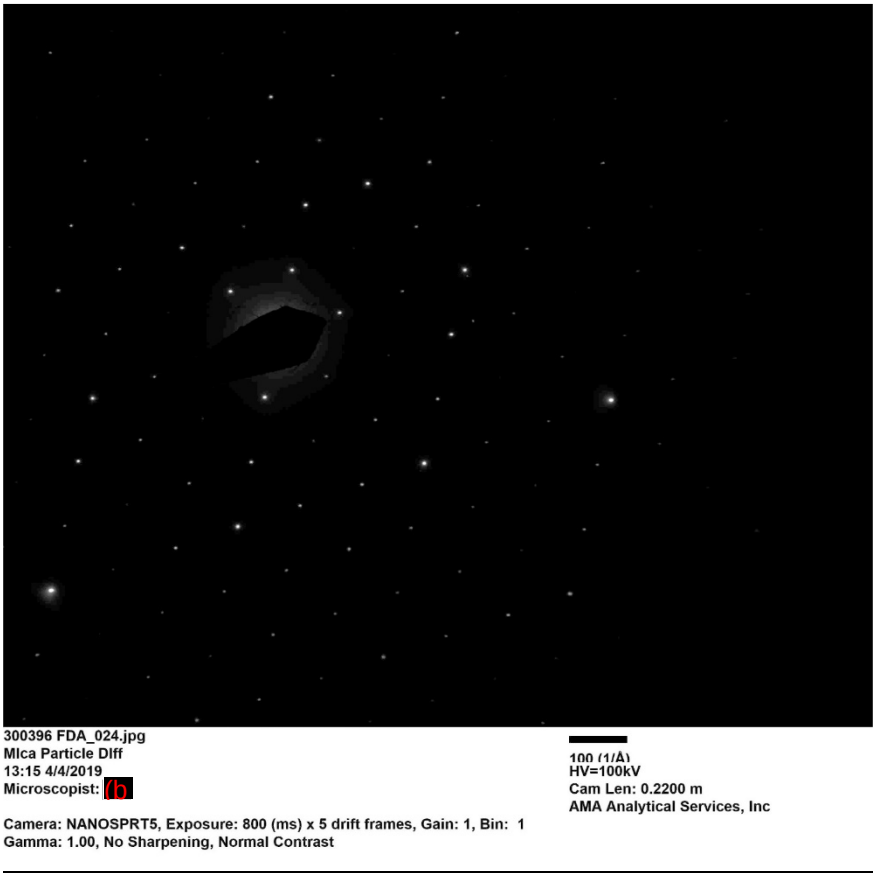
Sample 300396-2 Chemistry from talc ribbon pictured above



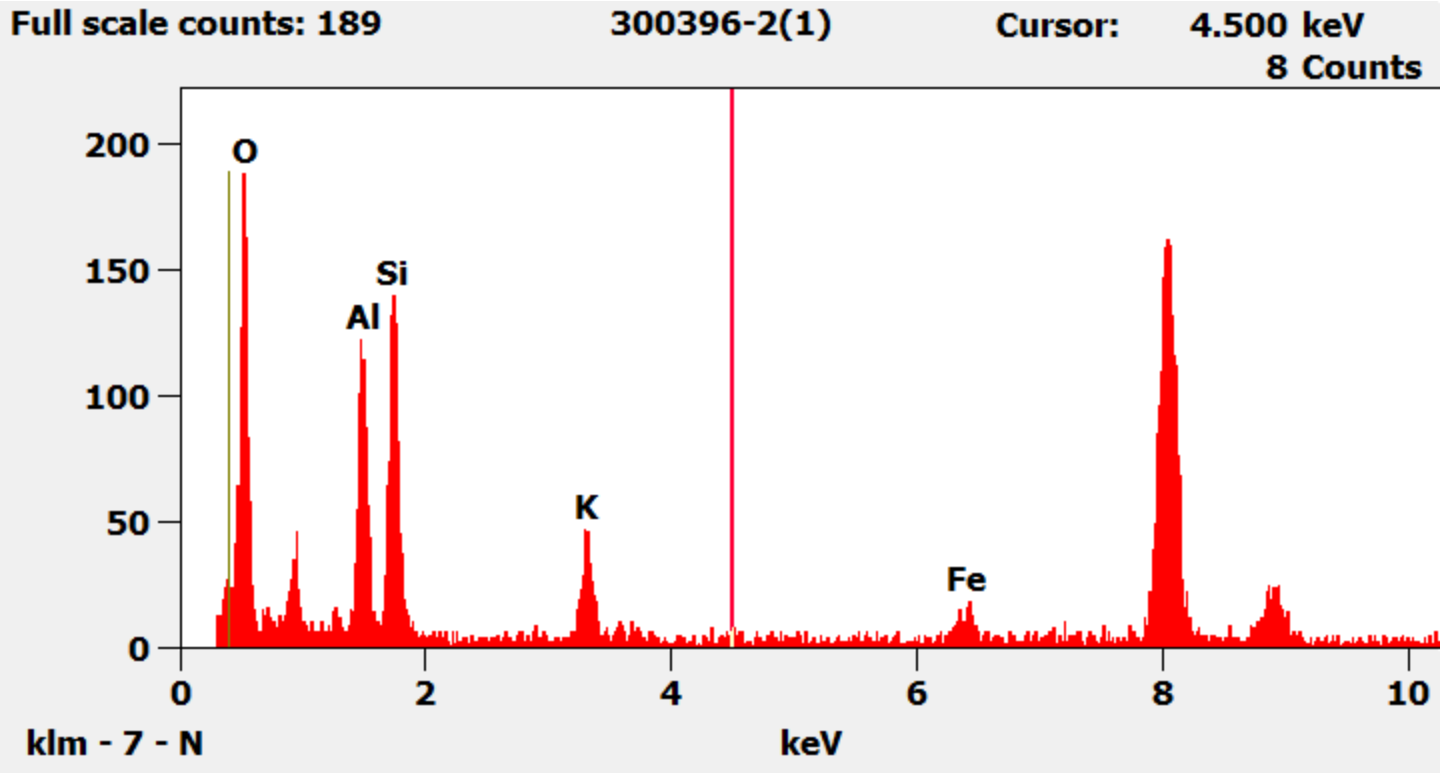
Sample 300396-2 Mica particle.



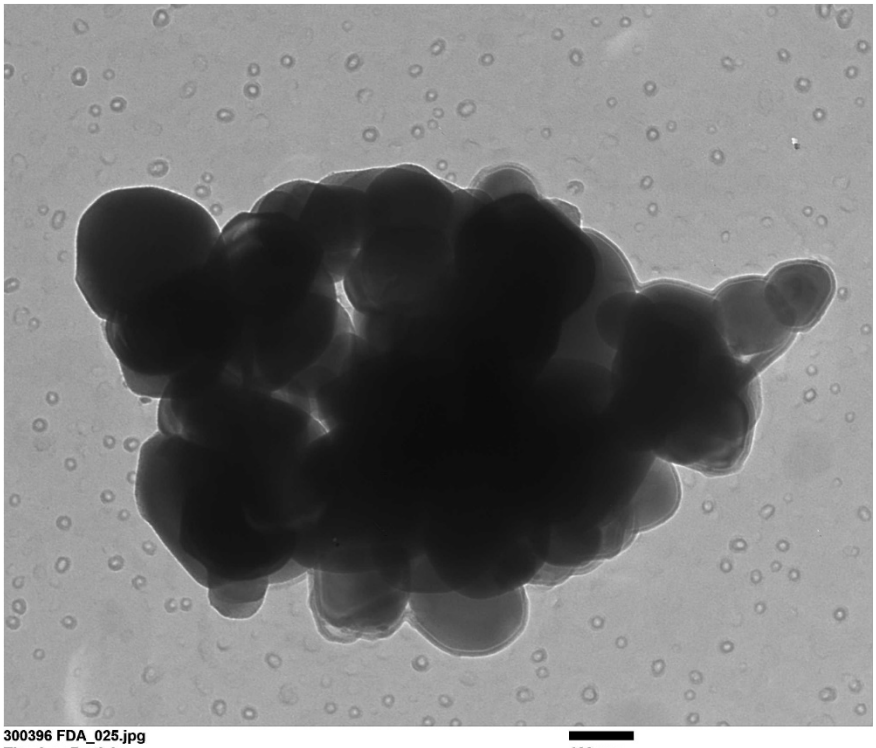
Sample 300396-2 Hexagonal diffraction pattern from mica particle pictured above.



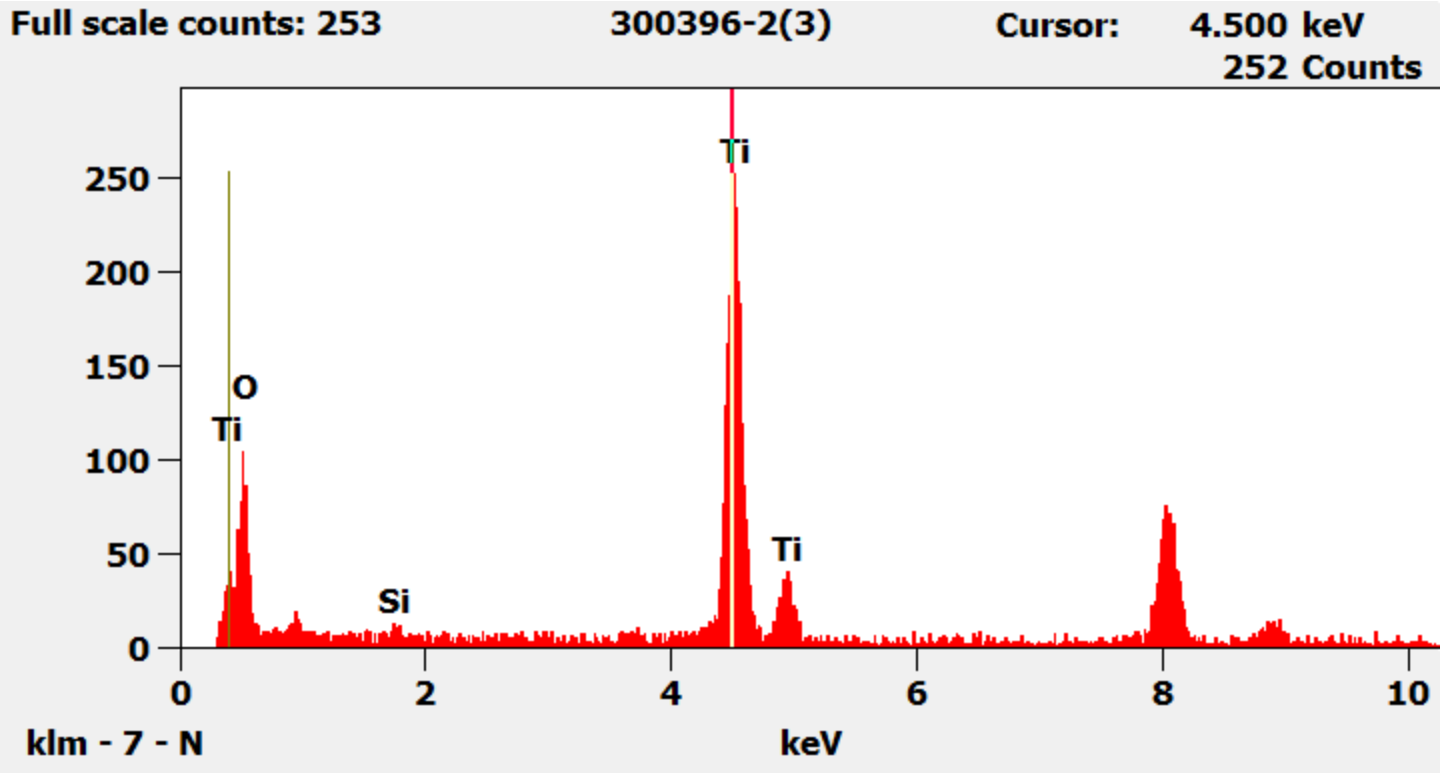
Sample 300396-2 Chemistry from mica particle pictured above.



Sample 300396-3 Titanium particle



Sample 300396-2 Chemistry from titanium particle pictured above.



300396-3, 3A, 3B, Client Sample D-34

PLM

All three aliquots of sample D-34 were analyzed by (b) (6) on March 29, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

300396-3	NAD
300396-3A	NAD
300396-3B	NAD

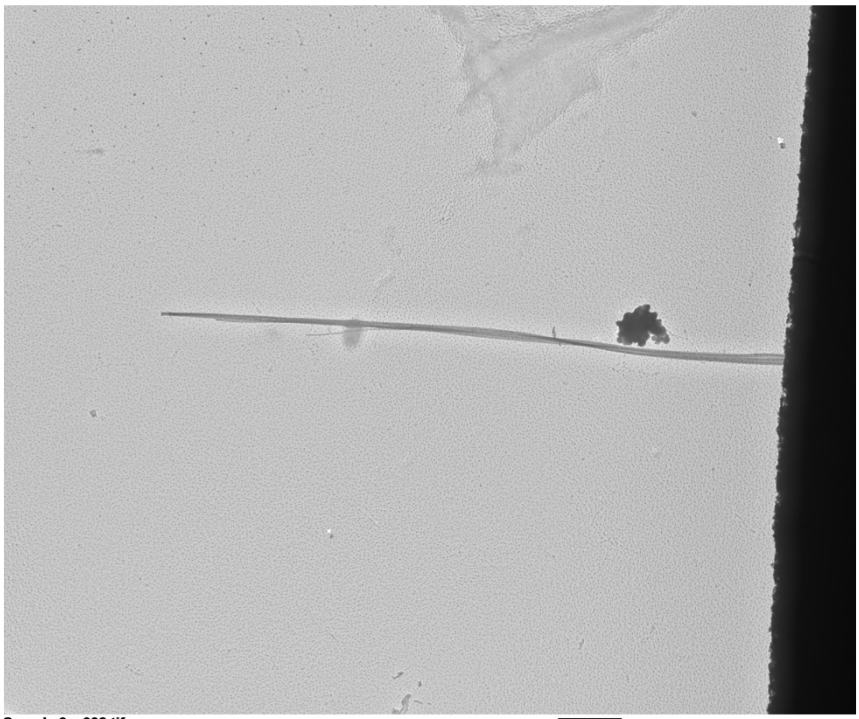
TEM

Sample 3 was analyzed by (b) (6) on April 4, 2019. (b) (6) analyzed sample 3A on April 15, 2019 and sample 3B on April 16, 2019. All three samples contained mica, talc, and titanium particles. One 9.9 x 0.15 micron chrysotile bundle was counted on sample 3A. Five (5) chrysotile structures were counted on sample 3B. The results were calculated using the equations detailed in the calculations section.

300396-3	NAD
300396-3A	< 0.00080%
300396-3B	0.00030%

Below are pictures, diffraction patterns, and chemistry of the counted Chrysotile particles. The mica, talc, and titanium particles are similar to those pictured in samples 1 and 2. The unidentified peaks in chemistry spectra are copper, zinc, and carbon. Those peaks are from the TEM specimen holder and specimen grid.

Sample 300396-3A Chrysotile structure

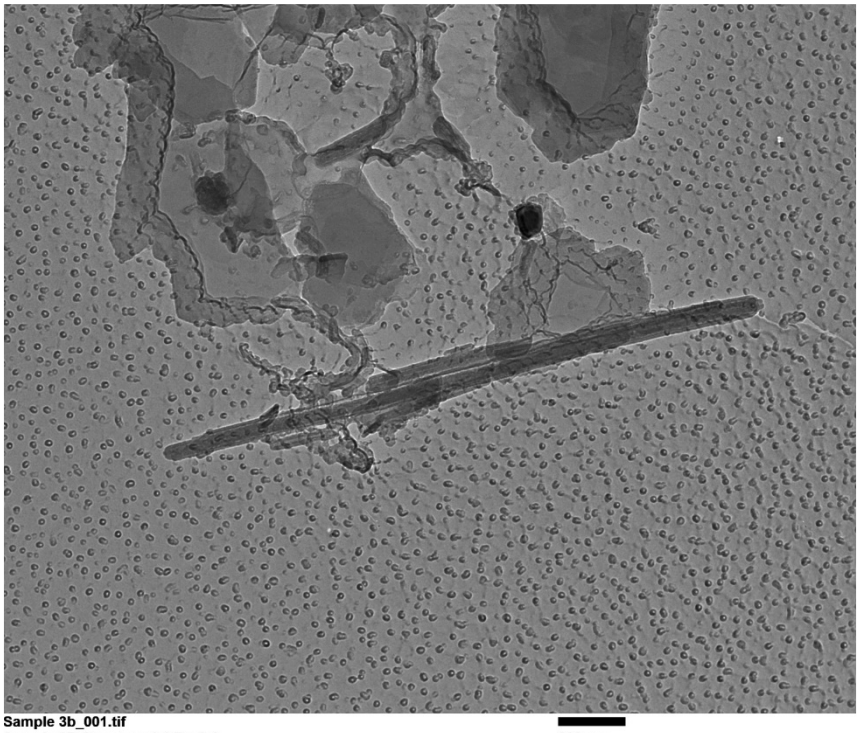


Sample 300396-3A Diffraction pattern from the chrysotile structure pictured above.

Sample 3a_001.tif
Sample 3a Structure 1
Chrysotile Diffraction
09:58 4/18/2019
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

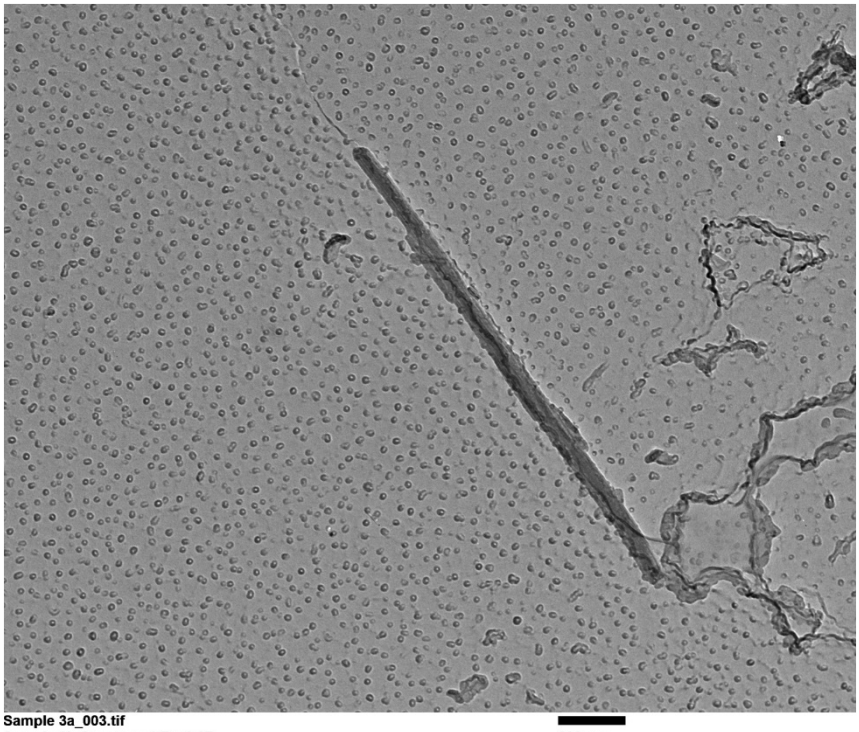
Sample 300396-3B Chrysotile structure #1



Sample 3b_001.tif
Sample 3B Structure 1 1.7 x 0.1
Chrysotile
Cal: 0.001029 µm/pix
10:16 4/18/2019
Microscopist: (b) (6)
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

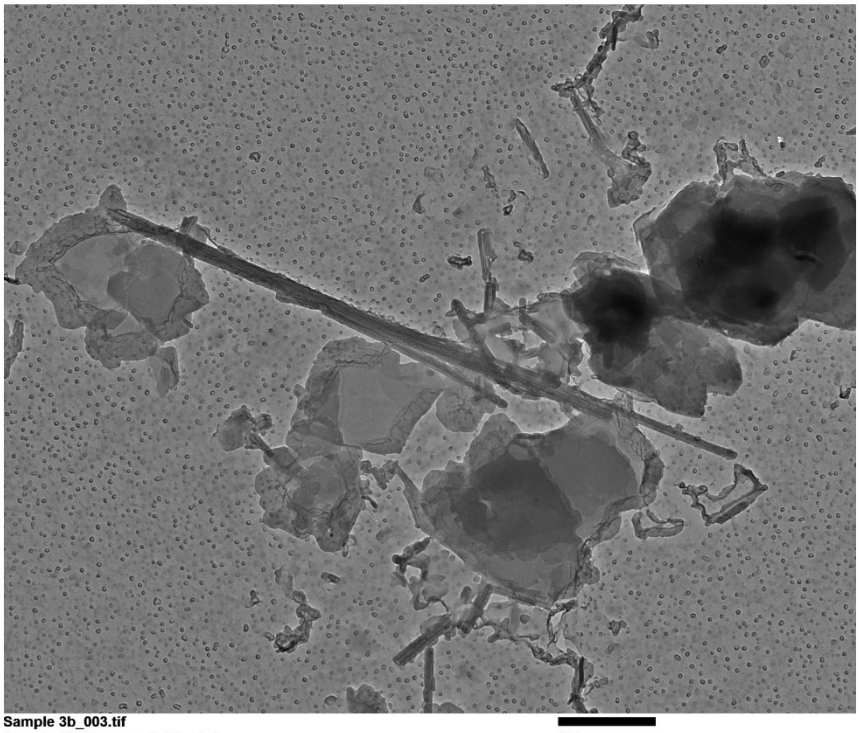
Sample 300396-3B Chrysotile structure #2



Sample 3a_003.tif
Sample 3b Structure 1.7 x 0.07
Chrysotile
Cal: 0.001029 µm/pix
10:09 4/18/2019
Microscopist: (b) (6)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 std. frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Sample 300396-3B Chrysotile structure #3



Sample 3b_003.tif
Sample 3B Structure 3 3.9 x 0.1
Chrysotile
Cal: 0.001774 µm/pix
10:24 4/18/2019
Microscopist: (b) (6)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

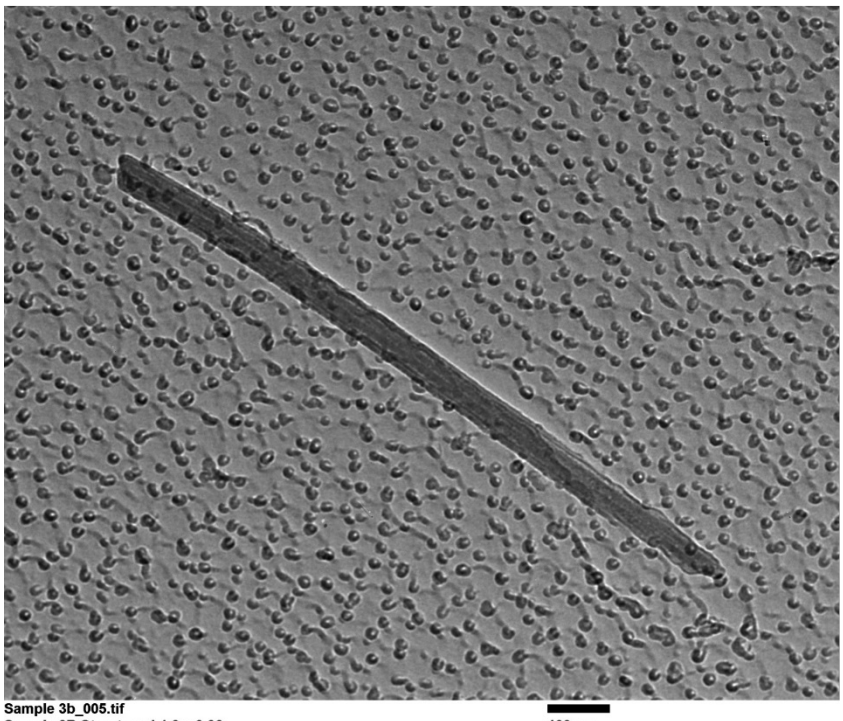
500 nm
HV=100kV
Direct Mag: 5800 x
AMA Analytical Services, Inc

Sample 300396-3B Chrysotile diffraction pattern from structure #3

Sample 3b_002.tif
Sample 3B Structure 3 3.9 x 0.1
Chrysotile Diffraction
10:21 4/18/2019
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

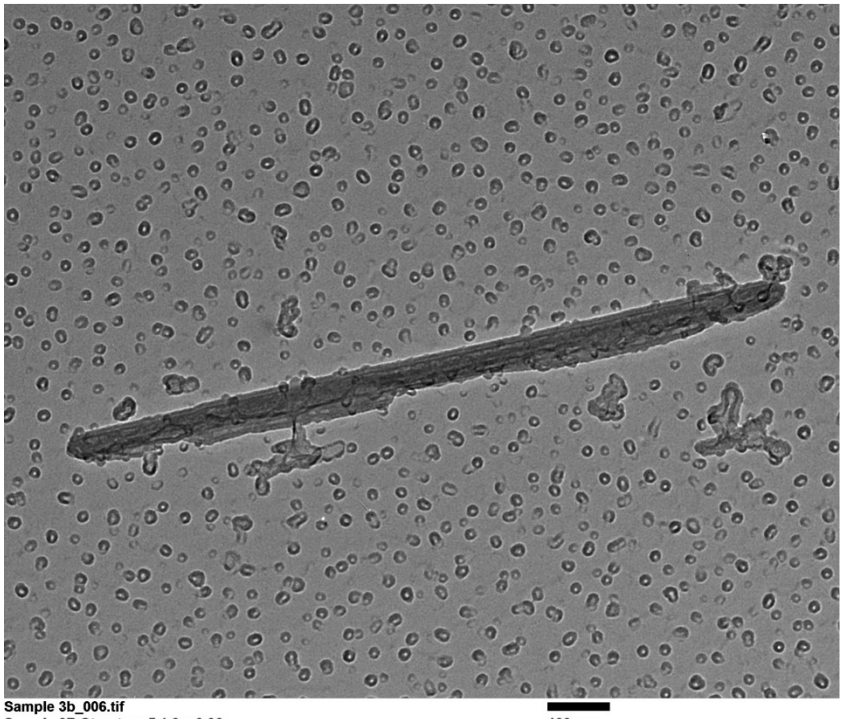
Sample 300396-3B Chrysotile structure #4



Sample 3b_005.tif
Sample 3B Structure 4 1.3 x 0.06
Chrysotile
Cal: 0.541520 nm/pix
10:31 4/18/2019
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

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

Sample 300396-3B Chrysotile structure #5



300396-4, 4A, 4B, Client Sample D35

PLM

All three aliquots of sample D35 were analyzed by (b) (6) on March 29, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

300396-4	NAD
300396-4A	NAD
300396-4B	NAD

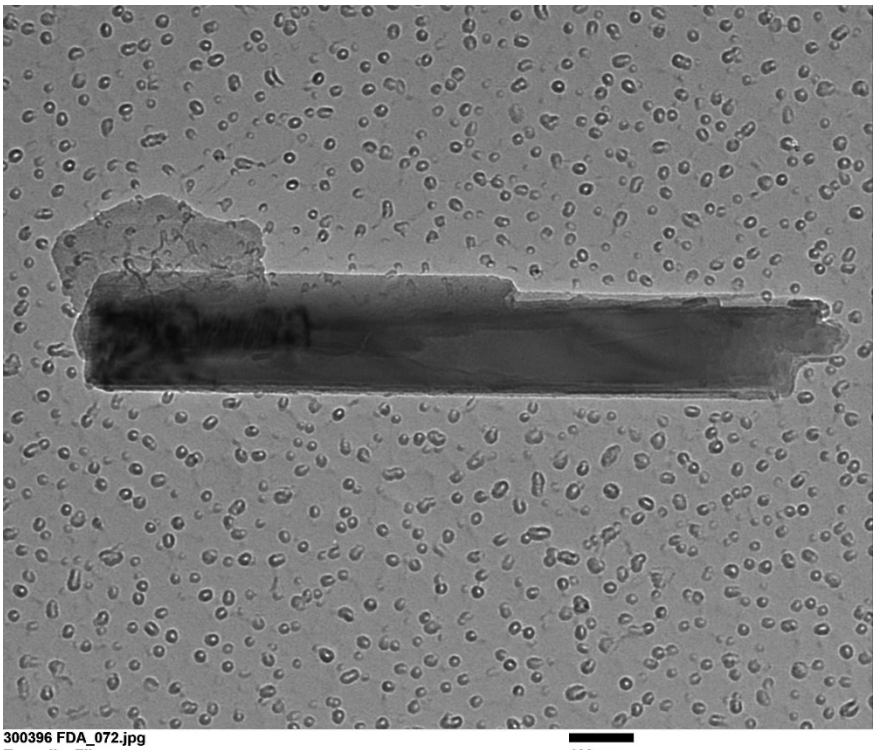
TEM

Samples 4 and 4A were analyzed by (b) (6) on April 11, 2019. He analyzed sample 4B on April 16, 2019. The primary particles observed were mica and talc. Some titanium particles were observed. The talc was mostly flakes but there were also some ribbons and fibers. Tremolite and chrysotile were observed and counted on all three samples. Two tremolite and two chrysotile structures were counted on sample 4. One tremolite and two chrysotile structures were counted on sample 4A. One tremolite and five chrysotile structures were counted on sample 4B. The results were calculated using the equations detailed in the calculations section.

300396-4	0.00574%
300396-4A	< 0.00013%
300396-4B	0.00371%

Below are pictures, diffraction patterns, and chemistry of the counted tremolite and chrysotile particles. Some of the talc fibers and ribbons are also pictured below. The unidentified peaks in chemistry spectra are copper, zinc, and carbon. Those peaks are from the TEM specimen holder and specimen grid.

Sample 300396-4 Tremolite fiber #1

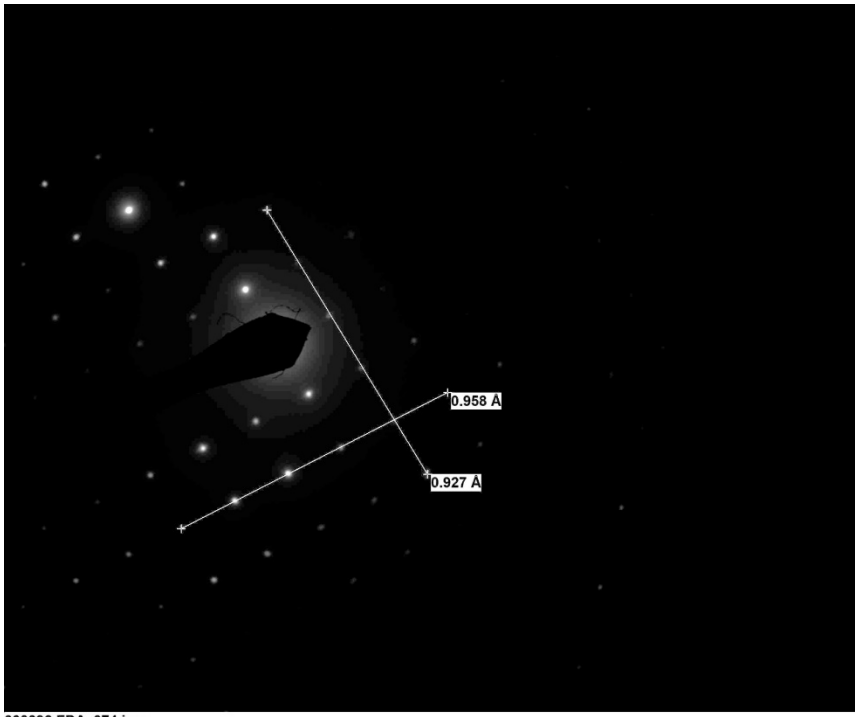


300396 FDA_072.jpg
Tremolite Fiber
Cal: 0.541520 nm/pix
10:44 4/11/2019

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

Sample 300396-4 Zone axis diffraction pattern from tremolite fiber #1 pictured above.

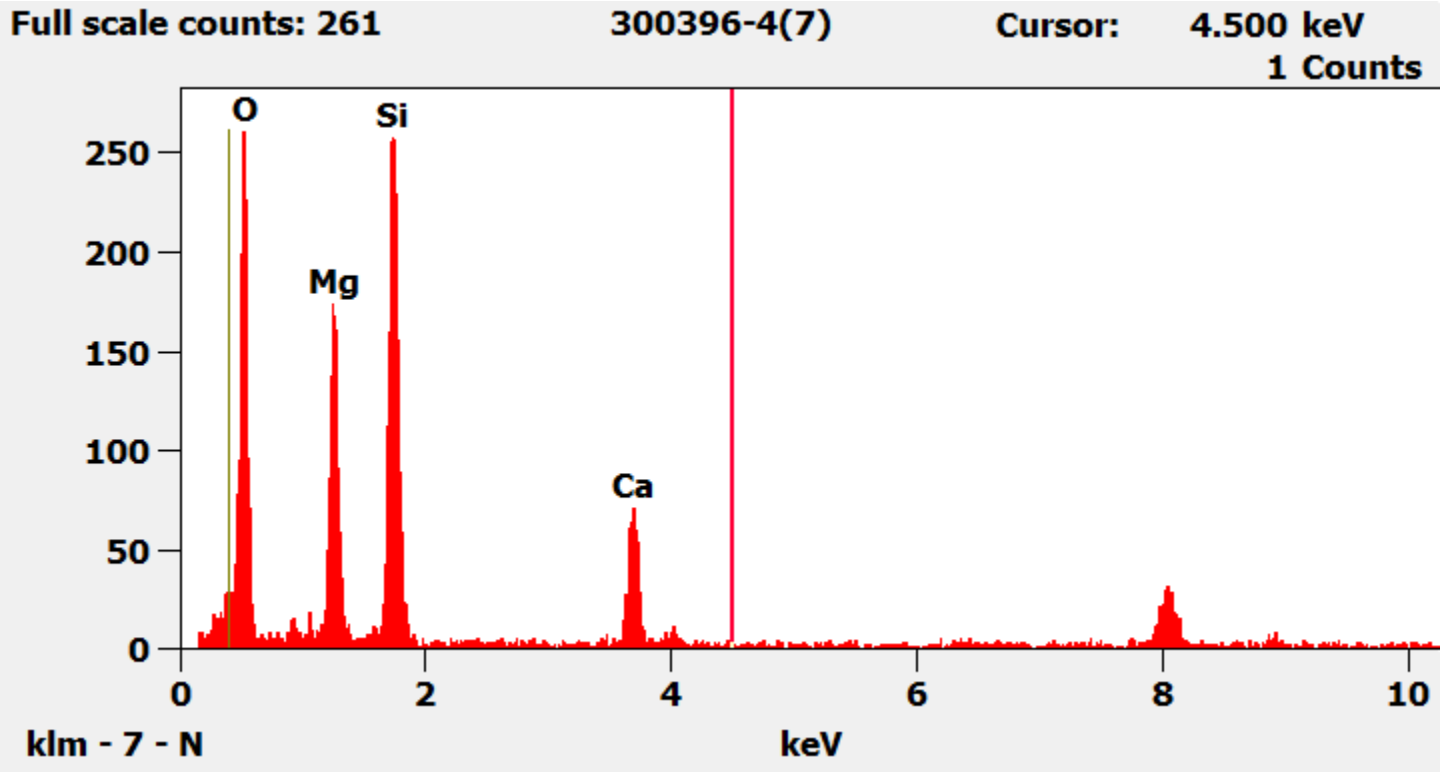


300396 FDA_074.jpg
Tremolite Fiber Zone Axis
[0 2 -2]
10:49 4/11/2019

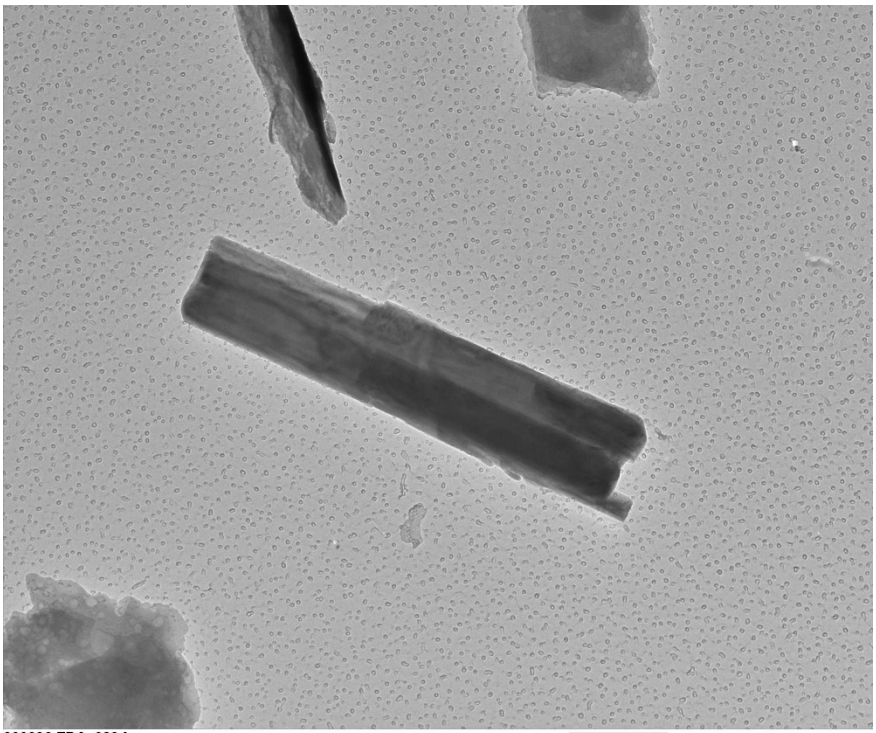
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Sample 300396-4 Chemistry from tremolite fiber #1



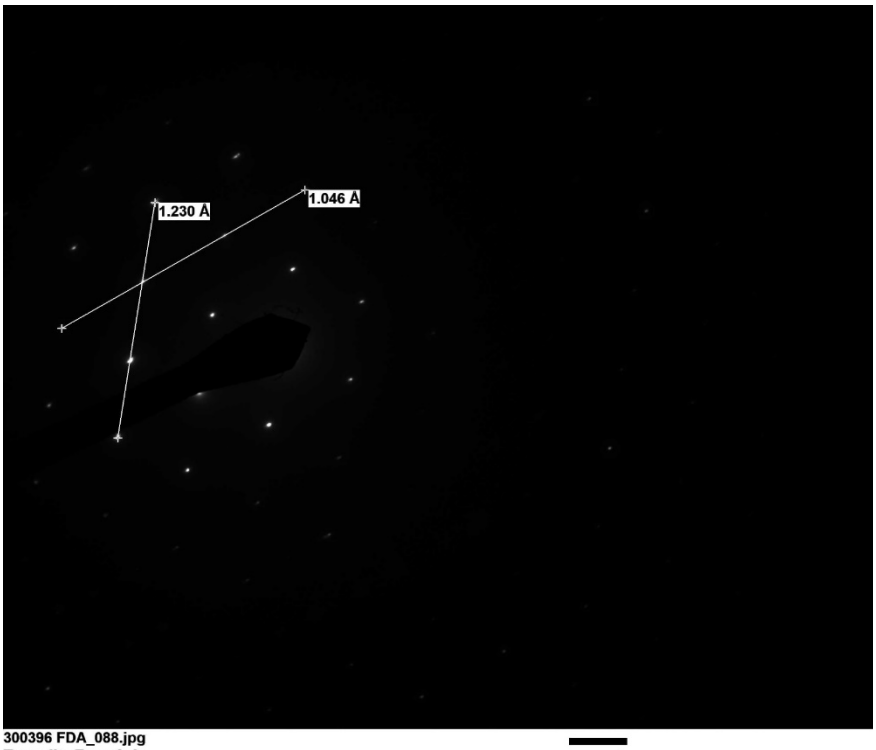
Sample 300396-4 Tremolite fiber 2



300396 FDA_089.jpg
Tremolite Fiber
[-1 1 -4]
Cal: 0.001429 µm/pix
12:34 4/11/2019
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

Sample 300396-4 Diffraction patterns from the tremolite fiber #2 pictured above.



300396 FDA_088.jpg
Tremolite Zone Axis
[-1 1 -4]
12:28 4/11/2019

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

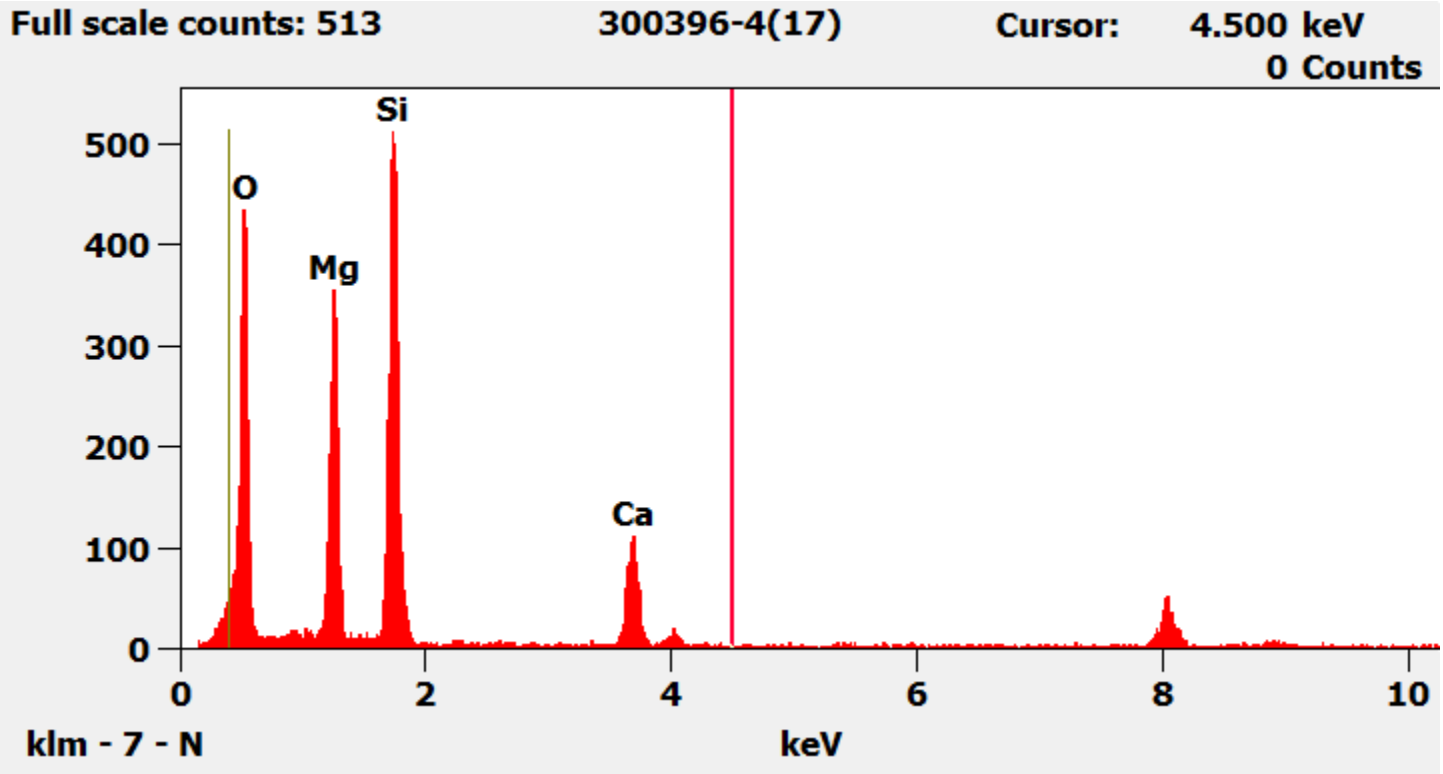
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

300396 FDA_087.jpg
Tremolite Diff
12:23 4/11/2019

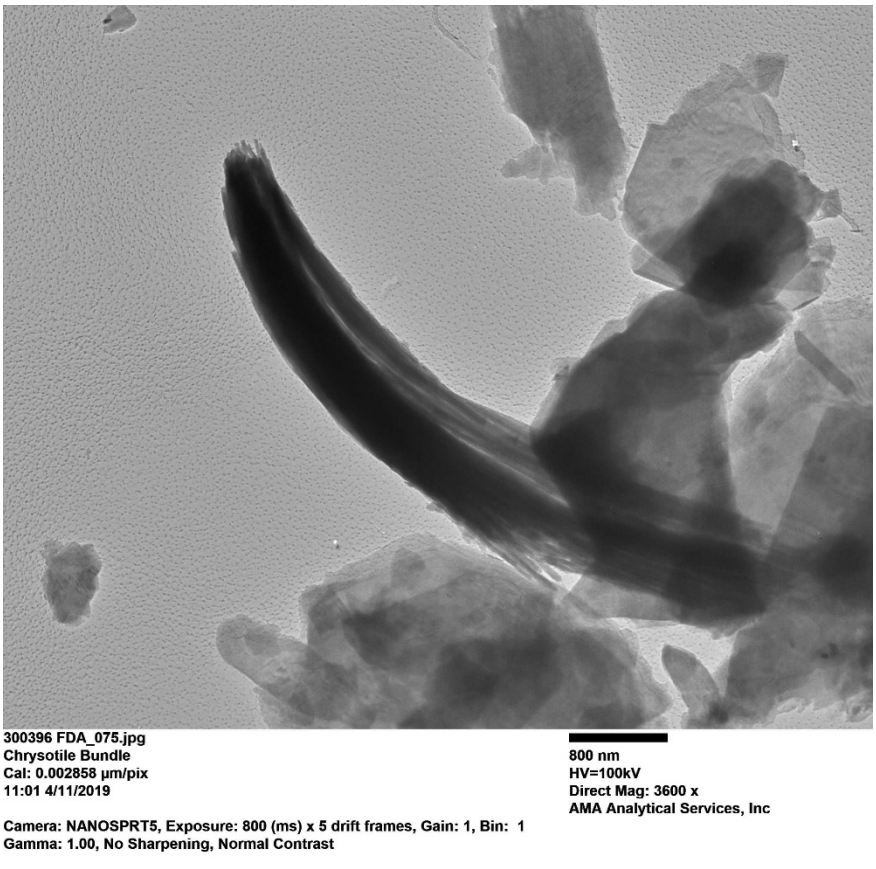
100 (1/Å)
HV=100kV
Cam Len: 0.2200 m
AMA Analytical Services, Inc

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

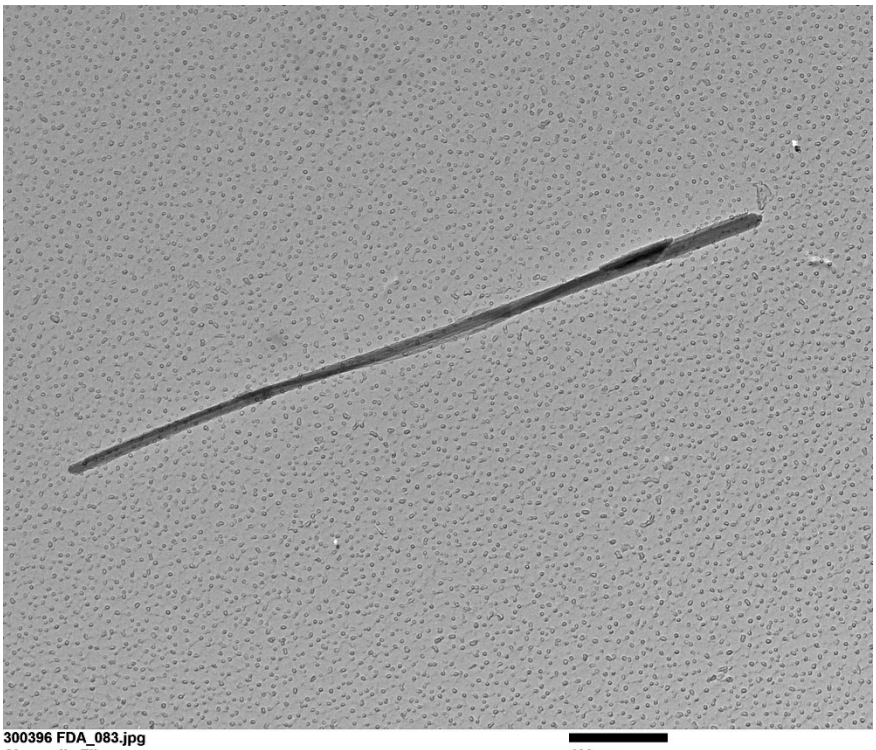
Sample 300396-4 Chemistry from tremolite fiber pictured above.



Sample 300396-4 Chrysotile structure #1



Sample 300396-4 Chrysotile structure #2

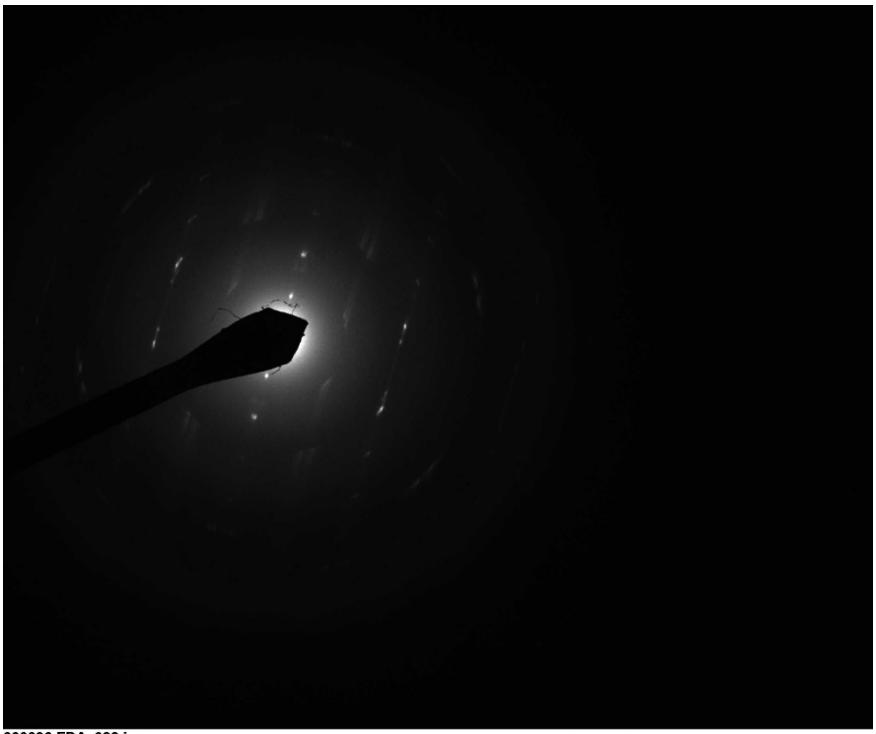


300396 FDA_083.jpg
Chrysotile Fiber
Cal: 0.001429 µm/pix
11:57 4/11/2019

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

Sample 300396-4 Diffraction pattern from chrysotile structure #2 pictured above

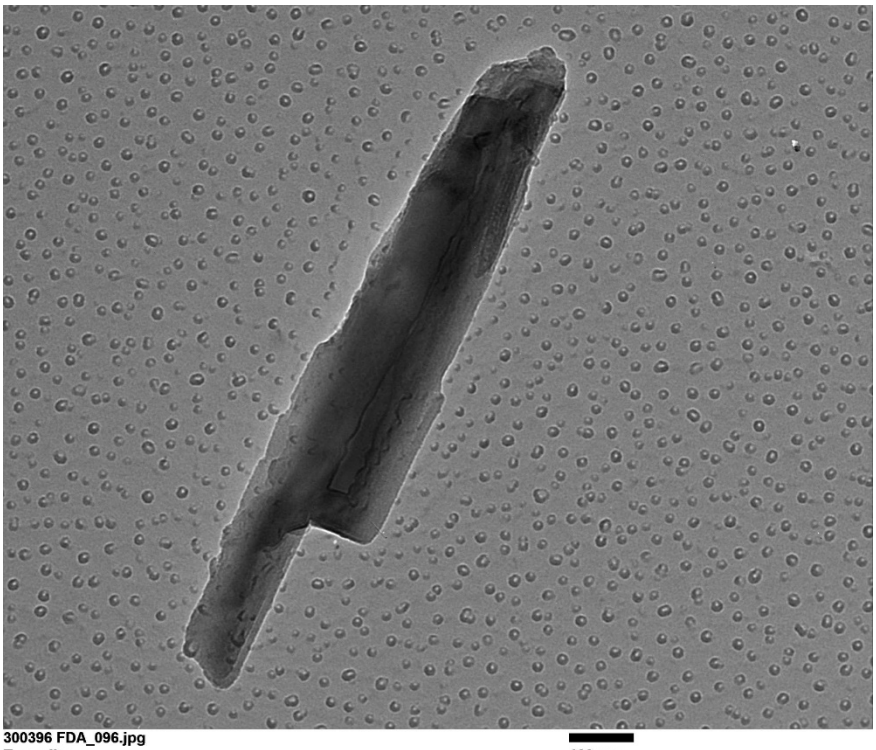


300396 FDA_082.jpg
Chrysotile Diffraction 2
11:46 4/11/2019

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

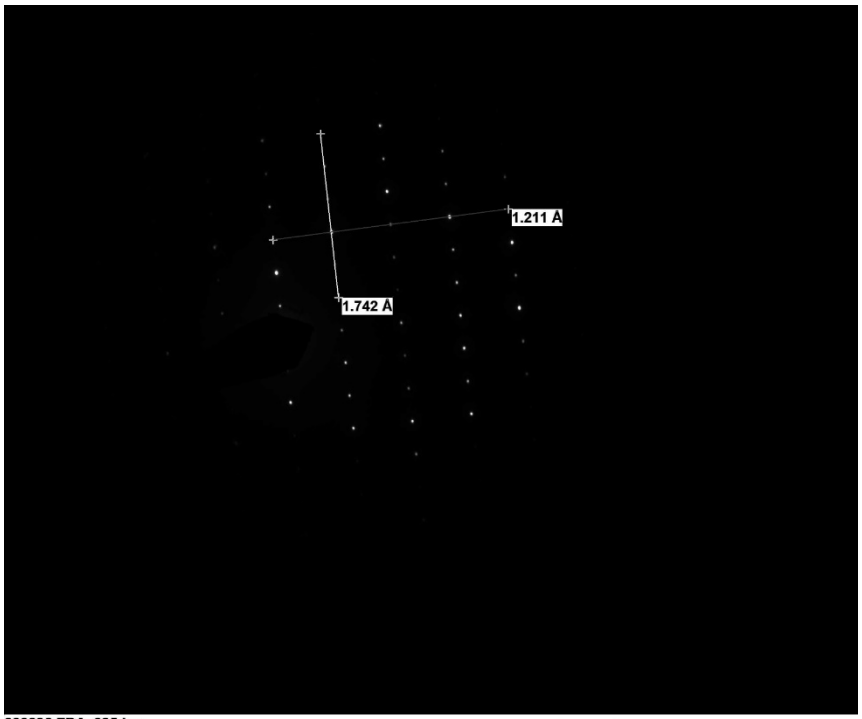
Sample 300396-4A Tremolite fiber



300396 FDA_096.jpg
Tremolite
[0 0 -4]
Cal: 0.541520 nm/pix
15:53 4/11/2019
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

Sample 300396-4A Diffraction pattern from tremolite fiber pictured above.

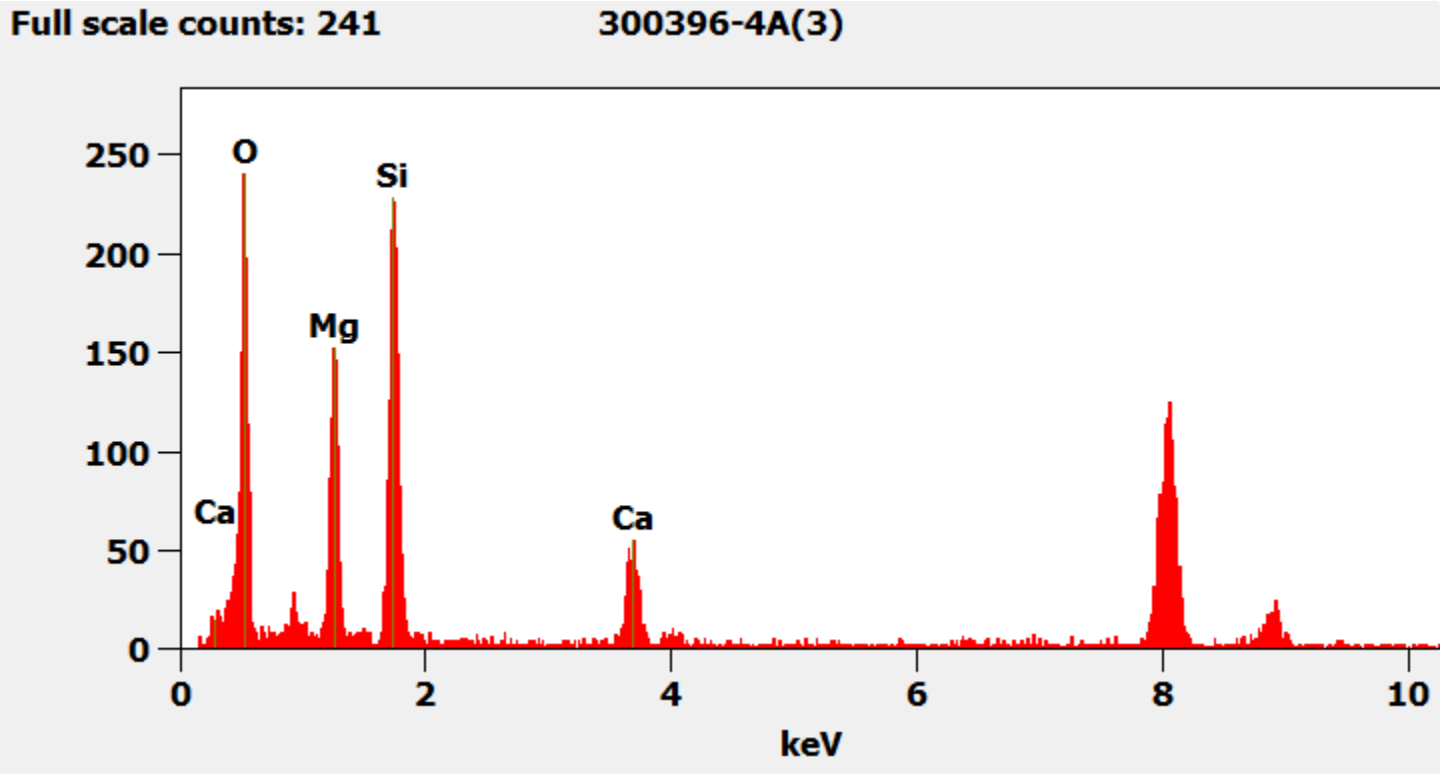


300396 FDA_095.jpg
Tremolite Zone Axis
[0 0 -4]
15:44 4/11/2019

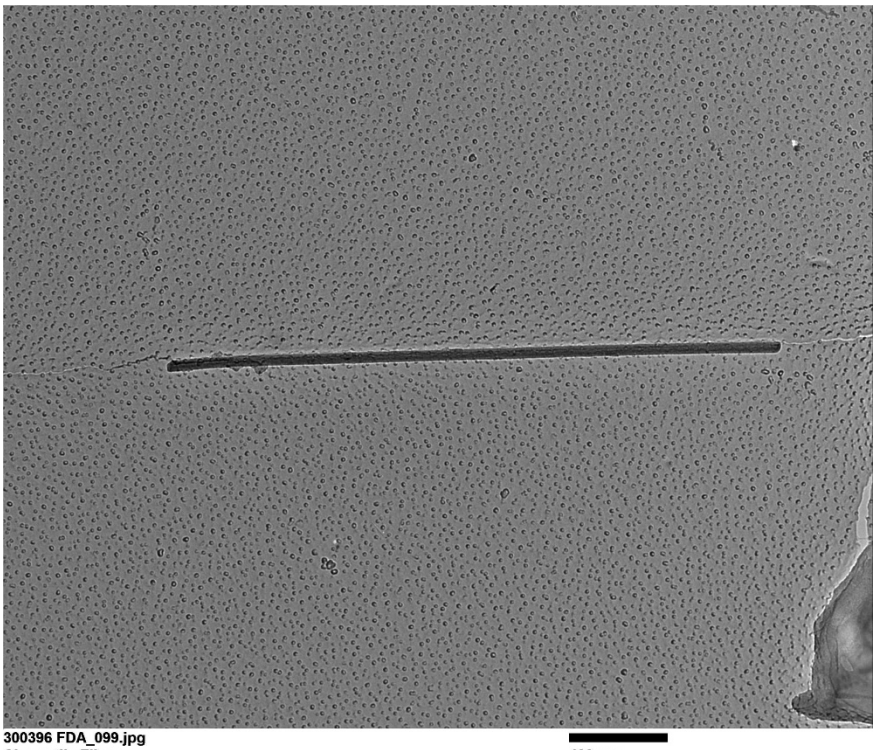
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

100 (1/Å)
HV=100kV
Cam Len: 0.2200 m
Tilt:25
AMA Analytical Services, Inc

Sample 300396-4A Chemistry from tremolite fiber pictured above



Sample 300396-4A Chrysotile structure #1



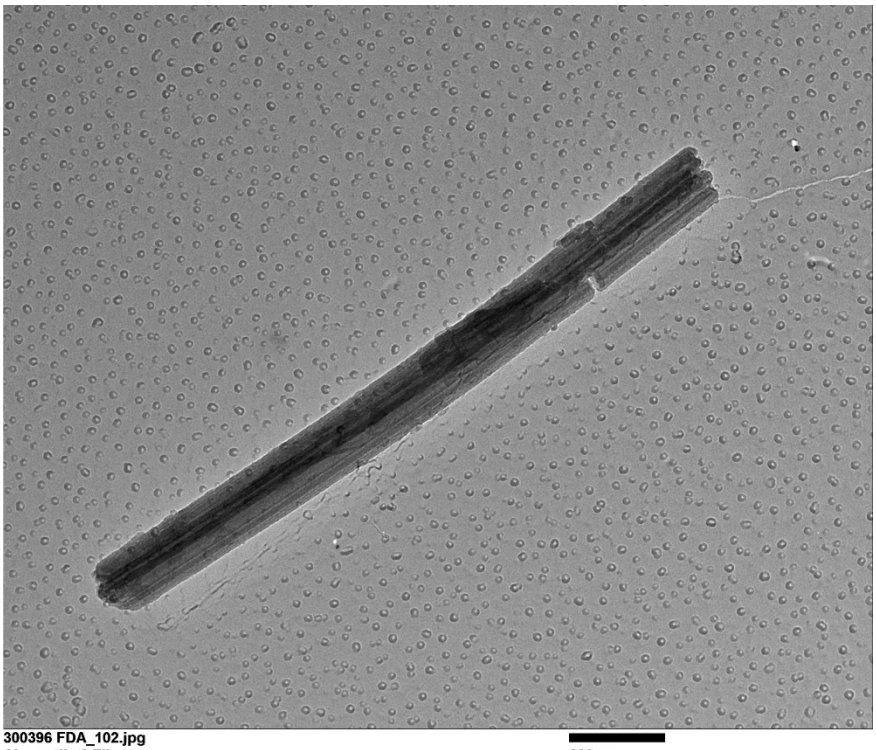
Sample 300396-4A Diffraction pattern from chrysotile structure #1 pictured above

300396 FDA_097.jpg
Chrysotile
16:07 4/11/2019

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

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

Sample 300396-4A Chrysotile structure #2



300396 FDA_102.jpg
Chrysotile 2 Fiber
Cal: 0.734921 nm/pix
16:27 4/11/2019

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

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

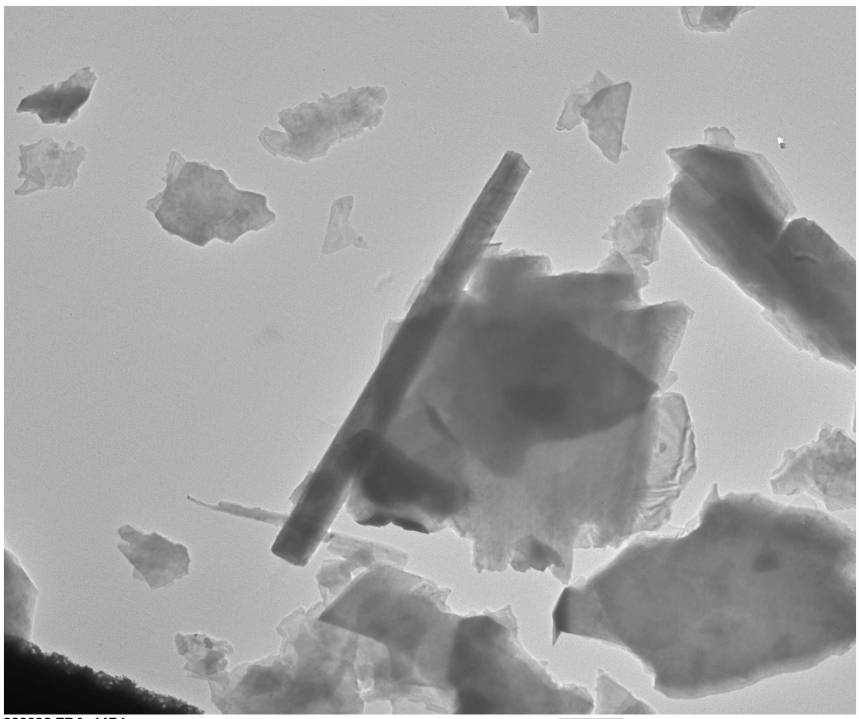
Sample 300396-4A Diffraction pattern from chrysotile structure #2 pictured above.

300396 FDA_100.jpg
Chrysotile 2 Diff
16:25 4/11/2019

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

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

Sample 300396-4B Tremolite fiber

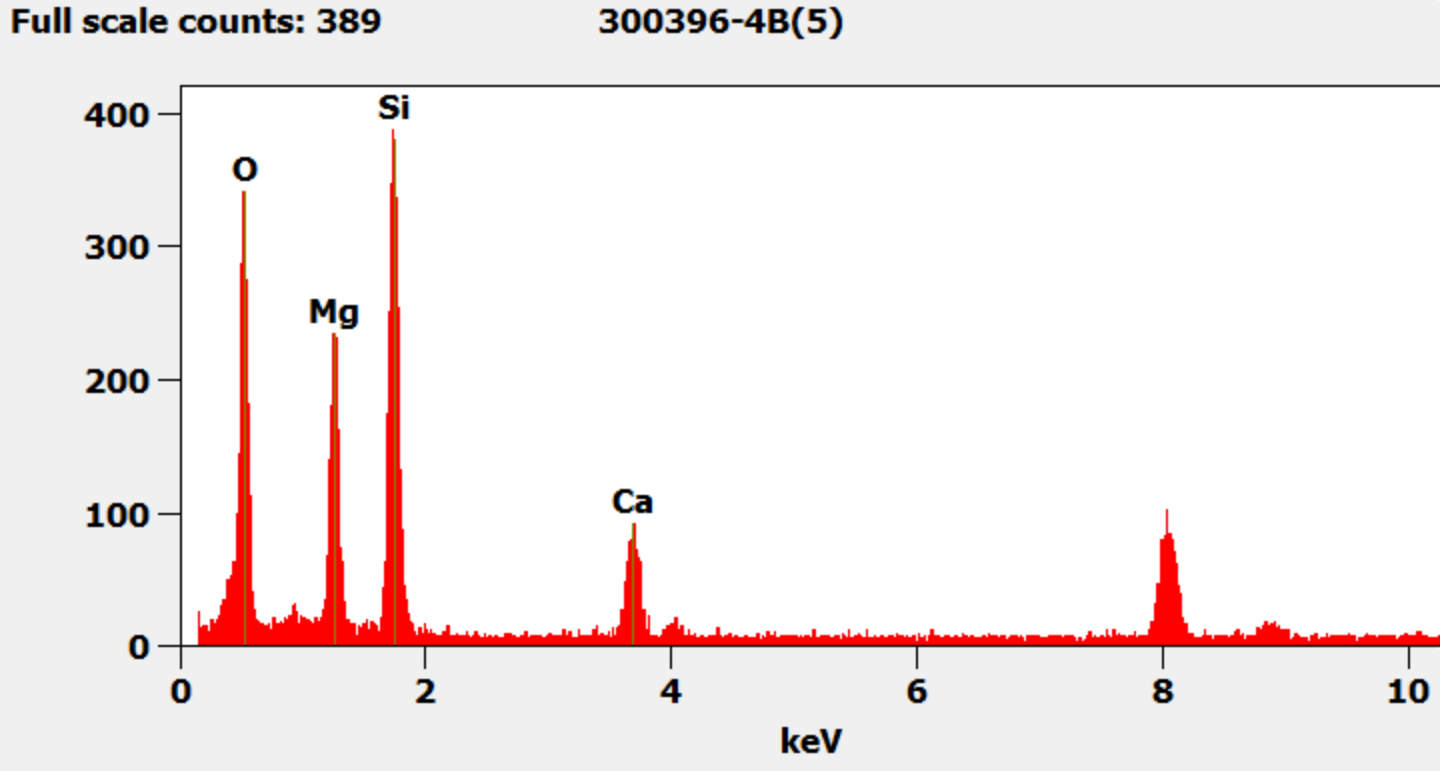


300396 FDA_117.jpg
Tremolite Fiber
[-4 0 -4]
Cal: 0.005415 µm/pix
11:30 4/16/2019
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

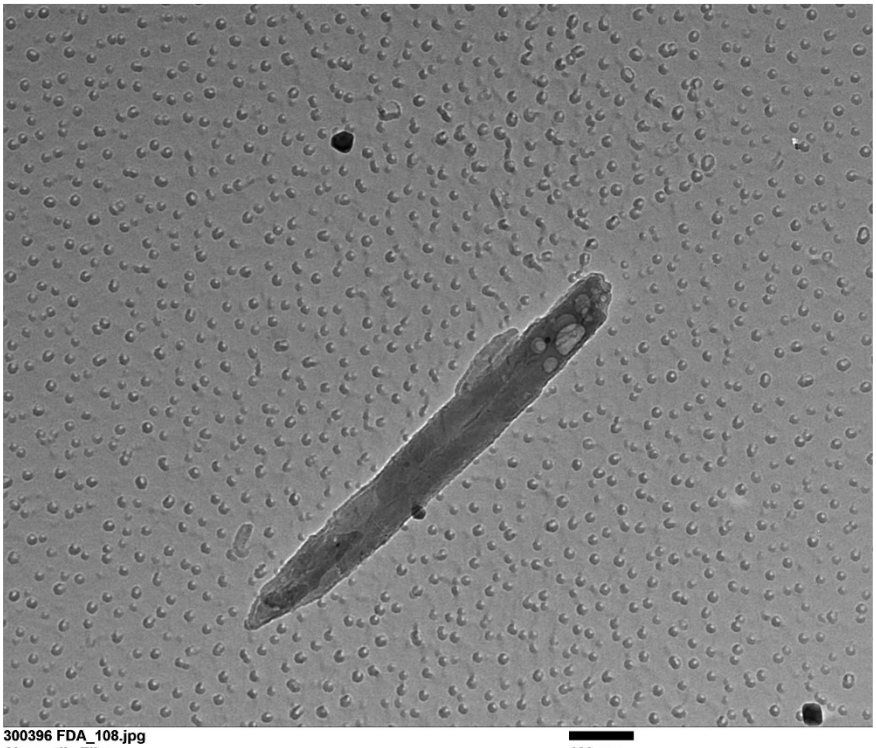
1 µm
HV=100kV
Direct Mag: 1900 x
AMA Analytical Services, Inc

Sample 300396-4B Diffraction pattern from tremolite fiber pictured above.

Sample 300396-4B Chemistry from tremolite fiber pictured above.



Sample 300396-4B Chrysotile structure #1



300396 FDA_108.jpg
Chrysotile Fiber
Cal: 0.541520 nm/pix
10:46 4/16/2019
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

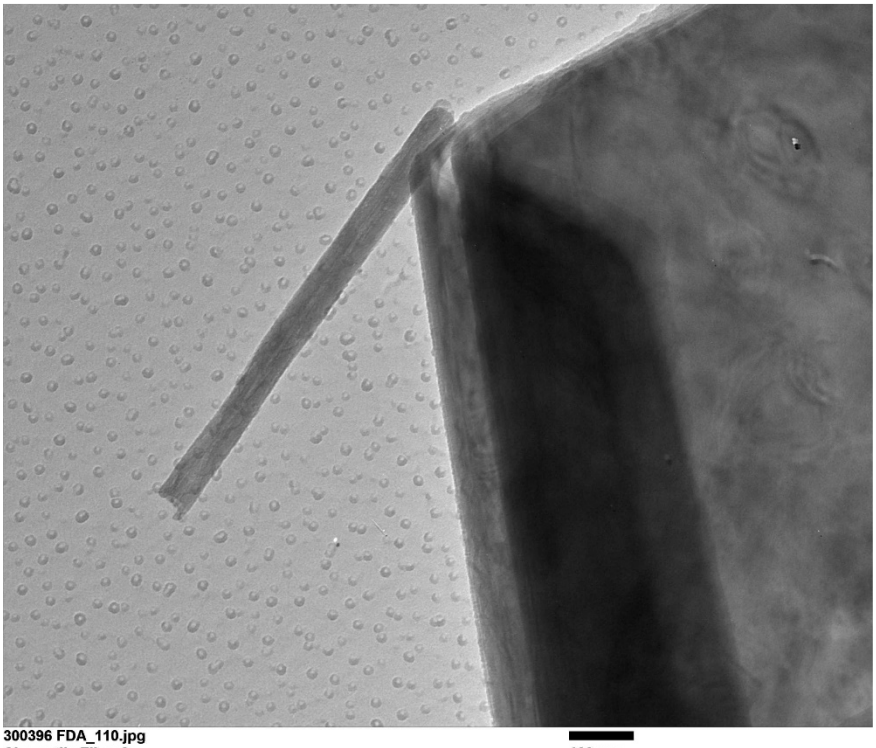
Sample 300396-4B Diffraction pattern from chrysotile structure #1 pictured above

300396 FDA_107.jpg
Chrysotile Diffraction
10:45 4/16/2019
Microscopist: (b

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

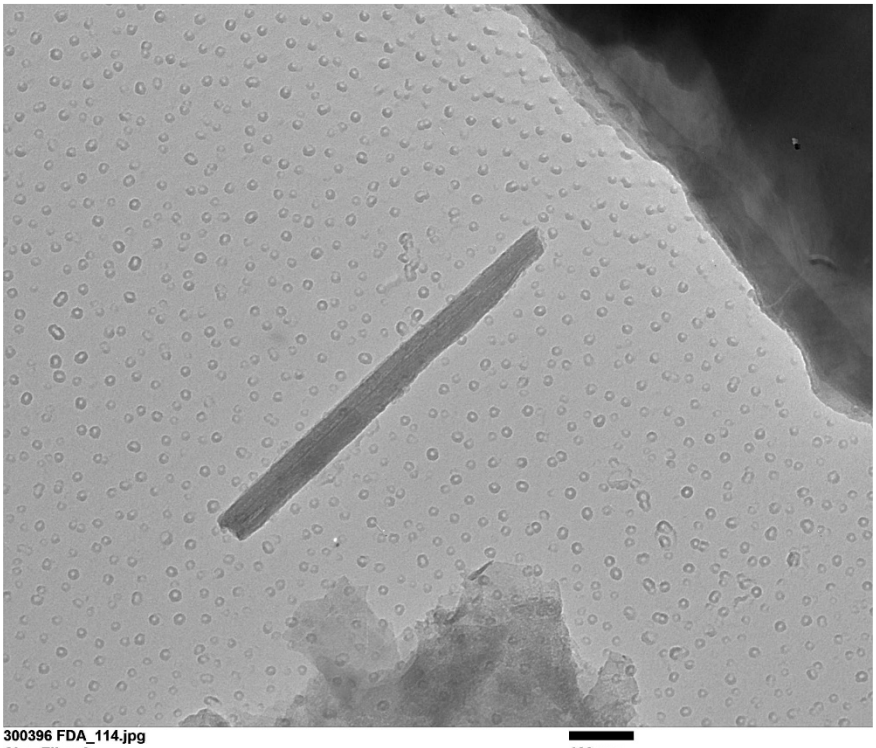
100 (1/Å)
HV=100kV
Cam Len: 0.2200 m
AMA Analytical Services, Inc

Sample 300396-4B Chrysotile structure #2

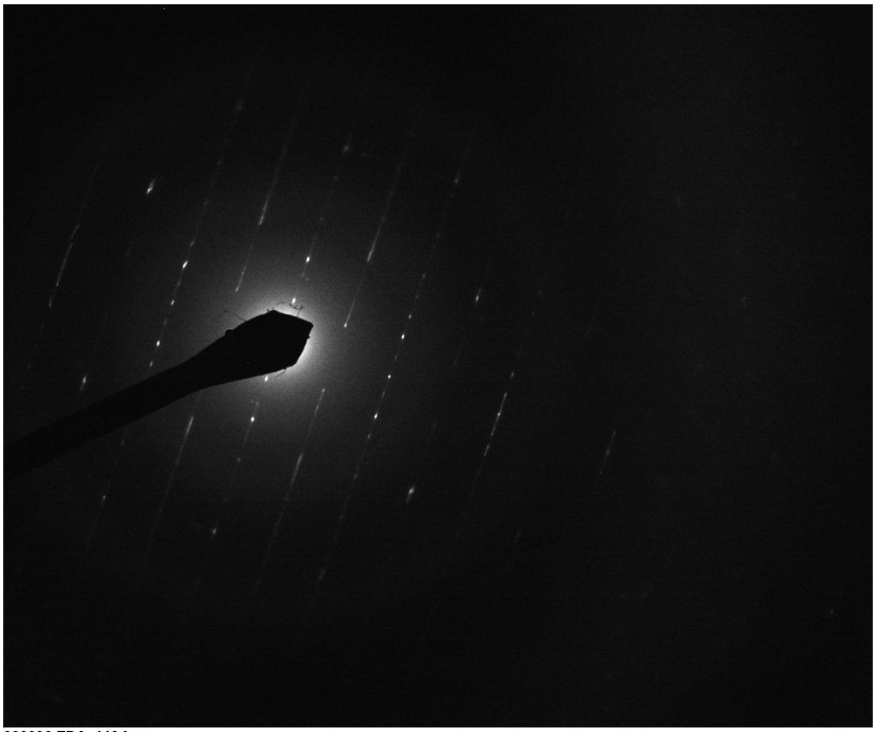


Sample 300396-4B Diffraction pattern from chrysotile structure #2 pictured above.

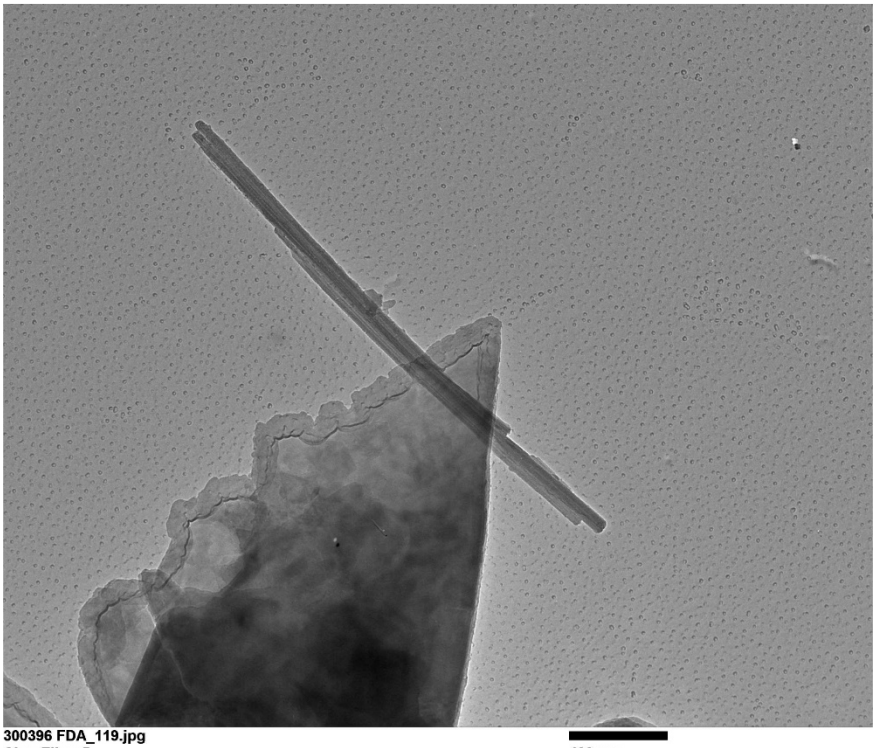
Sample 300396-4B Chrysotile structure #3



Sample 300396-4B Diffraction pattern from chrysotile fiber #3 pictured above.



Sample 300396-4B Chrysotile structure #5



300396 FDA_119.jpg
Chry Fiber 5
Cal: 0.001429 µm/pix
11:52 4/16/2019
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 drift frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

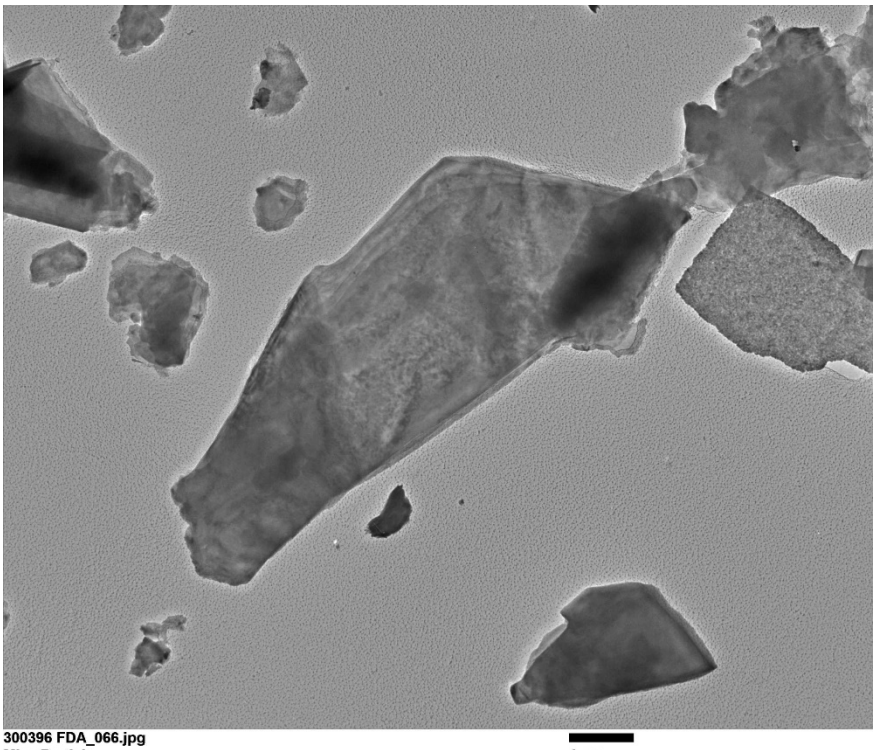
400 nm
HV=100kV
Direct Mag: 7200 x
AMA Analytical Services, Inc

Sample 300396-4B Diffraction pattern for Chrysotile structure #5 pictured above.

300396 FDA_118.jpg
Chry Diff 5
11:51 4/16/2019
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 300396-4 Mica particle

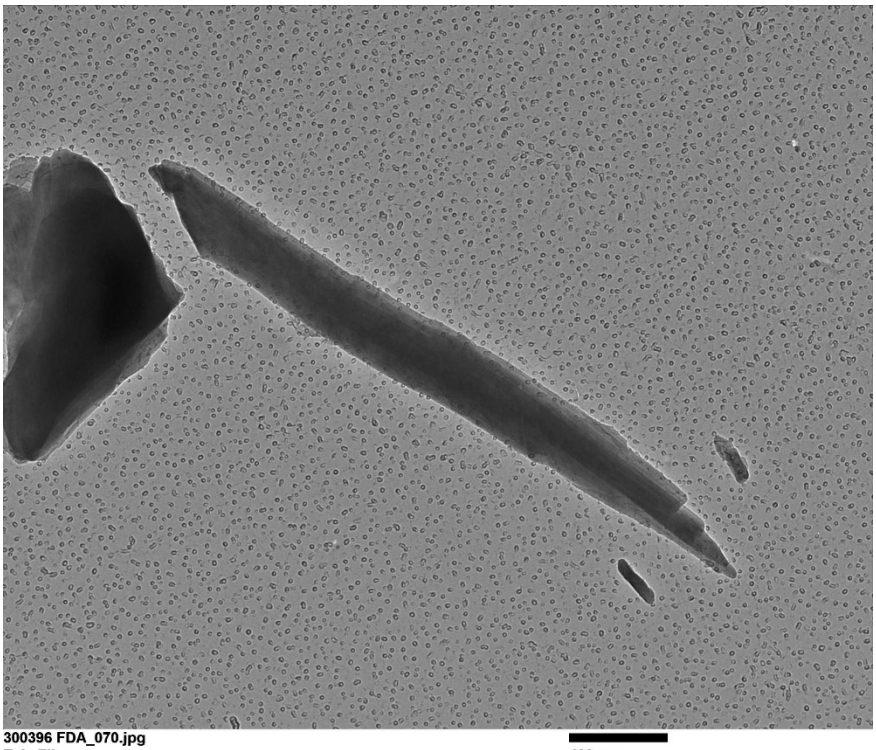


300396 FDA_066.jpg
Mica Particle
Cal: 0.005415 µm/pix
10:30 4/11/2019

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

Sample 300396-4 Talc fiber

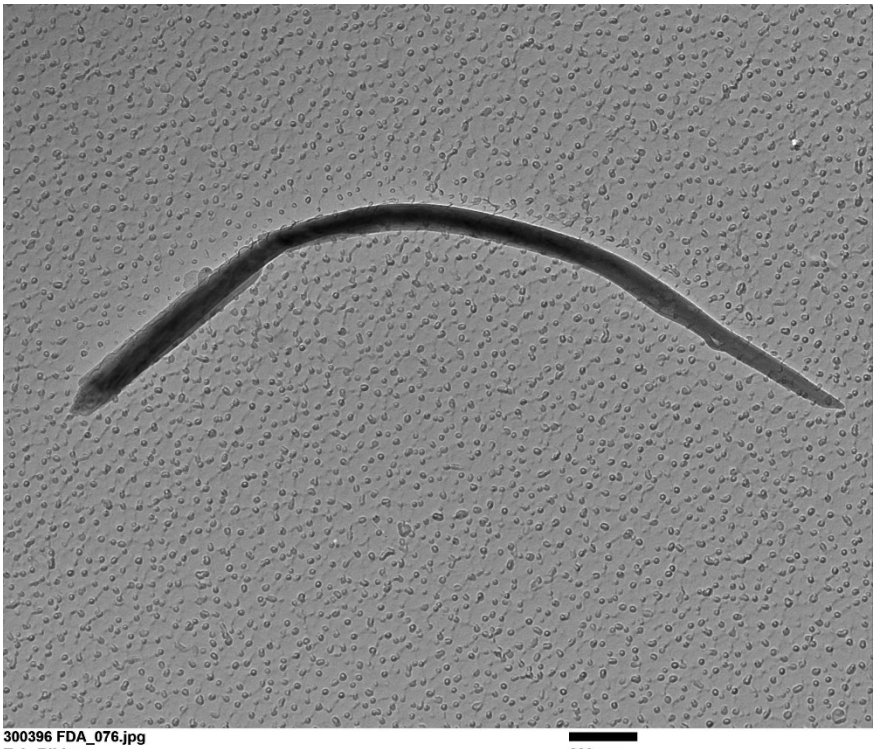


300396 FDA_070.jpg
Talc Fiber
Cal: 0.001429 µm/pix
10:39 4/11/2019

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

Sample 300396-4 Talc ribbon



<u>300396-5, 5A, 5B, Client Sample D36</u>

PLM

All three aliquots of sample D-36 were analyzed by (b) (6) on March 29, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

300396-5	NAD
300396-5A	NAD
300396-5B	NAD

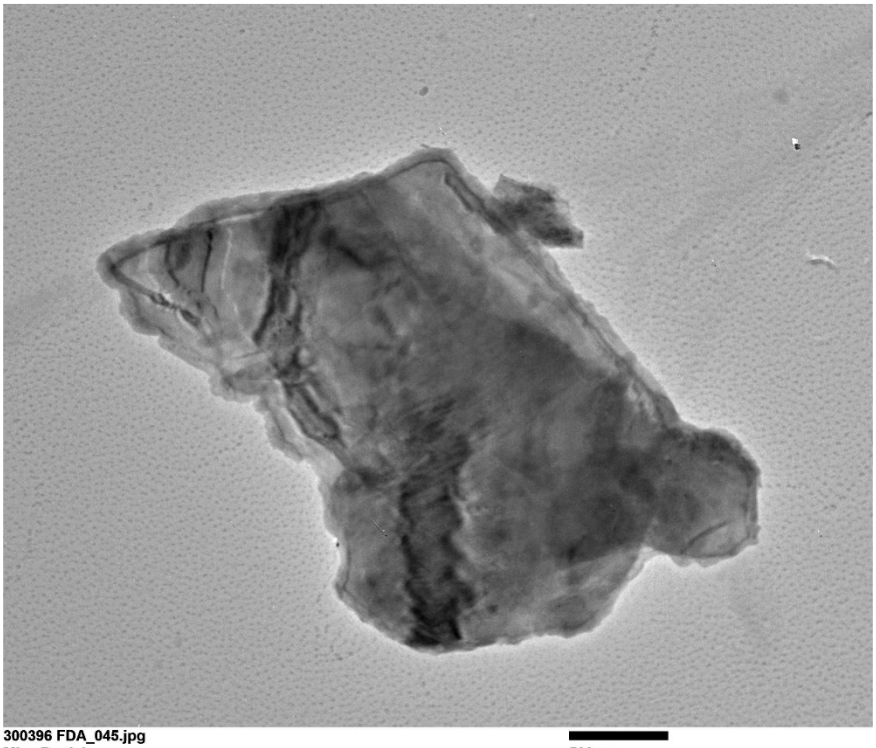
TEM

Sample 5 was analyzed by (b) (6) on April 13, 2019. Sample 5A was analyzed by (b) (6) on April 17, 2019 and sample 5B was analyzed by (b) (6) on April 18, 2019. The samples primarily consisted of mica particles with some titanium particles. No talc particles were observed. No asbestos or non-asbestos amphibole variants were detected in the samples. The results were calculated using the equations detailed in the calculations section.

300396-5	NAD
300396-5A	NAD
300396-5B	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.

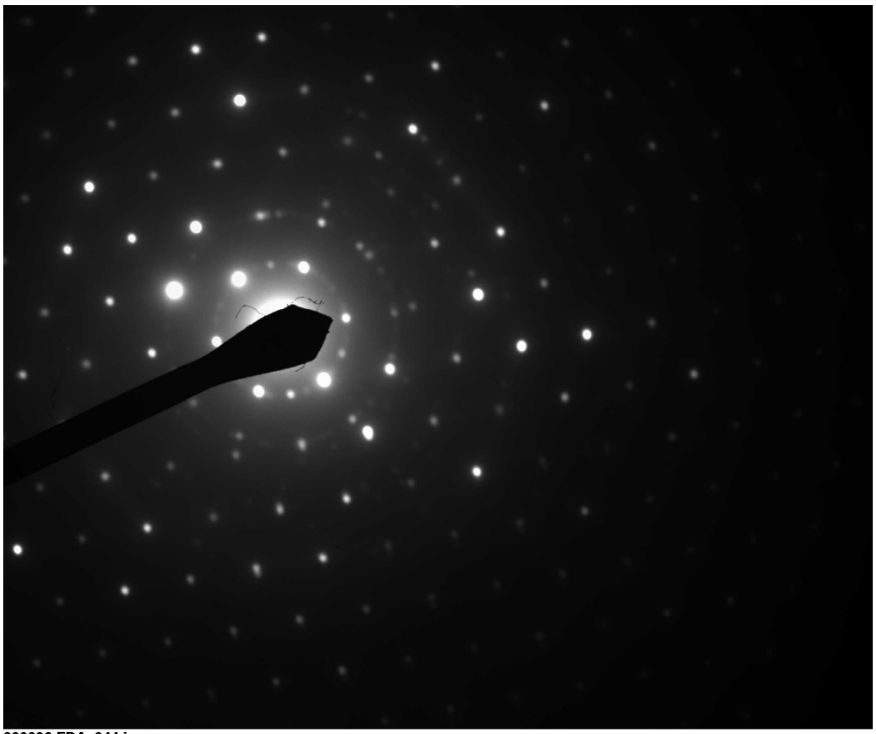
Sample 300396-5 Mica particle



300396 FDA_045.jpg
Mica Particle
Cal: 0.001774 µm/pix
12:18 4/5/2019
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

Sample 300396-5 Diffraction pattern from mica particle pictured above.

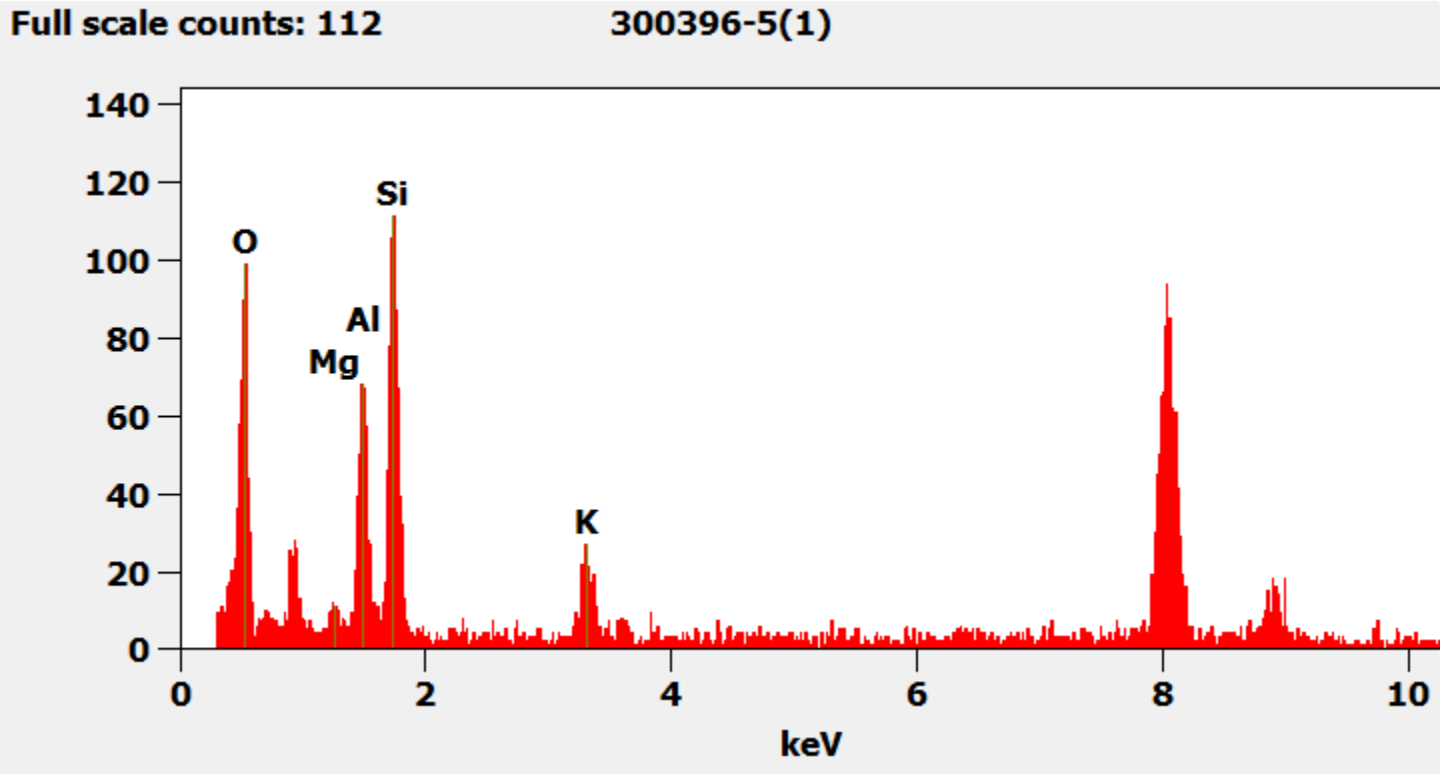


300396 FDA_044.jpg
Mica Particle Diff
12:17 4/5/2019
Microscopist: (b)

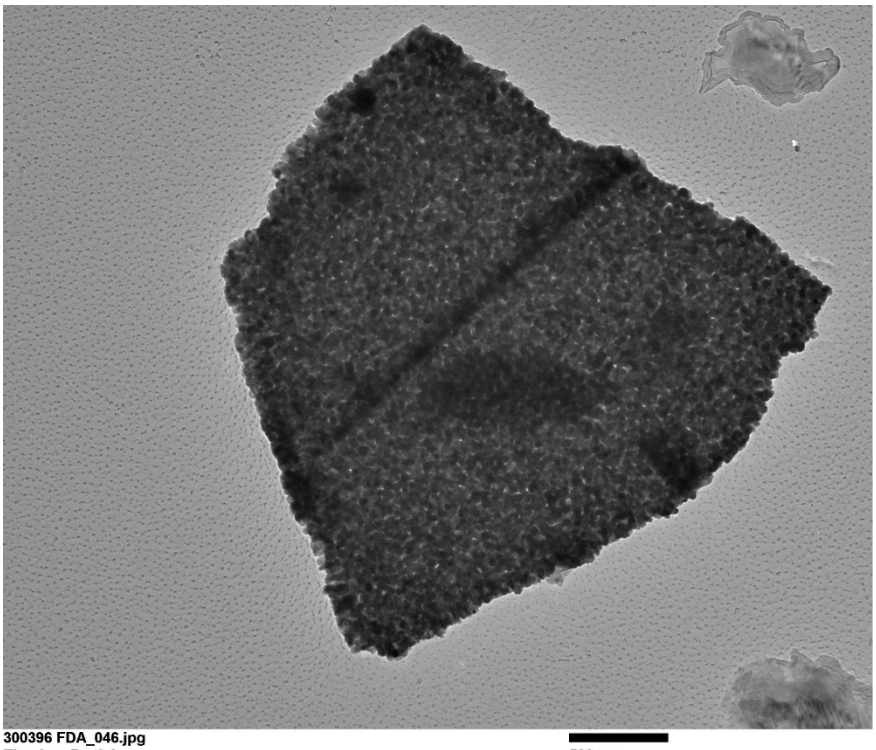
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 std. frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Sample 300396-5 Chemistry from mica particle pictured above.



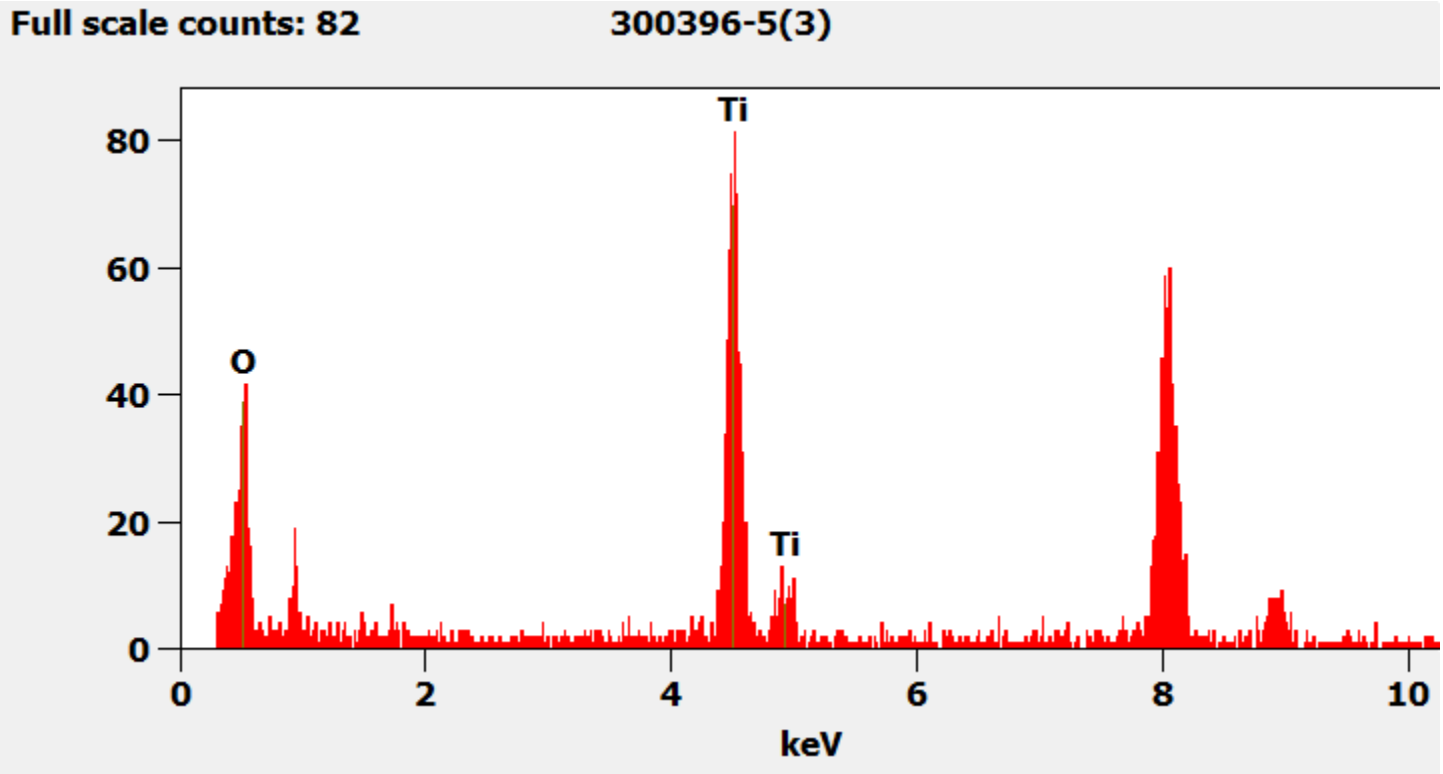
Sample 300396-5 Titanium particle



300396 FDA_046.jpg
Titanium Particle
Cal: 0.001774 µm/pix
12:22 4/5/2019
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

Sample 300396-5 Chemistry from titanium particle pictured above.



300396-6, 6A, 6B, Client Sample D-37

PLM

All three aliquots of sample D-37 were analyzed by (b) (6) on March 29, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

300396-6	NAD
300396-6A	NAD
300396-6B	NAD

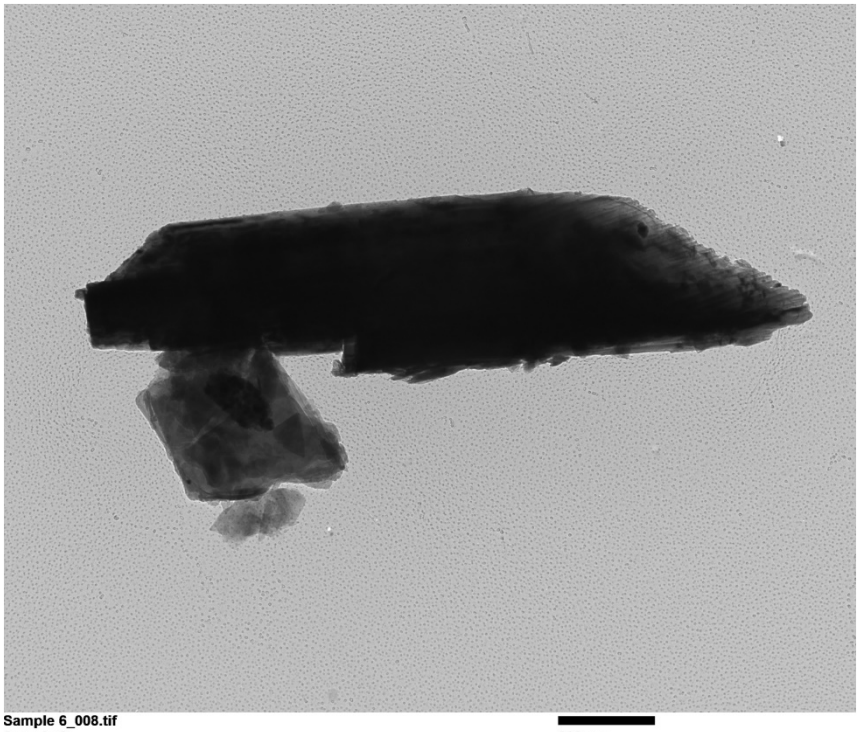
TEM

(b) (6) analyzed sample 6 on April 10, 2019 and samples 6A and 6B on April 11, 2019. The samples primarily consist of Mica, talc, and titanium particles. One particle was observed with chemistry that was consistent with tremolite. A zone axis pattern was obtained and, when indexed, was found to not match tremolite. No asbestos or non-asbestos amphibole variants were detected in the samples. The results were calculated using the equations detailed in the calculations section.

300396-6	NAD
300396-6A	NAD
300396-6B	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 300396-6 Particle with chemistry similar to tremolite.



Sample 6_008.tif
Sample 6
Particle with tremolite chemistry
Cal: 0.002858 µm/pix
13:06 4/10/2019
Microscopist: (b) (6)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 std. frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

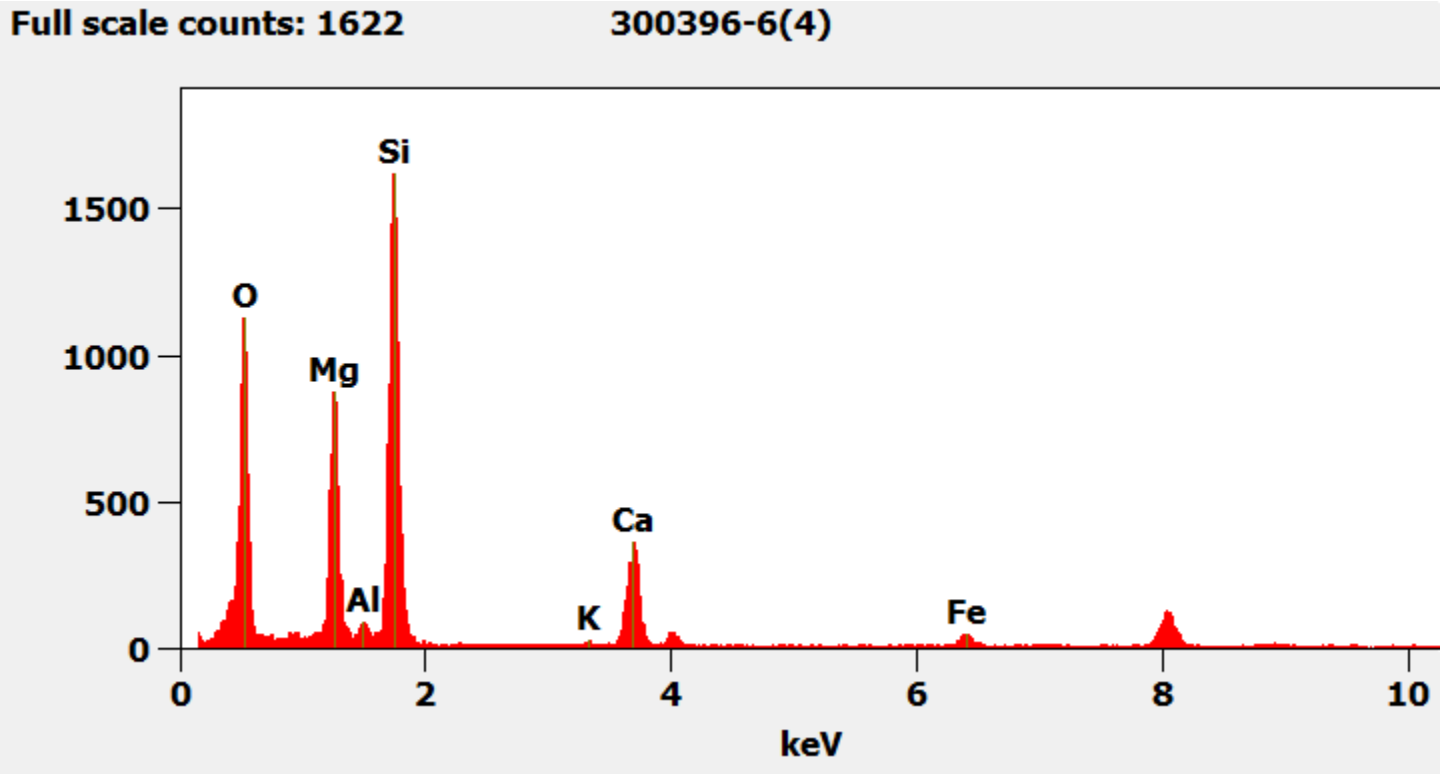
Sample 300396-6 Diffraction pattern for the particle pictured above

.tif
Sample 6
diffraction
12:42 4/10/2019

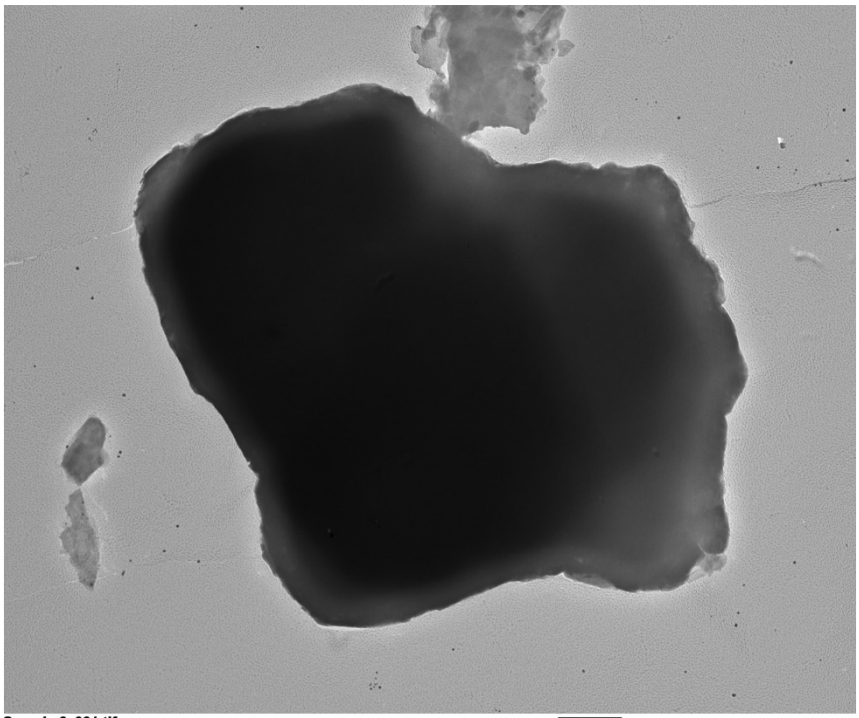
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 std. frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

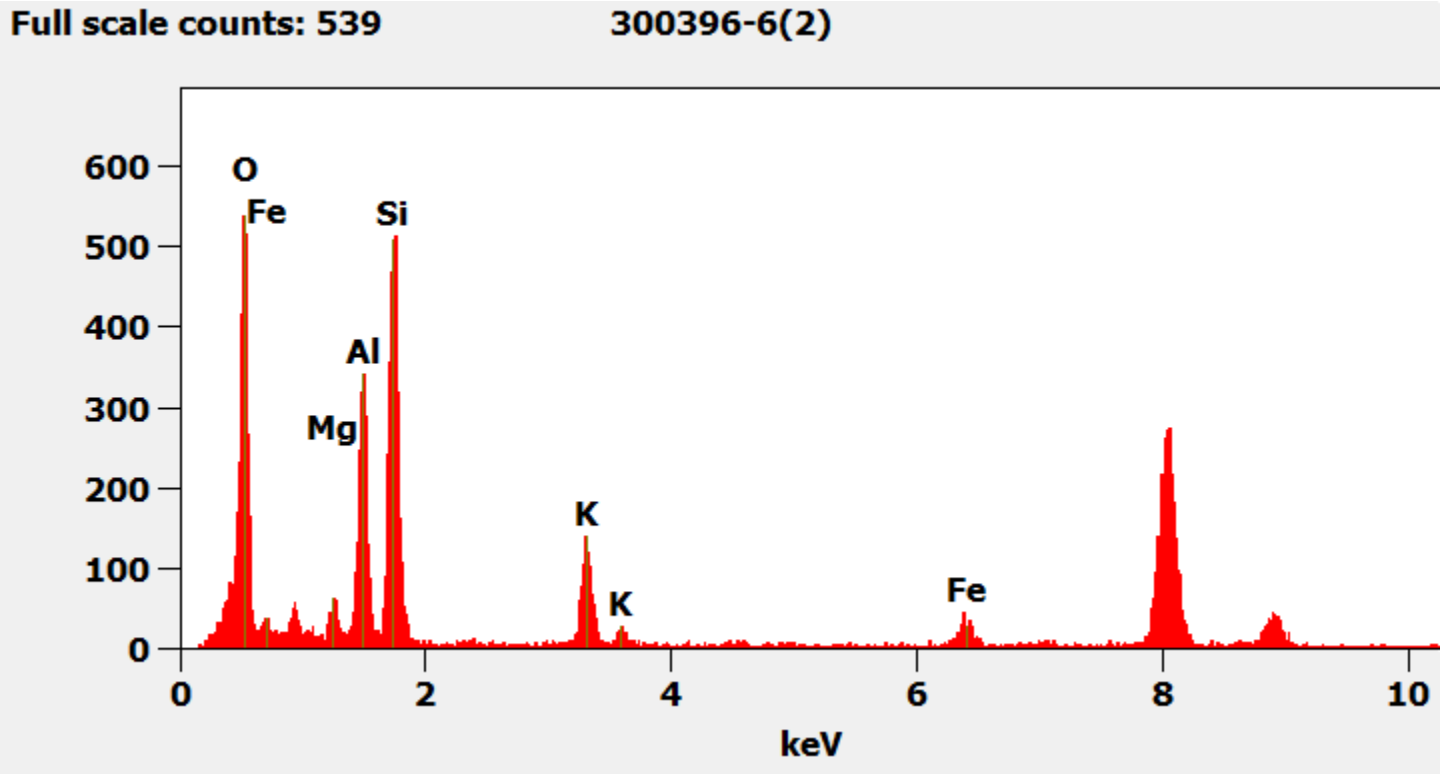
Sample 300396-6 Chemistry of particle pictured above.



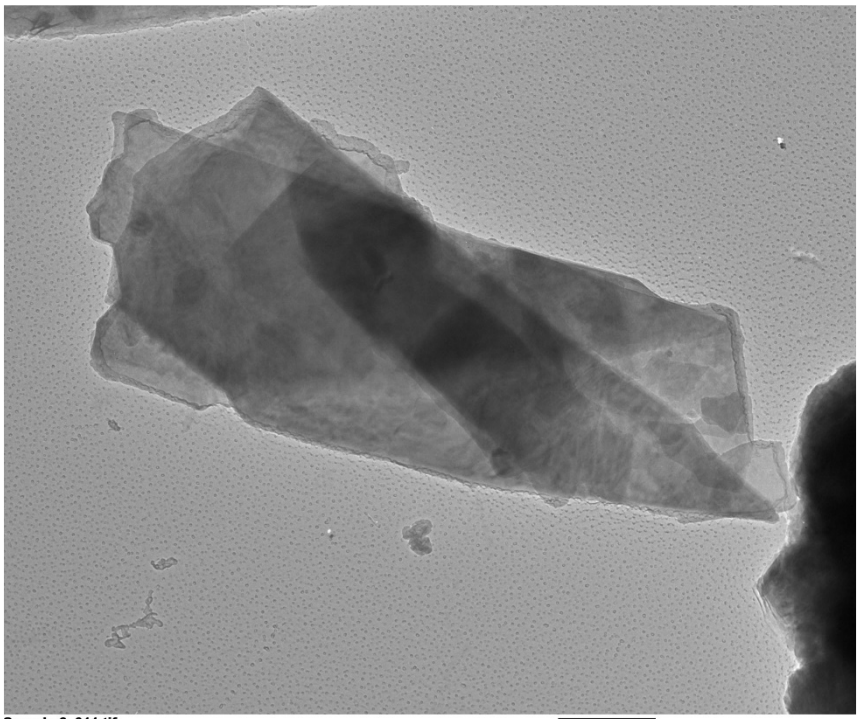
Sample 300396-6 Mica particle



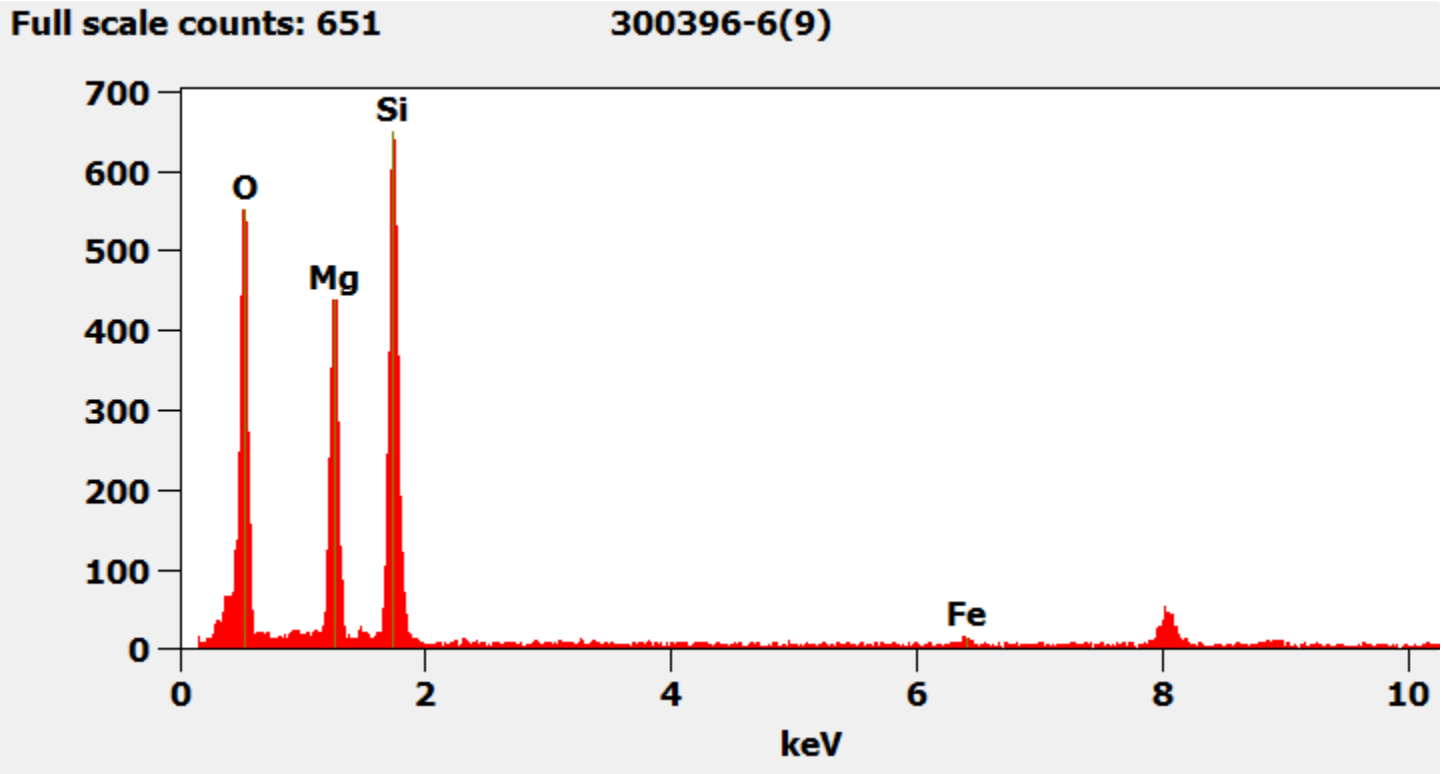
Sample 300396-6 Chemistry from mica particle pictured above.



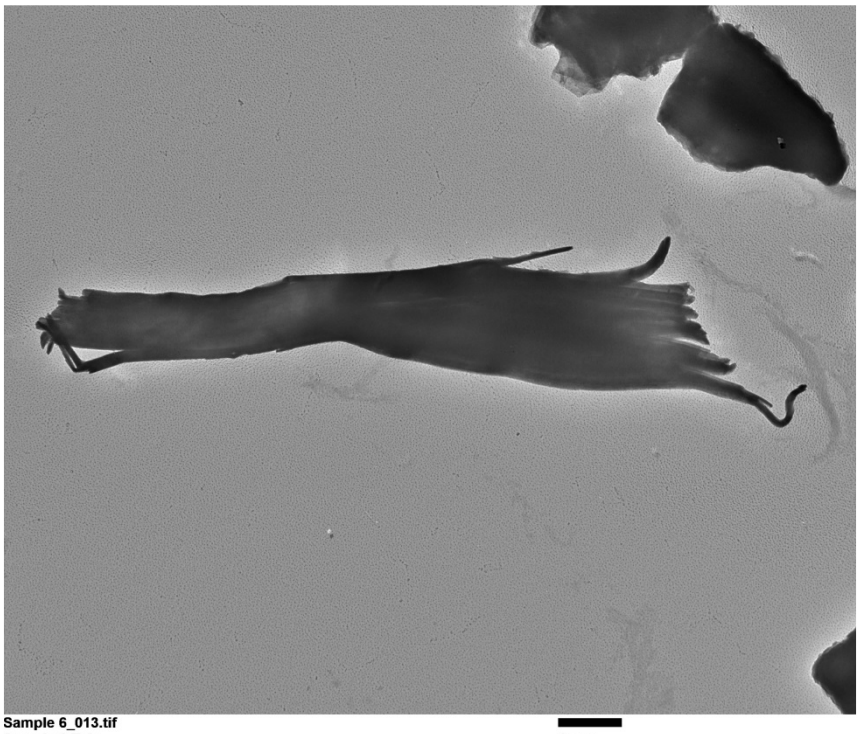
Sample 300396-6 Talc Particle



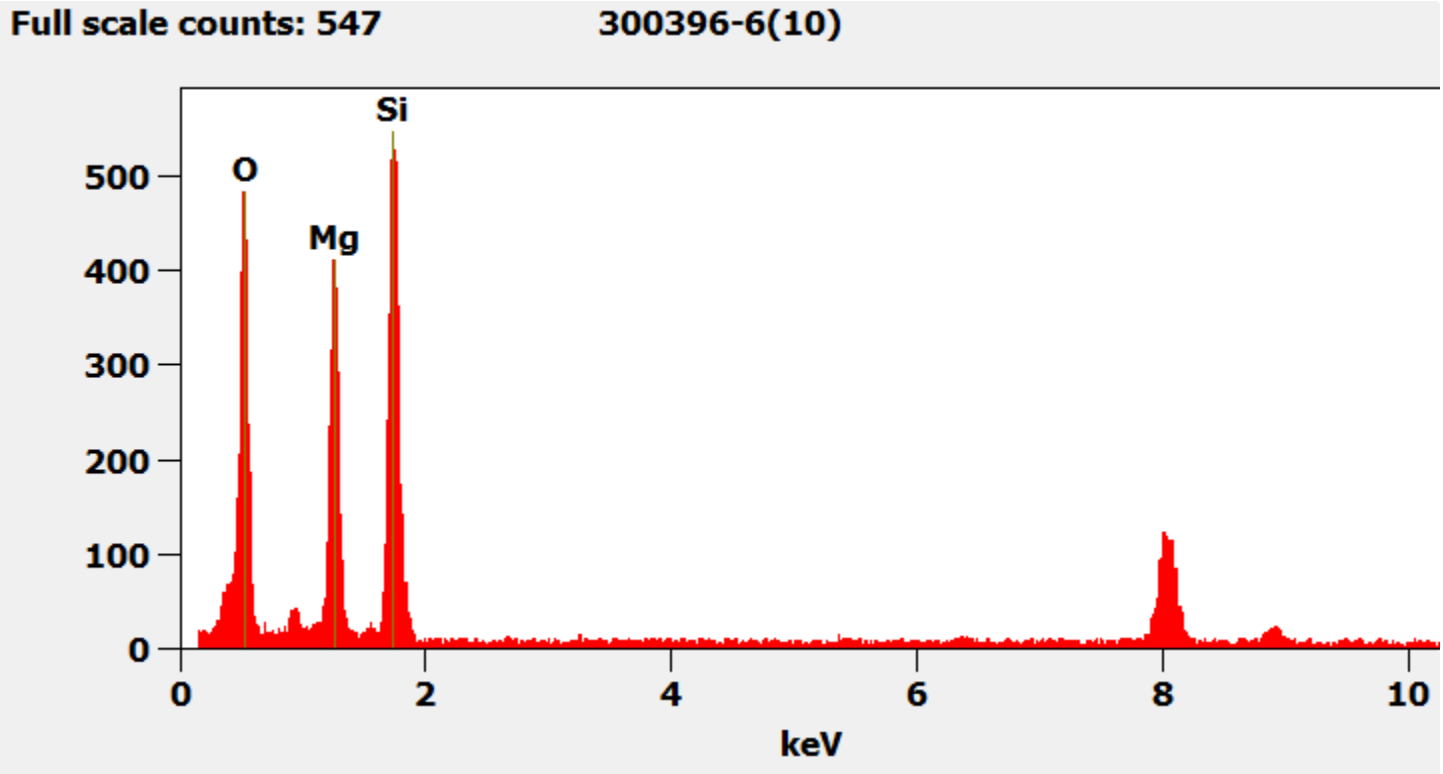
Sample 300396-6 Chemistry from talc particle pictured above.



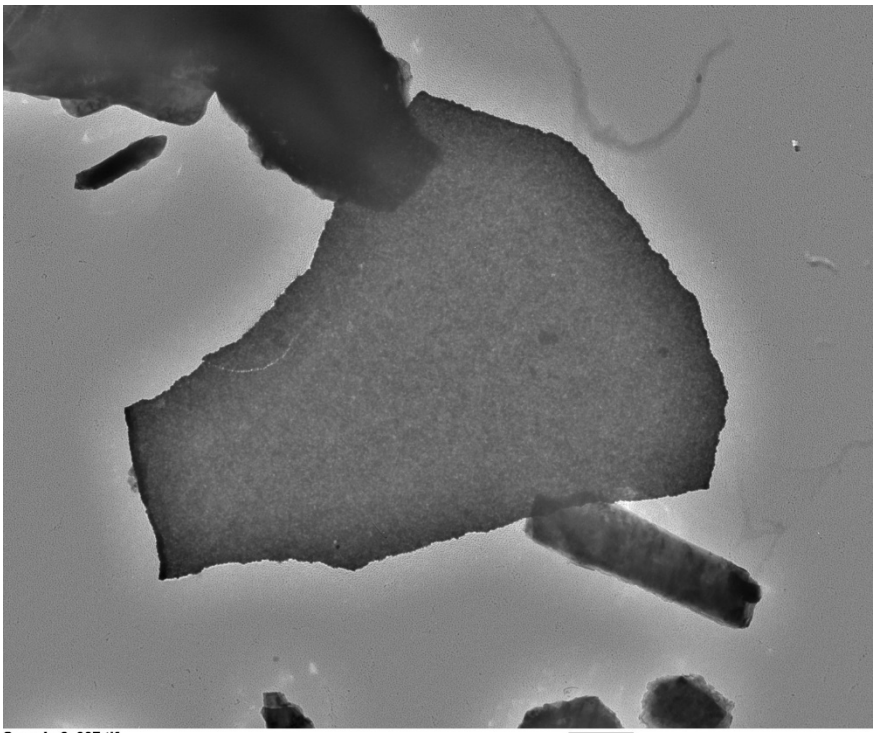
Sample 300396-6 Talc ribbon



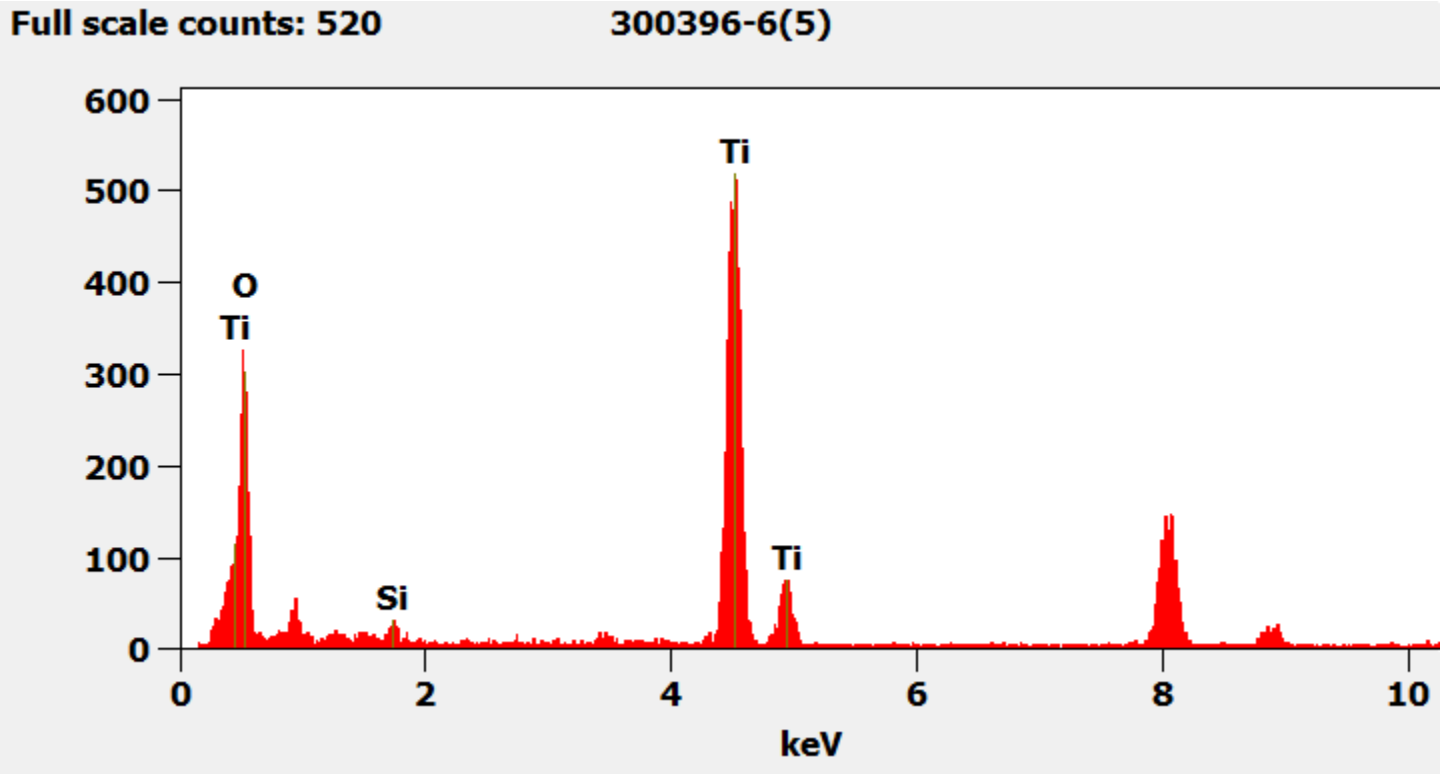
Sample 300396-6 Chemistry from talc ribbon pictured above.



Sample 300396-6 titanium particle



Sample 300396-6 Chemistry from titanium particle pictured above.



<u>300396-7, 7A, 7B, Client Sample D-38</u>

PLM

All three aliquots of sample D-38 were analyzed by (b) (6) on March 29, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

300396-7	NAD
300396-7A	NAD
300396-7B	NAD

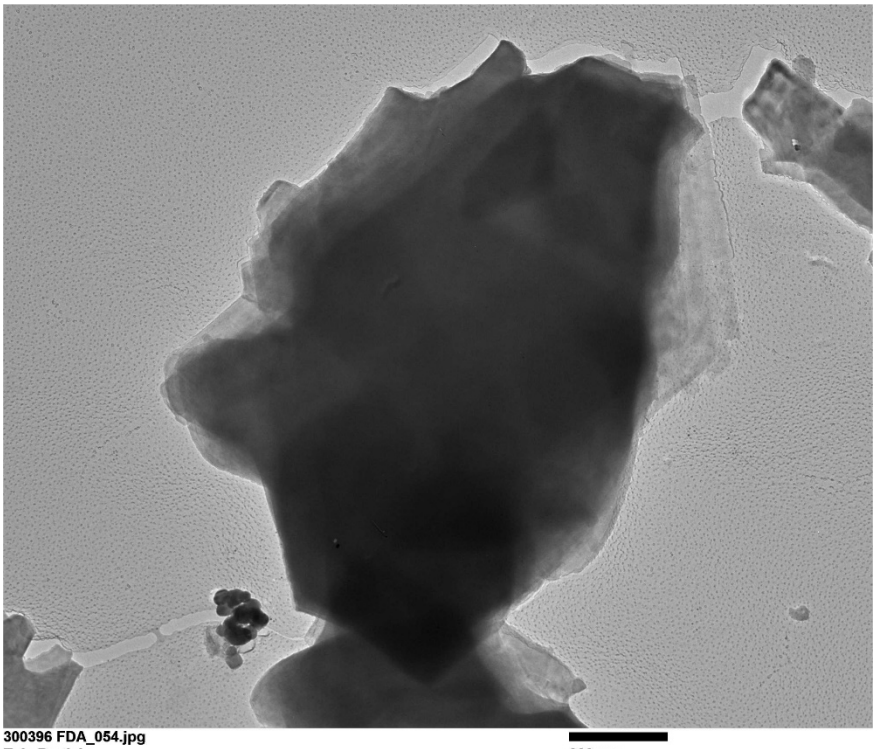
TEM

(b) (6) analyzed Sample 7 on April 11, 2019 and sample 7B on April 18, 2019. (b) (6) analyzed sample 7A on April 18, 2019. The sample consisted primarily of talc particles. These were mostly talc flakes but numerous talc fibers and ribbons were also observed. A few mica particles were observed. No asbestos or non-asbestos amphibole variants were detected in the samples. The results were calculated using the equations detailed in the calculations section.

300396-7	NAD
300396-7A	NAD
300396-7B	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 300396-7 Talc particle



300396 FDA_054.jpg
Talc Particle
Cal: 0.002858 µm/pix
19:46 4/10/2019
Microscopist: (b)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 std. frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

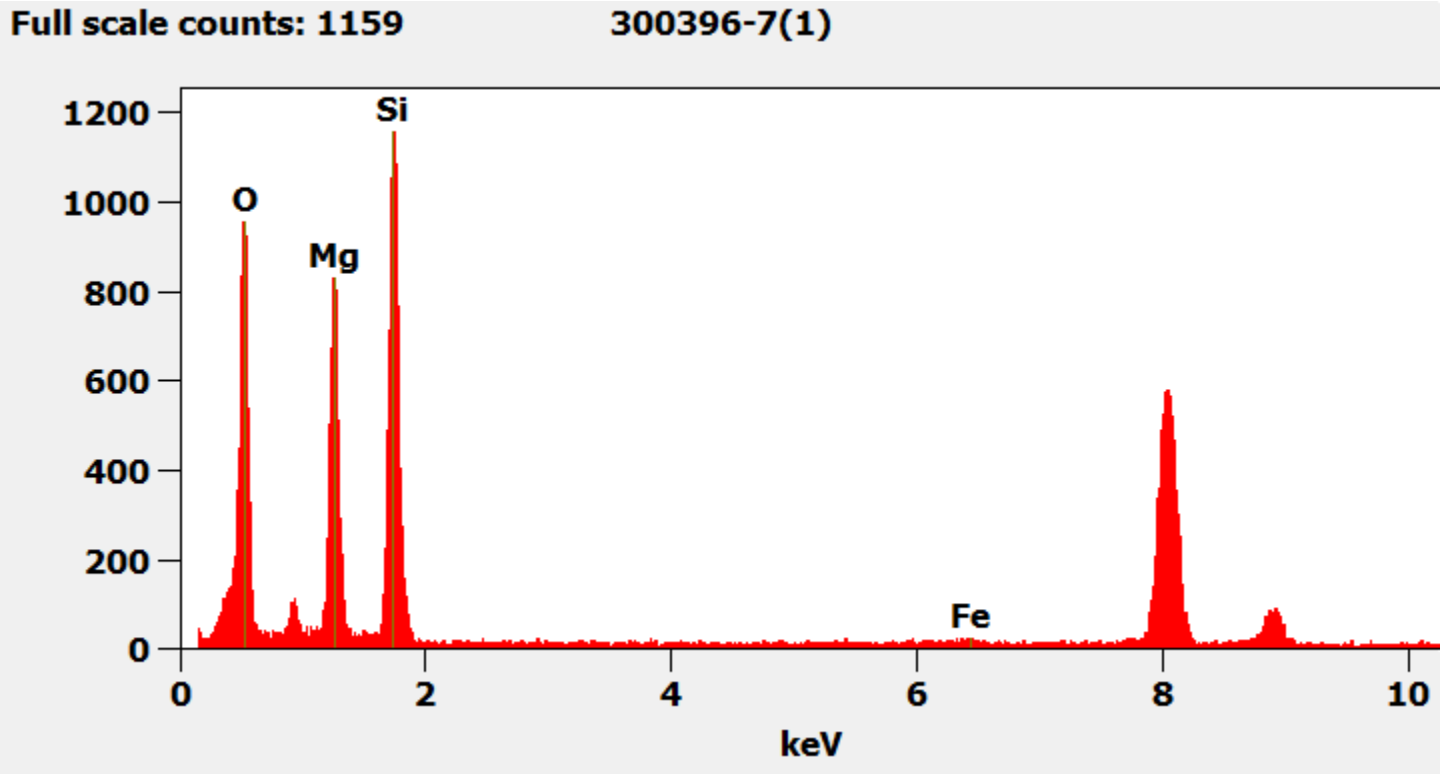
Sample 300396-7 Diffraction pattern from talc particle pictured above.

300396 FDA_055.jpg
Talc Particle Diff
19:47 4/10/2019
Microscopist: (b)

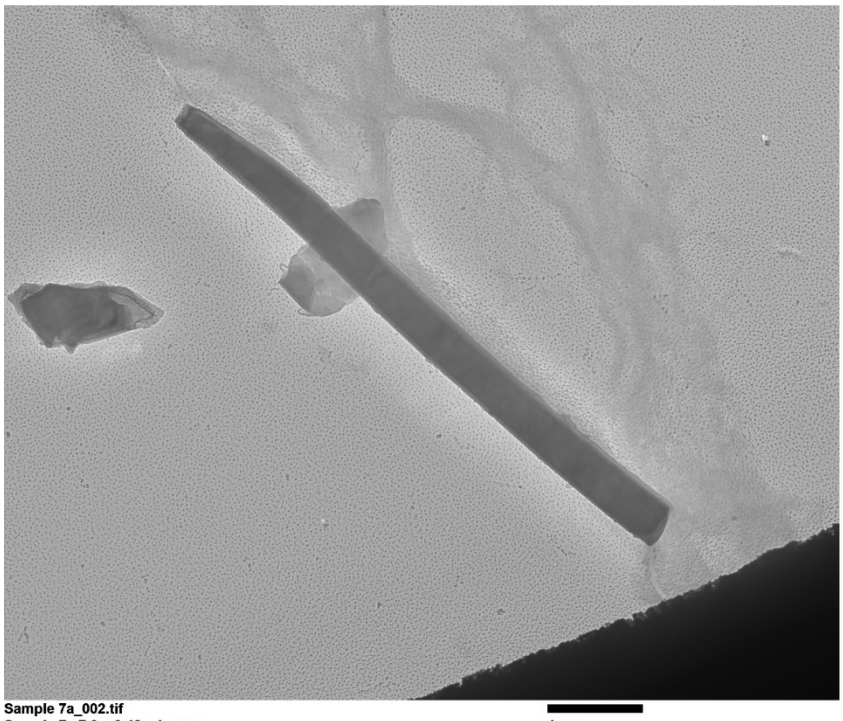
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 std. frames, Gain: 1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

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

Sample 300396-7 Chemistry from talc particle pictured above

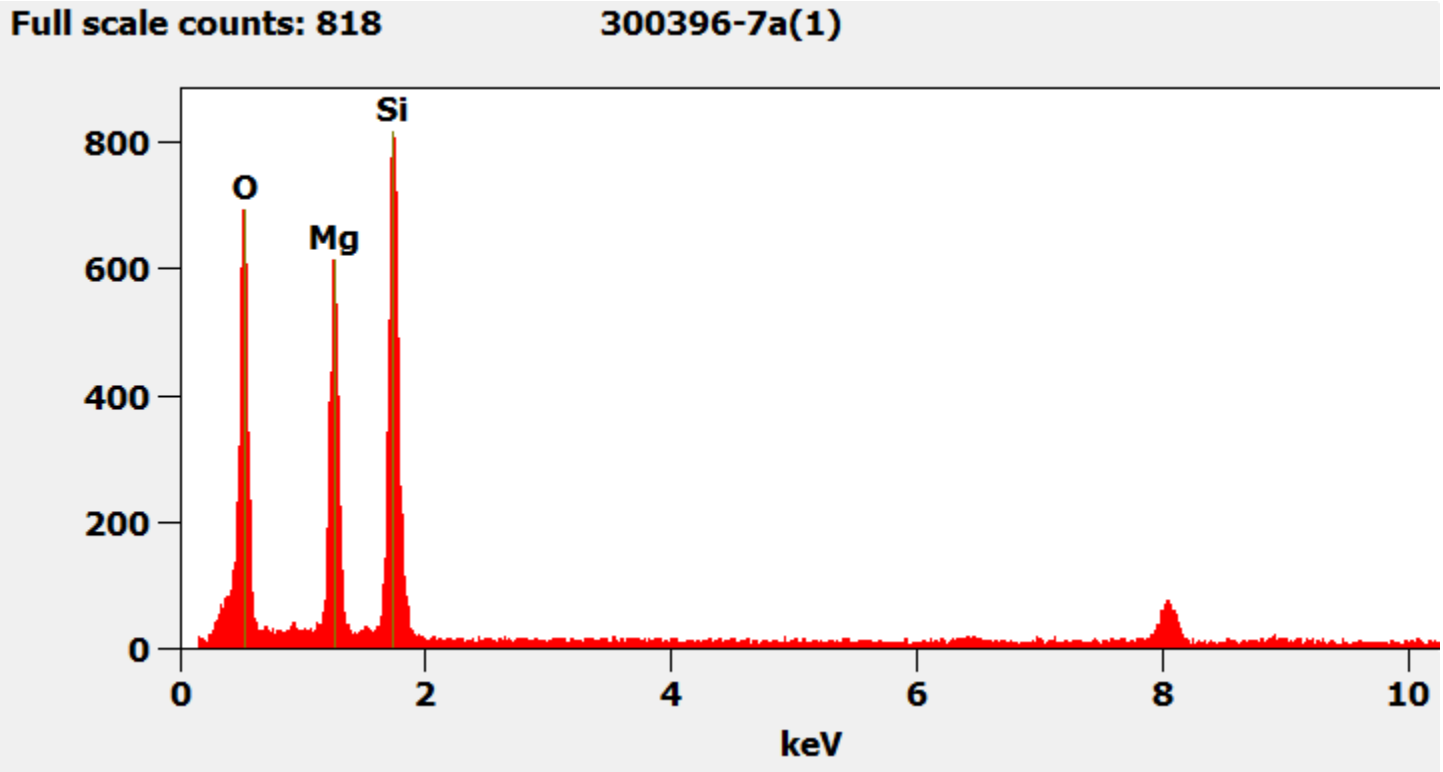


Sample 300396-7A Talc fiber



Sample 300396-7A Diffraction pattern from talc fiber pictured above.

Sample 300396-7A Chemistry from talc fiber pictured above.



300396-8, 8A, 8B, Client Sample D-39

PLM

All three aliquots of sample D-39 were analyzed by (b) (6) on March 29, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

300396-8	NAD
300396-8A	NAD
300396-8B	NAD

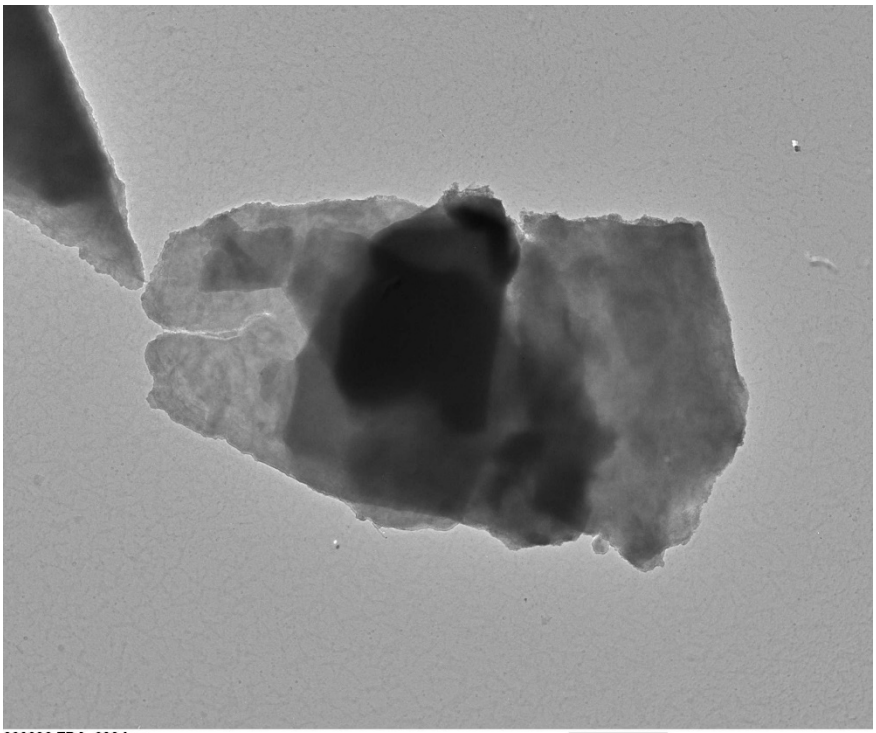
TEM

(b) (6) analyzed sample 8 on April 11, 2019, sample 8A on April 14, 2019 and sample 8B on April 17, 2019. The sample consisted of primarily talc 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.

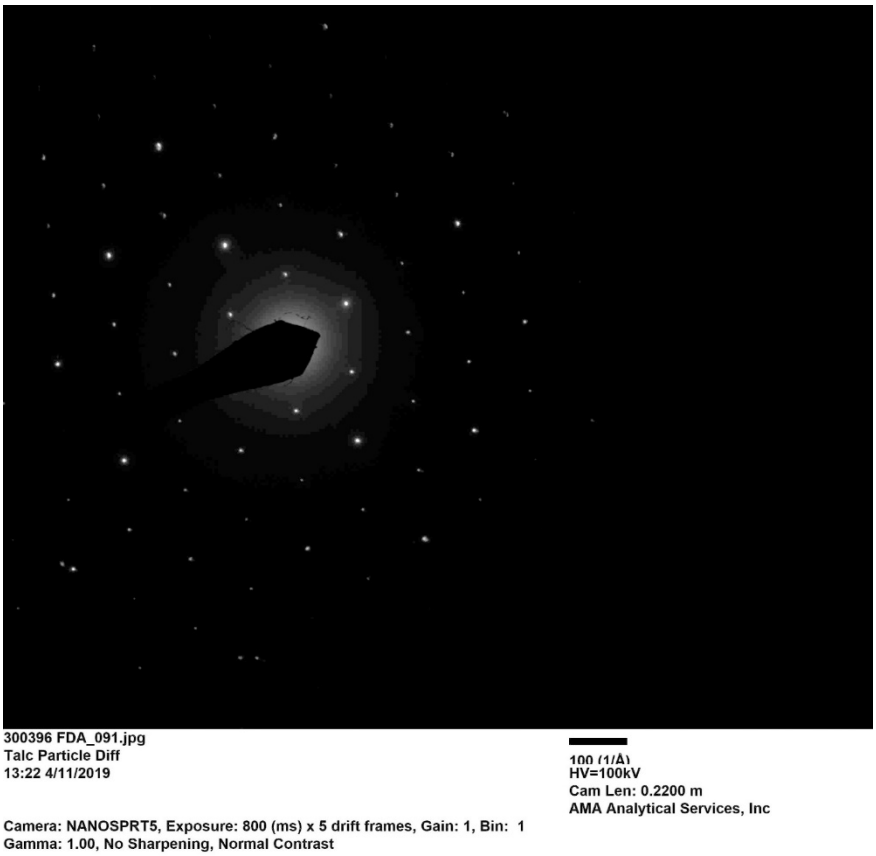
300396-8	NAD
300396-8A	NAD
300396-8B	NAD

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.

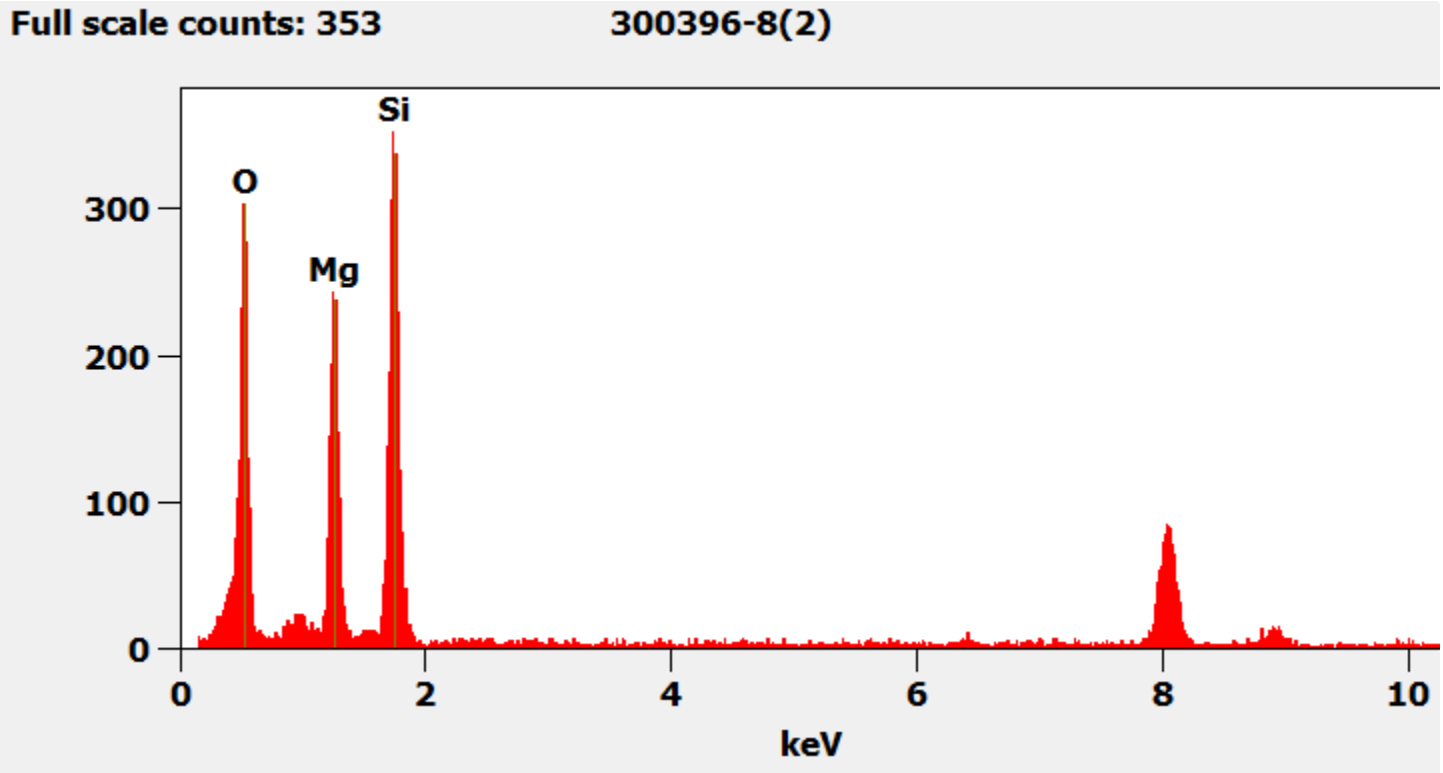
Sample 300396-8 Talc Particle



Sample 300396-8 Diffraction pattern from talc particle pictured above.



Sample 300396-8 Chemistry from talc particle pictured above.



QC Discussion:
During preparation, one blank control sample and one reference control sample were prepared. These samples were prepared alongside the customer samples. The blank sample was prepared using Sigma-Aldrich Talc Powder, <10 micron, and was analyzed by (b) (6) on April 25, 2019. No asbestos was detected on the blank sample. 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 April 25, 2019 and found to be within acceptable limits.

Our laboratory information management systems (LIMS) randomly selected sample 300396-3/D-34 for additional replicate QC analysis. Separate preparations were made for PLM and TEM analysis. The replicate QC analysis was performed by (b) (6) on March 29, 2019, 2019 for PLM analysis and by (b) (6) on April 25, 2019 for TEM analysis. The Chrysotile found in the replicate QC sample for D-34 is consistent with the findings of 300396-3A/D-34 and 300396-3B/D-34.

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 9/1/2018 and 3/31/2019
7) Replicate & Duplicate QC Chart for (b) (6) for samples analyzed between 9/1/2018 and 4/19/2019
8) Replicate & Duplicate QC Chart for (b) (6) for samples analyzed between 9/1/2018 and 4/19/2019
9) Replicate & Duplicate QC Chart for (b) (6) for samples analyzed between 9/1/2018 and 4/19/2019
10) 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.

4/25/2019

Andreas Saldivar — Date
Laboratory Director