

Chain of Custody: 625547

Client: US Food & Drug Adminitration Address: Office of Cosmetics & Colors

4300 River Road College Park, MD 20740

Attention: John Gasper

Job Name: Assignment DFPG #21-18

Job Location: Batch No. 02232021 (Batch #2A) Job Number: CLIN 0001

PO Number: 75F40119P10689

Date Submitted: 3/8/2021

Date Analyzed: 3/29/2021-4/23/2021

Report Date: 5/7/2021 Date Sampled: Not Provided Person Submitting: Martha Schwartz Revised: 5/17/2021 (Revision #3)

SUMMARY OF ANALYSIS

AMA Sample ID	Client Sample ID	TEM LOD Using ASTM D5756 Mass Calculation	TEM LOQ Using ASTM D5756 Mass Calculation	% Chrysotile by TEM Using ASTM D5756 Mass Calculation	% Tremolite by TEM Using ASTM D5756 Mass Calculation	% Total Chrysotile & Tremolite by TEM Using ASTM D5756 Mass Calculation	% Asbestos by PLM	% Organics	% Acid Soluable	% Other	Comments
625547-1B	02232021-1	0.00000238%	0.00000952%	ND	ND	< 0.00001%	ND	17.04%	9.52%	73.43%	
625547-1C	02232021-1	0.0000199%	0.00000797%	ND	ND	< 0.00001%	ND	17.08%	9.42%	73.51%	
625547-2A	02232021-2	0.00000175%	0.0000699%	ND	ND	< 0.00001%	ND	2.42%	2.50%	95.08%	
625547-2B	02232021-2	0.00000217%	0.0000868%	ND	ND	< 0.00001%	ND	2.35%	2.86%	94.79%	
625547-2C	02232021-2	0.00000221%	0.00000884%	ND	ND	< 0.00001%	ND	2.51%	2.65%	94.85%	
625547-3A	02232021-3	0.0000188%	0.00000751%	ND	ND	< 0.00001%	ND	17.46%	8.06%	74.48%	
625547-3B	02232021-3	0.0000186%	0.00000745%	ND	ND	< 0.00001%	ND	17.27%	9.16%	73.57%	
625547-3C	02232021-3	0.0000196%	0.00000782%	ND	ND	< 0.00001%	ND	17.27%	9.36%	73.37%	
625547-4A	02232021-4	0.0000158%	0.00000632%	ND	ND	< 0.00001%	ND	2.48%	4.78%	92.75%	
625547-4B	02232021-4	0.0000148%	0.00000593%	ND	ND	< 0.00001%	ND	2.47%	4.70%	92.83%	
625547-4C	02232021-4	0.0000148%	0.00000592%	ND	ND	< 0.00001%	ND	2.51%	4.18%	93.32%	
625547-5A	02232021-5	0.00000245%	0.00000981%	ND	ND	< 0.00001%	ND	20.40%	18.22%	61.39%	
625547-5B	02232021-5	0.00000324%	0.00001297%	ND	ND	< 0.00001%	ND	20.45%	19.17%	60.38%	
625547-5C	02232021-5	0.00000233%	0.00000931%	ND	ND	< 0.00001%	ND	20.45%	20.04%	59.51%	
625547-6A	02232021-6	0.00000293%	0.00001173%	ND	ND	< 0.00001%	ND	5.16%	7.04%	87.80%	
625547-6B	02232021-6	0.00000250%	0.00001002%	ND	ND	< 0.00001%	ND	5.14%	7.58%	87.28%	
625547-6C	02232021-6	0.00000258%	0.00001030%	ND	ND	< 0.00001%	ND	5.11%	7.75%	87.14%	
625547-7A	02232021-7	0.00000220%	0.00000882%	ND	ND	< 0.00001%	ND	29.42%	8.70%	61.87%	
625547-7B	02232021-7	0.00000191%	0.00000764%	ND	ND	< 0.00001%	ND	29.44%	9.42%	61.15%	
625547-7C	02232021-7	0.00000244%	0.00000975%	ND	ND	< 0.00001%	ND	29.46%	9.14%	61.40%	
625547-8A	02232021-8	0.00000245%	0.00000981%	ND	ND	< 0.00001%	ND	17.16%	9.98%	72.85%	
625547-8B	02232021-8	0.00000242%	0.00000967%	ND	ND	< 0.00001%	ND	17.09%	9.68%	73.22%	
625547-8C	02232021-8	0.00000283%	0.00001131%	ND	ND	< 0.00001%	ND	17.10%	10.54%	72.36%	
625547-9A	02232021-9	0.00000214%	0.00000855%	ND	ND	< 0.00001%	ND	4.42%	9.25%	86.33%	
625547-9B	02232021-9	0.00000324%	0.00001297%	ND	ND	< 0.00001%	ND	4.34%	10.60%	85.06%	
625547-9C	02232021-9	0.00000268%	0.00001071%	ND	ND	< 0.00001%	ND	4.35%	9.49%	86.16%	
625547-10A	02232021-10	0.00000216%	0.00000865%	ND	ND	< 0.00001%	ND	11.38%	9.89%	78.73%	
625547-10B	02232021-10	0.0000197%	0.0000787%	ND	ND	< 0.00001%	ND	11.31%	8.61%	80.08%	
625547-10C	02232021-10	0.00000205%	0.0000818%	ND	ND	< 0.00001%	ND	11.34%	8.71%	79.95%	
625547-11A	02232021-11	0.00000237%	0.0000949%	ND	ND	< 0.00001%	ND	14.61%	9.25%	76.15%	
625547-11B	02232021-11	0.00000197%	0.0000788%	ND	ND	< 0.00001%	ND	14.57%	7.77%	77.66%	
625547-11C	02232021-11	0.00000195%	0.00000779%	ND	ND	< 0.00001%	ND	14.57%	7.48%	77.95%	

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



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Revised: 5/17/2021 (Revision #3)

SUMMARY OF ANALYSIS

TEM LOD

Using ASTM D5756 Mass Calculation

TEM LOQ Using ASTM D5756 Mass Calculation

% Chrysotile by TEM Using ASTM D5756

Mass Calculation

% Tremolite by TEM Using ASTM D5756 Mass Calculation

Tremolite by TEM Using ASTM D5756 Mass Calculation

% Total Chrysotile &

Asbestos by PLM

% Acid Organics Soluable

Comments

Analyst(s):

TEM

Andreas Saldivar

Technical Director: Andreas Saldivar

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

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625547-1A, 1B, 1C/02232021-1











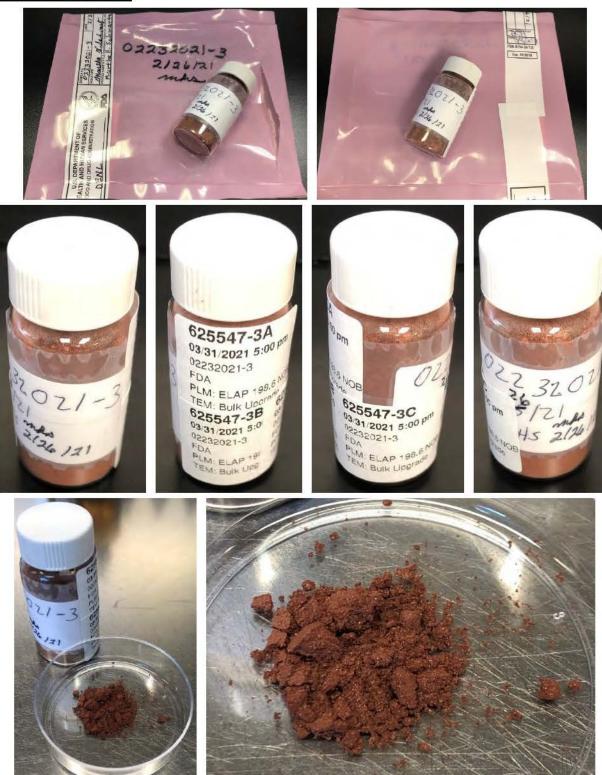




625547-2A, 2B, 2C/02232021-2



625547-3A, 3B, 3C/02232021-3



625547-4A, 4B, 4C/02232021-4



625547-5A, 5B, 5C/02232021-5







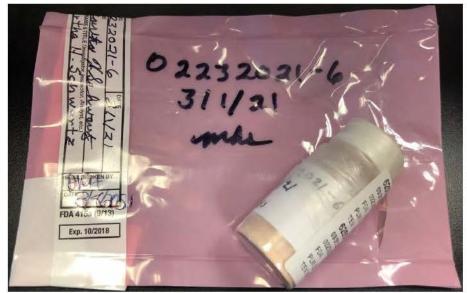






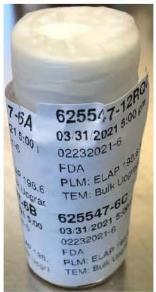


625547-6A, 6B, 6C/02232021-6



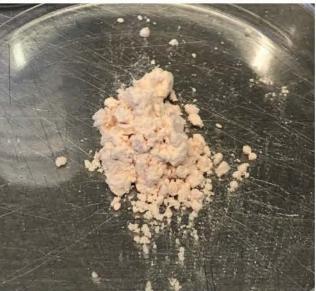












625547-7A, 7B, 7C/02232021-7



625547-8A, 8B, 8C/02232021-8



625547-9A, 9B, 9C/02232021-9



625547-10A, 10B, 10C/02232021-10



625547-11A, 11B, 11C/02232021-11



Re: FDA Office of Cosmetics & Colors Case Narrative for COC 625547

Sample Preparation

Samples were gravimetrically reduced and filtered by (b)(6) no. March 22, 2021 through March 24, 2021 for samples 625547-1A through 625547-4C and NB21-210; on March 25, 2021 through March 29, 2021 for samples 625547-5A through 625547-8C, 625547-12RQC and NB21-217; and on April 12, 2021 through April 14, 2021 for samples 625547-9A through 625547-11C, 625547-13RQC and NB21-257. PLM slide preparations were made by (b)(6) on April 13, 2021. TEM grid preparations were made by (b)(6) on: March 25, 2021 for samples 625547-1A through 625547-4C and NB21-210; on March 31, 2021 for samples 625547-5A through 625547-8C, 625547-12RQC and NB21-217; and on April 14, 2021 for samples 625547-9A through 625547-11C, 625547-13RQC and NB21-257. Sample preparation consisted of the following steps:

- 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 the corresponding 8mL glass vial. Record weight.
- 3) Burn samples at 480° C for at least 12-hours.
- Record Post-Ash weight.
- 5) Treat ashed sample with reagent grade hydrochloric acid.
- Filter acid reduced material with a pre-weighed disposable filtration apparatus onto a 47mm 0.4μm PolyCarbonate filter.
- Place disposable filtration apparatus with filter into drying oven for 3 hours and then record Post-Acid Reduced weight.
- 8) Make four PLM slide preparations from the PLM residue for each sample in 1.550 dispersion oil. Make additional preparations in 1.605, 1.625, 1.680 and 1.700 dispersion oil(s) as necessary for particle identification.
- Weigh a portion of the material from the TEM residue and place it into the corresponding pre-weighed 100mL jar.
- 10) Fill the 100mL jar with deionized water
- 11) Sonicate the jar for ~5-minutes.
- 12) Filter 0.1mL to 2mL of the solution onto a 47mm 0.22μm MCE filter.
- 13) Dry the filter for ~10-minutes then collapse, carbon coat, and place on a 3 TEM grids.

TEM grid preparations were examined prior to analysis and were rejected if they met the following criteria:

- 1) Less than 50% of the carbon coating was intact
- 2) The grid was too dark due to incomplete dissolution of the filter
- 3) Heavy particulate loading in excess of 25%
- 4) Light particulate loading below 10%
- 5) Uneven distribution of particulate

Problems Encountered During Preparation & Resolutions:

No problems were encountered during sample preparation. All gravimetric data was consistent among each group of aliquots and all TEM grid preparations were deemed acceptable for analysis.

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; each slide preparation consisted of two (2) coverslips for a total of eight (8) coverslips. 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.

Point Counting

If asbestos was observed on the slide preparations, the amount of asbestos was quantified using point count techniques. Point counting is form of quantifying PLM samples. One of the oculars of each PLM microscope is etched with a crosshair. When point counting, whatever is under the crosshair is counted as one point of whatever the material is. Four (4) slide preparations with a total of eight (8) coverslips are prepared for each sample. The microscope mechanical



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stage is used to randomly move the slide. After each movement, whatever is under the crosshair, provided the point is not empty, is counted. Fifty (50) non-empty points are counted on each of the eight (8) coverslips for a total of four hundred (400) points. The total asbestos points counted are divided by the total points counted to calculate the percentage.

Example:

11 points of asbestos were counted out of the 400 total points

Slide percentage = (11pts/400pts) * 100%

Slide percentage = 2.75%

This number is not the final asbestos percentage. To calculate the final percentage, this number must be corrected to account for the material lost during gravimetric reduction preparation. See the *Calculations* section below for additional details.

TEM Analysis

Analysis was performed in accordance with modified NY ELAP Method 198.4 protocols. The analysis was performed using JEOL JEM-100CX II and JEOL JEM-100CX transmission electron microscopes (TEM) equipped with Thermo Fisher NSS System 7 Energy Dispersive X-Ray Analyzers (EDXA), at magnifications of 19,000x. All TEM scopes are equipped with a Selective Area Electron Diffraction (SAED) setting that allows the operator to view the diffraction pattern of any mineral substance. Twenty (20) grid openings over two (2) grids were examined for each aliquot.

Modifications to the NY ELAP 198.4 Method were:

- 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 0.22μm MCE filter.
- 2) Any amphibole or chrysotile particle(s) observed were not quantified by visual estimation. The length and width of the observed particle(s) were measured, and the mass of each amphibole and chrysotile particle was calculated using the ASTM D5756 method.
- 3) All particles identified as amphibole 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

TEM ASTM D5756 Mass: Gravimetric Reduction Loss Percentages:

 $M = \pi/4 L * W^2 * D * 10^{-12}$ Organic: ((W1 - W2) * 100)/W1 Where: M: Mass Acid Soluble: ((W2 - W3) * 100)/W1

L: Length Other* Percent: ((W3/W1) * 100) – Calculated Asbestos %
W: Width *Other is defined as the non-asbestos, inorganic, acid insoluble portion of the sample

D: Density Where: W1: Weight of sample prior to ashing/acid wash

W2: Weight of sample after ashing

W3: Weight of sample after acid treatment

Asbestos Percent Calculation:

TEM PLM

 $EFA(mm^2) * 100ml * MA(g) * RW(g)$ (ASB * W3)/W1

VF(mI) * IW(g) * AA(mm²) * RJ(g)

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

Where: EFA: Effective filter area Where: W1: Weight of sample prior to ashing/acid wash

MA: Mass of asbestos W3: Weight of sample after acid treatment

RW: Weight of residue ASB: Calculated Point Count Result

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VF: Volume filtered

IW: Initial weight of the sample

AA: Area analyzed

RJ: Weight of residue placed into the jar

Note: All reported concentrations were calculated using the gravimetric data from the TEM preparations.

Limit of Detection and Quantification

We used the mass of a 0.5×0.04 -micron tremolite fiber as the basis for our calculations. Limit of detection (LOD) was defined as 1 fiber and limit of quantification (LOQ) was defined as 4 fibers.

Discussion and Interpretation of Analytical Findings:

625547-1A, 1B, 1C/Client Sample: 02232021-1

PLM

All three aliquots of sample 02232021-1 were analyzed by (b)(6) on April 23, 2021. No asbestos or non-asbestos amphibole variants were observed during analysis. The results were calculated using the equations detailed in the Calculations section above.

625547-1A	No Asbestos Detected
625547-1B	No Asbestos Detected
625547-1C	No Asbestos Detected

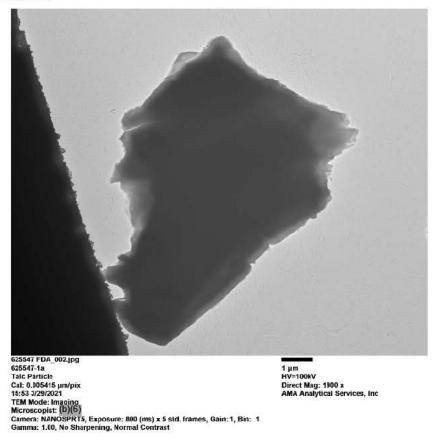
TEM

(b)(6) analyzed aliquot 1A March 29, 2021, and aliquots 1B and 1C on March 30, 2021. The primary particles observed were mica and talc; several particles containing titanium were also observed along with some scattered silica spheres and a few mica ribbons and talc fibers. No asbestos or non-asbestos amphibole variants were observed during analysis. The results were calculated using the equations detailed in the Calculations section above.

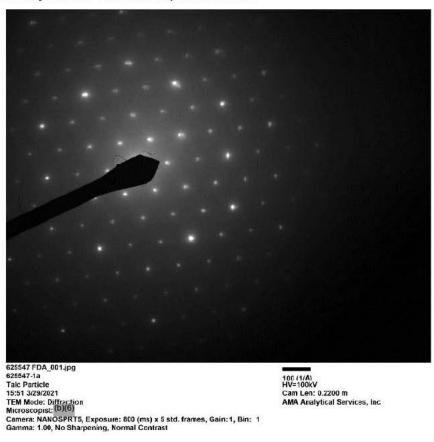
625547-1A	No Asbestos Detected
625547-1B	No Asbestos Detected
625547-1C	No Asbestos Detected

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

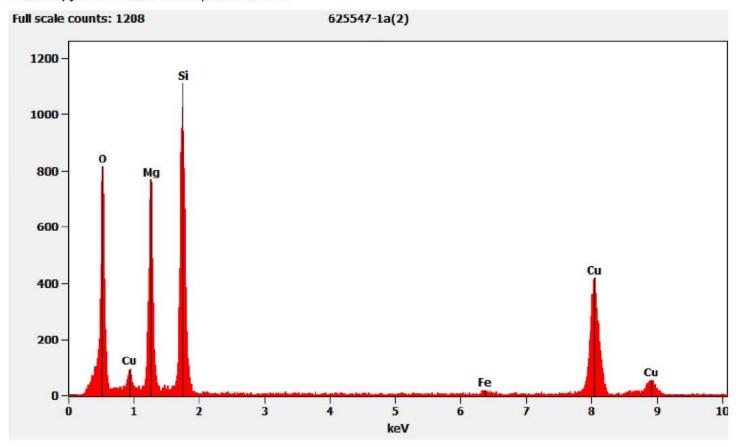
Sample 625547-1A, Talc Particle



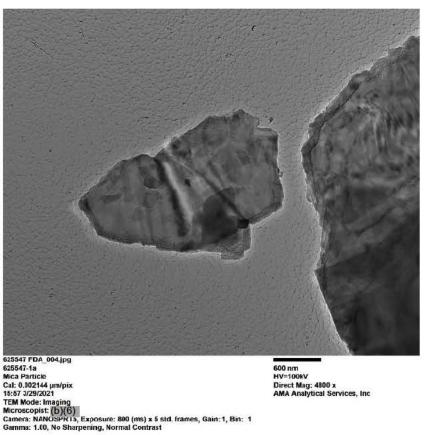
Hexagonal Diffraction Pattern from the Talc Particle pictured above



Chemistry from the Talc Particle pictured above



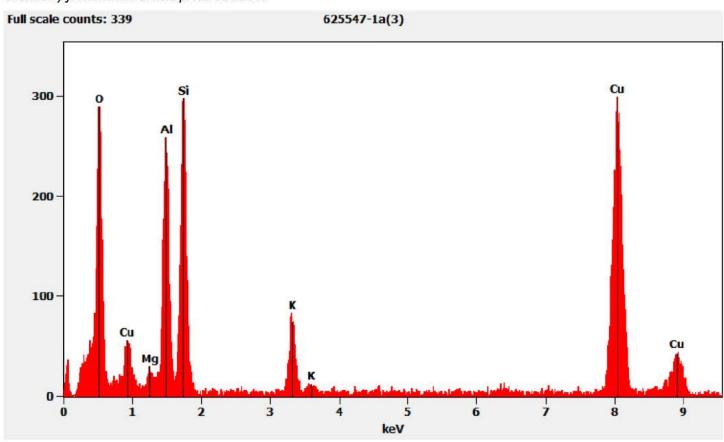
Sample 625547-1A, Mica Particle



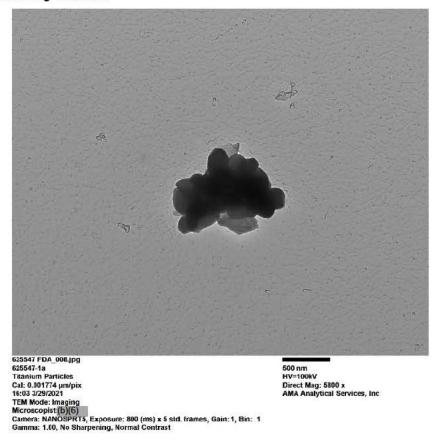
Hexagonal Diffraction Pattern from the Mica Particle pictured above



Chemistry from Mica Particle pictured above



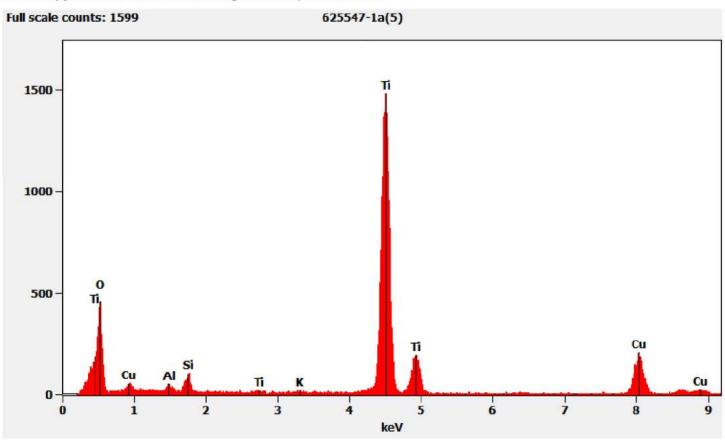
625547-1A, Particles Containing Titanium



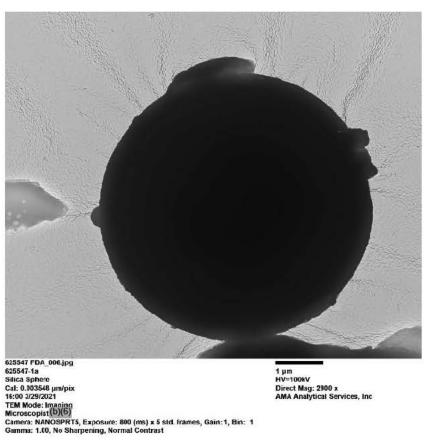
Diffraction Pattern from the Particles Containing Titanium pictured above



Chemistry from the Particles Containing Titanium pictured above



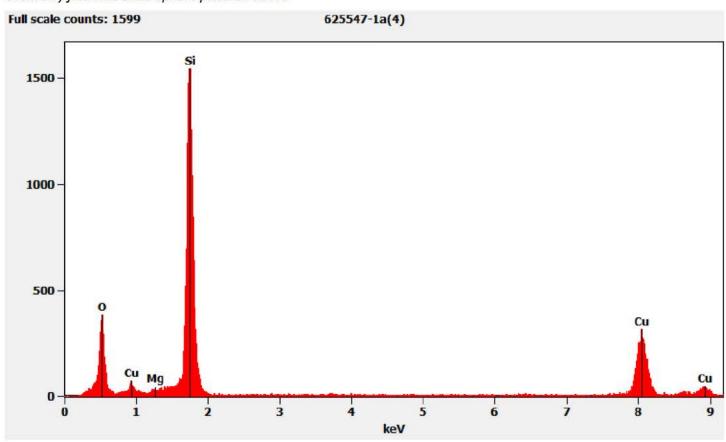
625547-1A, Silica Sphere



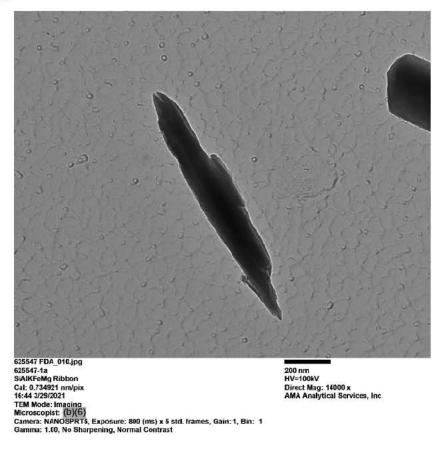
Diffraction Pattern from the Silica Sphere pictured above



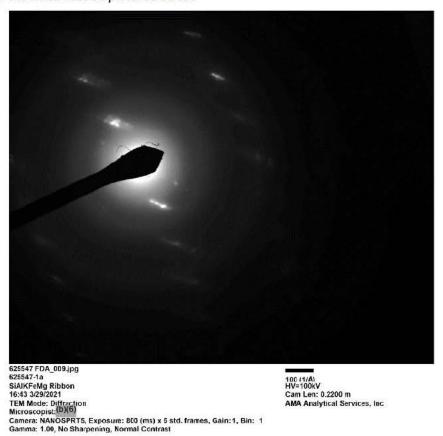
Chemistry from the Silica Sphere pictured above



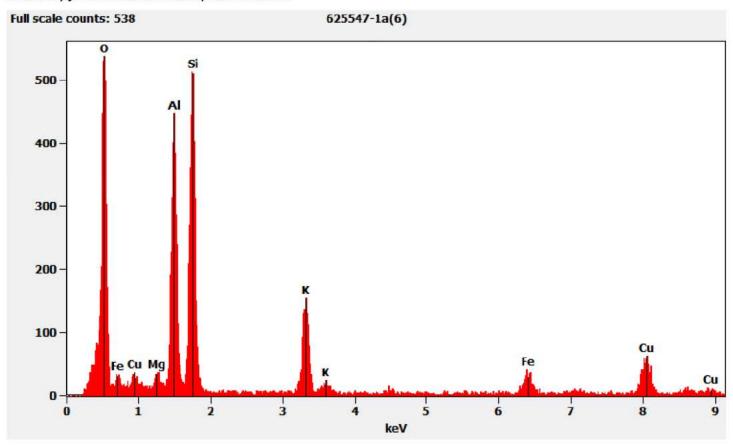
625547-1A, Mica Ribbon



Diffraction Pattern from the Mica Ribbon pictured above



Chemistry from the Mica Ribbon pictured above



625547-2A, 2B, 2C/Client Sample: 02232021-2

PLM

All three aliquots of sample 02232021-2 were analyzed by (b)(6) on April 23, 2021. No asbestos or non-asbestos amphibole variants were observed during analysis. The results were calculated using the equations detailed in the Calculations section above.

625547-2A	No Asbestos Detected
625547-2B	No Asbestos Detected
625547-2C	No Asbestos Detected

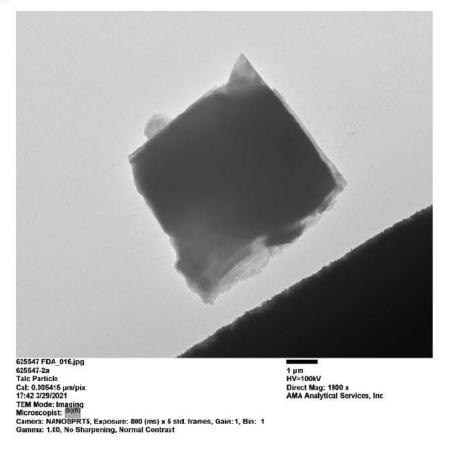
TEM

(b)(6) analyzed aliquot 2A on March 29, 2021 and aliquot 2C on March 30, 2021. (b)(6) analyzed aliquot 2B on March 30, 2021. The primary particles observed were talc and talc fibers; some talc ribbons were also observed. No asbestos or non-asbestos amphibole variants were observed during analysis. The results were calculated using the equations detailed in the Calculations section above.

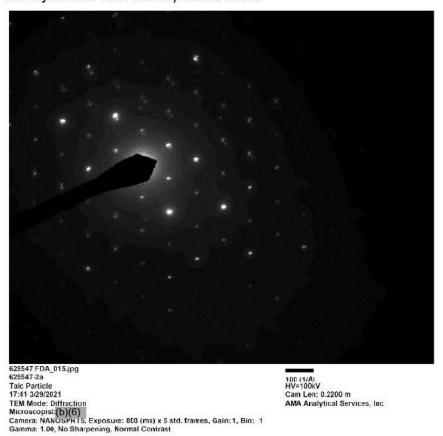
625547-2A	No Asbestos Detected
625547-2B	No Asbestos Detected
625547-2C	No Asbestos Detected

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

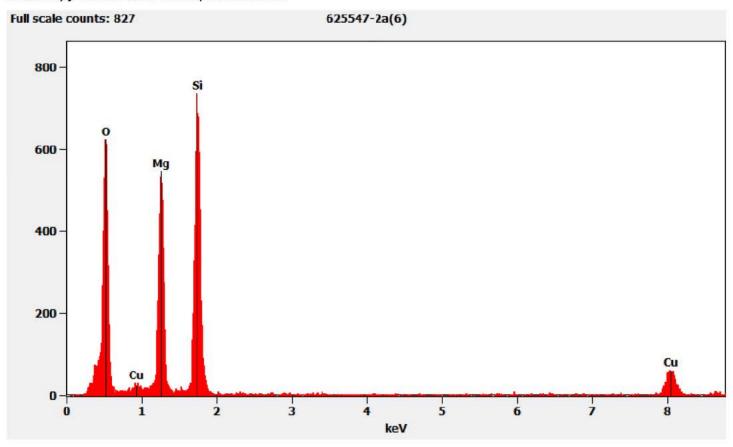
625547-2A, Talc Particle



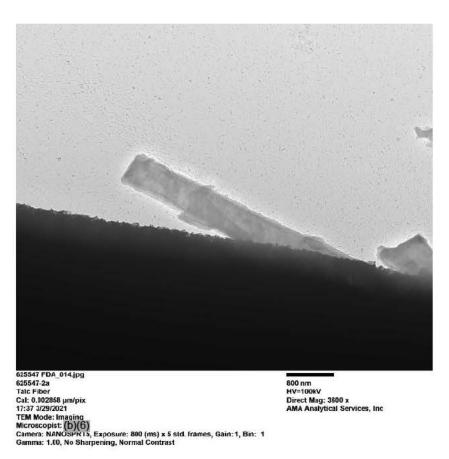
Hexagonal Diffraction Pattern from the Talc Particle pictured above



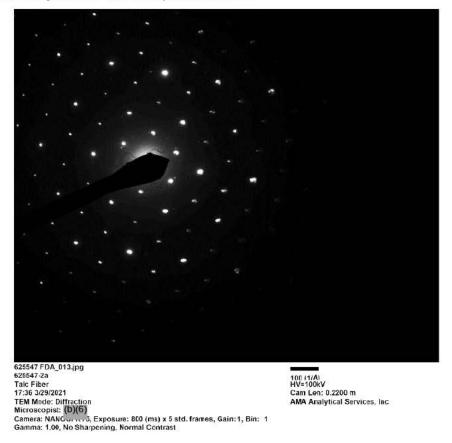
Chemistry from the Talc Particle pictured above



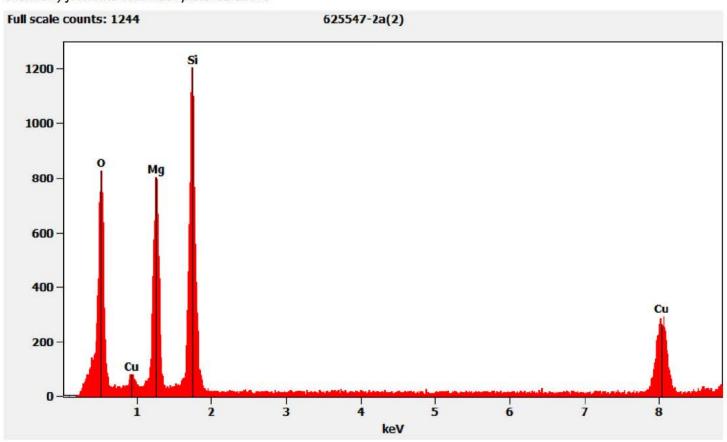
625547-2A, Talc Fiber



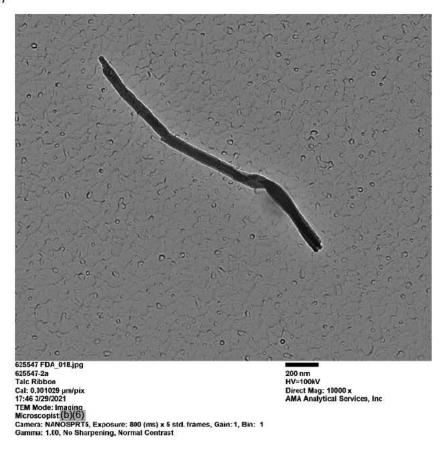
Hexagonal Diffraction Pattern from the Talc Fiber pictured above



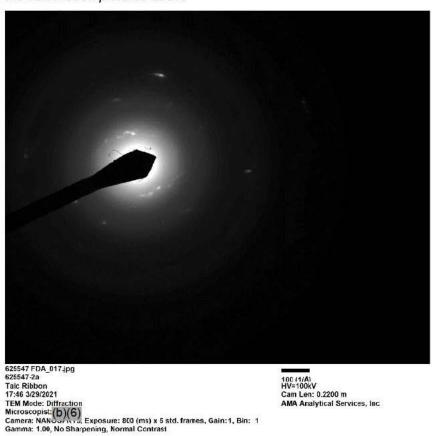
Chemistry from the Talc Fiber pictured above



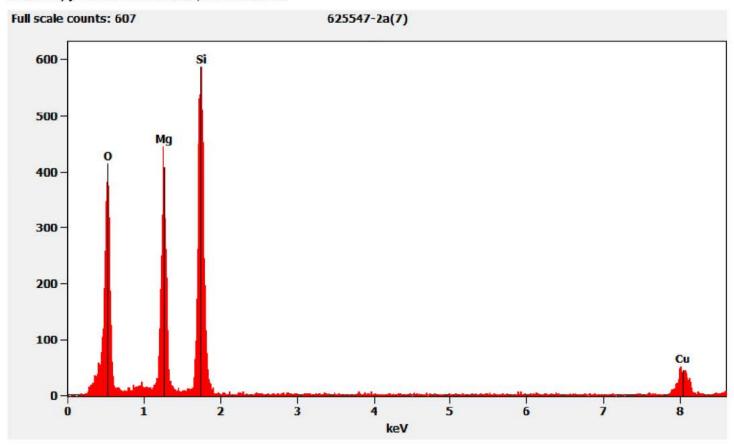
625547-2A, Talc Ribbon



Diffraction Pattern from the Talc Ribbon pictured above



Chemistry from the Talc Ribbon pictured above



625547-3A, 3B, 3C/Client Sample: 02232021-3

PLM

All three aliquots of sample 02232021-3 were analyzed by (b)(6) on April 23, 2021. No asbestos or non-asbestos amphibole variants were observed during analysis. The results were calculated using the equations detailed in the *Calculations* section above.

625547-3A	No Asbestos Detected
625547-3B	No Asbestos Detected
625547-3C	No Asbestos Detected

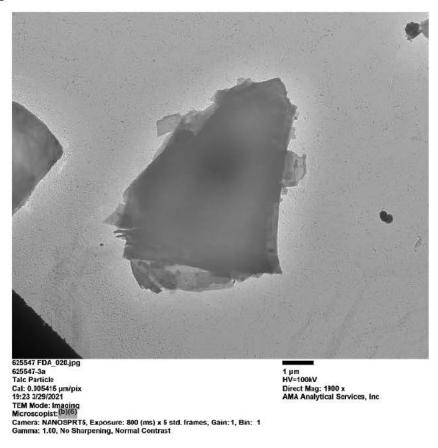
TEM

(b)(6) analyzed aliquot 3A on March 29, 2021 and aliquot 3B on March 30, 2021. The primary particles observed were talc and mica; several particles containing titanium were also observed along with a few talc ribbons, silica spheres, mica particles coated with iron, and magnesium aluminum silicate fibers. No asbestos or non-asbestos amphibole variants were observed during analysis. The results were calculated using the equations detailed in the Calculations section above.

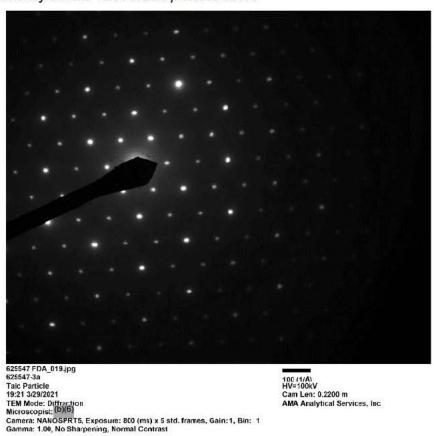
625547-3A	No Asbestos Detected
625547-3B	No Asbestos Detected
625547-3C	No Asbestos Detected

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

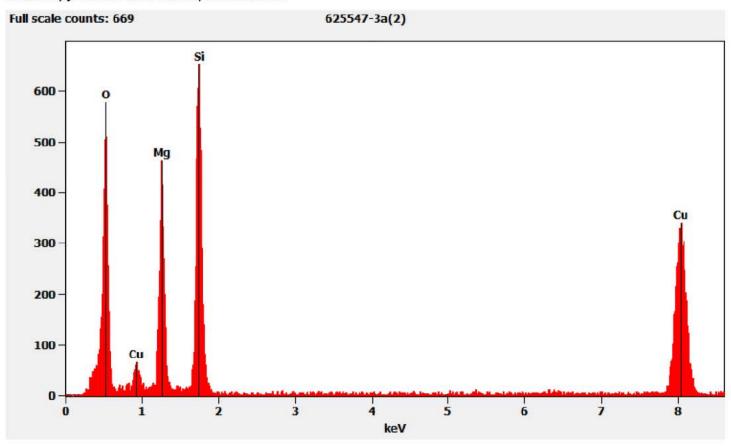
625547-3A, Talc Particle



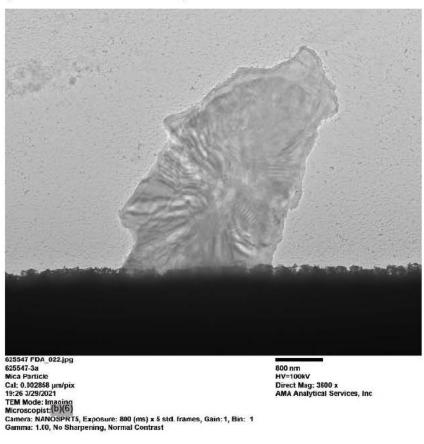
Hexagonal Diffraction Pattern from the Talc Particle pictured above



Chemistry from the Talc Particle pictured above



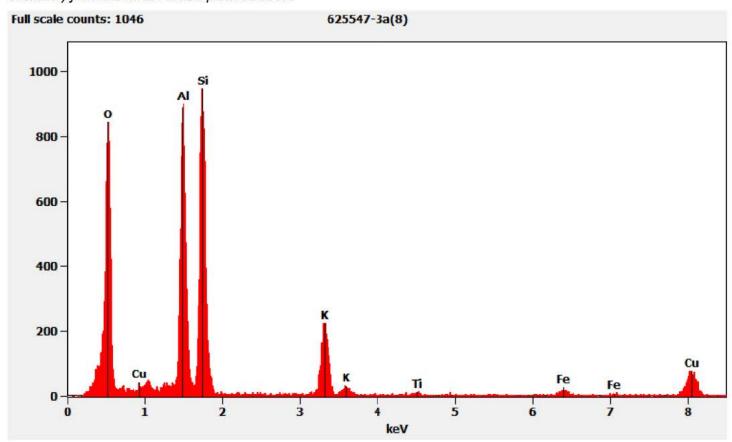
625547-3A, Mica Particle (Potassium Aluminum Silicate)



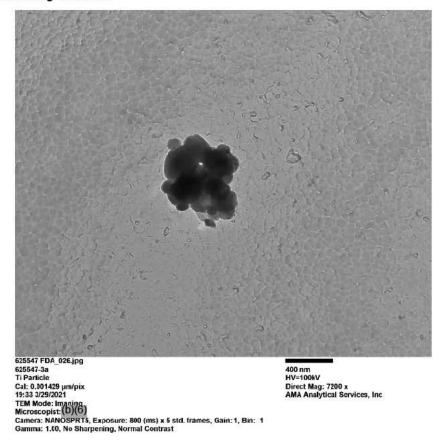
Hexagonal Diffraction Pattern from the Mica Particle pictured above



Chemistry from the Mica Particle pictured above



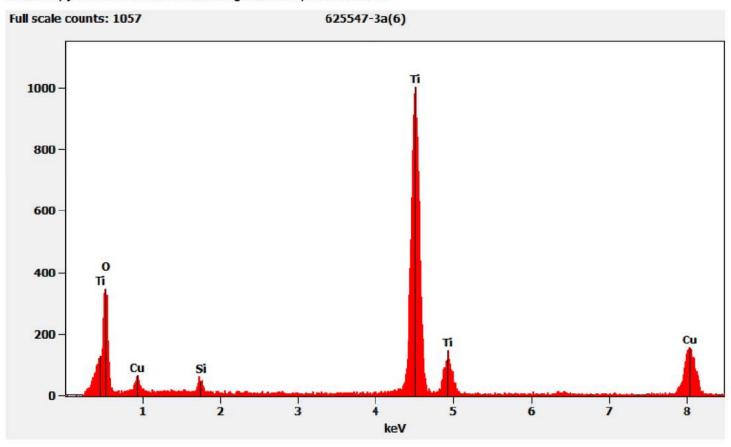
625547-3A, Particles Containing Titanium



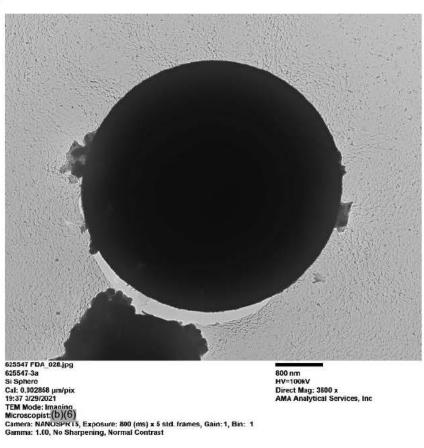
Diffraction Pattern from the Particles Containing Titanium pictured above



Chemistry from the Particles Containing Titanium pictured above



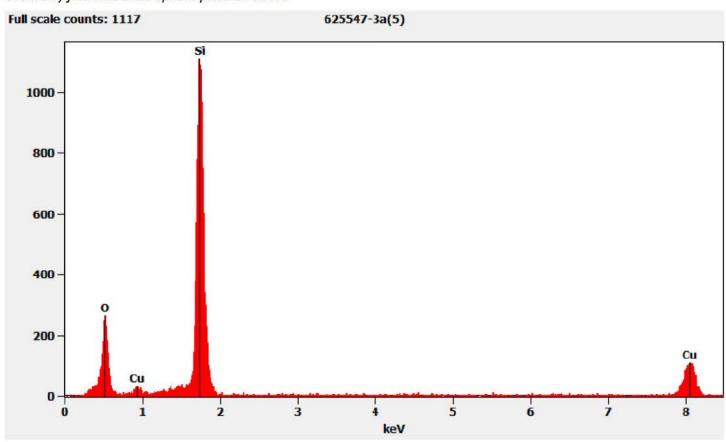
625547-3A, Silica Sphere



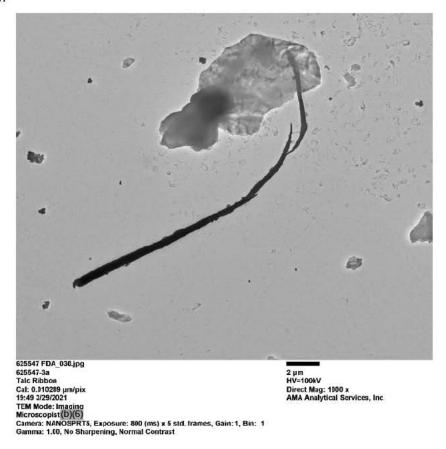
Diffraction Pattern from the Silica Sphere pictured above



Chemistry from the Silica Sphere pictured above



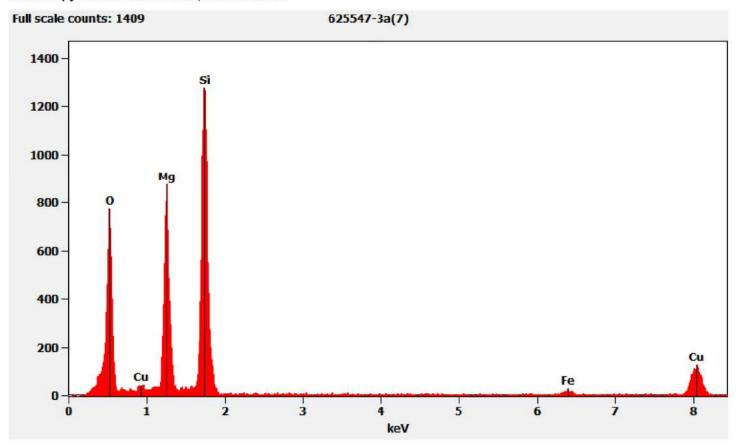
625547-3A, Talc Ribbon



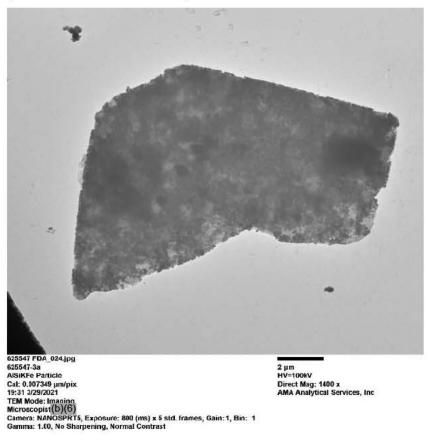
Diffraction Pattern from the Talc Ribbon pictured above



Chemistry from the Talc Ribbon pictured above



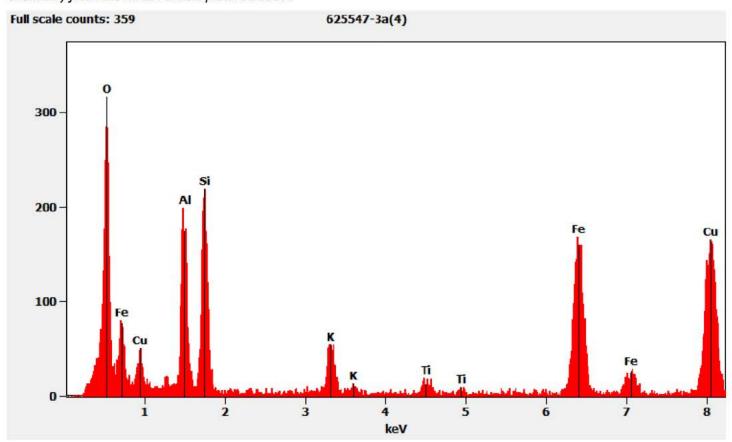
625547-3A, Mica Particle (Potassium Aluminum Silicate) coated with Iron Oxide



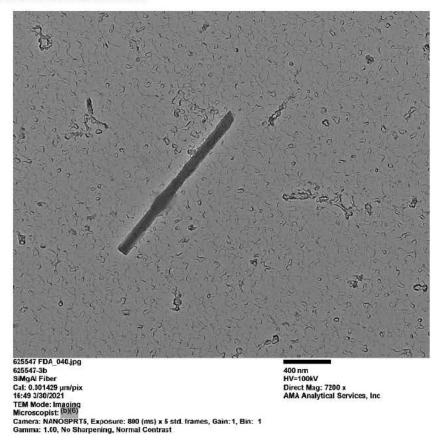
Diffraction Pattern from the Mica Particle pictured above



Chemistry from the Mica Particle pictured above



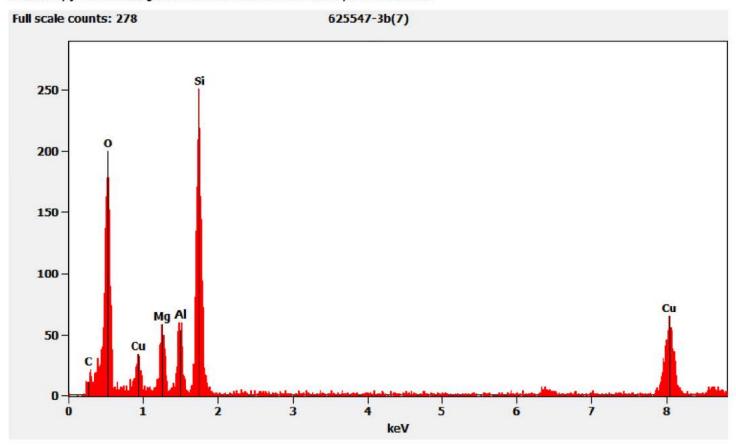
625547-3B, Magnesium Aluminum Silicate Fiber



Diffraction Pattern from the Magnesium Aluminum Silicate Fiber pictured above



Chemistry from the Magnesium Aluminum Silicate Fiber pictured above



625547-4A, 4B, 4C/Client Sample: 02232021-4

PLM

All three aliquots of sample 02232021-4 were analyzed by (b)(6) on April 23, 2021. No asbestos or non-asbestos amphibole variants were observed during analysis. The results were calculated using the equations detailed in the Calculations section above.

625547-4A	No Asbestos Detected
625547-4B	No Asbestos Detected
625547-4C	No Asbestos Detected

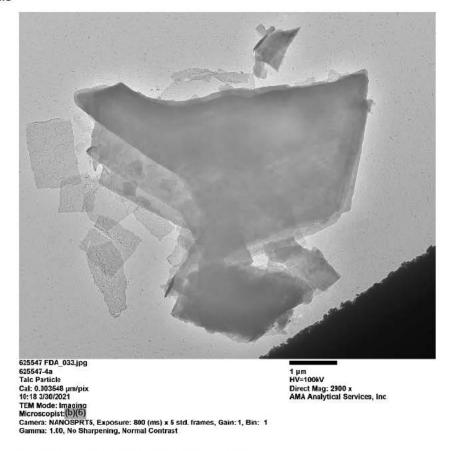
TEM

(b)(6) analyzed aliquot 2A on March 30, 2021 and aliquots 4B and 4C on March 31, 2021. The primary particle observed was talc; scattered silica particles were also observed as well as a few talc fibers. No asbestos or non-asbestos amphibole variants were observed during analysis. The results were calculated using the equations detailed in the Calculations section above.

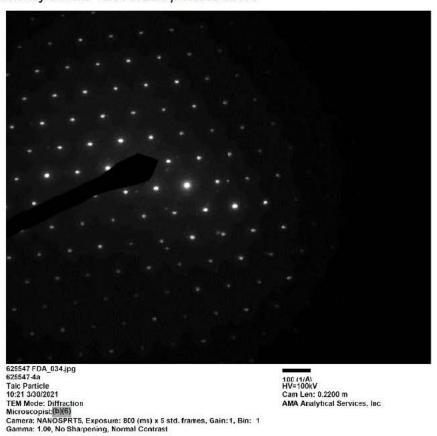
625547-4A	No Asbestos Detected
625547-4B	No Asbestos Detected
625547-4C	No Asbestos Detected

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

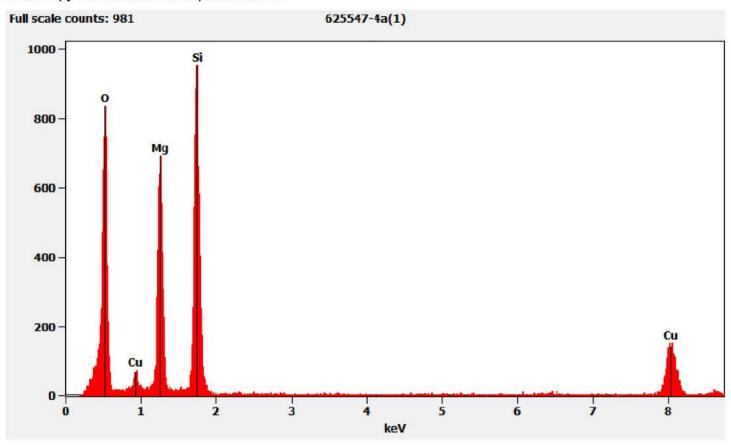
625547-4A, Talc Particle



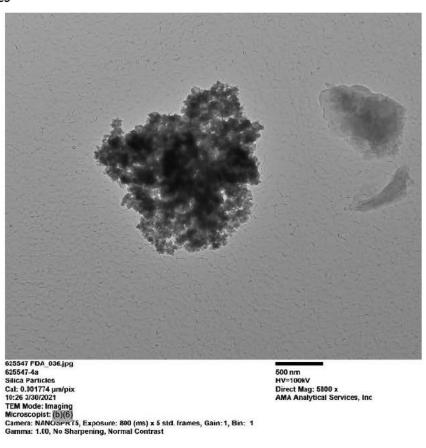
Hexagonal Diffraction Pattern from the Talc Particle pictured above



Chemistry from the Talc Particle pictured above



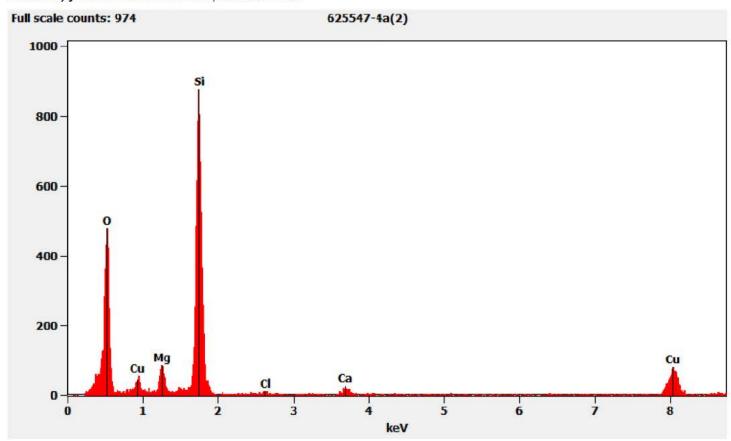
625547-4A, Silica Particles



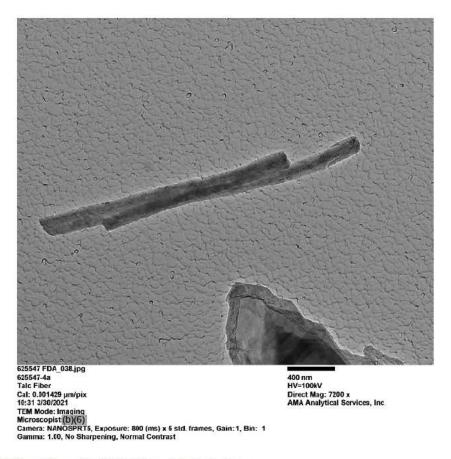
Diffraction Pattern from the Silica Particles pictured above



Chemistry from the Silica Particles pictured above



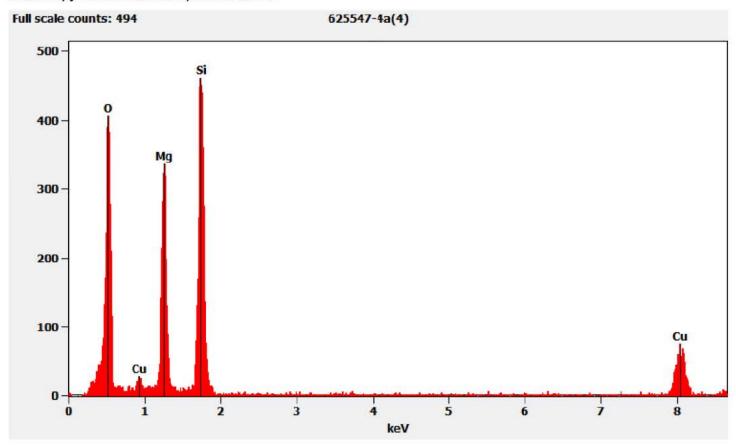
625547-4A, Talc Fiber



Hexagonal Diffraction Pattern from the Talc Fiber pictured above



Chemistry from the Talc Fiber pictured above



625547-5A, 5B, 5C/Client Sample: 02232021-5

PLM

All three aliquots of sample 02232021-5 were analyzed by (b)(6) on April 23, 2021. No asbestos or non-asbestos amphibole variants were observed during analysis. The results were calculated using the equations detailed in the Calculations section above.

625547-5A	No Asbestos Detected
625547-5B	No Asbestos Detected
625547-5C	No Asbestos Detected

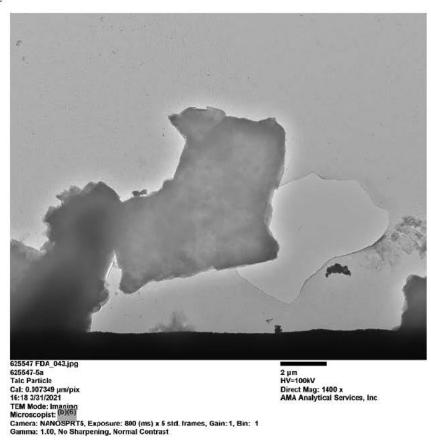
TEM

(b)(6) analyzed aliquot 5A on March 31, 2021. Andreas Saldivar analyzed aliquot 5B on April 2, 2021 and (b)(6) analyzed aliquot 5C on April 8, 2021. The primary particle observed was talc; several mica particles were also observed as well as particles containing titanium, scattered talc fibers/ribbons and a few barium sulfate particles. No asbestos or non-asbestos amphibole variants were observed during analysis. The results were calculated using the equations detailed in the Calculations section above.

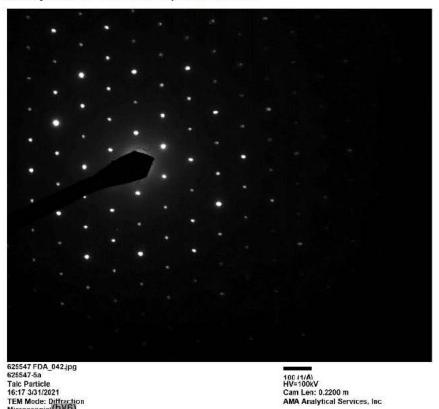
625547-5A No Asbestos Detected 625547-5B No Asbestos Detected 625547-5C No Asbestos Detected

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

625547-5A, Talc Particle

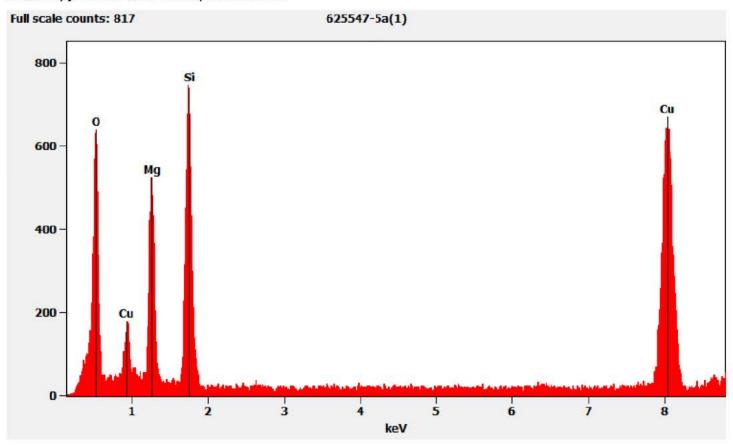


Hexagonal Diffraction Pattern from the Talc Particle pictured above

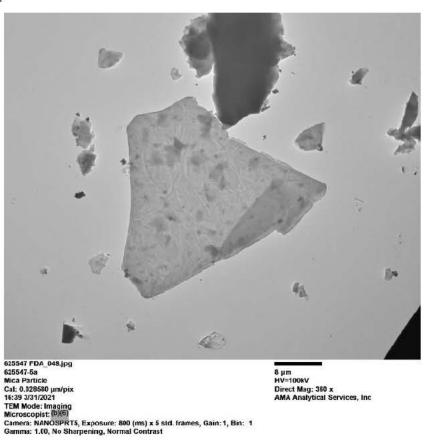


TEM Mode: Diffraction
Microscopist(b)(6)
Camera: NANOSPRT5, Exposure: 800 (ms) x 5 std. frames, Gain:1, Bin: 1
Gamma: 1.00, No Sharpening, Normal Contrast

Chemistry from the Talc Particle pictured above



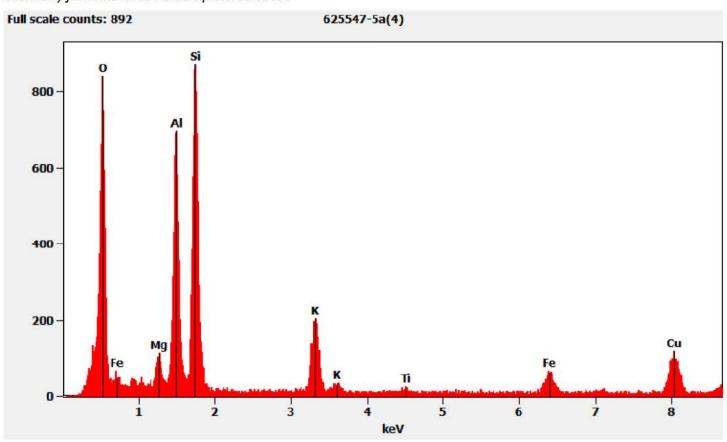
625547-5A, Mica Particle



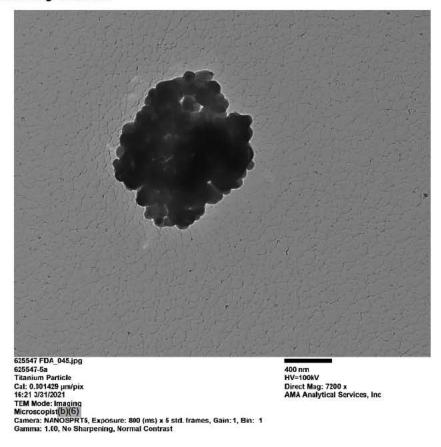
Hexagonal Diffraction Pattern from the Mica Particle pictured above



Chemistry from the Mica Particle pictured above



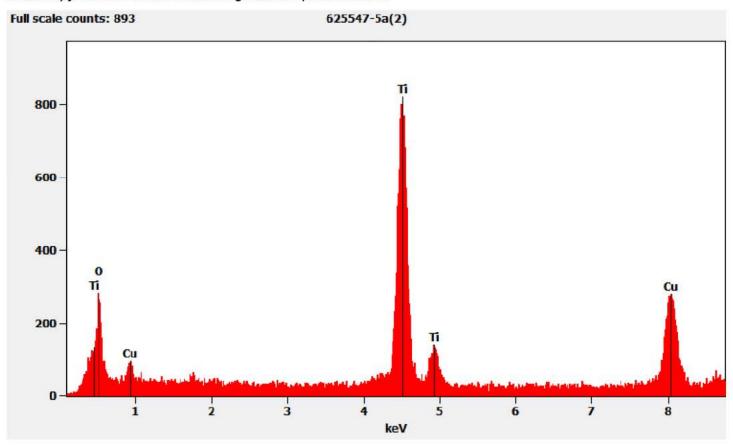
625547-5A, Particles Containing Titanium



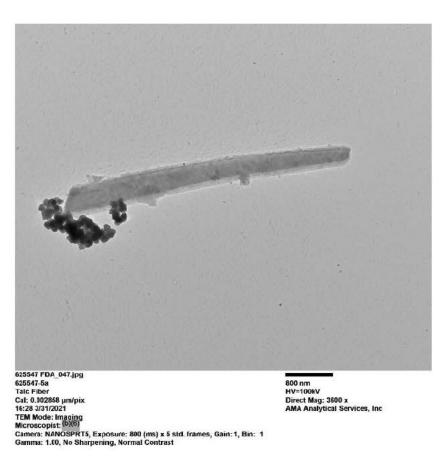
Diffraction Pattern from the Particles Containing Titanium pictured above



Chemistry from the Particles Containing Titanium pictured above



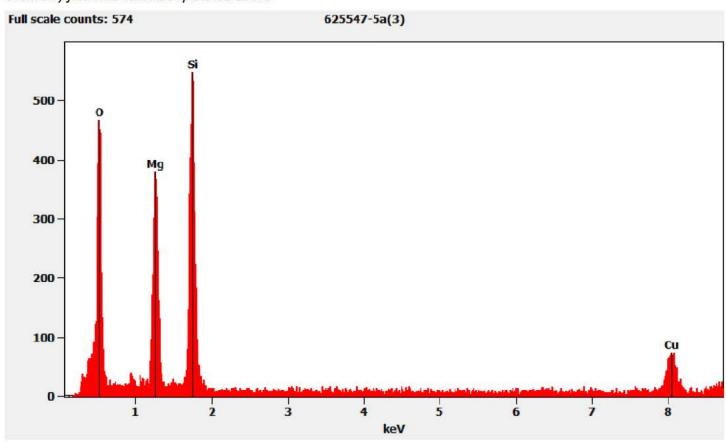
625547-5A, Talc Fiber



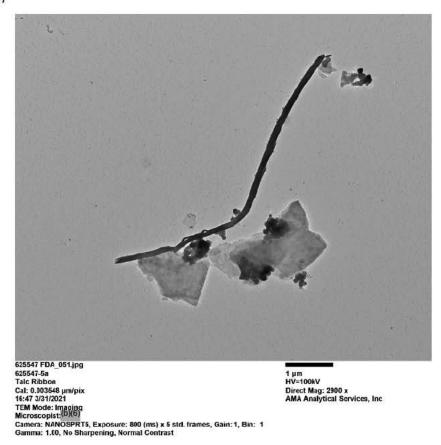
Hexagonal Diffraction Pattern from the Talc Fiber pictured above



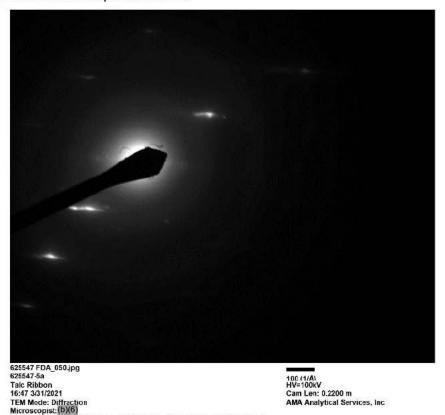
Chemistry from the Talc Fiber pictured above



625547-5A, Talc Ribbon

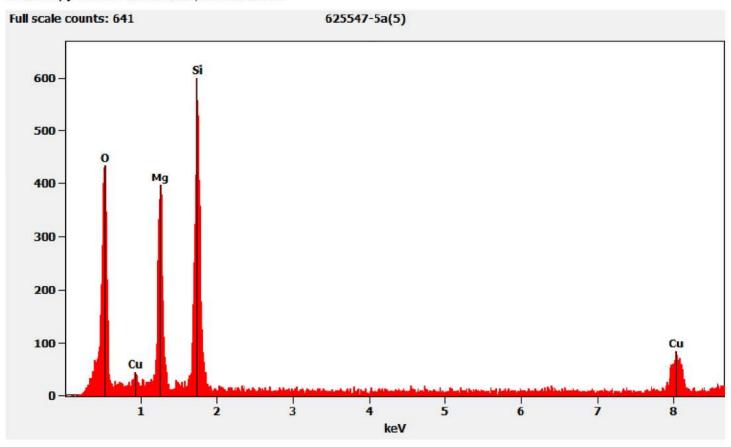


Diffraction Pattern from the Talc Ribbon pictured above

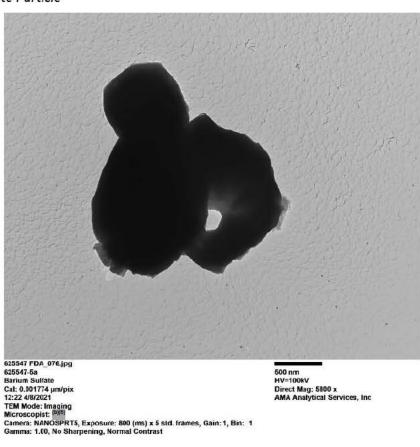


TEM Mode: Diffraction Microscopist (D)(6) Camera: NANOSPRT5, Exposure: 800 (ms) x 5 std. frames, Gain:1, Bin: 1 Gamma: 1.00, No Sharpening, Normal Contrast

Chemistry from the Talc Ribbon pictured above



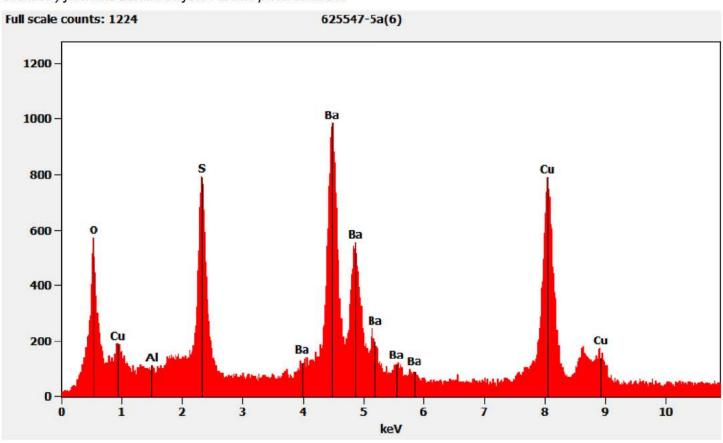
625547-5A, Barium Sulfate Particle



Diffraction Pattern from the Barium Sulfate Particle pictured above



Chemistry from the Barium Sulfate Particle pictured above



625547-6A, 6B, 6C/Client Sample: 02232021-6

PLM

All three aliquots of sample 02232021-6 were analyzed by (b)(6) on April 23, 2021. No asbestos or non-asbestos amphibole variants were observed during analysis. The results were calculated using the equations detailed in the Calculations section above.

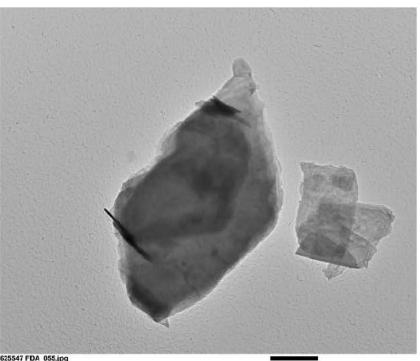
625547-6A	No Asbestos Detected
625547-6B	No Asbestos Detected
625547-6C	No Asbestos Detected

Andreas Saldivar analyzed aliquot 6C on April 9, 2021. The primary particles observed were talc and mica; silica spheres were also observed as well as a few talc fibers/ribbons. No asbestos or non-asbestos amphibole variants were observed during analysis. The results were calculated using the equations detailed in the Calculations section above.

625547-6A No Asbestos Detected 625547-6B No Asbestos Detected 625547-6C No Asbestos Detected

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

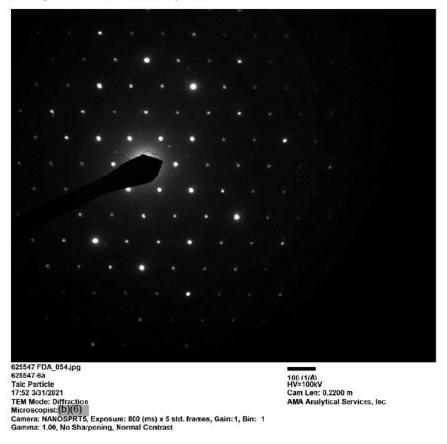
625547-6A, Talc Particle



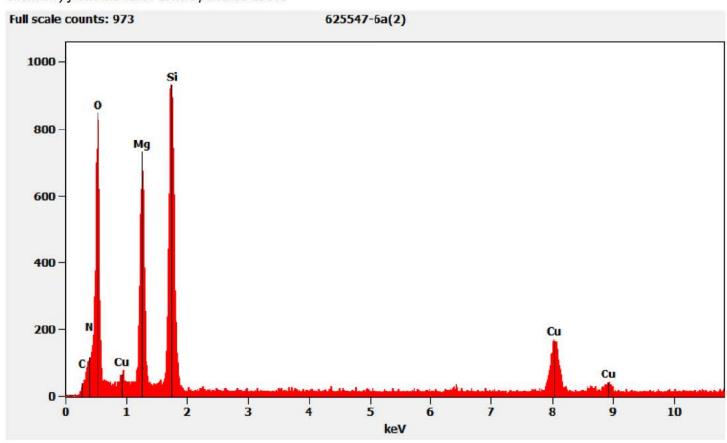
625547 FDA_055.jpg 625547-6a Talc Particle Cal: 0.002858 µm/pix 17:54 3/31/2021 TEM Mode: Imagino Microscopist:(D)(6)

Gamera: 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: 3800 x AMA Analytical Services, Inc

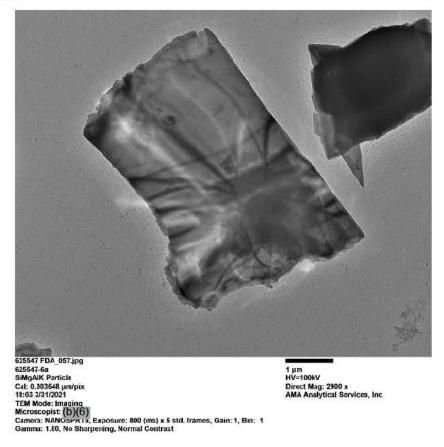
Hexagonal Diffraction Pattern from the Talc Particle pictured above



Chemistry from the Talc Particle pictured above



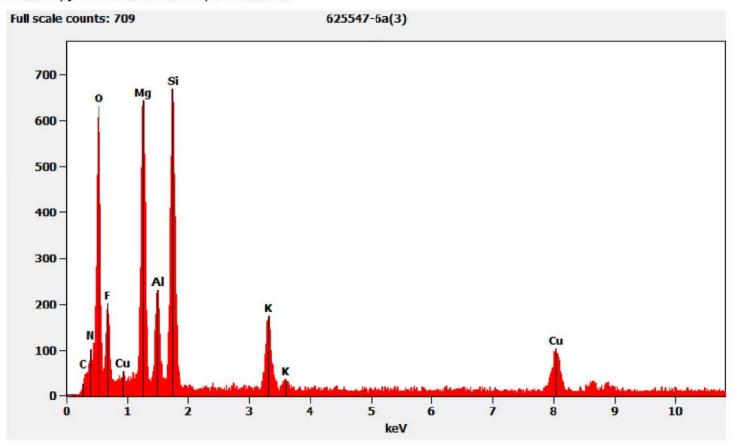
625547-6A, Mica Particle



Hexagonal Diffraction Pattern from the Mica Particle pictured above



Chemistry from the Mica Particle pictured above



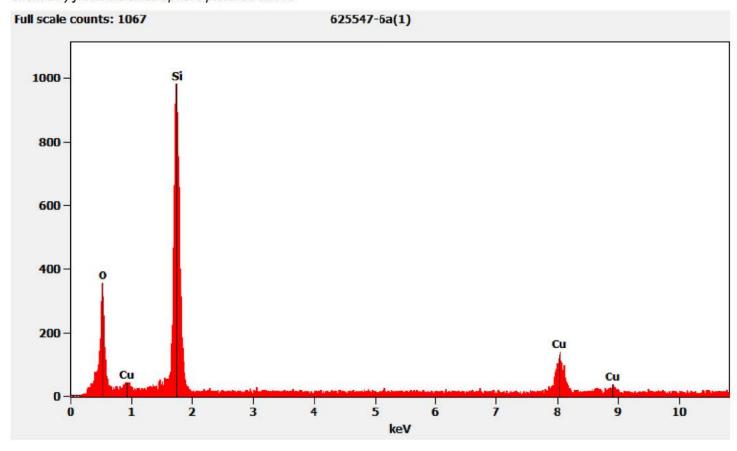
625547-6A, Silica Sphere



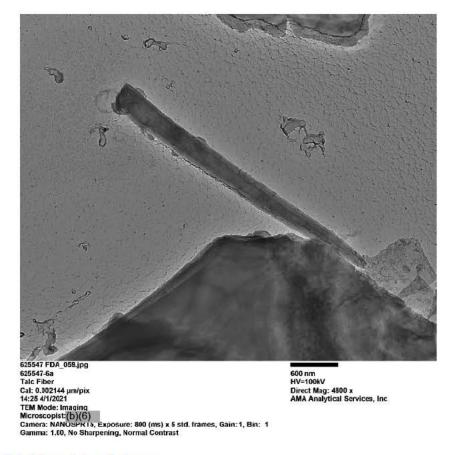
Diffraction Pattern from the Silica Sphere pictured above



Chemistry from the Silica Sphere pictured above



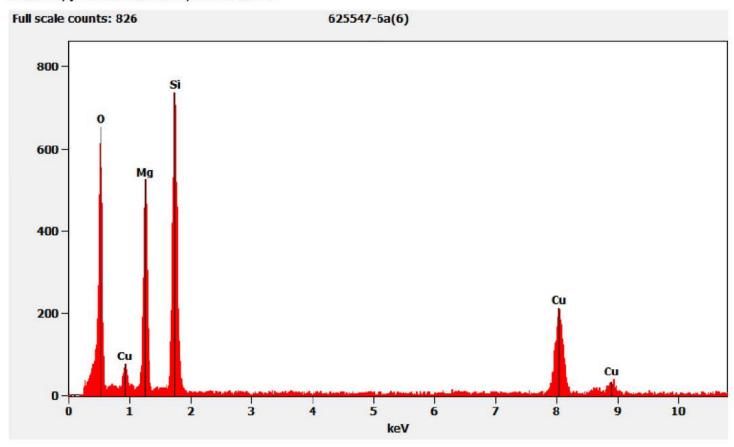
625547-6A, Talc Fiber



Hexagonal Diffraction Talc Fiber pictured above



Chemistry from the Talc Fiber pictured above



625547-7A, 7B, 7C/Client Sample: 02232021-7

PLM

All three aliquots of sample 02232021-7 were analyzed by (b)(6) on April 23, 2021. No asbestos or non-asbestos amphibole variants were observed during analysis. The results were calculated using the equations detailed in the *Calculations* section above.

625547-7A	No Asbestos Detected
625547-7B	No Asbestos Detected
625547-7C	No Asbestos Detected

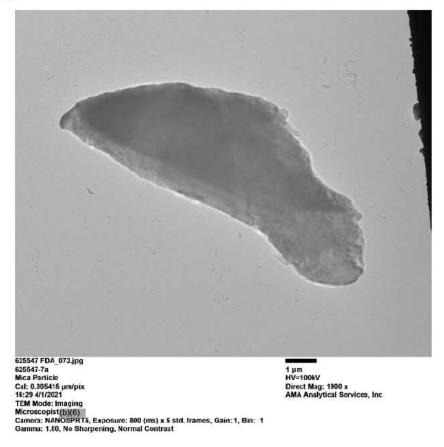
TEM

(b)(6) analyzed aliquot 7A on April 1, 2021 and aliquot 7C on April 9, 2021. Andreas Saldivar analyzed aliquot 7B on April 9, 2021. The primary particle observed was mica; particles containing titanium, potassium aluminum silicate and magnesium aluminum silicate particles were also observed as well as a few silica spheres, calcium phosphate particles and talc particles. No asbestos or non-asbestos amphibole variants were observed during analysis. The results were calculated using the equations detailed in the *Calculations* section above.

625547-7A	No Asbestos Detected
625547-7B	No Asbestos Detected
625547-7C	No Asbestos Detected

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

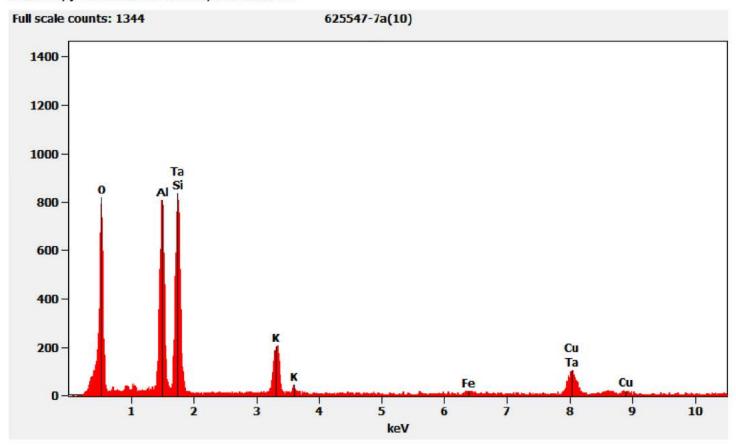
625547-7A, Mica Particle



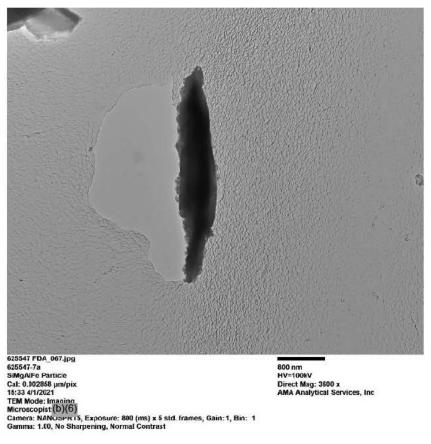
Hexagonal Diffraction Pattern from the Mica Particle pictured above



Chemistry from the Mica Particle pictured above



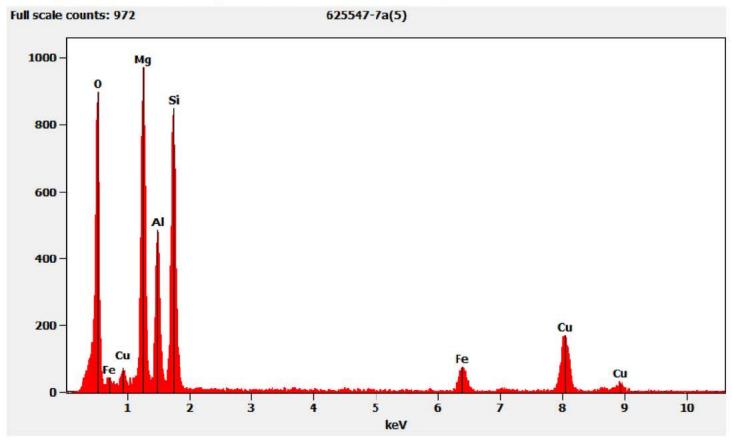
625547-7A, Magnesium Aluminum Silicate Particle coated with Iron Oxide



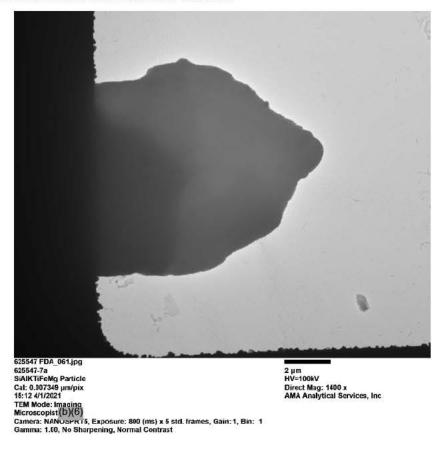
Hexagonal Diffraction Pattern from the Magnesium Aluminum Silicate Particle pictured above



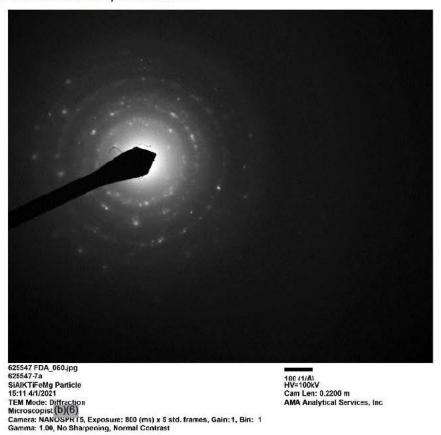
Chemistry from the Magnesium Aluminum Silicate Particle pictured above



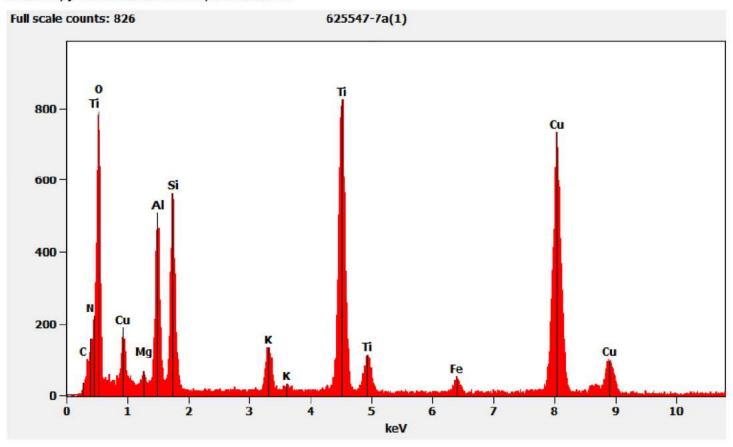
625547-7A, Mica Particle coated with Iron Oxide and Titanium



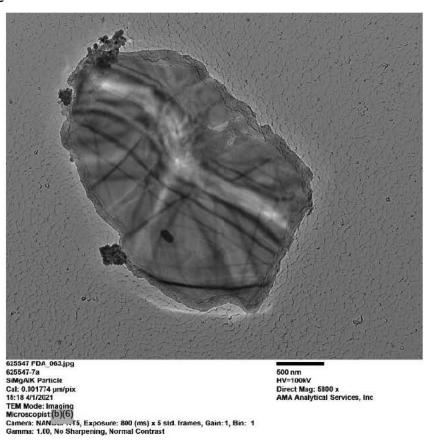
Diffraction Pattern from the Mica Particle pictured above



Chemistry from the Mica Particle pictured above



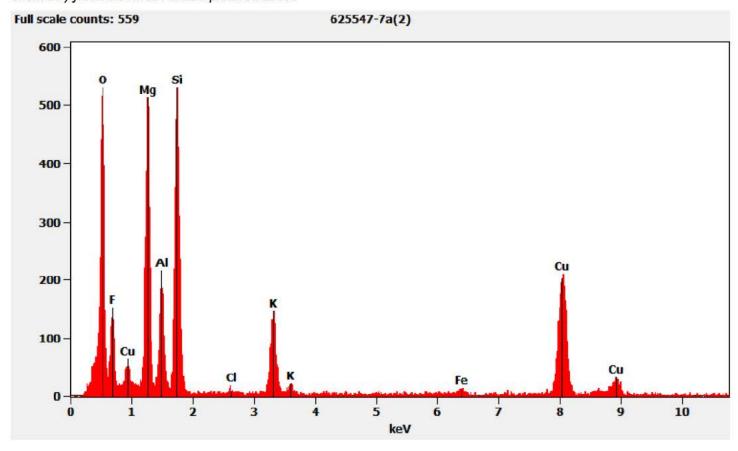
625547-7A, Mica Particle



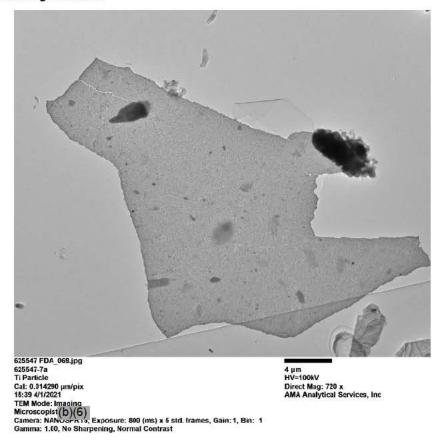
Hexagonal Diffraction Pattern from the Mica Particle pictured above



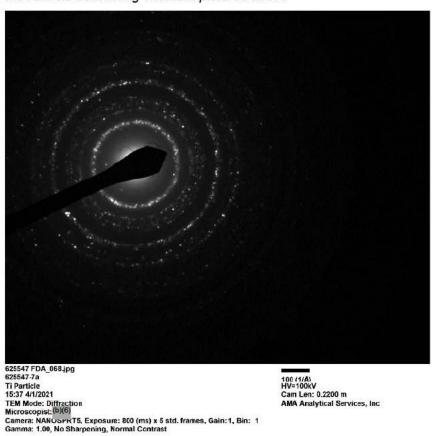
Chemistry from the Mica Particle pictured above



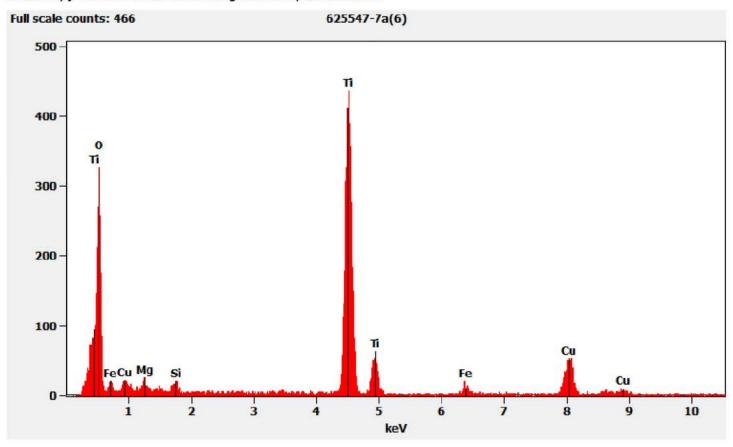
625547-7A, Particle Containing Titanium



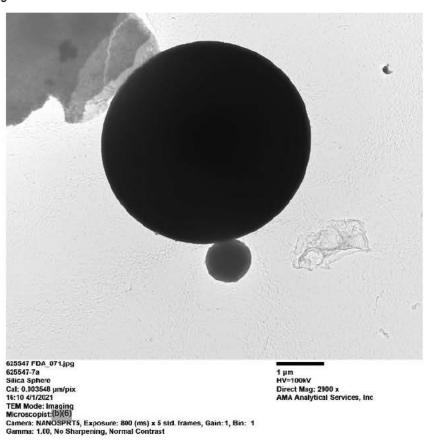
Diffraction Pattern from the Particle Containing Titanium pictured above



Chemistry from the Particle Containing Titanium pictured above



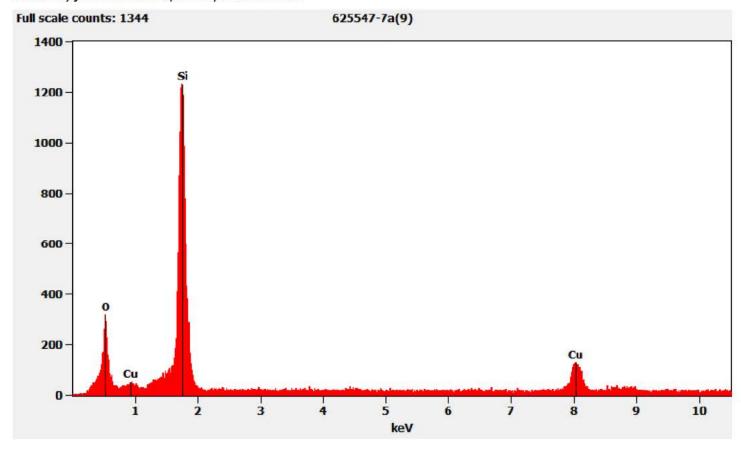
625547-7A, Silica Spheres



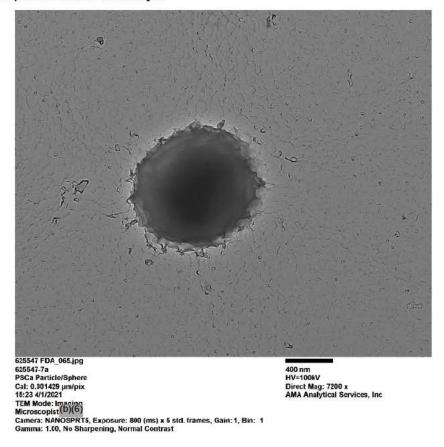
Diffraction Pattern from the Silica Spheres pictured above



Chemistry from the Silica Spheres pictured above



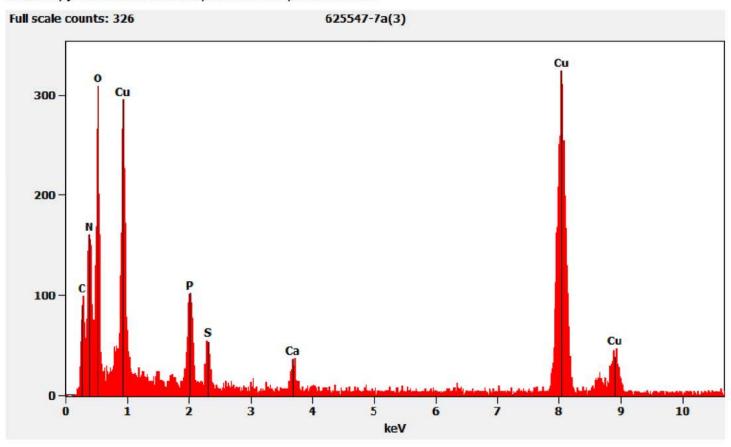
625547-7A, Calcium Phosphate Particle with Sulfur



Diffraction Pattern from the Calcium Phosphate Particle pictured above



Chemistry from the Calcium Phosphate Particle pictured above



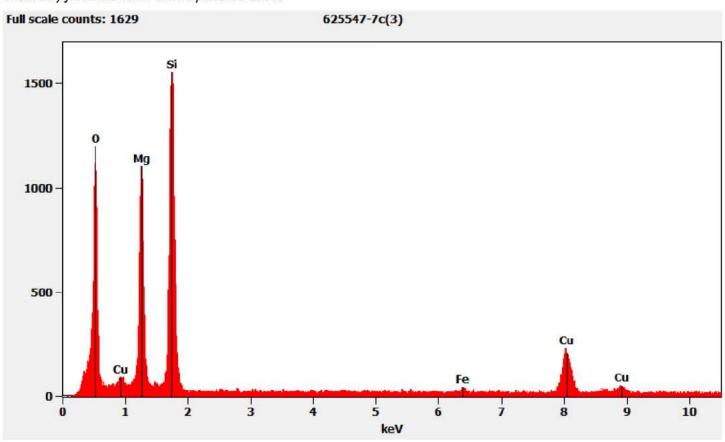
625547-7C, Talc Particle



Hexagonal Diffraction Pattern from the Talc Particle pictured above



Chemistry from the Talc Particle pictured above



625547-8A, 8B, 8C/Client Sample: 02232021-8

PLM

All three aliquots of sample 02232021-8 were analyzed by (b)(6) on April 23, 2021. No asbestos or nonasbestos amphibole variants were observed during analysis. The results were calculated using the equations detailed in the Calculations section above.

625547-8A 625547-8B	No Asbestos Detected	
	No Asbestos Detected	
625547-8C	No Asbestos Detected	

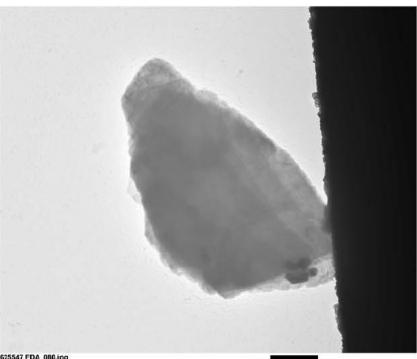
TEM

(b)(6)analyzed aliquot 8A on April 9, 2021. Andreas Saldivar analyzed aliquot 8B on April 13, 2021 and aliquot 8C on April 14, 2021. The primary particle observed was mica; talc and scattered particles containing titanium were also observed as well as a few silica spheres and talc ribbons. No asbestos or non-asbestos amphibole variants were observed during analysis. The results were calculated using the equations detailed in the Calculations section above.

625547-8A	No Asbestos Detected
625547-8B	No Asbestos Detected
625547-8C	No Asbestos Detected

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

625547-8A, Mica Particle



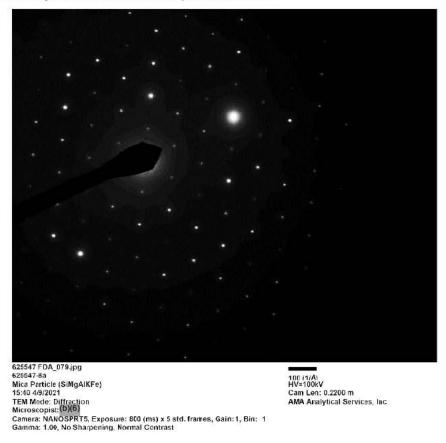
625547 FDA_080.jpg 625547-8a Mica Particle (SiMgAlKFe) Cal: 0.002858 µm/pix 15:41 4/9/2021 Microscopist: (b)(6)

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

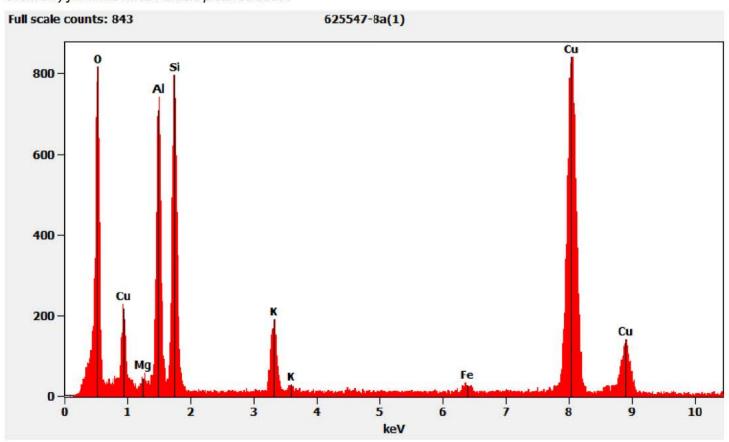
Gamma: 1.00, No Sharpening, Normal Contrast

HV=100kV Direct Mag: 3800 x AMA Analytical Services, Inc.

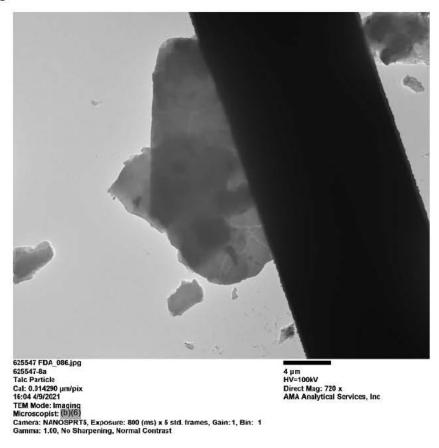
Hexagonal Diffraction Pattern from the Mica Particle pictured above



Chemistry from the Mica Particle pictured above



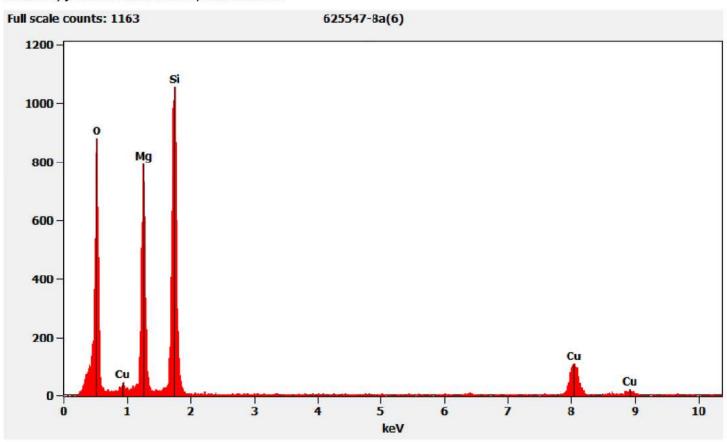
625547-8A, Talc Particle



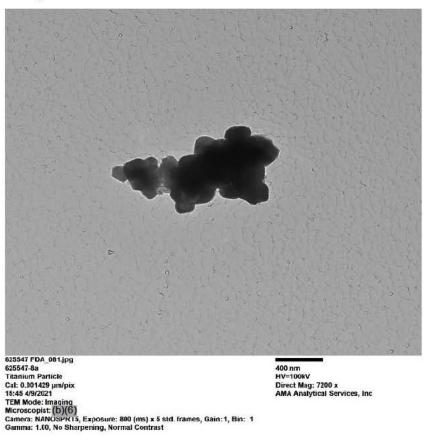
Hexagonal Diffraction Pattern from the Talc Particle pictured above



Chemistry from the Talc Particle pictured above



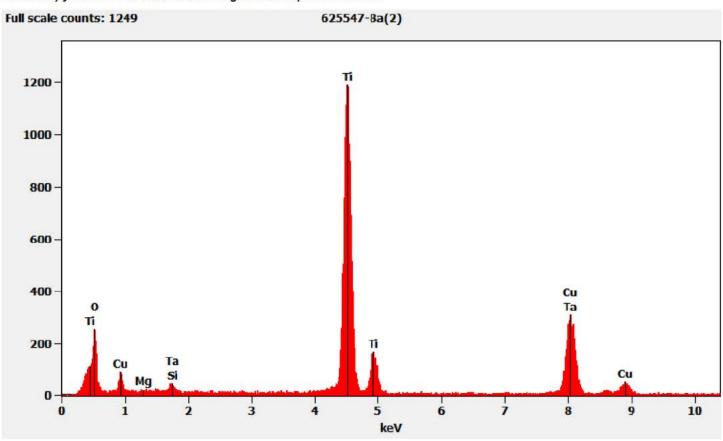
625547-8A, Particles Containing Titanium



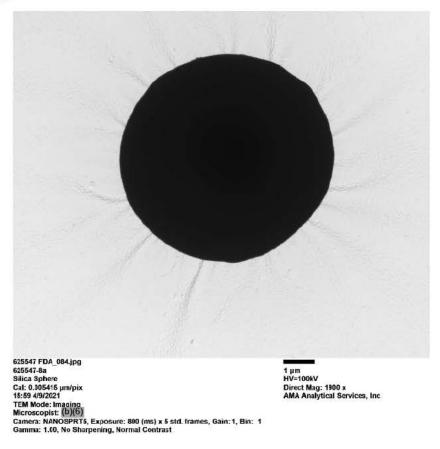
Diffraction Pattern from Particles Containing Titanium pictured above



Chemistry from the Particles Containing Titanium pictured above



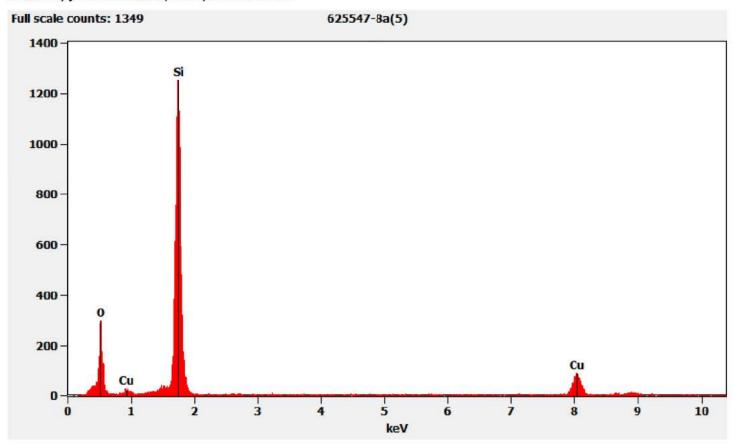
625547-8A, Silica Sphere



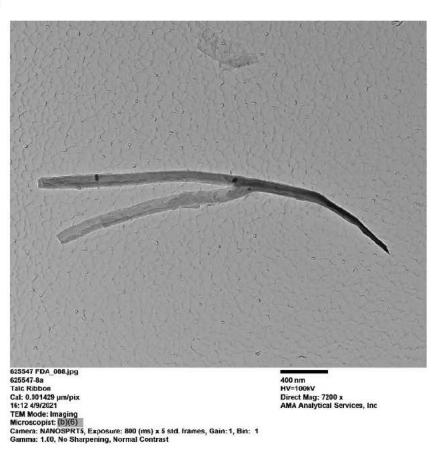
Diffraction Pattern from the Silica Sphere pictured above



Chemistry from the Silica Sphere pictured above



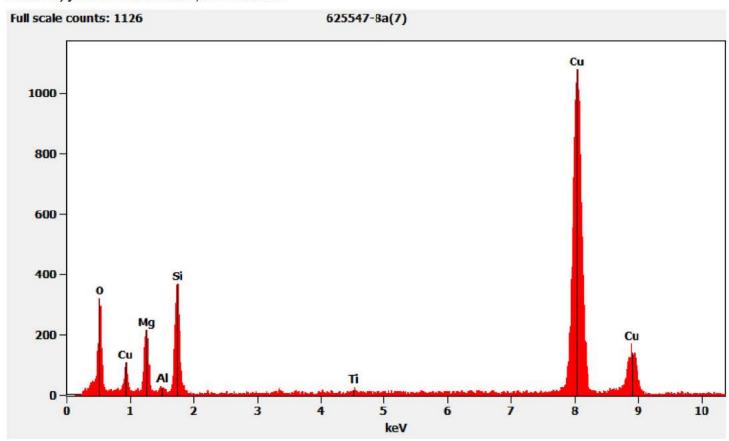
625547-8A, Talc Ribbon



Diffraction Pattern from the Talc Ribbon pictured above



Chemistry from the Talc Ribbon pictured above



625547-9A, 9B, 9C/Client Sample: 02232021-9

PLM

All three aliquots of sample 02232021-9 were analyzed by (b)(6)on April 23, 2021. No asbestos or nonasbestos amphibole variants were observed during analysis. The results were calculated using the equations detailed in the Calculations section above.

625547-9A	No Asbestos Detected		
625547-9B	No Asbestos Detected		
625547-9C	No Asbestos Detected		

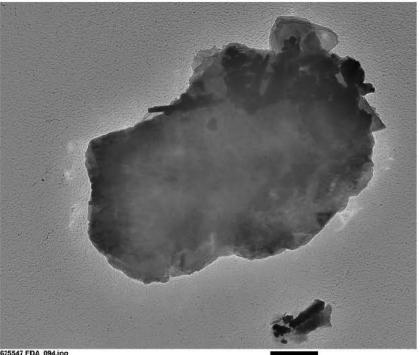
TEM

(b)(6)inalyzed aliquot 9A on April 20, 2021. (b)(6) analyzed aliquots 9B and 9C on April 21, 2021. The primary particle observed was mica; many iron oxide and particles containing titanium were also observed along with a few talc particles and scattered talc fibers and silica spheres. No asbestos or non-asbestos amphibole variants were observed during analysis. The results were calculated using the equations detailed in the Calculations section above.

625547-9A 625547-9B	No Asbestos Detected	
	No Asbestos Detected	
625547-9C	No Asbestos Detected	

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

625547-9A, Mica Particle



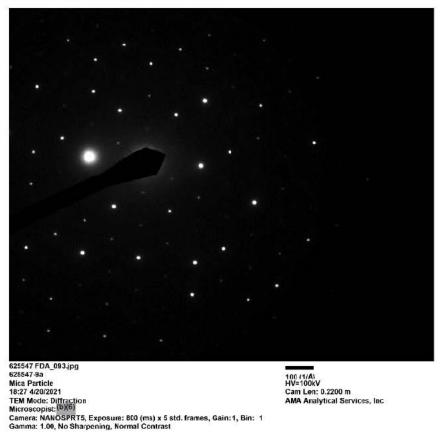
625547 FDA_094.jpg Mica Particle Cal: 0.002858 µm/pix 18:28 4/20/2021 TEM Mode: Imagin Microscopist:(b)(6)

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

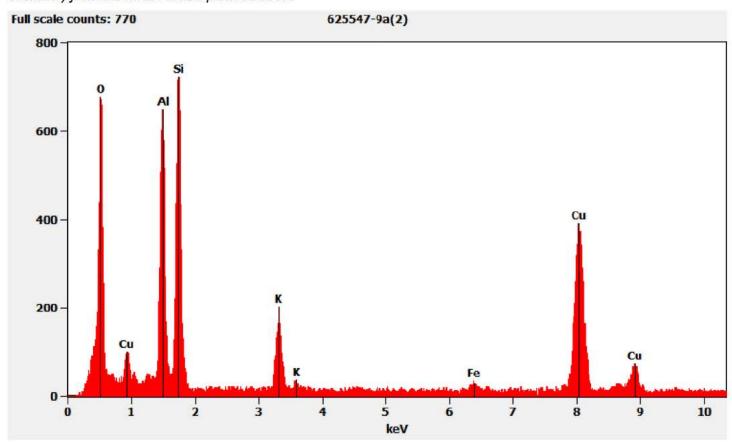
Gamma: 1.00, No Sharpening, Normal Contrast

Direct Mag: 3600 x AMA Analytical Services, Inc.

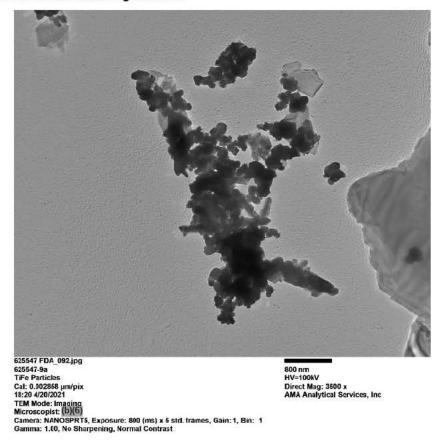
Hexagonal Diffraction Pattern from the Mica Particle pictured above



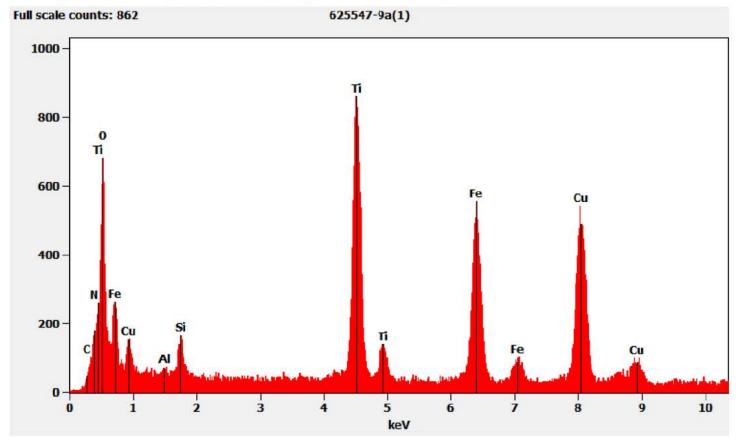
Chemistry from the Mica Particle pictured above



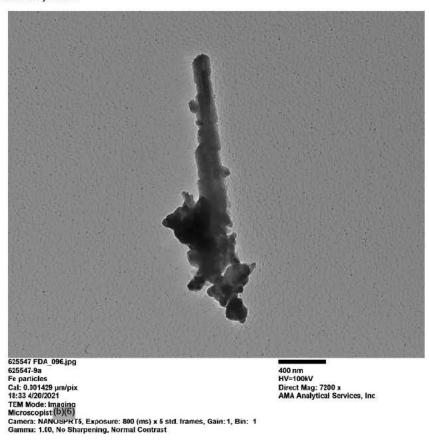
625547-9A, Iron Oxide & Particles Containing Titanium



Chemistry from the Iron Oxide & Particles Containing Titanium pictured above



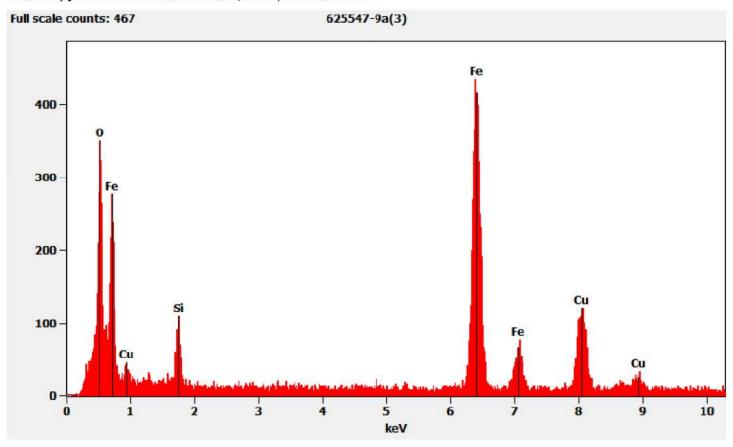
625547-9A, Iron Oxide Particles/Fiber



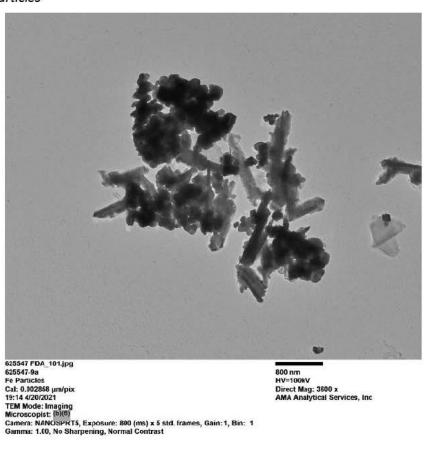
Diffraction Pattern from Iron Oxide Particles/Fiber pictured above



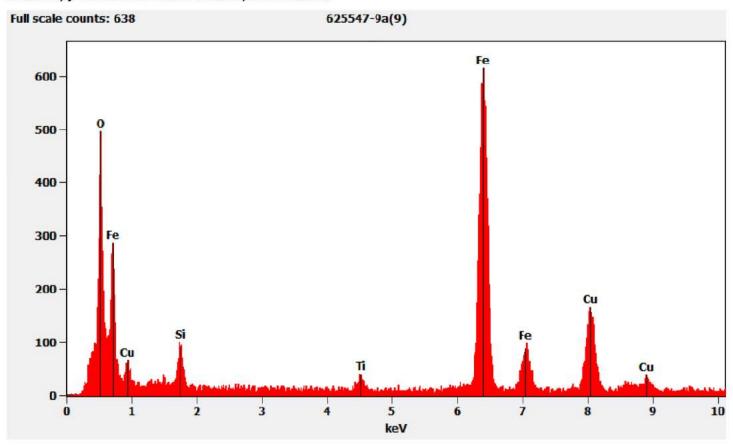
Chemistry from the Iron Oxide Particles/Fiber pictured above



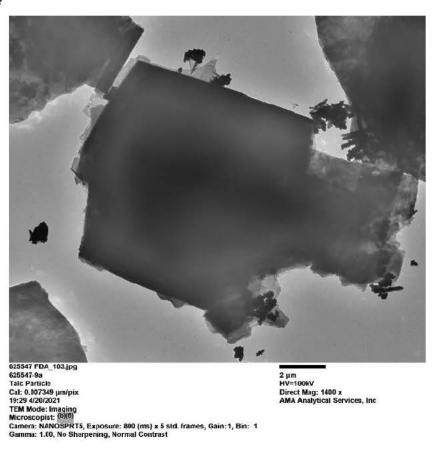
625547-9A, Iron Oxide Particles



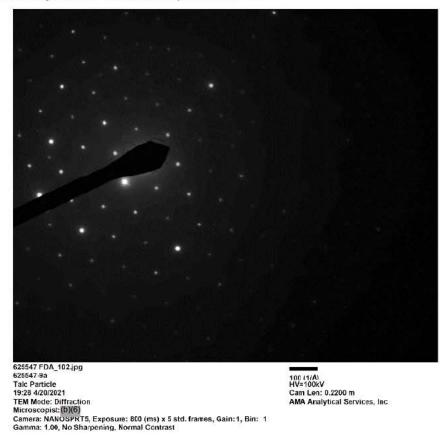
Chemistry from the Iron Oxide Particles pictured above



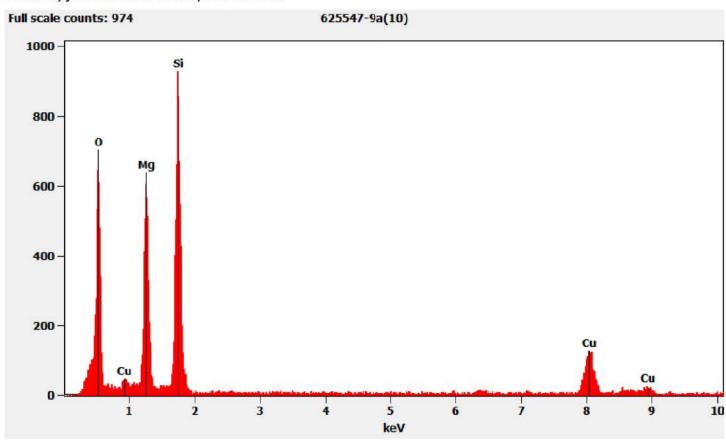
625547-9A, Talc Particle



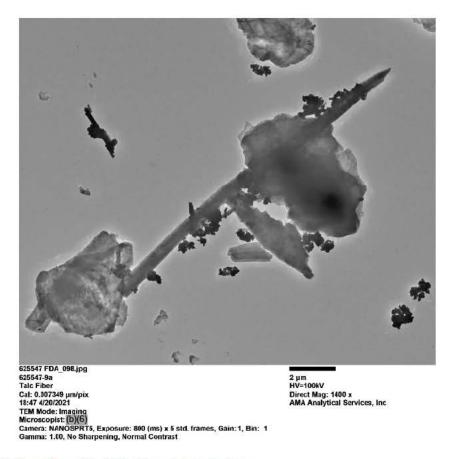
Hexagonal Diffraction Pattern from the Talc Particle pictured above



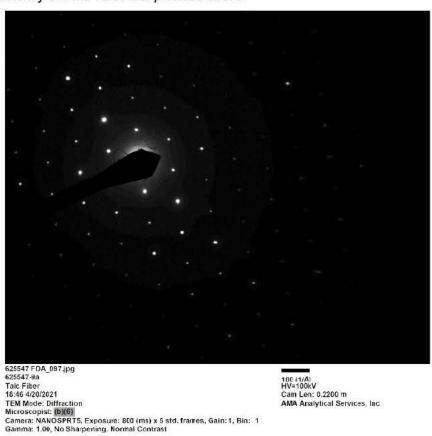
Chemistry from the Talc Particle pictured above



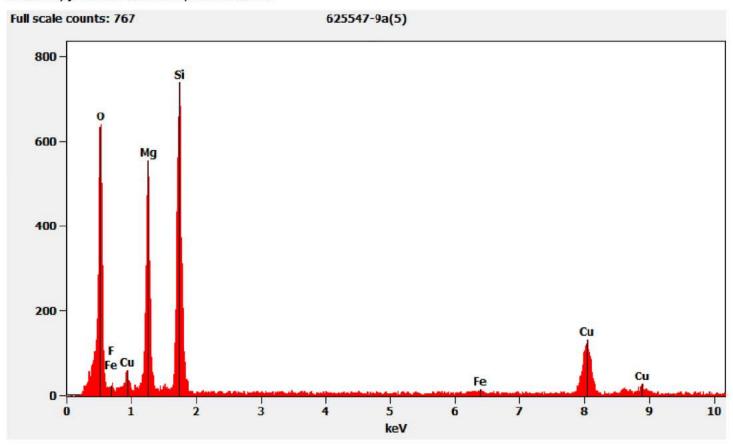
625547-9A, Talc Fiber



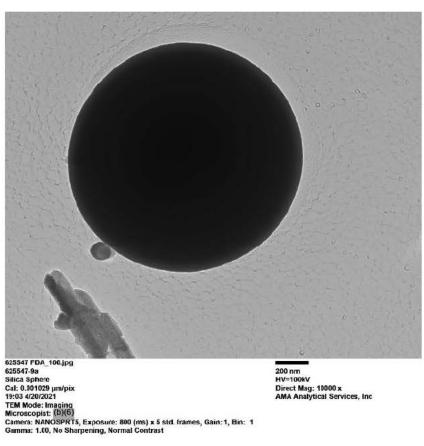
Hexagonal Diffraction Pattern from the Talc Fiber pictured above



Chemistry from the Talc Fiber pictured above



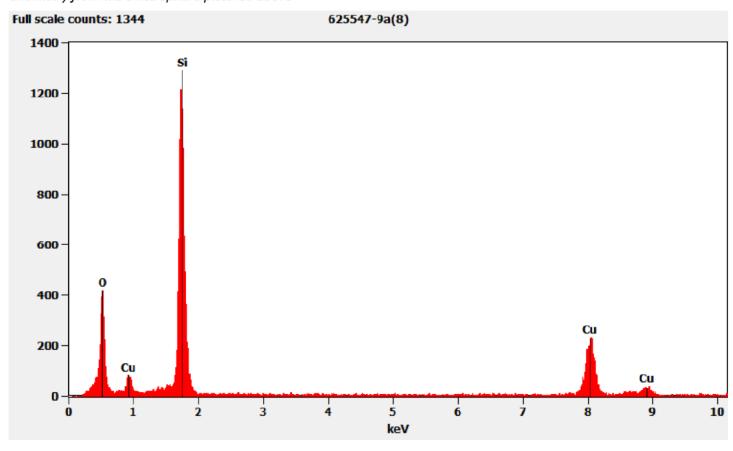
625547-9A, Silica Sphere



Diffraction Pattern from the Silica Sphere pictured above



Chemistry from the Silica Sphere pictured above



625547-10A, 10B, 10C/Client Sample: 02232021-10

PLM

All three aliquots of sample 02232021-10 were analyzed by (b)(6) on April 23, 2021. No asbestos or non-asbestos amphibole variants were observed during analysis. The results were calculated using the equations detailed in the *Calculations* section above.

625547-10A	No Asbestos Detected
625547-10B	No Asbestos Detected
625547-10C	No Asbestos Detected

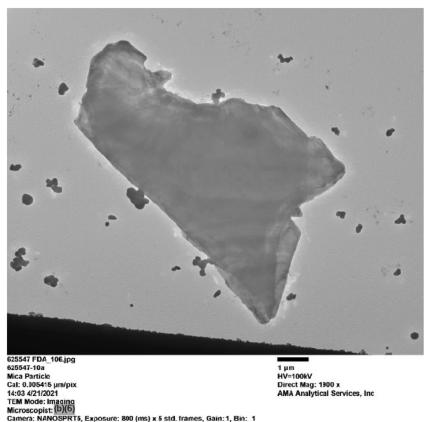
TEM

(b)(6) analyzed aliquot 10A on April 21, 2021. Andreas Saldivar analyzed aliquot 10B on April 21, 2021 and (b)(6) analyzed aliquot 10C on April 21, 2021. The primary particle observed was mica; many particles containing titanium and silica particles were also observed as well as some talc particles and a few talc fibers and silica spheres. No asbestos or non-asbestos amphibole variants were observed during analysis. The results were calculated using the equations detailed in the Calculations section above.

625547-10A	No Asbestos Detected
625547-10B	No Asbestos Detected
625547-10C	No Asbestos Detected

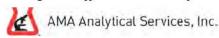
Below are pictures, diffraction patterns, and chemistry from some of the observed particles. The copper peaks in the chemistry spectra are from the TEM grid. The unidentified peaks in chemistry spectra are zinc and carbon, which are from the TEM specimen holder.

625547-10A, Mica Particle



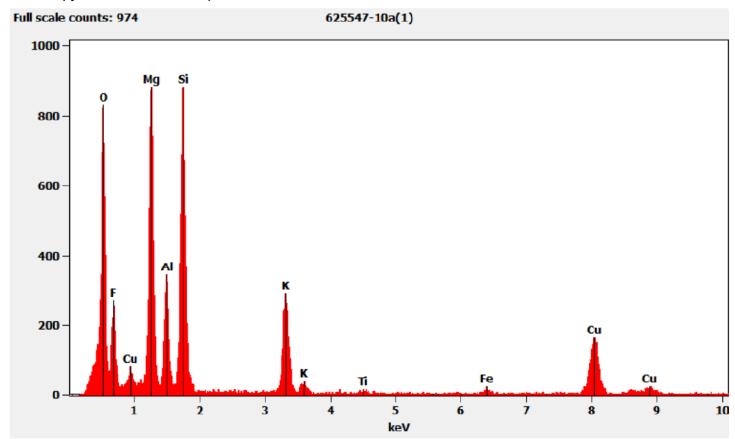
Hexagonal Diffraction Pattern from the Mica Particle pictured above

Gamma: 1.00, No Sharpening, Normal Contrast

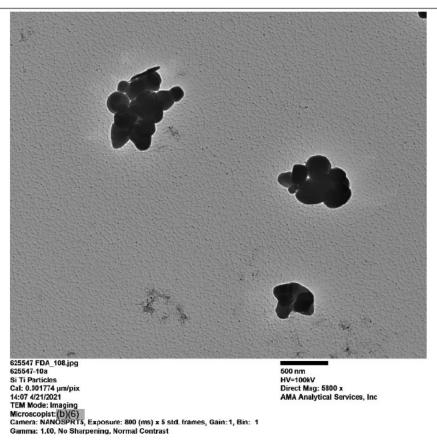




Chemistry from the Mica Particle pictured above



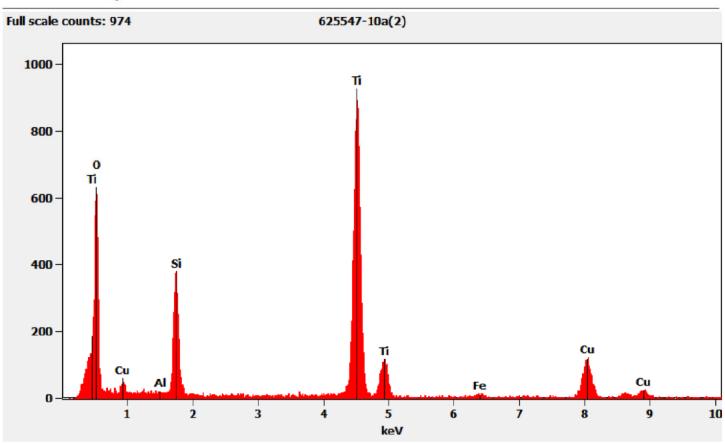
625547-10A, Particles Containing Titanium & Silica Particles



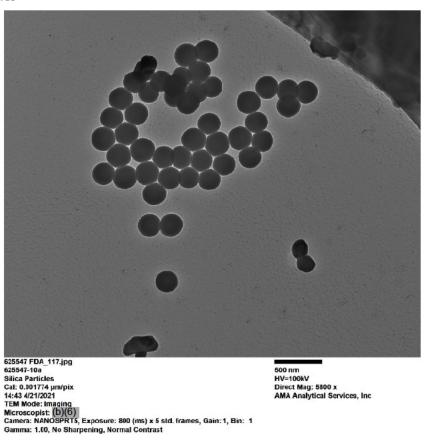
Hexagonal Diffraction Pattern from the Particles Containing Titanium & Silica Particles pictured above



Chemistry from the Particles Containing Titanium & Silica Particles pictured above



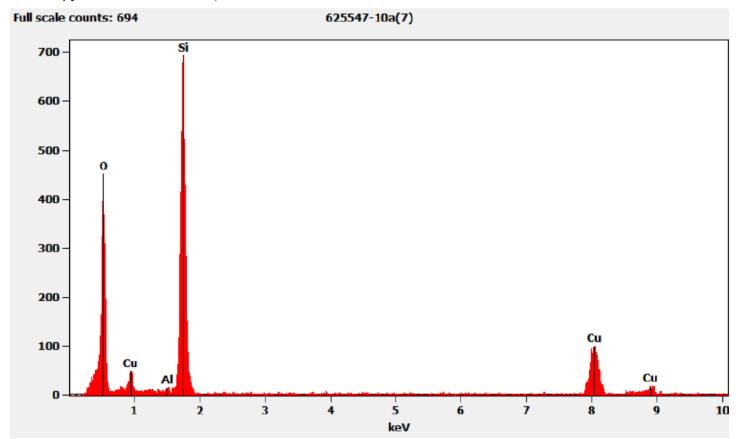
625547-10A, Silica Particles



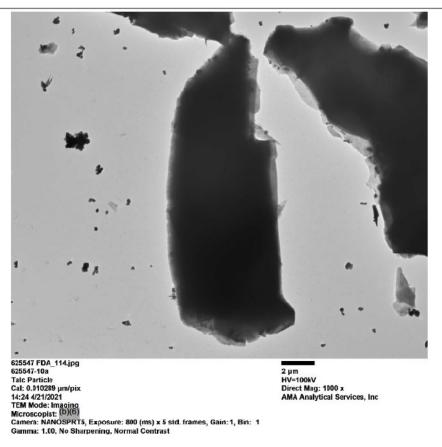
Diffraction Pattern from Silica Particles pictured above



Chemistry from the Silica Particles pictured above



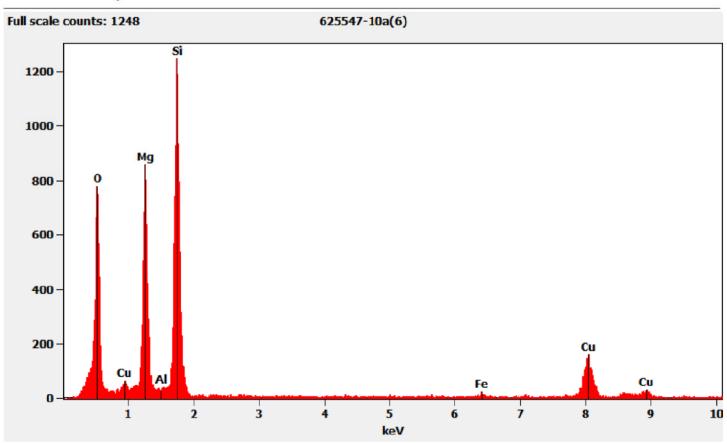
625547-10A, Talc Particle



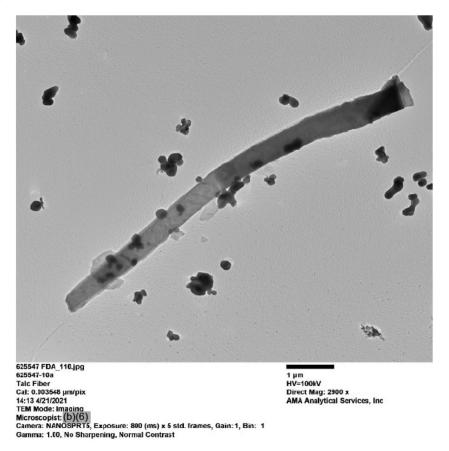
Hexagonal Diffraction Pattern from the Talc Particle pictured above



Chemistry from the Talc Particle pictured above

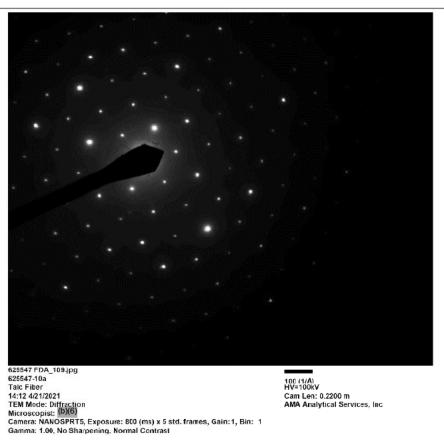


625547-10A, Talc Fiber

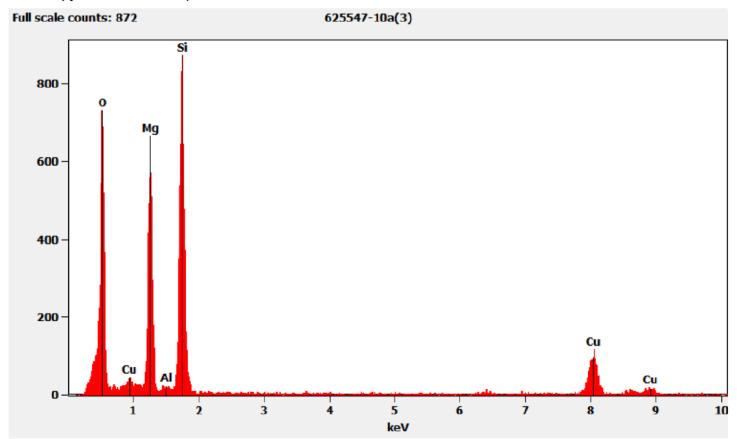


Hexagonal Diffraction Pattern from the Talc Fiber pictured above

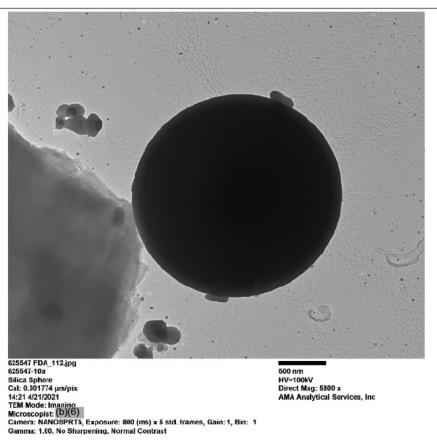




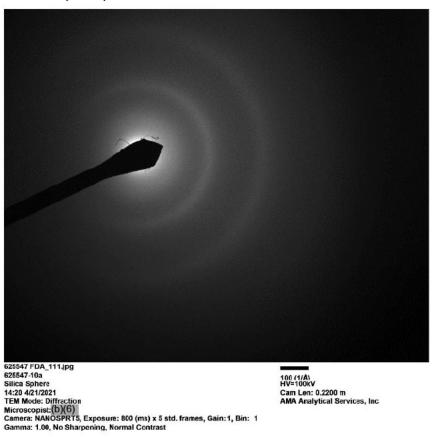
Chemistry from the Talc Fiber pictured above



625547-10A, Silica Sphere



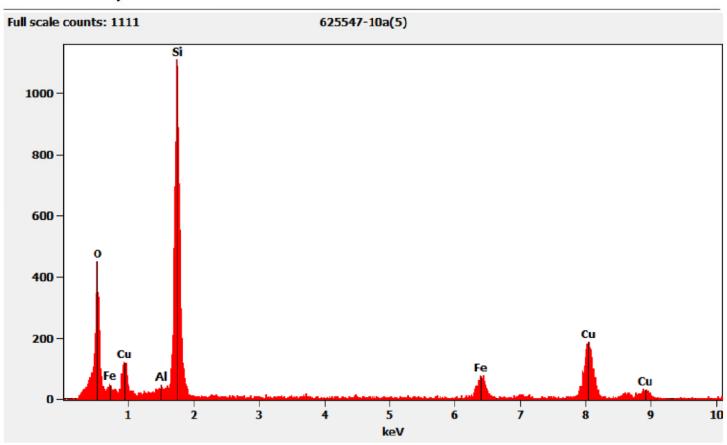
Diffraction Pattern from the Silica Sphere pictured above



Chemistry from the Silica Sphere pictured above



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625547-11A, 11B, 11C/Client Sample: 02232021-11

All three aliquots of sample 02232021-11 were analyzed by (b)(6)on April 23, 2021. No asbestos or non-asbestos amphibole variants were observed during analysis. The results were calculated using the equations detailed in the Calculations section above.

625547-11A No Asbestos Detected 625547-11B No Asbestos Detected 625547-11C No Asbestos Detected

TEM

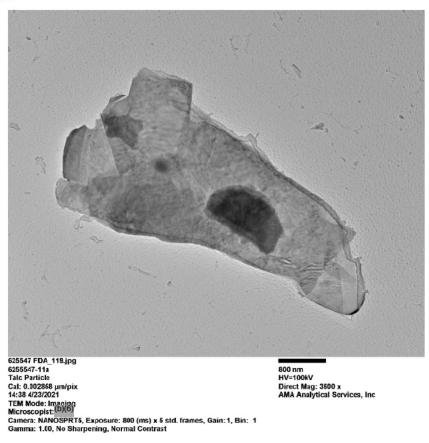
PLM

(b)(6)analyzed aliquot 11A on April 23, 2021. Andreas Saldivar analyzed aliquot 11B on April 21, 2021 and (b)(6)analyzed aliquot 11C on April 21, 2021. The primary particle observed was talc; talc ribbons/fibers were also observed as well as scattered silica spheres and particles containing titanium. No asbestos or non-asbestos amphibole variants were observed during analysis. The results were calculated using the equations detailed in the Calculations section above.

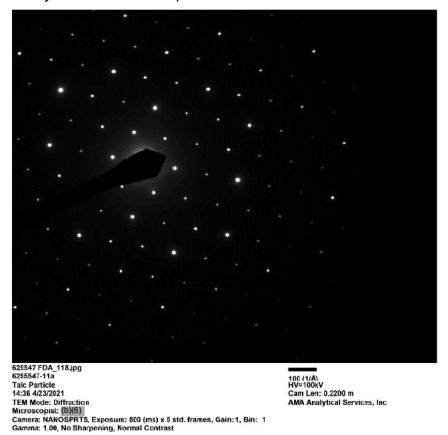
625547-11A No Asbestos Detected No Asbestos Detected 625547-11B 625547-11C No Asbestos Detected

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

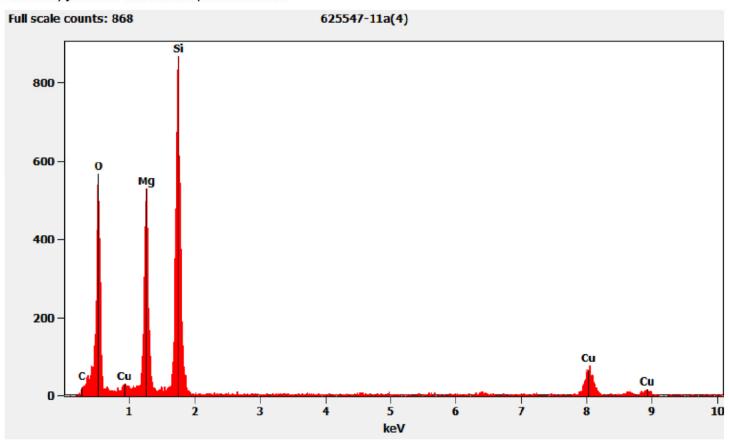
625547-11A, Talc Particle



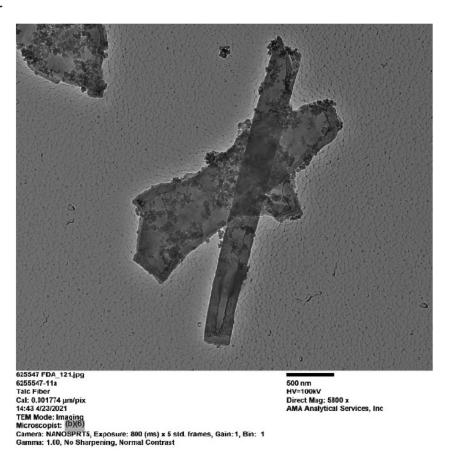
Hexagonal Diffraction Pattern from the Talc Particle pictured above



Chemistry from the Talc Particle pictured above



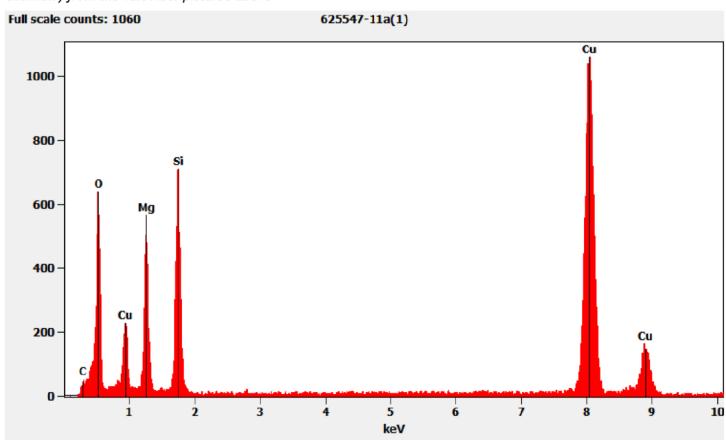
625547-11A, Talc Fiber



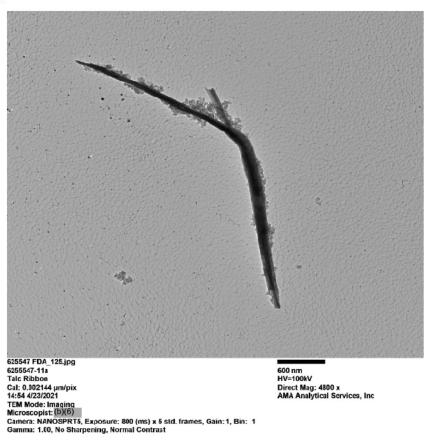
Hexagonal Diffraction Pattern from the Talc Fiber pictured above



Chemistry from the Talc Fiber pictured above



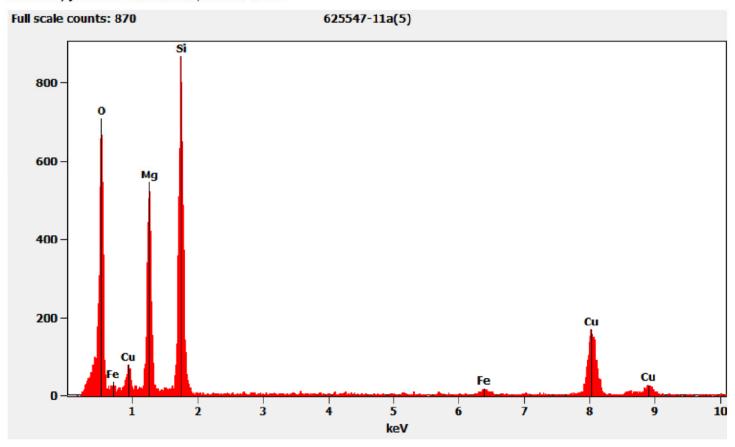
625547-11A, Talc Ribbon



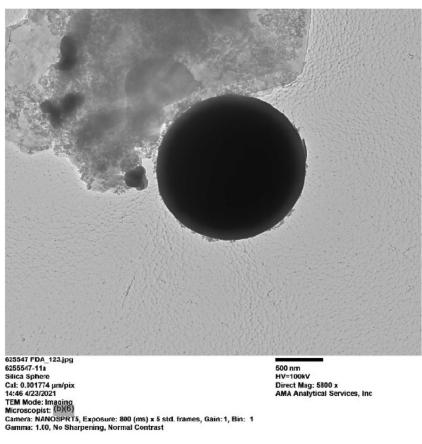
Diffraction Pattern from Talc Ribbon pictured above



Chemistry from the Talc Ribbon pictured above



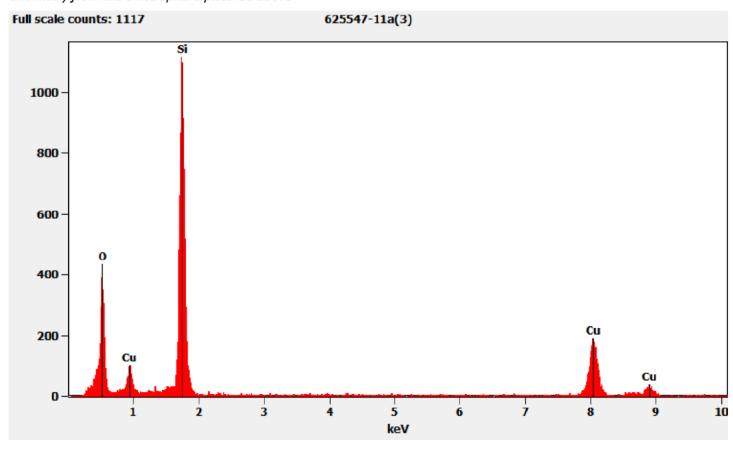
625547-11A, Silica Sphere



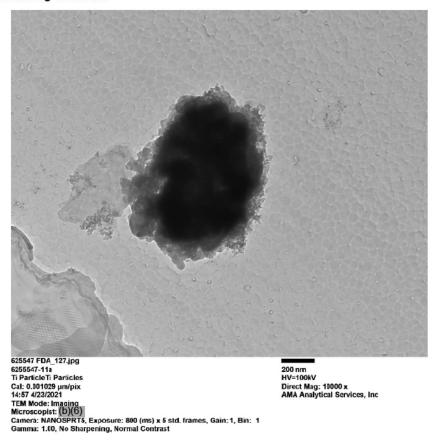
Diffraction Pattern from the Silica Sphere pictured above



Chemistry from the Silica Sphere pictured above



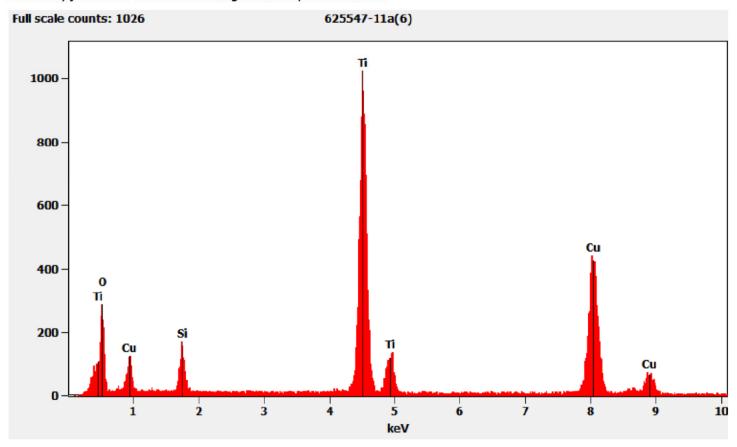
625547-11A, Particle Containing Titanium



Diffraction Pattern from the Particle Containing Titanium pictured above



Chemistry from the Particle Containing Titanium pictured above



QC Discussion:

Microscope alignment and calibration for both the PLM and TEM scopes, and EDXA unit calibration were performed on each day of analysis as specified by method requirements and standard laboratory operating procedures. The analytical balance used for gravimetric reduction is verified weekly at three (3) tare levels using three NIST-traceable weights – 10.0-g, 0.1-g, 0.5-g – and on each day of operation using the 0.1-g and 0.5-g weights tared with an 8-mL glass vial. The muffle furnace is verified monthly at a temperature of 480°C. All equipment was functioning within normal operating parameters

Matrix blank samples were prepared at rate of 10% or greater alongside the client samples with each series of samples that were put into the muffle furnace together. The matrix blank samples were prepared using Sigma-Aldrich Talc Powder 18654 (Cas No. 14807-96-6; EC No. 238-877-9, Lot 82330). Analysis of the matrix blank samples was only required if asbestos, or the non-asbestos versions of the regulated minerals, was found on the associated client samples unless otherwise noted. Matrix blank sample numbers NB21-210, NB21-217 and NB21-257 were not analyzed since no asbestos was observed on the associated client samples.

A talc reference control sample was randomly selected from our library of TEM grid preparations made from Sigma-Aldrich Talc Powder, <10 micron (Product No. 643604-500G; Batch No. 10830AJ) spiked with various levels of Chrysotile ranging from 0.4%-10%. One (1) reference control sample, sample number 625547/625947-RB1, was analyzed with this set. It was analyzed by (b)(6) on May 7, 2021 and found to be within acceptable limits.

Filtration blank samples were prepared alongside the client samples with each use of the filtration apparatus. Analysis of these samples was only required on those blanks associated with a client sample on which asbestos, or the non-asbestos versions of the regulated minerals, was found unless otherwise noted. Filtration blank sample numbers DI-Blank-01 through DI-Blank-11 were not analyzed since no asbestos was observed on the associated client samples.

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TEM grid preparation (EB) blank samples were prepared with each batch of carbon coated filters. AMA policy is to analyze these blank samples whenever asbestos, or the non-asbestos versions of the regulated minerals, is detected on an associated client sample or when the laboratory blank identification number ends in a "0" or "5." Since no asbestos was observed on any of the client samples, only EB Blank IDs 56510 and 56530 were analyzed. (b)(6) analyzed these samples on April 23, 2021. No asbestos was detected on the sample.

No samples in this set were randomly selected for additional duplicate QC analysis.

Our laboratory information management system (LIMS) randomly selected samples 625547-6/02232021-6 and 625547-10/02232021-10 for additional replicate QC analysis. Separate preparations were made for the PLM and TEM portions of analysis. The replicate QC analysis was performed by Lom Butruk on May 6, 2021 for PLM and by (b)(6) on May 6, 2021 for TEM. The QC results were consistent with the original findings.

Attachments:

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

- 1) Sample Log-In Sheet
- 2) Analytical Balance Verification Log
- 3) Daily PLM Scope Verification Log
- 4) Refractive Index Oil Verification Log
- 5) Daily TEM Scope Verification Log(s)
- 6) QC Results Summary for 625547
- 7) NB (Matrix) Blank Preparation Log
- 8) RB (Reference) Sample Bench Sheet(s)
- 9) EB (TEM Grid) Blank Preparation Log
- 10) EB (TEM Grid) Blank Bench Sheet(s)
- 11) Duplicate & Replicate QC Charts for (b)(6) for samples analyzed between 1/1/2021 & 4/23/2021
- 12) Duplicate & Replicate QC Charts for (b)(6) for samples analyzed between 1/1/2021 & 4/23/2021
- 13) Duplicate & Replicate QC Charts for (b)(6) for samples analyzed between 1/1/2021 & 4/23/2021
- 14) Duplicate & Replicate QC Charts for Andreas Saldivar for samples analyzed between 1/1/2019 & 4/23/2021
- 15) Raw Data Sheets
 - a. PLM Gravimetric Reduction Bench Sheet
 - b. TEM Gravimetric/Filtration Bench Sheet
 - c. PLM Analysis
 - d. TEM Analysis
 - e. Replicate QC Analysis

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.

Andreas Saldivar

D-4-

5/7/2021

President