AMA Analytical Services, Inc.
Focused On Results.

CERTIFICATE OF ANALYSIS

COC: 305281
Client: US Food & Drug Administration
Address: Office of Cosmetics & Colors
4300 River Road
College Park, Maryland 20740
Attn: John Gasper

Job Name: Task 2 - Analysis of Demonstration Samples
Job Location: Talc (x000756) A TK4428 FDA
Job Number: Not Provided
PO Number: HHSF223201810337P

Date Submitted: 11/14/2018
Date Analyzed: 1/31/2019
Report Date: 2/21/2019
Date Sampled: Not Provided
Person Submitting: John Gasper
Revised: 2/21/2019 (Revision #1)

SUMMARY OF ASBESTOS IN TALC ANALYSIS

AMA Sample ID	Client Sample ID	TEM LOD Range		TEM LOQ Range		% Chysotile by TEM	% Tremolite by TEM		% Total by TEM		% Organics	% Acid Soluble	% Chrysotile by PLM	% Tremolite by PLM
							ISO 22262-2	ASTM D5756	ISO 22262-2	ASTM D5756				
305281-3	D-18	0.00000057%	0.00000090%	0.00000229%	0.00000359%	ND	ND	ND	<0.00000229%	<0.00000359%	0.1	6.6	ND	ND
305281-3A	D-18	0.00000059%	0.00000093%	0.00000236%	0.00000371%	ND	0.00412%	0.00647%	0.00412%	0.00647%	0.1	7.4	ND	ND
305281-3B	D-18	0.00000056%	0.00000088%	0.00000225%	0.00000353%	ND	ND	ND	<0.00000225%	<0.00000353%	0.1	4.5	ND	ND
305281-4	D-30	0.00000684%	0.00001074%	0.00002734%	0.00004295%	ND	9.86344%	15.49345%	9.86%	15.5%	9.4	46	ND	ND
305281-4A	D-30	0.00000330%	0.00000519%	0.00001322%	0.00002076%	ND	1.35083%	1.82317%	1.35%	1.82%	8.4	41.2	ND	<0.13
305281-4B	D-30	0.00000453%	0.00000712%	0.00001813%	0.00002848%	ND	2.75310%	4.32456%	2.75%	4.32%	8.9	41.7	ND	ND
305281-5	D-31	0.00000103%	0.00000161%	0.00000410%	0.00000644%	0.00183%	0.15435%	0.24245%	0.156%	0.244%	13.4	14.7	ND	<0.18%
305281-5A	D-31	0.00000146%	0.00000229%	0.00000583%	0.00000916%	0.00035%	0.17068%	0.26810%	0.171%	0.268%	13.7	13.5	ND	<0.18%
305281-5B	D-31	0.00000075%	0.00000117%	0.00000298%	0.00000468%	0.00012%	0.13368%	0.20999%	0.134%	0.210%	13.5	13.7	ND	<0.18%
305281-6	D-96	0.00000084%	0.00000132%	0.00000335%	0.00000526%	ND	ND	ND	<0.00000335%	<0.00000526%	18.2	6	ND	ND
305281-6A	D-96	0.00000047%	0.00000074%	0.00000190%	0.00000298%	ND	ND	ND	<0.00000190%	<0.00000298%	17.6	5.2	ND	ND
305281-6B	D-96	0.00000073%	0.00000114%	0.00000290%	0.00000456%	ND	ND	ND	<0.00000290%	<0.00000456%	17.9	5.3	ND	ND

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/ISO 22262-2

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

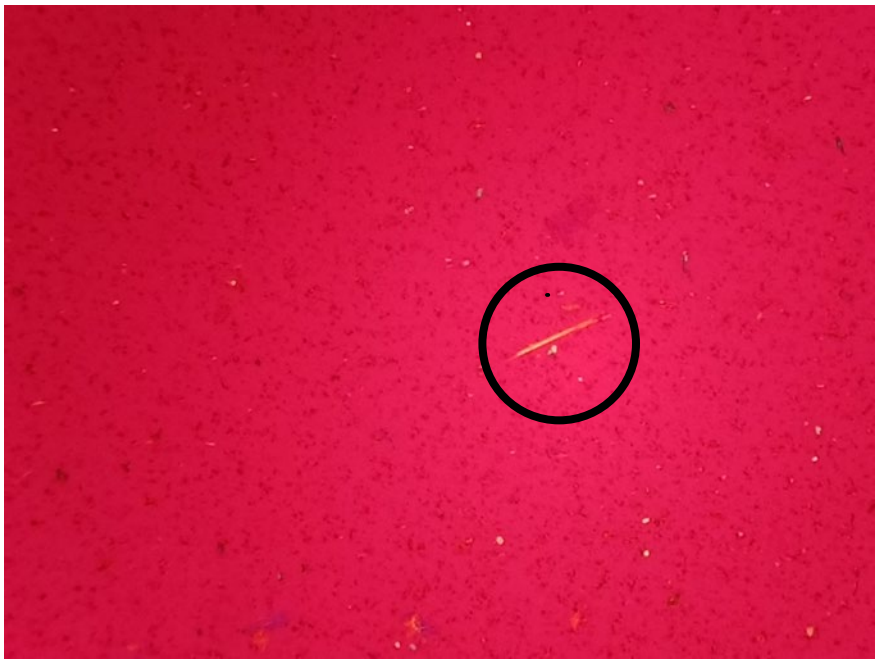
305281-4, 4A, 4B, Client Sample D30

PLM

All three aliquots of sample D30 were analyzed by (b) (6) on January 31, 2019. Tremolite was observed on aliquot 4a but no points were counted. No tremolite was detected on 4 and 4b. No other asbestos was detected.

305281-4	NAD
305281-4A	<0.13% Tremolite detected
305281-4B	NAD

305281-4A Tremolite 100x



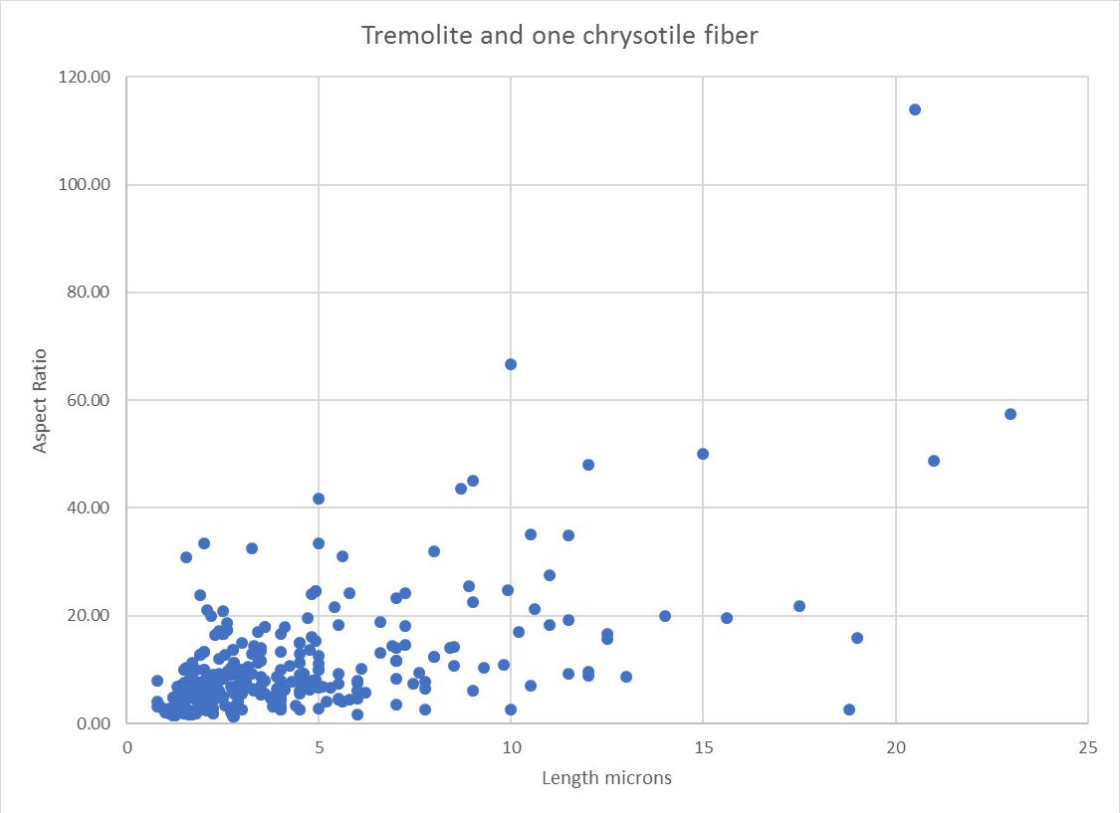
TEM

Sample 4 was analyzed by Andreas Saldivar on January 11, 2019. Samples 4A and 4B were analyzed by (b) (6) (b) (6) on January 25, 2019 and January 31, 2019 respectively. Talc is the main constituent in all three aliquots. The talc is primarily in plate form but there are also some talc fibers and ribbons. Tremolite was detected on all three aliquots. At least 100 tremolite particles were documented for each aliquot. The length and width of each particle was recorded and the mass was calculated using both the ASTM D5756 and ISO 22262-2 methods. The percentage of tremolite was calculated using the mass from both calculations and the range was reported.

305281-4	9.86% to 15.5%
305281-4a	1.35% to 1.82%
305281-4b	2.75% to 4.32%

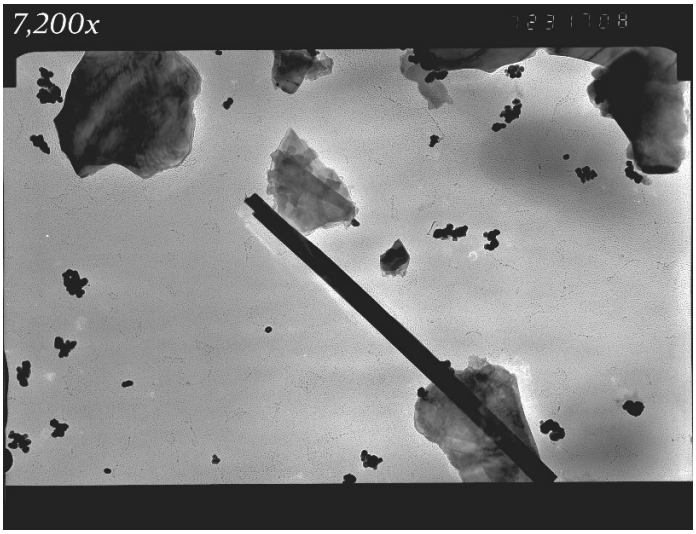
The range of tremolite in aliquot 4 is substantially higher than 4a and 4b. One large particle, #24, accounts for 85% of the total mass of tremolite counted in aliquot 4. When the mass of that particle is removed from the total the range of tremolite is 1.54% - 2.41%. The dramatic effect of this one particle on the total percent is a result of the dilution factors from preparation. The tremolite particles varied in morphology from small blocks to very long, high length to width aspect ratio fibers. The length to width aspect ratio of particles counted ranged from 1.27 to 113.9. The mean aspect ratio is 11.5. The chemistry of the tremolite observed consisted of O, Mg, Si, and Ca. A few particles had small amounts of Al and Fe. One chrysotile fiber was counted on sample 305281-4a.

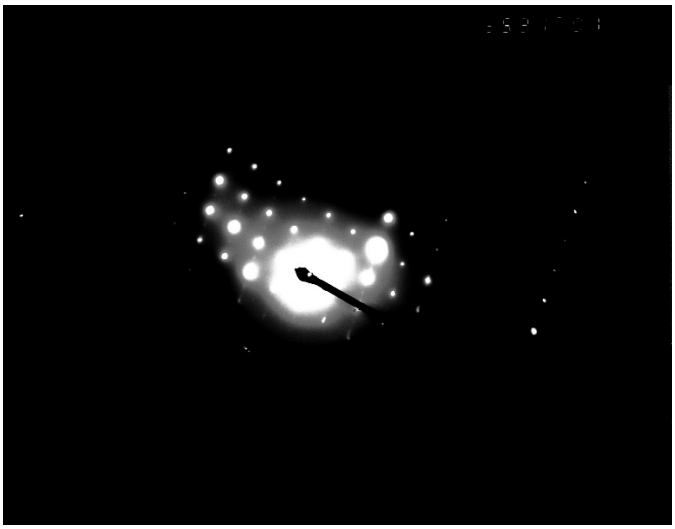
The following chart is a plot aspect ratio vs length for all the particles counted over all three aliquots.

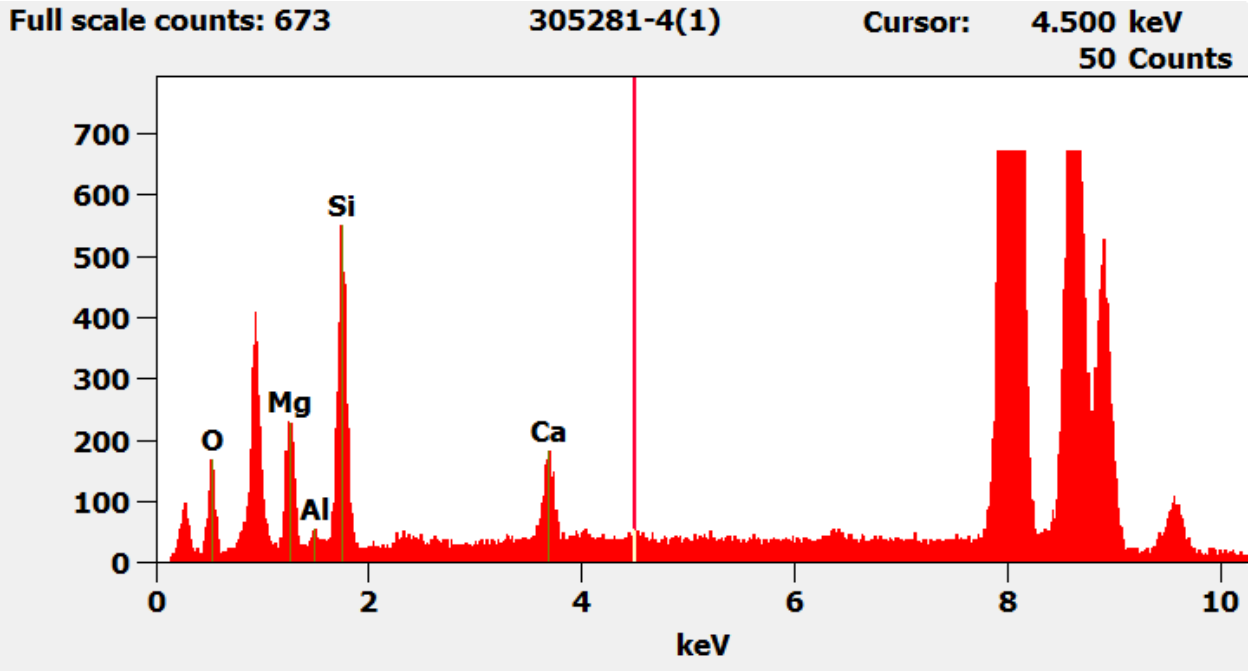


Below are pictures, diffraction patterns, and chemistry from some of the counted particles. The unidentified peaks in chemistry spectra are copper, zinc, and carbon. Those peaks are from the TEM specimen holder and specimen grid.

305281-4 Particle 2 8.9 x 0.35 microns Tremolite

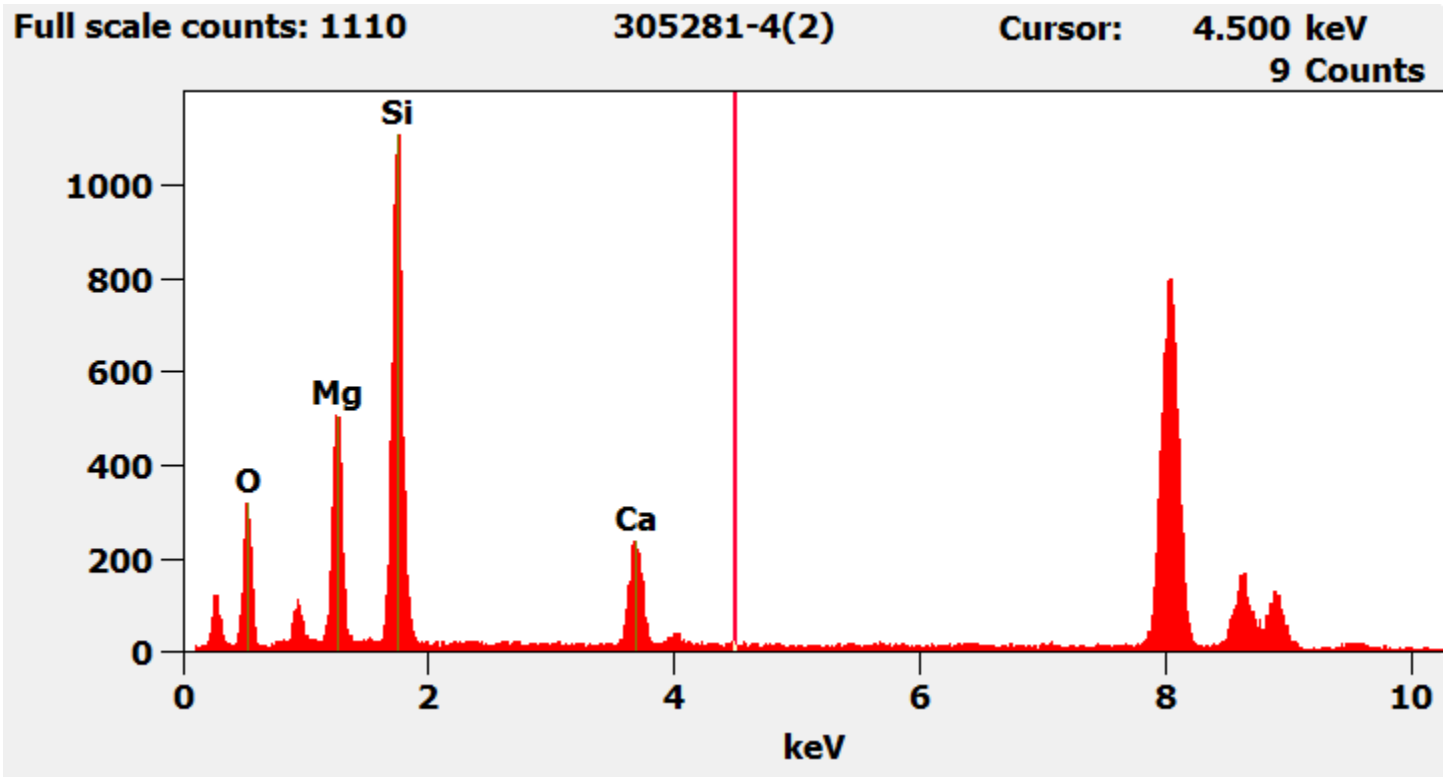






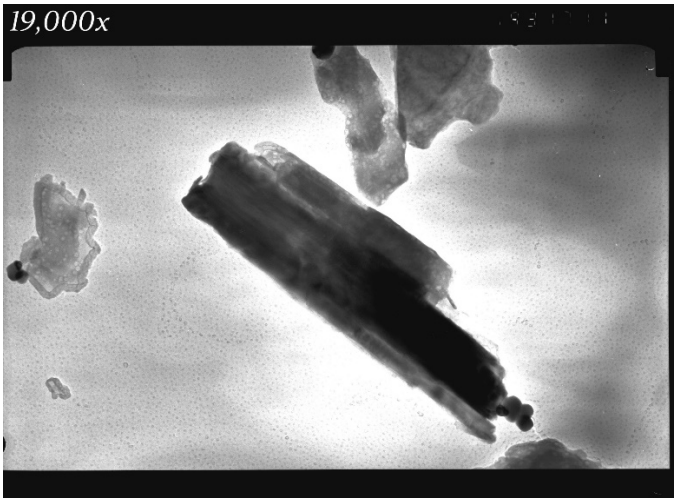
Element Line	Weight %	Weight % Error
O K	51.41	± 2.37
Mg K	18.09	± 0.54
Al K	1.98	± 0.29
Si K	25.57	± 0.35
Si L	---	---
Ca K	2.96	± 0.19
Ca L	---	---
Total	100.00	

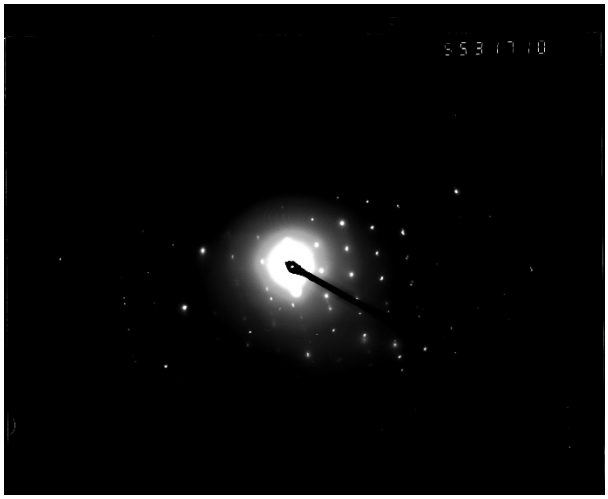
305281-4 Particle 3 1.6 x 0.2 microns Tremolite

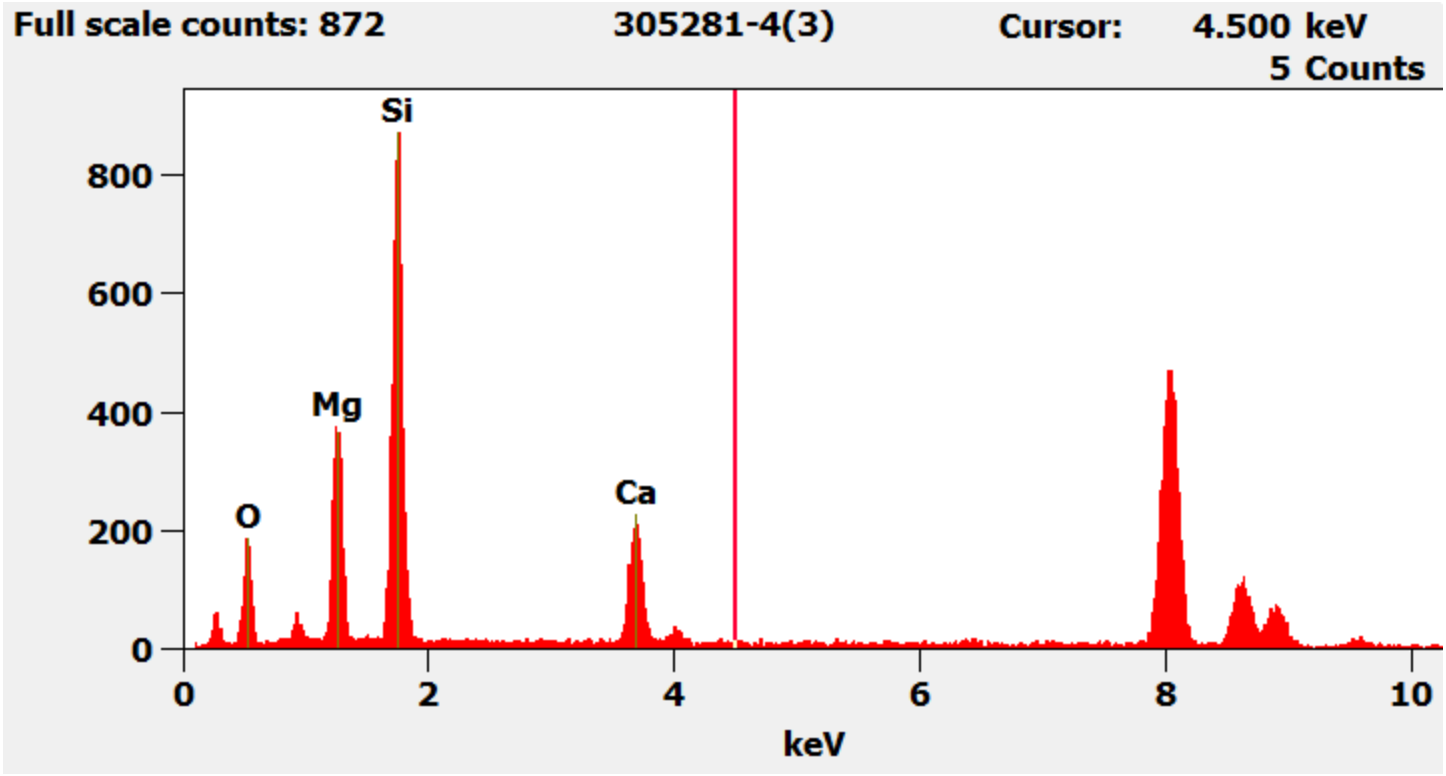


Element Line	Weight %	Weight % Error
O K	53.11	± 1.38
Mg K	16.92	± 0.30
Si K	27.08	± 0.30
Si L	---	---
Ca K	2.89	± 0.09
Ca L	---	---
Total	100.00	

305281-4 Particle 4 3.0 x 1.2 microns Tremolite

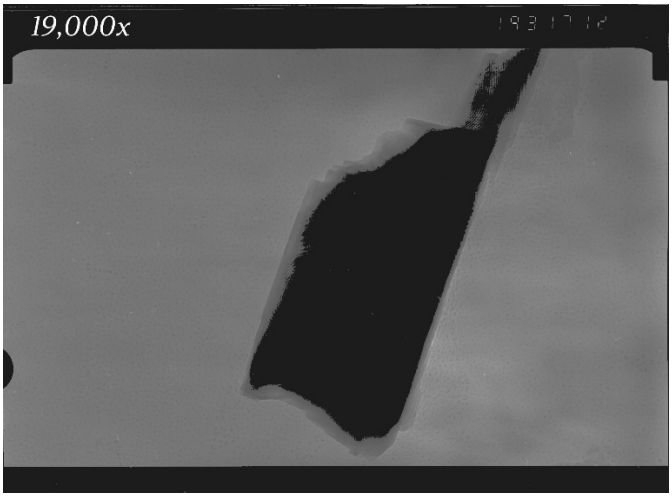


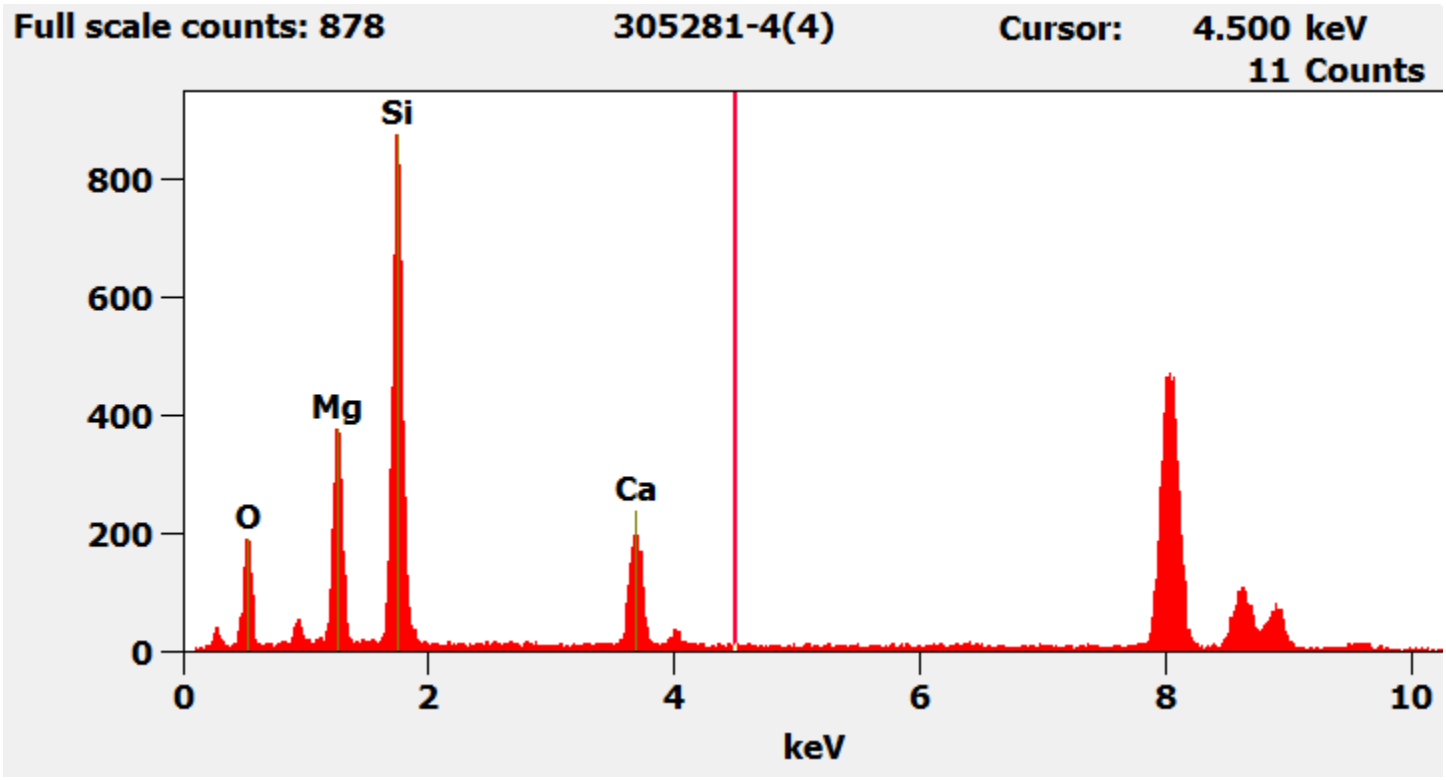




Element Line	Weight %	Weight % Error
O K	48.04	± 1.69
Mg K	17.67	± 0.35
Si K	30.30	± 0.38
Si L	---	---
Ca K	3.99	± 0.13
Ca L	---	---
Total	100.00	

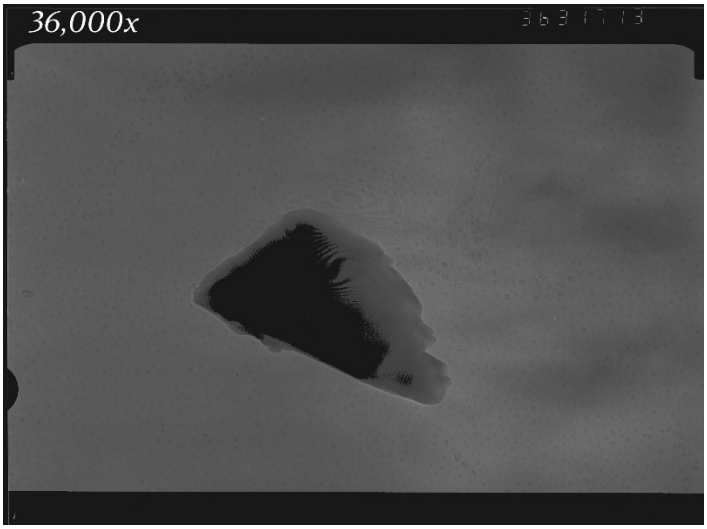
305281-4 Particle 5 2.7 x 1.3 microns Tremolite

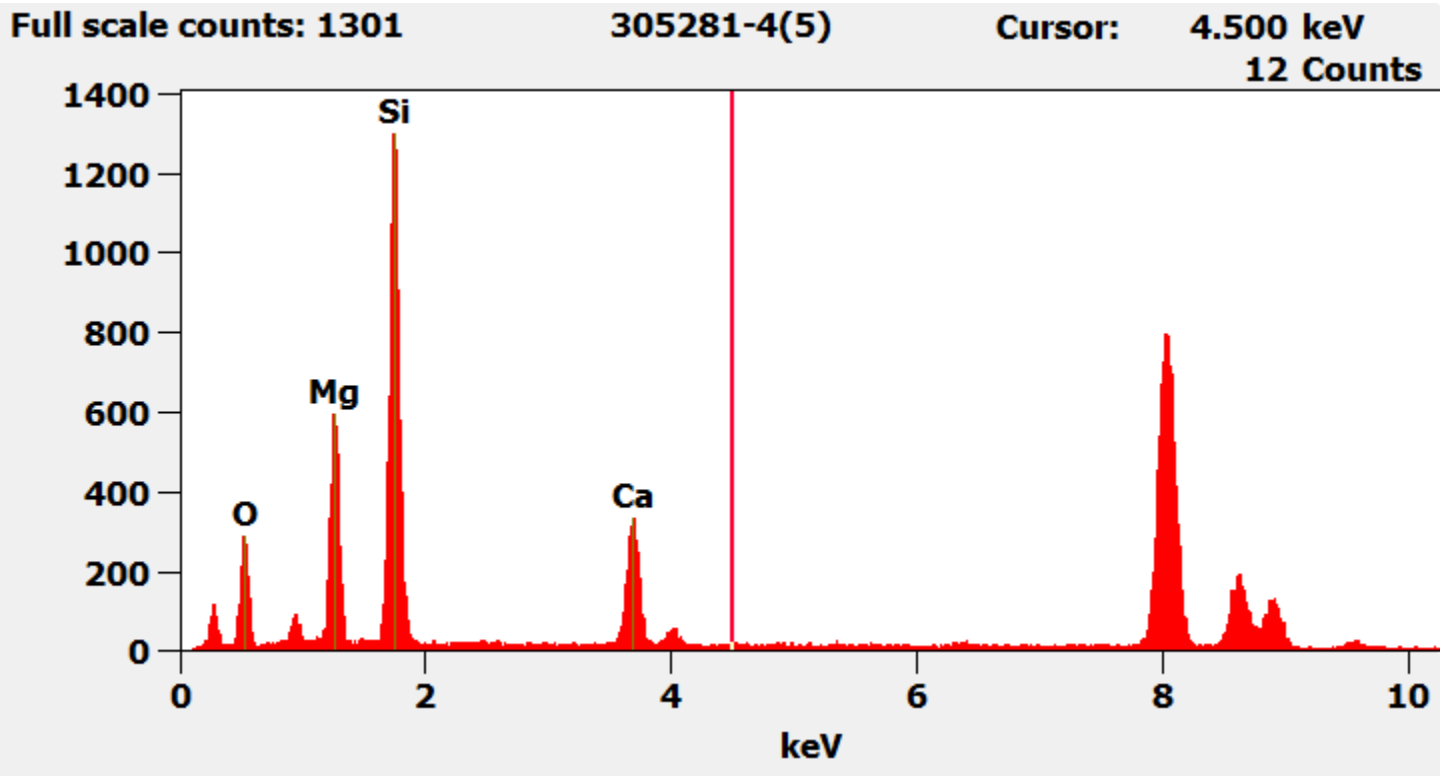




Element Line	Weight %	Weight % Error
O K	50.90	± 1.56
Mg K	16.75	± 0.34
Si K	28.70	± 0.35
Si L	---	---
Ca K	3.65	± 0.12
Ca L	---	---
Total	100.00	

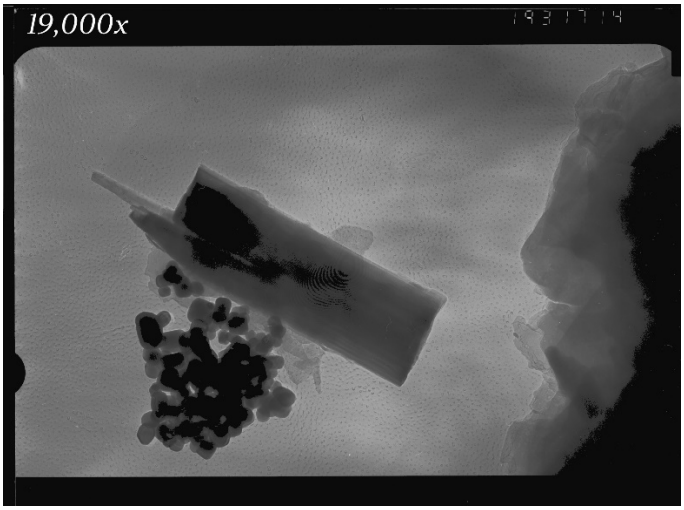
305281-4 Particle 6 1.2 x 0.8 microns Tremolite

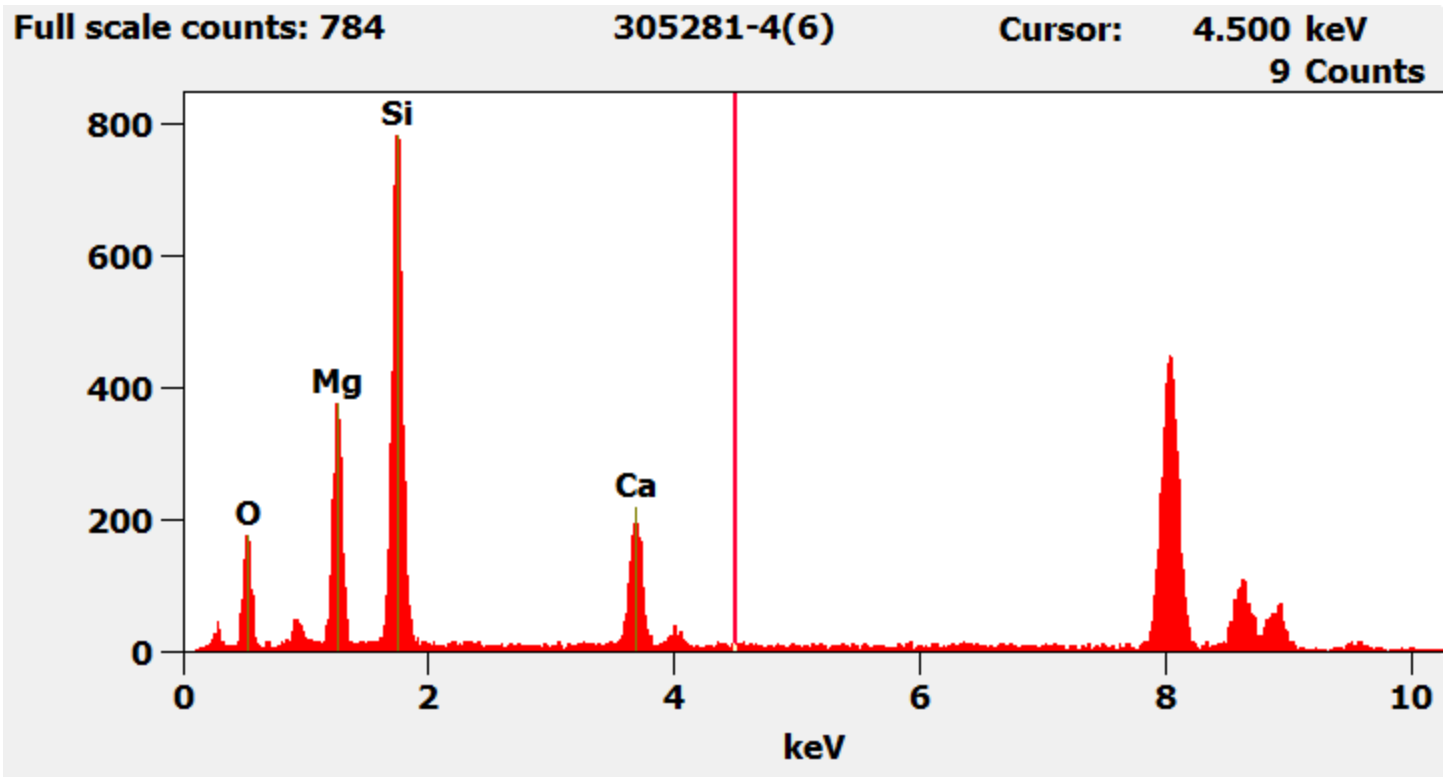




Element Line	Weight %	Weight % Error
O K	49.72	± 1.38
Mg K	17.25	± 0.29
Si K	29.30	± 0.30
Si L	---	---
Ca K	3.74	± 0.10
Ca L	---	---
Total	100.00	

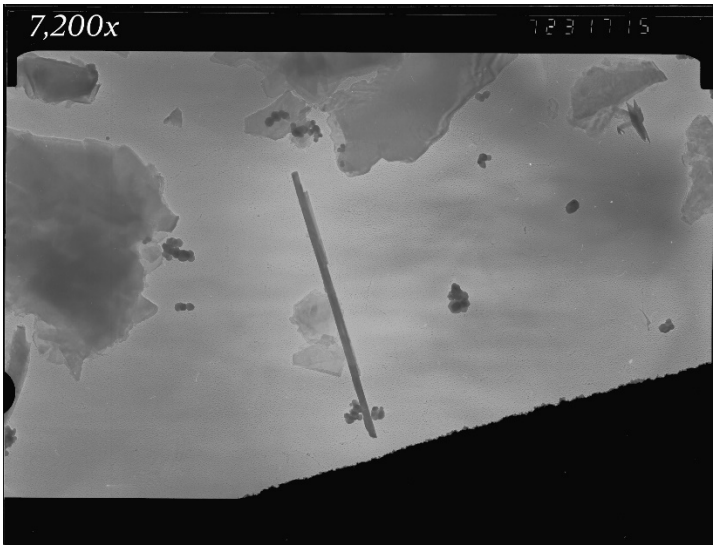
305281-4 Particle 7 2.3 x .8 microns Tremolite

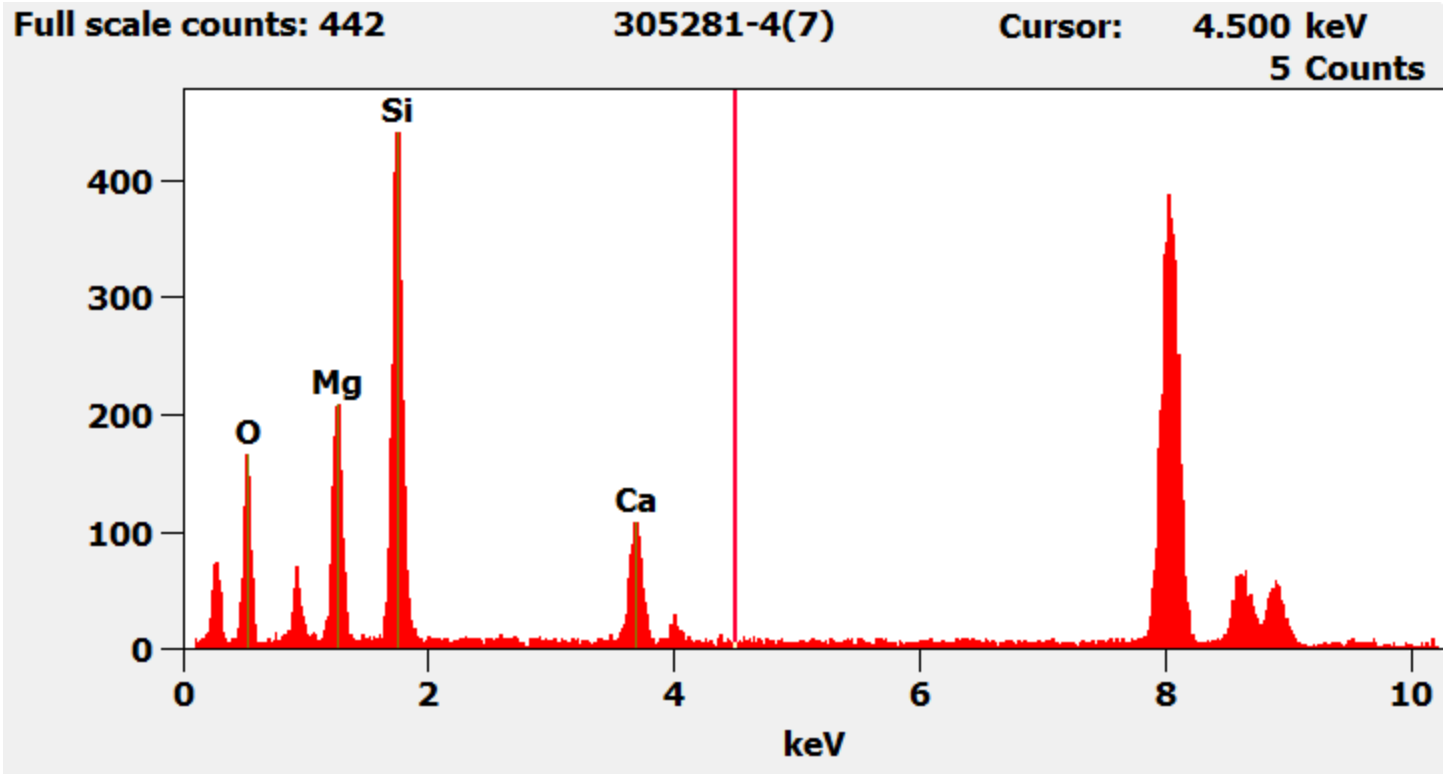




Element Line	Weight %	Weight % Error
O K	49.62	± 1.60
Mg K	17.26	± 0.35
Si K	29.41	± 0.37
Si L	---	---
Ca K	3.71	± 0.12
Ca L	---	---
Total	100.00	

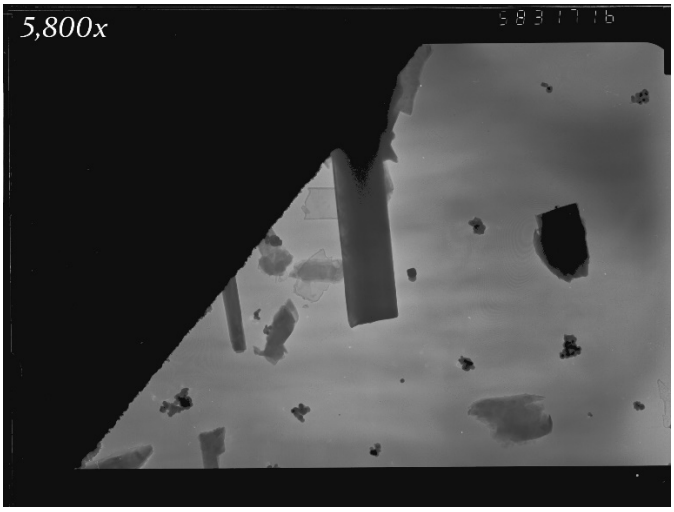
305281-4 Particle 8 5.6 x 0.18 microns Tremolite

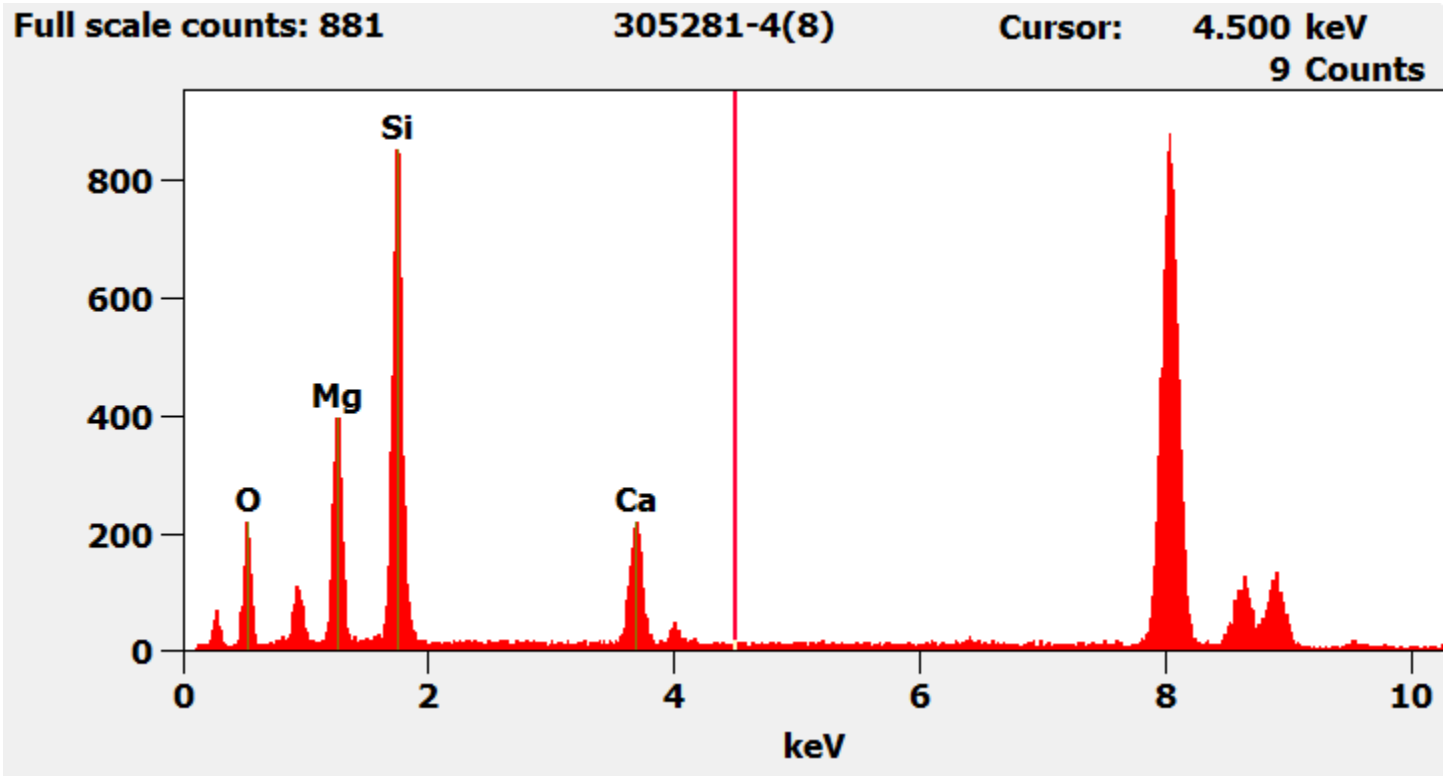




Element Line	Weight %	Weight % Error
O K	55.70	± 2.05
Mg K	16.34	± 0.44
Si K	25.17	± 0.42
Si L	---	---
Ca K	2.79	± 0.13
Ca L	---	---
Total	100.00	

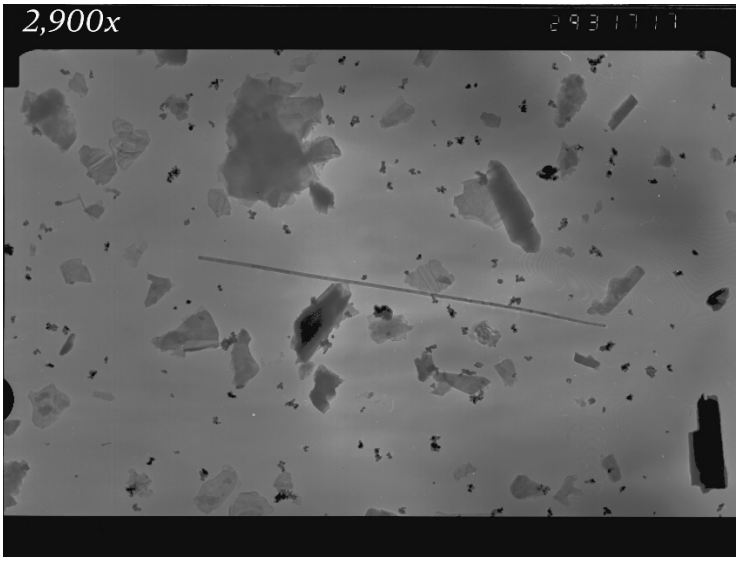
305281-4 Particle 9 5.8 x 1.3 microns Tremolite (#9 is the large particle. Particle 10 is also pictured to the left of 9)

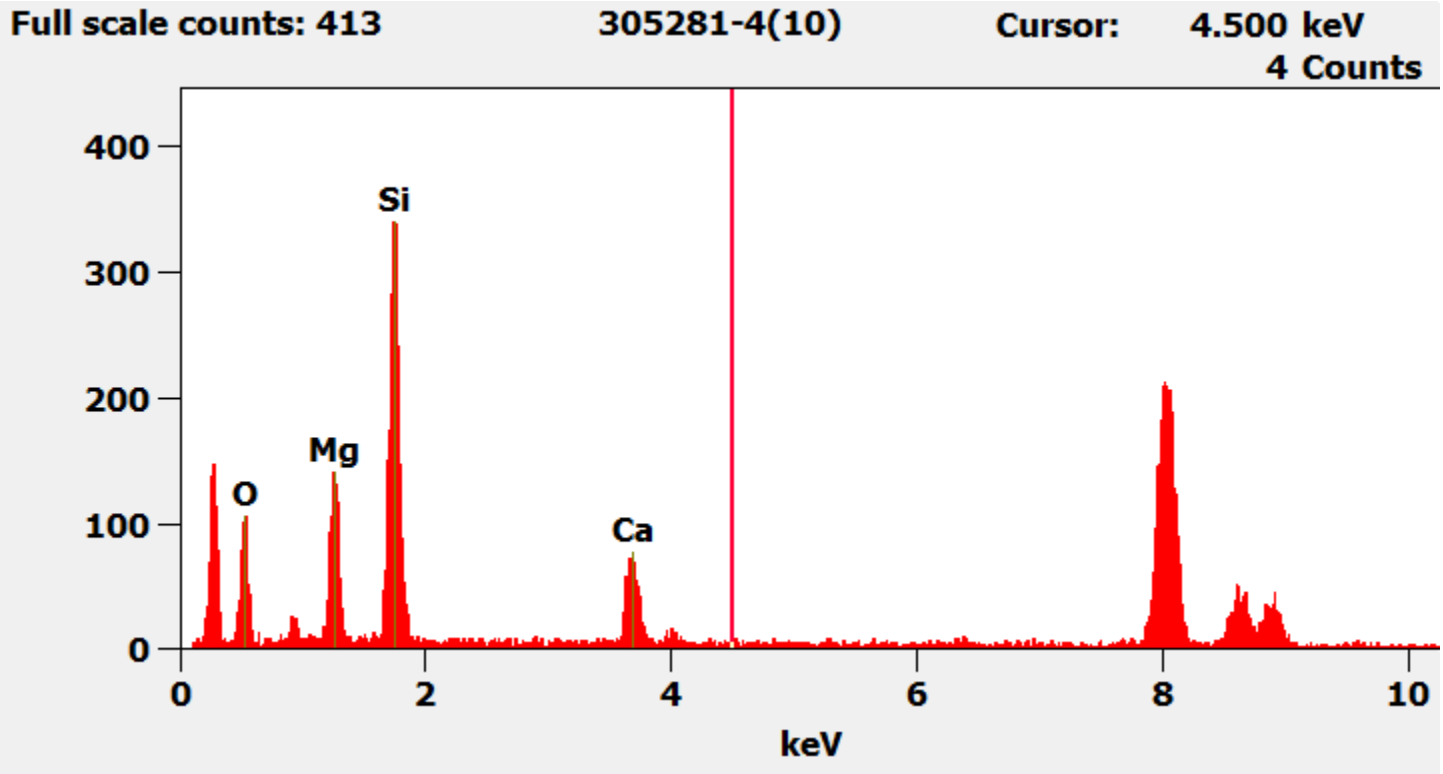




Element Line	Weight %	Weight % Error
O K	50.52	± 1.60
Mg K	18.00	± 0.35
Si K	27.86	± 0.35
Si L	---	---
Ca K	3.63	± 0.12
Ca L	---	---
Total	100.00	

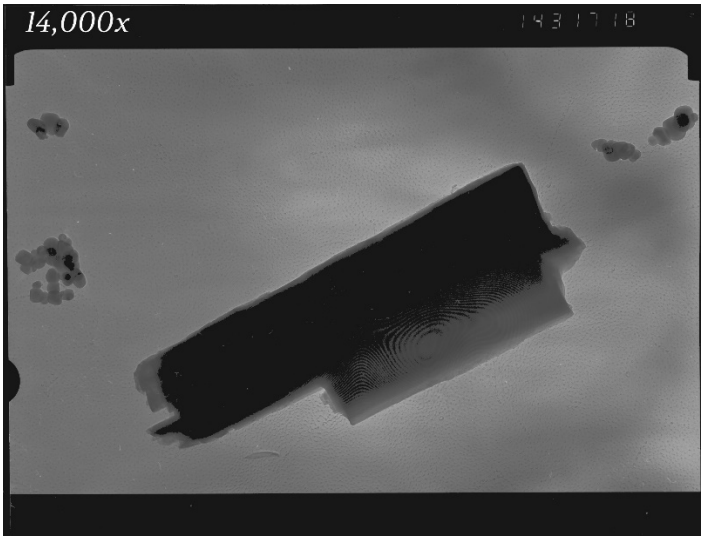
305281-4 Particle 11 20.5 x 0.18 microns Tremolite

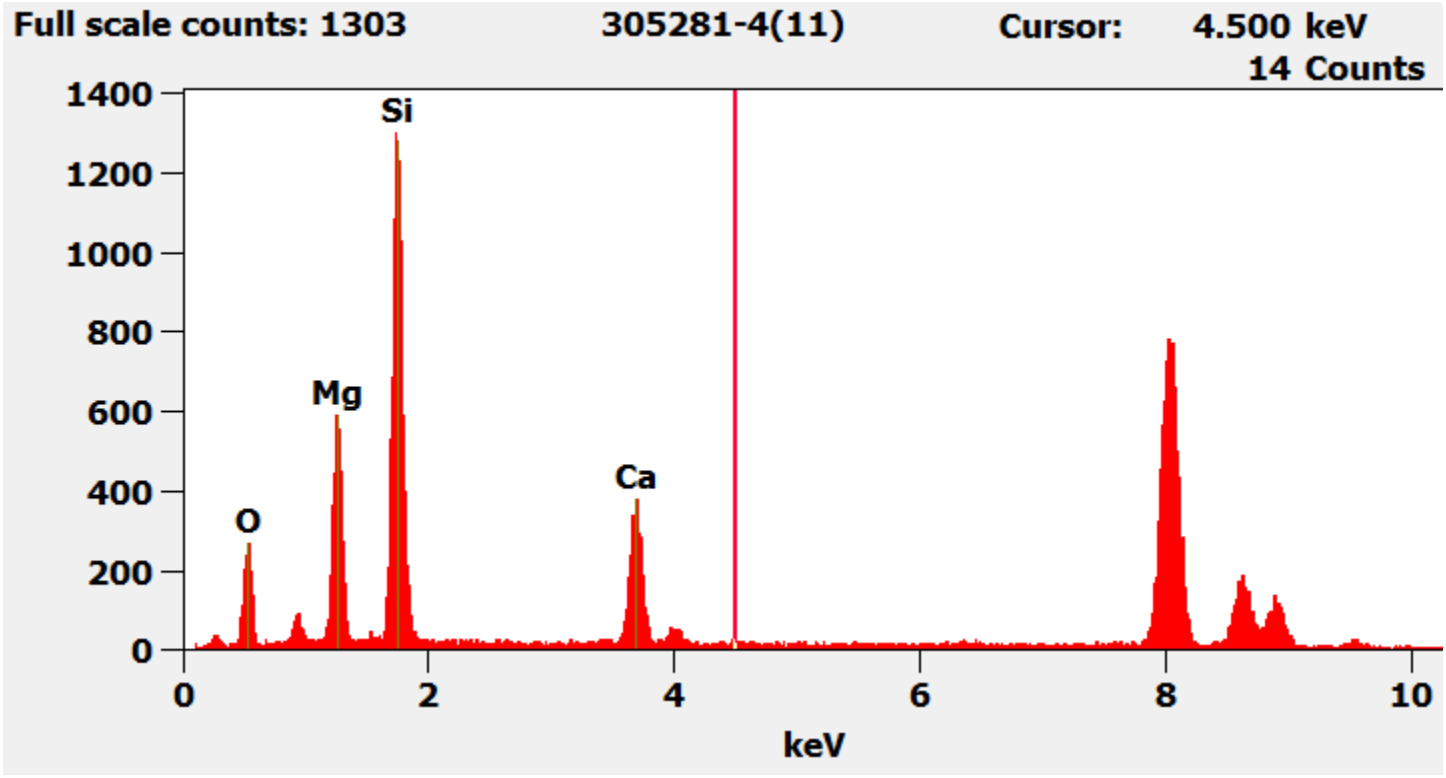




Element Line	Weight %	Weight % Error
O K	54.02	± 3.07
Mg K	14.93	± 0.54
Si K	27.96	± 0.56
Si L	---	---
Ca K	3.09	± 0.18
Ca L	---	---
Total	100.00	

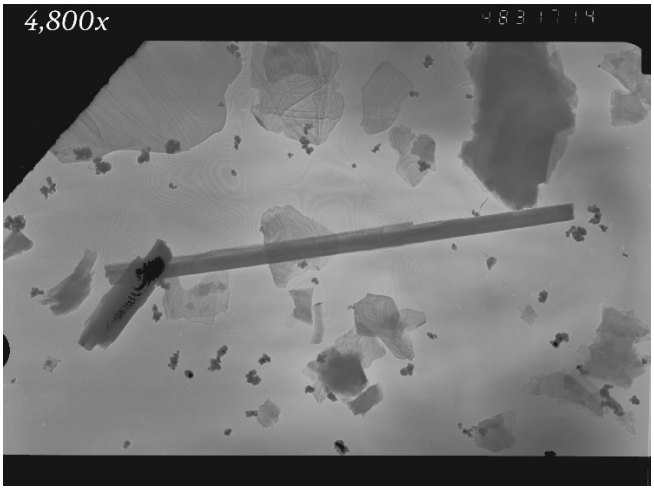
305281-4 Particle 12 4.5 x 1.7 microns Tremolite

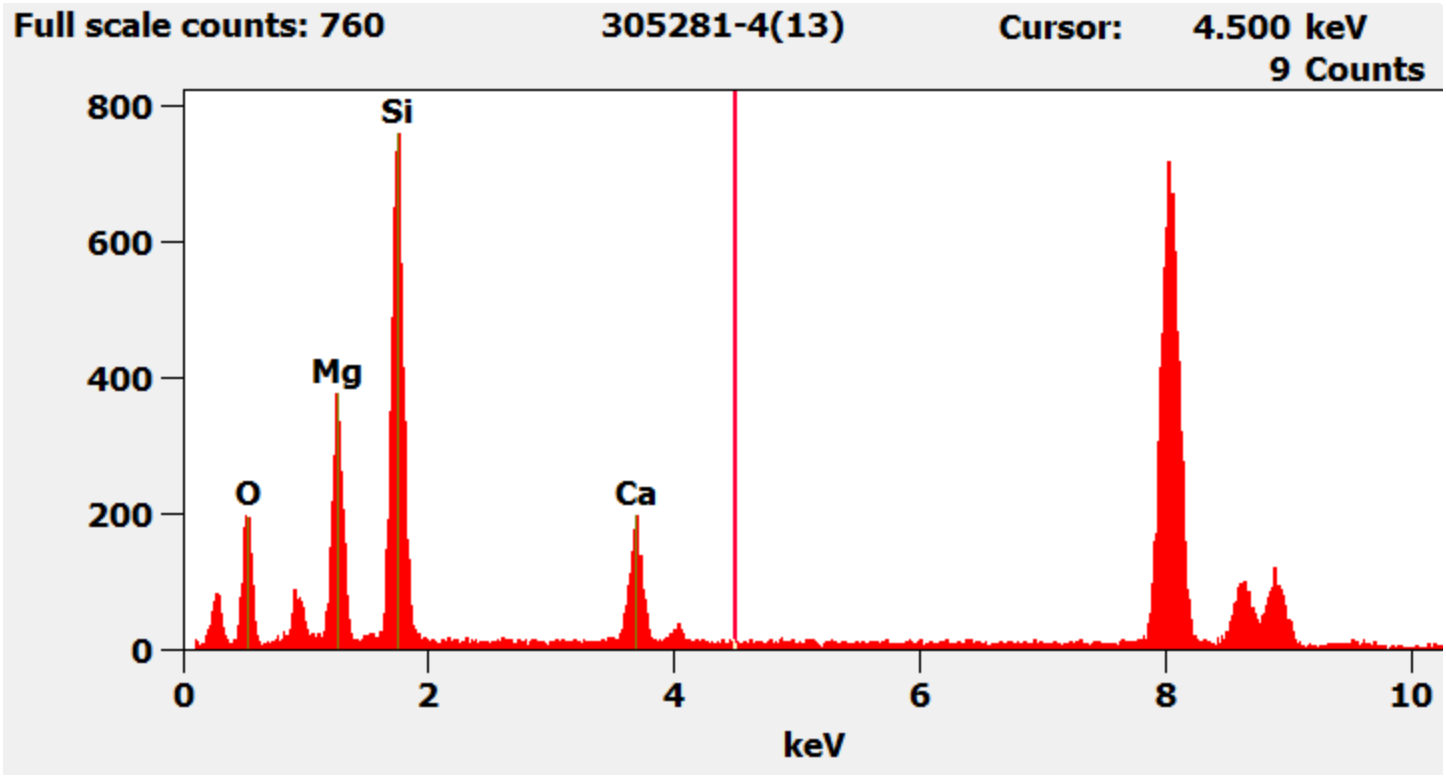




Element Line	Weight %	Weight % Error
O K	48.73	± 1.26
Mg K	17.21	± 0.28
Si K	29.82	± 0.30
Si L	---	---
Ca K	4.24	± 0.10
Ca L	---	---
Total	100.00	

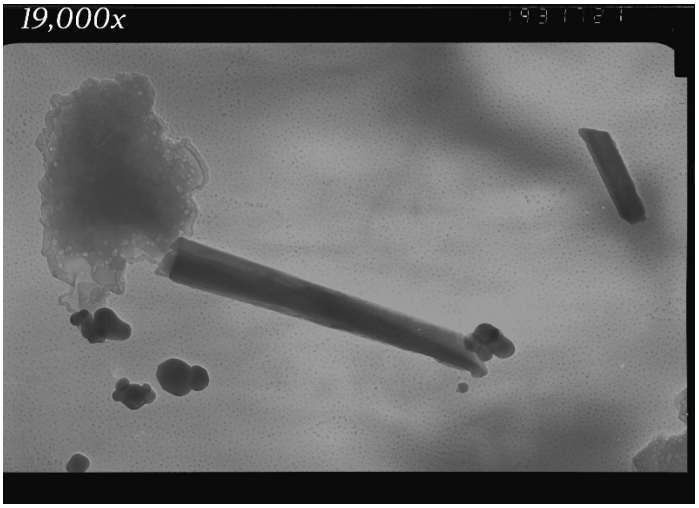
305281-4 Particle 13 & 14 4.4 x 1.3 microns 15.6 x 0.8 Tremolite

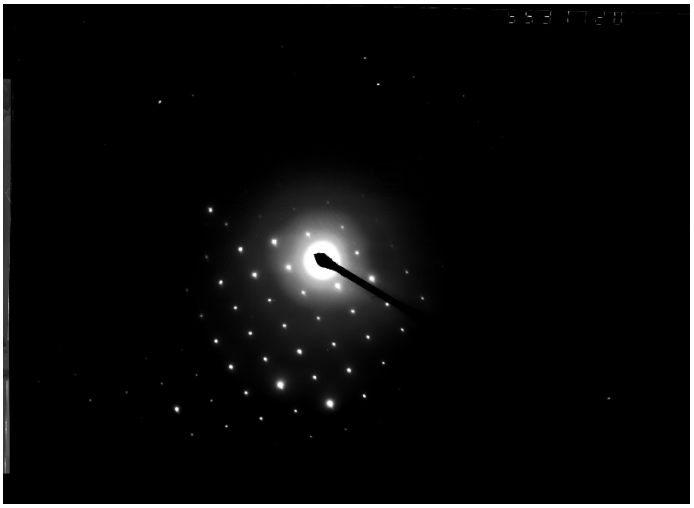


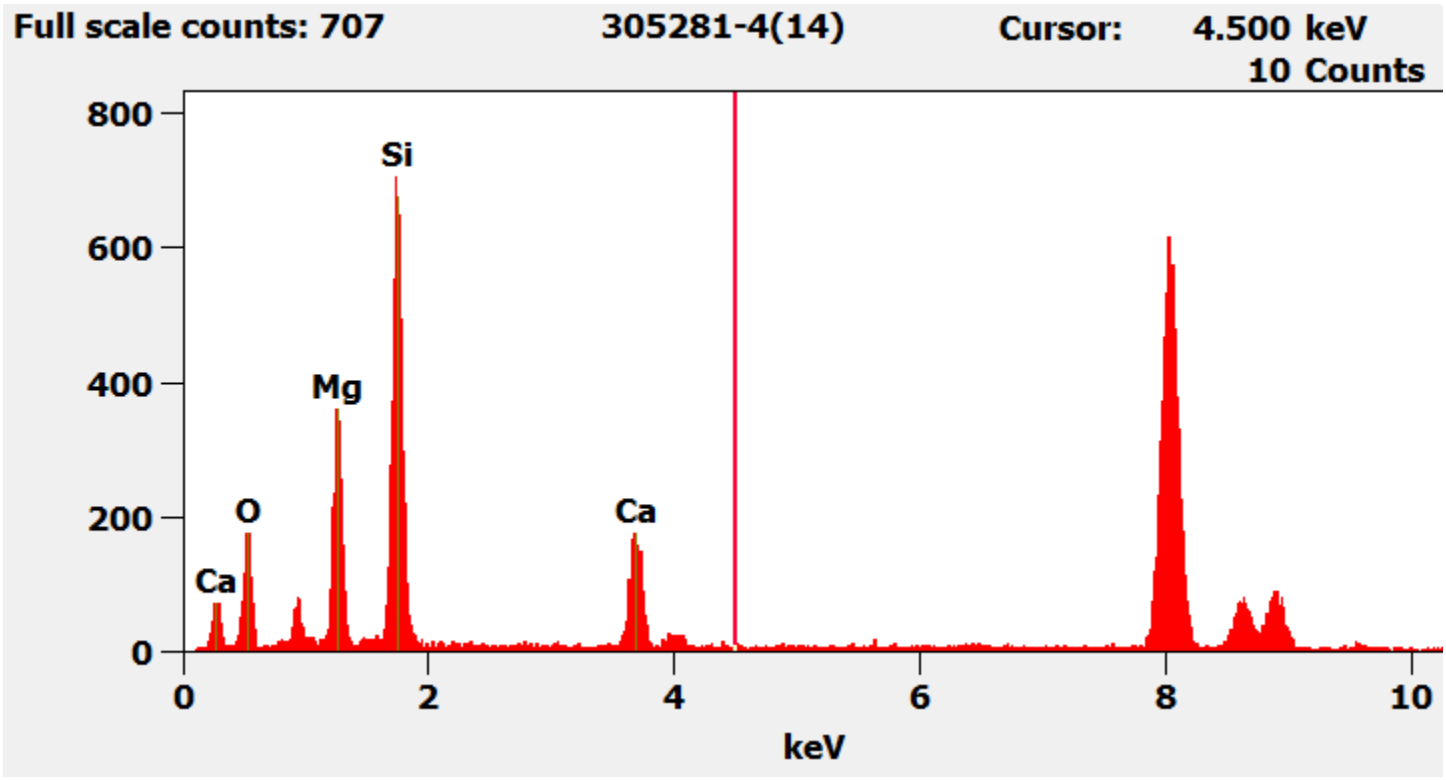


Element Line	Weight %	Weight % Error
O K	52.28	± 1.64
Mg K	17.40	± 0.35
Si K	27.41	± 0.36
Si L	---	---
Ca K	2.92	± 0.11
Ca L	---	---
Total	100.00	

305281-4 Particle 15 2.7 x 0.26 microns Tremolite

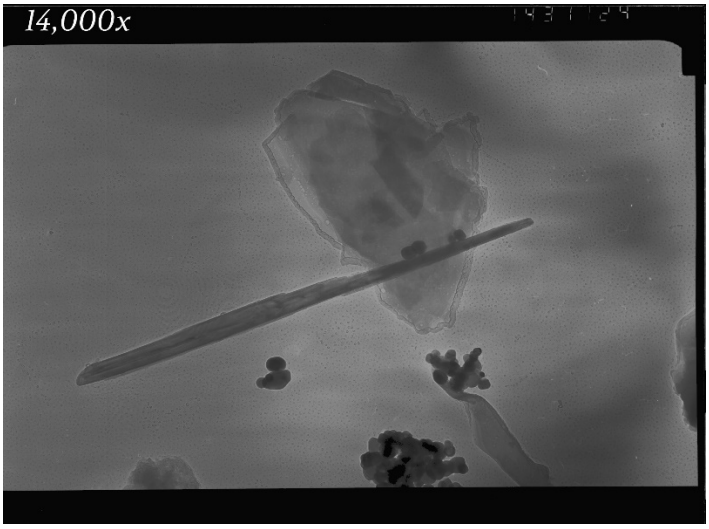


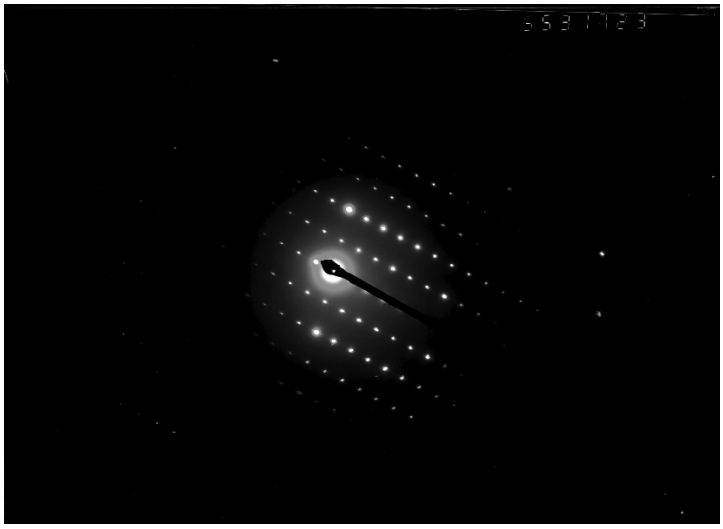


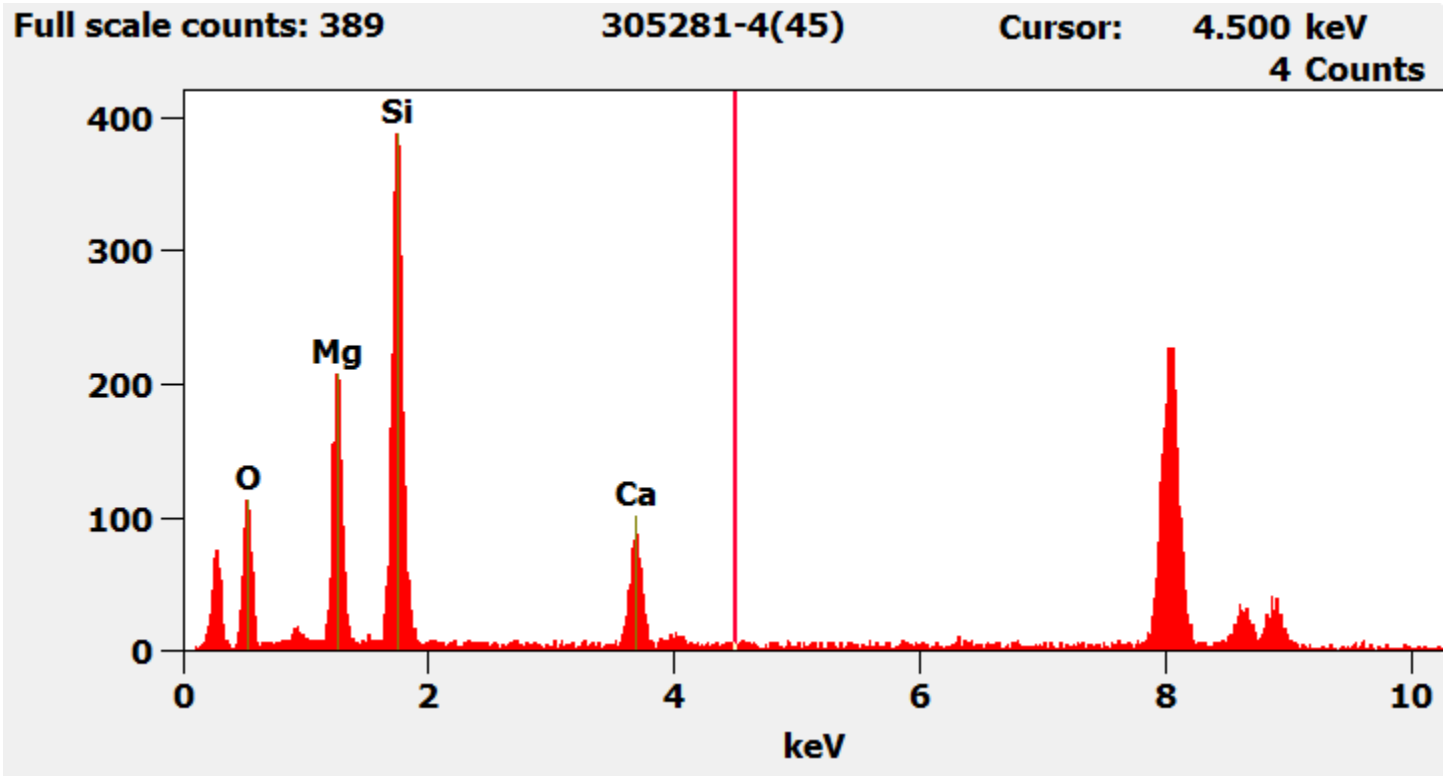


Element Line	Weight %	Weight % Error
O K	59.21	± 2.63
Mg K	17.88	± 0.68
Si K	19.08	± 0.52
Si L	---	---
Ca K	3.84	± 0.14
Ca L	---	---
Total	100.00	

305281-4 Particle 51 4.8 x 0.2 microns Tremolite







Element Line	Weight %	Weight % Error
O K	52.86	± 2.32
Mg K	17.64	± 0.46
Si K	26.86	± 0.48
Si L	---	---
Ca K	2.64	± 0.13
Ca L	---	---
Total	100.00	

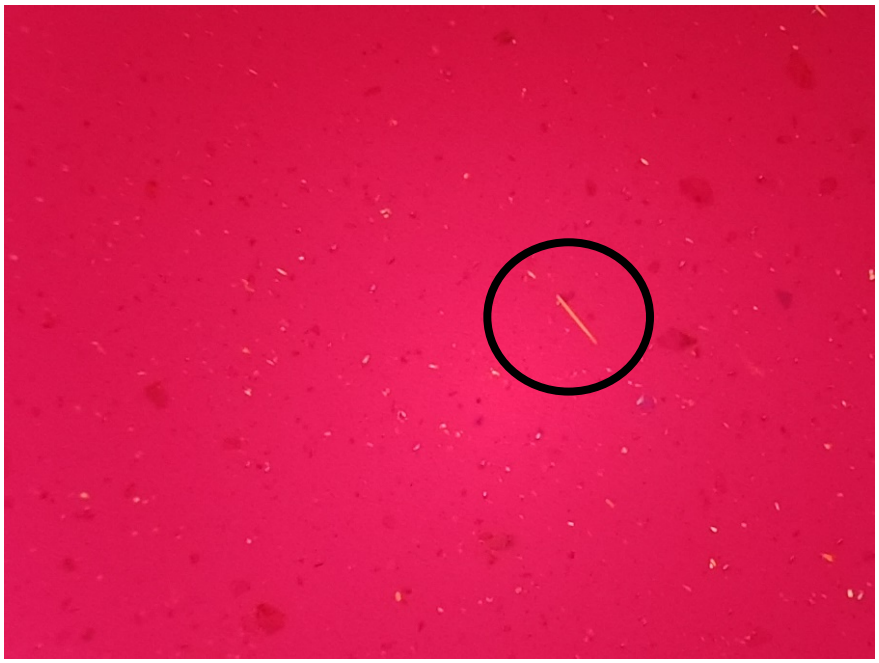
305281-5, 5A, 5B, Client Sample D31

PLM

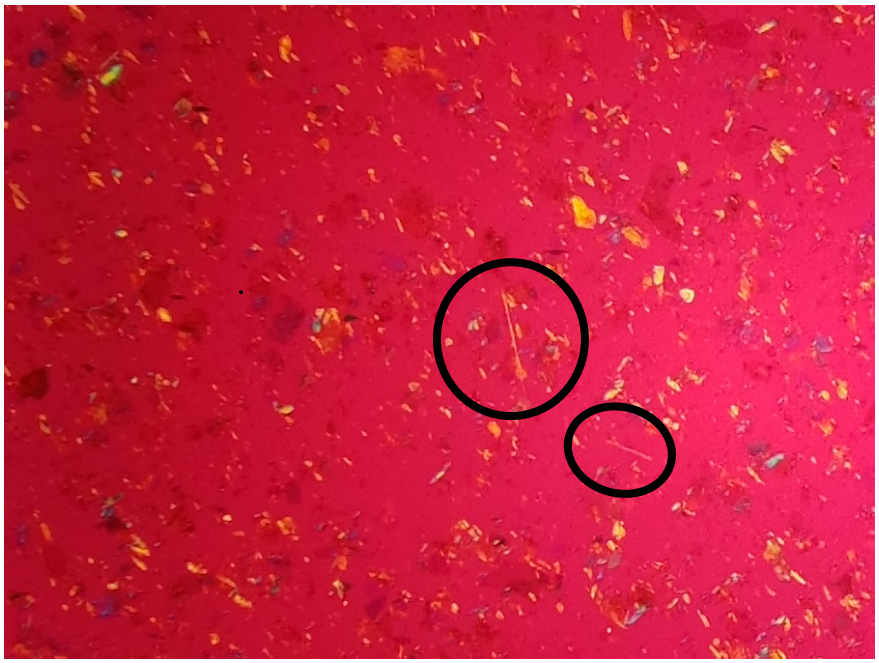
All three aliquots of sample D31 were analyzed by (b) (6) on January 31, 2019. Tremolite was observed on all three aliquots but no points were counted. No other asbestos was detected. The results were calculated using the equations detailed in the calculations section.

305281-5	<0.18% Tremolite detected
305281-5A	<0.18% Tremolite detected
305281-5B	<0.18% Tremolite detected

305281-5 Tremolite 100x



305281-5A Tremolite 100x



305281-5B Tremolite 100x

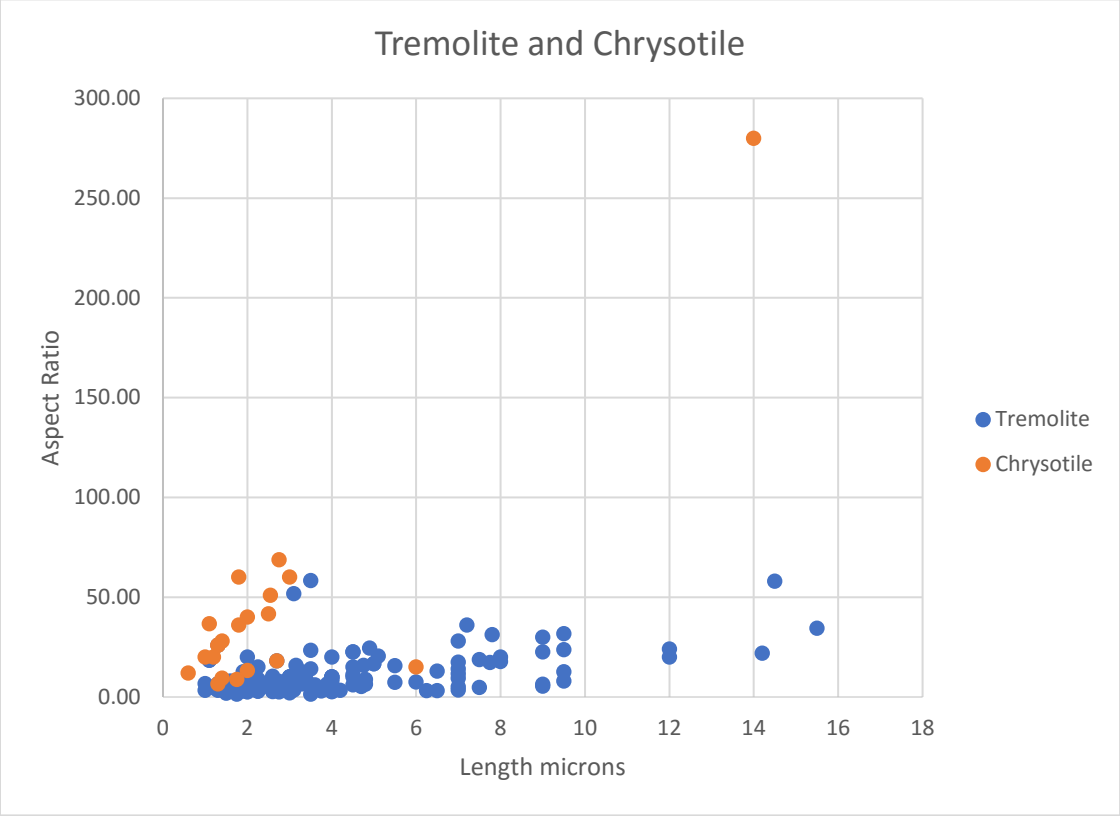
TEM

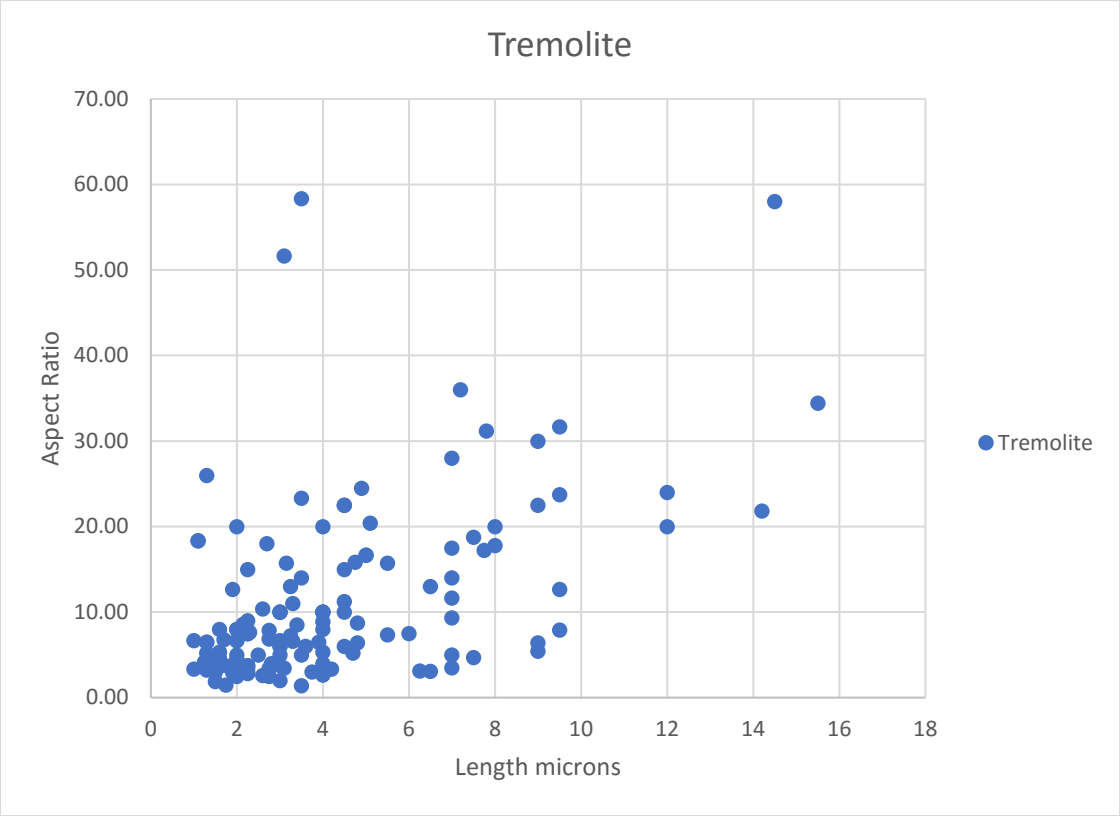
Samples 5, 5A and 5B were analyzed by (b) (6) on January 15-17, 2019. Talc is the main constituent in all three aliquots. The talc is primarily in plate form but there are also some talc fibers and ribbons. Tremolite and chrysotile was detected on all three aliquots. 20 grid openings were analyzed on all three aliquots. Sample 5 had 43 tremolite and 7 chrysotile particles counted. Sample 5A had 43 tremolite and 5 chrysotile particles. Sample 5B had 55 Tremolite and 9 chrysotile particles. The length and width of each particle was recorded. The mass of the tremolite was calculated using both the ASTM D5756 and ISO 22262-2 methods. The mass of the chrysotile was calculated using the ASTM D5756 method.

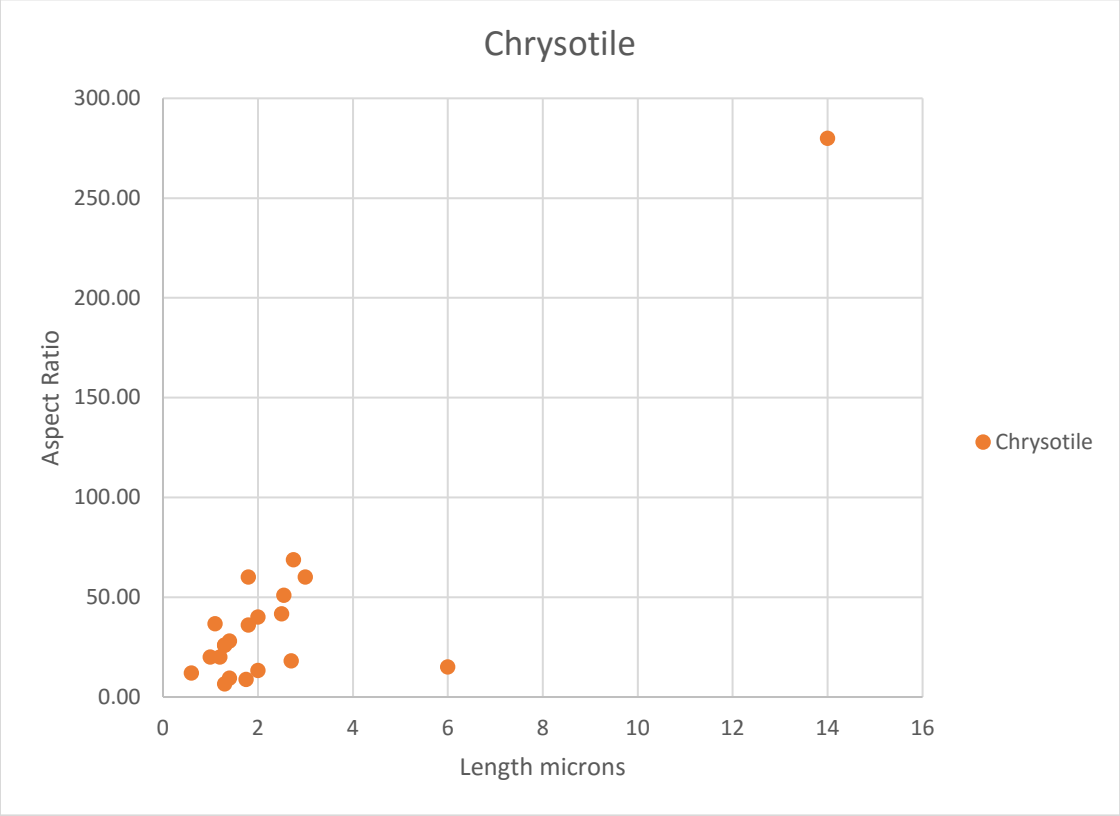
305281-5	0.156% to 0.244%
305281-5A	0.171% to 0.268%
305281-5B	0.134% to 0.210%

The tremolite particles varied in morphology from small blocks to very long, high length to width aspect ratio fibers. The length to width aspect ratio of particles counted ranged from 1.40 to 58.33. The mean aspect ratio is 11.0. The chemistry of the tremolite observed consisted of O, Mg, Si, and Ca. The chrysotile particles were high length to width aspect ratio fibers. The aspect ratio ranged from 6.5 to 280. The mean aspect ratio was 41.8. The chemistry of the chrysotile was O, Mg, Si, Fe.

The following charts are a plot of the aspect ratio vs length for all the particles counted over all three aliquots, for the tremolite only, and for the chrysotile only.

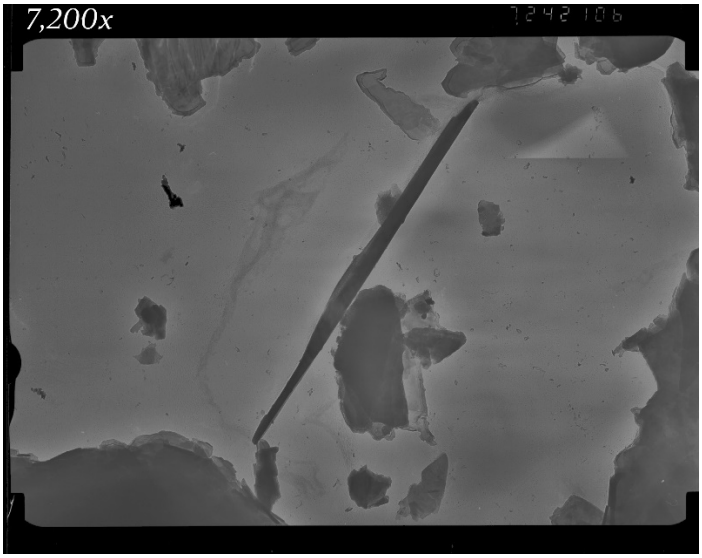

Tremolite and Chrysotile
300.00
250.00
200.00
150.00
100.00
50.00
0.00
Aspect Ratio
0
2
4
6
8
10
12
14
16
18
Length microns
Tremolite
Chrysotile

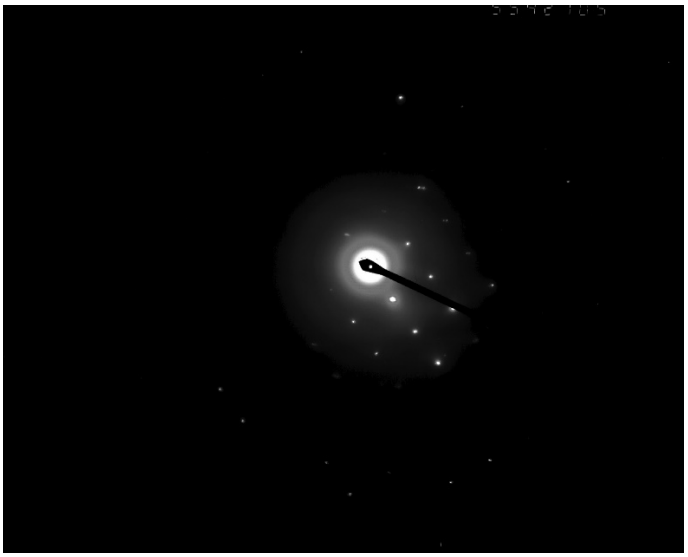

Tremolite
70.00
60.00
50.00
40.00
30.00
20.00
10.00
0.00
Aspect Ratio
0
2
4
6
8
10
12
14
16
18
Length microns
Tremolite

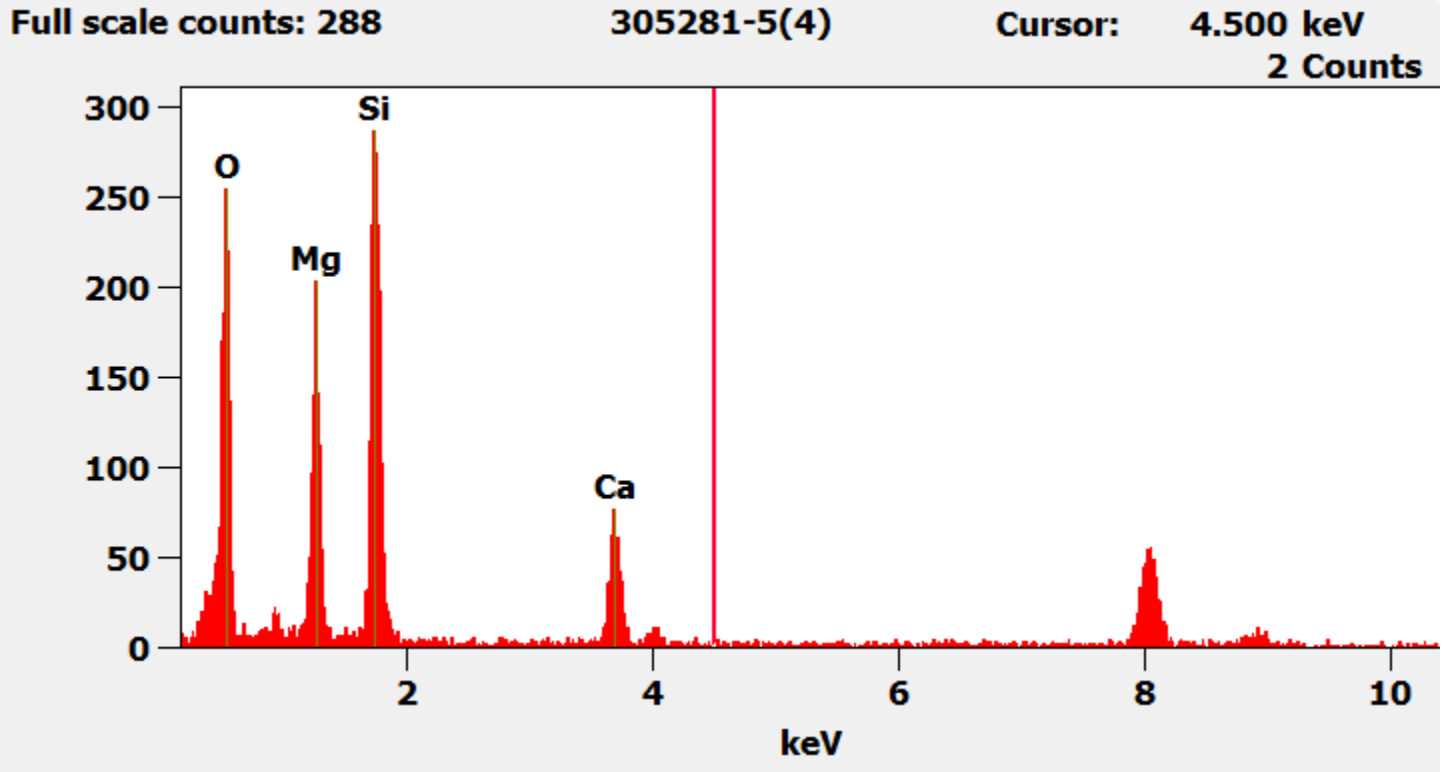


Below are pictures, diffraction patterns, and chemistry from some of the counted particles. The unidentified peaks in chemistry spectra are copper, zinc, and carbon. Those peaks are from the TEM specimen holder and specimen grid.

305281-5 Particle 3 8.0 x 0.40 microns Tremolite

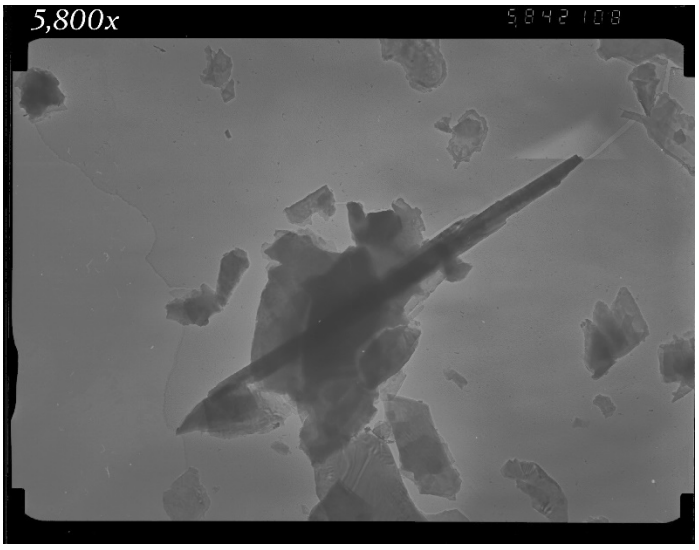


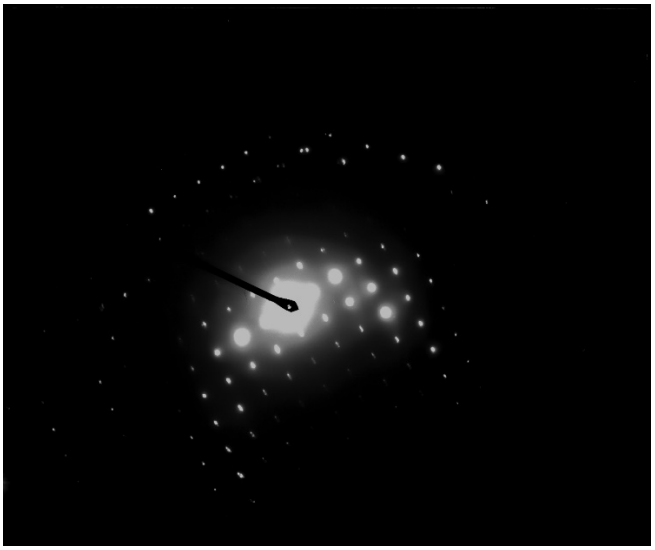


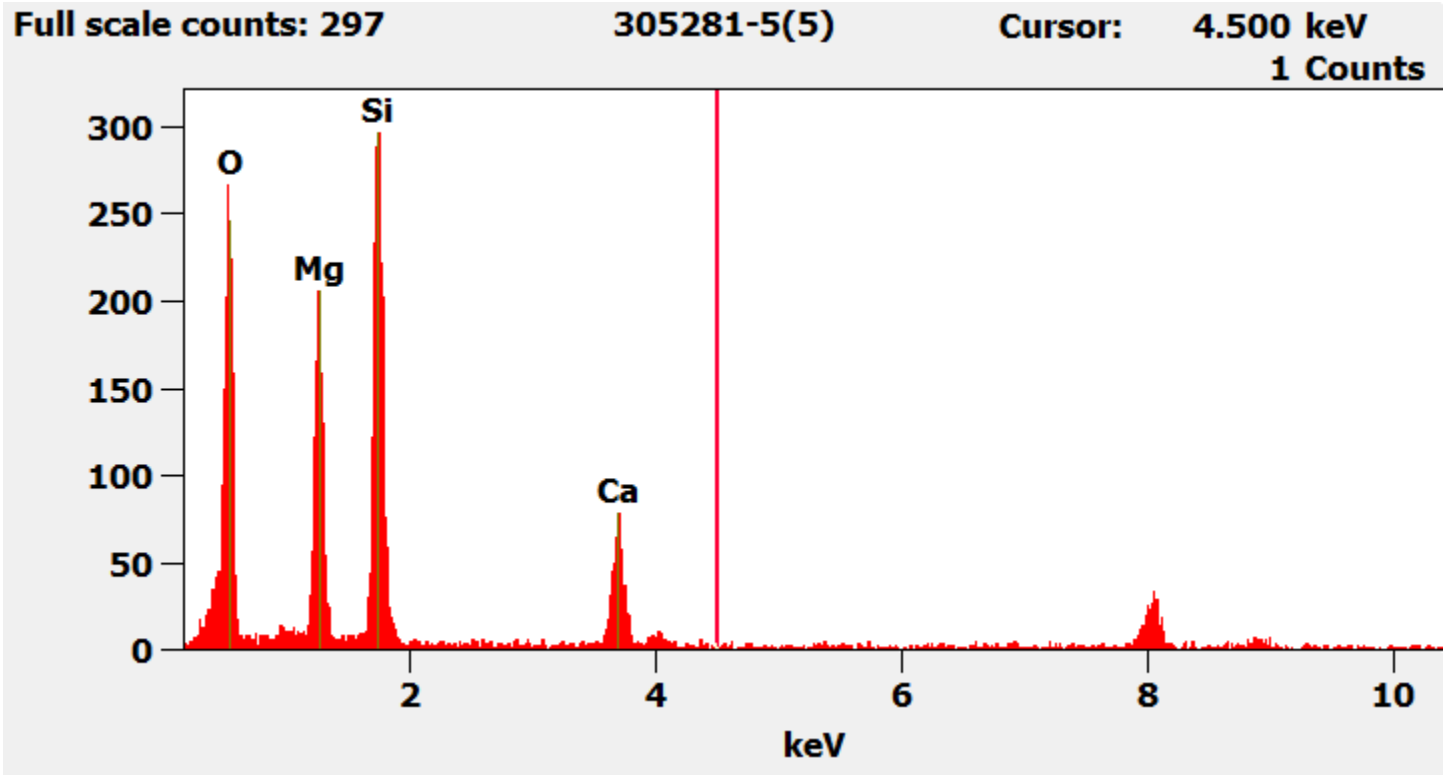


Element Line	Weight %	Weight % Error
O K	59.45	± 1.68
Mg K	16.67	± 0.51
Si K	22.15	± 0.47
Si L	---	---
Ca K	1.73	± 0.09
Ca L	---	---
Total	100.00	

305281-5 Particle 4 12.0 x 0.60 microns Tremolite

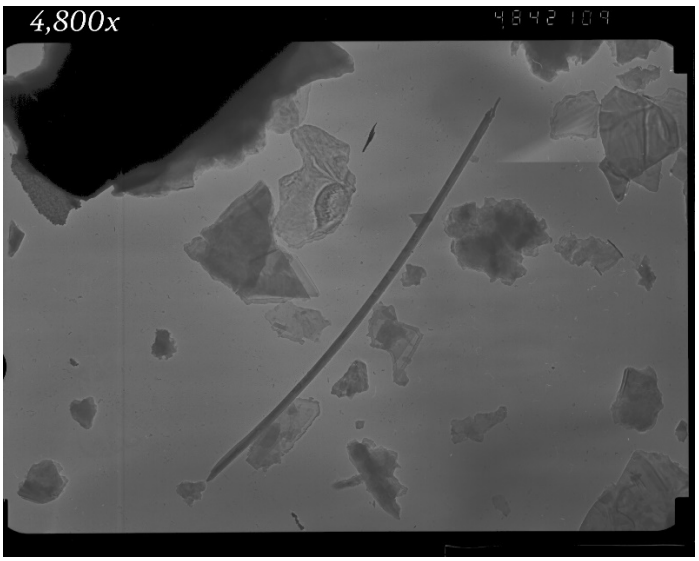


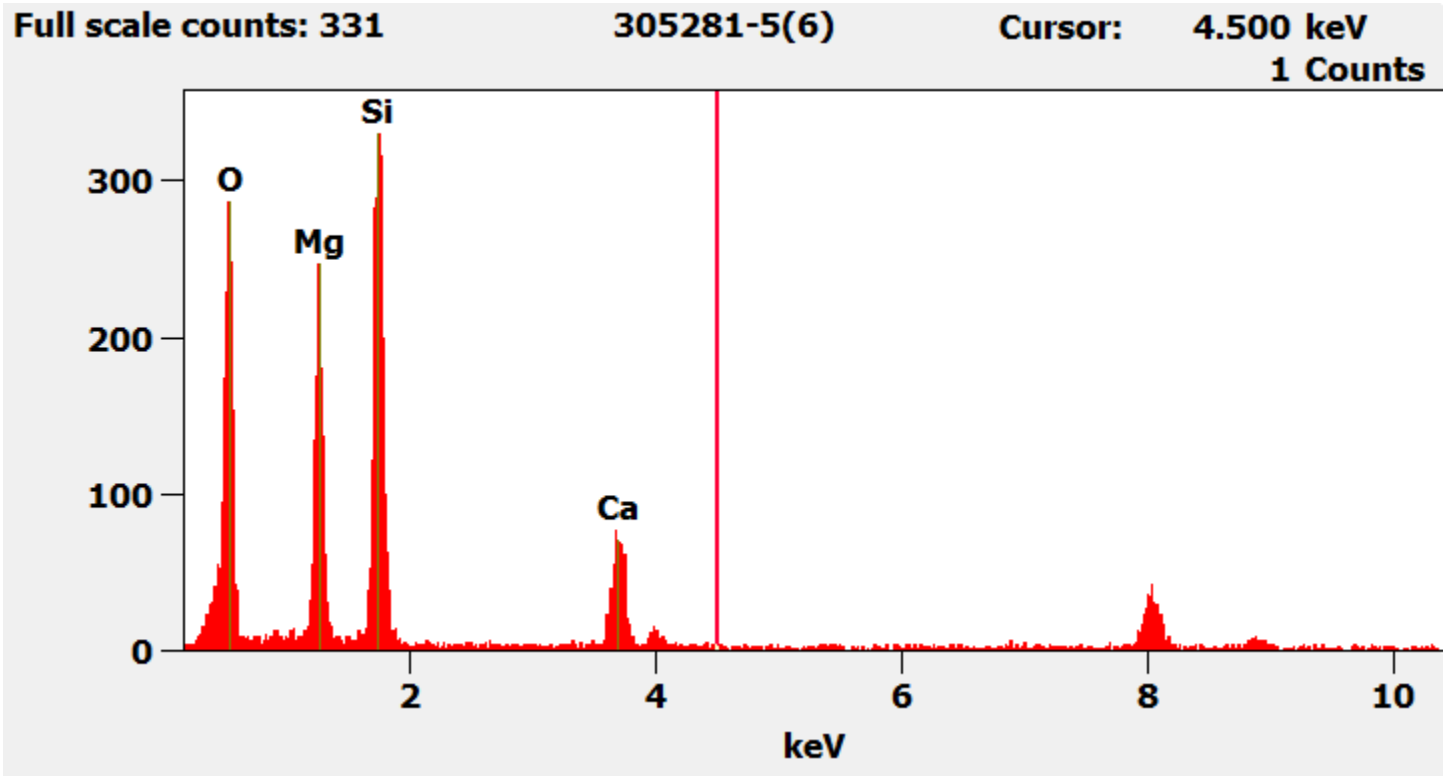




Element Line	Weight %	Weight % Error
O K	59.77	± 1.60
Mg K	17.11	± 0.51
Si K	21.58	± 0.45
Si L	---	---
Ca K	1.54	± 0.09
Ca L	---	---
Total	100.00	

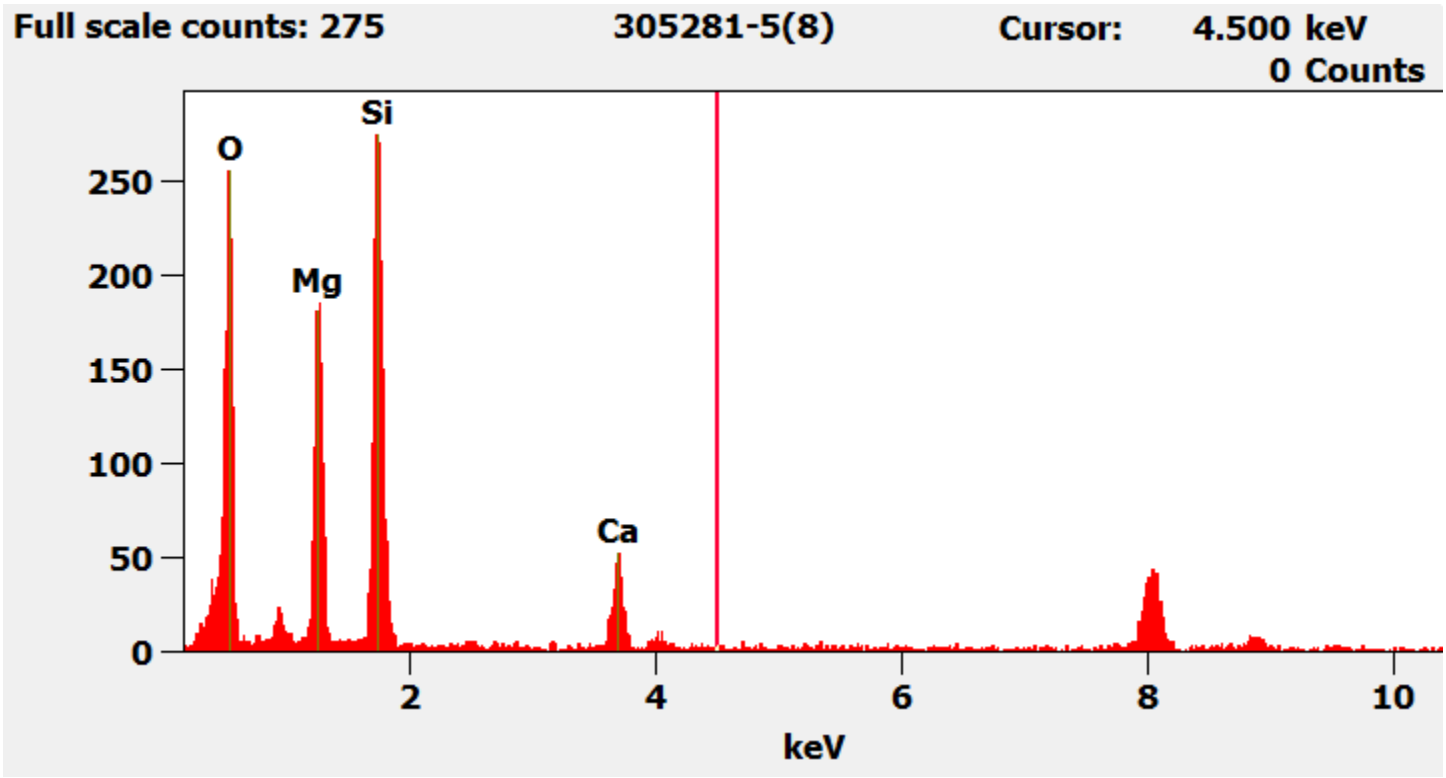
305281-5 Particle 5 12.0 x 0.60 microns Tremolite





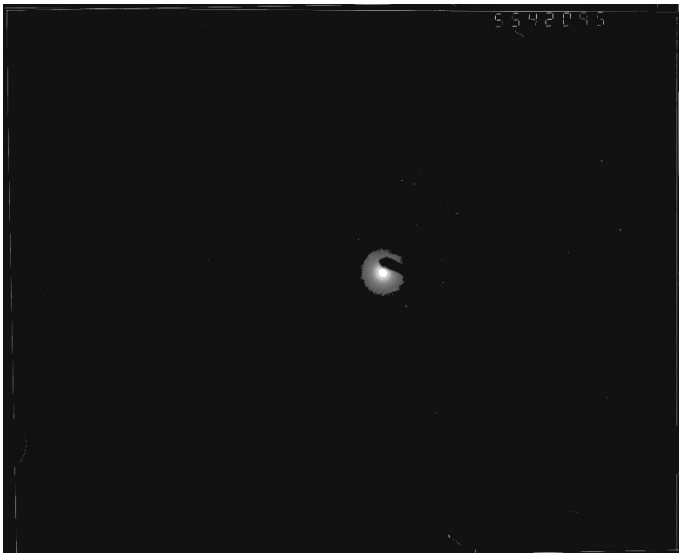
Element Line	Weight %	Weight % Error
O K	59.22	± 1.53
Mg K	17.13	± 0.47
Si K	22.06	± 0.43
Si L	---	---
Ca K	1.60	± 0.09
Ca L	---	---
Total	100.00	

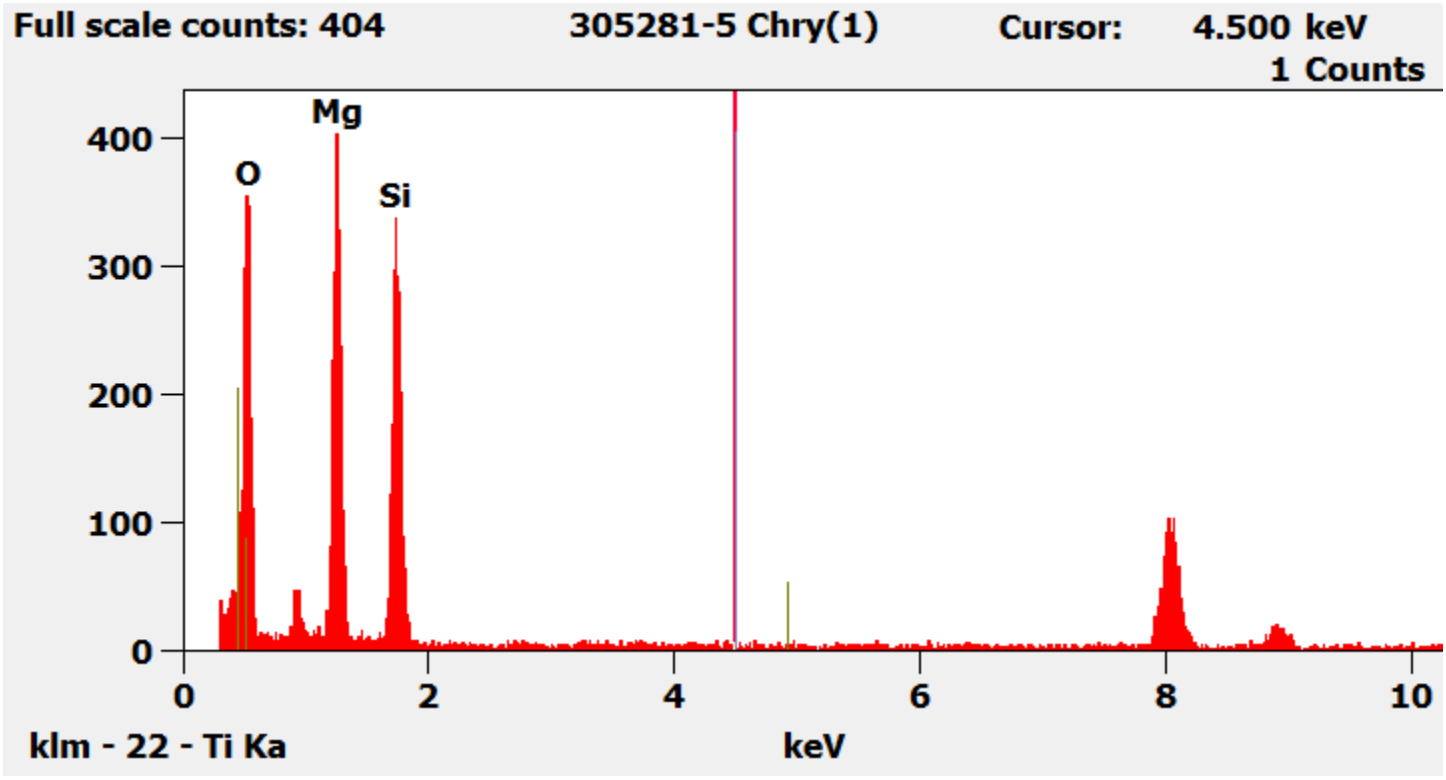
305281-5 Particle 6 1.70 x 0.25 microns Tremolite



Element Line	Weight %	Weight % Error
O K	58.34	± 2.26
Mg K	17.65	± 0.67
Si K	22.56	± 0.63
Si L	---	---
Ca K	1.45	± 0.12
Ca L	---	---
Total	100.00	

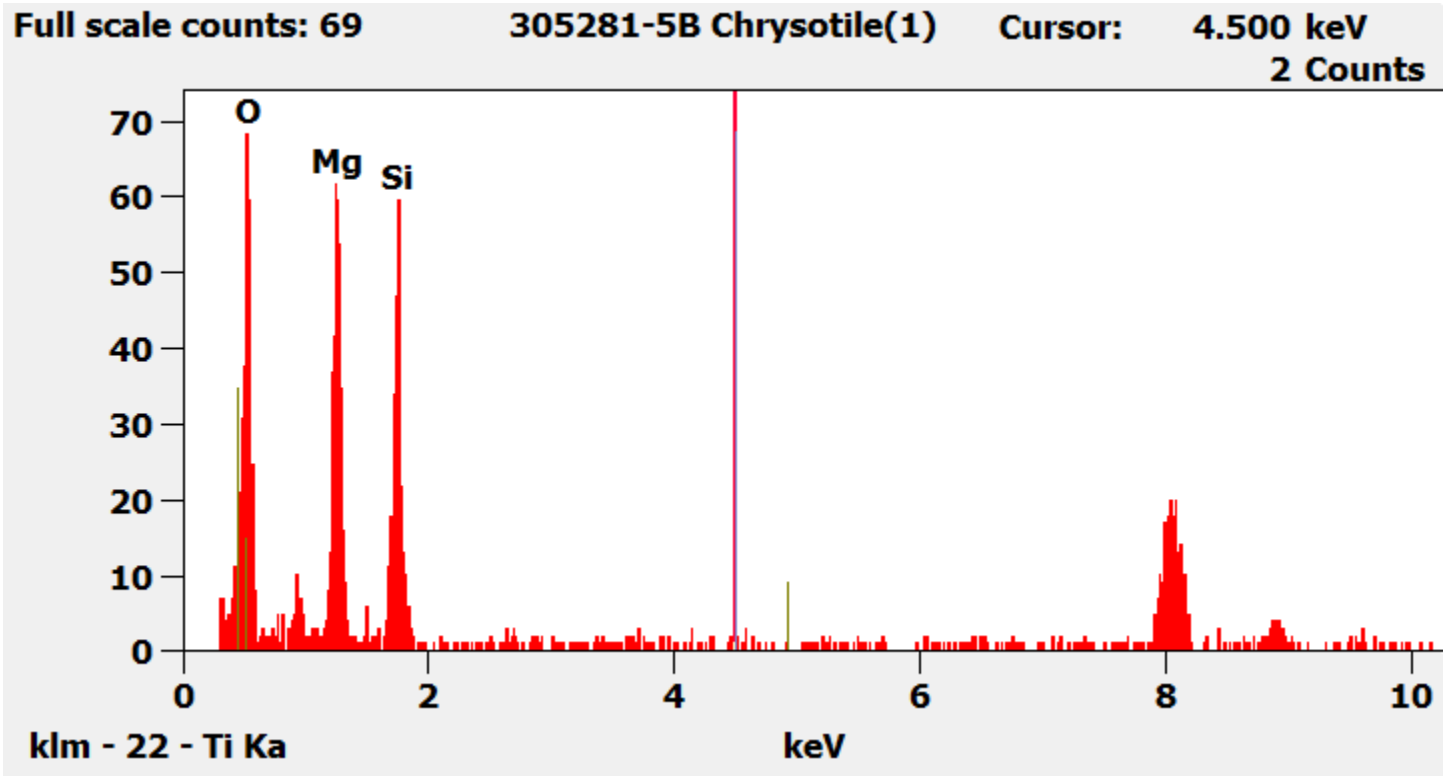
305281-5B Particle 49 1.40 x 0.15 microns Chrysotile





Element Line	Weight %	Weight % Error
O K	55.89	± 1.44
Mg K	24.04	± 0.49
Si K	20.07	± 0.42
Si L	---	---
Total	100.00	

305281-5B Particle 56 2.50 x 0.06 microns Chrysotile



Element Line	Weight %	Weight % Error
O K	60.28	± 3.44
Mg K	22.85	± 1.17
Si K	16.86	± 0.96
Si L	---	---
Total	100.00	