Asbestos in Talc Testing

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Presented at the

Testing Methods for Asbestos in Talc and Cosmetic Products Containing Talc Public Meeting

U.S. Food and Drug Administration

White Oak Campus

Bldg. 31 Conference Center, The Great Room

Silver Spring, Maryland

February 4th, 2020


IGNORE
ME


What
is
ASBESTOS?

Asbestos (a few definitions):

- EPA: *Asbestos is a mineral fiber that occurs in rock and soil*
- USGS: *"Asbestos" is not a mineralogical term, but rather a commercial and industrial term used to describe a group of specific silicate minerals that form bundles of long, very thin mineral fibers.*
- ASTM Soil: 3.1.2 asbestos, n— *a collective term that describes a group of naturally occurring, inorganic, highly-fibrous, silicate minerals that are easily separated into long, thin, flexible, strong fibers when crushed or processed.*
- **CFR 40 763.80: *The asbestiform varieties of: chrysotile (serpentine); crocidolite (riebeckite); amosite (cummingtonite grunerite); anthophyllite; tremolite; and actinolite.***

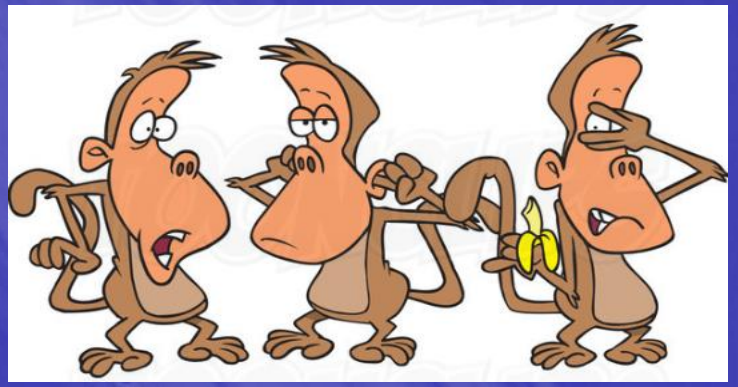
Asbestos (more as we know it):

- CFR 40 763.80: *The asbestiform varieties of: chrysotile (serpentine); crocidolite (riebeckite); amosite (cummingtonite grunerite); anthophyllite; tremolite; and actinolite.*

Asbestiform

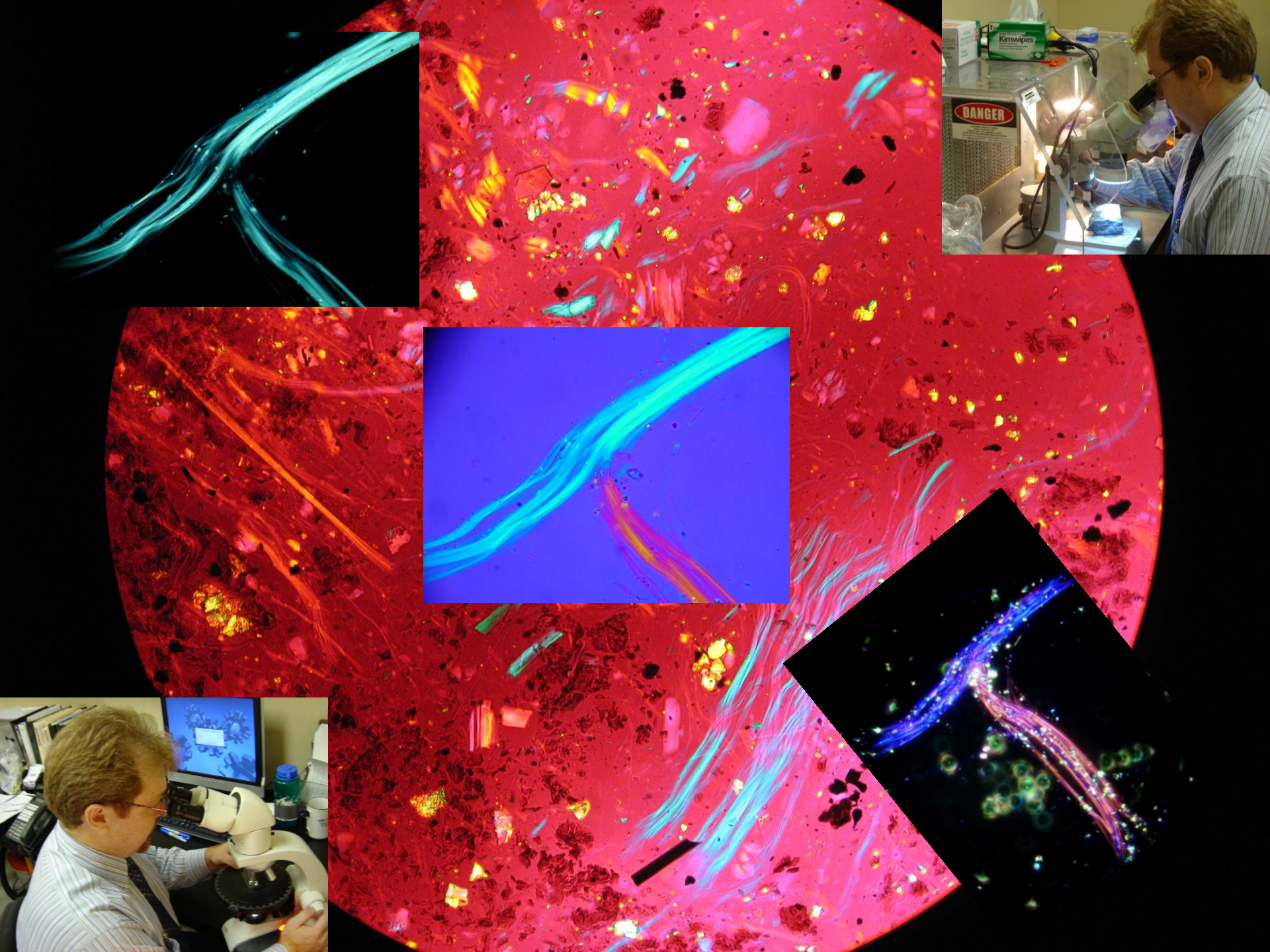
- 3.1.1 *asbestiform*, n— type of fibrous habit in which the fibers are separable into thinner fibers and ultimately into fibrils. Discussion—This habit accounts for greater flexibility and higher tensile strength than other habits of the same mineral.

Contradictions.



- Is a single fiber asbestos? Fibril?
- Can asbestos be a constituent of a non-asbestiform population, or is a “bundle” asbestiform regardless of aspect?
- Are particles or structures of the minerals regulated as asbestos that don’t meet *some* parameters of counting criteria asbestos?
- Are particles or structures of the minerals regulated as asbestos that don’t meet *all* parameters of a population asbestos?
- *Is asbestos a definable thing, or just a great concept?*


Asbestos Testing Tools


Kimwipes
DANGER

PLM Limitations

PLM is a useful tool for the determination of minerals present that constitute the majority of the rock, e.g., those minerals at or above 1-5% of the whole rock.

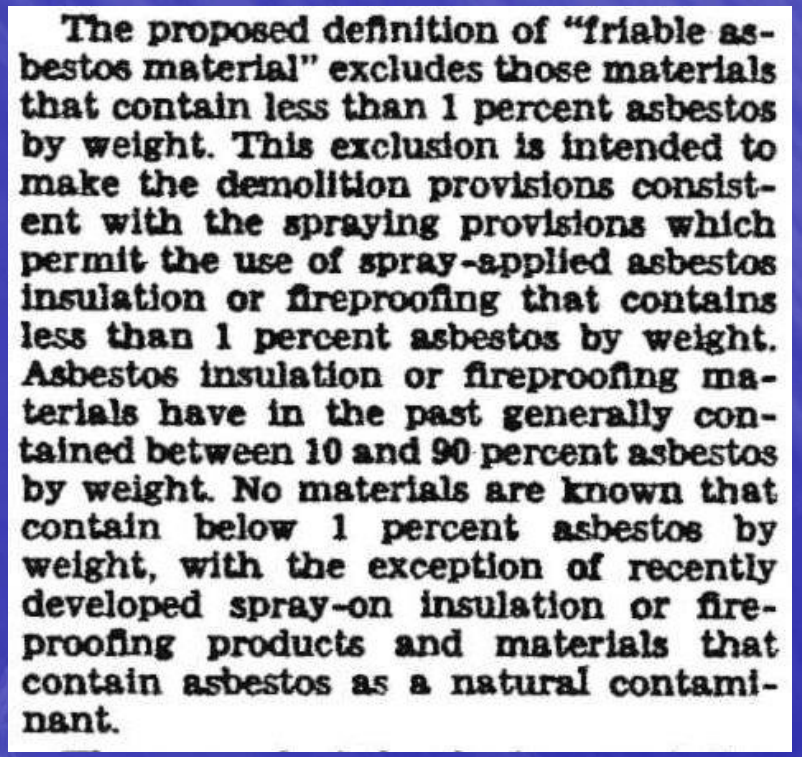
Although PLM is a microscopic technique, the determination of the asbestiform habit of a given mineral is possible by this technique, but only the resolvable size fraction.

Why PLM and 1%?

- A NESHAP for asbestos was proposed in 1973.
- The first draft had no mention of amount of asbestos in building materials, and producers and manufacturers provided input to EPA that if they included small amounts of "contaminant" asbestos, *you wouldn't be able to buy many building products and other materials.*
- EPA wanted to control materials manufactured with asbestos. A review of product manufacturers formulas determined materials made with asbestos would contain more than 1% by weight.

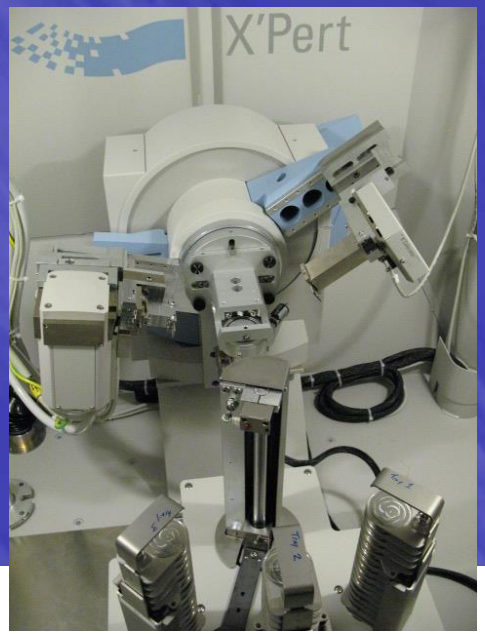
Page 38065 of the *Federal Register* on October 25, 1974:

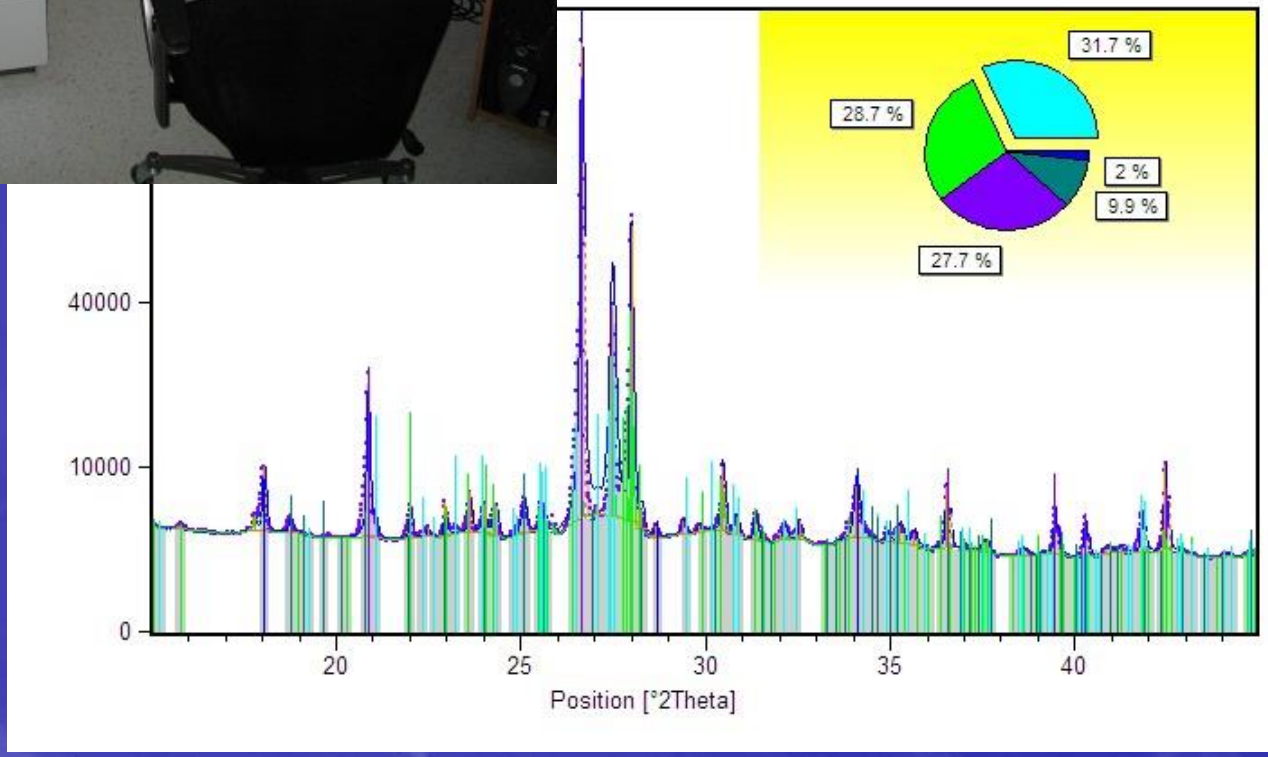
Essentially, the ban on spray fireproofing was given a 1% cut-off to allow products like Monokote 4, which had less than 1% Tremolite/Actinolite (Libby amphiboles) content.



The proposed definition of "friable asbestos material" excludes those materials that contain less than 1 percent asbestos by weight. This exclusion is intended to make the demolition provisions consistent with the spraying provisions which permit the use of spray-applied asbestos insulation or fireproofing that contains less than 1 percent asbestos by weight. Asbestos insulation or fireproofing materials have in the past generally contained between 10 and 90 percent asbestos by weight. No materials are known that contain below 1 percent asbestos by weight, with the exception of recently developed spray-on insulation or fireproofing products and materials that contain asbestos as a natural contaminant.


X'Pe

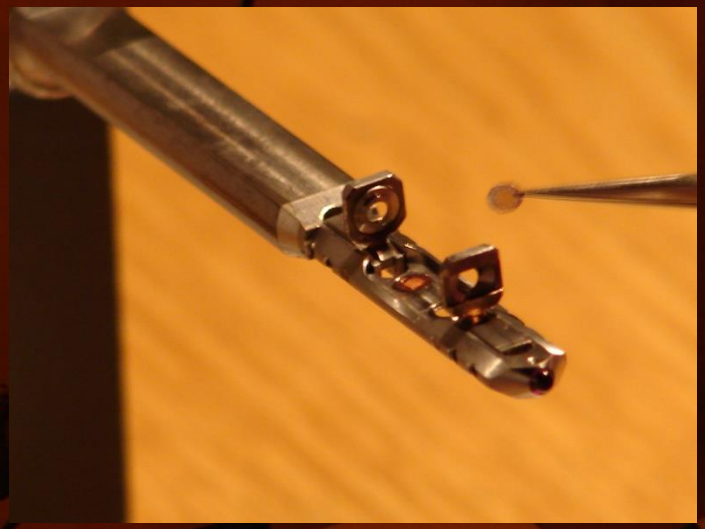

X'Pert

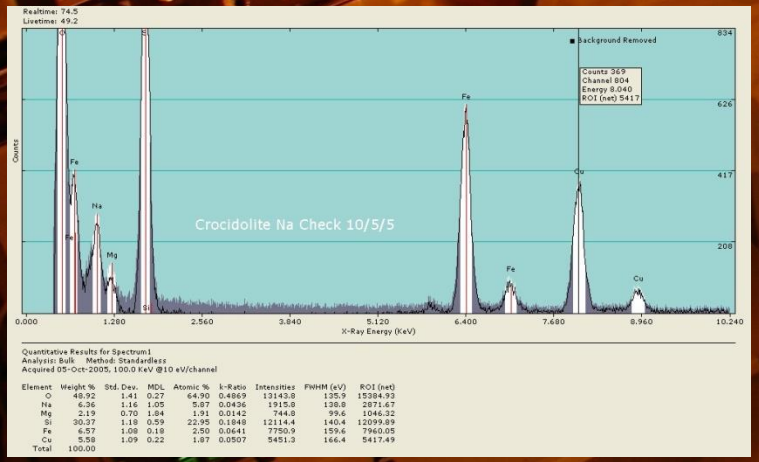

31.7 %
28.7 %
2 %
9.9 %
27.7 %
40000
10000
0
20
25
30
35
40
Position [°2Theta]

XRD Limitations

XRD is a useful tool for the determination of minerals present that constitute the majority of the rock, e.g., those minerals at or above 1-5% of the whole rock.

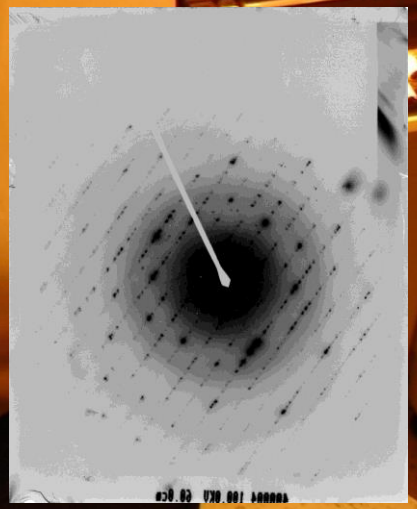
XRD is not a microscopic technique. Therefore, the determination of the asbestiform habit of a given mineral (most easily determined with the eyes) is veritably impossible by this technique alone.

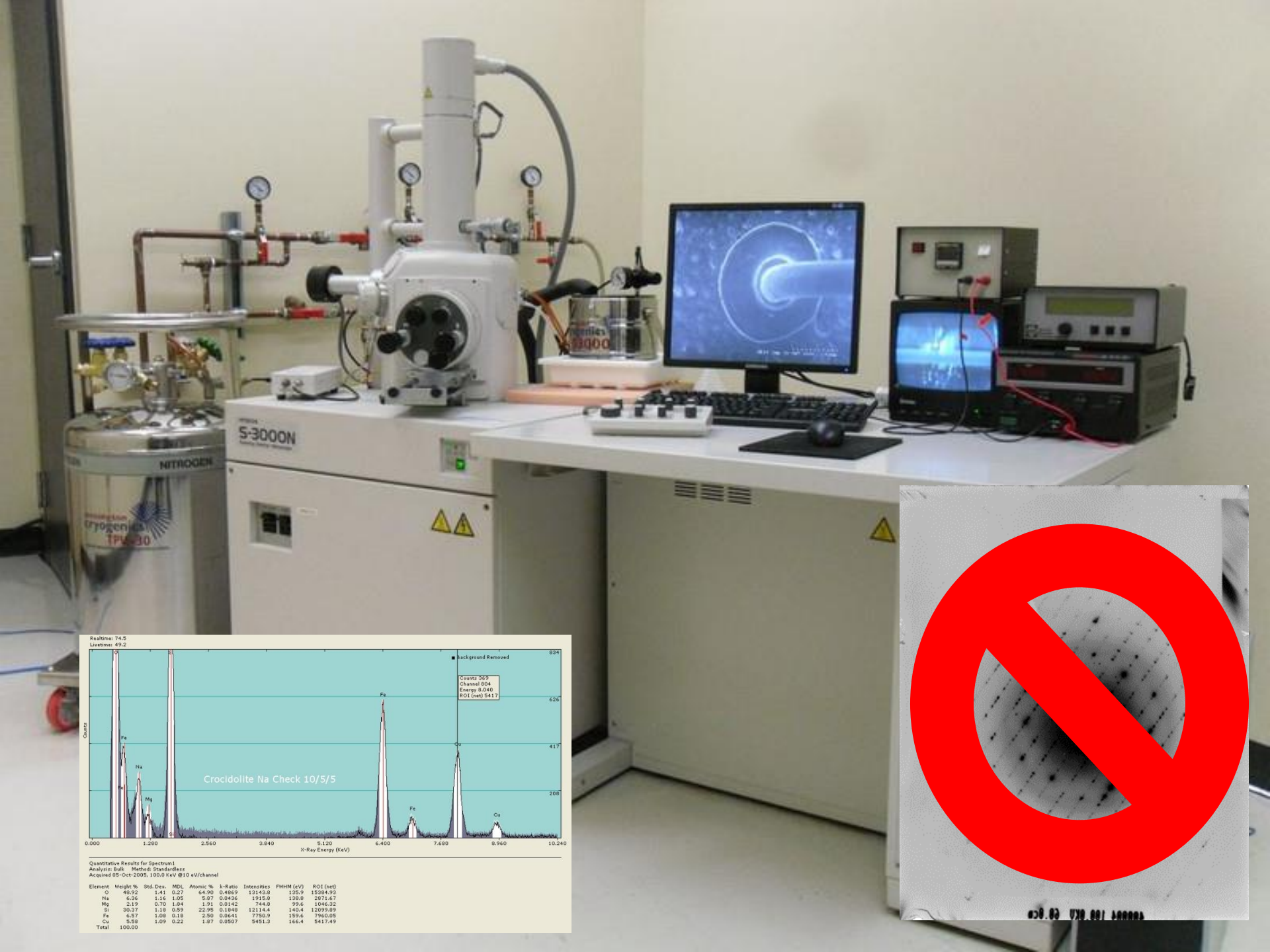




Quantitative Results for Spectrum1
Analysis: Bulk Method: Standardless
Acquired 05-Oct-2005, 100.0 KeV @10 eV/channel

Element	Weight %	Std. Dev.	MDL	Atomic %	k-Ratio	Intensities	FWHM (eV)	ROI (net)
O	48.92	1.41	0.27	64.90	0.4869	13143.8	135.9	15384.93
Na	6.36	1.16	1.05	5.87	0.0436	1915.8	138.8	2671.67
Mg	2.19	0.70	1.04	1.91	0.0142	744.8	99.6	1046.32
Si	30.37	1.18	0.59	22.95	0.1848	12114.4	140.4	12099.89
Fe	6.57	1.09	0.18	2.50	0.0641	7750.9	159.6	7960.05
Cu	5.58	1.09	0.22	1.87	0.0507	5451.3	166.4	5417.49
Total	100.00							

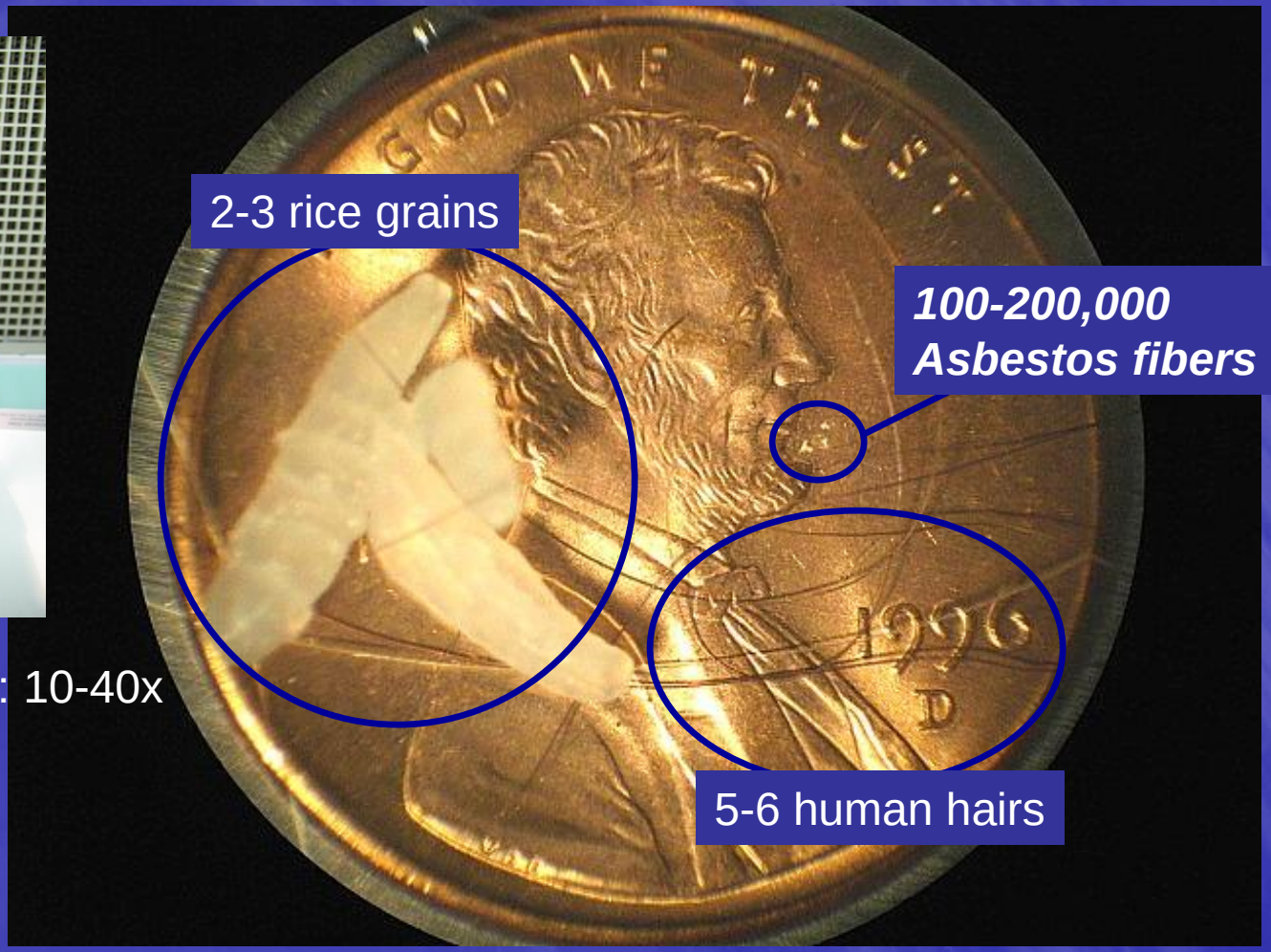



S-3000N
NITROGEN

Realtime: 74.5
Livetime: 49.2
Background Removed
Counts 369
Channel 804
Energy 8.040
ROI (net) 5417
Crocidolite Na Check 10/5/5
Counts
X-Ray Energy (KeV)
Quantitative Results for Spectrum1
Analysis: Bulk Method: Standardless
Acquired 05-Oct-2005, 100.0 KeV @10 eV/channel
Element Weight % Std. Dev. MDL Atomic % k-Ratio Intensities FWHM (eV) ROI (net)
O 48.92 1.41 0.27 64.90 0.4869 13143.8 135.9 15384.93
Na 6.36 1.16 1.05 5.87 0.0436 1915.8 138.8 2871.67
Mg 2.19 0.70 1.84 1.91 0.0142 744.8 99.6 1046.32
Si 30.37 1.18 0.59 22.95 0.1848 12114.4 140.4 12099.89
Fe 6.57 1.09 0.18 2.50 0.0641 7750.9 159.6 7960.05
Cu 5.58 1.09 0.22 1.87 0.0507 5451.3 166.4 5417.49
Total 100.00

How small is asbestos?

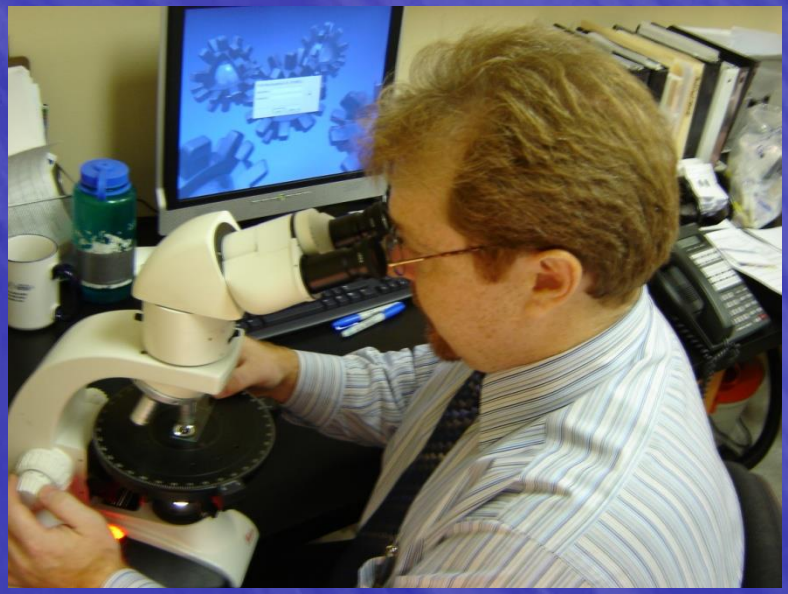
Stereomicroscope: 10-40x



How small is asbestos?

Polarized Light Microscope 400x

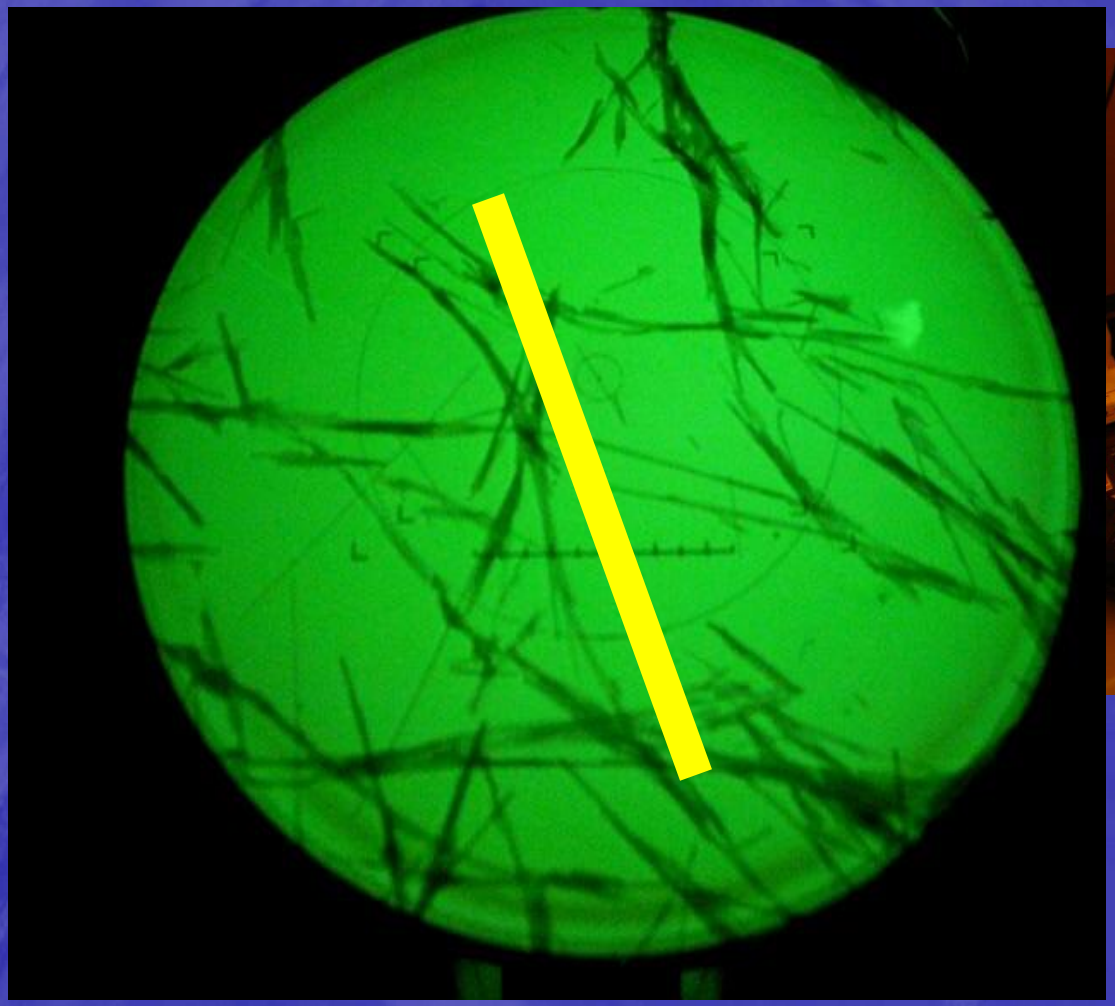
The large black fiber is human hair from the penny, approximately 40 microns thick.



The smaller chrysotile fiber bundle below it is actually **several fibers together.**

How small is asbestos?

In most asbestos fiber distributions, **less than one fiber out of 20** is large enough to see by Light Microscopes (PLM or PCM).



Transmission Electron Microscope 20,000x

The yellow bar is the smallest fiber size resolvable by light microscopes at 5µm long, and 0.25µm wide (PCMe).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ME

What is Talc?

A metamorphic sheet silicate mineral resulting from the metamorphism of magnesian minerals such as serpentine, pyroxene, amphiboles, olivine.

What is Asbestos?

The asbestiform varieties of sheet silicate chrysotile (serpentine), or double-chain silicates: crocidolite, amosite, anthophyllite, tremolite, and actinolite (amphiboles).

Talc Petrogenesis

- Type I, *ultramafic origin*: Begins as ultramafic rock, e.g., olivine or pyroxene that is serpentinized, and the resulting serpentine subsequently carbonitized by CO_2 and water to form talc and carbonates. Accessory minerals include magnesite, sulfides, chlorite, and **serpentine**.

- Type II, *mafic origin*: Begins as mafic rock, e.g., gabbro; serpentinized, and the resulting serpentine subsequently carbonitized by CO_2 and water to form talc and carbonates. Accessory minerals include magnesite, chlorite, parent minerals (e.g., gabbro) and **serpentine**.

- Type III, *metasedimentary origin*: begins as dolomite or magnesite, hydrothermally altered by silica-bearing fluids. Accessory minerals include dolomite, calcite, chlorite, quartz, and feldspars.

- Type IV, *metamorphic origin*: begins as carbonates, e.g., dolomite or silica -containing dolomitic marble. Recrystallization of host rock forms tremolite or actinolite within the host, which is subsequently steatized (altered to talc) by heat and pressure. Accessory minerals include carbonates, quartz, **serpentine**, **tremolite**, and **actinolite**; occasionally **anthophyllite** (McCarthy et.al., 2006).

ASBESTOS >

When fibrous

ASBESTOS >

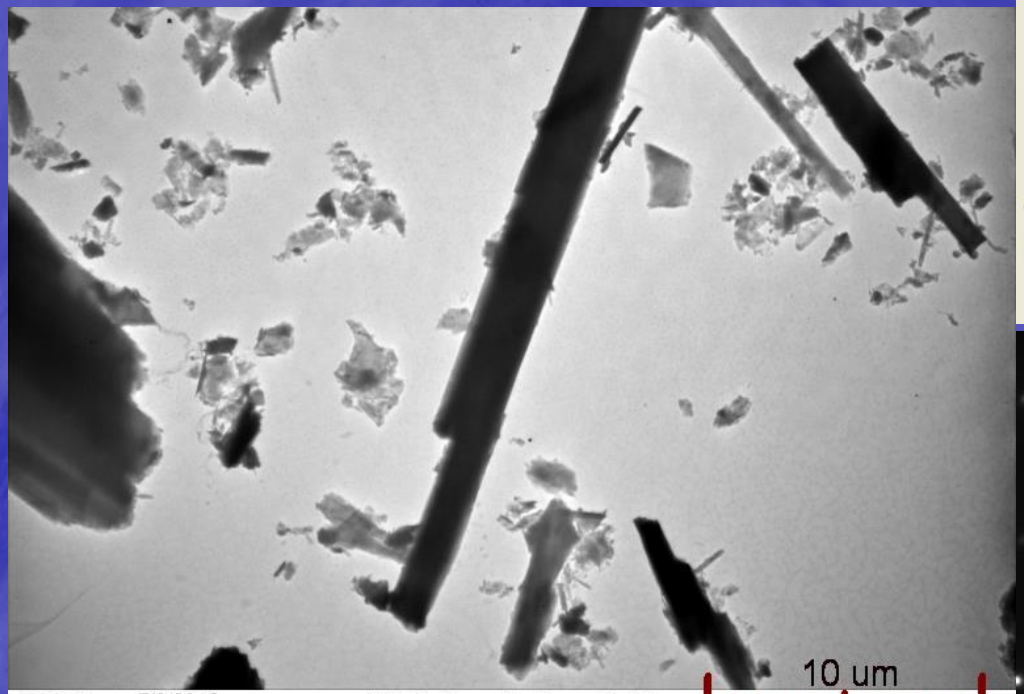
When fibrous

Table 1.4. Minerals commonly associated with talc

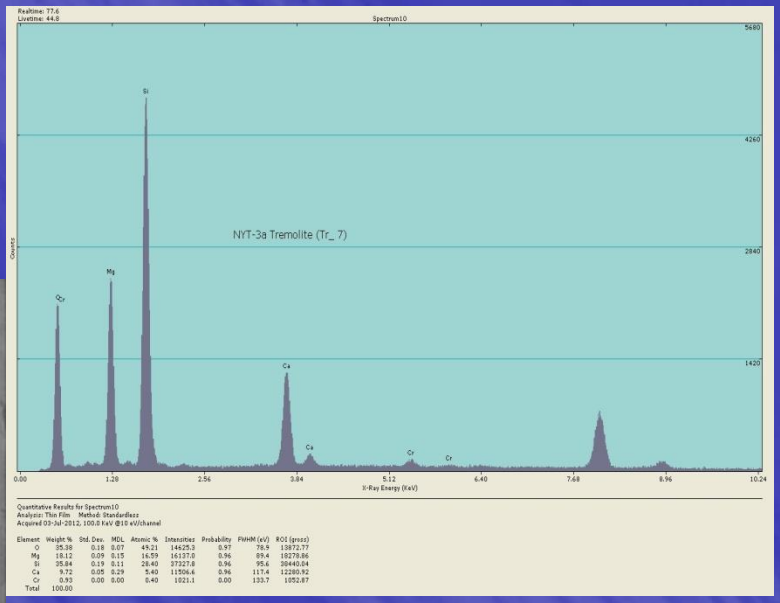
Mineral group	Name	Ideal formula
Carbonate	Dolomite	$(Ca,Mg)CO_3$
	Magnesite	$MgCO_3$
	Breunnerite	$(Mg,Fe)CO_3$
	Calcite	$CaCO_3$
	Siderite	$FeCO_3$
	Ankerite	$Ca(Fe,Mg,Mn)(CO_3)_2$
Phyllosilicates	Chlorite	$(Mg,Al,Fe)_{12}(Si,Al)_8O_{20}(OH)_{16}$
	Serpentine (lizardite and antigorite)	$Mg_3Si_2O_5(OH)_4$
	Phlogopite (mica)	$K_2(Mg,Fe)_6Si_6Al_2O_{20}(OH)_4$
	Sepiolite	$Mg_8Si_{12}O_{30}(OH)_4(H_2O)_4$
Amphibole[a]	Tremolite	$Ca_2Mg_5Si_8O_{22}(OH)_2$
	Anthophyllite	$(Mg,Fe)_7\ Si_8O_{22}(OH)_2$
	Actinolite	$Ca_2(Mg,Fe)_5\ Si_8O_{22}(OH)_2$
Tectosilicates	Quartz	SiO_2
	Feldspar	$(K,Na)AlSi_3O_8$
Oxides	Magnetite	Fe_3O_4
	Ilmenite	$FeTi\ O_3$
	Manganese oxide	MnO_2
	Rutile	$Ti\ O_2$
Sufides	Pyrite	FeS_2
	Pyrrhotite	FeS
	Pentlandite	$(Fe,Ni)_9S_8$
Other minerals	Tourmaline[b]	$NaFe_3Al_6(BO_3)_3Si_6O_{18}(OH)_3(OH)$
	Graphite	C

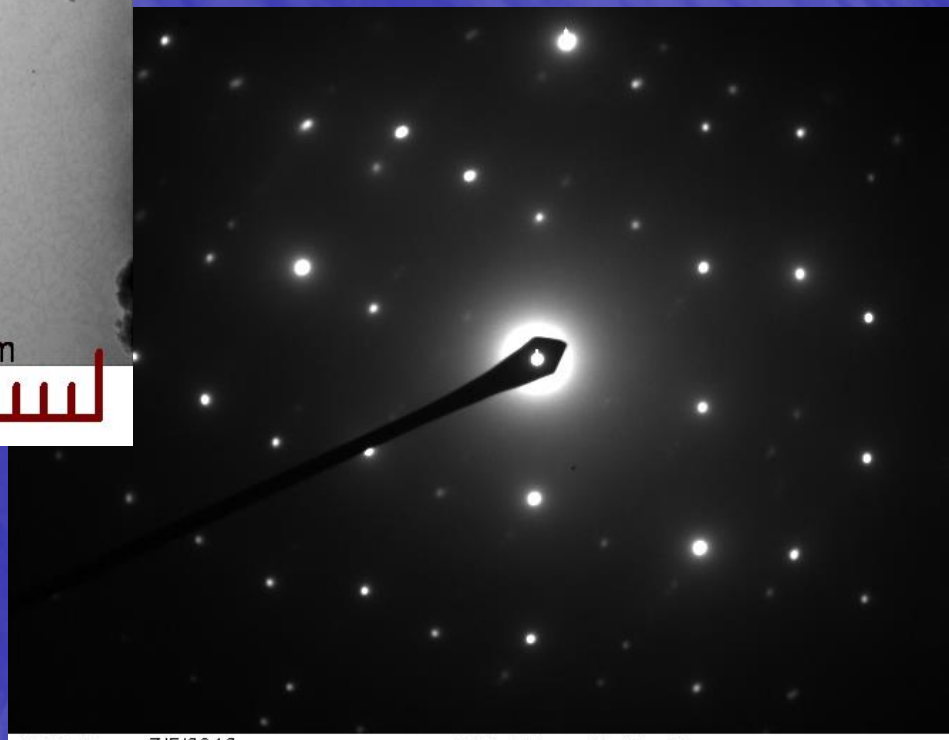
Compiled by the Working Group from Pooley & Rowlands (1975); Piniazkiewicz *et al.* (1994); Harben & Kuzvart (1996)

Tremolite



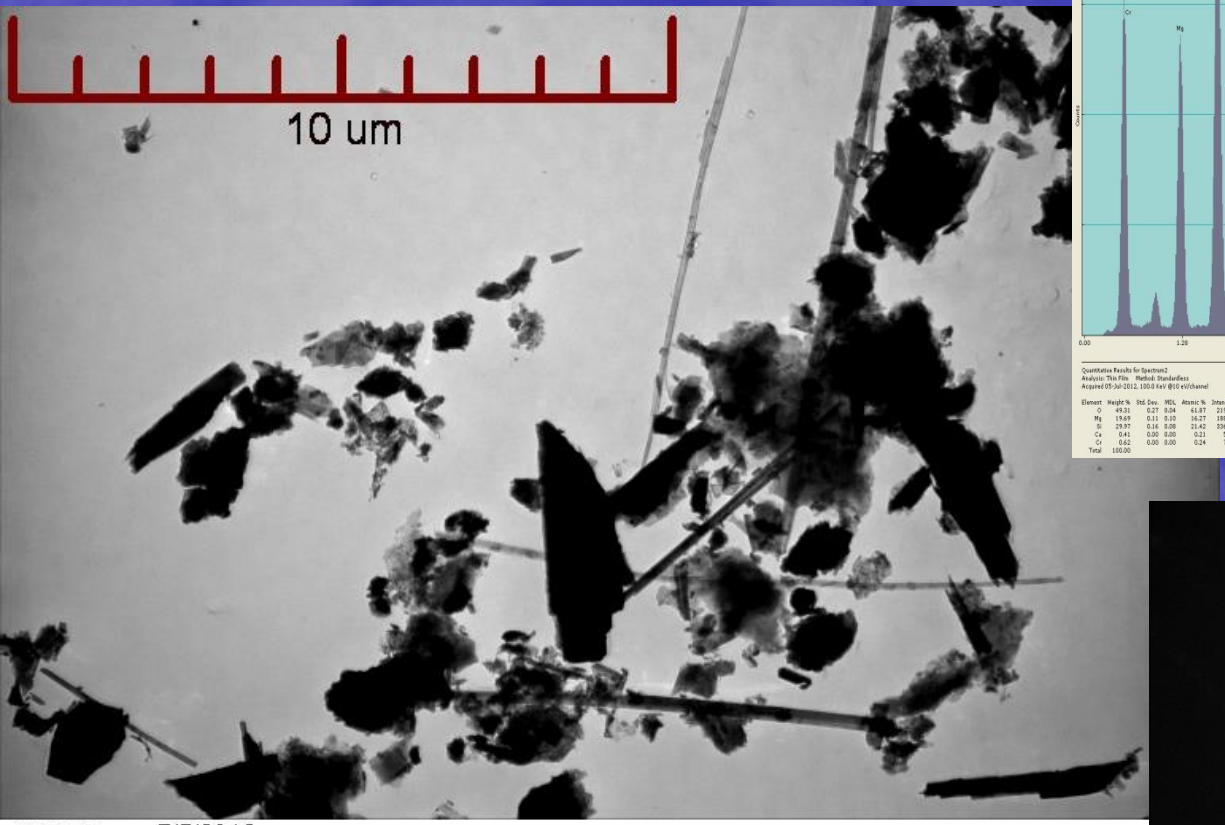
1200EX 7/3/2012 NYT-3a Tremolite Fibers (Tr_7)
HT: 100 kV Exposure Time: 3.00 Sec
TEM Magnification: 2KX





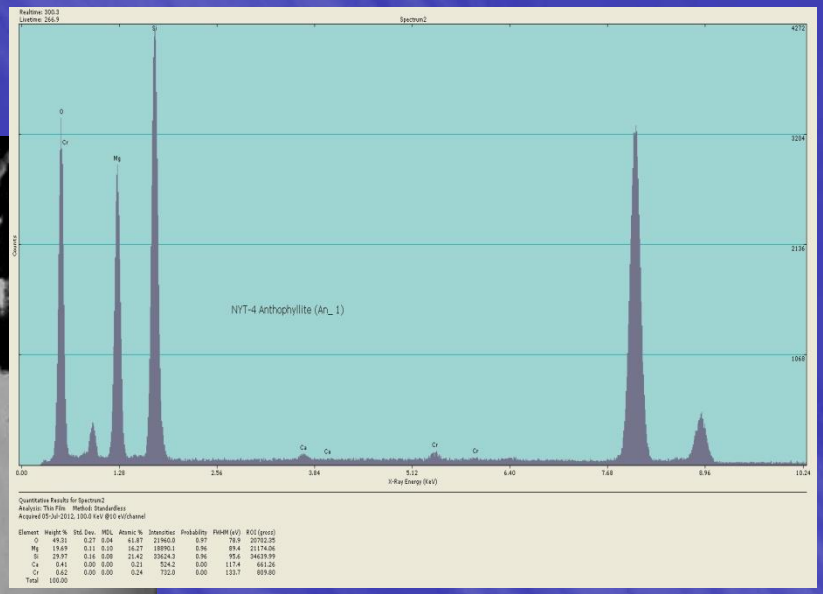
1200EX 7/5/2012 NYT-4: Tremolite (Tr_4)
HT: 100 kV Exposure Time: 9.00 Sec
TEM Magnification: KX

Anthophyllite



1200EX 7/5/2012
HT: 100 kV Exposure Time: 2.00 Sec
TEM Magnification: KX

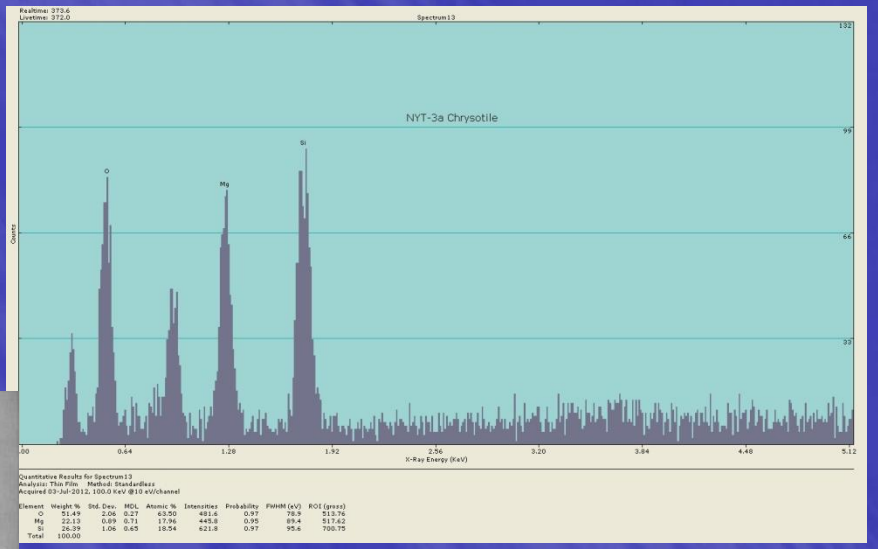
NYT-4: Anthophyllite asbestos fibers in NYTAL



1200EX 7/3/2012
HT: 100 kV Exposure Time: 9.00 Sec
TEM Camera Length: 80cm

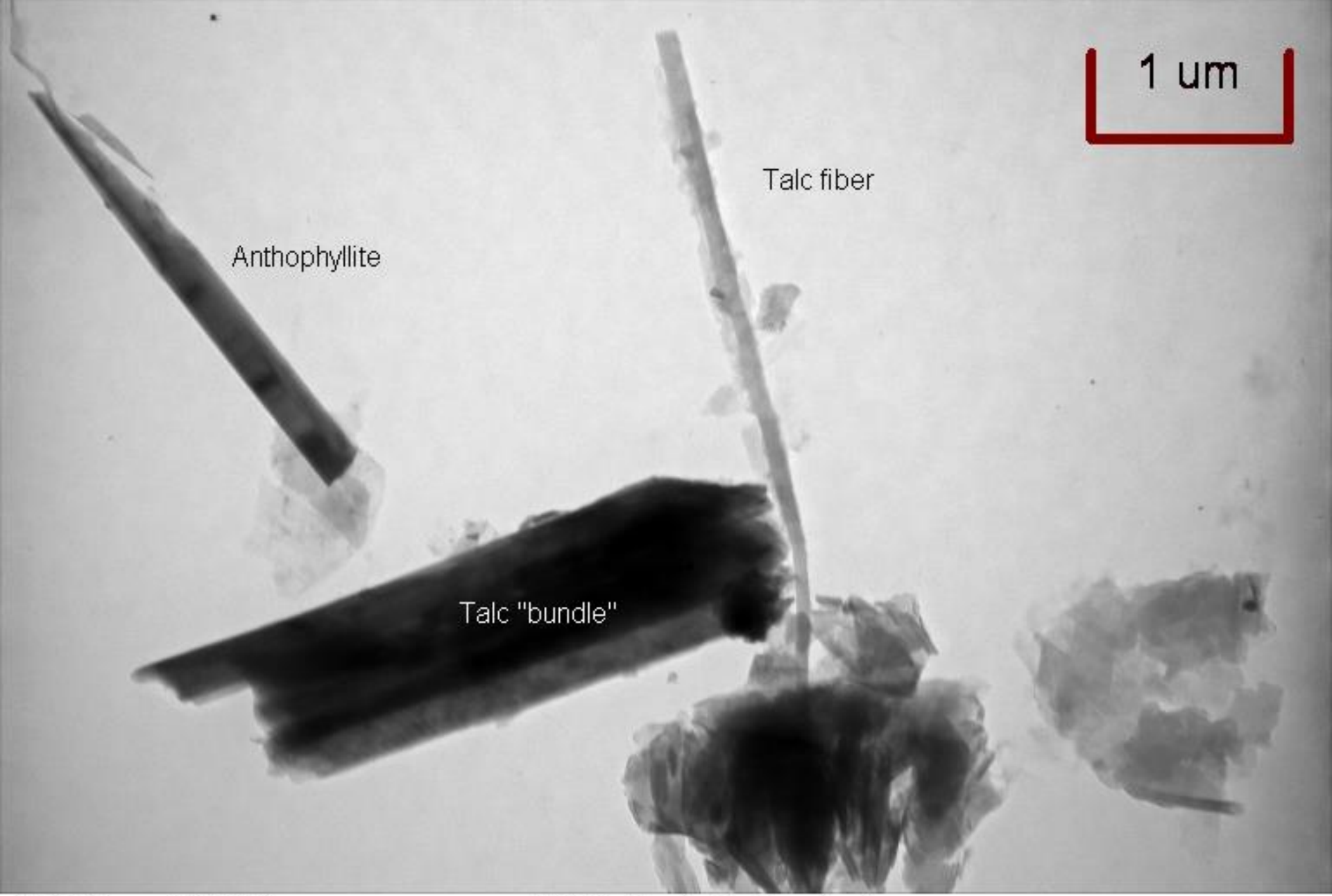
NYT-3a: An_2

Chrysotile


NYT-3a Chrysotile
O
Mg
Si
Counts
X-Ray Energy (KeV)


1200EX 7/3/2012
HT: 100 kV Exposure Time: 8.00 Sec
TEM Magnification: 30KX
NYT-3a: Chrysotile
1 um


1200EX 7/3/2012
HT: 100 kV Exposure Time: 10.00 Sec
Camera Length: 80cm
NYT-3a: Chrysotile



1200EX 7/5/2012
HT: 100 kV Exposure Time: 2.00 Sec
TEM Magnification: KX 12

NIST Anthophyllite Standard

1867a: Uncommon: SUPPLEMENTAL INFORMATION

Table 5. Descriptive information from observations with low-power microscopy and polarized light microscopy and approximate concentrations of accessory phases determined by XRD analysis are provided for information purposes only in Table 5.

Anthophyllite

Texture: asbestiform[a]
Color: tan in hand specimen, colorless in plane polarized light
Concentration of anthophyllite: > 80 % mass fraction Talc is estimated to be present at concentrations of approximately 5 % mf to 15 % mf.

Tremolite

Texture: asbestiform[a] Some of the fibers are loose and others are more tightly bound together. A small amount of material may be massive.
Color: white to pale green in hand specimen, colorless in plane polarized light
Concentration of tremolite: > 90 % mass fraction Talc is estimated to be present at concentrations less than 5 % mf.

Actinolite

Texture: asbestiform[a] Some of the fibers are loose and others are more tightly bound together. A considerable amount of material may be massive. The massive material may contain significant clinochlore (chlorite).
Color: white to green in hand specimen, colorless in plane polarized light
Concentration of actinolite: > 80 % mass fraction of the fibrous material Clinochlore (chlorite) is estimated to be present at concentrations that can exceed 20 % mf if a considerable amount of massive material is present. The massive material can be easily segregated, leaving primarily actinolite in the fibrous portion. A small amount (< 5 % mass fraction) of talc may also be present in the fibrous component.

[a] Asbestiform: crystallizes with the habit of asbestos. These asbestos minerals possess properties such as long fiber length and high tensile strength. Under the light microscope, **some portion** of these samples exhibit the asbestiform habit as defined by several of the following characteristics: 1) mean aspect ratios ranging from 20:1 to 100:1 or higher for fibers longer than 5 µm, 2) very thin fibrils, usually less than 0.5 µm in width, 3) parallel fibers occurring in bundles, 4) fiber bundles displaying splayed ends, 5) fibers in the form of thin needles, 6) matted masses of individual fibers, and 7) fibers showing curvature.

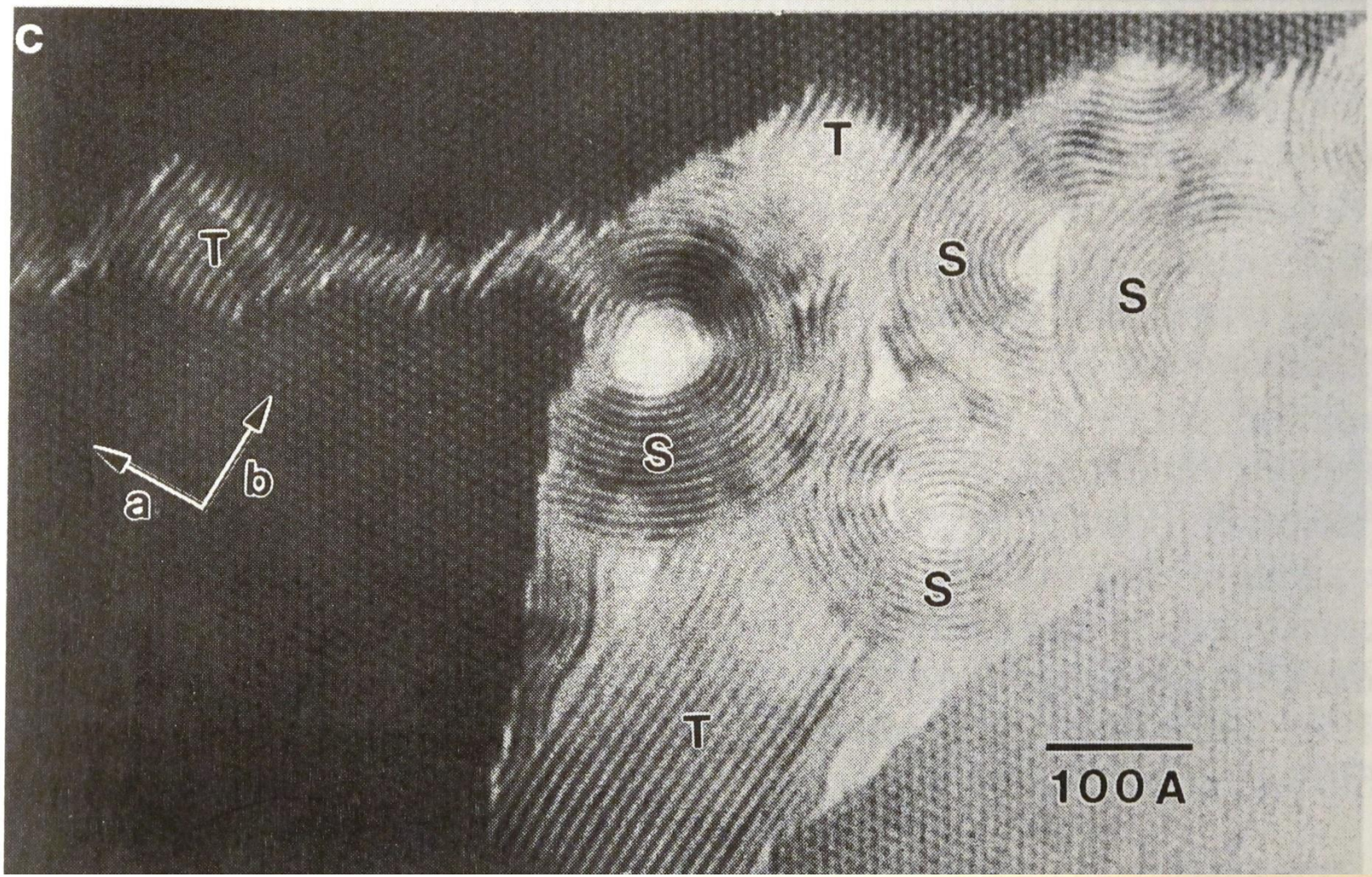


Figure 25. Interfibril grain boundaries in anthophyllite asbestos from a small, abondoned mine at Pelham, Massachusetts (Veblen, 1980). (a) Two adjacent fibrils showing amphibole-amphibole contact; where the boundary changes orientation at the upper left, it assumes a wedgelike, wide-chain pyribole structure. (b) Three adjacent fibrils with the layer silicate talc (T) in their grain boundaries. (c) A very low-angle grain boundary (~1° rotation between the fibrils). The amphibole at the top is one fibril, and the patches in the lower left and lower right both belong to the other fibril. The boundary contains talc (T) and serpentine mostly of the chrysotile persuasion (S), though a few planar serpentine (lizardite) layers are also present (distinguished from talc by smaller spacing between layers).

Mineralogy of Amphiboles and 1:1 Layer Silicates, Veblen and Wylie, 1993

"Because these grain boundaries contain layer silicates, it is likely that the amphibole asbestos surface with which a biologic system will interact in many cases may not actually be amphibole.

The likelihood of layer silicate interfaces varies, depending on the sample, and contact with a number of different layer silicates is possible.

Because of the potential biologic ramifications of the interfibril layer silicates in amphibole asbestos, their analysis with state-of-the-art AEM methods is much needed."

SAI Talc Testing from 2015 - 2019

- From 2015 to 2020, we tested over 600 cosmetic talc products for asbestos, and over 100 were found to contain asbestos fibers.

Brands found to contain asbestos fibers have included…

- Avon
- Claire's
- Johnson & Johnson
- Mennen
- Cashmere Bouquet
- Colgate for Men
- Jean Nate
- Clubman
- Old Spice
- Chanel
- Justice
- Coty
- Dr. Scholl's
- Woods of Windsor

Proposed Method for the Determination of Asbestos Fiber Structures Consistent with Airborne Counting Criteria in Mineral Powders, e.g., Talc; Quantified in Structures per Gram by Transmission and/or Scanning Electron Microscopy

by:

S. Fitzgerald, PG

Scientific Analytical Institute

September 24, 2018

ISO TC 146, SC3, WG1

Sydney, Australia

Groundhog Day Round Robin

- Over the past decade (+), we have repeatedly been able to determine that talc and/or talc-based products can contain fiber-structures of tremolite, anthophyllite, and/or chrysotile asbestos using TEM alone

- Current methods for the determination of asbestos in talc are inadequate, resulting in products in the open market that contain releasable asbestos fibers, manufactured with talc certified as “asbestos-free” (USP)

- Testing by XRD and/or PLM cannot reliably detect the asbestos structures found in these products by TEM & SEM

Groundhog Day Round Robin

- The preparation technique developed is capable of resolving these low levels of asbestos in talc & talc-based products, and the tools & definitions employed were derived from current ASTM & ISO protocols
- The preparation and analysis used is quite similar to the current ASTM WK30039 Transmission Electron Microscopy (TEM) Analysis of Mineral Powders for Asbestos, & ISO 222262-2
- Final analyses can detect and quantify asbestos by TEM or SEM
- This method can and should be a stand-alone method (XRD and/or PLM should not be required)

Groundhog Day Round Robin

- As it is clear that we need such a method, a sample exchange (GRR) was designed to give us insight as to the quantitative and qualitative repeatability
- Seven samples were chosen to prepare duplicates of talc-based products previously-determined to contain countable asbestos structures
- 10 laboratories agreed to participate by analyzing TEM grids prepared of these duplicates, using ISO 10312 counting protocol
- No participants were aware of what product or products were prepared for this exchange; only that we were looking for countable structures in talc.

GRR Sample A:

		str/mm2							
Sample	Lab	tre<	tre>	Chy<	Chy>	Anth <	Anth >	Total	str/ filter
A	1	3.9	15.6	1.9				21.5	28,380
A	2	28	32				12	72	95,040
A	3								0
A	4	5.2	1.2					6.4	8,448
A	5	41.25	15	3.75				60	79,200
A	6	60	20	44	4			128	168,960
A	7	30.8	11.55	23.1	3.85			69.3	91,476
A	8								0
A	9	5	35					40	52,800
A	10								0
A	Ave:	24.87857	18.62143	18.1875	3.925		12	56.74286	
							STDEV:	36.90807	
							CV:	0.650444	

GRR Sample B:

		str/mm2							
Sample	Lab	tre<	tre>	Chy<	Chy>	Anth <	Anth >	Total	str/ filter
B	1	0.99	6.9					7.9	10,428
B	2	24	68		8			100	132,000
B	3								0
B	4	6	3.2					9.2	12,144
B	5	24	48		4			76	100,320
B	6	52	12	32				96	126,720
B	7	19.25	11.55	7.7				38.5	50,820
B	8								0
B	9	4	32					36	47,520
B	10								0
B	Ave:	18.60571	25.95	19.85	6			51.94286	
							STDEV:	35.91831	
							CV:	0.691497	

GRR Sample C:

		str/mm2							
Sample	Lab	tre<	tre>	Chy<	Chy>	Anth <	Anth >	Total	str/ filter
C	1	0.98	4.9					5.6	7,392
C	2	28	36					64	84,480
C	3								0
C	4	4.4	3.2					7.6	10,032
C	5	12	20		4			36	47,520
C	6	20	20	8				48	63,360
C	7	7.69	19.23	15.38				42.3	55,836
C	8								0
C	9		12					12	15,840
C	10								0
C	Ave:	12.17833	16.47571	11.69	4			30.78571	
							STDEV:	20.99009	
							CV:	0.681813	

GRR Sample D:

		str/mm2							
Sample	Lab	tre<	tre>	Chy<	Chy>	Anth <	Anth >	Total	str/ filter
D	1		12					12	15,840
D	2	14	32	2		2		50	66,000
D	3								0
D	4	3.2	2					5.2	6,864
D	5	44	36		8			88	116,160
D	6	40	32	32	4			104	137,280
D	7	26.95	3.85	11.55	3.85			46.2	60,984
D	8								0
D	9	4	8					12	15,840
D	10								0
D	Ave:	22.025	17.97857	15.18333	5.283333	2		45.34286	
							STDEV:	36.09048	
							CV:	0.795946	

GRR Sample E:

		str/mm2							
Sample	Lab	tre<	tre>	Chy<	Chy>	Anth <	Anth >	**Total**	str/ filter
E	1		15.3					15.3	20,196
E	2	12.78	27.39				1.83	42	55,440
E	3								0
E	4	2.4	4					6.4	8,448
E	5	20	28					48	63,360
E	6	24	16					40	52,800
E	7	7.69	30.76	3.85				42.3	55,836
E	8								0
E	9	4	20					24	31,680
E	10								0
E	Ave:	11.81167	20.20714	3.85			1.83	31.14286	
							STDEV:	14.73188	
							CV:	0.473042	

GRR Sample F:

		str/mm2							
Sample	Lab	tre<	tre>	Chy<	Chy>	Anth <	Anth >	Total	str/ filter
F	1	1.9						1.9	2,508
F	2	1.52	1.9	0.38				3.8	5,016
F	3								0
F	4	0.4	0.1					0.5	660
F	5	3.9	2.6					6.5	8,580
F	6								0
F	7			3.85	3.85			7.7	10,164
F	8								0
F	9								0
F	10								0
F	Ave:	1.93	1.533333	2.115	3.85			4.08	
							STDEV:	2.705846	
							CV:	0.663197	

GRR Sample G:

		str/mm2							
Sample	Lab	tre<	tre>	Chy<	Chy>	Anth <	Anth >	Total	str/ filter
G	1		1.1					1.1	1,452
G	2	3.375	0.75			0.375		4.5	5,940
G	3								0
G	4	0.37	0.22					0.56	739
G	5	9.4	4.7					14.1	18,612
G	6								0
G	7		3.8					3.8	5,016
G	8								0
G	9				4			4	5,280
G	10								0
G	Ave:	4.381667	2.114	#DIV/0!	4	0.375		4.676667	
							STDEV:	4.468339	
							CV:	0.955454	

GRR Sample A

Justice

Just Shine Shimmer Powder

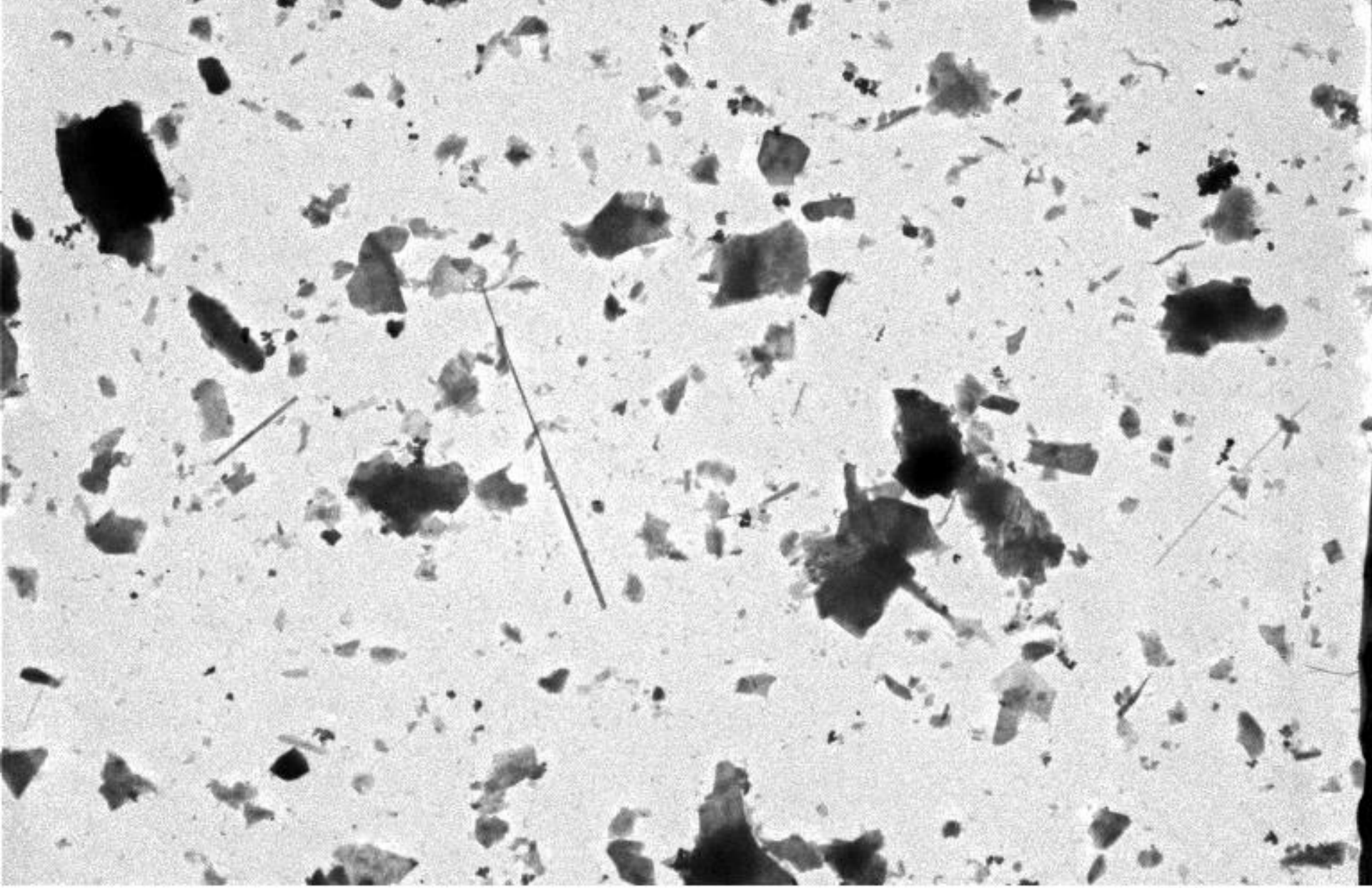
Purchased May 4, 2017

In Raleigh, NC

Original Concentration:

106 Million Tremolite Str/gram

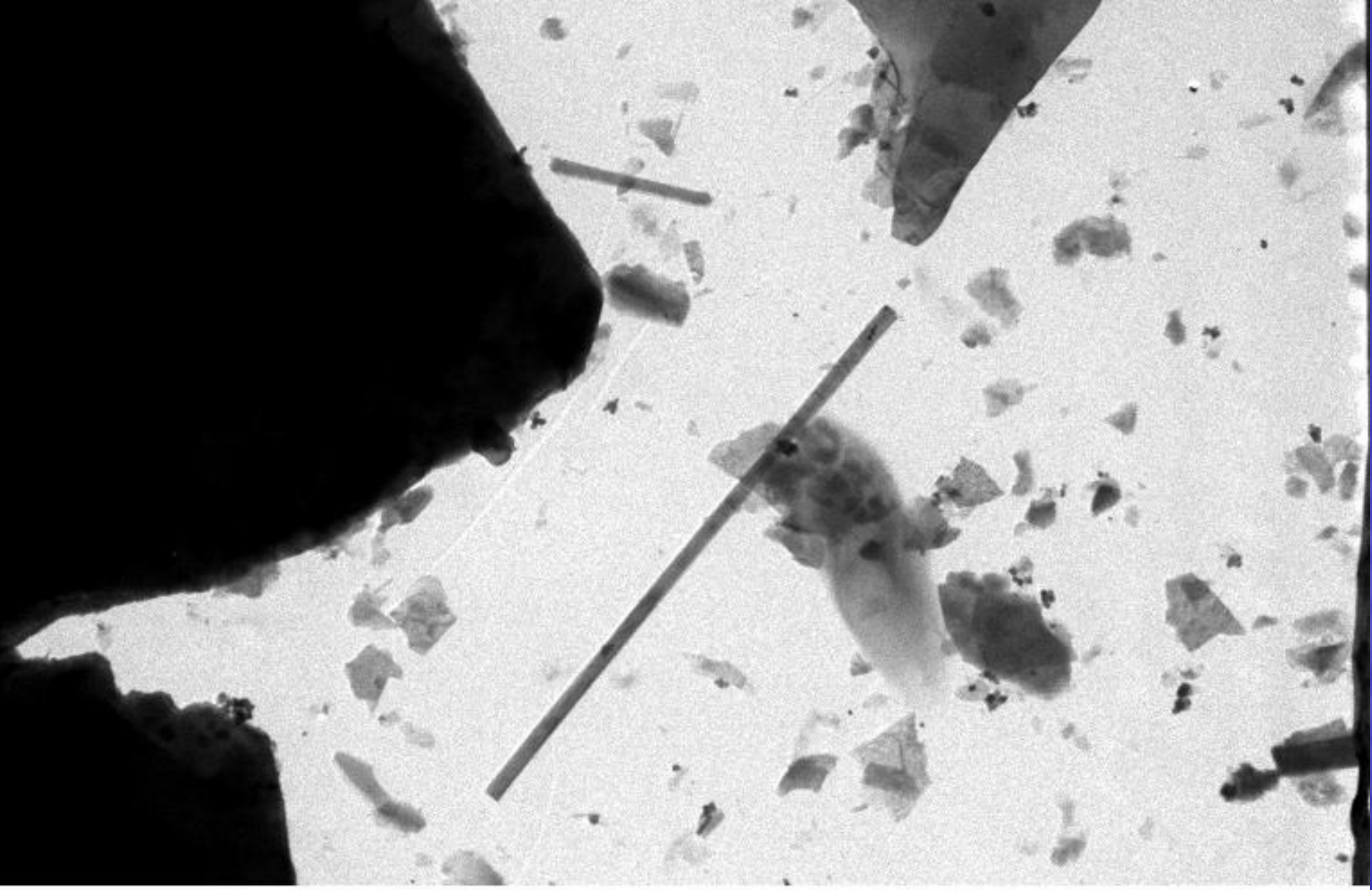
GRR Ave: ***234 Million Asbestos Str/gram***

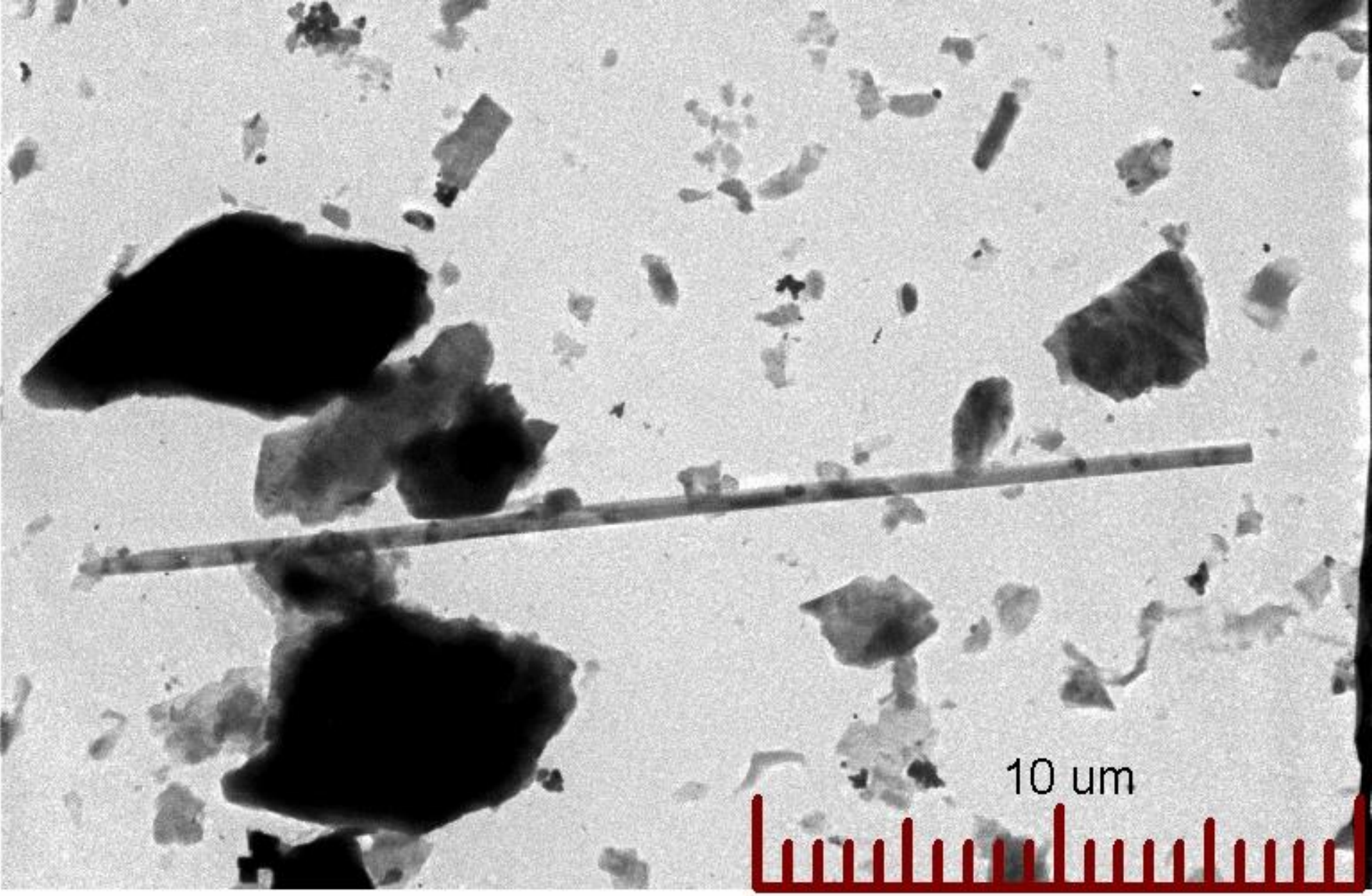


5/17/2017
HT: 100KV
TEM Magnification: 1,500x

Tremolite Asbestos in Children's Make-up

10 um


5/17/2017
HT: 100KV
TEM Magnification: 2,500x
Exposure Time: 2.00 Sec
10 um

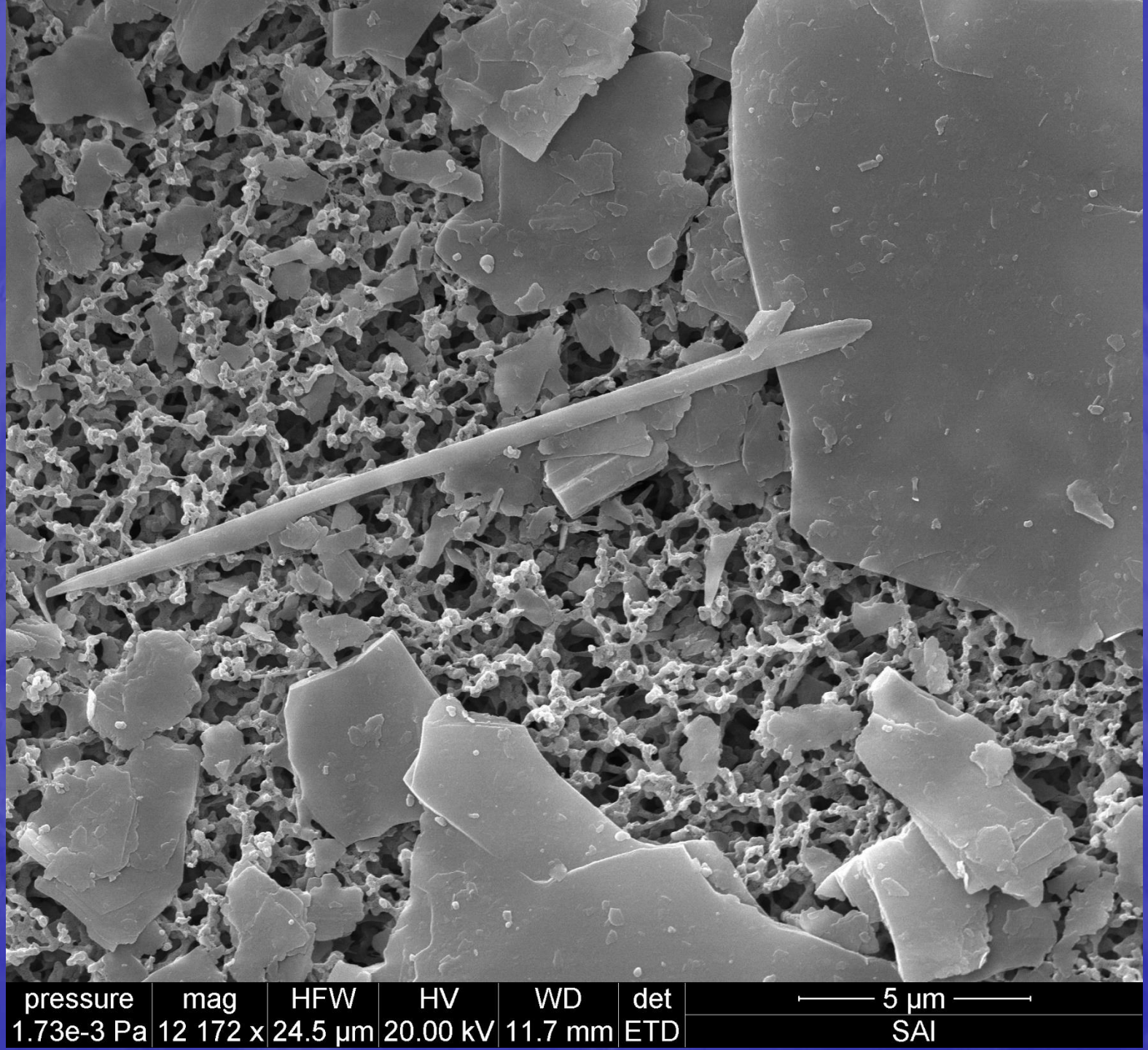


5/17/2017

HT: 100KV

TEM Magnification: 3,000x

Tremolite Asbestos in Children's Make-up


pressure
1.73e-3 Pa
mag
12 172 x
HFW
24.5 µm
HV
20.00 kV
WD
11.7 mm
det
ETD
5 µm
SAI

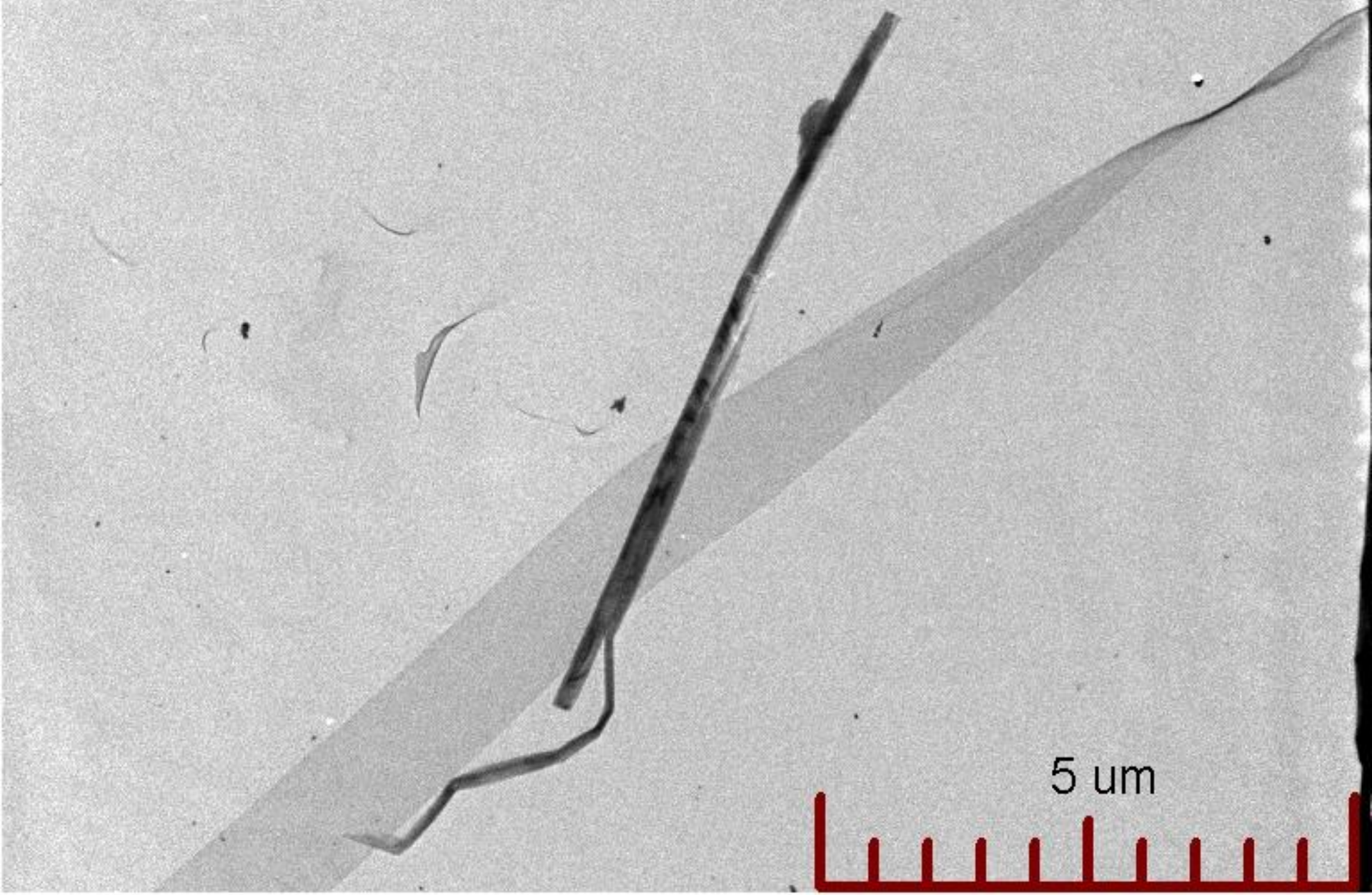
GRR Sample C

Claire's Black shiny Makeup Kit

Light tan Compact powder

Purchased December 02, 2017 In Los Angeles

GRR Ave: 158 Million Asbestos Str/gram

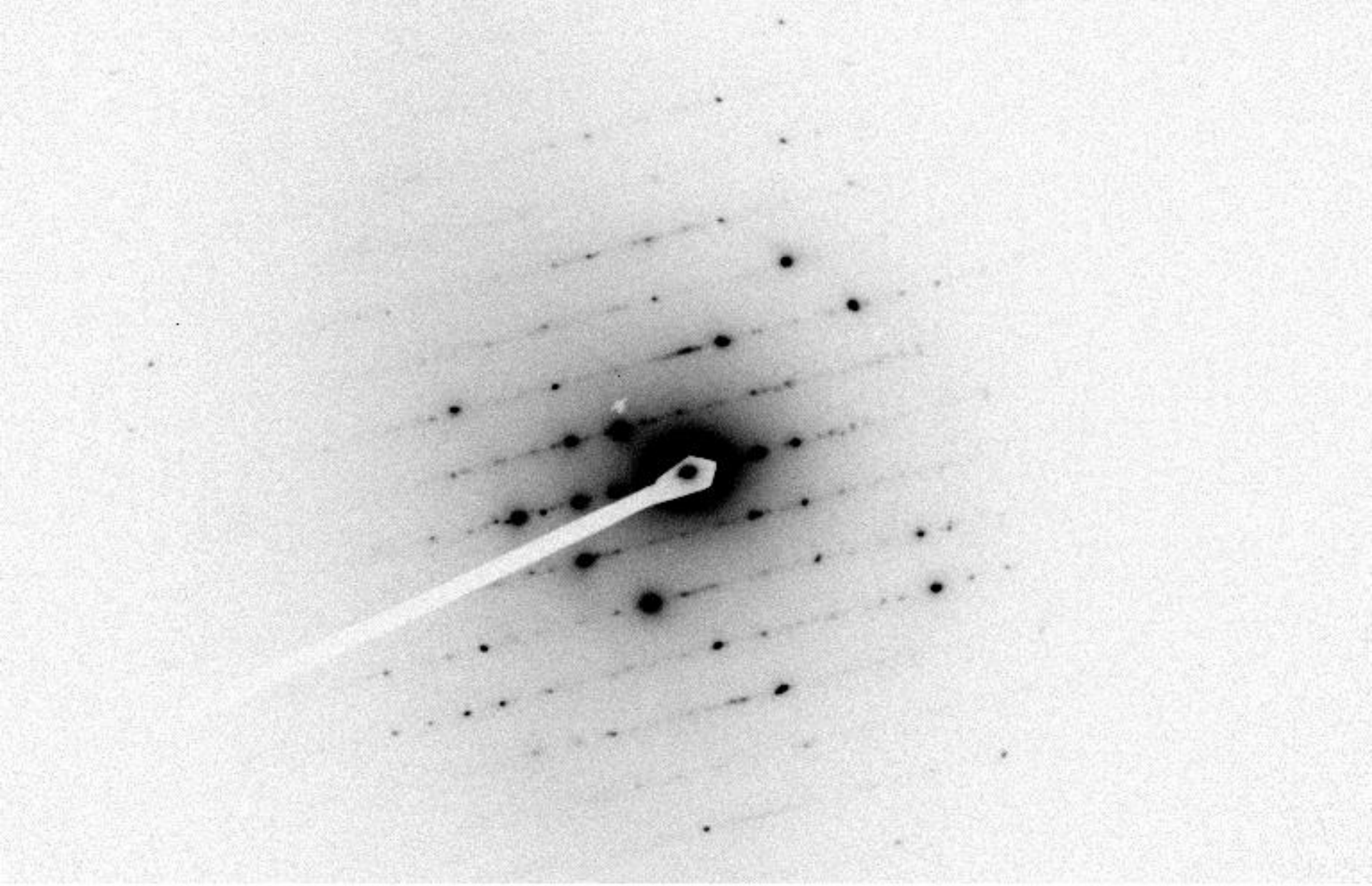


12/14/2017

HT: 100KV

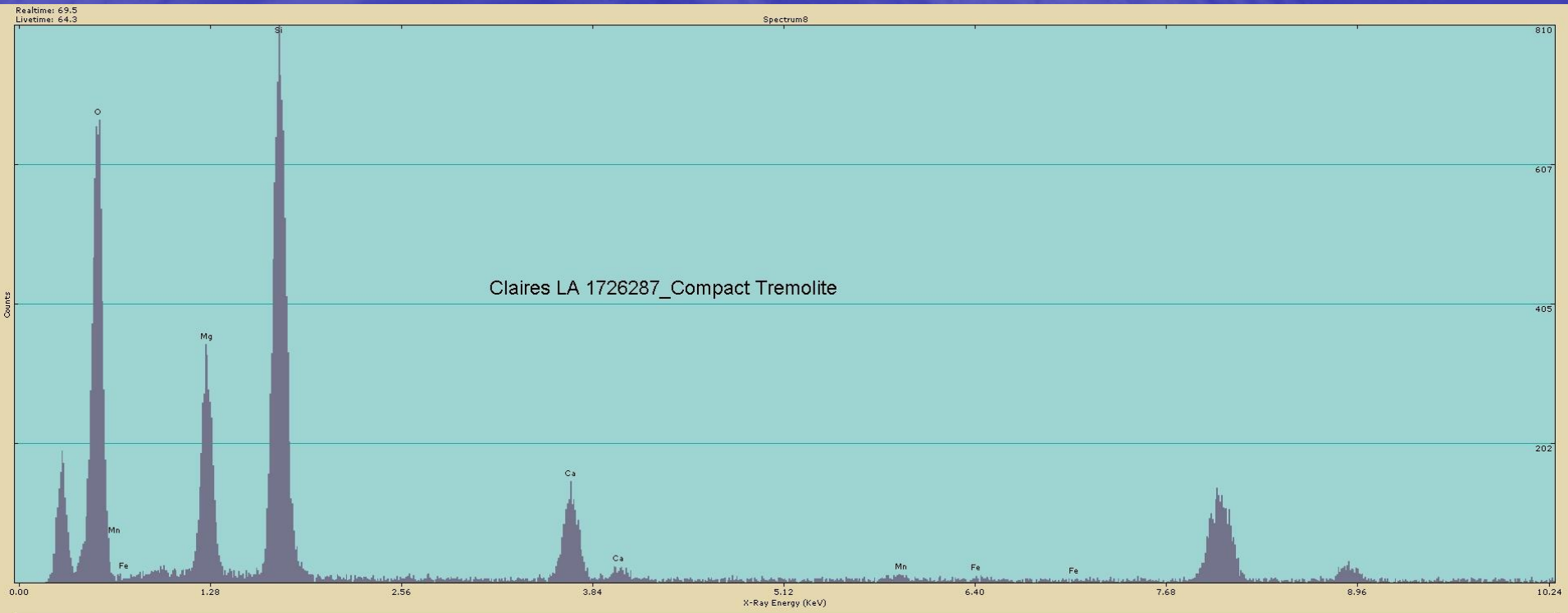
TEM Magnification: 6,000x

Claires LA 1726287_Compact Tremolite



12/14/2017
HT: 100KV
TEM Magnification: 50cm

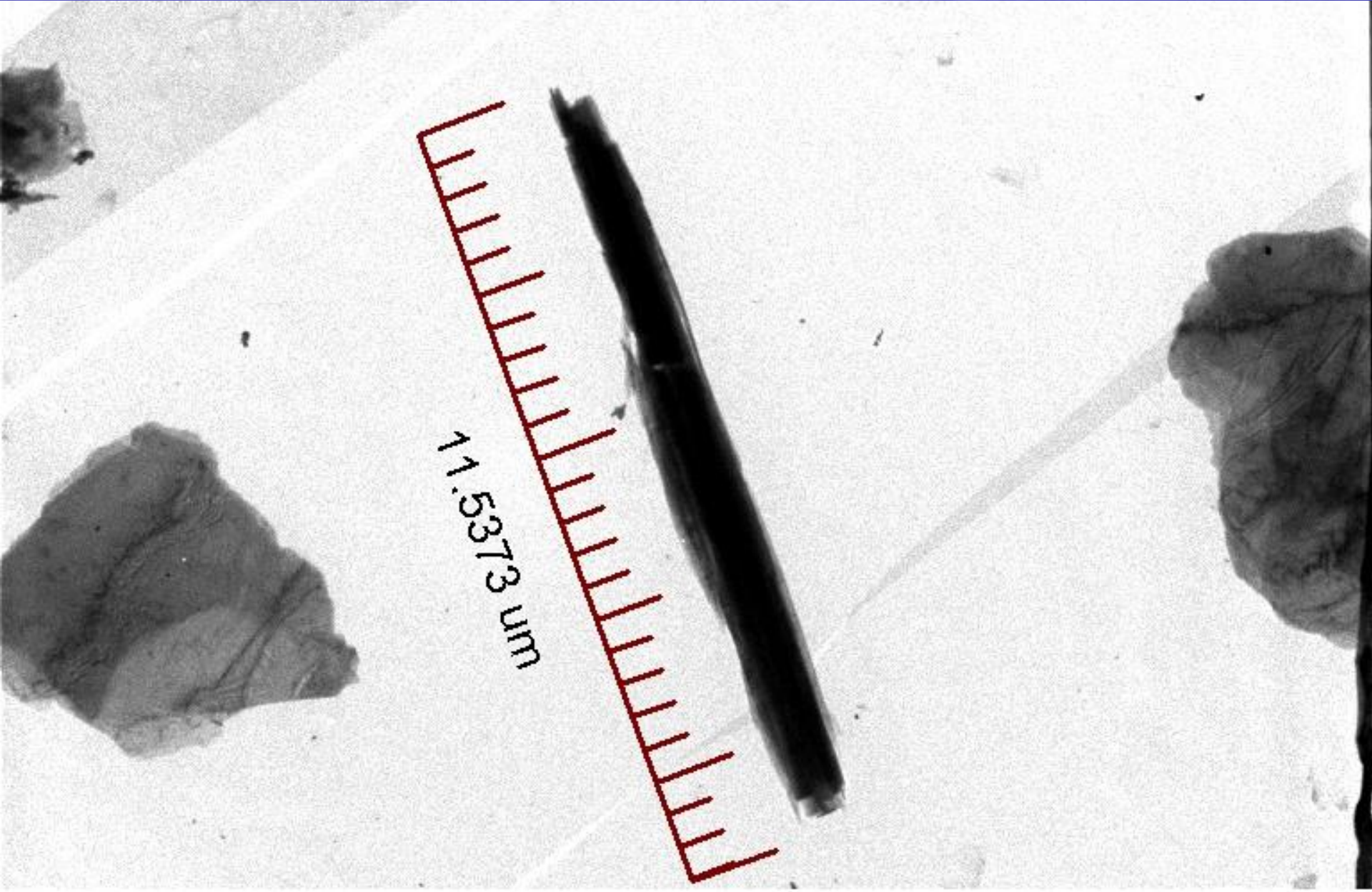
Claires LA 1726287_Compact Tremolite



Quantitative Results for Spectrum8
Analysis: Bulk Method: Standardless
Acquired 14-Dec-2017, 30.0 KeV @10 eV/channel

Element	Weight %	Std. Dev.	MDL	Atomic %	Oxide %		k-Ratio	Intensities	Probability	FWHM (eV)
O	49.99	1.92	0.28	63.34	0.00		0.0000	0.0	0.00	77.7
Mg	14.78	0.91	0.81	12.32	24.51	(MgO)	0.0647	2104.4	0.96	88.2
Si	30.49	1.23	0.41	22.01	65.23	(SiO2)	0.1675	6417.7	0.97	94.5
Ca	4.24	1.01	0.50	2.15	9.33	(CaO3)	0.0353	1206.8	0.97	116.5
Mn ?	0.41	0.17	2.09	0.15	0.82	(Mn2O7)	0.0035	74.0	0.00	137.0
Fe ?	0.09	0.05	4.15	0.03	0.12	(FeO)	0.0008	16.8	0.00	141.9
Total	100.00				100.00					

? These elements are statistically insignificant.

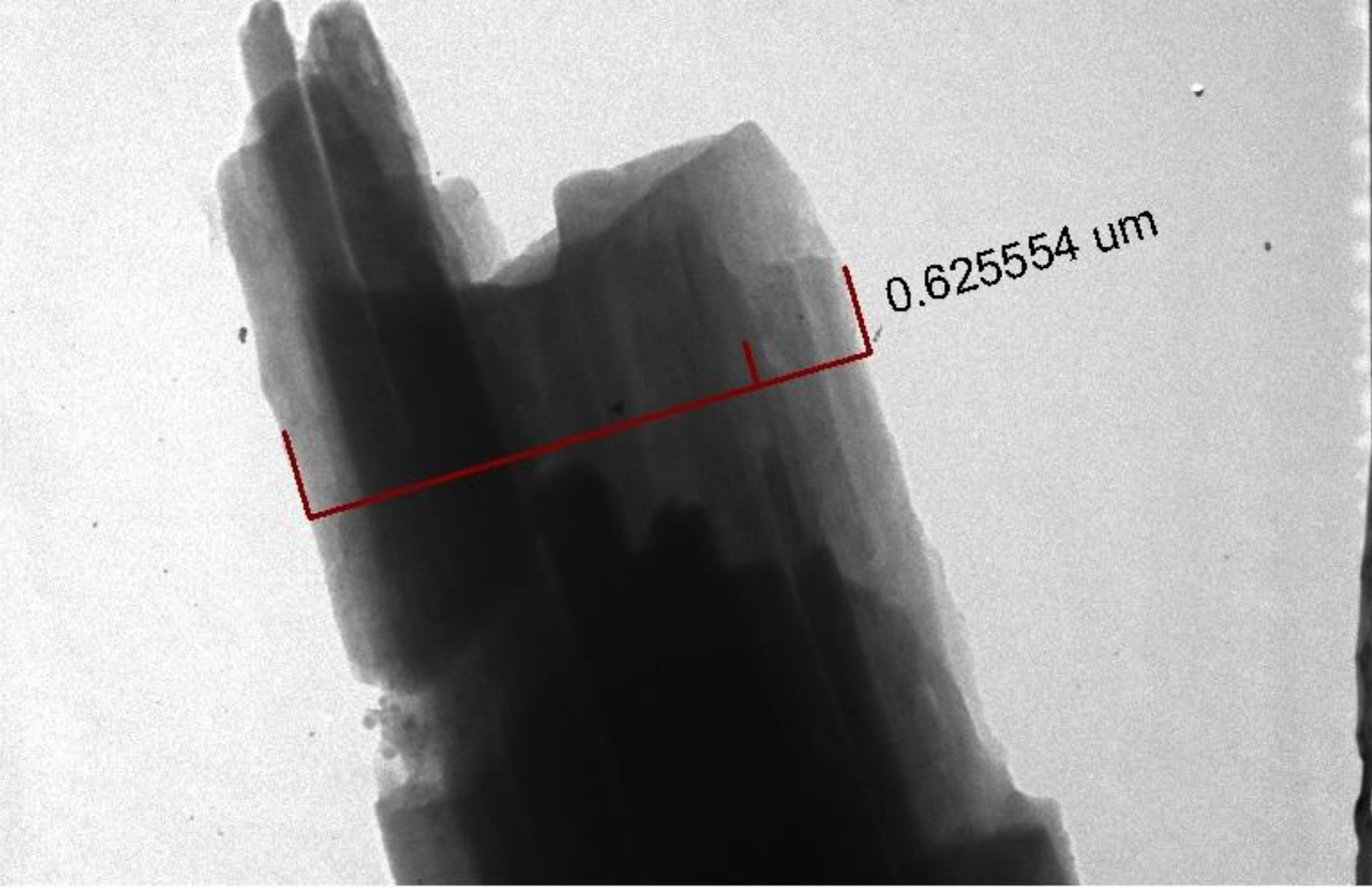


12/14/2017

HT: 100KV

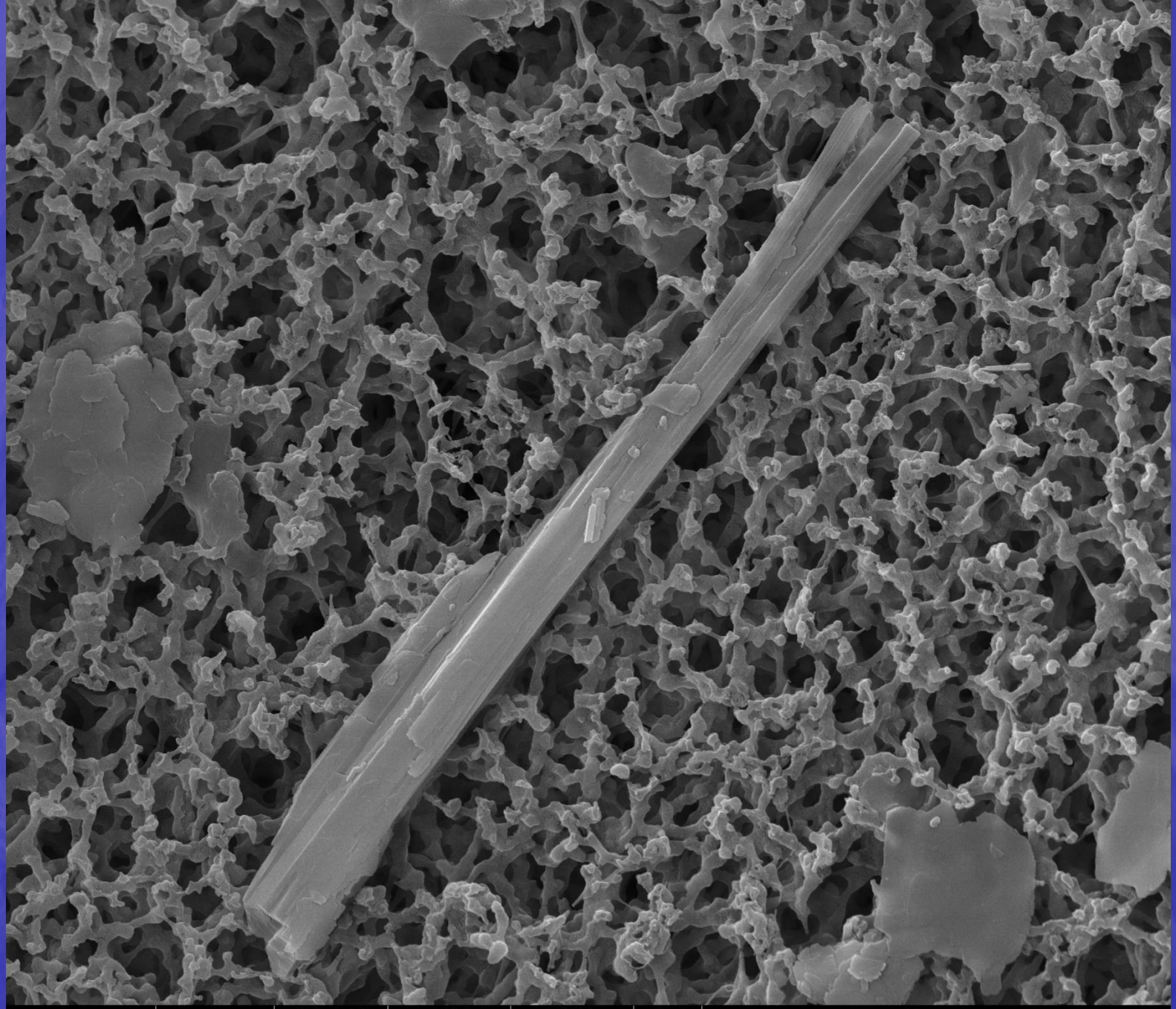
TEM Magnification: 4,000x

Claires LA 1726287_Compact Tremolite

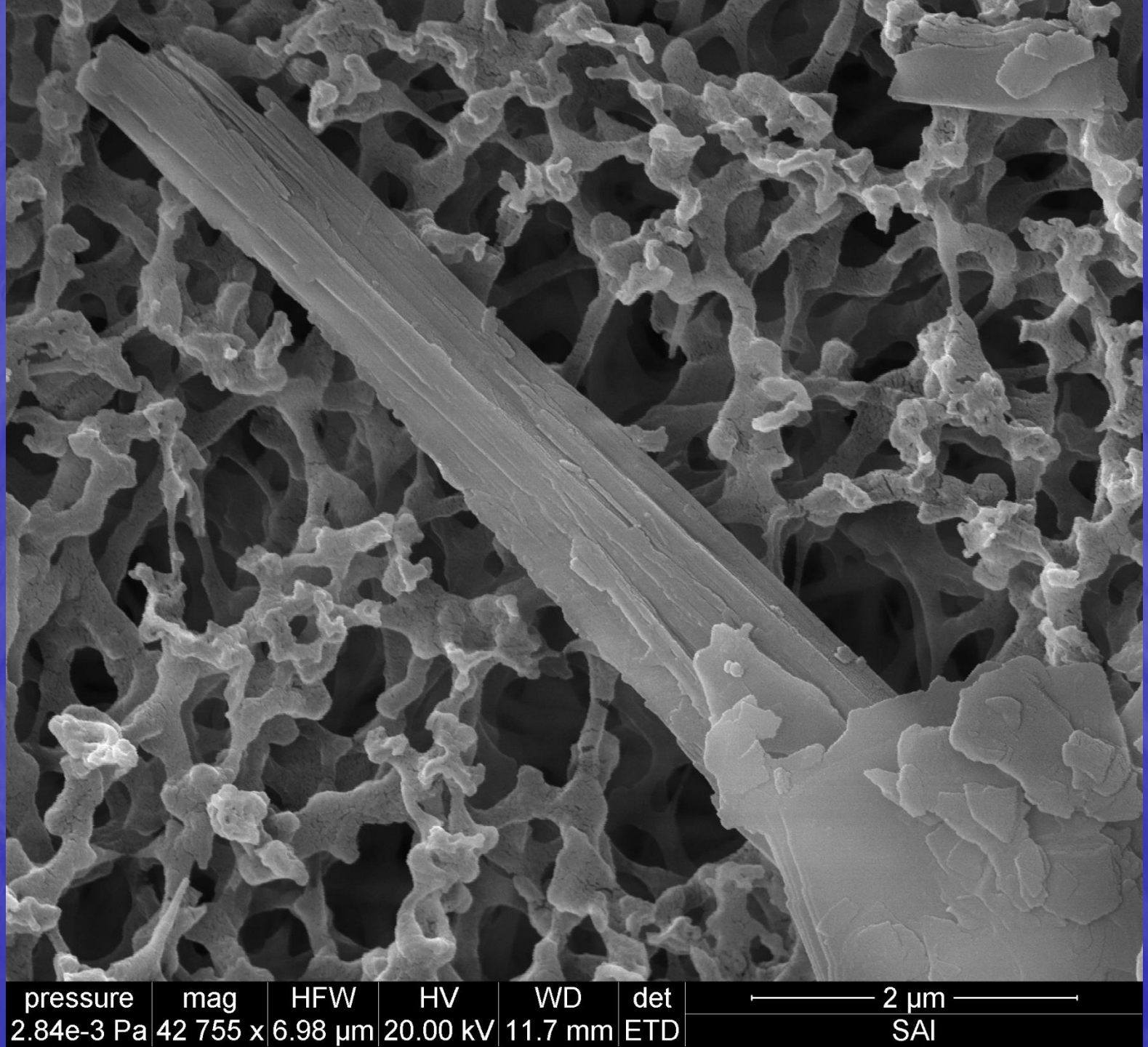


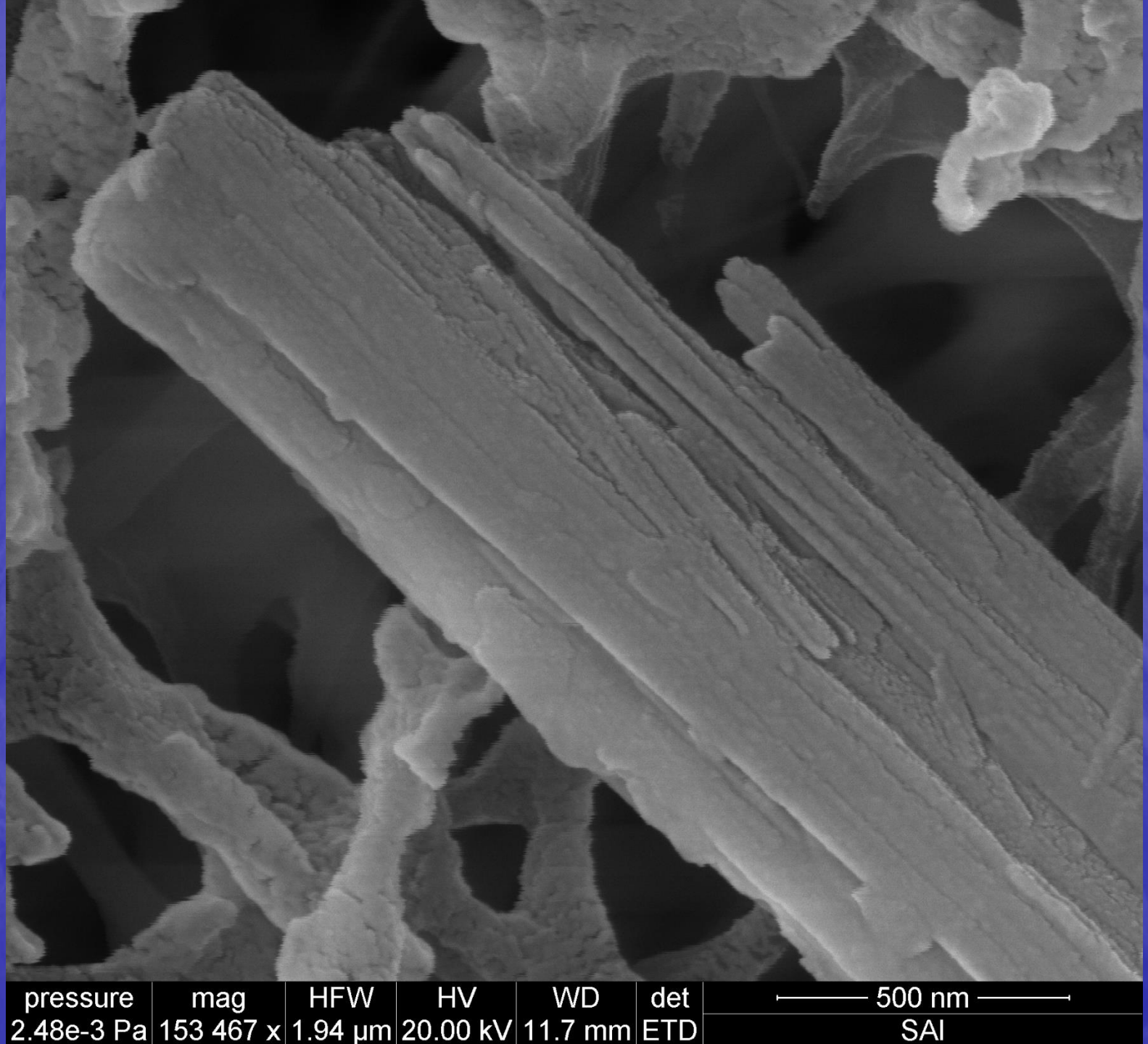
12/14/2017
HT: 100KV
TEM Magnification: 50,000x

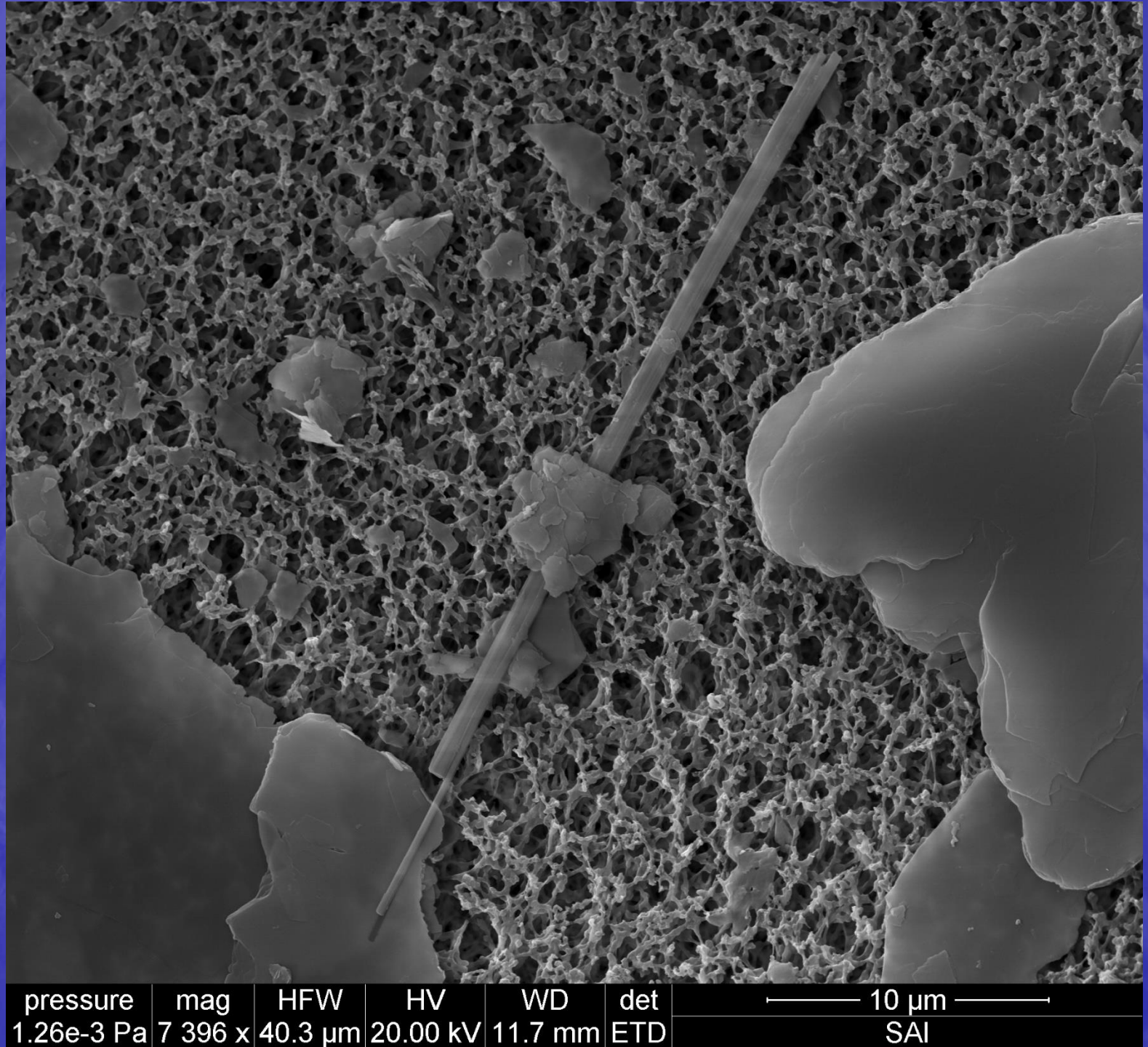
Claires LA 1726287_Compact Tremolite

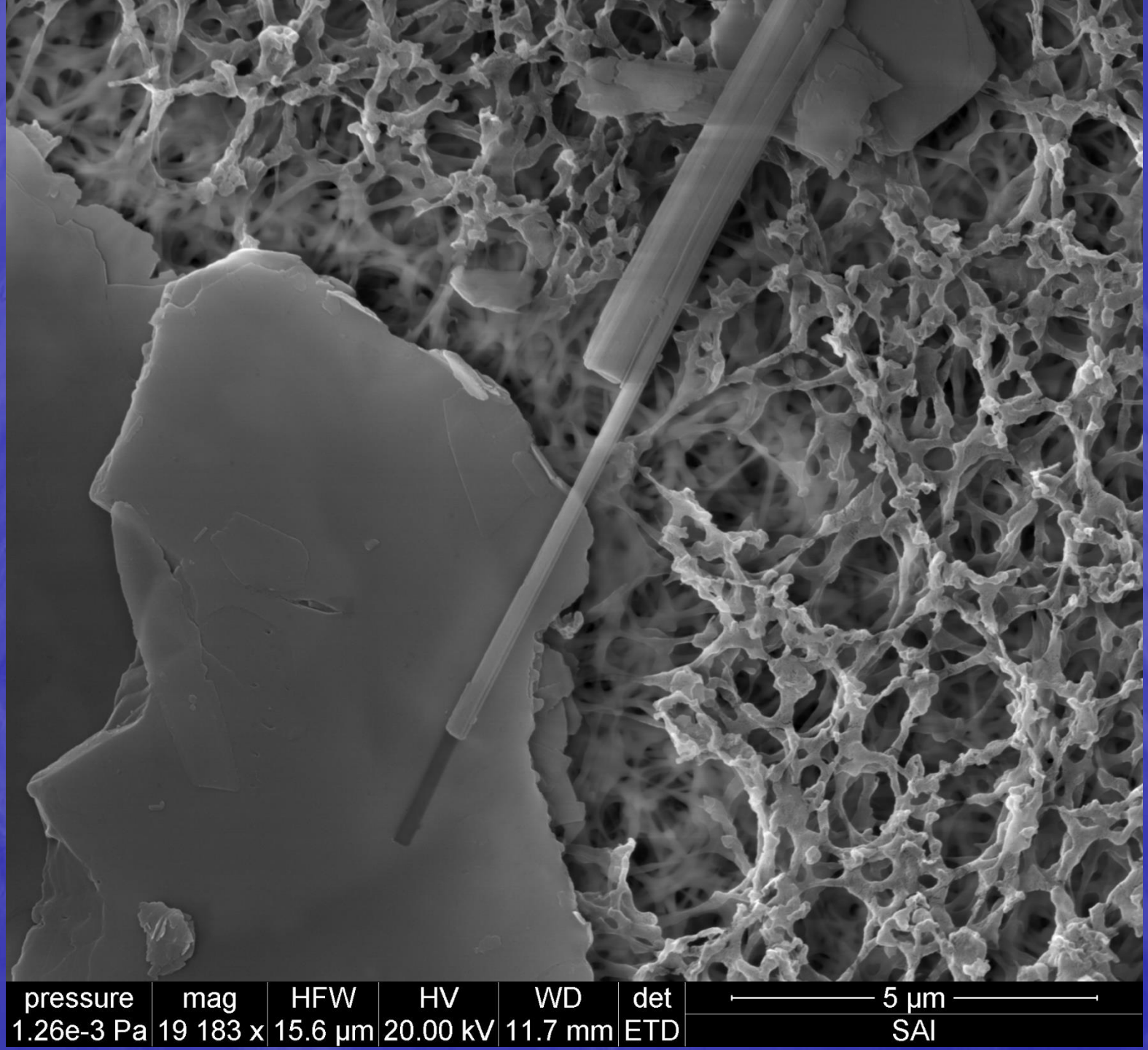

pressure
4.87e-3 Pa
mag
16 598 x
HFW
18.0 µm
HV
20.00 kV
WD
11.7 mm
det
ETD
5 µm
SAI


pressure
3.25e-3 Pa
mag
60 465 x
HFW
4.94 µm
HV
20.00 kV
WD
11.7 mm
det
ETD
1 µm
SAI


pressure
2.84e-3 Pa
mag
42 755 x
HFW
6.98 µm
HV
20.00 kV
WD
11.7 mm
det
ETD
2 µm
SAI


pressure
2.48e-3 Pa
mag
153 467 x
HFW
1.94 µm
HV
20.00 kV
WD
11.7 mm
det
ETD
500 nm
SAI


pressure
1.26e-3 Pa
mag
7 396 x
HFW
40.3 µm
HV
20.00 kV
WD
11.7 mm
det
ETD
10 µm
SAI

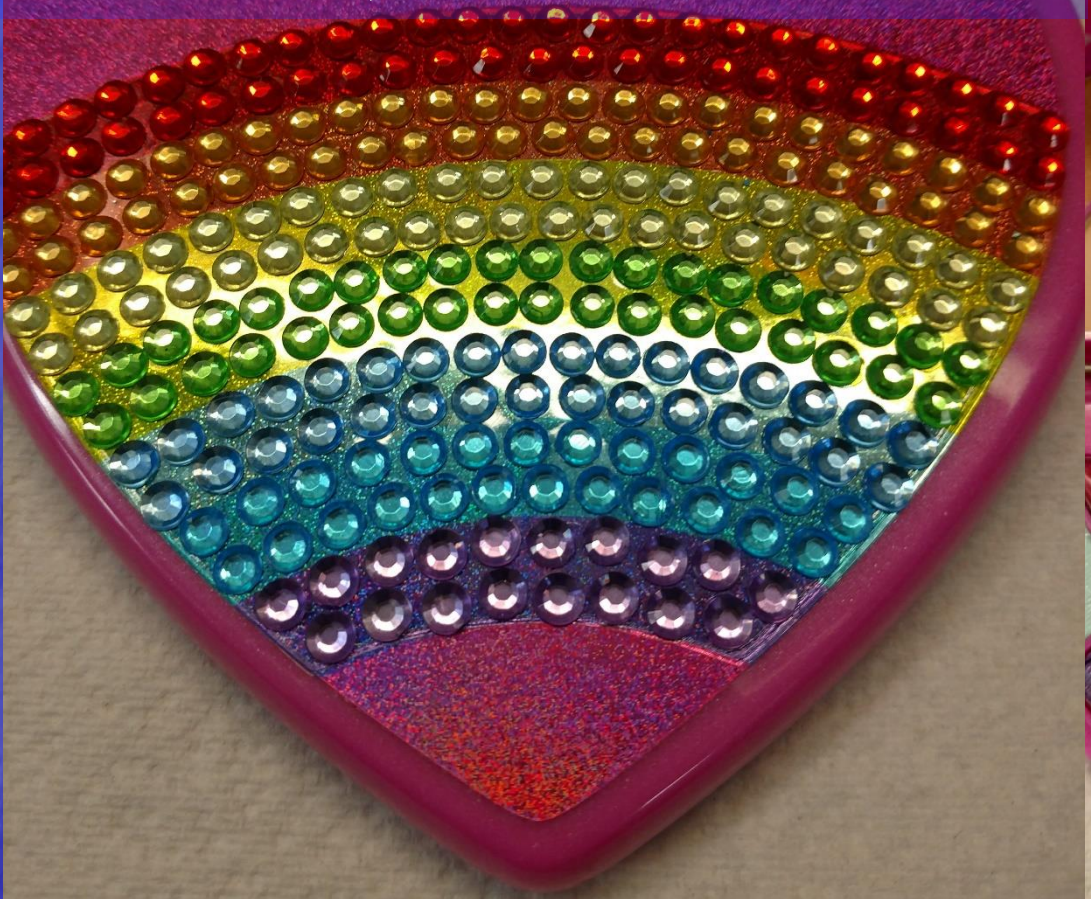

pressure
1.26e-3 Pa
mag
19 183 x
HFW
15.6 µm
HV
20.00 kV
WD
11.7 mm
det
ETD
5 µm
SAI

GRR Sample D

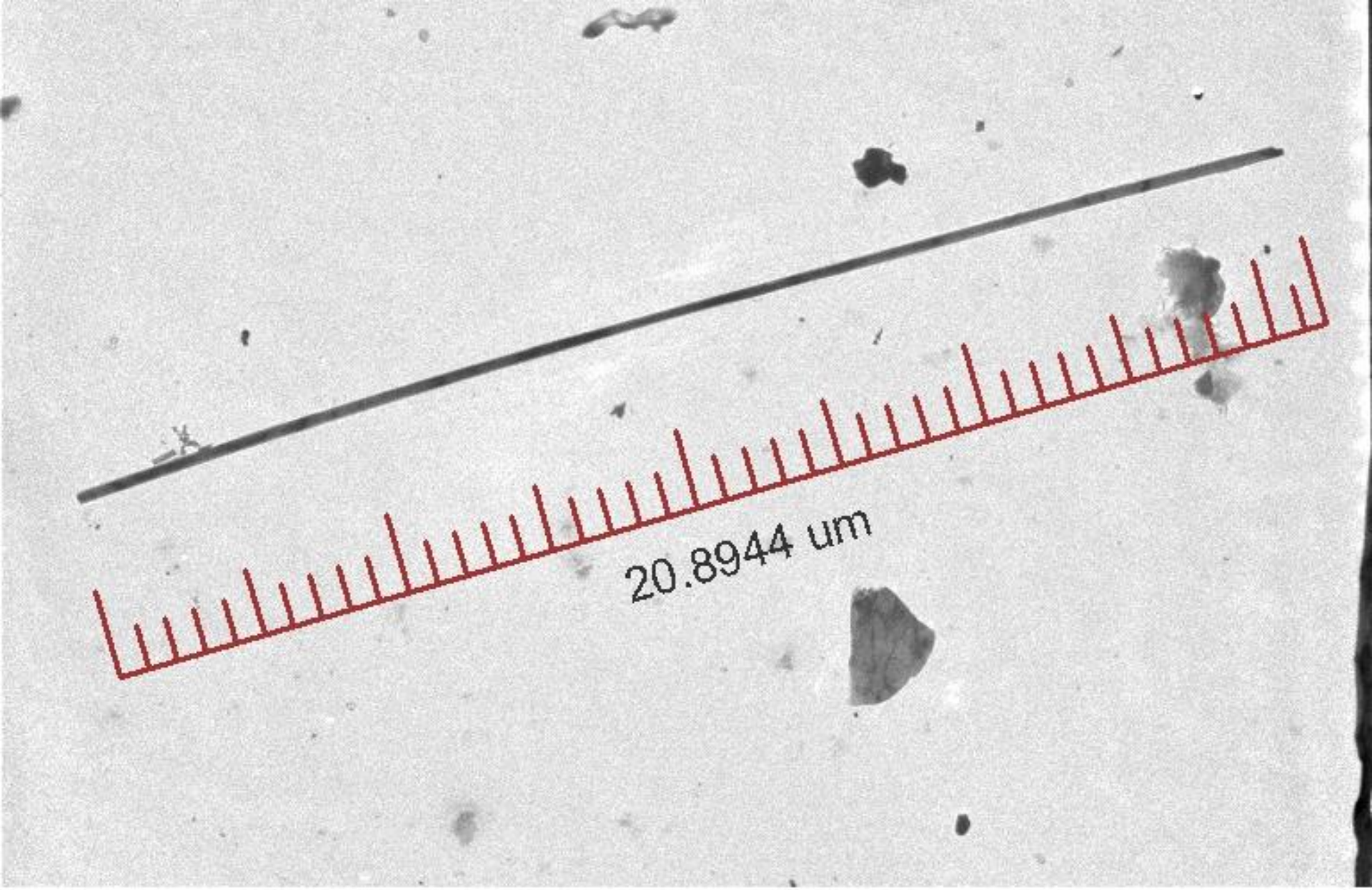
Claire's Bejeweled Heart: eyeshadow

Purchased December 01, 2017

In Greensboro, NC



GRR Ave: 212 Million Asbestos Str/gram



12/12/2017

HT: 100KV

TEM Magnification: 3,000x

Claires GSO 1725538_4_Purple_scan Tremolite

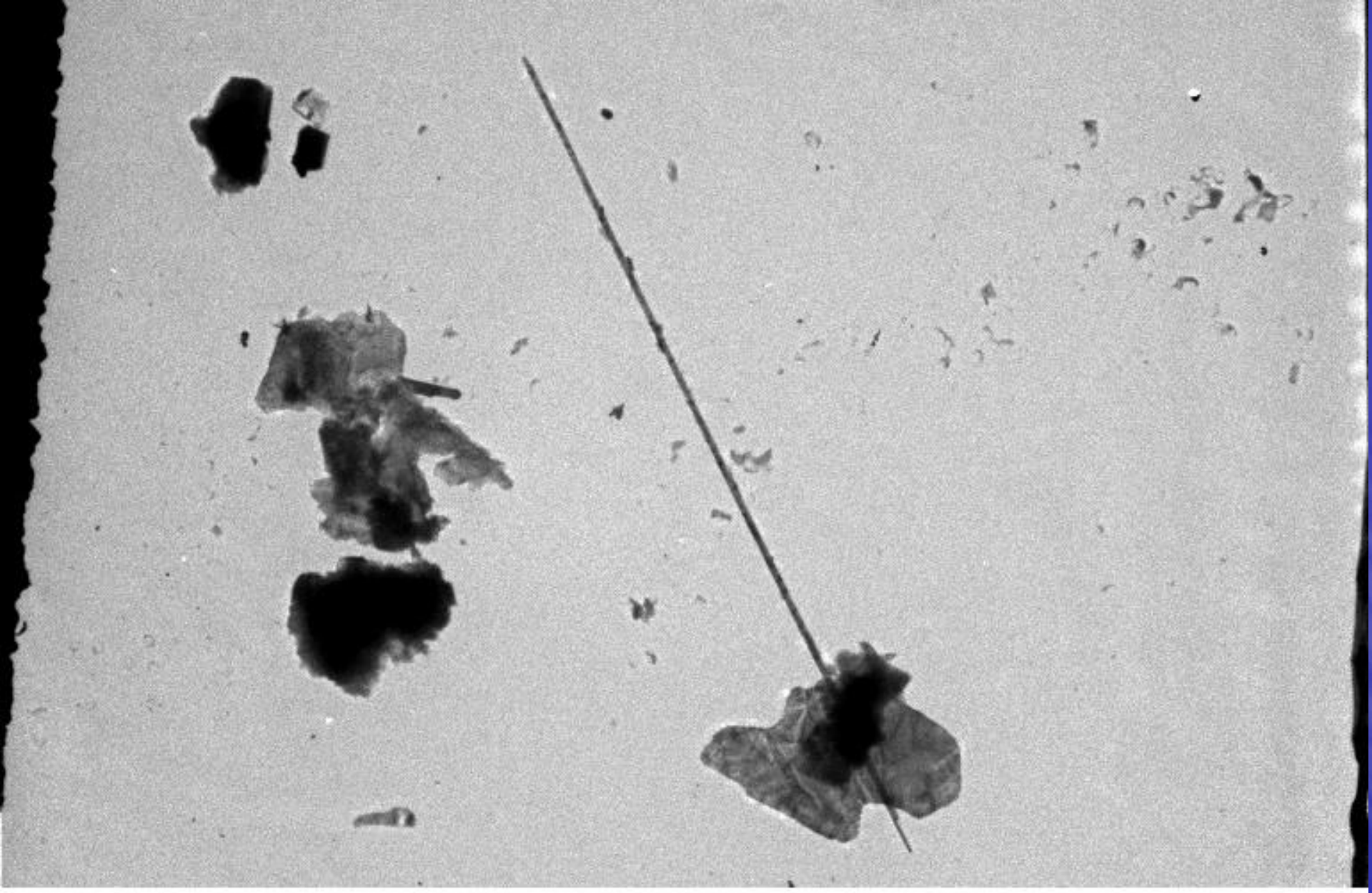
GRR Sample E

Claire's Sparkely Heart: Blush

Purchased December 05, 2017

In Atlanta, GA

GRR Ave: 152 Million Asbestos Str/gram



12/15/2017
HT: 100KV
TEM Magnification: 1,200x

Claires 1726301_Compact_Tremolite

20 um

GRR Ave: 25 Million Asbestos Str/gram

GRR Sample F

Claire's Bejeweled Heart: eyeshadow

Purchased December 02, 2017

In Indianapolis, IN


Claires IND 1725717_6_ Orange scan Tremolite
12/13/2017
HT: 100KV
TEM Magnification: 8,000x
5 um

GRR Sample G

Claire's Black shiny Makeup Kit

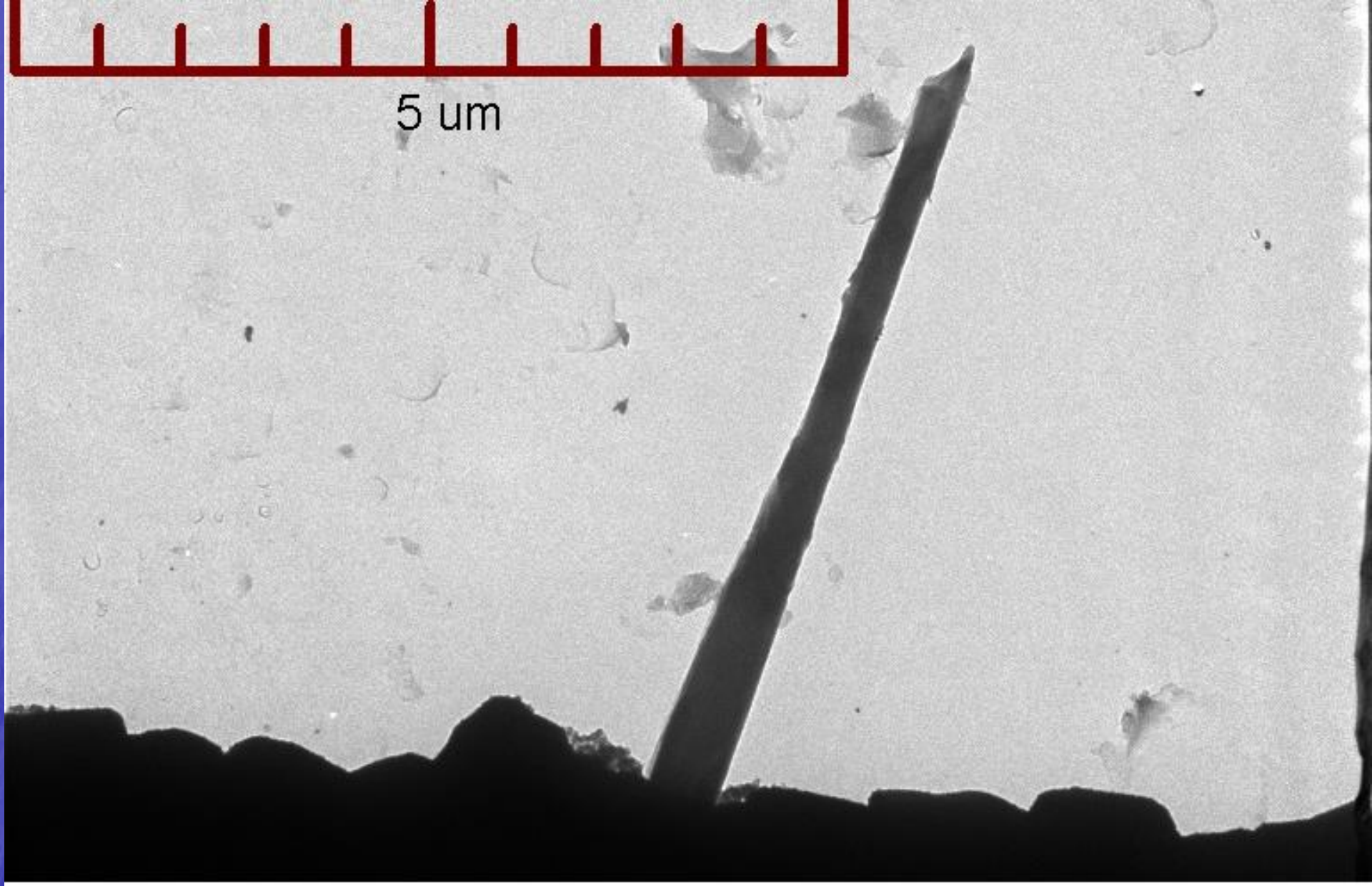
Pink eye shadow

Purchased December 02, 2017

In Los Angeles, CA

GRR Ave:

15.2 Million Asbestos Str/gram

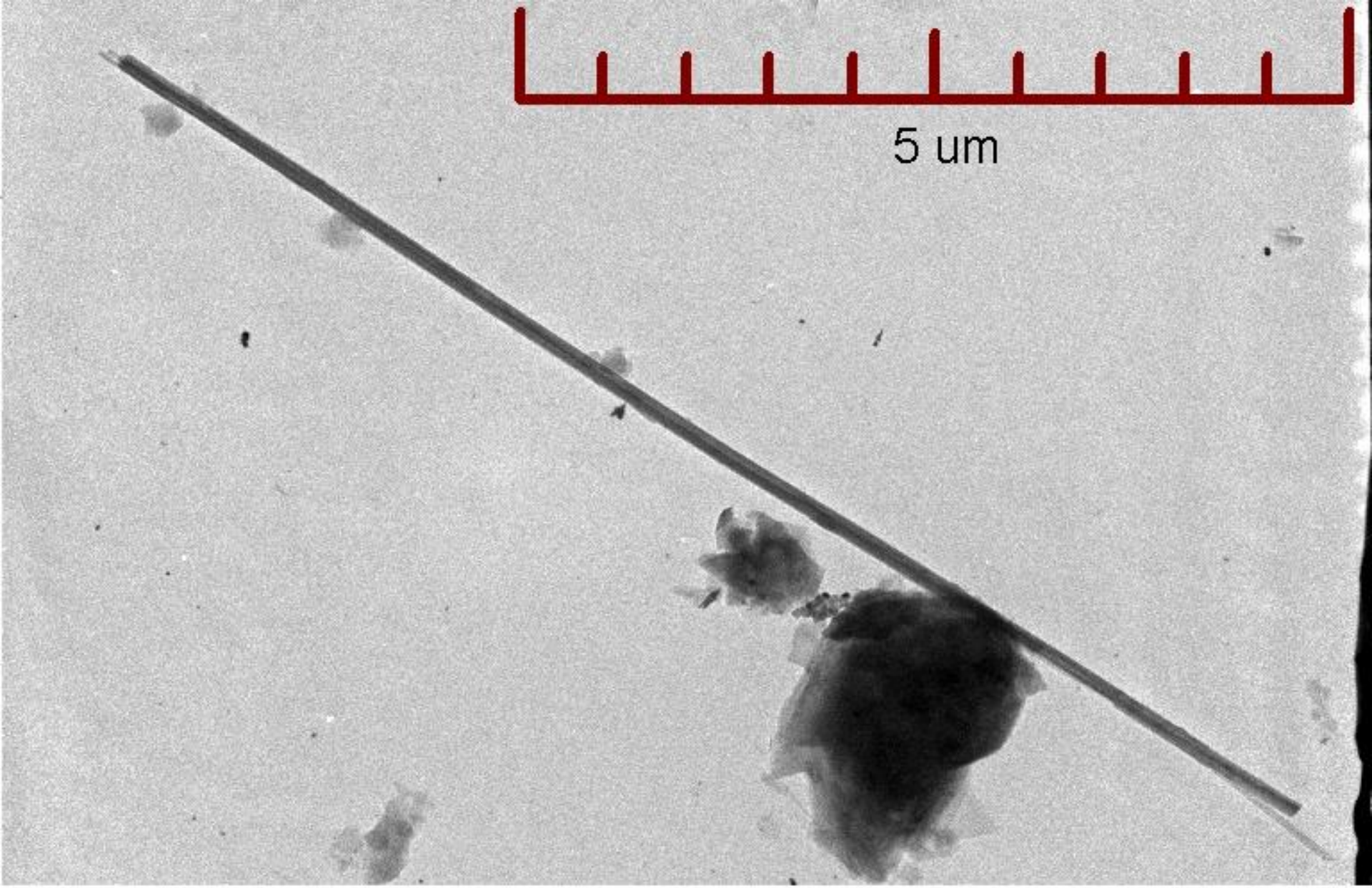


12/14/2017

HT: 100KV

Claires LA 1726287_1_Pink_Tremolite

TEM Magnification: 10,000x

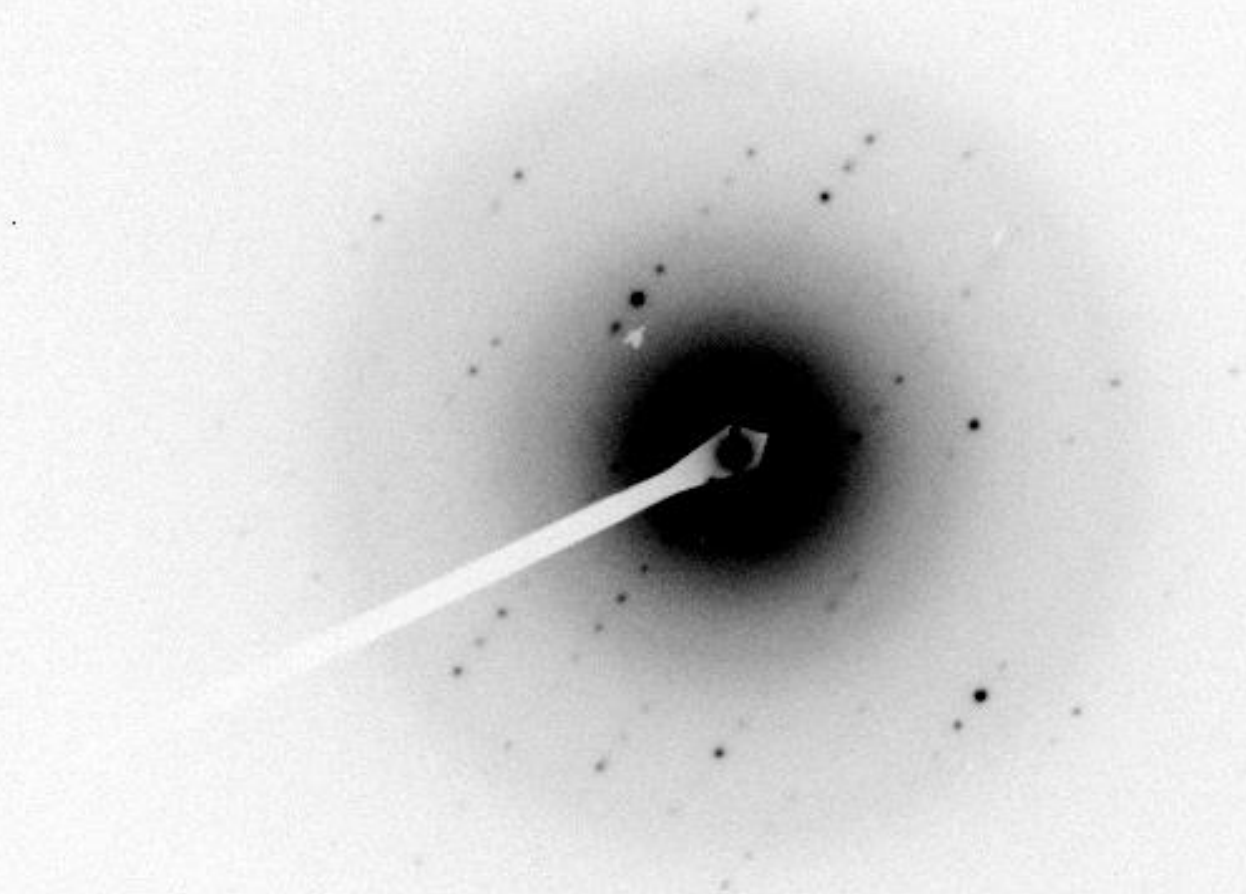


12/14/2017

HT: 100KV

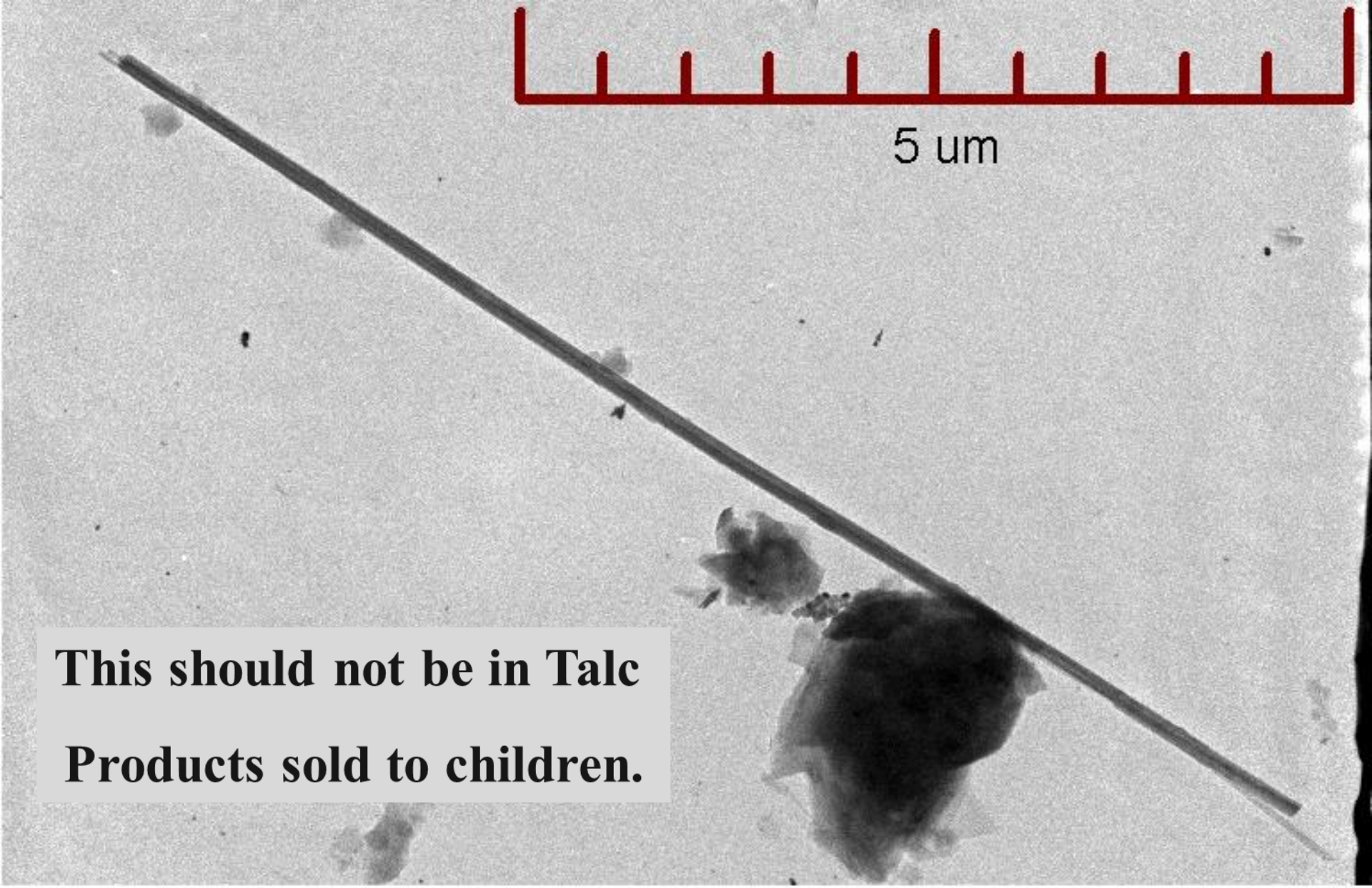
TEM Magnification: 10,000x

Claires LA 1726287_1_Pink_Anthophyllite



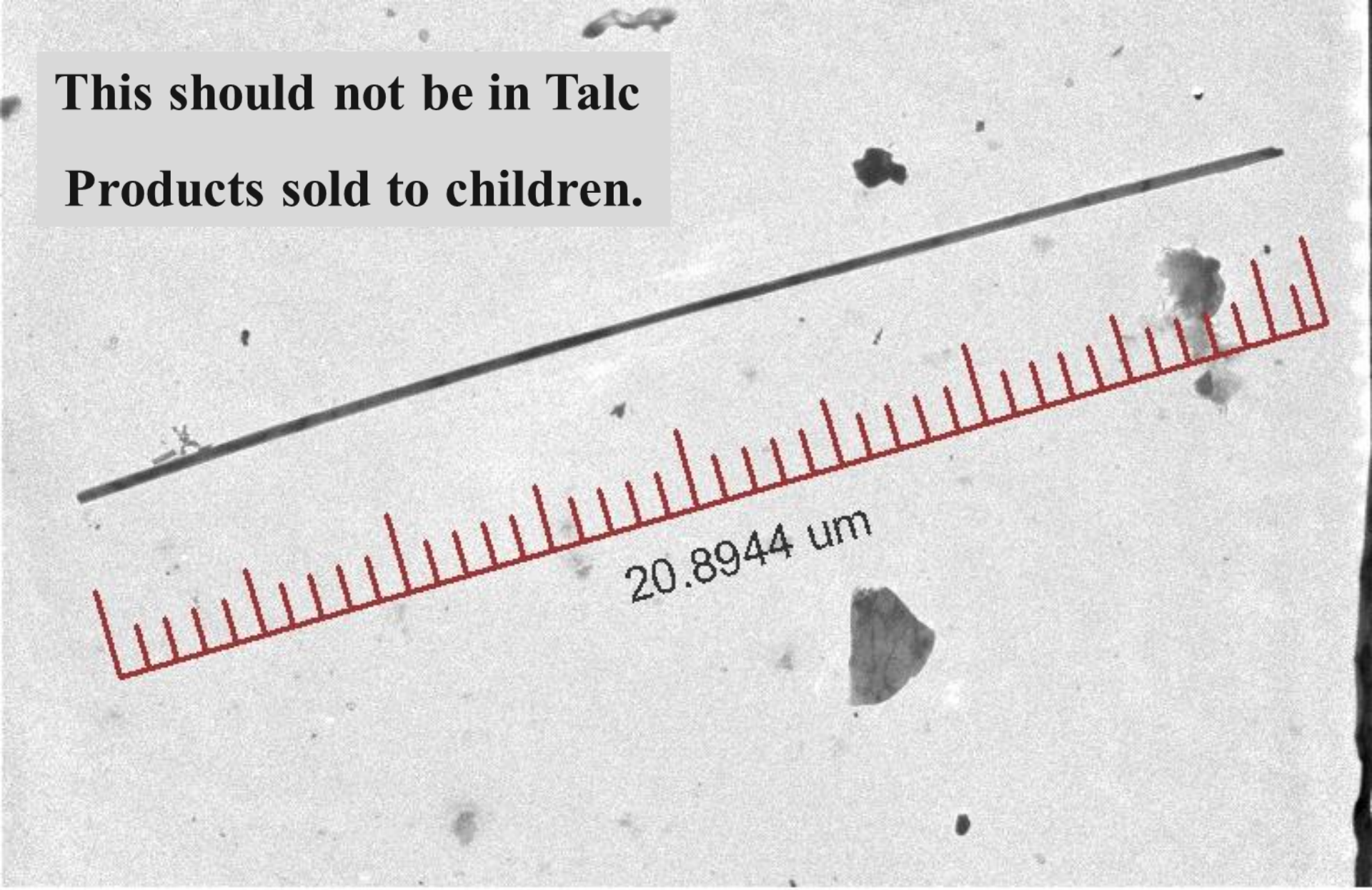
12/14/2017
HT: 100KV
TEM Magnification: 50cm

Claires LA 1726287_1_Pink_Anthophyllite



12/14/2017
HT: 100KV
TEM Magnification: 10,000x

Claires LA 1726287_1_Pink_Anthophyllite



12/12/2017
HT: 100KV
TEM Magnification: 3,000x

Claires GSO 1725538_4_Purple_scan Tremolite

We Need a Method That WORKS.

Asbestos in Talc by Electron Microscopy

Method for the Determination of Asbestos Fiber Structures Consistent with Airborne Counting Criteria in Mineral Powders, e.g., Talc; Quantified in Structures per Gram by Transmission and/or Scanning Electron Microscopy

Procédé pour la détermination et la quantification de l'amiante dans des poudres minérales, par exemple le talc, en accord avec les critères de comptage des structures par grammes en suspension dans l'air par microscopie électronique

S. Fitzgerald, PG

Key Words: asbestos, talc, talcum, asbestiform, electron microscopy, TEM, SEM, tremolite, anthophyllite, richterite, cosmetic, pharmaceutical, fibrous structures, serpentine, amphibole

Purpose/Rationale

Most previous methods for the determination and quantification of asbestos were specifically designed for asbestos as a constituent/ingredient intentionally added to bulk building products by the manufacturer [1]. Further, extant bulk or talc protocols specify analytical tools that are inadequate for the determination of relative low content and/or very fine fibrous minerals that commonly co-mineralize in nature with other mineral resources, such as vermiculite or talc. Specifically, it is inappropriate to label talc as "free of asbestos" by using methods incapable of resolving asbestos structures, as they can and do occur in talc reserves [2, 3]. Light microscopy (e.g., Polarized; PLM) is only suitable for larger asbestos fiber bundles, and only with content minimums consistently repeatable at a visual estimated 1% or more by weight, calibrated to volume. Methods such as this, as well as X-Ray Diffraction (XRD), are incapable of resolving fractions of fractions of a percent of asbestos and could leave tens of thousands of individual fibers undetected and unbound in fine mineral powders [10]. Finally, extant protocols, and those trained to use them, are more geared toward commercial asbestos minerals (primarily chrysotile, amosite [asbestiform granite-cummingtonite], and crocidolite). To the contrary, anthophyllite, tremolite, and other calcic amphiboles (e.g., richterite, winchite, etc.) are the asbestos varieties more commonly found with talc or vermiculite in the earth, often intergrown with the minerals of interest at the finest level. We now know such methods are woefully inadequate for the determination and quantification of asbestos in talc, and in need of modernization [3]. This holds especially true if one considers the definition of asbestos provided by NIOSH in 1972, where asbestos was defined as *any fiber* of chrysotile, crocidolite, amosite, anthophyllite, tremolite, or actinolite with an aspect ratio >3:1, greater than 5µm in length [8].

Further, asbestos content in disaggregated powders or soils at levels much lower than PLM and/or XRD can detect have proven capable of releasing significant levels of airborne fiber concentrations. For example:

"In particular, it has been shown that as little as 0.001% of asbestos in loose clay soil can produce around 0.1 fiber per ml of asbestos in air while at the same time respirable dust concentration is around 5 mg/m³" [4–6], and

"Recent data from the Libby site and other sites provide evidence that soil/debris containing significantly less than 1 percent asbestos can release unacceptable air concentrations of all types of asbestos fibers (i.e., serpentine/chrysotile and amphibole/tremolite)." [7]

This standard is therefore written to engage the tools by which modern science can best determine and quantify these fine fibers and low relative concentrations with precision and accuracy, by employing transmission and/or scanning electron microscopy (TEM/SEM) techniques, which have been proven to be effective beyond all other analytical tools in differentiating asbestos fibers, specifically as it occurs as accessory minerals in resources such as vermiculite or talc, at the size fraction known to be of greatest impact to human health.

Although the inadequacy of PLM or XRD for the specific determination of talc and asbestos was recognized and addressed with the publication of an earlier McCrone TEM talc protocol by Kremer and Millette in *A Standard TEM Procedure for Identification and Quantitation of Asbestiform Minerals in Talc* [9] in The Microscope in 1990, some key elements of that specification were found lacking in research testing for development of this standard. To that method's credit, the need for analytical electron microscopy (AEM) and greater analytical sensitivity was identified [2, 9], as it recognized that "manufacturers have become concerned with the potential presence of these minerals in their products in any measurable quantity". To that method's detriment, however, the use of formvar drop-mount preparations has proven less reliable than full filter dispersion for repeatability, and end report quantifications used full fiber-volume calculations to provide very small percentages that have been found difficult to repeat [8], where quantification in asbestos fiber-structures per unit weight is more repeatable and relatable to exposure potential.

Rationale References

1. U.S. Environmental Protection Agency. Test Method EPA/600/R-93/116: Method for the Determination of Asbestos in Bulk Building Materials, 1993.

2. CTFA Cosmetic Talc J4-1. Cosmetic, Toiletry and Fragrance Association, Inc., Nititakis, J.M. and McEwen G.N. (eds.), Washington D.C., 1982. CTFA Compendium Method J 4-1. Asbestiform amphiboles minerals in cosmetic talc. In: Cosmetic Ingredients Test Methods. Cosmetic, Toiletry and Fragrance Association: Washington D.C., pp 1–6, 1990.

3. Block, L.H.; Beckes, D.; Ferret, J.; Meeker, G.; Miller, A.; Osterberg, R.; Patil, D.; Pier, J.; Rieman, S.; Rutstein, M.; Tomaino, G.; Van Orden, D.; Webber, J.; Weibrid, J.; Wolfgang, S. and Moore, K. "Modernization of Asbestos Testing in USP Talc," Stimuli

Subcommittee on Economic and Consumer Policy:
Carcinogens in Talc and Asbestos Detection

12/10/19

Bill Longo Testifies, 12/10/19

- Industry Standard DL: 10-14 Million Fibers/ gram
- HLS: 4,500 Million Fibers/ gram
- Would not find chrysotile
- “Ban use of talc in cosmetic products”

Latest from the FDA

EXECUTIVE SUMMARY[1]

PRELIMINARY RECOMMENDATIONS ON TESTING METHODS FOR ASBESTOS IN TALC AND CONSUMER PRODUCTS CONTAINING TALC

January 6, 2020

In the fall of 2018, the United States Food and Drug Administration (US FDA) formed the Interagency Working Group on Asbestos in Consumer Products (IWGACP), with representatives from eight federal agencies[2], to support the development of standardized testing methods for asbestos and other mineral particles of health concern in talc that could potentially affect consumer product safety.[3] The IWGACP was formed in response to reports of the presence of asbestos in talc-containing cosmetic products, with talc being the presumptive source of asbestos. Since 2017, there have been several voluntary recalls of cosmetic products by retailers in the US and globally (Canada, Netherlands, Taiwan) due to the presence of asbestos.

The optimal analytical approach should address potential interference by sample matrices and thereby ensure sensitivity at levels or concentrations that are protective of public health. In addition, multiple sampling and analysis methods will be required to provide all the information that is needed to make health protective identification and classification of asbestos and other EMPs of potential concern. To improve agreement in data interpretation among stakeholders and resolve inconsistencies in applying published methods and counting criteria, IWGACP recommends minimum content and format for analytical reports. IWGACP also suggests written protocols that specify appropriate instruments, methods, and counting rules for the detection, quantification, and classification of EMPs. In conclusion, the IWGACP recommends:

1. Adoption of the term EMP as *"any mineral particle with a minimum aspect ratio of 3:1"*, consistent with how this term is defined in the NIOSH Bulletin 62, to resolve ambiguity and disagreement in mineral (asbestos versus non-asbestos) identification.

2. Testing laboratories report all EMPs having length ≥ 0.5 µm (500 nm).

3. That test methods specify reportable EMPs identified as amphibole or chrysotile particles as covered minerals.

4. Test methods require the counting and reporting of covered EMPs as a function of sample mass. When counting, IWGACP recommends referring to guidelines such as ISO 10312 to classify primary and secondary structures. Individual fibers in secondary structures can be counted recording the dimensions of each fiber.

5. Use of TEM at nominally 20,000x magnification, in addition to PLM, to resolve the issues of sensitivity that cause reporting of false negatives for covered EMPs. IWGACP strongly recommends using TEM with energy dispersive X-ray spectroscopy (EDS) and selected area electron diffraction (SAED) analyses to reliably detect and identify chrysotile and asbestiform and non-asbestiform amphibole minerals, including EMPs whose narrowest width is <200 nm (the limit of resolution for light microscopy). SEM might be useful as a complementary method but has significant shortcomings for identification of chrysotile and visualization of the narrowest particles in the population that can only be overcome by using TEM.

6. That "mass percent," a unit that is frequently used to express content of asbestos in commercial bulk materials, is not appropriate for measurement of EMPs in talc and consumer products containing talc because weight percent does not correlate with the number of fibers, and one large fiber could dominate the mass percent value.

7. Although IWGACP concludes that criteria for differential counting and classification of EMPs meeting criteria in #2 would be beneficial, no specific recommendations were agreed upon during deliberations. Therefore, at this time the IWGACP recommends reporting and counting all EMPs of covered minerals under a single classification with additional information that would allow further classification based on measurements such as mineral type and dimensions in the future.

In addition, the IWGACP has identified the following as areas for directing efforts to promote reliability of the analytical methods for asbestos and other EMPs of health concern in talc and talc-containing consumer products:

- Validation of analytical methods (XRD, PLM, TEM) specific to talc and consumer products containing talc that minimize false positive and false negative results.
- Research and validation of methods of sampling that maximize sample representativeness and minimize error and false positives and false negatives.
- Research on methods for sample preparation, in particular, treatments (e.g. "concentration methods") that improve sensitivity while leaving covered minerals unchanged with respect to identity and dimensions.
- Development of talc-specific reference standards with known concentrations of specific EMPs that can be used to assess laboratory and analyst proficiency, increase inter-laboratory concurrence in method validation, minimize reporting errors, and potentially provide for improved reliability of quantitative analysis.

Asbestos in Talc Testing

Sean Fitzgerald, PG

Presented at the

Testing Methods for Asbestos in Talc and Cosmetic Products Containing Talc Public Meeting

U.S. Food and Drug Administration

White Oak Campus

Bldg. 31 Conference Center, The Great Room

Silver Spring, Maryland

February 4th, 2020