



Sent via e-mail only

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May 29, 2020

Michael P. Kutney, P.G.
Environmental Group Manager
Pottsville District Mining Office
Pennsylvania Department of Environmental Protection
5 West Laurel Boulevard
Pottsville, PA 17901

Richard Tallman
Civil Engineer (General), E.I.T.
Pottsville District Mining Office
Pennsylvania Department of Environmental Protection
5 West Laurel Boulevard
Pottsville, PA 17901

**Re: Response to Comments – April 17, 2020 Department Letter
Rock Hill Quarry
Hanson Aggregates Pennsylvania LLC
SMP # 7974SM1
East Rockhill Twp., Bucks Co., PA**

Mr. Kutney & Mr. Tallman:

Hanson Aggregates Pennsylvania LLC (Hanson) is providing this correspondence to address the items required for submission by May 31, 2020 in the Department's April 17, 2020 letter. Specifically, the correspondence attached addresses the following:

- All items included in Comments 1, 2 and 3 of the Department's March 2, 2020 Qualitative Geologic Survey Report (QGSR) comment letter;
- The Department's request for a Revegetation Plan describing disturbed areas capable of supporting plant growth and a plan to revegetate them congruent with the current operational restrictions; and
- The Department's request for a Draft Air Monitoring Plan that incorporates monitoring for airborne asbestos exposure during periods of limited activity at the quarry as well as during inactivity.

Hanson has also included the following direct responses to the following two (2) questions included in the Department's March 2, 2020 QGSR comment letter. The Department's comment is repeated below in *italics*, followed by Hanson's response.

Please explain why drilling and sampling for asbestos was not proposed in other locations in the quarry, and explain what consideration is being given to the question of whether avoiding asbestos entirely would be easier and safer than mitigating its effect.

Hanson Response: Drilling and sampling for asbestos was completed per the PA DEP approved Workplan. The Workplan scope was designed to investigate and clear a specific area intended for the next phase of mining. A significant amount of field investigation, sample collection and data analysis has been completed at the site to characterize the occurrence of asbestiform actinolite. Field observation and sample collection from boulders staged at various locations across the site and the aggregate products produced in 2018 are indicative of the diabase mined historically across many locations at the site. The geologic logging and sample analysis of core borings installed in 2019 were conducted to assess the presence of NOA within the next phase of mining at the site. The results indicate the overall absence of NOA or its very limited presence in trace concentrations in sporadic actinolite mineral veins formed within the diabase rock. Given the trace concentrations of NOA detected at the site, it is premature to consider mining avoidance until future air sampling and data evaluation is conducted to assess potential for airborne NOA fibers from mining and related activities at the site. Furthermore, Hanson has previously detailed the use of engineering controls that are widely recognized by regulatory agencies and utilized throughout the mining industry to prevent fugitive dust emissions and meet applicable standards.

What is the expected annual production of aggregate from the quarry? What is the total tonnage of aggregate planned for mining at the quarry?

Hanson Response: Currently, Hanson is only planning to remove the requisite 500 tons from the site to maintain an active Large Noncoal Surface Mining Permit. The source of the 500 tons will be the previously crushed aggregate stockpiles on the northwest portion of the permit area. Therefore, no processing will be conducted at the site until such time Hanson receives approval from the Department to resume production of crushed aggregate. The total tonnage of aggregate planned for mining at the quarry is proprietary and confidential information. There is likely 100 years of reserves within the current permit area depending on the achieved annual production rate.

Additional correspondence to be submitted by June 30, 2020 will include the analytical results and discussion of Department requested TEM analyses, petrographic analyses and reanalysis of the water samples using EPA Method 100.1.

Please feel free to contact me at (610) 366-4819 should you wish to discuss this submission.

Regards,



Andrew J. Gutshall, P.G.
Area Environmental Manager

encl: EARTHRES letter to Andrew J. Gutshall, P.G. dated May 29, 2020
RJ Lee Group letter to Andrew J. Gutshall, P.G. dated May 29, 2020

cc: John Stefanko, PADEP (e-mail only)
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Michael C. Lewis, CHMM, Hanson (e-mail only)
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Environmental File

EARTHRES Response to Comments Letter

May 29, 2020



May 29, 2020

Andrew J. Gutshall, P.G.
Area Environmental Manager
Hanson Aggregates Pennsylvania LLC
7660 Imperial Way
Allentown, PA 18195

**RE: Response to PADEP Technical Comments dated March 2, 2020
Regarding the “Qualitative Geologic Survey Report” dated November 15, 2019
Rock Hill Quarry (Pierson Materials/Hanson Aggregates)
East Rockhill Township, Bucks County, PA**

Dear Mr. Gutshall:

EARTHRES is providing the following comment responses to the Pennsylvania Department of Environmental Protection (PA DEP) technical comment letter of March 2, 2020, which was addressed to your attention. The comments concern the Qualitative Geologic Survey Report (QGSR) that EARTHRES submitted to the PA DEP Pottsville District Mining Office on behalf of Hanson Aggregates Pennsylvania, LLC (Hanson). The comments provided by PA DEP are repeated below *in italics*, followed by comment responses in **bold**.

2. Section 3.0, Section 3.1, and Section 4.0:

- *How was the diabase determined to be York Haven Type?*

RESPONSE:

As discussed within the QGSR, the diabase in the Newark Basin has been extensively studied and mapped by the Pennsylvania Geological Survey, universities, and other researchers. Please refer to the geologic map prepared by Smith et al., 1975¹, which shows the distribution of the three (3) diabase types in the region (Attachment 1, Geologic Map). As shown on the map, the diabase in the vicinity of Quakertown and in the location of Rock Hill Quarry is identified as York Haven Type. EARTHRES’ field observations and data from the Quarry as described below are consistent with this mapping.

- *What site-specific observations were made to make the interpretation that the diabase is homogeneous as well as the lithology of the site in general?*

¹ Smith, Robert C., Rose, Arthur W., and Lanning, Robert M., 1975, Geology and Geochemistry of Triassic Diabase in Pennsylvania: Department of Geosciences, Pennsylvania State University, University Park, Pennsylvania.

RESPONSE:

Site-specific observations of the geology include visual inspection of quarry highwalls, detailed observation and logging of 500 feet of rock core, observation of several hundred boulders, as well as observation of crushed aggregate stockpiles. These observations all fit with the published description of the York Haven diabase as described within the QGSR and indicate a homogeneous diabase without geologic facies changes, ductile shear zones, or brittle shear zones that would constitute separate units. Thin-section/petrographic analysis completed for the site indicates the mineralogy to consist of 95 percent pyroxene and plagioclase feldspar, which is consistent with York Haven diabase (see Attachment 2, Petrographic Analysis). Petrographic analysis will be completed on two (2) additional diabase samples and results will be provided in a subsequent submission to further substantiate this interpretation.

3. Please Explain or Provide:

- *Please verify the permitted acreage. Section 4.0 states the permitted area is 55 acres. Department records shows the permit acreage is 103.2 acres.*

RESPONSE:

EARTHRES has verified that the permit acreage is 103.2 acres. The reference contained within Section 4.0 and cited above of 55 acres refers to the limit of mining acreage.

- *On what basis is the asbestos detection in DB-1 anomalous? What field observations, beyond visual, were used to make this determination? The literature cited is general in nature and was not addressing NOA. (Core Sampling Results, Section 6.2)*

RESPONSE:

Sample DB-1 was originally intended to characterize the diabase matrix. The detection of naturally occurring asbestos (NOA) in the sample was viewed as an anomaly because published literature, diabase mineralogy, and field observations indicate NOA is not present in the diabase matrix. Upon further visual inspection, DB-1 was found to be located in a mineralized vein zone, which contributed to the detection of NOA as discussed within Section 5.3 of the QGSR. A petrographic analysis of DB-1 will be completed to provide further characterization of the sample.

Please explain how the vein volume assessment is an overestimate when mineralized veins are ubiquitous at the site. Proposed mining would presumably encounter all the veins.

RESPONSE:

The QGSR does not expressly or implicitly indicate that mineral veins within the diabase rock are ubiquitous. In fact, this terminology is misleading as it is ambiguous and implies

that mineral veins are present everywhere and constitute a large percentage of the rock mass. Site observations do not support the conclusion that mineralized veins, let alone actinolite veins that are asbestiform, are ubiquitous or comprise a large percentage of the rock mass. For example, only 33 out of hundreds of boulders examined indicated mineral veining, none of which was identified as asbestiform in hand sample and only seven (7) of which had trace detections of NOA. Further, quarry face mapping indicated only nine (9) veins observed along 150 feet of highwall along Bench 1 and only four (4) veins observed along 200 feet of highwall along Bench 2. Only one vein (Vein #7) was identified as actinolite in hand sample and was found to contain NOA at low levels.

To put these numbers into perspective, the relative amount of vein material was calculated using the vein width data provided in the Bench Face Data Mapping tables contained in the report. The vein widths of each of the thirteen (13) veins observed were added together and compared to the total footage of high wall inspected (see Attachment 3, Highwall Mapping Vein Assessment table). That data indicates a combined vein width of 5.58 feet for all thirteen (13) veins over the 350 feet of highwall inspected. Based on this data, veins comprise 1.6% of the rock exposed along the highwall. However, only one vein (Vein #7) was identified as actinolite and had a width of 8 inches or 0.66 feet. Based on this width, actinolite veining comprises only 0.19% of the rock exposed along the highwall. This estimate is comparable but lower than the actinolite vein percentage determined from the rock core data (see Table 5, Actinolite Vein Total Core Volume - Arithmetic Mean of 0.43%).

Furthermore, laboratory analysis of Vein #7 indicated it to contain 0.10% NOA. If this concentration is applied to the actinolite vein percentage calculated from the face, then the exposed highwall would contain only 0.00019% NOA:

$$\begin{aligned}\text{NOA on exposed highwall} &= \text{Fraction of Actinolite Veins} \times \text{Concentration of NOA in Veins} \\ &= 0.0019 \times 0.001 \\ &= 0.0000019 \text{ or } 0.00019\%\end{aligned}$$

These field observations and supporting calculations indicate that mineral veining and more importantly asbestiform actinolite veining is limited at the site. This conclusion is consistent with the limited amount of actinolite veining quantified in the rock cores and the trace concentrations of NOA detected in laboratory samples.

The vein volume assessment is considered an overestimate as the vein percentages for all cores were equally weighted in the assessment, even though CB-1, in particular, is located within a cluster of veins and has a significantly higher vein percentage than the other cores. Furthermore, comparison of actinolite vein percentage estimates based on the rock core data (0.43%) and face mapping (0.19%) indicates the cores encountered roughly twice the amount of actinolite veining. This data demonstrates that drilling successfully targeted actinolite veining. For these reasons, the core vein volume assessment is reasonably considered a conservative, overestimate.

- *The Department inquired about the goals of the drilling program prior to its approval. EarthRes replied, “The goal of the drilling program is to further evaluate the presence or absence of trace amounts of naturally occurring asbestos (NOA) in the rock to be mined. Success will be determined through laboratory analysis for NOA in the mineral vein samples identified in the cores as specified in the work plan.” Typically drilling results are compared with mapping results and an evaluation is made regarding whether there is a need for more data collection. In this case it appears that drilling data does not match the limited mapping. Estimates of mineralized veins, based on drilling data (Section 4.4), were made about the volume of veins and the NOA within them. However the drill core logs do not appear to intersect veins that are mapped. As mentioned above, veins at the site are ubiquitous, although comparatively few were logged. It appears that using drill core data, the volume of veins, and therefore the volume of NOA, are underestimated.*

RESPONSE:

As discussed above, term ubiquitous is misleading and implies that mineral veins are present everywhere and constitute a large percentage of the rock mass. The field observations (including observation of hundreds of boulders and the accessible quarry faces discussed above) and supporting calculations indicate that mineral veining and more importantly asbestiform actinolite veining is limited at the site. With regard to mapping of specific veins, geologic observations from the site indicate a one to one correlation of veins on the rock face with veins in the rock core may not to be observed due to variation of individual veins as they progress through the diabase host rock. This is demonstrated by observation of Vein #7 on Bench 1, which is noted to pinch out (reduce in thickness and terminate) as it progresses up the highwall. Similar variations in expression of this vein and others may be expected as they propagate away from the rock face. However, projection of the face veins along their structural trend is believed to be useful for identification of general zones of veining. This is demonstrated by results of coring from CB-1 which was located to intercept veining and contained almost 3 times the amount of actinolite vein volume as CB-2, which was not located near mapped veins (see Table 5, QGSR). In determining the core vein volume average, all of the rock core results were weighted evenly. For this reason and others cited in the response above, the core vein volume assessment is reasonably considered a conservative, overestimate.

- *Please explain, in detail, how EarthRes determined that, “it is indicated that the CB-1 #1 sample is from mineral Vein #7” (Section 5.5). Visually, they appear dissimilar. The drill log describes voids, but the Bench Face Mapping Data does not mention voids. The Bench Face Mapping tables states that Vein #7 is approximately 8 inches wide. Intersecting the vein (via drilling) at an angle, with respect to both the vertical and horizontal axes, will result in the vein appearing longer in the drill core. The photos provided do not show a vein of actinolite greater than 8 inches. Based on the Geologic Features and Boring Location Plan, two or three other veins (Nos. 1, 3, and 4) should have been intersected before Vein #7. These veins were not*

correlated in the drill logs. Looking at the bench face photographs, it would appear that more than 19.9' (the sample depth of CB-1) horizontal feet exists between Vein #7, and vein 1, 3, and 4. Properly orienting the core and surveying the bore hole would reduce uncertainty.

RESPONSE:

Vein #7 was projected along its strike and dip to evaluate the depth at which it would intersect core CB-1. CB-1#1 was then inferred to be from mineral Vein #7 based on its proximity to the intersection depth and having a description that most closely matched Vein #7, notably the presence of actinolite visible in hand sample. As discussed in the above response, Vein #7 as observed on the highwall is noted to vary in its width and could reasonably be expected to vary as it propagates into the wall toward CB-1. The small voids present at CB-1#1 is likely the result of entrapped air during crystallization of the vein material. Voids may or may not be present in Vein #7 at the rock face, but could reasonably be expected in a vein of hydrothermal origin. Small voids may be weathered on the face or otherwise unnoticeable or may have developed as the vein width decreased. The relationship between the other veins (Nos. 1, 3, and 4) would have to be determined in the same way via projection. The veins will not maintain the same separation observed at the bench face because the veins are not parallel (they strike in slightly different directions). However, as mentioned in the response above one to one correlation of veins on the rock face with veins in the rock core may not to be observed due to variation of individual veins as they progress through the diabase host rock.

- *The noncoal regulations require complete and accurate characterization of the geology and hydrology of the area proposed for mining. Every effort should be made to fully characterize the amount, type and location of asbestos at the Rock Hill quarry.*

RESPONSE:

Sampling efforts from 2018 and 2019 have generated 90 rock samples and 99 analyses for NOA with which to characterize the site. Approximately half of the analyses (N=49) were from targeted samples including hand samples, core samples, and boulder vein samples, and half (N=50) were from composite samples including stockpiles, crusher fines, and drill cuttings. NOA was detected at trace levels in both types of samples, with a slightly higher concentration in the Target Samples (see Table 7, Average All, QGSR). The results indicate the overall absence of NOA or its very limited presence in trace concentrations in actinolite mineral veins formed within the diabase rock at the site. This finding is consistent with site-specific observations of the geology including visual inspection of quarry highwalls, detailed observation and logging of 500 feet of rock core, observation of several hundred boulders, as well as observation of crushed aggregate stockpiles, none of which indicated asbestos visible in hand sample. This finding is also consistent with the extensive literature review

completed for the site which indicates the diabase is homogeneous and consists primarily of non-asbestos forming minerals.

In addition to these responses, EARTHRES is providing revised tables for the QGSR report, which were found to contain a decimal error in the calculations that artificially inflated the NOA estimates. The revised tables are included in Attachment D, Revised QGSR Tables.

I trust that the information provided herein sufficiently responds to the comments presented in the PADEP letter dated March 2, 2020. If you have any questions regarding the comment responses, please contact me at (800) 264-4553.

Sincerely,
Earthres Group, Inc.



Matthew S. Weikel, P.G.
Technical Manager

Attachments:

Attachment 1 – Geologic Map

Attachment 2 – Petrographic Analysis

Attachment 3 – Highwall Mapping Vein Assessment Table

Attachment 4 – Revised QGSR Tables

cc: Matthew Burns, Lehigh Hanson (via email)
David Raphael, K&L Gates (via email)

*Andrew J. Gutshall, P.G.
May 29, 2020*

ATTACHMENT 1
GEOLOGIC MAP

FIGURE 1. Distribution of three types of diabase in Pennsylvania.

Smith, Robert C., Rose, Arthur W., and Lanning, Robert M., 1975, Geology and Geochemistry of Triassic Diabase in Pennsylvania: Department of Geosciences, Pennsylvania State University, University Park, Pennsylvania.

(Adapted to show Site Location)

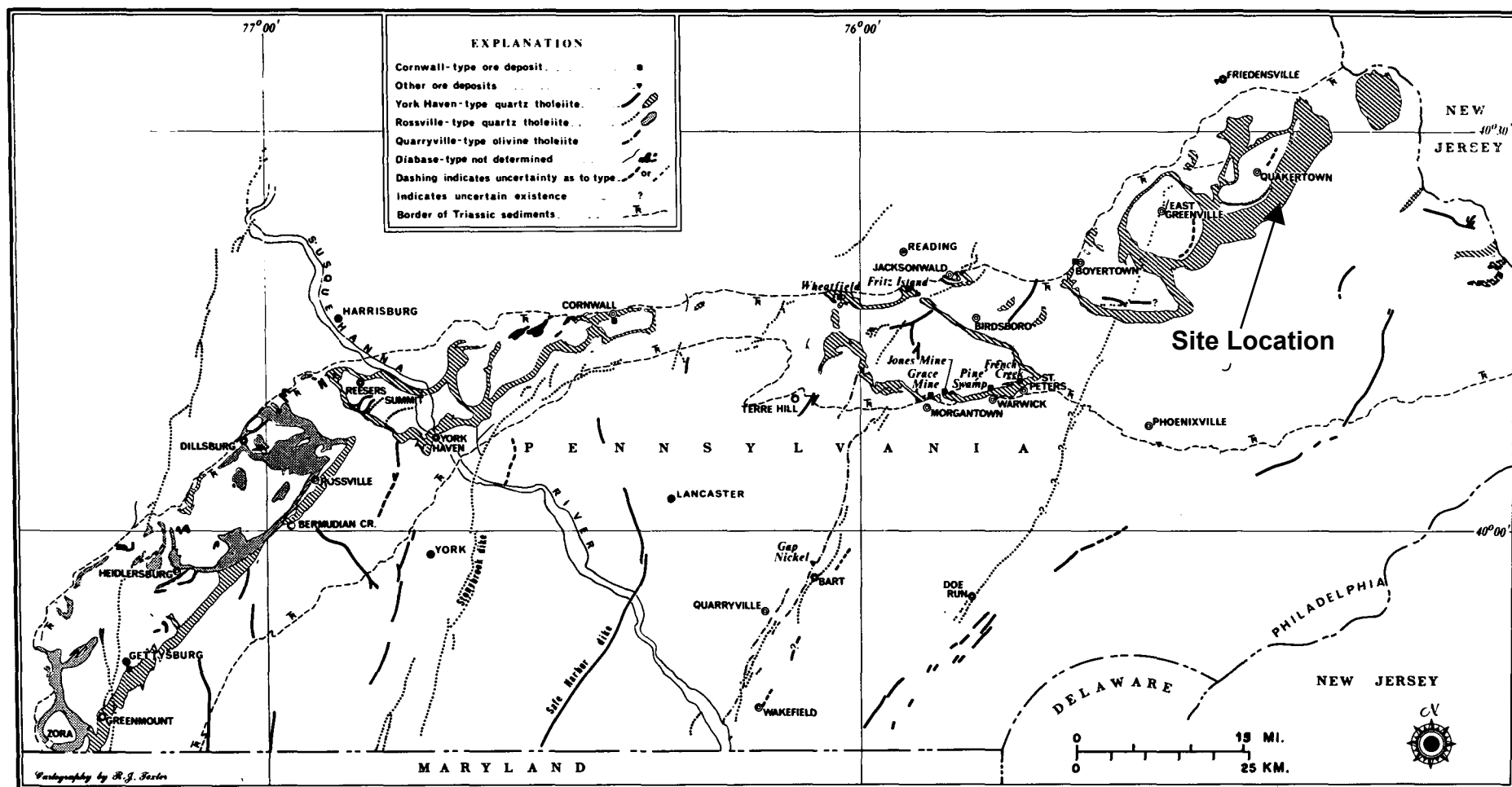


Figure 1. Distribution of three types of diabase in Pennsylvania. Map based on field observations and chemical analyses by R. C. Smith II and R. M. Lanning, state geological map (Gray and others, 1960), Longwill and Wood (1965), Peter W. Weigand (1970, personal commun.), D. B. MacLachlan (1973, personal commun.), and other sources.

*Andrew J. Gutshall, P.G.
May 29, 2020*

ATTACHMENT 2

PETROGRAPHIC ANALYSIS

Petrographic Examination of Stone

17-1357

By: Michael Trim

10/26/2017



Client: R.E. Pierson Construction Co.
Project Number: 170791
Date Sampled: 9/27/2017
Date Completed: 10/26/2017

Project: 2017 Quality Control
Lab Number: 17-1357
Location: East Rockhill Quarry
Petrographer: Michael Trim

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Sample Background

The sample investigated by this analysis is stone from the East Rockhill Quarry as provided by R.E. Pierson. The purpose of this examination is to identify and describe the stone.



Specimen selected for petrographic analysis

Initial Observations

After visual examination of the received sample, it was determined that it was represented by one type of rock. One representative hand specimen of stone was selected and utilized for the duration of this analysis. The sample is dark gray to nearly black in color, and is composed of mostly black and white grains. The rock exhibits features indicative of a fine grained intermediate – mafic intrusive igneous rock. The specimen has one face with a brown crust.



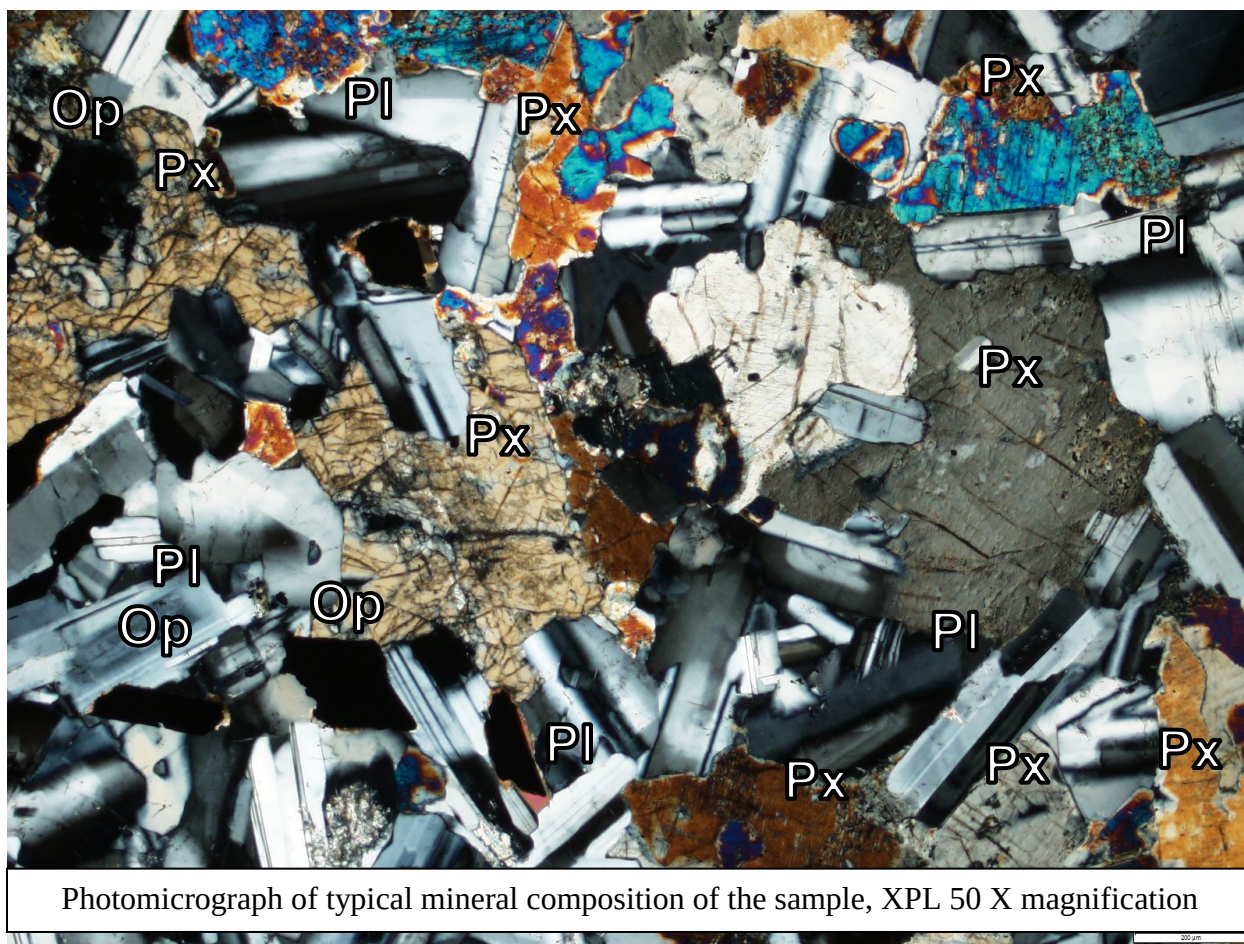
Image showing the observable mineralogy of the specimen

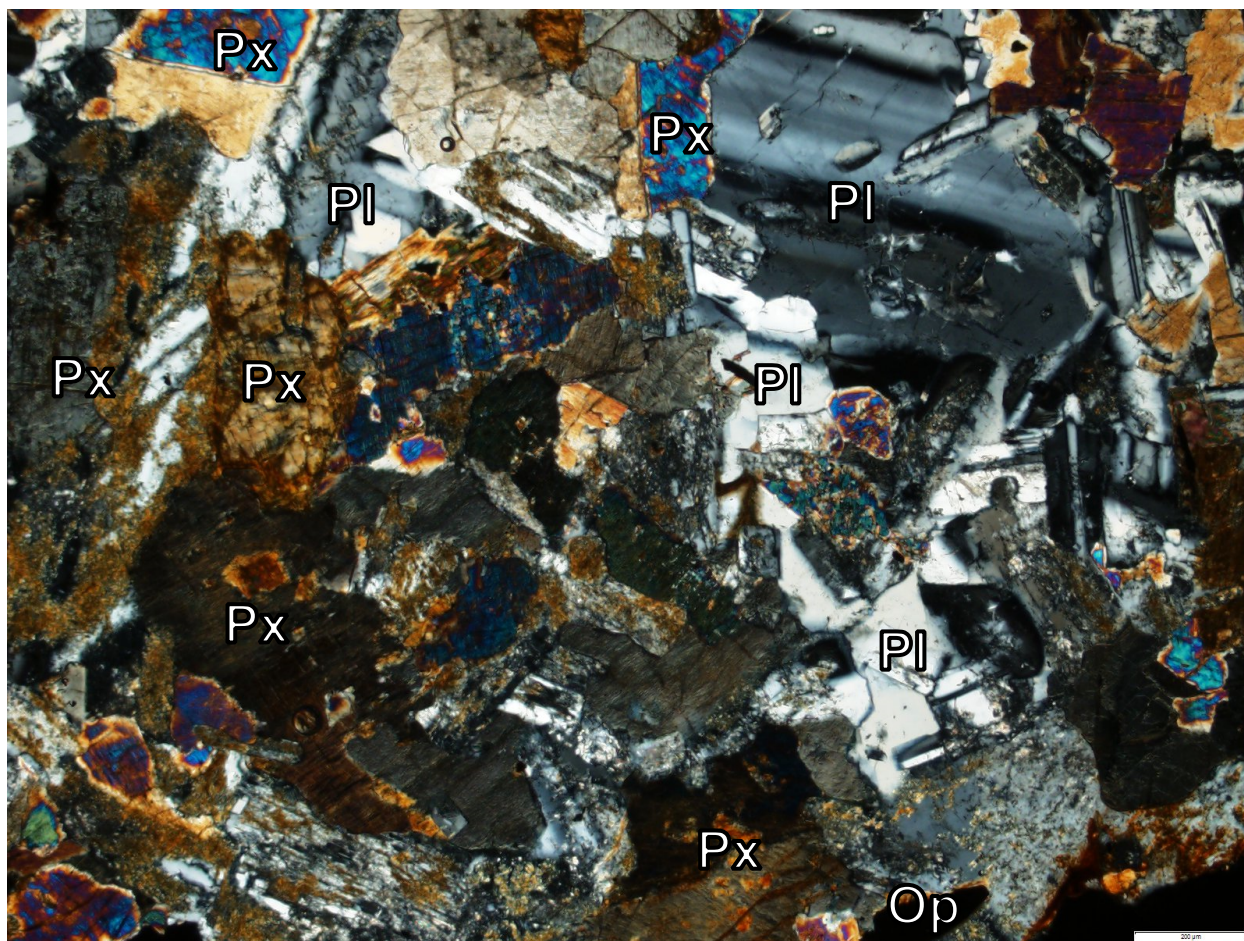
Sample Preparation

A small sample was cut from the larger specimen and trimmed into two approximately 30 x 45 mm billets. One billet is representative of the interior of the specimen and the other is representative of the crust described earlier. This billet was ground, affixed to a glass slide, and ground again to a thickness of 30 μm .

Identification and Description

Based on the initial observations made on the specimens and analysis of the representative thin sections, the stone was determined to be diabase. Diabase is a fine grained mafic intrusive igneous rock commonly deposited in dikes or sills near the surface of the earth. Diabase may also commonly be referred to as dolerite or microgabbro. The primary mineralogy is plagioclase (Pl, labradorite, An₅₅) and pyroxene (Px), with a small (< 5%) amount of opaque minerals (Op). The opaque minerals are too fine grained to be identified. Pyroxene and small amounts of plagioclase present at or near the exposed surface of the stone, particularly where the brown crust described earlier is present, are beginning to alter to clay. This alteration is commonly referred to as a weathering rind, which is chemical alteration of the outermost portion of stone due to weathering. This alteration has a maximum observed depth of ≤ 5 mm and is occurring primarily in the pyroxene, which is more atmospherically unstable than plagioclase. This material has a Skid Resistance Rating (SRL) of H according to the provided specification of skid ratings.





Photomicrograph of mineral composition in the weathering rind, XPL 50 X magnification

*Andrew J. Gutshall, P.G.
May 29, 2020*

ATTACHMENT 3

HIGHWALL MAPPING VEIN ASSESSMENT TABLE

Highwall Mapping Vein Assessment Table
Hanson Aggregates Pennsylvania, LLC Rock Hill Quarry

Vein	Vein Width (inches)	Vein Width ¹ (feet)	Description
1	6	0.50	weathered zone, no veins visible
2	0.25	0.02	whitish, thin coating on diabase surface
3	18	1.50	weathered fault/shear zone with white and green minerals noted
4	8	0.67	weathered zone with whitish veining
5	0.25	0.02	whitish, very thin coating
6	0.5	0.04	white haloed, dark colored vein
7	8	0.67	weathered Actinolite vein
8	0.5	0.04	white haloed, dark colored vein
9	4	0.33	banded white/green vein
10	1	0.08	white vein
11	0.5	0.04	white vein
12	12	1.00	weathered vein with white veins
13	8	0.67	weathered vein with white vein

Total Vein Width 67.0 5.58

¹The maximum observed vein width was utilized where vein width varied. A value of 1/2 the minimum reported vein width of 0.5 inches was utilized for coatings.

Vein Type	Total Vein Width (feet)	Percentage of Veins in Exposed Highwall ²	Comments
All Veins	5.58	1.60%	All veins inclusive of weathered zones and non-actinolite veins
Actinolite Veins	0.667	0.19%	Vein #7 was the only vein with Actinolite identified in hand sample

²Percentage of calculated as footage of veins ÷ 350 feet of exposed highwall x 100.

*Andrew J. Gutshall, P.G.
May 29, 2020*

ATTACHMENT 4

REVISED QGSR TABLES

Table 5
Summary of Asbestos Testing in Rock Core Borings
Hanson Aggregates Pennsylvania, LLC Rock Hill Quarry

Core	Total Drilled Length of Core feet	<u>Actinolite Vein Volume</u> Total Core Volume	Asbestos Concentration PLM
CB-1	91	1.09%	0.0027%
CB-2	90	0.32%	0.00028%
CB-3	160.5	0.18%	0.000091%
CB-4	160	0.13%	0.000063%
Arithmetic Mean		0.43%	0.00078%
Geometric Mean		0.30%	0.00026%

Notes: 1) See individual core vein summary tables in Appendix E for details on each core boring.

2) Asbestos Concentration calculated as: $\frac{\sum (\text{Volume of Actinolite Vein (ft}^3) \times \text{Percent Asbestos by PLM)}}{\text{Total Volume of Core (ft}^3)}$

3) Value of 1/2 the detection limit was used for non-detects.

Core CB-1 Vein Calculation								Percent Asbestos via PLM**
Depth of Vein	Thickness (<i>T</i>)	Length across Core (<i>L</i>)	Width (<i>W</i>)	Vein Angle	Vein Volume	*Lithologic Description	Comments	
(ft)	(inches)	(inches)	(inches)	(Degree)	(ft3)			
19.5-20.9	2.63	6.00	2.00	55	0.0143172	Plagioclase, Chlorite, Albite, Actinolite	CB-1#1 at 19.9'	0.20%
74-79.6	0.06	65.00	2.00	88	0.0036929	Hornblende, Pyroxene	DB-1 at 78.0'	0.60%
90.4-90.8	2.50	3.25	2.00	50	0.0073858	Actinolite	CB-1#3 at 90.4'	ND
Total					0.1445635	1.09% Actinolite Veining		0.0027%

Notes:

*Lithology and total depth (*D*) of corehole was obtained from May 2019 boring logs in Appendix G.

**Percent asbestos as determined by RJ Lee Group by PLM US EPA 600/R-93/116

ND: Not Detected; Percent asbestos calculations utilized one half of the detection limit or 0.05%

Total Core Volume = $V = \pi r^2 D$, where *r* is 1/2*W* and *D* is total depth of core hole = 1.985310347 ft^3

Core CB-2 Vein Calculation								Percent Asbestos
Depth of Vein	Thickness (T)	Length across Core (L)	Width (W)	Vein Angle	Vein Volume	*Lithologic Description	Comments	via PLM**
(ft)	(inches)	(inches)	(inches)	(Degree)	(ft3)			
84.3-84.5	0.50	4.00	2.00	60	0.0018181	Plagioclase, Mica, Actinolite	CB-2#4 at 84.3	ND
85.0-86.3	1.00	5.00	2.00	60	0.0045451	Plagioclase, Mica, Quartz, Actinolite	CB-2#5 at 85.2', CB-2#6 at 86.0'	ND, 0.10%
Total					0.0253796	0.32% Actinolite Veining		0.00028%

Notes:

*Lithology and total depth (*D*) of corehole was obtained from May 2019 boring logs in Appendix G.

**Percent asbestos as determined by RJ Lee Group by PLM US EPA 600/R-93/116

ND: Not Detected; Percent asbestos calculations utilized one half of the detection limit or 0.05%

Total Core Volume = $V = \pi r^2 D$, where *r* is 1/2*W* and *D* is total depth of core hole = 1.96349375 ft³

Core CB-3 Vein Calculation								Percent Asbestos via PLM**
Depth of Vein	Thickness (<i>T</i>)	Length across Core (<i>L</i>)	Width (<i>W</i>)	Vein Angle	Vein Volume	*Lithologic Description	Comments	
(ft)	(inches)	(inches)	(inches)	(Degree)	(ft3)			
18.0-18.2	0.13	2.13	2.00	50	0.0002415	Plagioclase	CB-3#7 at 17.8'	ND
36.4	0.13	2.25	2.00	40	0.0002557	Quartz	DB-3 at 30.5'	ND
146.2-146.8	2.00	3.50	2.00	55	0.0063632	Quartz, Actinolite, Augite	CB-3#8 at 146.2'	ND
Total					0.0171472	0.18% Actinolite Veining		0.000091%

Notes:

*Lithology and total depth (*D*) of corehole was obtained from May 2019 boring logs in Appendix G.

**Percent asbestos as determined by RJ Lee Group by PLM US EPA 600/R-93/116

ND: Not Detected; Percent asbestos calculations utilized one half of the detection limit or 0.05%

Total Core Volume = $V = \pi r^2 D$, where *r* is 1/2*W* and *D* is total depth of core hole = 3.501563854 ft^3

Core CB-4 Vein Calculation								Percent Asbestos
Depth of Vein	Thickness (<i>T</i>)	Length across Core (<i>L</i>)	Width (<i>W</i>)	Vein Angle	Vein Volume	*Lithologic Description	Comments	via PLM**
(ft)	(inches)	(inches)	(inches)	(Degree)	(ft3)			
70.3	0.13	2.63	2.00	50	0.0002983	Quartz	DB-4 at 69.0'	ND
147.5-148.0	0.88	5.50	2.00	75	0.0043747	Plagioclase, Actinolite, Chlorite	CB-4#10 at 147.5'	ND
Total					0.0207371	0.13% Actinolite Veining		0.000063%

Notes:

*Lithology and total depth (*D*) of corehole was obtained from May 2019 boring logs in Appendix G.

**Percent asbestos as determined by RJ Lee Group by PLM US EPA 600/R-93/116

ND: Not Detected; Percent asbestos calculations utilized one half of the detection limit or 0.05%

Total Core Volume = $V = \pi r^2 D$, where *r* is 1/2*W* and *D* is total depth of core hole = 3.490655556 ft³

RJ Lee Group Response to Comments Letter

May 29, 2020

May 29, 2020

Andrew Gutshall
Hanson Aggregates Pennsylvania, LLC.
7660 Imperial Way
Allentown, PA 18195
RE: Hanson Project
RJ Lee Group Project Number: LLH901997

Mr. Gutshall,

The RJ Lee Group has drafted responses to the several comments from the PADEP regarding the 2019 QGSR report. These responses are provided below:

- Why RJ Lee Group, Inc. “modified” EPA Method 100.1 to count the 5µm length fibers as opposed to the protocol of 0.5 µm length as specified in EPA Method 100.1.
 - Accepted risk models (see EPA IRIS at https://cfpub.epa.gov/ncea/iris2/chemicalLanding.cfm?substance_nmbr=371) utilize data derived from epidemiology studies that measured airborne asbestos concentrations using the size parameters of length, width, and aspect ratio. The fiber dimensions used to determine the risk for asbestos-related diseases are those airborne asbestos fibers that are 5 µm or longer and aspect ratio >3:1. The method was modified to include only fibers longer than 5 µm in order to provide some comparison to assess the risk presented if those fibers were to become airborne.
- Why RJ Lee Group, Inc. did not use the same parameters (length, etc.) as EMSL used in its analysis and the impact that would have on the reported results.
 - For the analysis of the water samples, the length parameter used by RJLG and EMSL was >5 µm and >0.5 µm, respectively. The concern with the water samples is that fibers in the water would become suspended in the air after drying. Therefore, RJLG used a size of 5 µm which is consistent with the toxicological and epidemiological studies referenced above that studied risks associated with airborne fibers.
- Why would the subsequent investigation of the surface water (per the QGSSP) begun on April 25, 2019 be performed to apparently less stringent parameters than the Previous Site Investigation?
 - It is not necessarily that the water testing begun April 29, 2019 is less stringent, but rather it has been modified to focus on fibers of lengths that present risk when airborne and would be included in air monitoring testing if they were to become airborne.
- Please explain why the 1.0% limit is the correct standard to use for Naturally Occurring Asbestos?

- Currently, there is no relevant federal or Pennsylvania standard for natural occurrences of asbestos. However, federal standards (OSHA, MSHA, and EPA) do exist for bulk substances containing 1% or more asbestos by weight being designated as an asbestos containing material (ACM). Since this is a federal standard, each state is required to use it or a lower one if it exists. California has set 0.25% as a limit for asbestos specifically in the case of aggregate produced and sold from serpentinite, mafic, and ultramafic sources. As there is no scientific consensus on a “correct” limit for “Naturally Occurring Asbestos”, to utilize the 1.0% limit in this case is most consistent with the federal standards applied across the country. While 1% is the limit for the definition of asbestos containing materials, we have reported, and will continue to report, quantities of asbestos well below 1% in the materials analyzed. If the presence of asbestos below 1% is significant for the management of this resource, that information is available.
- Please explain why PLM was chosen as the approach for determining the amount of NOA at the Rock Hill Quarry?
 - The approach utilized includes multiple macro and micro observation tools for the identification and quantification of NOA in the samples from Rock Hill Quarry and does not rely on any single technique. The analysis begins with stereobinocular macro to micro scale observation of the as received materials, prior to grinding, to determine if coarse fibrous features are present. In addition, both PLM and TEM have been utilized for the identification and quantification of NOA in a subset of samples. Where no NOA was detected by PLM, TEM was used to confirm that finding. In every instance where TEM observed NOA and PLM did not, and the amount observed by TEM was well below the detection limit for the PLM. None of the TEM results quantified NOA greater than what was observed by PLM. The determination of the asbestos content in bulk materials is most accurately made using PLM and is the primary technique implemented in EPA 600/R-93/116 as well as CARB 435. TEM has been utilized in these samples in order to provide a thorough characterization of the minerals observed, as well as to provide an assessment of particles that may not be visible in the PLM analysis. The majority of the mass of asbestos is included in particles visible in the PLM and these larger particles are likely to be excluded from the TEM analysis due to the extremely small mass of material analyzed as well as the very small area of the prepared sample filter that is analyzed by TEM. Because of the nature of the bulk materials being analyzed, PLM is used as the primary quantifying analytical technique, but can be supplemented by TEM for further characterization and confirmation of mineral identifications.
- Regulations have been cited in the QGSR as sources for a definition of Asbestos Containing Material which has been also used to define a material containing Naturally Occurring Asbestos. These regulations also prescribe methods of analysis. Please explain why an analysis method (EPA 600/R-93/116) that counts as asbestos those fibers with an aspect ratio of >20:1 may be preferable to the methods cited in the regulations that count fibers as asbestos that have an aspect ratio of 3:1 or 5:1?
 - EPA 600/R-93/116 is not a method to enumerate and count individual asbestos fibers and does not define a fiber based on strict dimensional parameters. EPA 600/R-93/116 is the method recommended by EPA for the analysis of bulk materials to determine the

asbestos content of those types of materials. The method was recommended in Federal Register, 59, p. 38970-38971 as an improvement upon the promulgated method (40 CFR §763 Appendix E to Subpart E of Part 763) and also contains a definition for “asbestiform” that was lacking in the promulgated method. The definition of asbestiform is significant, as it is recognized that non-asbestiform particles of amphibole, and other minerals, pose a significant interference in the accurate determination of the amount of asbestos present in a material analyzed by the method. There are no specific criteria for aspect ratio in the definition of a fiber in EPA 600/R-93/116. Aspect ratio is only one characteristic of an asbestiform material that is useful for the determination of whether or not it is asbestos. The other characteristics listed in the asbestiform definition need to be considered when the possibility exists that non-asbestiform materials may be present in the material being analyzed. This is exactly the case in any situation where naturally occurring asbestos may be of concern.

- Why would EPA Method 600/R-93/116 be used for analysis if it was not promulgated by the EPA, particularly if the regulations cited for the definition of Asbestos Containing Materials contain methods of analysis?
 - The promulgated methods are presumptive tests used in environments where asbestos was known to have existed in the form of asbestos containing building materials and were the result of negotiated rulemaking by the EPA. While EPA 600/R-93/116 is also designed to analyze building materials, it is more broadly defined, improved over the promulgated methods, and capable of capturing the characteristics necessary of a material to determine if it is in fact asbestiform or not. The promulgated methods are very narrowly defined and do not take into consideration the unique situation that exists with natural occurrences of asbestos where, if an asbestos mineral is present, it will very likely exist with non-asbestiform particles or other interfering minerals. Because of this, a more robust approach is needed to accurately determine if, and how much, asbestos might be present in a material. EPA 600/R-93/116 also provides guidance on how to utilize TEM for the analysis of bulk materials that is not present in the promulgated method. This is crucial for the identification of asbestos in naturally occurring situations as it provides a means to verify mineral identifications made by PLM. In addition, it is the clearer asbestiform definition and expanded implementation of TEM that make EPA 600/R-93/116 the preferable method for the analysis of naturally occurring asbestos over the promulgated method. Other regulated methods, CARB 435, refer to EPA 600/R-93/116 as an acceptable method for the analysis of asbestos in rocks.
- Why EPA Method 600/R-93/116 is appropriate for analyzing for Naturally Occurring Asbestos when Federal Register, Volume 59, Number 146 (Monday, August 1, 1994) which introduced the method states the “approach should be considered” for use in analyzing (1) “Floor tiles...” and (2) “...hard wall and acoustical plaster, stucco or other similar multi-layered materials...”
 - The two statements outlined above denote how the EPA 600/R-93/116 method is significantly improved over the promulgated method in those specific types of materials. These two specific materials present unique analytical challenges that were recognized after the promulgated method was implemented. Because of the narrow definition of the promulgated method, it was necessary to provide an improved method

(600/R-93/116) to accurately analyze those types of materials, but the improvements were not limited to just floor tile and layered systems. It is also important to note that the EPA 600/R-93/116 contains a clearer definition of asbestiform and is an improvement over the promulgated method for accurately determining the presence of asbestos in a material. EPA 600/R-93/116 also provides guidance on how to utilize TEM for the analysis of bulk materials that is not present in the promulgated method. Other regulated methods for the analysis of asbestos in rocks, e.g. CARB 435, refer to EPA 600/R-93/116.

- By inspection of the submitted data for aggregate samples in the QGSR, it appears that a 10:1 aspect ratio was the governing parameter in determining if a structure was asbestos or a cleavage fragment as there is only one instance found that shows a 10:1 aspect ratio that was deemed asbestos. All other structures at 10:1 or less aspect ratios were deemed to be cleavage fragments or non-asbestos. Please explain why this is a logical assumption and consistent with the regulatory definitions cited in the QGSR. Secondly, if one was using EPA 600/R-93/116, which states an aspect ratio of 20:1 should be used, why was a 10:1 ratio apparently used?
 - It appears there may be a misunderstanding of the analytical parameters and definition of asbestiform provided in EPA 600/R-93/116. As discussed above, the definition of fiber in EPA 600/R-93/116 has no specific dimensional parameters, and the definition for asbestiform is for the description of the population of fibers that might be observed in a material. The TEM analysis of bulk materials was performed with the following criteria for enumerating fibers of any kind. Fibers observed in the TEM analysis of bulk samples are counted based on their morphological parameters: length ≥ 0.5 micrometers, aspect ratio $\geq 5:1$ (per ASTM D-5756). When a fiber is observed, the mineral composition of that fiber is identified as either chrysotile, amphibole, or non-asbestos using selected area electron diffraction and energy dispersive x-ray spectroscopy. The information gathered from the fiber is compared to reference materials for the purpose of mineral identification. Of the amphibole particles identified, a determination of whether or not a fiber is asbestiform is made based on the asbestiform definition provided in EPA 600/R-93/116. If an amphibole fiber is recognized as asbestiform it is counted as amphibole asbestos. If an amphibole fiber is recognized as non-asbestiform it is counted as amphibole cleavage.

The observation that asbestos fibers have aspect ratios generally greater than 10:1 is consistent with the asbestiform definition in EPA 600/R-93/116:

Asbestiform (morphology) Said of a mineral that is like asbestos, i.e., crystallized with the habit of asbestos. Some asbestiform minerals may lack the properties which make asbestos commercially valuable, such as long fiber length and high tensile strength. With the light microscope, the asbestiform habit is generally recognized by the following characteristics:

- Mean aspect ratios ranging from 20:1 to 100:1 or higher for fibers longer than 5 μ m. Aspect ratios should be determined for fibers, not bundles.
- Very thin fibrils, usually less than 0.5 micrometers in width, and
- Two or more of the following:
 - Parallel fibers occurring in bundles,
 - Fiber bundles displaying splayed ends,
 - Matted masses of individual fibers, and/or
 - Fibers showing curvature

These characteristics refer to the population of fibers as observed in a bulk sample. It is not unusual to observe occasional particles having aspect ratios of 10:1 or less, but it is unlikely that the asbestos component(s) would be dominated by particles (individual fibers) having aspect ratios of <20:1 for fibers longer than 5 μ m. If a sample contains a fibrous component of which most of the fibers have aspect ratios of <20:1 and that do not display the additional asbestiform characteristics, by definition the component should not be considered asbestos.

The observation that many of the counted structures had aspect ratios <10:1 indicates that the analyzed material is not entirely asbestiform, but there are fibers present that have much higher aspect ratios and lengths longer than 5 μ m indicating there is a trace amount of asbestiform material present as well. This is not unusual for a naturally occurring asbestos material, as typically a range of morphologies can be present and intimately intermixed. The data collected indicate that the material present at Rock Hill is predominantly non-asbestiform, however trace amounts of asbestiform material were observed and reported. If fibers with aspect ratio between 5:1 and 10:1 were observed to possess asbestiform characteristics they too would have been reported as asbestos.

- Please explain the significance, in terms of protecting human health and safety, of the presence of asbestos fibers in water at the site. Citing drinking water standards seems irrelevant since none of the onsite water is proposed for human consumption.
 - The task was to evaluate water samples collected on site for the presence of asbestos and it is beyond the scope of our work to determine what impact that might have on human health and safety. In the instance that the water available on site is to be used for dust mitigation, it is useful to understand if asbestos is present in that water as there could be potential to disperse asbestos, if present in the water, over the site which then could become entrained as dust after drying. There is no limit of asbestos in water that the EPA 100.1 result can be compared against. EPA method 100.2 is an official EPA method that measures the presence of asbestos (10 μ m and longer) in water that will be used for human drinking. The EPA drinking water limit for these fibers is 7 million fibers per liter of water. The water at Rock Hill quarry was never intended to be used for

drinking water, however, if it were, it would meet this EPA drinking water quality standard. The surface water at the Rock Hill quarry is intended to be used for dust suppression applications. In government (OSHA and EPA) asbestos risk assessments derived from toxicological and epidemiological studies, the fiber dimensions used to determine the risk for asbestos-related diseases were those airborne asbestos fibers that were 5 µm or longer, with a 3:1 or greater aspect ratio as seen by a phase contrast optical microscope at 400X. This microscope only sees fibers that are 0.25 µm wide or wider. For this reason, the water samples at Rock Hill quarry were additionally analyzed for these risk fiber dimensions so that an assessment could be made on whether the dust suppression water could contribute to possible airborne levels of these risk fibers.

- Asbestos concentrations from the samples collected by the Department are in some cases different than those collected by EarthRes. In light of the comments above noting apparent discrepancies between asbestos definitions and laboratory reporting, please reevaluate the asbestos concentrations as appropriate. (Core Vein Volume Assessment Section 6.2)
 - The direct comparison between laboratories can only be made on the samples where both laboratories utilized the same analytical techniques. In the group of samples submitted to EMSL by PADEP, only PLM results are available from RJLG. In all of these samples, with the exception of the Vein #7 sample, the results are identical for asbestos. For non-asbestos fibers, EMSL appears to have visually estimated the quantity and as a result produces an overestimate when compared to the point counting results obtained by RJLG for non-asbestos fibers. TEM analysis of bulk materials for asbestos is known to have significant limitations and produce results that are of limited precision and accuracy.
 - Given the disparate results for the Vein #7 sample between laboratories, a second aliquot of the sample was tested by PLM using EPA 600/R-93/116 with quantification by a 1000-point count and found to contain 0.2% actinolite asbestos and 47.7% non-asbestiform amphibole. While still significantly different than the EMSL result, the magnitude of this result is comparable, and indicates one of the challenges associated with analysis of geological materials.

- The simplest way to resolve asbestiform definitional discrepancies, it appears in this instance, is by simple addition of the amphibole asbestos and amphibole cleavage fragment wt% results reported by EarthRes for any given sample. This is possible because RJLG has included all particles that meet the aspect ratio criteria for fibers identified in the TEM analyses. Where another laboratory has not differentiated asbestiform from non-asbestiform morphologies and included both as “asbestos” using the same counting criteria the “asbestos” wt% determined is the sum of both morphologies.

If you have any questions, please do not hesitate to contact me directly.

Sincerely,

A handwritten signature in black ink, appearing to read 'Bryan Bandli', with a stylized flourish at the end.

Bryan Bandli, Ph.D.
Principal Investigator
bbandli@rjleegroup.com

Laboratory Report

K & L Gates
17 North Second Street
Harrisburg, PA 17101
United States
Attention: David Raphael
Telephone: 717-231-4504

Report Date 05/26/2020
Sample Receipt Date 05/21/2020
RJ Lee Group Job No. LLH901997-21
Authorization/P.O. No.
Client Job No./Name

Analysis: Asbestos in Bulk Samples by Point Count
Method: EPA/600/R-93/116

RJLG Sample Number	Client Sample Number	Homogeneous	# of Layers	Asbestos Detected(%)	Non-Asbestos Fibers(%)	Non-Fibrous Materials(%)	Matrix Material	Analyst - Analysis Date
3158813.HPL	#3 - Vein 7	Yes	1	0.20 AC	47.7 OF	52.10	CA,OP, M	EAF-05/22/2020
Description:	Gray Powder 1000 Point Count. Detection Limit of 0.1%.							
Weight Loss: 0.0%	0.2% Asbestiform Actinolite and 47.7% Non-Asbestiform Actinolite observed.							

Client Job No./Name:

RJ Lee Group Job No: LLH901997-21

RJLG Sample Number	Client Sample Number	Homogeneous	# of Layers	Asbestos Detected(%)	Non-Asbestos Fibers(%)	Non-Fibrous Materials(%)	Matrix Material	Analyst - Analysis Date
-----------------------	-------------------------	-------------	-------------	-------------------------	---------------------------	-----------------------------	--------------------	----------------------------



Authorized Signature:

Elizabeth Fischer

ASBESTOS

AM = Amosite
 AC = Actinolite
 AN = Anthophyllite
 CH = Chrysotile
 CR = Crocidolite
 TR = Tremolite

NON-ASBESTOS

CE = Cellulose
 MW = Mineral Wool
 FG = Fibrous Glass
 SF = Synthetic Fibers
 H = Hair
 W = Wollastonite
 OF = Other Fibers

NON-FIBROUS MATERIALS

AM = Amphibole
 B = Binder
 CA = Carbonates
 CL = Clay
 F = Feldspar
 G = Gypsum
 HY = Hydromagnesite
 M = Miscellaneous
 MI = Mica
 OP = Opaque
 OR = Organic
 P = Perlite
 Q = Quartz
 T = Tar
 V = Vermiculite

DISCLAIMER NOTES

- "ND" indicates no asbestos was detected; the method detection limit is 0.1%.
- "Trace" or "<" indicates asbestos was identified in the sample, but the concentration is less than the method quantitation limit. PLM coefficients of variance range from approximately 1.8 at the quantitation limit of 0.25% to 0.32 at high fiber concentrations.
- Samples are archived for three months following analysis and are then properly discarded.
- These results are submitted pursuant to RJ Lee Group's current terms and conditions of sale, including the company's standard warranty and limitation of liability provisions. No responsibility or liability is assumed for the manner in which these results are used or interpreted.
- This test report relates to the items tested.
- This report is not valid unless it bears the name of a NVLAP Lab Code 101208-0 approved signatory.
- Any reproduction of this document must be in full in order for the report to be valid.
- This report may not be used to claim product endorsement by NVLAP Lab Code 101208-0, any agency of the U.S. Government or any other laboratory accrediting agency.
- Polarized-light microscopy is not consistently reliable in detecting asbestos in floor coverings and similar nonfriable organically bound materials. Quantitative transmission electron microscopy is currently the only method that can be used to determine if this material can be considered or treated as "non-asbestos-containing."
- Sample(s) for this project were analyzed at our: Monroeville, PA (AIHA LAP, LLC. #100364, NY ELAP #10884) facility.
- If RJ Lee Group, Inc. did not collect the samples analyzed, the verifiability of the laboratorys results are limited to the reported values.

2020 Revegetation Plan

**Rock Hill Quarry
SMP No. 7974SM1
May 29, 2019**

As requested by the Department, Hanson is proposing to revegetate disturbed areas at the site that are capable of supporting plant growth. In accordance with information previously submitted to the Department in Module 23: Revegetation, Hanson will collect one (1) composite soil sample to submit for agronomic crop fertility testing. The sample will be sent to the Penn State Extension Agricultural Analytical Services Laboratory in University Park, PA for analysis. The laboratory results will include a fertility report describing lime and fertilizer recommendations.

Hanson will then conduct hydroseeding of approximately six (6) acres utilizing a perennial ryegrass seed mixture to support stabilization on these areas. Please note that fertilizers, soil conditioners and seed may be applied at the same time.

The disturbed areas proposed for stabilization are outlined on the 2020 Revegetation Plan drawing (attached). The five (5) areas consists of approximately six (6) acres. The remaining portions of the surface mine permit area are either undisturbed or have been previously stabilized with crushed stone or previous hydroseeding.



LEGEND



PERMIT BOUNDARY



REVEGETATION AREA



Hanson
HEIDELBERGCEMENT Group

DRAWN BY:	AJG
DATE:	05/20/2020
SCALE:	1" = 500'
0 250 500 SCALE IN FEET	

TITLE:	<u>2020 REVEGETATION PLAN</u>
LOCATION:	ROCK HILL QUARRY SMP # 7974SM1 EAST ROCKHILL TWP., BUCKS CO., PA
SOURCE:	04/23/2020 KESPRY DRONE ORTHOPHOTOGRAPH BACKGROUND IMAGE IS FROM GOOGLE EARTH PRO (06/15/2018)

Draft Air Monitoring Plan

Rock Hill Quarry SMP No. 7974SM1 May 29, 2019

As requested by the Department, Hanson has prepared the following Draft Air Monitoring Plan (hereafter referred to as Plan) that incorporates monitoring for potential airborne asbestos fibers during periods of limited activity at the quarry as well as during inactivity. The scope of this Plan includes those activities Hanson, or its subcontractors, perform at the site. The limited activities are described below along with task-based air sampling protocols.

During the pending limited activity period at the Rock Hill Quarry, the following site tasks must be conducted:

Monthly NPDES water sample collection

Hanson personnel or a subcontractor will access the site at least once per month to collect water samples from the NPDES discharge point(s). It is possible that additional trips in a month would be necessary to collect follow-up water samples. Since the site visit duration, typically by only one person, would last less than 30 minutes, Hanson does not propose air monitoring to be conducted during these events. Hanson or its subcontractor will obey the posted speed limit of 15 miles per hour (mph) intended to prevent fugitive dust emissions that could potentially be generated by the passenger vehicle driving to the sample points.

Routine site inspection conducted via drive through;

At any time, Hanson or a subcontractor may access the site to conduct routine inspections for evidence of trespassing, site vandalism or regular checks on the stormwater management controls. Since the site visit duration would typically last less than 30 minutes, Hanson does not propose air monitoring to be conducted during these events. Hanson or its subcontractor will obey the posted speed limit of 15 mph intended to prevent fugitive dust emissions that could potentially be generated by the passenger vehicle.

Annual removal of 500 tons of crushed aggregate from existing stockpiles; and

As required by the Department, Hanson will conduct removal of a minimum 500 tons of previously crushed aggregate material on an annual basis. The event will likely last one (1) day; however, some equipment (e.g., loader, etc.) may be transported to the site ahead of the loadout event. As such, all vehicle movement at the site will obey the posted speed limit of 15 mph intended to prevent fugitive dust emissions.

During the day of the 500 ton loadout event, Hanson will conduct air monitoring to assess airborne particulate for the potential presence of naturally occurring asbestos (NOA). The air monitoring will be conducted at two (2) upwind and three (3) downwind locations relative to the aggregate loading operation and be located at or near the perimeter of the disturbed area of the Surface Mine Permit boundary (or as close as possible). The sample collection will follow the attached protocol titled Naturally Occurring Asbestos (NOA) Perimeter Monitoring Practices (dated May 29, 2020).

A letter report will be prepared and submitted to the Department no later than 30 days after the receipt of the analytical data from the laboratory. The report will include a description of the sample methodology, weather conditions, pump serial numbers, initial/final flow rates, sample numbers, sample locations, sample start/end times, laboratory analysis results, and recommendations (if necessary).

Non-scheduled site maintenance (e.g., ponds, roads, etc.)

At any time, Hanson or a subcontractor may access the site to conduct maintenance to stormwater ponds or site roads. Should the nature of the work disturb any dry aggregate or earthen material and last more than four (4) hours, the air monitor sampling methodology presented above will be utilized to monitor the site activity. If the maintenance event is to last less than four (4) hours, water sprays will be used to prevent fugitive dust emissions that could be generated by the maintenance activities. Natural precipitation should also be considered sufficient to prevent fugitive dust emissions. Hanson or its subcontractor will also obey posted speed limit of 15 mph intended to prevent additional fugitive dust emissions from moving vehicles.

Idle Site Background Conditions Monitoring

To address the Department's concerns regarding potential exposure to airborne NOA at the site, Hanson proposes to conduct a one-time background air monitoring event. This air monitoring event will last a total of two (2) days and consist of four (4) samples per day with two (2) upwind and three (3) downwind locations relative to the site permit boundary. The sample collection will follow the attached protocol titled Naturally Occurring Asbestos (NOA) Perimeter Monitoring Practices (dated May 29, 2020). The background sampling will be completed prior to the first 500-ton loadout event.

Hanson Aggregates Pennsylvania LLC



Effective Date XX/XX/XXXX

Rev XX

Naturally Occurring Asbestos (NOA) Perimeter Monitoring Practices ROCK HILL QUARRY

Approver			xx/xx/xxxx	
	Name	Signature	Date	
AMENDMENT HISTORY				
Rev No	Date	Description of Amendments	Approver	Sections
00	xx/xx/xxxx	Initial Document		N/A

1. Purpose

The purpose of this document is to outline practices for periodically assessing potential naturally occurring asbestos (NOA) fibers in air at the perimeter of the Rock Hill Quarry, in East Rockhill Township, Bucks County, Pennsylvania. This document defines air sampling and analysis techniques to be implemented through certified and licensed third-party specialists.

2. Scope

The scope of this document is limited to the Rock Hill Quarry operations. It is not applicable to assessments conducted beyond the facility's perimeter. The focus of this assessment is to establish a plan for assessing the potential presence of NOA fibers in air at the perimeter of the Rock Hill Quarry.

3. Definitions

Asbestos – A generic term for several asbestiform hydrated silicates. The term asbestos is limited to the following mineral fibers: chrysotile, amosite, crocidolite, anthophyllite asbestos, tremolite asbestos, and actinolite asbestos.

Asbestos Fiber – A particle form of asbestos longer than 5 micrometers (μm) with a length-to-diameter ratio of at least 3:1.

Phase Contrast Microscopy (PCM) – Analysis counts fibers that are present on filters in order to give a time-weighted average of the concentration of those fibers for the volume of air sampled. PCM results are fiber concentrations and do not distinguish between asbestos and non-asbestos fibers.

Transmission Electron Microscopy (TEM) - Analysis identifies specific asbestos fibers in air (and bulk) samples. TEM is capable of analyzing samples at high magnification (20,000X and higher) and identifies asbestos fibers by morphology, crystalline structure and elemental analysis.

4. Naturally Occurring Asbestos (NOA) Air Sampling Practices

4.1. Number and Locations for Air Sampling

For each sampling event, a minimum of five (5) samples will be collected:

- Two (2) upwind; and
- Three (3) downwind.

General air sampling locations will be selected based upon:

- Site-specific activities ongoing during the sampling period;
- Historic prevailing wind direction; and
- Wind direction and site-specific weather conditions at the time of sampling.

Wind direction and wind speed will be monitored during each sampling event. If wind direction changes materially during a sampling event in any one sampling phase, the time and change in direction will be documented to reflect the change and provide data for analysis and comparison. In addition, if wind direction change is considered extreme during any sampling

event, the sampling location may be adjusted to reflect the change based on judgement of the field sampling technician.

- Wind direction and speed will be measured using a hand-held anemometer and recorded on field sampling data sheets.

In all cases, based upon professional judgment and knowledge of offsite concerns, sampling areas may also be adjusted to provide more representative data and consideration of special conditions. Any change in location will be properly documented to reflect the location, time, and change in wind direction.

4.2. Air Sample Collection

Sampling will be in accordance with the National Institute for Occupational Safety and Health (NIOSH) Manual for Analytical Methods (Method 7400 or Method 7402 for Asbestos and other Fibers). Air samples will be collected from fixed sampling locations with low-flow pumps and each sampling apparatus shall include a cassette that contains a 25-millimeter (mm) diameter Mixed Cellulose Ester (MCE) filter with a pore size of 0.8 or 0.45 micrometers (um). Samples shall be:

- Set to operate at approximately three (3) to four (4) liters per minute (lpm);
- Placed approximately five (5) feet above the ground surface (e.g. designed to approximate the breathing area of a worker or passerby to assess exposure);
- Calibrated prior to and following each sampling event using a cassette reserved for calibration (from the same lot of sample cassettes to be used for sample collection); and
- Sampled for durations lengthy enough to assure an adequate sample volume to achieve the desired laboratory reporting limits.

(Note: the site is a remote location where power for high volume sampling pumps is not available.)

Attachment A includes the field sampling data sheet and log sheet forms that will be used to document sampling activities.

4.3. Air Sample Analysis

Laboratory analysis of the collected samples will be conducted via a certified and licensed third-party. Analysis will be in accordance with the NIOSH Manual for Analytical Methods (Method 7400 or Method 7402 for Asbestos and other Fibers).

- Method 7400 Phase Contrast Microscopy (PCM): PCM will be used to initially analyze all samples. (See Attachment B – Method 7400.)
- Method 7402 Transmission Electron Microscopy (TEM): When PCM results indicate a potential exposure exceeding the 0.01 f/cc action level, samples will be further analyzed using TEM to identify asbestos fibers. (See Attachment C – Method 7402.)
 - Action level based upon the US Environmental Protection Agency (EPA)'s clearance standard for asbestos clean-up.

4.4. Third-Party Requirements

Third-party personnel collecting samples will be both certified for asbestos work and licensed according to applicable State of Pennsylvania licensing requirements, as applicable.

Third party laboratories conducting analysis shall be accredited through a recognized accreditation body, such as the American Industrial Hygiene Association (AIHA) or the National Voluntary Laboratory Accreditation Program (NVLAP), or a similar accreditation body.

4.5. Reports and Records

Exposure monitoring results will be formally documented within a summary report. Reports are to be maintained for a period of no less than five (5) years past the sampling date.

5. Responsibilities

Quarry Management

Quarry Management is responsible for the implementation of the sampling through:

- Providing for the contractual use of third parties conducting the exposure monitoring; and
- Maintaining result summary documentations.

Environment & Sustainability

Environment & Sustainability (E&S) representatives are responsible for supporting management during sampling implementation, coordinating for any necessary Occupational Health support with Corporate, and assisting in result communication.

6. References

1. National Institute of Occupational Health and Safety (NIOSH) Manual for Analytical Methods:
 - 1.1. Method 7400
 - 1.2. Method 7402

DRAFT

ATTACHMENTS

[illegible]



AIR SAMPLING RECORD
Field Sampling Data

Lehigh Hanson
HEIDELBERGCEMENT Group

Date: _____

Facility: _____

Temp: _____ Humidity: _____

Conditions (include wind direction/speed): _____

Sample Area and Description: _____

Activities being performed: _____

SAMPLING DATA

Time Start: _____ Time End: _____ Total Time (min): _____

Flowrate: _____ Volume: _____

Chemicals Monitored: _____

Pump: _____ Pump ID: _____

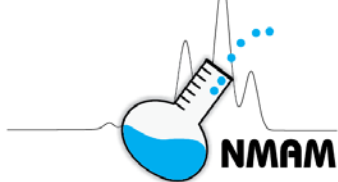
Media: _____ Media ID: _____

Secondary Calibrator / ID: _____

Comments / Unusual or Upset Conditions / Task or Work Activity Considerations:

Performed by: _____

Signature: _____



Formula: Various

MW: Various

CAS: Table 3

RTECS: Various

METHOD: 7400, Issue 3

EVALUATION: FULL

Issue 1: 15 May 1989

Issue 3: 14 June 2019

OSHA: 0.1 asbestos fiber (>5 µm long and ≥3:1 aspect ratio)/cc; 1 f/cc, 30 min excursion; carcinogen

PROPERTIES: solid, fibrous, crystalline, anisotropic

NIOSH: 0.1 fiber (>5 µm long and ≥3:1 aspect ratio)/cc, for a 400 L sample; carcinogen

MSHA: As OSHA

SYNONYMS: actinolite or ferroactinolite; amosite; anthophyllite; chrysotile; crocidolite; tremolite; amphibole asbestos; refractory ceramic fibers; fibrous glass

SAMPLING		MEASUREMENT	
SAMPLER:	FILTER (0. 45- to 1.2-µm mixed cellulose ester membrane, 25-mm; conductive cowl on cassette)	TECHNIQUE:	LIGHT MICROSCOPY, PHASE CONTRAST
FLOW RATE*:	0.5 to 16 L/min	ANALYTE:	fibers (manual count)
VOL-MIN*:	400 L @ 0.1 fiber/cc	SAMPLE PREPARATION:	Treatment of filter by acetone or dimethylformamide (DMF)/acetic acid, followed by triacetin or Euparal mounting medium [2-4]
-MAX*:	(step 4, Sampling)	COUNTING RULES:	Described in previous version of this method as "A" rules [1,5]
	*Adjust to give 100 to 1300 fiber/mm ²	EQUIPMENT:	1. positive phase-contrast microscope; 2. graticule (100-µm field of view); 3. phase-shift test slide
SHIPMENT:	routine (pack to reduce mechanical and static electrical shock) (step 6, Sampling)	CALIBRATION:	Phase-shift test slide
SAMPLE STABILITY:	stable	RANGE:	100 to 1300 fibers/mm ² filter area
BLANKS:	2 to 10 field blanks per set	ESTIMATED LOD:	7 fibers/mm ² filter area
ACCURACY		PRECISION (S_r):	0.10 to 0.12 [1]; see Evaluation of Method
RANGE STUDIED:	80 to 100 fibers counted		
BIAS:	see Evaluation of Method		
OVERALL PRECISION (Ŝ_T):	0.115 to 0.13 [1]		
ACCURACY:	see Evaluation of Method		

APPLICABILITY: The quantitative working range is 0.04 to 0.5 fiber/cc for a 1000-L air sample. The LOD depends on sample volume and quantity of interfering dust, and is <0.01 fiber/cc if free of interferences. The method gives an index of airborne fibers. This method can be used in conjunction with electron microscopy (e.g., Method 7402) for assistance in identification of fibers. For fibers with diameters >1 µm, polarizing light microscopy (as in NIOSH Method 7403) may be used to identify and eliminate interfering non-crystalline fibers [6]. Asbestos fibers thinner than about 0.05-0.15 µm diameter, depending on asbestos type, will not be detected by this method [7-10]. This method may be used for other materials with alternate counting rules.

INTERFERENCES: If the method is used to detect a specific type of fiber, any other fiber may interfere because all particles meeting the counting criteria are counted. Chain-like particles may appear fibrous. High levels of non-fibrous dust particles may obscure fibers in the field of view and increase the detection limit.

OTHER METHODS: This revision replaces Method 7400, issue 2 (dated 08/15/1994).

REAGENTS:

1. Acetone*, reagent grade.
NOTE: Dimethylformamide (DMF)*, reagent grade/glacial acetic acid can be used as an alternative filter clearing reagent.
2. Triacetin (glycerol triacetate), reagent grade.
NOTE: Euparal (synthetic Canada Balsam) can be used as an alternative mounting media.

*See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: Field monitor, 25-mm, 3-piece cassette with 50-mm electrically conductive extension cowl and mixed cellulose ester (MCE) filter, 0.45- to 1.2- μ m pore size, and backup pad.
NOTE 1: Analyze representative filters for fiber background before use to check for clarity and background. Discard the filter lot if mean is ≥ 5 fibers per 100 graticule fields. These are defined as laboratory blanks. Manufacturer-provided quality assurance checks on filter blanks are normally adequate as long as field blanks are analyzed as described below.
NOTE 2: The electrically conductive extension cowl reduces electrostatic effects [11]. Ground the cowl when possible during sampling.
NOTE 3: 0.8- μ m pore size filters are commonly used for personal sampling. However, 0.45- μ m filters are recommended for sampling when performing TEM analysis on the same samples. Check personal sampling pumps before use with 0.45- μ m filters to ensure they can operate at the higher pressure drop. Perform calibration with same type of filter as used for sampling.
2. Sampling pump, battery or line-powered vacuum, of sufficient capacity to meet flow rate requirements and, for personal sampling pumps, applicable ISO Standard [12], with flexible connecting tubing.
NOTE: See Step 4 in Sampling section for flow rate.
3. Wire, multi-stranded, 22-gauge; 1" hose clamp to attach wire to cassette for grounding, if needed.
4. Tape, shrink- or adhesive-, or cellulose bands.
5. Slides, glass, pre-cleaned, 25- x 75-mm.
6. Cover slips, 22- x 22-mm, No. 1 $\frac{1}{2}$, unless otherwise specified by microscope manufacturer.
7. Cover slips, as above, imprinted with a relocatable grid, where required for quality assurance or training purposes.
8. Lacquer or nail polish.

9. Knife, #10 surgical steel, curved blade, or scissors.
10. Forceps (tweezers).
11. Flash vaporization system for clearing filters on glass slides using acetone. (See ref. [11] for specifications or see manufacturer's instructions for equivalent devices.). Use a drying oven or warming plate located in fume cabinet if using DMF/glacial acetic acid.
12. Micropipettes or microsyringes, 5- μ L and 100- to 500- μ L.
13. Microscope, positive phase (dark) contrast, with green or blue filter, adjustable field iris, 8x to 10x eyepiece, and 40x to 45x phase objective (total magnification ca. 400x); numerical aperture (NA) = 0.65 to 0.75.
14. Graticule, Walton-Beckett type (Type G22 and G24 are optimized for different counting rules per Appendix C.) with 100- μ m projected diameter circular field (area = 0.00785 mm²) at the specimen plane. Alternative graticules may be used where similar performance has been demonstrated; e.g., the RIB graticule.
NOTE: The graticule is custom-made for each microscope. See APPENDIX A for the custom-ordering process.
15. Phase contrast test slide. A slide with blocks of visible ruled lines where at least one block of lines is certified as invisible under the microscope set up conditions given below.
16. Telescope, ocular phase-ring centering.
17. Stage micrometer (0.01-mm divisions).

SPECIAL PRECAUTIONS: Wear appropriate personal protection during sampling activities and analysis. It is essential that suitable gloves, eye protection, laboratory coat, etc., be used when working with the chemicals. Acetone is toxic at high exposures and is extremely flammable. Take precautions not to ignite it. Heating of acetone in volumes greater than 1 mL must be done in a ventilated laboratory fume hood using a flameless, spark-free heat source. DMF is toxic by inhalation; heating in a drying oven or warming plate located in an operating fume exhaust hood will reduce potential exposure. DMF is also toxic via absorption through the skin.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line. Use a flow meter whose calibration is traceable to national or international standards.
NOTE: See NMAM guidance chapters for discussion on sampling.
2. Cassette assemblies shall be tested to ensure they are not likely to fall apart during sampling. (This testing can be undertaken by the manufacturer.) A press may be useful in ensuring a tight fit, but shall not cause the filter to be cut. To reduce contamination, seal the crease between the cassette base and the cowl with a shrink band or light-colored adhesive tape. Commercial pre-assembled cassettes may already include a taped seal. For personal sampling, fasten the uncapped open-face cassette to the worker's lapel. The open face shall be oriented downward.

NOTE: Electrically grounding the cowl is highly recommended during area sampling, where possible, but especially under conditions of low relative humidity. Use a hose clamp to secure one end of the wire (Equipment, Item 3) to the monitor's cowl. Connect the other end to an earth ground (e.g., cold water pipe). It is recognized that circumstances do not always allow this procedure.

3. Submit at least two field blanks (or 10% of the total samples, whichever is greater) for each set of samples. Handle field blanks in a manner representative of actual handling of associated samples in the set. Open field blank cassettes at the same time as other cassettes just prior to sampling. Store top covers and cassettes in a clean area (e.g., a closed bag or box) with the top covers from the sampling cassettes during the sampling period.
4. Sample at 0.5 L/min or greater [13]. Adjust sampling flow rate, Q [L/min], and time, t (min), to produce a fiber density, E , of 100 to 1300 fibers/mm² (3.85×10^4 to 5×10^5 fibers per 25-mm filter with effective collection area $A_c = 385$ mm²) for optimum counting. These variables are related to the concentration of fibers in air, L (fibers/cc), of the fibrous aerosol being sampled by:

$$t = \frac{A_c * E}{Q * L * 10^3}$$

NOTE 1: The purpose of adjusting sampling times is to obtain optimum fiber loading on the filter. The collection efficiency does not appear to be a function of flow rate in the range of 0.5 to 16 L/min for asbestos fibers [14]. However, counting efficiency is a function of filter loading, with lower loadings typically resulting in higher proportional concentrations [14-16]. A sampling rate of 1 to 4 L/min for 8 h is appropriate in atmospheres containing about 0.1 fiber/cc in the absence of significant amounts of non-asbestos dust. Dusty atmospheres require smaller sample volumes (≤ 400 L) to obtain countable samples. In such cases take short, consecutive samples and average the results over the total collection time. For documenting episodic exposures, use high flow rates (7 to 16 L/min) over shorter sampling times. In relatively clean atmospheres, where targeted fiber concentrations are much less than 0.1 fiber/cc, use larger sample volumes (3000 to 10000 L) to achieve quantifiable loadings. Take care, however, not to overload the filter with background dust. If $\geq 50\%$ of the filter surface is covered with particles, the filter may be too overloaded to count and will bias the measured fiber concentration.

NOTE 2: OSHA regulations specify a minimum sampling volume of 48 L for an excursion measurement, and a maximum sampling rate of 2.5 L/min for all personal asbestos sampling [5].

5. At the end of sampling, shut off the pump, record the time, remove the cassette from the tube attaching it to the pump, and replace top cover and end plugs. Capping the cassette before shutting off the pump and removing the tubing will cause a vacuum within the cassette and damage to the filter.
6. Ship samples with conductive cowl attached in a rigid container with packing material to protect cassettes from jostling or damage.

NOTE: Do not use untreated polystyrene foam in shipping container because electrostatic forces may cause fiber loss from sample filter.

SAMPLE PREPARATION:

Two acceptable procedures for clarifying either a whole filter or a portion of a filter are described below. These procedures are the Acetone clearance procedure and the DMF/acetic acid clearance procedure. In either procedure, the filter material is placed on a glass microscope slide. It is then made transparent (clarified). Then, either triacetin or Euparal are placed on the clarified filter and a cover slip is placed on top.

NOTE 1: The object is to produce samples with a smooth (non-grainy) background in a medium with refractive index ≤ 1.46 . An early method (P&CAM 239 in APPENDIX F) used dimethyl phthalate and diethyl oxalate [17]. This method may still be used as an alternative to those described below, but because the preparations are only stable for one to two days, the procedure is not further discussed.

NOTE 2: Other procedures use a technique for clearing the filter, either acetone (section A) or DMF/glacial acetic acid (section B), followed by mounting the cleared preparation (section C or D). Acetone collapses the filter into a gel, and has the advantage of being the fastest procedure to prepare the filter for the mounting medium. However, acetone can leave residual polymeric structures in the filter that may be counted as fibers. DMF/acetic acid dissolves the filter and does not leave residual structures to the same extent as acetone collapse.

NOTE 3: After the filter is clarified, either triacetin (section C) or Euparal (section D) are acceptable alternative mounting media. Triacetin provides reasonably permanent mounts when the amount used is restricted to $\leq 3.5 \mu\text{L}$, otherwise fiber migration has been observed in slides made with larger quantities [4]. Euparal provides permanent mounts for long-term storage (> 5 years) regardless of the amount used. Either mounting medium can be used with either filter preparation procedure, and all combinations have been demonstrated to provide equivalent fiber counts [4].

7. Prepare samples in a clean area away from any bulk samples which might contaminate the samples. Ensure that the glass slides and cover slips are free of dust and fibers.
8. Determine and record the effective sample collection or filtration area and record the information referenced against the sample ID number.
9. Cut wedges of about 1/4 for a 25-mm diameter filter using a curved-blade surgical steel knife using a rocking motion to prevent tearing. Do not use a blade that was used to open the cassette. Scissors are an option for cutting the filter. The entire filter can be used instead of a wedge, but with the understanding that the whole sample is lost if there is a problem in preparation. Place the wedge or entire filter, dust side up, on slide using forceps. To prevent cross-contamination of samples, blades or scissors should be cleaned between samples.

NOTE 1: Filters can be cut while still in the base of the cassette, or removed and cut on a special cut-stand. If a cut-stand is used it shall be cleaned between filters to ensure no cross-contamination.

NOTE 2: Static electricity will usually keep the wedge on the slide.

A. ACETONE CLEARANCE PROCEDURE

NOTE 1: The aluminum "hot block" or similar flash vaporization techniques may be used outside the laboratory [2].

NOTE 2: Excessive water in the acetone may slow the clearing of the filter. Also, filters that have been exposed to high humidity or water prior to clearing may have a grainy background.

NOTE 3: Rest the "hot block" on a ceramic plate and isolate it from any surface susceptible to heat damage unless it is designed with appropriate safety features to prevent fire or damage.

- A1. If the temperature can be varied, adjust the "hot block" to ca. 70 °C [2], otherwise switch on until ready.
- A2. Mount a wedge cut from the sample filter on a clean glass slide. Insert slide with wedge into the receiving slot at base of the "hot block." Immediately place the tip of a micropipette or microsyringe containing ca. 250 μL acetone (use the minimum volume needed to consistently clear the filter sections) into the inlet port of the PTFE cap on top of the "hot block" and inject the acetone into the vaporization chamber with a slow, steady pressure on the plunger button while holding the micropipette or microsyringe firmly in place. After waiting 3 to 5 seconds for the filter to clear, remove the micropipette or microsyringe and glass slide from their ports.

NOTE: Using excess acetone, or delivering it too fast into the "hot block" may cause material to be washed off the surface of the filter. Using insufficient acetone may cause the filter to curl.

CAUTION: Although the volume of acetone used is small, use safety precautions. Work in a well-ventilated area (e.g., laboratory fume hood). Take care not to ignite the acetone. Continuous use of this device in an unventilated space may produce unhealthful levels or even explosive acetone vapor concentrations.

B. DMF/ACETIC ACID CLEARANCE PROCEDURE

- B1. Make a mixture of 1.4mL (35% volume) of DMF, 0.6mL (15% volume) of glacial acetic acid and 2mL (50% volume) of distilled water in a dark brown vial [3]. This should be done in a fume hood to minimize exposure to chemicals. This solution must be made at least once per week and the vial shall be stored in accordance with applicable laboratory safety procedures (e.g., kept in a vented chemical cabinet rated for flammable materials).
- B2. Turn on the drying oven or warming plate (vented or located in a laboratory fume hood) and allow to come to working temperature. Place a cut filter wedge on a glass slide using forceps. Using $20 \pm 5 \mu\text{L}$ DMF solution, place several drops along the edge of the wedge and allow the solution to wick onto the filter to avoid washing fibers off the filter. Warm the slide at $60 (+2/-5) ^\circ\text{C}$ for 30 minutes in the drying oven or on the warming plate.

C. TRIACETIN MOUNTS AFTER CLEARING AND COLLAPSE

- C1. Using a 5- μL micropipette or microsyringe, immediately place 3.0 to 3.5 μL triacetin on the wedge. Gently lower a clean cover slip onto the wedge at a slight angle to reduce bubble formation. Avoid excess pressure and movement of the cover glass. Do not wait longer than 30 seconds before applying the coverslip.
NOTE: If too many bubbles form or the amount of triacetin is insufficient, the cover slip may become detached within a few hours. If excessive triacetin remains at the edge of the filter under the cover slip, fiber migration may occur [4].
- C2. Mark the outline of the filter segment just inside the edge of the segment with a glass marking pen (such as a permanent ink marker) to aid in microscopic evaluation and to ensure the edge is avoided in the examination. Markings on the bottom of the slide are visible, but outside the depth of focus of fibers and this must be accounted for when positioning the microscope for counting. However, marking the bottom avoids any pressure on the coverslip. If marking the coverslip is preferred, it must be done very lightly and carefully.
- C3. Glue the edges of the cover slip to the slide using lacquer or nail polish [18]. Counting may proceed immediately after clearing and mounting are completed.
NOTE: If clearing is slow, warm the slide on a hotplate (surface temperature $50 ^\circ\text{C}$) for up to 15 min in order to hasten clearing. Heat carefully to prevent gas bubble formation.

D. EUPARAL MOUNTS AFTER CLEARING

- D1. Add 1 drop of Euparal solution on the middle of the wedge and 1 drop to the center of a cover slip. In order to avoid trapping air bubbles, gently lower the cover slip onto the slide at a slight angle. Warm the slide at $60 (+2/-5) ^\circ\text{C}$ for 1 hour to polymerize the Euparal resin.
- D2. Mark the outline of the filter segment just inside the edge of the segment with a glass-marking pen (such as a permanent ink marker) to aid in microscopic evaluation and to ensure the edge is avoided in the examination. Markings on the bottom of the slide are visible, but outside the depth of focus of fibers and this must be accounted for when positioning the microscope for counting. However, marking the bottom avoids any pressure on the coverslip. If marking the coverslip is preferred, it must be done very lightly and carefully.
- D3. It is not necessary to seal the edges of the cover slip with nail polish if Euparal is used. Counting may proceed immediately after clearing and mounting are completed.

CALIBRATION AND QUALITY CONTROL:

10. Microscope adjustments. Follow the manufacturer's instructions for centering and focusing the objectives, condenser, stage, and lamp, if applicable. At least once daily, and preferably at regular intervals throughout the day, use a phase telescope ocular (or Bertrand lens, for some microscopes) to ensure that the phase rings (annular diaphragm and phase-shifting elements) are concentric. With each microscope, keep a paper or electronic logbook in which to record the dates of microscope cleanings and major servicing.

a. Each time a sample is examined, do the following:

- i. Adjust the light source for even illumination across the field of view at the condenser iris. Use Köhler illumination, if available. With some microscopes, the illumination may have to be set up with bright field optics before aligning the phase contrast elements.
- ii. Focus on the particulate material to be examined (do not focus on the marking line).
- iii. Using the condenser focus, make sure that the field iris is in focus, centered on the sample, and open only enough to fully illuminate the field of view.

b. Check the phase-shift detection limit of the microscope periodically for each microscopist/microscope combination:

- i. Center the certified phase-contrast test slide under the phase objective.
- ii. Bring the blocks of grooved lines into focus in the graticule area.

NOTE: The slide contains seven blocks of grooves (ca. 20 grooves per block) in descending order of visibility. For asbestos counting, it is intended that some blocks of lines are completely visible and one or more are completely invisible when centered in the graticule area (blocks in between may be partially visible). The visibility of the blocks must match the statements in the accompanying certificate [19]. A microscope which fails to meet this requirement for a test slide has resolution either too low or too high for fiber counting.

- iii. If image quality deteriorates, clean the microscope optics. If the problem persists, consult the microscope manufacturer.

c. Measure the projected diameter of the Walton-Beckett type graticule to assure that it is $100\text{ }\mu\text{m} \pm 2\text{ }\mu\text{m}$. Use the measured value to calculate the actual area of graticule. Check the measured value at the minimum and maximum interpupillary distance of the eyepieces. If the diameter changes, then the diameter may change any time a different microscopist uses the microscope and changes the interpupillary distance. If service is performed on the microscope, or components of the microscope are changed, measure the projected diameter again as changes may occur if the tube length or the location of the graticule in the eyepiece is changed.

- i. Center the stage micrometer under the 40X objective of the microscope.
- ii. Bring the scale into focus in the graticule area
- iii. The lines in the scale will appear wide. Using the mechanical stage of the microscope, manipulate the slide until the graticule is at one edge of a major line of the stage micrometer.
- iv. Estimate the diameter of the graticule using edges of the lines of the stage micrometer. Do not attempt to use the center of the micrometer lines.
- v. If the diameter is greater than $102\text{ }\mu\text{m}$ or less than $98\text{ }\mu\text{m}$, determine if the inter-pupillary distance is correct for the analyst. If it is, then the microscope must be adjusted by someone qualified to service the microscope, or the graticule replaced by one meeting the projected diameter specification.

11. Determine a microscopist's ability to observe, measure, and count fibers.

NOTE: Prior to examining samples, microscopists shall undergo training to observe, measure, and count fibers and the results of such training shall be available for inspection. A procedure for such training, which allows proficiency to be demonstrated quantitatively, may be found in Appendix B.

12. Document the laboratory's precision for each microscopist for replicate fiber counts.

- a. Maintain as part of the laboratory quality assurance program a set of reference slides to be used on a daily basis [20]. These slides shall consist of filter preparations including a range of loadings and background dust levels from a variety of sources including both field and reference samples (e.g., PAT, AAR, or commercial samples). The Quality Assurance Officer shall maintain custody of the reference slides and shall supply each microscopist with a minimum of one reference slide per workday. Change the labels on the reference slides periodically so that the microscopist does not become familiar with the samples.
 - b. From blind repeat counts on reference slides, estimate the laboratory intra- and inter-microscopist precision. Obtain separate values of relative standard deviation (S_r) for each sample matrix analyzed in each of the following ranges: 5 to 20 fibers in 100 graticule fields, >20 to 50 fibers in 100 graticule fields, and >50 to 100 fibers in 100 graticule fields. Maintain control charts for each of these data files.
 - c. Alternatively, a laboratory may develop an analytical uncertainty model from intra- and inter-microscopist precision measurements (but, in practice, most laboratories will choose option b).
- NOTE: Certain sample matrices (e.g., asbestos cement) have been shown to give poor precision [23].
13. Prepare and count field blanks along with the field samples. Report counts on each field blank.

NOTE 1: The identity of blank filters shall be unknown to the microscopist until all counts have been completed.

NOTE 2: If a field blank yields greater than 7 fibers per 100 graticule fields, report possible contamination of the samples.
 14. Perform blind recounts by the same microscopist on 10% of filters counted (slides relabeled by a person other than the microscopist). Use the following test to determine whether a pair of counts by the same microscopist on the same filter shall be rejected because of possible bias: Discard the data if the absolute value of the difference between the square roots of the two counts (in fiber/mm²) exceeds $2.77XS'_r$ where X is the average of the square roots of the two fiber counts (in fiber/mm²) and $S'_r = (S_r/2)$ where S_r is the intra-microscopist relative standard deviation for the appropriate count range (in fibers) determined in step 12. For more complete discussions see reference [20].

NOTE 1: Fiber counting as the measurement of randomly placed fibers may be described by a Poisson distribution, so that a square root transformation of the fiber count data will result in approximately normally distributed data [20].

NOTE 2: If a pair of counts is rejected by this test, recount all other samples in the set and test the new counts against the first counts. Discard all rejected paired counts. It is not necessary to use this statistic on blank counts.

NOTE 3: Do not use an intra-microscopist variation calculated from standard relocatable test slides (Appendix B) for this test.
 15. The microscopist is a critical part of this analytical procedure. Care must be taken to provide a non-stressful and comfortable environment for fiber counting. Use an ergonomically designed chair, with the microscope eyepiece situated at a comfortable height for viewing. Set external lighting at a similar level to the illumination level in the microscope to reduce eye fatigue. In addition, microscopists shall take 10- to 20-minute breaks from the microscope every one or two hours to limit fatigue [21]. During these breaks, eye and upper back, and neck exercises can be performed to relieve strain.
 16. All laboratories engaged in asbestos counting shall participate in a proficiency testing program such as the AIHA Industrial Hygiene Proficiency Analytical Testing (IHPAT) Program for asbestos.
 17. Each laboratory analyzing asbestos samples shall implement an interlaboratory quality assurance program that as a minimum includes participation of at least two other independent laboratories. Each laboratory shall participate in round robin testing at least once every 6 months with at least all the other laboratories in its interlaboratory quality assurance group. Each laboratory shall submit slides typical of its own work load for use in this program. The round robin shall be designed and results analyzed using appropriate statistical methodology.

MEASUREMENT:

18. Center the slide on the stage of the calibrated microscope under the objective lens. Focus the microscope on the sample-containing plane of the filter.
19. Adjust the microscope (Step 10).
20. Counting rules: (same as P&CAM 239 rules [1,17,22]: see examples in APPENDIX C).
 - a. Count any fiber longer than 5 μm which lies entirely within the graticule area.
 - i. Count only fibers longer than 5 μm .
 - ii. Measure length of curved fibers along the curve. Count only fibers with a length-to-width ratio equal to or greater than 3:1.
 - b. For fibers which cross the boundary of the graticule field:
 - i. Count as 1/2 fiber any fiber with only one end lying within the graticule area, provided that the fiber meets the criteria of rule "a" above.
 - ii. Do not count any fiber which crosses the graticule boundary more than once.
 - iii. Reject and do not count all other fibers.
 - c. Count bundles of fibers as one fiber unless individual fibers can be identified by observing both ends of a fiber.
 - d. Count enough graticule fields to yield 100 fibers. Count a minimum of 20 fields. Stop at 100 graticule fields regardless of count.
21. Start counting from the tip of the filter wedge and progress along a radial line to the outer edge. Shift up or down on the filter, and continue in the reverse direction. Select graticule fields randomly. Ensure that, as a minimum, each analysis covers one radial line from the filter center to the outer edge of the filter. When an agglomerate or bubble covers ca. 1/8 or more of the graticule field, reject the graticule field and select another. [35] Do not report rejected graticule fields in the total number counted.

NOTE 1: When counting a graticule field, continuously scan a range of focal planes by moving the fine focus knob to detect very fine fibers which have become embedded in the filter. The small-diameter fibers will be very faint but are an important contribution to the total count. A minimum counting time of 15 s per field is appropriate for accurate counting.

NOTE 2: Do not count at the outside edge of the filter, or at edges where the filter was cut, as the fiber distribution may be disturbed in these areas. Move in at least 1 mm from the edge (i.e., inside the marked line).

NOTE 3: Under certain conditions, electrostatic charge may affect the sampling of fibers. These electrostatic effects are most likely to occur when the relative humidity is low (below 20%), and when sampling is performed near the source of aerosol. The result is that deposition of fibers on the filter is reduced, especially near the edge of the filter. If such a pattern is noted during fiber counting, choose fields as close to the center of the filter as possible [11].

NOTE 4: Counts are to be recorded on a paper or electronic data sheet that provides, as a minimum, record of the counts for each field, filter identification number, analyst's name, date, total fibers counted, total fields counted, mean count, fiber density, and commentary. The average count is calculated by dividing the total fiber count by the number of fields observed. Fiber density (fibers/ mm^2) is defined as the average count (fibers/field) divided by the field (graticule) area (mm^2/field).

CALCULATIONS AND REPORTING OF RESULTS:

22. Calculate and report fiber density on the filter, E (fibers/ mm^2), by dividing the mean fiber count per graticule field, F/n_f , minus the mean field blank count per graticule field, B/n_b , by the graticule field area, A_f :

$$E = \frac{\left(\frac{F}{n_f}\right) - \left(\frac{B}{n_b}\right)}{A_f}, \text{ fibers/mm}^2$$

NOTE 1: $A_r=0.00785 \text{ mm}^2$ is the area for a graticule with a projected diameter of $100 \mu\text{m}$. The actual area shall be calculated using the measured diameter of the graticule. The measured diameter shall be $100 \pm 2 \mu\text{m}$ at the proper magnification. The diameter of the graticule shall be re-measured whenever the microscope is serviced or if necessary if there is a change in measured size with inter-pupillary distance (e.g., between different microscopists).

NOTE 2: Fiber counts above 1300 fibers/mm^2 and fiber counts from samples with $>50\%$ of filter area covered with particulate shall be reported as "uncountable" or "probably biased." Fiber counts outside the $100\text{--}1300 \text{ fiber/mm}^2$ range shall be reported as having "greater than optimal variability" and as being "probably biased."

23. Calculate and report the concentration, (C) in fibers/cc, of fibers in the air volume sampled, (V) in liters, using the effective collection area of the filter, A_c (nominally 385 mm^2 for a 25-mm filter):

$$C = \frac{EA_c}{V * 10^3}$$

NOTE: The area varies according to the cassette inner diameter, where it contacts the filter, which could vary between manufacturers and even between production runs. For most accurate results the value of A_c may be checked and adjusted accordingly, but, in practice, the variation in this parameter is very small in comparison to other sources of uncertainty.

24. Report intra- and interlaboratory relative standard deviations with each set of results.

NOTE: Precision depends on the total number of fibers counted [1,24]. Relative standard deviation is documented in references [1,24-25] for fiber counts up to 100 fibers in 100 graticule fields. Comparability of interlaboratory results is discussed below. As a first approximation, use 213% above and 49% below the count as the upper and lower confidence limits for fiber counts greater than 20 (Figure 1).

EVALUATION OF METHOD:

Summary of Method Revisions:

Each revision or release clarified minor points of grammar and procedure as well as major changes. This summary lists the major changes only.

The initial US Public Health Service/NIOSH method for counting fibers was published in 1968 [26]. It was based upon the British Hygiene standard [27] and procedures developed by the British Asbestos Research Council. The procedure was published in the NIOSH Criteria for a Recommended Standard [28] Section VIII, Appendix I (1972).

An informal draft version was used until 1976 when NIOSH issued it as P & CAM 239. It was published in the NIOSH Manual of Analytical Methods, 2nd ed., Vol. 1, P&CAM 239, U.S. Department of Health, Education, and Welfare Pub. (NIOSH) 11-157-A (1977) [17].

NIOSH Method 7400 Issued 15 February 1984

In 1984, the NIOSH P&CAM 239 Fiber counting method was renamed Method 7400 and rewritten to be in the standard format for inclusion in the NIOSH Manual of Analytical Methods. This revision included changes in three areas:

1. Sampling: The change from a 37-mm to a 25-mm filter improved sensitivity for similar air volumes. The change in flow rates allowed for 2 m^3 full-shift samples to be collected, providing that the filter is not overloaded with non-fibrous particulates. The collection efficiency of the sampler is not a function of flow rate in the range 0.5 to 16 L/min [17].
2. Sample preparation technique: The acetone vapor-triacetin preparation technique was included as a faster, more permanent mounting technique than the dimethyl phthalate/diethyl oxalate method of

P&CAM 239 [2,7,17]. The aluminum “hot block” technique minimizes the amount of acetone needed to prepare each sample.

3. Measurement:

- a. The Walton-Beckett graticule standardizes the area observed [21,29-30].
- b. A phase shift test slide standardizes microscope optics [31].
- c. Because of past inaccuracies associated with low fiber counts, the minimum recommended loading was increased to 100 fibers/mm² filter area (a total of 78.5 fibers counted in 100 fields, each with field area = 0.00785 mm²). Lower levels generally result in an overestimate of the fiber count when compared to results in the recommended analytical range [15]. The recommended loadings are expected to yield intra-microscopist S_r in the range of 0.10 to 0.17 [32, 36].
- d. An alternative set of counting rules (B rules) was introduced to improve precision of counting.

NIOSH Method 7400 Issued 15 February 1984, Revision #1 15 May 1985

Revision #1 included a discussion of interlaboratory variability and comparison of the A and B counting rules.

NIOSH Method 7400 Issued 02 February 1984, Revision #2 15 May 1986

Revision # 2 added an expanded discussion of interlaboratory variability and comparison of the A and B counting rules. It moved the “B” rules to an appendix as alternate counting rules (which should be used when counting man-made mineral fibers, e.g., fibrous glass, refractory ceramic fibers, etc.). It added three Appendices:

- a. Appendix A: Calibration of the Walton Beckett Graticule
- b. Appendix B: Examples of Counting Rules
- c. Appendix C: Alternate Counting Rules

NIOSH Method 7400 Issued 02 February 1984, Issue 1: Revision #3 15 May 1989

Issue 1: Revision #3 corrected some technical issues and clarified the method language.

NIOSH Method 7400 Issued 02 February 1984, Issue 2: 15 August 1994

Issue 2 refined the method language, added some procedural notes and added an additional Appendix D listing Equivalent Limits of Detection and Quantitation.

NIOSH Method 7400 Issued 02 February 1984, Issue 3: 29 April 2019

Issue 3 of this Method incorporates:

1. More accurate values for the minimum width limits for asbestos fiber detection. A value of 0.15 μm for chrysotile and 0.05 μm for amphibole asbestos is based on published research [7,8]. A recent user check confirms that these values are achievable [10]. However, not all microscopists can see finer chrysotile fibers between 0.15 μm and 0.2 μm without adequate training [33-34]. Note that PCM does not distinguish between asbestos types.
2. DMF/acetic acid as an alternative filter clearing procedure to acetone, and Euparal as an alternative mounting medium to triacetin. Either mounting medium is used with either clearing procedure. The DMF/acetic acid procedure does not leave undissolved residues from the filter medium, which may appear as fibers. Euparal produces permanent mounts regardless of the quantity of medium used [3]. These alternatives have been shown in a user-check to provide fiber counts that are not significantly different from counts in slides made from the original dimethyl phthalate/diethyl oxalate procedure [4]. This revision also allows the continued use of dimethyl phthalate/diethyl oxalate, if preferred.

3. A generic description of phase-shift test slides, since changes in the manufacture have led to confusion [19]. The description now only requires the slides to have a certificate stating that there are fully visible blocks and invisible blocks, so that the required phase-shift is bracketed. The microscopist is expected to be able to visualize a slide according to its certificate. Newer slides have been shown in a user-check to provide fiber counts that are not significantly different from counts in slides where the original version of the phase shift test slide was used for calibration [10].
4. Standard relocatable test slides as an additional non-mandatory quality assurance Appendix, and as documentation of inter- and intra-microscopist precision without the contribution from the selection of counting fields. Several user-checks have demonstrated increases in average analyst performance after training on these slides [33-34].
5. Guidance on the calculation of an expanded uncertainty budget. This guidance allows users to determine expanded uncertainty as required under international standards [35].
6. Re-numbering of appendices.

INTERLABORATORY COMPARABILITY

An international collaborative study involved 16 laboratories using prepared slides from the asbestos cement, milling, mining, textile, and friction material industries [23]. The relative standard deviations (S_r) varied with sample type and laboratory. The ranges were:

Rules	Intralaboratory S_r	Interlaboratory S_r	Overall S_r
AIA (NIOSH A Rules)*	0.12 to 0.40	0.27 to 0.85	0.46
Modified CRS (NIOSH B Rules)*	0.11 to 0.29	0.20 to 0.35	0.25

*Under AIA (Asbestos International Association) rules, only fibers having a diameter less than 3 μm are counted and fibers attached to particles larger than 3 μm are not counted. NIOSH A Rules are otherwise similar to the AIA rules.

A NIOSH study conducted using field samples of asbestos gave intralaboratory S_r in the range 0.17 to 0.25 and an interlaboratory S_r of 0.45 [20]. This agrees well with other recent studies [21,23-24,36].

At this time, there is no independent means for assessing the overall accuracy of this method. One measure of reliability is to estimate how well the count for a single sample agrees with the mean count from a large number of laboratories. The following discussion indicates how this estimation can be carried out based on measurements of the interlaboratory variability, as well as showing how the results of this method relate to the theoretically attainable counting precision and to measured intra- and interlaboratory S_r (NOTE: The following discussion does not include bias estimates and cannot be taken to indicate that lightly loaded samples are as accurate as properly loaded ones).

Theoretically, the process of counting randomly (Poisson) distributed fibers on a filter surface will give a S_r that depends on the number, N , of fibers counted: $S_r = \frac{1}{\sqrt{N}}$

Thus, S_r is 0.1 for 100 fibers and 0.32 for 10 fibers counted. The actual S_r found in a number of studies is greater than these theoretical numbers [15,25,30,36].

An additional component of variability comes primarily from subjective interlaboratory differences. In a study of ten microscopists in a continuing sample exchange program, Ogden [24] found this subjective component of intralaboratory S_r to be approximately 0.2 and estimated the overall S_r by the term:

$$\frac{\sqrt{N + (0.2 + N)^2}}{N}$$

Ogden found that the 90% confidence interval of the individual intralaboratory counts in relation to the means were $+2 S_r$ and $-1.5 S_r$. In this program, one sample out of ten was a quality control sample. For

laboratories not engaged in an intensive quality assurance program, the subjective component of variability can be higher.

In a study of field sample results in 46 laboratories, the Asbestos Information Association (AIA) also found that the variability had both a constant component and one that depended on the fiber count [18]. These results gave a subjective interlaboratory component of S_r (on the same basis as Ogden's) for field samples of ca. 0.45. A similar value was obtained for 12 laboratories analyzing a set of 24 field samples [36]. This value falls slightly above the range of S_r (0.25 to 0.42 for 1984–85) found for 80 reference laboratories in the NIOSH PAT program for laboratory-generated samples [25].

A number of factors influence intralaboratory S_r for a given laboratory, such as that laboratory's actual counting performance and the type of samples being analyzed. In the absence of other information, such as from an interlaboratory quality assurance program using field samples, the value for the subjective component of variability is chosen as 0.45. It is recommended that each laboratory establish an interlaboratory quality assurance program to improve their performance and thus reduce the intralaboratory S_r .

The above relative standard deviations apply when the population mean has been determined. It is more useful, however, for laboratories to estimate the 90% confidence for of the count distribution for both interlaboratory and intralaboratory results [24].

For example, if a sample yields a count of 24 fibers, Figure 1 indicates that the mean interlaboratory count will fall within the range of 227% above and 52% below that value 90% of the time. These percentages can be applied directly to the air concentrations as well. If, for instance, this sample (24 fibers counted) represented a 500-L volume, then the measured concentration is 0.02 fibers/cc (assuming 100 fields counted, 25-mm filter, 0.00785 mm² counting field area). If this same sample were counted by a group of laboratories, there is a 90% probability that the mean would fall between 0.01 and 0.08 fiber/cc. These limits are to be reported in any comparison of results between laboratories.

Note that the interlaboratory S_r of 0.45 used to derive Figure 1 is used as an estimate for a random group of laboratories. If several laboratories belonging to a quality assurance group can show that their interlaboratory S_r is smaller, then it is more correct to use that smaller S_r . However, the estimated interlaboratory S_r of 0.45 is to be used in the absence of such information. Note also that it has been found that interlaboratory S_r can be higher for certain types of samples, such as asbestos cement [23].

Quite often the estimated airborne concentration from an asbestos analysis is used to compare to a regulatory standard. For instance, if one is trying to show compliance with an 0.5 fiber/cc standard using a single sample on which 100 fibers have been counted, then Figure 1 indicates that the 0.5 fiber/cc standard must be 213% higher than the measured air concentration. This indicates that if one measures a fiber concentration of 0.16 fiber/cc (100 fibers counted), then the mean fiber count by a group of laboratories (of which the compliance laboratory might be one) has a 95% chance of being less than 0.5 fibers/cc; i.e., $0.16 + 2.13 \times 0.16 = 0.5$.

It can be seen from Figure 1 that the Poisson component of the variability is not very important unless the number of fibers counted is small. Therefore, a further approximation is to simply use +213% and -49% as the upper and lower confidence values of the mean for a 100-fiber count.

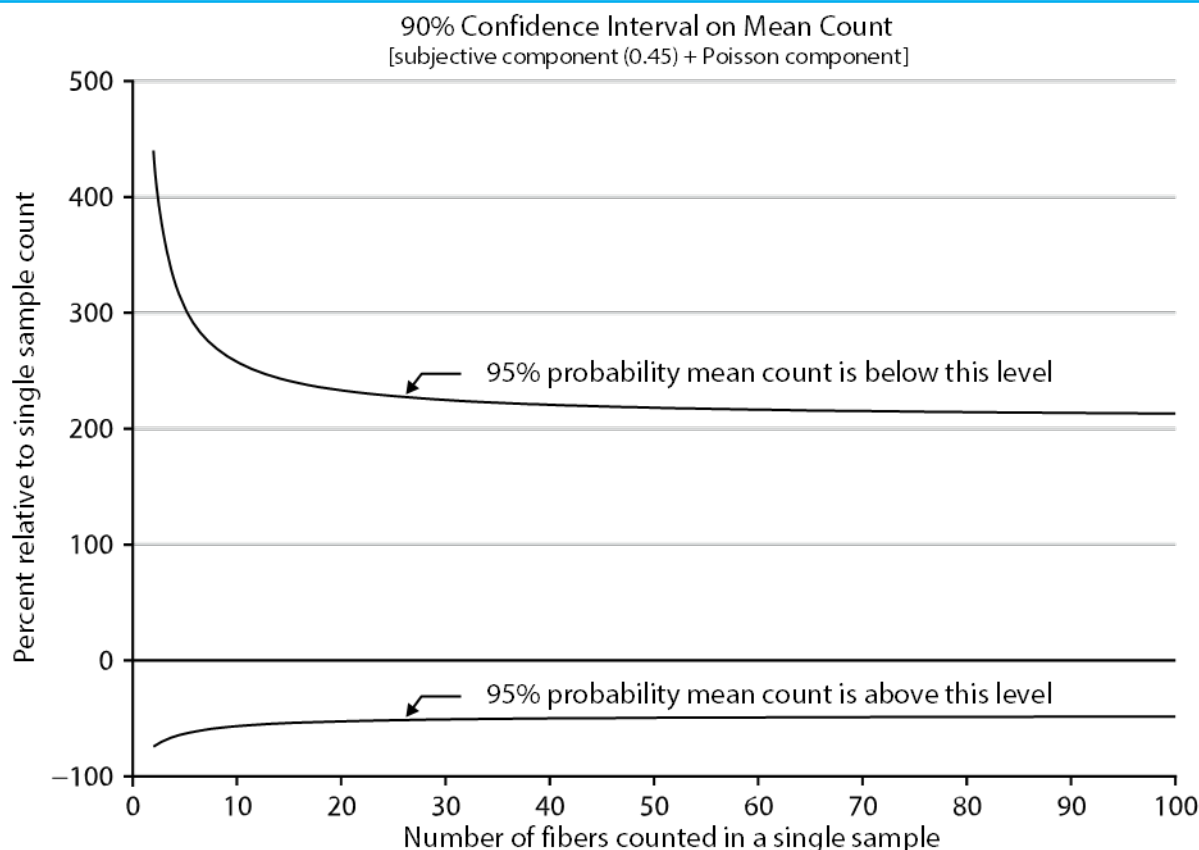


Figure 1. Interlaboratory precision of fiber counts

The curves in Figure 1 are defined by the following equations:

$$UCL = \frac{2X + 2.25 + \sqrt{(2.25 + 2X)^2 - 4(1 - 2.25S_r^2)X^2}}{2(1 - 2.25S_r^2)}$$

and

$$LCL = \frac{2X + 4 - \sqrt{((4 + 2x)^2 - 4(1 - 4S_r^2)X^2)}}{2(1 - 4S_r^2)}$$

where S_r = subjective interlaboratory relative standard deviation, which is close to the total interlaboratory S_r when approximately 100 fibers are counted, X = total fibers counted on sample, UCL = upper 95% confidence limit, and LCL = lower 95% confidence limit.

NOTE: The range between these two limits represents 90% of the total range.

It is also possible to use a similar approach to determining expanded uncertainty within a single laboratory.

The formula:

$$S_r = \frac{\sqrt{N + (0.2N)^2}}{N}$$

suggests that if n is the number of counts taken in a single reading, then the distribution of

$$\frac{n - N}{\sqrt{n + S_r^2 N^2}}$$

though unknown, depends only weakly on N, because the mean and variance are independent of N. In fact, from many readings by a number of microscopists, with N equal to the mean across the microscopists, confidence intervals were determined [24] as:

$$-1.8 \leq \frac{n - N}{\sqrt{N + S_r^2 N^2}} \leq +2.6$$

By solving the two quadratic equations at the two extreme limits for N in terms of n, confidence limits enclosing the consensus mean N, given single measurements n are easily obtained: $LCL[n] < N < UCL[n]$ (95% Confidence Limit), where LCL and UCL are given by:

$$LCL[n] = \frac{2n + 2.6^2 - \sqrt{(2.6^2 + 2n)^2 - 4(1 - 2.6^2 S_r^2)n^2}}{2(1 - 2.6^2 S_r^2)}$$

and

$$UCL[n] = \frac{2n + 1.8^2 + \sqrt{(1.8^2 + 2n)^2 - 4(1 - 1.8^2 S_r^2)n^2}}{2(1 - 1.8^2 S_r^2)}$$

NOTE: In these formulae the confidence level is expanded from 90% to 95%.

From these equations, the confidence limits accounting for both subjective microscopist and Poisson components for various fiber counts are calculated (Table 1) by taking $S_r = 0.2$. The intra- and inter-microscopist variability may be greater if quality control is poor.

Table 1. Intra-microscopist ($S_r=0.2$) 95% confidence interval bracketing the consensus mean for various numbers (n) of fibers in a single count

Number (n) for fibers	Lower confidence limit (LCL)	Upper confidence limit (UCL)	Expanded uncertainty (%)
5	1.6	13	(-68;160)
7	2.6	16	(-63;129)
10	4.2	21	(-58;110)
20	10	37	(-50;85)
50	29	85	(-42;70)
100	62	163	(-38;63)
200	127	319	(-36;60)

NOTE: Also shown is the confidence interval expressed as expanded uncertainty asymmetric around n and stated relative to n.

UNCERTAINTY BUDGET EXCLUDING COUNTING ERRORS

Various factors aside from counting errors can introduce random variation in measured values of asbestos concentrations [35]. Such factors are identified in Table 2.

Table 2. Example of an uncertainty budget for non-counting variable

Variable	Estimated uncertainty	Uncertainty squared
Sampling flowrate	0.03	0.0008
Sampling time	0.02	0.0004
Master stage micrometer	0.01	0.0001
Calibration of submaster	0.01	0.0001
Calibration of graticule	0.02	0.0004
Area of exposed filter	0.05	0.0025
Sum of squares	-	0.0043
Square root of sum of squares = Overall uncertainty	-	0.066
Expanded uncertainty (coverage factor $k = 2$)	-	0.13 or 13 %

The uncertainty in Table 2 contributes in quadrature to the expanded uncertainty. Therefore, as 13% is small relative to the values in Table 1, it may be reasonable to conclude that the contribution of non-counting errors to the total uncertainties is not significant in comparison to the Poisson and subjective errors.

NOTE 1: Non-counting errors are negligible only if consistently controlled.

NOTE 2: Inhomogeneous deposition of dust on the filter leads to gross errors, the magnitude of which cannot be estimated. Counting 20 or more fields ensures that minor divergence from randomness does not bias the result.

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Table 3. CAS numbers for analytes

Name	CAS No.
Actinolite	[77536-66-4]
Ferroactinolite	[15669-07-5]
Amosite	[12172-73-5]
Anthophyllite	[77536-67-5]
Chrysotile	[12001-29-5]
Crocidolite	[12001-28-4]
Tremolite	[77536-68-6]
Amphibole asbestos	[1332-21-4]
Refractory ceramic fibers	[142844-00-6]
Fibrous glass	[14808-60-7]

APPENDIX A: CALIBRATION OF THE GRATICULE

Before ordering any graticule, the following calibration must be done to obtain a counting area (D) 100 µm in diameter at the image plane. The diameter, d_c (mm), of the circular counting area and the disc diameter must be specified when ordering the graticule.

1. Insert any available graticule into the eyepiece and focus so that the graticule lines are sharp and clear.
2. Set the appropriate inter-pupillary distance and, if applicable, reset the binocular head adjustment so that the magnification remains constant.
3. Install the 40x to 45x phase objective.
4. Place a stage micrometer on the microscope object stage and focus the microscope on the graduated lines.
5. Measure the magnified grid length of the graticule, L_o (µm), using the stage micrometer.
6. Remove the graticule from the microscope and measure its actual grid length, L_a (mm). This can best be accomplished by using a stage fitted with verniers.
7. Calculate the circle diameter, d_c (mm), for the Walton-Beckett graticule: $d_c = \frac{L_a}{L_o} \times D$
EXAMPLE: if $L_o = 112$ µm, $L_a = 4.5$ mm, and $D = 100$ µm, then $d_c = 4.02$ mm.
8. Check the field diameter, D (acceptable range 100 µm ± 2 µm) with a stage micrometer upon receipt of the graticule from the manufacturer. Determine field area (acceptable range 0.00754 mm² to 0.00817 mm²).

APPENDIX B: DETERMINATION OF A MICROSCOPIST'S ABILITY TO OBSERVE, MEASURE AND COUNT FIBERS USING RELOCATABLE GRIDDED COVERSLEIPS (Non-mandatory)

1. Reference slides are commercially available, made from chrysotile or amosite proficiency test filters obtained from the Industrial Hygiene Proficiency Analytical Testing (IHPAT) scheme of the American Industrial Hygiene Association Proficiency Analytical Testing, LLC [37]. Each slide has been mounted

with a permanent Euparal medium and covered with a relocatable gridded cover slip. The fibers visible in each grid opening have been identified and their locations marked on a drawing of each opening. The identity, number and position of each fiber have been verified by a second microscopist. The accuracy of this verification has been shown to be within 3% of consensus values determined by multiple microscopists, separately and together [38]. Such slides are referred to in this method as standard relocatable test slides, and they are applied in various ways to improve and assess the quality of fiber counts.

NOTE: It is also possible to make standard relocatable test slides in the laboratory from PAT or field samples by using commercially available relocatable gridded cover slips. Either filter clearance or slide mounting procedure can be used with these cover-slips, but the DMF/acetic acid-Euparal procedure has been demonstrated to provide slides with long-term stability. The fibers in each grid are identified, marked and verified by at least two microscopists as above.

2. Count the fibers in each designated field and refer the counts to the accompanying slide descriptions. Calculate a score from the number of absolute discrepancies between the reported and verified fibers in each field as shown in:

$$Score = \left(1 - \left(\frac{\text{number of discrepancies}}{\text{number of verified fibers}}\right)\right) \times 100$$

3. Microscopists shall obtain a discrepancy score of more than 70 for a slide containing amosite fibers and more than 50 for a slide containing chrysotile fibers from the AIHA IHPAT scheme (or more than 70 for a slide prepared from chrysotile field samples) before proceeding. These scoring criteria are based on the results that have been achieved in round-robin studies and proficiency test programs. If these scores are not achieved, the Quality Assurance Officer is expected to work with the microscopist to review the slide descriptions to determine the cause and then attempt to rectify the situation by having the microscopist repeat the microscope set-up or by re-training.

NOTE: The number of absolute discrepancies has been shown to be linearly related to the sum of counting errors [37]. The positive discrepancies are mainly due to sizing extra fibers (especially amphiboles). The negative discrepancies are mainly due to oversight of fibers (especially chrysotile). Chrysotile filters from the AIHA IHPAT program contain a large number of fibers with narrow widths near the limit of detection and thus provide a very robust test of visual acuity and concentration. In theory, it should be possible for microscopists to obtain high discrepancy scores using slides made from chrysotile PAT samples, but a discrepancy score above 50 is sufficient to ensure the microscopist will be able to see the majority of the wider fibers typical of field samples.

4. The repeated fiber counts obtained from standard relocatable test slides can be used to document the laboratory's precision for each microscopist, per step 13. If repeated counts from specific fields are used, the resulting intra- and inter-microscopist precision will be improved because there will not be a contribution from the selection of field areas to be counted.

APPENDIX C: COMPARISON OF COUNTING RULES

Figure 2 shows a Walton-Beckett graticule as seen through the microscope. This is a Type G22 graticule with 3:1 ratios, providing optimal assistance for these counting rules. The rules will be discussed as they apply to the labeled objects in the figure.

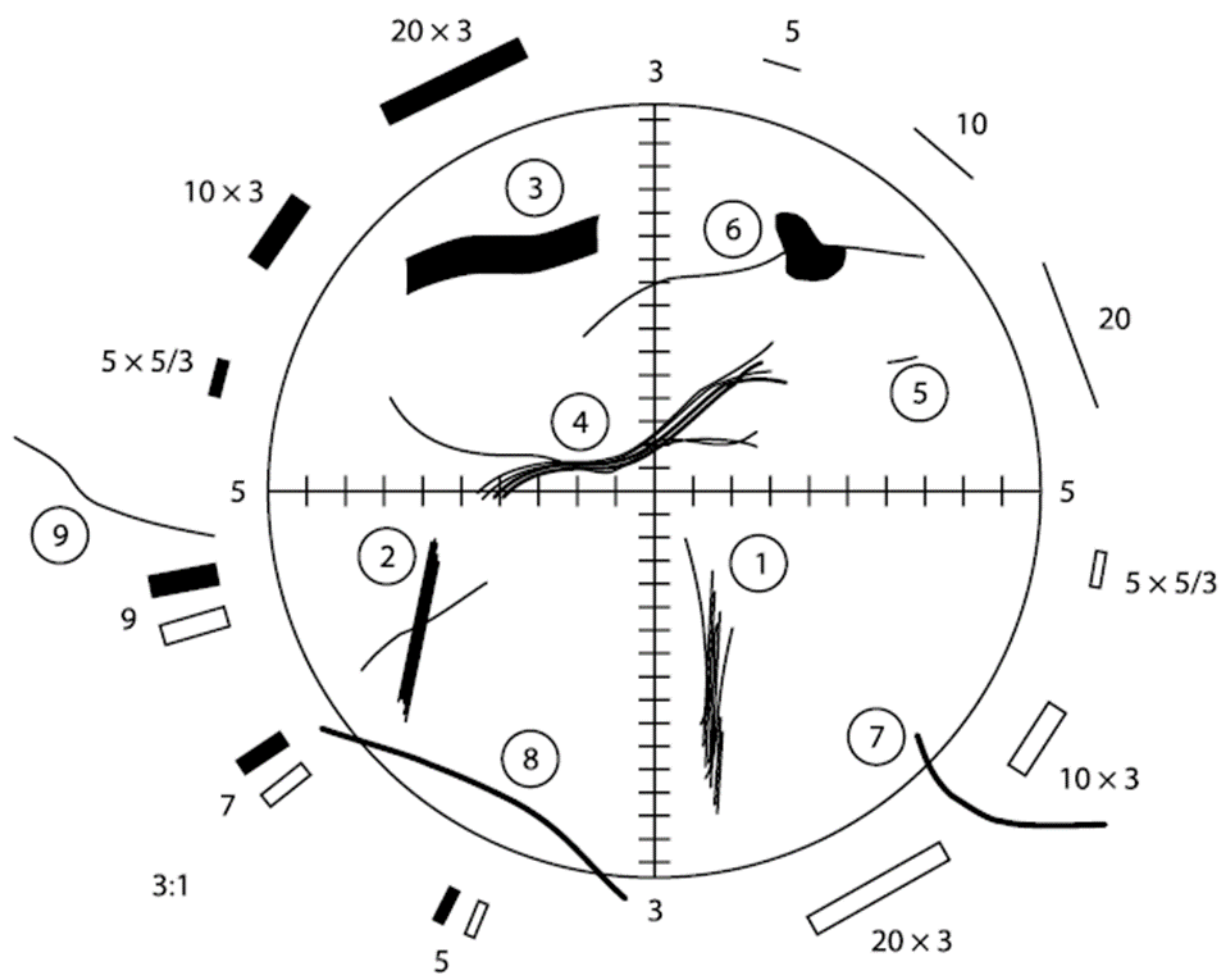


Figure 2 Walton-Beckett graticule with fibers

These rules are sometimes referred to as the "A" rules:

Object	Count	Discussion
1	1 fiber	Optically observable asbestos fibers are actually bundles of fine fibrils. If the fibrils seem to be from the same bundle, the object is counted as a single fiber. Note, however, that all objects meeting length and aspect ratio criteria are counted whether or not they appear to be asbestos.
2	2 fibers	If fibers meeting the length and aspect ratio criteria (length > 5 μm and length-to-width ratio > 3 to 1) overlap, but do not seem to be part of the same bundle, they are counted as separate fibers.
3	1 fiber	Although the object has a relatively large diameter (> 3 μm), it is counted as a fiber under the rules. There is no upper limit on the fiber diameter in the counting rules. Note that fiber width is measured at the widest compact section of the object.
4	1 fiber	Although long fine fibrils may extend from the body of a fiber, these fibrils are considered part of the fiber if they seem to have originally been part of the bundle.
5	Do not count	If the object is $\leq 5 \mu\text{m}$ long, it is not counted
6	1 fiber	A fiber partially obscured by a particle is counted as one fiber. If the fiber ends emanating from a particle do not seem to be from the same fiber and each end meets the length and aspect ratio criteria, they are counted as separate fibers.
7	$\frac{1}{2}$ fiber	A fiber which crosses into the graticule area one time is counted as $\frac{1}{2}$ fiber.
8	Do not count	Ignore fibers that cross the graticule boundary more than once.
9	Do not count	Ignore fibers that lie outside the graticule boundary.

NOTE 1: OSHA Method ID-160 has slightly different counting rules, e.g. Rule 5: If the object is $\leq 5 \mu\text{m}$, it is not counted, would become: If the object is $< 5 \mu\text{m}$, it is not counted. In practice, these minor differences would not likely modify calculated concentrations to the extent that decisions based on those concentrations would be affected, but this opinion has not been tested.

NOTE 2: Fibers wider than $3 \mu\text{m}$ are included in this method. Some other methods for counting asbestos fibers restrict the count to only those fibers of $3 \mu\text{m}$ width or less, as these are the fibers most likely to penetrate to the bronchial and lower airways. However, the inlet efficiency of the cowl has a similar effect in that it limits the maximum diameter of fibers entering the cassette, so the effect of this additional dimensional counting rule on the total fiber count is expected to be small. Any bias from not applying an upper width limit is also conservative (i.e. concentrations will be assessed higher).

APPENDIX D: ALTERNATE COUNTING RULES FOR NON-ASBESTOS FIBERS

Other counting rules may be more appropriate for measurement of specific non-asbestos fiber types, such as fibrous glass. These include the rules given below ("B" rules from NIOSH Method 7400, Revision #2, dated 08/15/1987), the World Health Organization reference method for man-made mineral fiber [39], and the NIOSH fibrous glass criteria document method [40]. The upper diameter limit in these methods prevents measurements of non-thoracic fibers. It is important to note that the aspect ratio limits included in these methods vary. Graticules designed for counting according to these rules are available (e.g. Walton-Beckett Type G24).

OSHA recommends using these alternate, or "B" counting rules as given below for all man-made vitreous fibers (MMVF). It is emphasized that hybridization of different sets of counting rules is not permitted. Report specifically which set of counting rules are used with the analytical results.

"B" Counting Rules

1. Count only *ends* of fibers. Each fiber must be longer than $5 \mu\text{m}$ and less than $3 \mu\text{m}$ diameter.
2. Count only ends of fibers with a length-to-width ratio equal to or greater than 5:1.
3. Count each fiber end which falls within the graticule area as one end, provided that the fiber meets rules 1 and 2 above. Add split ends to the count as appropriate if the split fiber segment also meets the criteria of rules 1 and 2 above.
4. Count visibly free ends which meet rules 1 and 2 above when the fiber appears to be attached to another particle, regardless of the size of the other particle. Count the end of a fiber obscured by another particle if the particle covering the fiber end is less than $3 \mu\text{m}$ in diameter.
5. Count free ends of fibers emanating from large clumps and bundles up to a maximum of 10 ends (5 fibers), provided that each segment meets rules 1 and 2 above.
6. Count enough graticule fields to yield 200 ends. Count a minimum of 20 graticule fields. Stop at 100 graticule fields, regardless of count.
7. Divide total end count by 2 to yield fiber count.

APPENDIX E: EQUIVALENT LIMITS OF DETECTION AND QUANTITATION

Fiber density on filter* (Fibers per 100 fields)	Fiber density on filter* (Fibers /mm²)	Fiber concentration in air, f/cc (400-L air sample)	Fiber concentration in air, f/cc (1000-L air sample)
200	255	0.25	0.10
100	127	0.125	0.05
LOQ 80	102	0.10	0.04
50	64	0.0625	0.025
25	32	0.03	0.0125
20	25	0.025	0.010
10	12.7	0.0125	0.005
8	10.2	0.010	0.004
LOD 5.5	7	0.00675	0.0027

*Assumes 385 mm² effective filter collection area, and field area = 0.00785 mm², for relatively “clean” (little particulate aside from fibers) filters

APPENDIX F: P&CAM 239 ASBESTOS FIBER IN AIR [17]

Analyte: Asbestos fibers	Method No.: P&CAM 239
Matrix: Air	Range: 0.1-60 fibers/cm ³
Procedure: Filter collection, microscopic count	Precision (CV_r): 0.24 to 0.38
Date Issued: 3/30/77	Classification: D (Operational)

1. Principle of the Method

- 1.1. This method describes the equipment and procedures for collecting, mounting, and counting asbestos fibers on cellulose ester membrane filters in the evaluation of personal samples of airborne asbestos fibers. The purpose of the method is to determine an employee's index of exposure to airborne asbestos fibers. The method is primarily a personal monitoring technique, but can be used for area monitoring.
- 1.2. The sample is collected by drawing air through a membrane filter by means of a battery powered personal sampling pump. The filter is transformed from an opaque solid membrane to a transparent optically homogeneous gel. The fibers are sized and counted using a phase-contrast microscope at 400-450X magnification.
- 1.3. Definitions. Asbestos fiber, for counting purposes, means a particulate which has a physical dimension longer than 5 micrometers and with a length to diameter ratio of 3 to 1 or greater. Asbestos includes chrysotile, cummingtonite-grunerite (amosite), crocidolite, fibrous tremolite, fibrous anthophyllite, and fibrous actinolite.
- 1.4. Any laboratory attempting to use this procedure should have at least one counter attend a training course conducted by an experienced, proficient laboratory. Novice, untutored counters, using only published instructions, can easily obtain counts of half those performed by experienced, proficient counters. Large differences between laboratories can be caused by: 1) differences in technique and observing ability among counters and 2) small, but significant, differences between microscopes meeting the basic specifications of Section 6.2. The following procedures are recommended:

- 1.4.1. All microscopists who perform asbestos counting should meet together for an "asbestos counting workshop" at least quarterly. This is best accomplished with counters from several laboratories using their own microscopes.
- 1.4.2. Each microscopist should count the same series of slides and with the results being compared.
- 1.4.3. Differences between counters should be resolved with side-by-side counting of the fields by the different counters.
- 1.4.4. Individuals who are found to be persistent outliers over several sessions should be encouraged to seek other tasks in their respective laboratories.

2. Range and Sensitivity

- 2.1. The usable range is primarily a function of sample volume, microscope count field area, and background airborne particulates. The influence of these variables is discussed in 8.1.3. For a microscope count field area of 0.003 mm^2 (see Figure 1) and a pump flow rate of 1.7 lpm, the optimal fiber densities would be produced over the range of 0.4 fiber/cm³ (8-hour sample) to about 60 fibers/cm³ (15-minute sample). For a field area of 0.006 mm^2 (see Figure 2) and a pump flow rate of 1.7 lpm, the optimal range is 0.2 fiber/cm³ (8-hour sample) to about 30 fibers/cm³ (15-minute sample). In each case, the optimal detection limits are inversely proportional to pump flow rate. The upper detection limit can be extended by using sample times less than 15 minutes or using lower flow rates. The lower detection limit can be extended by increasing the flow rate up to about 2.5 lpm. Filter surface fiber densities less than optimal (less than about 0.5 to 1.0 fiber per count field) are still adequate, but will lead to decreased precision for the method (increased coefficient of variation, see Section 4). The minimum total fiber count in 100 fields considered adequate for reliable quantitation is 10 fibers. Thus, the lower limit of reliable quantitation is 0.1 fiber/cm³ (100,000 fibers/m³). For this level, a flow rate of about 2.5 lpm is recommended. For a field area of 0.003 mm^2 , the minimum sample time would be about 2 hours. For a field area of 0.006 mm^2 , the minimum sample time would be about 1 hour.
- 2.2. This method considers only fibers with a length to diameter ratio of 3 to 1 or greater and a length greater than 5 micrometers.

3. Interferences

In an atmosphere known to contain asbestos, all particulates with a length to diameter ratio of 3 to 1 or greater, and a length greater than 5 micrometers should, in the absence of other information, be considered to be asbestos fibers and counted as such.

4. Precision and Accuracy

- 4.1. In the past decade, there have appeared a number of articles examining sources of variation in the asbestos sampling and counting procedure. These include: Lynch et al. (11.1), Weidner and Ayer (11.2), Conway and Holland (11.3), Leidel and Busch (11.4), Beckett and Attfield (11.5), and Rajhans and Bragg (11.6). The sources of variation will be discussed by stages in the membrane filter evaluation procedure.
- 4.2. **Sources of Variation in the Sampling Process.** These include variations in pump flow rate, proximity of the filter to the employee's body, and filter location (left to right) in the employee's breathing zone.
 - 4.2.1. Section 9.1 requires that the personal sampling pump be calibrated with sufficient accuracy such that the 95% confidence limits on the flow rate are $\pm 10\%$. This is equivalent to a coefficient of variation (CV) of about 5%. However, this CV makes a negligible contribution to the total CV for the method due to the relatively large CV of the counting procedure.
 - 4.2.2. Conway and Holland (11.3) concluded that positioning of the filter cassette on the wearer (regarding the angular portions of the filter and their proximity to the wearer) is not a significant factor in determining the fiber distribution on filters.
 - 4.2.3. Weidner and Ayer (11.2) concluded that there is no appreciable difference between samples collected on either the right or left sides of a breathing zone or between samples collected side-by-side, especially for samples with concentrations less than 2.5 fibers/cm³.

4.3. Sources of Variation in the Counting Procedure

4.3.1. Random variations exist in the fiber distribution on a filter wedge (intra-wedge variability). The industrial hygiene literature has seen considerable debate in the last 20 years concerning whether or not the distribution of mineral dust or asbestos fibers on a filter surface is adequately described by a Poisson distribution probability density function. Leidel and Busch (11.4) found excellent agreement between empirical error variance and theoretical variance calculated from the assumption of Poisson distributed true counts. They concluded that there was not excessive variation among count fields for a filter wedge and that clumping of fibers (non-random coalescence) did not occur.

4.3.2. Variations exist in the fiber distribution on the total filter surface (inter-wedge variability) due to the random or non-random distribution of fibers across the total surface of the filter. This type of variation is easily confused with intra-wedge variations. The count procedure does not require counting of multiple sectors of the filter. There may be significant differences between average counts for different wedges, or the fiber distribution variations for the total filter surface may be greater than the variations of the Poisson distribution. If either of these occur experimentally, one must use the experimental variations to estimate the minimum precision of the count procedure. The minimum precision is governed by the variations of the fiber distribution on the total surface of the filter.

Conway and Holhind (11.3) concluded the distribution of fibers on filters is not uniform and, the distribution of fiber counts is more disperse than Poisson. For their filters which had significant variations in fiber concentrations between sectors (as much as 50-60% of the total filter mean), they described the following relation for the standard deviation of the total number of fibers counted on a wedge (N)

$$\text{empirical } s(N) = 1.6(N)^{1/2}$$

where N is about 100. The Poisson standard deviation would be:

$$\text{Poisson } \sigma(N) = (N)^{1/2}$$

Rajhans and Bragg (11.6) in Series I of their study found significant variation between filter segments and rejected the Poisson distribution for the total filter surface. However, in Series II of their study, utilizing various experimental modifications, they found no significant variation between filter segments and no reason to reject the assumption of Poisson distributed fiber counts.

4.3.3. Systematic variations due to differences between microscopes were studied by Leidel and Busch (11.4). In their study using five different brands of microscopes, they found no significant differences among four, but the fifth gave counts approximately 45% higher on the average than the other four.

4.3.4. Variations due to differences between counters should be examined at three levels: experienced counters occasionally counting, experienced counters routinely counting, and inexperienced (new or untutored) counters. Leidel and Busch (11.4) studied five experienced counters, with one counting only occasionally. There were no significant differences among three of the counters, but a fourth was 16% lower than the first three. The fifth, who occasionally counted, averaged 27% higher than the first three. Conway and Holland (11.3) studied three experienced counters and three inexperienced counters. They found statistically significant differences between the means of both the experienced and inexperienced counters that typically were in the range plus or minus 5 to 15%. They concluded that experience as a fiber counter is not a significant parameter affecting intercounter variations. Rajhans and Bragg (11.6) found no significant differences among means of five experienced counters in Series I of their study. But in their carefully controlled Series II, an analysis of variance showed significant variations between counters that were plus or minus 1 to 15%.

4.3.5. Variations between laboratories are most likely due to systematic biases and are not a significant additional source of random variations. Any additional variations are most likely due to differences in counting technique. Beckett and Attfield (11.5) observed that standard

counters improved greatly after personal instruction; also new counters, after instruction, tended to overcompensate and get exceedingly high counts. Additionally, they found that counts from an experienced laboratory that had not had contact with other laboratories performing the same analysis were as far from the standard values as were the counts by new counters.

- 4.4. Sources of variations between samples taken at different times on one employee during one work shift can affect the exposure estimate for that employee. These are primarily due to a) differences in exposure concentrations during the day, b) differences in location of the employee within the plant, and c) differences in work operation performed by the employee during the day. These sources of variation can be controlled by proper choice of sampling strategy. Refer to Leidel and Busch (11.7) and Leidel, Busch, and Lynch (11.8) for an extended discussion of sampling strategies. Interday temporal variations can affect the exposure estimates obtained on different days. Refer to Leidel, Busch, and Crouse (11.9) for a discussion of this type of variation.
- 4.5. Until recently, the total coefficient of variation (CV_T) for the sampling and counting procedure was best estimated from the work of Conway and Holland (11.3). The conclusions of their study included:
 - 4.5.1. The precision of their procedure for filters not containing an abundance of fine fibers can be estimated by a coefficient of variation of 16.2%. This value includes variation among counters and observed interaction effects.
 - 4.5.2. The accuracy of the procedure for similar filters may be estimated for a 100-fiber count by a coefficient of variation of 21.4%. This assumes that the contribution of the overall variance from the nonuniform fiber distribution is additive.
 - 4.5.3. A high percentage of very fine fibers on the filter can significantly affect the standard deviation and confidence limits for counts by different counters. After combining variations in fiber concentrations over the entire filter with those for different counters, it was concluded:
 1. For filters with a low concentration of fine fibers, the coefficient of variation is estimated at 21% and the 95% confidence interval is $\pm 43\%$.
 2. For filters with a high concentration of fine fibers, the coefficient of variation is estimated at 25% and the 95% confidence interval is $\pm 50\%$.

Lynch, Kronoveter, and Leidel (11.1) have also reported on variations of the method. Their intralaboratory study utilized the data from a large number of dust counts made by different methods by experienced counters over a period of years in an epidemiologic study of the asbestos products industry. They concluded that the standard deviation of counts of fibers longer than 5 micrometers on membrane filters could be estimated from the relation $\sigma = (N)^{0.591}$. Thus for counts of about 100 fibers, the coefficient of variation could be estimated at about 15.2% and the 95% confidence limits at $\pm 30.4\%$.

These values are lower than the values reported by Conway and Holland (11.3). Recently, the Johns-Manville Corporation conducted an in-house investigation of the asbestos count method. (11.10). The study data contained total fiber counts for over 100 filters with each filter counted by two to five counters. From the Johns-Manville data, NIOSH calculated over 100 estimates of the count CV for the method (11.11). The NIOSH CV estimates included random intrafilter variations and intercounter variations, but did not include random pump flow rate variations. It was found that the count coefficient of variation (all random variations except for pump variations) was a function of the total fiber count. NIOSH then included a CV of 0.05 for random pump variations (see Section 9.1) in the CV-estimator equation to obtain a CV_T -estimator. The CV_T -estimator line is plotted on Figure 3 for total fiber counts in the range 10 to 100 fibers. Or the following equation can be used: $CV_T = [\text{antilog}_{10}(-0.215 - 0.203(\log_{10} FB)) + 0.0025]^2$ where FB is total fiber count as discussed in Section 10.

Figure 3 demonstrates that for a total fiber count of 100, the best CV_T is attainable with the appropriate sampling times given in 8.1.3 and the count rules in 8.3.9. When making

decisions regarding compliance with the OSHA asbestos exposure standards in 29 CFR 1910.1001, the statistical procedures given in Leidel et al. (11.11) should be followed. The procedures are based on statistical theory and assumptions given in References 11.12, 11.13.

Because of the possibility of systematic biases due to differences between microscopes, counters, and laboratories as discussed above, it is strongly recommended that any laboratory counting asbestos should participate in an interlaboratory quality control program that includes the counting of standard reference filters. These standard filters are available from NIOSH through the Proficiency Analytical Testing (PAT) Program. The PAT Program is used by the American Industrial Hygiene Association (AIHA) as part of its Laboratory Accreditation Program. Each laboratory's quality control program must include protocols for routinely adjusting, and calibrating sampling and counting equipment plus training and evaluation programs for counters.

5. **Advantages and Disadvantages of the Method**

- 5.1. The method is intended to give an index of employee exposure to airborne asbestos fibers of specified dimensional characteristics.
- 5.2. It is not meant to count all asbestos fibers in all size ranges or to differentiate asbestos from other fibrous particulates.

6. **Apparatus**

6.1. Sampling Equipment

The personal sampling equipment train consists of 1) personal sampling pump, 2) tubing, 3) clothing spring clip, 4) tubing-to-field monitor metal adaptor, and 5) field monitor (filter and holder).

- 6.1.1. Personal Sampling Pump. The pump must be capable of sampling at 1.0 to 2.5 liters per minute (lpm) against a flow resistance of 7.5 inches of water (1.4 mm Hg) for 8 continuous hours on a fully charged battery.
- 6.1.2. Tubing. Laboratory tubing such as rubber or plastic with 6-mm bore and about 100 cm length.
- 6.1.3. Clothing Spring Clip. The clip attaches the rubber tubing to the lapel or shirt of the individual being monitored.
- 6.1.4. Tubing-to-field Monitor Adaptor. A short metal adaptor with ridges on one end to grip the inside of the tubing. The other end is designed for a pressure fit into the field monitor.
- 6.1.5. Field Monitor (Filter and Holder). The only field monitor currently considered acceptable by NIOSH is manufactured by the Millipore Corporation. The unit consists of 1) a three section styrene plastic case designated Millipore Aerosol Monitor Case, 2) a 37-mm diameter plain white cellulose ester membrane filter designated Millipore AA (pore size of 0.8 micrometer), 3) a support pad, and 4) two plastic sealing caps. If a large number of samples are to be taken, it may be less expensive to reuse the plastic cases. Great care must be taken in the cleaning and reassembly process. The outside mating surfaces of the field monitors may be covered with a "shrink-fit" band to provide proper sealing and a writing surface for filter identification.

6.2. **Optical Equipment and Microscope Features**

- 6.2.1. Microscope body with binocular head.
- 6.2.2. 10X Huygenian eyepieces are recommended. Other eyepieces can be substituted if necessary. Wide field eyepieces can be used; however, wide field eyepieces may yield a count field area less than 0.003 mm² with the Porton reticle. This is not always desirable from the standpoint of obtaining optimum sampling times (see Section 8.1.3). If wide field eyepieces are used, it is preferable to use the Patterson Globe and Circle reticle to obtain a larger count field area.
- 6.2.3. Koehler illumination (preferably built-in with provisions for adjusting light intensity).
- 6.2.4. A Porton reticle is recommended. Others such as the Patterson Globe and Circle can be substituted.
- 6.2.5. Mechanical stage.

- 6.2.6. Phase-Contrast condenser with a numerical aperture (N.A.) equal to or greater than the N.A. of the objective.
- 6.2.7. 40-45X phase contrast achromatic objective (N.A. 0.65 to 0.75).
- 6.2.8. Phase-ring centering telescope or Bertrand lens.
- 6.2.9. Green or blue filter, if recommended by microscope manufacturer.
- 6.2.10. Stage micrometer with 0.01 mm subdivisions.
- 6.2.11. For general guidance on phase contrast microscopy, consult Needham (11.12), Clark (11.15) and McCrone (11.14).

6.3. **Filter Mounting Equipment.** Experience has shown that certain equipment is useful for efficient sample mounting. The following items are recommended for extracting and mounting a portion of the filter for counting.

- 6.3.1. Microscope slides. 2.5 by 7.5 cm glass slides are most commonly used. Sample number, data, initials, etc., can be conveniently written on a frosted endslide.
- 6.3.2. Cover Slips. Cover slips are a necessary part of the slide mount and optical system. The shape should be appropriate for the size of the filter wedge. The appropriate cover slip depends upon the objective to be used. Ordinarily, objectives are optically corrected for a #1½ (0.17 millimeter) thickness cover slip. Improper cover glass thickness will detract from the final image quality.
- 6.3.3. Scalpel. A scalpel is needed to cut out a portion of the filter to be examined. A number-ten curved blade scalpel is recommended.
- 6.3.4. Tweezers. A pair of fine-tipped tweezers is used to remove the membrane filter slice from the field monitor and place it upon the slide.
- 6.3.5. Lens Tissue. To insure cleanliness, a lint-free tissue is recommended. This tissue should also be used for wiping mounting tools and for cleaning slides and coverslips.
- 6.3.6. Glass Rod. A fire-polished glass rod may be used to spread the mounting solution on the slide.
- 6.3.7. Wheaton Balsam Bottle. This special glass container has a glass top which prevents contamination of the mounting solution. A glass rod is included for dispensing the solution.

7. Reagents

Chemicals should be reagent grade, free from particles and color, conforming to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.

- 7.1. Dimethyl phthalate
- 7.2. Diethyl oxalate

Avoid getting the mounting solution on the skin. Wash skin promptly with soap and water if skin contact occurs.

8. Procedure

8.1. Sampling

8.1.1. General Information

Guidelines for the monitoring of employee exposures to industrial atmospheres are given in Reference 11.8. The Federal requirements for monitoring employee exposure to airborne asbestos are found in 29 CFR 1910.1001.

8.1.2. Mounting the Sampling Pump on the Worker

Fasten the sampling pump to the worker's belt and fasten the field monitor to the lapel or shirt front (as close to the breathing zone as is practical). Remove the top cover of the plastic monitor, then invert the monitor making certain the exposed filter is facing downward. Turn the pump on and adjust to the calibrated flow rate (1.0 to 2.5 lpm). Record the following information in a logbook.

- 1. Filter number
- 2. Pump start time and date
- 3. Flow rate
- 4. Subject's name and job title

5. Type of operation or process
 6. Ventilation controls and is the worker wearing a respirator approved for asbestos?
- The pump should be checked periodically during the sampling period for proper operation and flow rate.

8.1.3. Optimum Sampling Times

The requirement for the minimum count of 100 fibers or 20 fields in 8. 3. 9 was determined to be the best compromise to achieve adequate precision for the airborne fiber estimate and reasonable counting times. An optimum fiber density of about 1 to 5 fibers per microscope count field is recommended. To estimate appropriate sampling times for feasible counting and optimal counting, one must consider the following constraints:

1. microscope count field area (generally 0.003 to 0.006 mm²)
2. pump flow rate (typically 2.5 lpm maximum)
3. average airborne fiber concentrations
4. counting rule range of 20 to 100 fields
5. adequate fiber density to obtain a minimum count of 10 fibers in 100 fields, which is the least total fiber count that yields an acceptable count precision
6. background airborne particulate levels that can reduce the count precision due to an obscuring of fibers on the filter surface

The preceding constraints were considered in drawing Figures 1 and 2. These figures were developed from the following relationship:

$$\text{sampling time} = \frac{\left(\frac{FB}{FL}\right) \left(\frac{ECA}{MFA}\right)}{(FR)(AC)(1000)} \text{ minutes}$$

where:

FB/FL = 1 to 5 fibers/field

ECA = effective collecting area of filters (855 mm² for 37-mm filter with effective diameter of 33 mm)

MFA = microscope field area (generally 0.003 to 0.006 mm²)

FR = Pump flow rate (generally 1.0 to 2.5 lpm)

AC = Air concentration of fibers in fibers/cm³.

Figure 1 (microscope field area = 0.003 mm²) and Figure 2 (microscope field area = 0.006 mm²) show optimum and feasible sampling times for a pump flow rate of 1.7 lpm. Each individual responsible for sampling asbestos should prepare a similar chart for his particular pump flow rate and microscope field area before sampling is performed to aid in estimating proper sampling times. On Figures 1 and 2, the areas with solid shading lines are generally the optimum conditions for counting. The broken shading lines are for conditions very close to optimal.

However, feasible counting conditions may extend down to about 0.1 fiber/field and above 5 fibers/field. Recommended sampling times are most strongly influenced by background airborne particulate levels, once all the other constraints have been estimated. For heavy particulate levels, it may be necessary to limit each filter to about 60 to 180 minutes sampling duration. Each individual responsible for sampling should work closely with the microscopist to attain as high as possible filter surface fiber densities (up to about 5 fibers/field), while avoiding filter surface background particulate levels that create very difficult or impossible counting conditions. If one has very little idea of airborne fiber and particulate levels, the best procedure is to take several long samples (as one 8-hour or two consecutive 4-hour samples) in conjunction with several short samples (as four consecutive 2-hour or eight consecutive 1-hour samples). If the longer samples prove very difficult to count, the microscopist will have the shorter samples to fall back on.

From Figures 1 and 2, it can be seen that there are certain sampling times which will yield optimum fiber densities on the filter for almost all airborne fiber concentrations from 1 to 10 fibers/cm³. These optimum times have been calculated and are presented in Figure 4. Note

that the optimum times given by Figure 4 are approximate and can be varied by as much as $\pm 25\%$. The nomogram is intended as a guide to be used where no prior knowledge of the air concentration is available.

8.1.4. End of Sampling Period

Remove the field monitor, replace the plastic top cover and the small end caps, and store the monitor. Always shut off the pump when changing monitors to avoid contaminating or damaging the pump. Record the pump shutoff time and flow rate in the logbook.

8.1.5. Blanks

With each batch (25 to 50 filters) of samples sent for analysis, submit two unopened field monitors which have been subjected to the same treatment as the samples except that they were not exposed to the sampling environment. Label these as blanks. If the blanks yield fiber counts greater than 5 fibers/ 100 fields, then the entire sampling procedure should be examined carefully for the cause of contamination. The mounting solution of Section 8.2.1 should also be examined for contamination and/or crystal growth.

8.1.6. Shipping

The field monitors in which the samples are collected should be shipped in a rigid container with sufficient packing material to prevent crushing.

8.1.7. Numbers of Samples

When sampling for the Federal ceiling standard of 10 fibers ($>5 \mu\text{m}$)/ cm^3 , [29 CFR 1910.1001(b) (3), effective July 7, 1972], only one sample (15 minutes maximum duration) is necessary, theoretically. However, several samples should be taken during expected periods of peak air concentrations to allow for detection of gross sampling or counting errors.

When sampling for determination of noncompliance with the Federal 8-hour TWA standard of 2 fibers ($>5 \mu\text{m}$)/ cm^3 , [29 CFR 1910.1001(b) (2)], one should continuously sample as large a portion of the work day as is feasible for airborne concentrations of about 2 to 10 fibers/ cm^3 . However, for a lower airborne concentration such as 0.5 fiber/ cm^3 , one sample might require 4 to 8 hours sampling time in order to get the proper filter fiber density (Section 8.1.3). For this situation, the 8-hour TWA exposure would be determined from one 8-hour or two 4-hour samples as appropriate.

8.2. Sample Preparation

8.2.1. Preparation of Mounting Solution

A very important part of the sample evaluation is the mounting process. This process involves a special mounting medium of prescribed viscosity. The proper viscosity is important in order to expedite filter dissolving and still minimize particle migration. After the sample has been mounted, an elapsed time of approximately sixty minutes is needed before the sample is ready for evaluation.

Combine the dimethyl phthalate and diethyl oxalate in a one to one ratio by volume and pour into a Wheaton balsam bottle. Add approximately 0.05 (± 0.005) grams of new membrane filter per milliliter of solution to reach the necessary viscosity. The mixture must be stirred periodically until the filters have dissolved and a homogeneous mixture is formed. The normal shelf life of the mounting solution is about three months. Twenty milliliters of mounting solution will prepare approximately 300 samples.

8.2.2. Sample Mounting

Cleanliness is important! A dirty working area may result in sample contamination and erroneous counts. The following steps should be followed when mounting a sample.

1. Clean the slides and cover slips with lens tissue. Lay each slide down on a clean surface with the frosted end up. It is a good practice to rest one edge of the cover slip on the slide and the other edge on the working surface. By doing this, you keep the bottom surface (the one which contacts the filter) from becoming contaminated.
2. Wipe all the mounting tools clean with lens tissue and place them on a clean surface (such as lens tissue). All tools should be wiped clean prior to mounting each sample.

3. Using the glass rod supplied with the Wheaton balsam bottle, apply a drop of mounting solution onto the center of the slide. It may be necessary to adjust the quantity of solution so that after the cover slip has been placed on top, the solution extends only slightly beyond the filter boundary. If the quantity is greater than this, particle migration may occur.
4. Using another glass rod, spread the mounting media into a triangular shape. The size of this triangle should coincide with the dimension of the filter wedge.
5. Separate the middle and bottom sections of the field monitor case to expose the filter. Cut a triangular wedge from the center to the edge of the filter using the scalpel. The size of the wedge should approximate one-eighth of the filter surface. The filter can be very carefully removed from the cassette for cutting, but this should only be done with great care.
6. Grasp the filter wedge with the tweezers on the perimeter of the filter which was clamped between the monitor case sections. Do not touch the filter with your fingers. Place the wedge, **sample side up**, upon the mounting medium.
7. Pick up a clean cover slip with tweezers and carefully place it on the filter wedge. Once this contact has been made, **do not reposition the cover slip**.
8. Label the slide with the sample number and current date before proceeding to the next filter: On the bottom (backside) of the slide, trace the perimeter of the filter wedge with a felt tip marking pen. This will enable the counter, after the filter has become transparent, to stay within the filter perimeter when counting.
9. The sample should become transparent within fifteen minutes. If the filter appears cloudy, it may be necessary to press very lightly on the cover slip. This is rarely necessary; however, counting should not be started until an hour after the mounting. This allows the microscopic texture of the filter to become invisible to microscope viewing.
10. Discard the sample mount after two days if it has not been counted. Crystals appearing similar to asbestos fibers may begin to grow at the mounting media/air interfaces. They seldom present any problems if the slide is examined before two days. In any case, stay away from the filter's edges when counting and sizing.

8.3. Counting of Fibers

- 8.3.1. Place the slide on the mechanical stage of the microscope and position the center of the wedge under the objective lens and focus upon the sample. Start counting from one end of the wedge and progress along a radial line to the other end (count in either direction from perimeter to wedge tip). Random fields are selected, without looking into the eyepieces, by slightly advancing the slide in one direction with the mechanical stage control.
- 8.3.2. It is essential to continually scan over a range of focal planes (generally the upper 10 to 15 micrometers of the filter surface) with the fine focus control during each field count. This is especially necessary for asbestos fibers due to their impaction into the filter matrix.
- 8.3.3. On most airborne samples, asbestos fibers will generally have fiber diameters less than one micrometer. Therefore, it is necessary to look carefully for faint fiber images.
- 8.3.4. Regularly check phase ring alignment.
- 8.3.5. When an agglomerate (mass of material) covers a significant portion of the field of view (approx 1/6 or greater)--reject the field and select another. (Do not include it in the number of fields counted.) However, report the fact as it may have meaning on other data collection.
- 8.3.6. Bundles of fibers are counted as one fiber unless both ends of the fiber can be clearly resolved.
- 8.3.7. Count only fibers with a length to width ratio greater than or equal to 3:1.
- 8.3.8. Count only fibers greater than 5 micrometers in length. (Be as accurate as possible in accepting fibers near this length.) Measure curved fibers along the curve to estimate the total length.
- 8.3.9. Count as many fields as necessary to yield a total count of at least 100 fibers. Exceptions: a) count at least 20 fields even if you count more than 100 fibers, and b) stop at 100 fields even if you haven't reached 100 fibers.

8.3.10. For fibers that cross either one or two sides of the counting field, the following procedure is used to obtain a representative count.

COUNT any fiber greater than 5 micrometers in length that lies entirely within the counting area. COUNT as "1/2 fiber" any fiber with only one end lying within the counting area. DO NOT COUNT any fiber crossing any two sides.

Reject and do not count all other fibers. Refer to Figures 5 through 10. Note that the fibers in Figures 5 through 10 **are not representative** of the appearance of most asbestos fibers. Most fibers have a very faint image.

9. Calibration and Standards

9.1. Sampling Train Calibration

The accurate calibration of the sampling pump is essential to the correct calculation of the air volume sampled. The frequency of calibration is dependent on the use, care, and handling to which the pump is subjected. Pumps must be recalibrated if they have just been repaired, misused, or received from the manufacturer. If the pump receives hard usage, more frequent calibration may be necessary. Ordinarily, pumps should be calibrated in the laboratory both before they are used in the field and after they have been used to collect a large number of field samples.

The accuracy of calibration is dependent upon the type of instrument used as a reference. The choice of a calibration instrument will depend largely on where the calibration is performed. For laboratory testing, a 1-liter buret used as a soap bubble flow meter or wet-test meter is recommended. Other standard calibrating instruments, such as a spirometer, Marriott's bottle, or dry gas meter can be used. The calibration should be of sufficient precision that the 95% confidence limits on the flow rate are $\pm 10\%$ (95% of the flow rates will fall within $\pm 10\%$ of the calibrated value). Instructions for calibration with the soap bubble flow meter follow. The sampling train used (pump, hose, filter cassette) in the pump calibration should be the same as the one used in the field.

9.1.1. Check the voltage of the pump battery with a voltmeter both with the pump off and while it is operating to assure adequate voltage for calibration. If necessary, charge the battery to manufacturer's specifications.

9.1.2. Fill a beaker with 10 ml of soap solution.

9.1.3. Connect the filter cassette inlet to the top of the buret with a length of hose.

9.1.4. Turn the pump on and moisten the inside of the soap bubble meter by immersing the open end of the buret into the soap solution and drawing bubbles up the inside of the buret. Perform this task until the bubbles are able to travel the entire length of the buret without breaking.

9.1.5. Adjust the pump rotameter to provide a flow between 1.5 to 2.5 lpm.

9.1.6. With a water manometer, check that the pressure drop across the filter is less than 13 inches of water (about 1 inch of mercury).

9.1.7. Start a soap bubble up the buret and measure the time it takes for the bubble to travel a minimum volume of 1 liter.

9.1.8. Repeat the procedure in 9.1.7 at least three times, average the results, and calculate the calibrated flowrate by dividing the volume traveled by the soap bubble by the elapsed time. If the range between the highest and lowest of the three flow rates is greater than about 0.33 lpm, then the calibration should be repeated since it is likely that the precision is not adequate.

9.1.9. Data required for the calibration include the volume measured, elapsed time, pressure drop, air temperature, atmospheric pressure (or elevation), pump serial number, date, and name of person performing the calibration.

9.1.10. Corrections to the flow rate for pumps with rotameters may be necessary if the pressure (elevation) or temperature where the samples are collected (actual flow rate) differs significantly from that where the calibration was performed (indicated flow rate). Actual flow rates at time of sampling may be calculated for a linear scale rotameter by using the following correction formula:

$$Q_{actual} = Q_{indicated} \sqrt{\left(\frac{P_{cal}}{P_{actual}}\right) * \left(\frac{T_{actual}}{T_{cal}}\right)}$$

where both pressure (P) and temperature (T) are in absolute units such as:

psia = psig + 14.7

deg Rankin = deg Fahrenheit + 460

deg Kelvin = deg Celsius + 273

9.2. Microscope Setup

9.2.1. Porton Reticle and the Counting Field

The asbestos fiber count procedure consists of comparing fiber length to the diameters of calibrated circles of a Porton reticle, and counting all fibers greater than 5 micrometers in length lying within a given counting field area. The Porton reticle is a glass plate inscribed with a series of circles and rectangles. The left half of the reticle is divided into six rectangles constituting the counting field. The counting field is illustrated in Figures 5 through 10.

9.2.2. Placement in Eyepiece

The Porton reticle is placed inside the Huygenian eyepiece where it rests on the field limiting diaphragm. If other types of eyepieces are used, it may be necessary to insert a counting collar for retaining the reticle. The reticle should always be kept clean, since dirt on the reticle is in focus and could complicate the counting and sizing process.

9.2.3. Stage Micrometer

The Porton reticle cannot be used for counting until it has been properly calibrated with a stage micrometer. Most stage micrometer scales are approximately two millimeters long and are divided into units of one-hundredth of a millimeter (ten micrometers).

9.2.4. Microscope Adjustment

When adjusting the microscope, follow the manufacturer's instructions while observing the following guidelines.

1. The light source image must be in focus and centered on the condenser iris or annular diaphragm.
2. The particulate material to be examined must be in focus.
3. The illuminator field iris must be in focus, centered on the sample, and opened only to the point where the field of view is illuminated.
4. The phase rings (annular diaphragm and phase-shifting elements) must be concentric.

9.2.5. Porton Reticle Calibration Procedure

Each eyepiece-objective-reticle combination on the microscope must be calibrated. Should any of the three be changed (disassembly, replacement, zoom adjustment, etc.), the combination must be recalibrated. Calibration may change if interpupillary distance is changed. For proper calibration, the following procedure should be followed closely. With a 10X objective in place, place the stage micrometer on the mechanical stage, focus the millimeter scale, and center the image. Change to the 40-45X objective and adjust the first millimeter scale division to coincide with the left boundary of the Porton rectangle. Measure the distance between the left and extreme right boundaries of the Porton rectangle, estimating any portion of the final division. This measurement represents 200 L units. The rectangle is 100 L units on the short vertical dimension. The calculated "L" is inserted into the formula $D = L(2^N)^{1/2}$ where "N" is the circle number (indicated on the reticle) and "D" is the circle diameter. Since the circle diameters vary logarithmically, every other circle doubles in diameter. For example, circle number three is twice the diameter of number one; number four is twice the diameter of number two. When the circle sizes have been determined, the counting field area which consists of the left six smaller rectangles can be calculated from the relation $10,000 L^2$. This completes the reticle calibration for this specific objective-eyepiece-reticle combination.

Example for Porton Reticle

The following calibration was obtained for a pair of 10X Huygenian eyepieces and a 43X objective:

200 L = 0.148 mm = 148 micrometers

100 L = 0.074 mm = 74 micrometers

One L-unit = 0.74 micrometers

Thus Circle #1 has a diameter $D = L(2^N)^{1/2} = 0.74(2^1)^{1/2} = 0.74(1.414) = 1.05$ micrometers.

Then our circle diameter calibration table looks like:

Diameter of Circle #1 = 1.05 micrometers

#2 = 1.48

#3 = 2.09

#4 = 2.96

#5 = 4.19

#6 = 5.92

Field area = (10,000) (L²) = (100 L) (100 L) = (0.074) (0.074) = 0.0055 mm²

Thus fibers with a length greater than a distance halfway between the diameters of the #5 and #6 circles would be counted.

If a Patterson Globe and Circle reticle is used, a different calculation procedure is required. The circle diameters are related as follows. The #25 circle diameter is (0.1) (reticle length).

The circle diameters are proportional to the ratio of their numbers. Thus the #20 circle diameter is (20/25) or 0.8 times the #25 circle diameter.

10. Calculations

- 10.1. The average airborne asbestos fiber concentration estimated by the filter sample may be calculated from the following formula:

$$AC = \frac{\left(\left(\frac{FB}{FL} \right) - \left(\frac{BFB}{BFL} \right) \right) (ECA)}{(1000)(FR)(T)(MFA)}$$

where:

AC = Airborne fiber concentration in (fibers >5 µm)/cm³.

BFB = Total number of fibers counted in the BFL fields of the blank or control filters in fibers >5 µm.

BFL = Total number of fields counted on the blank or control filters.

ECA = Effective collecting area of filter (855 mm² for a 37-mm filter with effective diameter of 33 mm).

FR = Pump flow rate in liters/min (lpm).

FB = Total number of fibers counted in the FL fields in fibers >5 µm.

FL = Total number of fields counted on the filter.

MFA = Microscope count field area in mm² (generally 0.003 to 0.006).

T = Sample collection time in minutes.

- 10.2. Recount criteria. It is very desirable for a counter to conduct a "blind recount" for about 1 in every 10 filter wedges (slides) counted. Alternatively, a second counter could perform the blind recount. In training sessions for novice counters, the trainee should conduct a blind recount for filter wedges counted by an experienced, proficient counter. In all cases, we will observe differences between the first and second counts of the same filter wedge. Most of these differences will be due to chance alone, that is, due to the random variability (precision) of the count method. Statistical recount criteria enable us to decide whether observed differences can reasonably be explained due to chance alone or are probably due to systematic differences between counters or microscopes or due to some other biasing factor.

The following recount criterion is for a pair of counts that estimate some airborne fiber concentration (AC) in fibers/cm³. The criterion is given at the type-1 error level. That is, there is a 5% maximum risk that we will reject a pair of counts for the reason that one might be biased, when the large observed difference is really due to chance.

Reject a pair of counts because one might be biased if:

$$(AC_2 - AC_1) \text{ exceeds } 2.77(\overline{AC})(CV_{FB})$$

where:

AC_1 = lower estimated airborne fiber concentration

AC_2 = higher estimated airborne fiber concentration

$\overline{AC}$ = average of the two airborne concentration estimates

CV_{FB} = average CV for the two concentration estimates which are a function of the total fiber count (FB) in each case. Use the relation in Section 4 or Figure 3.

For a pair of counts on the same filter, reject the pair because one might be biased if:

$$(FB_2 - FB_1) \text{ exceeds } 2.77(\overline{FB})(CV_{FB}^-)$$

where:

FB_1 = lower fiber count on the filter (total fibers)

FB_2 = higher fiber count on the filter (total fibers)

$\overline{FB}$ = average of the two total fiber counts

$CV_{FB}^- = CV_T$ for the value FB. Use the relation in Section 4 or Figure 3.

11. References

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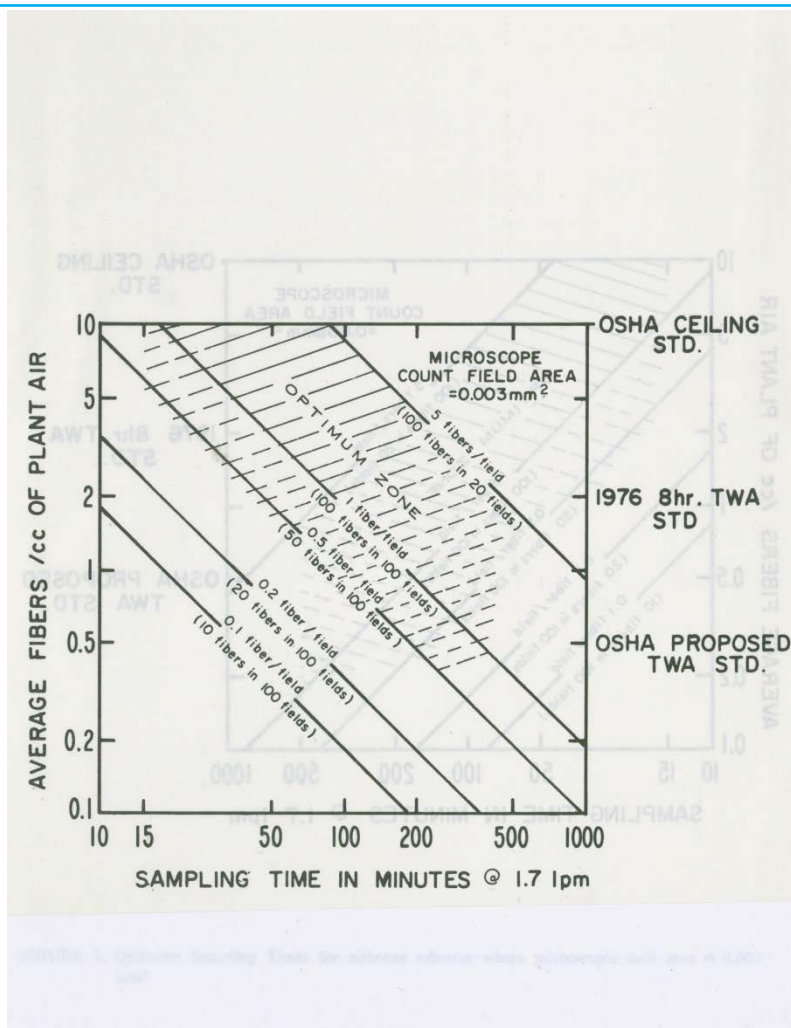


Figure 1 Optimum Sampling Times for airborne asbestos where microscopic field area = 0.003 mm²

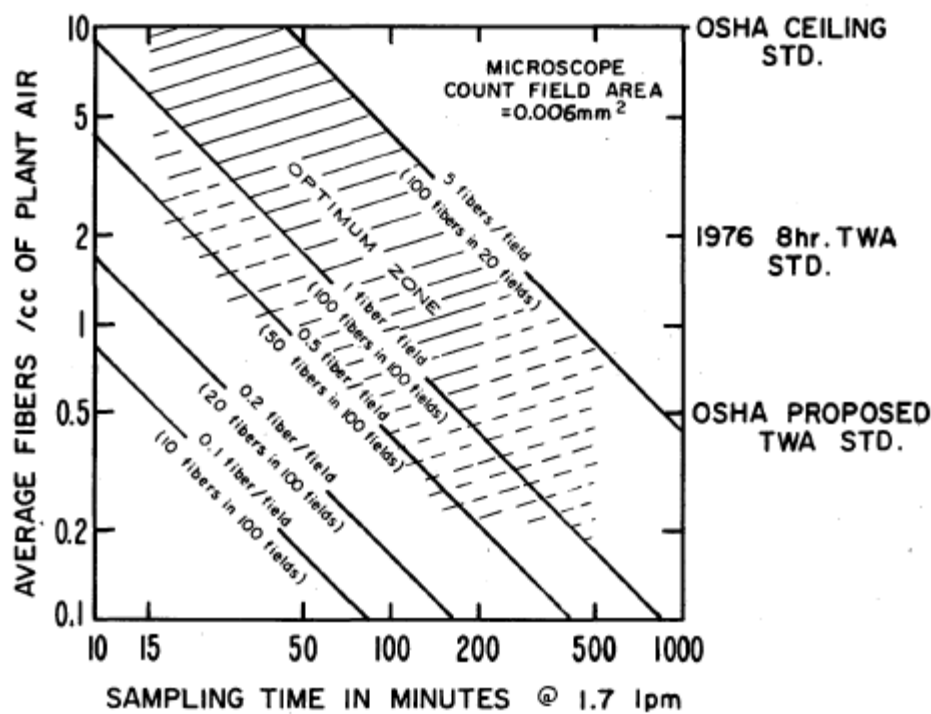


Figure 2 Optimum sampling times for airborne asbestos where microscopic field area = 0.006 mm²

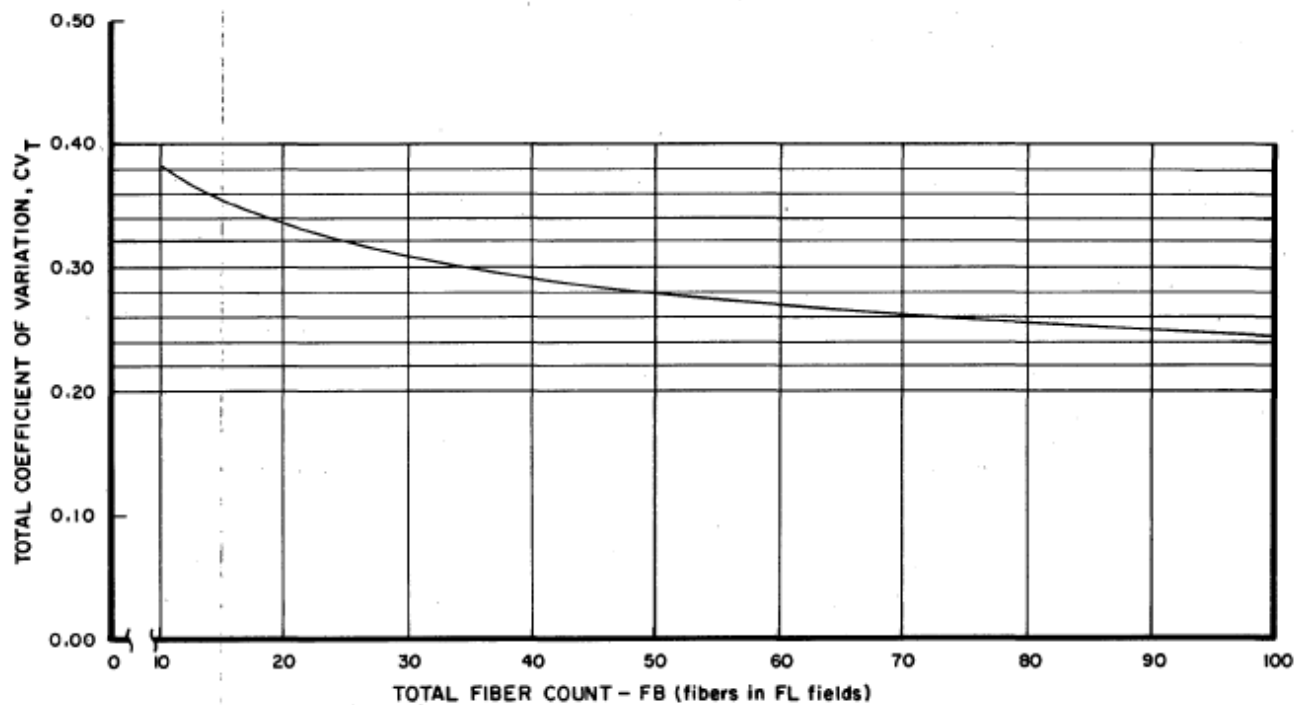


Figure 3 Total coefficient of variation as a function of total fiber count

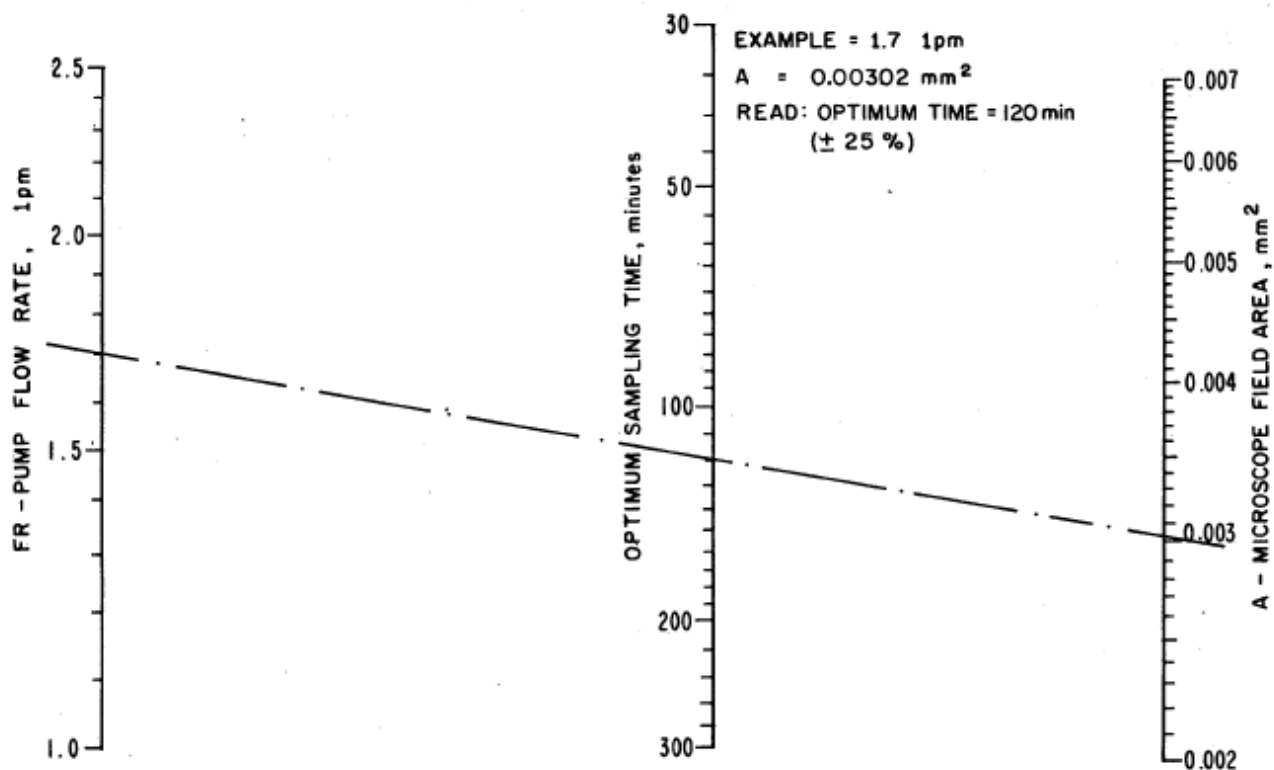


Figure 4 Nomogram of optimum sampling times for airborne asbestos fibers in concentrations of 1 to 10 fibers/cm³

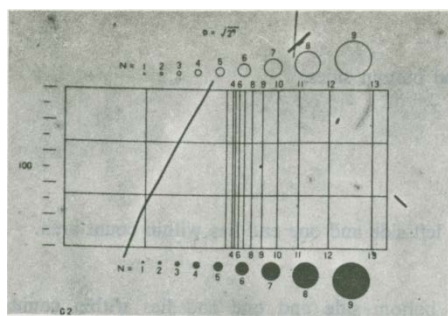


Figure 5 DO NOT COUNT. Fiber crosses top and bottom sides.

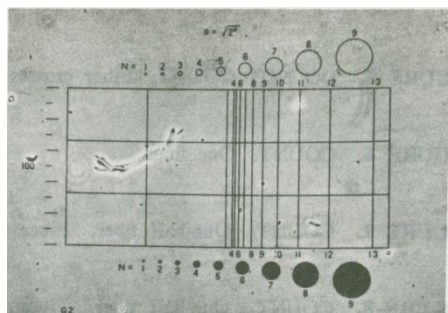


Figure 6 COUNT. One fiber.

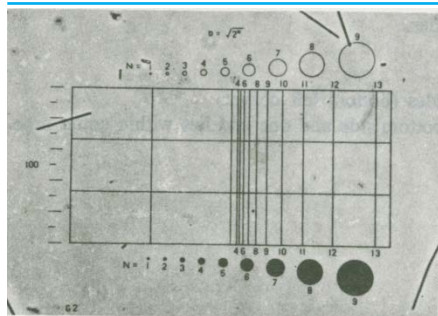


Figure 7 COUNT. One-half fiber. Fiber crosses left side and one end lies within the count area.

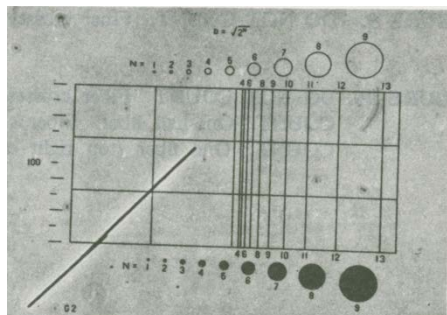


Figure 8 COUNT. One-half fiber. Fiber crosses bottom side and one end lies within count area.

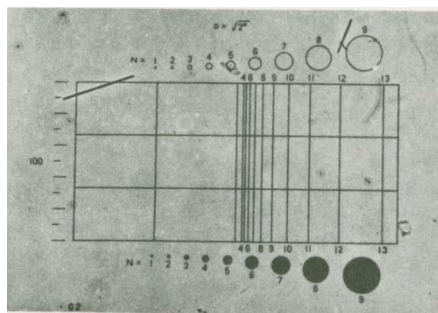


Figure 9 DO NOT COUNT. Fiber crosses two sides.

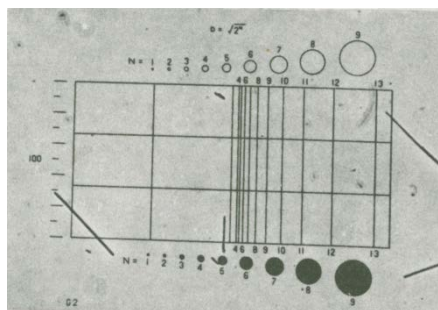


Figure 10 DO NOT COUNT. Fiber crosses two sides (bottom left corner). COUNT. One-half fiber. Fiber crosses bottom side and one end lies within count area. COUNT. One fiber (top right corner).

FORMULA: Various

MW: Various

CAS: Various

RTECS: Various

METHOD: 7402

EVALUATION: PARTIAL

Issue 1: 15 May 1989

Issue 2: 15 August 1994

OSHA : 0.1 asbestos fibers (>5 µm long)/cc;
1 f/cc/30 min excursion; carcinogen

MSHA: 2 asbestos fibers/cc

NIOSH: 0.1 f/cc (fibers > 5 µm long)/400 L; carcinogen

ACGIH: 0.2 crocidolite; 0.5 amosite; 2 chrysotile
and other asbestos, fibers/cc; carcinogen

PROPERTIES: solid, fibrous, crystalline,
anisotropic

SYNONYMS [CAS#]: actinolite [77536-66-4] or ferroactinolite [15669-07-5]; amosite [12172-73-5]; anthophyllite [77536-67-5]; chrysotile [12001-29-5]; serpentine [18786-24-8]; crocidolite [12001-28-4]; tremolite [77536-68-6]; amphibole asbestos [1332-21-4].

SAMPLING		MEASUREMENT	
SAMPLER:	FILTER (0.45- to 1.2-µm cellulose ester membrane, 25-mm diameter; conductive cassette)	TECHNIQUE:	MICROSCOPY, TRANSMISSION ELECTRON (TEM)
FLOW RATE:	0.5 to 16 L/min	ANALYTE:	asbestos fibers
VOL-MIN*:	400 L @ 0.1 fiber/cc	SAMPLE PREPARATION:	modified Jaffe wick
-MAX*:	(step 4, sampling) *Adjust for 100 to 1300 fibers/mm ²	EQUIPMENT:	transmission electron microscope; energy dispersive X-ray system (EDX) analyzer
SHIPMENT:	routine (pack to reduce shock)	CALIBRATION:	qualitative electron diffraction; calibration of TEM magnification and EDX system
SAMPLE STABILITY:	stable	RANGE:	100 to 1300 fibers/mm ² filter area [1]
BLANKS:	2 to 10 field blanks per set	ESTIMATED LOD:	1 confirmed asbestos fiber above 95% of expected mean blank value
ACCURACY		PRECISION ($\hat{S}_p$):	0.28 when 65% of fibers are asbestos; 0.20 when adjusted fiber count is applied to PCM count [2].
RANGE STUDIED:	80 to 100 fibers counted		
BIAS:	not determined		
OVERALL PRECISION ($\hat{S}_{pr}$):	see EVALUATION OF METHOD		
ACCURACY:	not determined		

APPLICABILITY: The quantitative working range is 0.04 to 0.5 fiber/cc for a 1000-L air sample. The LOD depends on sample volume and quantity of interfering dust, and is <0.01 fiber/cc for atmospheres free of interferences. This method is used to determine asbestos fibers in the optically visible range and is intended to complement the results obtained by phase contrast microscopy (Method 7400).

INTERFERENCES: Other amphibole particles that have aspect ratios greater than 3:1 and elemental compositions similar to the asbestos minerals may interfere in the TEM analysis. Some non-amphibole minerals may give electron diffraction patterns similar to amphiboles. High concentrations of background dust interfere with fiber identification. Some non-asbestos amphibole minerals may give electron diffraction patterns similar to asbestos amphiboles.

OTHER METHODS: This method is designed for use with Method 7400 (phase contrast microscopy).

REAGENTS:

1. Acetone. (See SPECIAL PRECAUTIONS.)

EQUIPMENT:

1. Sampler: field monitor, 25-mm, three-piece cassette with ca. 50-mm electrically-conductive extension cowl, cellulose ester membrane filter, 0.45- to 1.2- μ m pore size, and backup pad.
NOTE 1: Analyze representative filters for fiber background before use. Discard the filter lot if mean count is >5 fibers/100 fields. These are defined as laboratory blanks.
NOTE 2: Use an electrically-conductive extension cowl to reduce electrostatic effects on fiber sampling and during sample shipment. Ground the cowl when possible during sampling.
NOTE 3: 0.8- μ m pore size filters are recommended for personal sampling. 0.45- μ m filters are recommended for sampling when performing TEM analysis on the samples because the particles deposit closer to the filter surface. However, the higher pressure drop through these filters normally preclude their use with personal sampling pumps.
2. Personal sampling pump, 0.5 to 16 L/min, with flexible connecting tubing.
3. Microscope, transmission electron, operated at ca. 100 kV, with electron diffraction and energy-dispersive X-ray capabilities, and having a fluorescent screen with inscribed or overlaid calibrated scale (Step 15).
NOTE: The scale is most efficient if it consists of a series of lines inscribed on the screen or partial circles every 2 cm distant from the center.
4. Diffraction grating replica with known number of lines/mm.
5. Slides, glass, pre-cleaned, 25- x 75-mm.
6. Knife, surgical steel, curved-blade.
7. Tweezers.
8. Grids, 200-mesh TEM copper, (optional: carbon-coated).
9. Petri dishes, 15-mm depth. The top and bottom of the petri dish must fit snugly together. To assure a tight fit, grind the top and bottom pieces together with an abrasive such as carborundum to produce a ground-glass contact surface.
10. Foam, clean polyurethane, spongy, 12-mm thick.
11. Filters, Whatman No. 1 qualitative paper or equivalent, or lens paper.
12. Vacuum evaporator.
13. Cork borer, (about 8-mm).
14. Pen, waterproof, marking.
15. Reinforcement, page, gummed.
16. Asbestos standard bulk materials for reference; e.g. SRM #1866, available from the National Institute of Standards and Technology.
17. Carbon rods, sharpened to 1 mm x 8 mm.
18. Microscope, light, phase contrast (PCM), with Walton-Beckett graticule (see method 7400).
19. Grounding wire, 22-gauge, multi-strand.
20. Tape, shrink- or adhesive-.

SPECIAL PRECAUTIONS: Acetone is extremely flammable (flash point = 0 °F). Take precautions not to ignite it. Heating of acetone must be done in a fume hood using a flameless, spark-free heat source. Asbestos is a confirmed human carcinogen. Handle only in a well-ventilated fume hood.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. For personal sampling, fasten sampler to worker's lapel near worker's mouth. Remove the top cover from cowl extension ("open-face") and orient sampler face down. Wrap joint between extender and monitor body with tape to help hold the cassette together and provide a marking surface to identify the cassette. Where possible, especially at low %RH, attach sampler to electrical ground to reduce electrostatic effects during sampling.
3. Submit at least two field blanks (or 10% of the total samples, whichever is greater) for each set of samples. Remove top covers from the field blank cassettes and store top covers and cassettes in a clean area (e.g., closed bag or box) during sampling. Replace top covers when sampling is completed.
4. Sample at 0.5 to 16 L/min [3]. Adjust sampling rate, Q (L/min), and time, t (min), to produce fiber density, E , of 100 to 1300 fibers/mm² [$3.85 \cdot 10^4$ to $5 \cdot 10^5$ fibers per 25-mm filter with effective collection area ($A_c = 385 \text{ mm}^2$)] for optimum accuracy. Do not exceed ca. 0.5 mg total dust loading on the filter. These variables are related to the action level (one-half the current standard), L (fibers/cc), of the fibrous aerosol being sampled by:

$$t = \frac{A_c \cdot E}{Q \cdot L \cdot 10^3}, \text{ min.}$$

NOTE: The purpose of adjusting sampling times is to obtain optimum fiber loading on the filter. A sampling rate of 1 to 4 L/min for 8 h (700 to 2800 L) is appropriate in atmospheres containing ca. 0.1 fiber/cc in the absence of significant amounts of non-asbestos dust. Dusty atmospheres require smaller sample volumes (≤ 400 L) to obtain countable samples. In such cases take short, consecutive samples and average the results over the total collection time. For documenting episodic exposures, use high rates (7 to 16 L/min) over shorter sampling times. In relatively clean atmospheres, where targeted fiber concentrations are much less than 0.1 fiber/cc, use larger sample volumes (3000 to 10000 L) to achieve quantifiable loadings. Take care, however, not to overload the filter with background dust [3].

5. At the end of sampling, replace top cover and small end caps.
6. Ship samples upright with conductive cowl attached in a rigid container with packing material to prevent jostling or damage.

NOTE: Do not use untreated polystyrene foam in the shipping container because electrostatic forces may cause fiber loss from sample filter.

SAMPLE PREPARATION:

7. Remove circular sections from any of three quadrants of each sample and blank filter using a cork borer [4]. The use of three grid preparations reduces the effect of local variations in dust deposit on the filter.
8. Affix the circular filter sections to a clean glass slide with a gummed page reinforcement. Label the slide with a waterproof marking pen.
NOTE: Up to eight filter sections may be attached to the same slide.
9. Place the slide in a petri dish which contains several paper filters soaked with 2 to 3 mL acetone. Cover the dish. Wait 2 to 4 min for the sample filter(s) to fuse and clear.
NOTE: The "hot block" clearing technique [5] of Method 7400 or the DMF clearing technique [6] may be used instead of steps 8 and 9.
10. Transfer the slide to a rotating stage inside the bell jar of a vacuum evaporator. Evaporate a 1-by 5-mm section of a graphite rod onto the cleared filter(s). Remove the slide to a clean, dry, covered petri dish [4].
11. Prepare a second petri dish as a Jaffe wick washer with the wicking substrate prepared from filter or lens paper placed on top of a 12-mm thick disk of clean, spongy polyurethane foam [7].

Cut a V-notch on the edge of the foam and filter paper. Use the V-notch as a reservoir for adding solvent.

NOTE: The wicking substrate should be thin enough to fit into the petri dish without touching the lid.

12. Place the TEM grid on the filter or lens paper. Label the grids by marking with a pencil on the filter paper or by putting registration marks on the petri dish halves and marking with a waterproof marker on the dish lid. In a fume hood, fill the dish with acetone until the wicking substrate is saturated.

NOTE: The level of acetone should be just high enough to saturate the filter paper without creating puddles.

13. Remove about a quarter section of the carbon-coated filter from the glass slide using a surgical knife and tweezers. Carefully place the excised filter, carbon side down, on the appropriately-labeled grid in the acetone-saturated petri dish. When all filter sections have been transferred, slowly add more solvent to the wedge-shaped trough to raise the acetone level as high as possible without disturbing the sample preparations. Cover the petri dish. Elevate one side of the petri dish by placing a slide under it (allowing drops of condensed acetone to form near the edge rather than in the center where they would drip onto the grid preparation).

CALIBRATION AND QUALITY CONTROL:

14. Determine the TEM magnification on the fluorescent screen:
 - a. Define a field of view on the fluorescent screen either by markings or physical boundaries.
NOTE: The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric) [7].
 - b. Insert a diffraction grating replica into the specimen holder and place into the microscope. Orient the replica so that the grating lines fall perpendicular to the scale on the TEM fluorescent screen. Ensure that goniometer stage tilt is zero.
 - c. Adjust microscope magnification to 10,000X. Measure the distance (mm) between the same relative positions (e.g., between left edges) of two widely-separated lines on the grating replica. Count the number of spaces between the lines.
NOTE: On most microscopes the magnification is substantially constant only within the central 8- to 10-cm diameter region of the fluorescent screen.
 - d. Calculate the true magnification (M) on the fluorescent screen:

$$m = \frac{X \cdot G}{Y}$$

where: X = total distance (mm) between the two grating lines;

G = calibration constant of the grating replica (lines/mm);

Y = number of grating replica spaces counted

- e. After calibration, note the apparent sizes of 0.25 and 5.0 μm on the fluorescent screen. (These dimensions are the boundary limits for counting asbestos fibers by phase contrast microscopy.)
15. Measure 20 grid openings at random on a 200-mesh copper grid by placing a grid on a glass slide and examining it under the PCM. Use the Walton-Beckett graticule to measure the grid opening dimensions. Calculate an average graticule field dimension from the data and use this number to calculate the graticule field area for an average grid opening.
NOTE: A grid opening is considered as one graticule field.
16. Obtain reference selected area electron diffraction (SAED) or microdiffraction patterns from standard asbestos materials prepared for TEM analysis.
NOTE: This is a visual reference technique. No quantitative SAED analysis is required [7]. Microdiffraction may produce clearer patterns on very small fibers or fibers partially obscured by other material.
 - a. Set the specimen holder at zero tilt.

- b. Center a fiber, focus, and center the smallest field-limiting aperture on the fiber. Obtain a diffraction pattern. Photograph each distinctive pattern and keep the photo for comparison to unknowns.
 NOTE: Not all fibers will present diffraction patterns. The objective lens current may need adjustment to give optimum pattern visibility. There are many more amphiboles which give diffraction patterns similar to the analytes named on p. 7402-1. Some, but not all, of these can be eliminated by chemical separations. Also, some non-amphiboles (e.g., pyroxenes, some talc fibers) may interfere.
17. Acquire energy-dispersive X-ray (EDX) spectra on approximately 5 fibers having diameters between 0.25 and 0.5 μm of each asbestos variety obtained from standard reference materials [7].
 NOTE: The sample may require tilting to obtain adequate signal. Use same tilt angle for all spectra.
 - a. Prepare TEM grids of all asbestos varieties.
 - b. Use acquisition times (at least 100 sec) sufficient to show a silicon peak at least 75% of the monitor screen height at a vertical scale of ≥ 500 counts per channel.
 - c. Estimate the elemental peak heights visually as follows:
 - (1) Normalize all peaks to silicon (assigned an arbitrary value of 10).
 - (2) Visually interpret all other peaks present and assign values relative to the silicon peak.
 - (3) Determine an elemental profile for the fiber using the elements Na, Mg, Si, Ca, and Fe. Example: 0-4-10-3-<1 [7].
 NOTE: In fibers other than asbestos, determination of Al, K, Ti, S, P, and F may also be required for fiber characterization.
 - (4) Determine a typical range of profiles for each asbestos variety and record the profiles for comparison to unknowns.

MEASUREMENT:

18. Perform a diffraction pattern inspection on all sample fibers counted under the TEM, using the procedures given in step 17. Assign the diffraction pattern to one of the following structures:
 - a. chrysotile;
 - b. amphibole;
 - c. ambiguous;
 - d. none.
 NOTE: There are some crystalline substances which exhibit diffraction patterns similar to those of asbestos fibers. Many of these, (brucite, halloysite, etc.) can be eliminated from consideration by chemistry. There are, however, several minerals (e.g., pyroxenes, massive amphiboles, and talc fibers) which are chemically similar to asbestos and can be considered interferences. The presence of these substances may warrant the use of more powerful diffraction pattern analysis before positive identification can be made. If interferences are suspected, morphology can play an important role in making positive identification.
19. Obtain EDX spectra in either the TEM or STEM modes from fibers on field samples using the procedure of step 18. Using the diffraction pattern and EDX spectrum, classify the fiber:
 - a. For a chrysotile structure, obtain EDX spectra on the first five fibers and one out of ten thereafter. Label the range profiles from 0-5-10-0-0 to 0-10-10-0-0 as "chrysotile."
 - b. For an amphibole structure, obtain EDX spectra on the first 10 fibers and one out of ten thereafter. Label profiles ca. 0-2-10-0-7 as "possible amosite"; profiles ca. 1-1-10-0-6 as "possible crocidolite"; profiles ca. 0-4-10-3-<1 as "possible tremolite"; and profiles ca. 0-3-10-0-1 as "possible anthophyllite."
 NOTE: The range of profiles for the amphiboles will vary up to ± 1 unit for each of the elements present according to the relative detector efficiency of the spectrometer.
 - c. For an ambiguous structure, obtain EDX spectra on all fibers. Label profiles similar to the chrysotile profile as "possible chrysotile." Label profiles similar to the various amphiboles as "possible amphiboles." Label all others as "unknown" or "non-asbestos."

20. Counting and Sizing:

- a. Insert the sample grid into the specimen grid holder and scan the grid at zero tilt at low magnification (ca. 300 to 500X). Ensure that the carbon film is intact and unbroken over ca. 75% of the grid openings.
- b. In order to determine how the grids should be sampled, estimate the number of fibers per grid opening during a low-magnification scan (500 to 1000X). This will allow the analyst to cover most of the area of the grids during the fiber count and analysis. Use the following rules when picking grid openings to count [7,8]:
 - (1) Light loading (<5 fibers per grid opening): count total of 40 grid openings.
 - (2) Moderate loading (5 to 25 fibers per grid opening): count minimum of 40 grid openings or 100 fibers.
 - (3) Heavy loading (>25 fibers per opening): count a minimum of 100 fibers and at least 6 grid openings.

Note that these grid openings should be selected approximately equally among the three grid preparations and as randomly as possible from each grid.

- c. Count only grid openings that have the carbon film intact. At 500 to 1000X magnification, begin counting at one end of the grid and systematically traverse the grid by rows, reversing direction at row ends. Select the number of fields per traverse based on the loading indicated in the initial scan. Count at least 2 field blanks per sample set to document possible contamination of the samples. Count fibers using the following rules:
 - (1) Count all particles with diameter greater than 0.25 μm that meet the definition of a fiber (aspect ratio $\geq 3:1$, longer than 5 μm). Use the guideline of counting all fibers that would have been counted under phase contrast light microscopy (Method 7400). Use higher magnification (10000X) to determine fiber dimensions and countability under the acceptance criteria. Analyze a minimum of 10% of the fibers, and at least 3 asbestos fibers, by EDX and SAED to confirm the presence of asbestos. Fibers of similar morphology under high magnification can be identified as asbestos without SAED. Particles which are of questionable morphology should be analyzed by SAED and EDX to aid in identification.
 - (2) Count fibers which are partially obscured by the grid as half fibers.
NOTE: If a fiber is partially obscured by the grid bar at the edge of the field of view, count it as a half fiber only if more than 2.5 μm of fiber is visible.
 - (3) Size each fiber as it is counted and record the diameter and length:
 - (a) Move the fiber to the center of the screen. Read the length of the fiber directly from the scale on the screen.
NOTE 1: Data can be recorded directly off the screen in μm and later converted to μm by computer.
NOTE 2: For fibers which extend beyond the field of view, the fiber must be moved and superimposed upon the scale until its entire length has been measured.
 - (b) When a fiber has been sized, return to the lower magnification and continue the traverse of the grid area to the next fiber.
- d. Record the following fiber counts:
 - (1) f_s, f_b = number of asbestos fibers in the grid openings analyzed on the sample filter and corresponding field blank, respectively.
 - (2) F_s, F_b = number of fibers, regardless of identification, in the grid openings analyzed on the sample filter and corresponding field blank, respectively.

CALCULATIONS:

21. Calculate and report the fraction of optically visible asbestos fibers on the filter, $(f_s - f_b)/(F_s - F_b)$. Apply this fraction to fiber counts obtained by PCM on the same filter or on other filters for which the TEM sample is representative. The final result is an asbestos fiber count. The type of asbestos present should also be reported.
22. As an integral part of the report, give the model and manufacturer of the TEM as well as the model and manufacturer of the EDX system.

EVALUATION OF METHOD:

The TEM method, using the direct count of asbestos fibers, has been shown to have a precision of 0.275 (s_p) in an evaluation of mixed amosite and wollastonite fibers. The estimate of the asbestos fraction, however, had a precision of 0.11 (s_p). When this fraction was applied to the PCM count, the overall precision of the combined analysis was 0.20 [2].

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