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A Report on the Fiber Content of Eighty Industrial Talc Samples Obtained from, and Using the Procedures of, the Occupational Safety and Health Administration

Prepared for

Occupational Safety and Health Administration
Department of Labor
Washington, D. C. 20212

Prepared by the Staff of the
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May 1977

U.S. DEPARTMENT OF LABOR
Occupational Safety and Health Administration
WASHINGTON, D.C. 20210

Office of the Assistant Secretary

20 MAY 1977

Enclosed is a copy of A Report of the Fiber Content Eighty Industrial Talc Samples Obtained from, and Using the Procedures of the Occupational Safety and Health Administrtion (OSHA).

The analysis discussed in the report was conducted by the National Bureau of Standards at the request of OSHA. The purpose of this study was to determine the fiber content of industrial talcs and thereby answer parties regulated by OSHA who challenged data of both OSHA and the National Institute for Occupational Safety and Health. The enclosed final report was received by OSHA on May 10, 1977. I hope you find that this report addresses your concerns and consider it, as does OSHA, a means to assure healthful working conditions for employees.

Sincerely,

Eula Bingham BRC

Eula Bingham
Assistant Secretary
Occupational Safety and Health

Enclosure

TALC SAMPLE NUMBER	COMPANY AND SAMPLE DESIGNATION
1	Vanderbilt Talc Company Nytal 300
2	Vanderbilt Talc Company Nytal 400
3	Vanderbilt Talc Company 5X
4	Vanderbilt Talc Company 325
5	Vanderbilt Talc Company X
6	Vanderbilt Talc Company FT
7	Vanderbilt Talc Company 3X
34	Hitchcock Corporation Murphy, N.C., Product Talc
35	Windsor Minerals, Gassets "37" July 29, 1975
36	Windsor Minerals, West Windsor Mineral Ray Grind Composite
37	Windsor Minerals, Ludlow "36" July 29-30, 1976
38	Windsor Minerals, "Gassetts" 36, July 28-30, 1975
39	Vermont Talc Company - Bagging Bulk Sample, 7/16/75, 2nd shift
40	Vermont Talc Company - Bagging Bulk Sample, vertah XXXD 9/17/76 1st shift

TALC SAMPLE NUMBER	COMPANY AND SAMPLE DESIGNATION
41	Southern Talc Co. (Cohutta Mill) Chatsworth, Ga. Product (Powder) Sample Mine, MESA I.D. #04-00493
42	Packer Station #3, Vanderbilt Talc, Mill #1 Gouverneur, N. Y. 1st shift 11/4/75 (FT)
43	Packer Station #2, Vanderbilt Talc, Mill #1 Gouverneur, N. Y. (Nytal 100), 1st shift 11/4/75
44	Ceramitalc HDT, Vanderbilt Talc Co. 1st shift 11/4/75
45	Bagging Station #3, Vanderbilt Talc Co., Mill #1, Gouverneur, N. Y. 11/4/75 (Nytal 200)
46	Hitchcock Corp. #5 Mine MESA ID# 3100222
47	Hitchcock Corp., #3 Mine MESA ID# 3100683
48	Wrau Mine 3 mill, Int. Minerals and chemicals MESA ID# 31-0008
49	Bagging Station, Vanderbilt Talc Co. 2nd shift, Mill #1 Gouverneur, N. Y. (Nytal 300) 11/3/75
50	Bagging Station, Vanderbilt Talc Co., 2nd shift, 11/3/75 (Nytal 200)
51	Southern Talc Co., Southern Mill Product Talc sample, Chatsworth Ga. Rock Cliff Mine, MESA ID# 0900492, 9/3/74
52	Southern Talc Co., Southern Mill, Chatsworth, Ga. ore from Rock Cliff MESA ID# 90-00492
53	Southern Talc Co., Southern Mill Ore from Jude Hole Mine MESA ID# 09-00492

TALC SAMPLE NUMBER	COMPANY AND SAMPLE DESIGNATION
54	Winterborg Mine & Mill, Ore from American Talc Co., Alpine, Ala. Willowreek Mine, MESA ID# 01-00433 9/4/75
55	Thompson-Weinman Co., Cartersville Plant, Cartersville, Ga., 9/5/74, Ore from Brady Mine, MESA ID# 09-00224
56	Southern Talc Co., Cohutta Mill Chatsworth, Ga. 9/3/74, Ore from Ernest Mine, MESA ID# 09-00493
57	American Talc Co., Alpine, Ga. Winterboro Mine & Mill, 9/5/75 MESA ID# 01-00433, Final Prod.
58	Pioneer Talc Co. Inc., Garren Pit. Prod. #6 Allamore, Hudspeth Co., Texas MESA ID# 41-00869
59	Pioneer talc Co., Inc., Apache Pit Product, #4, Texas MESA ID# 41-00869
60	Pioneer Talc Co. Inc., #4 Texola Pit Product, Texas MESA ID# 41-00869
61	Southern Clay Products, #2 Bottom Product, Allamore, Hudspeth Co., Texas #18
62	Milwhite Co. Inc., Van Horn, Cumberson Co., Texas MESA ID# 41-01527 Ground Calcined Product
63	Milwhite Co. Inc., #11, Van Horn, Culberson Co. Texas, Eagle Hat Pit Crude MESA ID# 41-01527
64	Pioneer Talc Co., Allamore, Hudspeth Co. Texas, #8 Garren Ceramic MESA ID# 41-00869

TALC SAMPLE NUMBER	COMPANY AND SAMPLE DESIGNATION
65	Milwhite Co. Inc., Van Horn, Texas #14 Tumble Down Pit Product, #14 MESA ID# 41-01527
66	Eastern Magnesia Talc Emtal 42, Co. Vermont 8/28/75
67	Pfizer (Barretts Mill) Montana minus 12 Micron
68	Pfizer (Barretts Mill) Montana minus 325 mesh
69	Pfizer (Barretts Mill) Montana minus 20 microns
70	Cyprus Industrial Minerals (Three Forks Mill) Montana minus 325 mesh
71	Cyprus Industrial Minerals (Three Forks Mill) Montana Mistron Vapor (minus 10 micron)
72	Cyprus Industrial Minerals (Three Forks Mill) Montana Floor Sweepings (Bagging Area)
73	Cyprus Industrial Minerals (Yellowstone Mine) Montana Drill Hole
74	Southern Talc (Southern Mill) Ga. 98% passing 200 mesh
75	Southern Talc (Southern Mill) Ga. 99% passing 325 mesh (#1767)
76	Southern Talc (Cohutta Mill) Ga. 95% passing 325 mesh
78	Milwhite Talc Co. Inc., (Tex.) Finished CM4 product (#2)
79	Westex Talc Co. (TDM) Dark - (Milwhite Inc.) (#1) Tex.

TALC SAMPLE NUMBER	COMPANY AND SAMPLE DESIGNATION
80	Milwhite Co., Inc. (Tex) Marshel mix - 92-200 mesh (#3)
81	Westex Talc Co. (Milwhite Inc.) (Tex.) White mine 93-200 mesh (#4)
82	Southern Clay Products - Tex. Garren Mine #2 (#5)
83	Texas Talc Co. - TPI (Tex.) Mine (Texas Pacific) (#6)
84	Texas Talc Co. Tex. David Mine (#7)
86	Cyprus Industrial Minerals Co. Loyce Fine Mine (Tex.) #9
87	Pioneer Talc Co. Inc. (Tex.) Texola Mine (#10)
88	Pioneer Talc Co. Inc. (Tex.) Dees Mine (#11)
89	Milwhite Co. Inc. Bryant Soapstone Mine Arkansas
91	Laws Mill Micro talc from White Eagle Mine Standard Industrial Minerals
92	Laws Mill Desert Talc Product Standard Industrial Minerals
93	Laws Mill Holiday Talc Product Standard Industrial Minerals
94	Keeler T-076 Tecopa Mine (100% Panamint) Cyprus Industrial Minerals, Montana
95	Keller HGO-55 Blend 4 Panamint, 3 Oasis, 3 White Eagle Cyprus Industrial Minerals, Montana
96	Keeler Sierra Cloud Blend 4 Panamint, 1 White Eagle Cyprus Industrial Minerals, Montana

TALC SAMPLE NUMBER	COMPANY AND SAMPLE DESIGNATION
97	Keeler, MSC Mistron Panamint + 0.5% stearite Cyprus Industrial Minerals
98	Keeler Sierralite Talc City (Frisco) Cyprus Industrial Minerals
99	Keeler Glacier 200 Talc City (Tailings) Cyprus Industrial Minerals
100	Keeler USP Oasis Mine (100%) Cyprus Industrial Minerals
101	Keeler White Eagle Screenings Cyprus Industrial Minerals
102	Keeler Suggar Mine Cyprus Industrial Minerals
103	Pfizer Inc. (Cal) Talcron CP 44-31 Eclipse, Bonnie, Mongolian
104	Pfizer Inc. (Cal) Cercron CF 96-38 Eclipse, Acme, Mongolian
105	Pfizer Inc. (Cal) Cercron CF 96-36 White Eagle, Mongolian, Apex
106	Cyprus Industrial Minerals (LA) Furnace Creek Tecopa (100% Panamint)
107	Cyprus Industrial Minerals (LA) Vanderbilt Co., crude Soft LB
108	American Talc (Alabama) 84 Color Dark 200 Mesh Willow Creek Mine
109	American Talc (Alabama) 92 Color 99% 400 Mesh Willow Creek Mine

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A REPORT ON THE FIBER CONTENT OF EIGHTY INDUSTRIAL TALC SAMPLES OBTAINED FROM, AND USING THE PROCEDURES OF, THE OCCUPATIONAL SAFETY AND HEALTH ADMINISTRATION

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May 1977

U.S. DEPARTMENT OF COMMERCE, Juanita M. Kreps, *Secretary*

Dr. Betsy Ancker-Johnson, *Assistant Secretary for Science and Technology*

NATIONAL BUREAU OF STANDARDS, Ernest Ambler, *Acting Director*

This document has been prepared for the Occupational Safety and Health Administration of the Department of Labor. Responsibility for its use rests with that agency.

I. INTRODUCTION

A. Purpose of Study

This report has been prepared in response to a request received by Dr. John D. Hoffman, Director of the Institute for Materials Research of the National Bureau of Standards (NBS), in a letter dated September 1, 1976, from Dr. Morton Corn, Assistant Secretary of Labor, Occupational Safety and Health Administration (OSHA). In that letter, Dr. Corn stated that analysis of talc samples for their asbestos content was being performed by OSHA and the National Institute of Occupational Safety and Health (NIOSH) but that the methodology was being challenged by some of those regulated by OSHA.

Dr. Corn indicated that his request to NBS was composed of two tasks:

(1) to resolve the variability in the definition of asbestos fibers in talc, and

(2) to determine the asbestos content of some 80 talc samples to be provided by OSHA.

Copies of the letter containing this request and the subsequent correspondence between Drs. Hoffman and Corn which provide further background information, are included in this report as Appendix I.

It was agreed by both parties that the first of these tasks, *i.e.*, resolving the variability of the definition of asbestos fibers, would be a complex, long-term program which would require input from a number of sources both in the private and public sectors. In view of this, it was agreed that the more limited task, that of determining the asbestos content of the OSHA talc samples, would be addressed by NBS first. As Dr. Corn pointed out in his letter of October 8, 1976, this was recognized not to be a research task but would involve an analysis performed according to the procedures given in 29 CFR 1910.1001.

29 CFR 1910.1001 [39 FR 23502, June 27, 1974, as amended in 41 FR 11505, March 19, 1976] deals exclusively with airborne asbestos and does not describe procedures for, nor make any reference to asbestos in talc. It does, however, include the following two paragraphs which are pertinent to the work discussed in this report:

> "(a) *Definitions.* For the purpose of this section, (1) 'Asbestos' includes chrysotile, amosite, crocidolite, tremolite, anthophyllite, and actinolite.
>
> (2) 'Asbestos fibers' means asbestos fibers longer than 5 micrometers.
>
> "(e) *Method of measurement.* All determinations of airborne concentrations of asbestos fibers shall be made by the membrane filter method at 400-450 X (magnification) (4 millimeter objective) with phase contrast illumination."

These regulations do not, however, describe a measurement *procedure* but rather prescribe a *method* of measurement (*viz.*, phase contrast microscopy). A *procedure* contains a detailed listing of the sampling of the material, the specific experimental steps to be performed during an analysis, and, often, descriptions of the mathematical calculations to be performed and of the format for reporting results and their associated errors. A *method* of measurement is defined by a very general statement of the type of measurement to be made, from which a specific *procedure* is developed.

Since 29 CFR 1910.1001 stipulates only that the method of phase contrast microscopy is to be used for the determination of asbestos, NBS requested a detailed procedure from OSHA. In response, NBS was provided with a copy of the OSHA document "Asbestos Fiber in Air" - Method No. P&CAM 239,

issued March 30, 1976. NBS was also informed that the determination of asbestos, including asbestos in talc, was performed at the OSHA Salt Lake City, Utah, laboratory (OSHA-SLC), using P&CAM 239. Method No. P&CAM 239, however, deals only with the determination of asbestos in air using phase contrast microscopy to examine membrane filters. Since the determination of asbestos in talc is not described in the document, NBS was told to contact Mr. Willard C. Dixon at OSHA-SLC to obtain an exact description of the procedure used on talc samples.

To become familiar with the asbestos in talc procedure, an NBS scientist then visited the OSHA-SLC laboratory and obtained verbal and written descriptions of the procedure used. He spent approximately one and one-half days at the laboratory observing the procedures and techniques used and discussing them with the OSHA employees. At the conclusion of the visit he wrote a detailed report of what he had observed. This report was submitted to Mr. Dixon for comment. A copy of the report is included as Appendix II to this document. The portions of the report that are enclosed in boxes are the comments added by OSHA-SLC personnel.

NBS scientists also discussed the analysis of talc with a number of other persons. Those contacted are listed in Appendix III to this report.

Prior to commencing the actual analysis of the 80 talc samples, we were assured by OSHA personnel that 29 CFR 1910.1001, P&CAM 239 and the annotated NBS trip report were the only documents appropriate for documenting the analytical procedure to be employed, and that the process followed by NBS in developing the detailed procedure was proper.

The detailed procedure followed at NBS is given in Section II of this report; however, a brief description of the procedure, and some comments, are given below:

Phase contrast microscopy, with a mounting medium having an index of refraction of 1.546, is used. Both fibers (a fiber being defined as having a minimum length of 5 μm, a maximum diameter of 5 μm, and a minimum length to diameter ratio of 3:1) and other particles are counted.

Early in the study we determined that the identification of "asbestos" with the procedure used was extremely difficult for the following reasons:

(1) Since phase contrast microscopy is only a contrast mechanism, it does not indicate the *degree* of difference between the refractive index of the liquid and a particle or fiber. For example, if a specimen is mounted in a liquid which matches a refractive index of chrysotile, then the particles and fibers counted would include all fibers which do not have that particular refractive index, *e.g.*, talc, anthophyllite, wollastonite, fiber glass, etc. The same would be true for any other liquid used.

(2) Most mineral species have three refractive indices, therefore, any mineral fiber not lying in the correct orientation for the selected liquid will not match and would be visible and would be counted.

(3) The amphiboles are end members in solid solution. As a result there may be a large range of refractive indices from one end member to another. One example of such a series would be the tremolite-actinolite solid solution.

(4) In addition, the most common methods of talc formation are the hydrothermal alteration of ultrabasic rocks such as serpentine and tremolite and the thermal metamorphism of siliceous dolomites. Therefore, during the formation of talc in contact with other minerals, there may be extensive interconversions between talc and the minerals serpentine, tremolite and anthophyllite. These interconversions may give rise to single particles which have a combination of the talc, anthophyllite, and serpentine mineral phases.*

(5) There are many materials which may be present in talc which have overlapping refractive indices, *e.g.*,

Mineral	Refractive Index
Wollastonite	1.63
Tremolite	1.599 - 1.637
Quartz	1.55
Talc	1.539 - 1.589
Chrysotile	1.493 - 1.567

(6) Even if the refractive indices were known, positive identification of minerals could not be made since there may be interferences from other materials.

* Deer, W. A., Howie, R. A., and Zaussman, J., *Rock Forming Minerals*, Vol. 3, Longmans, Green and Co. Ltd, London, England (1967) pp. 126-128.

In his first letter to NBS, Dr. Corn requested a determination of the "number of fibers per unit weight or unit volume. Also, the asbestos content as percent of total weight...". This was to be based upon the definition of asbestos to be resolved by NBS. Since the definition of asbestos is to be investigated as part of the longer study, reporting results obtained in these analyses in terms of "asbestos" was not possible. The procedure used by OSHA-SLC did not use weight or volume of total sample, but instead used percent fibers as a function of total numbers of particles on a slide; that convention was followed at NBS. Thus, all data given in this report are on the basis of numbers or percent of fibers per total number of entities (particles plus fibers) counted (this is referred to as number percent).

B. Goal of the Study

In view of the current limitations both in the definition of asbestos and in the ability to distinguish minerals by the OSHA methods using the existing definition of asbestos, NBS established as the goal for this portion of the study to analyze the 80 talc samples supplied by OSHA to NBS and to report the number percent (and, if possible, the limits of error) of *fibers* using the phase contrast optical microscopy procedure discussed above and outlined in detail in Section II below.

II. Analysis Procedure

1. Equipment used by NBS
   a. A Leitz* Ortholux 1 microscope equipped with 10x and 40x phase contrast objectives and a phase contrast condenser.
   b. Cargille refractive index liquid (mounting medium) $n^{25\ °C} = 1.546$
   c. Porton Reticle
   d. 10x Periplan oculars

2. Specimen Mounting Procedure
   Two drops of the mounting medium were placed on a clean microscope slide.
   The end of a new (clean) metal wire (paper clip) was dipped into the mounting medium on the slide and then into the sample which was contained in a bottle.
   The material adhering to the wire was blended into the mounting medium on the slide.
   A cover slip was then placed over the preparation.

Note: No attempt was made to homogenize the contents of the sample bottle prior to drawing the specimen. A vigorous blending or mixing could itself lead to mechanical breakdown of particles into fragments with fibrous appearance or the separation of one fiber into many fibers. NBS did not attempt to homogenize

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* In order to describe materials and experimental procedures adequately, it is occasionally necessary to identify the sources of commercial products by the manufacturer's name. In no instance does such identification imply endorsement by the National Bureau of Standards, nor does it imply that the particular product is necessarily the best available for that purpose.

the samples as the OSHA-SLC procedure does not include homogenization.

3. Sample Notation

Upon receipt of the samples, a scientist who was not involved in the analysis divided the samples and assigned internal NBS numbers. This prevented the analysts from knowing which of the 80 samples they were dealing with and permitted blind replicate analyses.

Note: In the original transfer of the samples from OSHA to NBS a list was inadvertently included which gave the sample identifications. As soon as this was discovered, the list was sealed in an envelope and placed in the NBS Security Office safe until it was returned to OSHA. The only person at NBS who saw the list did not participate in any any of the analytical work.

4. Microscopy Techniques

Phase contrast microscopy was the method used by NBS, since it is the only technique specified both in 29 CFR 1910.1001 and in OSHA Method No. P&CAM 239. The detailed procedure followed by NBS is described below. Any steps which differ from the OSHA-SLC procedure are noted.

(1) Before the analysis of a specimen was performed, the microscope illumination was adjusted for Kohler illumination, the annular diaphragm and phase-shifting elements were aligned and the Porton reticle was calibrated against a stage micrometer. The configuration of a Porton reticle is shown in figure 1. A detailed description of both the calibration and use of this reticle is given in P&CAM 239.

(2) The counting field was defined as the six rectangles on the left half of the Porton reticle.

(3) Fiber dimensions were determined by comparing the length and width to the diameters of the calibrated circles on the Porton reticle.

(4) The unknown samples were mounted as described above in a Cargille liquid with a refractive index of 1.546.

Note: OSHA mounted their samples in liquids with $n^{25\ °C}$ equal to 1.546 and 1.47. The 1.546 index was used to identify quartz and chrysotile which appear blue in bright-field observation with their Zeiss phase-contrast microscope, while the background is light brown and most other materials are brown or black. The 1.47 refractive index liquid is carried over from techniques for identifying airborne fibers collected on membrane filters as opposed to the 1.546 liquid used for talc analysis.

(5) Counting fields were selected by advancing the mechanical stage in a pattern of traverses of the slide in one axis and steps along the other axis resulting in a rectangular zig-zag pattern. This procedure was followed until the necessary number of fields was counted.

(6) NBS used the definition of (asbestos) fibers, discussed above, *viz.*, a fiber is greater than or equal to 5 μm in length, has a length to width ratio of at least 3 to 1, and has a maximum diameter of 5 μm. Any particle meeting these criteria was counted as a fiber.

(7) The samples were continuously viewed over the range of focal planes covering the sample thickness during the counting.

(8) A preliminary scan of the slide was performed to attempt to find fibers in the specimen. If a fiber was found the following counting procedure was performed on newly prepared slides. Fibers were counted in as many fields as necessary to yield a total fiber count of 100 with the following exceptions:

(a) The analysts counted at least 20 fields even if they counted more than 100 fibers, and

(b) they stopped at 100 fields even if they did not count 100 fibers.

(9) For fibers and particles that crossed either one or two sides of the counting field, the following procedure was used to obtain a representative count:

The analysts counted all particles or any fibers of the correct dimensions which:

1. Were entirely within the counting area, or
2. Crossed the left or bottom sides, or
3. Crossed the upper or lower left corner, or
4. Crossed both the top and bottom sides.

Any other fibers and particles were not counted.

(10) When particle agglomerates covered a large portion of the field of view, the field was rejected and replaced by a new one.

(11) The measurements at NBS were performed @ 500x magnification. OSHA measurements were done @ 400x to 450x magnification, but NBS could not duplicate that magnification with the equipment available. Calculations performed indicate that the differences in magnification would not result in any significant changes in the number of fibers and particles identified.

(12) The numerical aperture, NA, of the NBS objective was 0.65. OSHA specifies a NA range of 0.65 to 0.75. With an objective having NA = 0.65, and at a total system magnification of 500x, the smallest diameter of a suspected fiber which could be seen was approximately 0.4 μm. Hence, any fiber 5 μm long but having a diameter less than 0.4 μm, could never be found unambiguously. The class of particles defined as fibers and visible by the procedure is depicted as the cross hatched portion shown in figure 2.

Note: The OSHA-SLC laboratory, in addition to phase-contrast microscopy, sometimes uses polarized light microscopy with a retardation plate to distinguish asbestos fibers from talc plates oriented on end and from other non-asbestos fibers such as glass or organic fibers which may have been present in the samples. Since these techniques are not part of the phase-contrast method they were not used at NBS.

NBS used two analysts working independently and following the procedure given above in obtaining the results on the OSHA-supplied talc samples. While the analysts did not have, prior to this study, an extended period of experience with the specific methodology employed, they have had extensive experience in a wide range of particle characterization techniques, including general optical microscopy.

III. Preliminary Investigations of Samples

A. Macroscopic Description of Samples

A qualitative description of the 80 samples supplied to NBS by OSHA is given in table 1. The sample numbers are those assigned by OSHA. The number after the description refers to the number of the color chip in the Inter-Society Color Council-NBS Color Name Chart (Supplement to NBS Circular 553).

B. Scanning of Samples

Specimens of the 80 talc samples were mounted on slides by each analyst independently, using the "dip" method described in Section II.2 above. These specimens, each containing ~1000 particles, were then scanned rapidly, *i.e.*, only the presence or absence of fibers was noted and fiber counts were not made at this time. As a result of these scans, specimens taken from 31 samples were identified by both analysts as containing at least one fiber, specimens taken from an additional 21 samples were identified as containing at least one fiber by one analyst but no fiber was seen by the other analyst, and no fiber was seen by either analyst for specimens taken from the remaining 28 samples.

IV. Counting and Analysis of Samples

A. Samples Noted by Both Analysts as Containing Fibers

As noted above, 31 samples were identified by both analysts as containing fibers during scanning. Data for these samples are given in table 2. The observed counts for each specimen by each of the two analysts, listed as F/(P+F) (*i.e.*, fibers divided by non-fibrous particles + fibers) are shown. In addition the estimated percent of fibers (p), and the lower (L) and upper (U) limits of a 95 percent confidence interval for the "true" value of p for the slide on which the

count was made are given.* It should be noted that the confidence interval refers only to the portion of fibers on the slide on which the count was made, and not necessarily to the true average of fibers in the entire sample. (Estimations of the latter would require that the slide constitute a random sample of a homogeneous material under examination.)

It is seen that for eight of these 31 materials, the confidence intervals for replicate slides failed to overlap, indicating a lack of statistical compatibility between these replicate determinations. To study in more detail the consistency (or lack thereof) of the results from replicate slides, a more exact statistical analysis was performed by considering the actual fiber and particle counts for each material as the elements of a contingency

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* The calculation of confidence limits, and other statistical analyses in this report, are valid under the assumptions (i) that the fibers are randomly located among the particles on a slide, and (ii) that the stopping rule of Section II.4(8) above introduces negligible bias.

The confidence interval is based on the relation:

Prob [at most c occurrences of event E in N trials]

$$= \text{Prob} \left[F \geq \frac{n_2}{n_1} \frac{P}{1-P} \right]$$

where P is the probability of an event E, F is the F statistic with n_1 and n_2 degrees of freedom, and

$$n_1 = 2(c+1)$$

$$n_2 = 2(N-c)$$

This relation is cited in Fisher, R. A., and Yates, F., *Statistical Tables*, Hafner Publishing Company, N. Y. (1963) p. 3.

table* and calculating the corresponding chi-square value*. A significant value for chi-square, at the 5 percent level of significance, was taken to indicate statistical incompatibility between the replicate slides. The samples for which significance at the 5 percent level was thus determined are identified by an asterisk in table 2. Of the 31 materials, 17 showed incompatibility between replicate slides at the 5 percent level of significance.

The fact that over 50 percent of the results of these 31 samples were incompatible suggests problems either of inhomogeneity, or of difficulty in the determination of fiber morphology, or both. If the sample is inhomogeneous, the reason for the statistical incompatibility is obvious. The problem of determining fiber or particle morphology is very difficult as there is often a great deal of subjectivity in deciding whether or not a particle meets the criteria to be classed as a fiber. Photomicrographic examples of (1) a specimen containing unambiguous fibers, (2) a specimen in which no fiber can be discerned, and (3) a specimen which required a great deal of judgment are shown in figure 3, a, b, and c respectively. In figure 3 six different fields of view are shown for each specimen, three using an optical microscope and three using a scanning electron microscope (SEM). (The SEM fields do not correspond to the optical fields, but were chosen randomly.)

In the case of talc samples, one must also judge which of the apparent fibers are talc platelets seen on edge. It is possible to "roll over" some of these platelets by moving the cover plate slightly, but while doing so, one also may "roll" another platelet into a position where it would subsequently be viewed from the edge.

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* Snedecor, G. W., and Cochran, W. G., *Statistical Methods*, 6th Ed., The Iowa State University Press, Ames, Iowa (1967). Eqn. 8.10.3, p. 217.

B. Samples Noted Initially by Both Analysts as not Containing Fibers

Because of our concern over the incompatibility of results in the 31 samples in which both analysts agreed there were fibers, we decided to reexamine a portion of the group of 28 samples originally classed by both analysts as not containing fibers. Ten samples were selected at random from the pool of 28 samples and were scanned a second time.

(Before discussing the results of additional scanning and counting in this group of samples, it is useful to calculate the probability of seeing no fibers in a count of N objects (particles or fibers). The probability is a function of the true fiber content of the material from which the slide is prepared. The calculation is based on the assumption that the specimen (*i.e.*, the portion of the material on the slide) is a random sample from the material being measured. According to the binominal distribution,* the probability (zero fibers in count of N) equals $(1-P)^N$ where $P=\frac{p}{100}$, and p is the number percent fiber content of the material. Table 3 lists values of this probability for various values of p, for N=200, 500, 1000, and 2000. It is evident that the probability of seeing no fibers in two slides of 1000 counts each (N=2000) is extremely small unless the true fiber content of the material is less than 0.25 percent. (Even for N=200, the probability of seeing no fibers is very small unless the fiber content of the material is less than 2.5 percent). One would therefore expect the materials in this group to have fiber contents not greater than 0.25 percent.

It is also interesting to note that for materials with very low fiber content, say 0.01 percent, there is only an 18 percent probability of finding at least one fiber in a count of 2000. In order to reach a probability of 95

* *Ibid.*, Chapter 8.

percent of finding at least one fiber in such a material, one would have to examine at least 30,000 particles. For a material with a 0.1 percent fiber content, the number of particles required to find at least one fiber with 95 percent probability is 3000.)

As a result of the second scan on newly prepared slides, four samples were still identified as fiber-free, but fibers were observed in the remaining six samples. The results are given in table 4.

Four samples showed fiber contents larger than 0.25 percent by both analysts. Furthermore, for two of these samples, the two new slides gave incompatible results (as shown by the chi-square test). These are indicated by an asterisk in table 4.

The results obtained from this reexamination of the materials in the group of initial negative scans thus support the interpretations given by the analysis of the samples in the first group: inhomogeneity and/or subjectivity in deciding fiber morphology.

C. Samples Noted as Containing Fibers by Only one Analyst During the Initial Scan

The remaining pool of 21 samples, which were noted as containing fibers by only one of the analysts during the initial scan, was then examined. In all of these cases the percent of fibers was estimated to be low and the arguments given above about the probability of finding fibers as a function of total number of particles are equally valid. In this case, eight samples were selected for fiber counts. Of the eight samples selected, seven provided incompatible replicates, given further support to the interpretation made on the basis of the other two groups of samples. These data are given in table 5. Again, incompatible results are indicated with an asterix.

V. Conclusions

In this report the results of the determination of the fiber content of 80 OSHA-supplied samples of talc are presented, along with a statistical interpretation of these results. Of the 45 samples on which fiber counts were made, the results for replicate analyses on 26 of the samples were statistically incompatible. This incompatibility was not confined to any particular concentration range of fibers. As a result, NBS deemed it inadvisable to report uncertainty limits for the fiber content determinations obtained on the samples measured. Fiber counts could be made using the present procedure on the 35 samples which were not counted, but it is doubtful that any useful additional information would be obtained.

The variability of these results raises several questions regarding the OSHA procedure, particularly sampling technique, sample homogeneity, and determining fiber morphology. It is the opinion at NBS that, even under favorable circumstances (*e.g.*, homogeneous samples, easily identified fibers, etc.), the existing OSHA procedure is useful only for determining "fiber" content and not "asbestos" content. Although careful manipulation of the mounting medium might make it possible to identify some of the fibers as "asbestos", the problem of the definition of "asbestos" still remains. NBS believes that the resolution of the measurement problem, including the definition and identification of asbestos, will be accomplished only by significant changes in the procedure and probably the method as well.

In order to complete the tasks requested by Dr. Corn in his letter of September 1, 1976, it will be necessary to arrive at an acceptable definition of asbestos and to develop the necessary measurement techniques and standards. Once that has been achieved, a more meaningful analysis of the 80 OSHA-supplied talc samples can be accomplished.

Table 1

Description of bulk material

Sample	Description
1	fine white powder (263)
2	fine white powder (263)
3	fine white powder (263)
4	fine white powder (263)
5	fine white powder (263)
6	fine white powder (263)
7	fine white powder (263)
34	fine white powder (263)
35	coarse grains (sand-like) (92) inhomogeneous
36	fine off-white powder (93)
37	beige fine-grained non-homogeneous some dark material, some light (93)
38	beige fine-grained non-homogeneous some dark material, some light material (93)
39	off-white powder (92)
40	fine very light grey powder (92)
41	fine light grey powder (264)
42	fine white powder (263)
43	fine white powder (263)
44	fine white powder (263)

Table 1 (continued)

Sample	Description
45	fine white powder (263)
46	fine white powder (263)
47	fine white powder (263)
48	light brown powder (264)
49	off-white powder clumps together in small aggregates (263)
50	off-white powder clumps together in small aggregates (263)
51	light grey powder (154)
52	grey granular material, non-homogeneous (155)
53	light grey powder (fine) (154)
54	fine white powder (263)
55	fine off-white powder (92)
56	grey granular material, non-homogeneous (154)
57	fine white powder, some aggregation (263)
58	fine off-white powder (263)
59	off-white powder clumps together in small aggregates (92)
60	off-white powder clumps together in small aggregates (263)
61	dark grey granular, some small fragments (265)
62	fine off-white powder (263)
63	fine off-white powder (9)

Table 1 (continued)

Sample	Description
64	fine brown-grey powder (264)
65	fine grey powder (265)
66	fine off-white powder (264)
67	off-white powder, some aggregation (263)
68	fine white powder (263)
69	off-white powder, some aggregation (263)
70	fine off-white powder (92)
71	off-white powder, some aggregation (263)
72	off-white powder, some aggregation (92)
73	small light grey pebbles and powder, inhomogeneous (264)
74	fine light grey powder (264)
75	fine off-white powder (264)
76	fine grey powder (154)
78	light grey to brown powder (264)
79	dark grey rock, some large fragments but mostly small grains (265)
80	fine grey powder (264)
81	off-white powder (fine)(9)
82	dark grey rock, mostly large fragments with some granular material (265)

Table 1 (continued)

Sample	Description
83	dark grey rock, mostly large fragments with some granular and powdered material (265)
84	dark grey rock almost all large fragments, very little granular or powdered material (266)
86	dark grey granular material with some large fragments (265)
87	white and pink rocks, very little granular or powdered material (9)
88	coarse granular light brown several large fragments with some powder (93)
89	grey-brown granular, inhomogeneous (93)
91	fine off-white powder (263)
92	white rock with powder (263)
93	grey and brown rocks (93)
94	fine white powder (263)
95	off-white powder, some aggregation (263)
96	fine white powder (263)
97	fine white powder (263)
98	fine off-white powder (92)
99	fine beige powder (153)
100	fine white powder (263)
101	white to light grey rocks with a fairly large amount of powder (92)

Table 1 (continued)

Sample	Description
102	large grey rocks with white powder @ the bottom (153)
103	fine white powder (263)
104	off-white powder (263)
105	fine white powder (263)
106	fine white powder (263)
107	large grey and light brown rocks, some powder in the bottom, very inhomogeneous (92)
108	fine white powder (263)
109	white powder, extensive aggregation (263)

Table 2

Results of the 31 Samples Identified as Containing Fibers by Both Analysts on the Initial Scan

Sample No.	Analyst 1 Count [F/(P+F)]	Analyst 1 p (%)	Analyst 1 95% Confidence Intervals L (%)	Analyst 1 95% Confidence Intervals U (%)	Analyst 2 Count [F/(P+F)]	Analyst 2 p (%)	Analyst 2 95% Confidence Intervals L (%)	Analyst 2 95% Confidence Intervals U (%)
1	102/760	13.4	11.1	16.0	125/833	15.0	12.7	17.6
2*	135/809	16.7	14.2	19.4	142/1138	12.5	10.6	14.5
3	100/480	20.8	17.3	24.7	103/445	23.1	19.3	27.3
4	105/660	15.9	13.2	18.9	109/670	16.3	13.6	19.3
5	100/711	14.1	11.6	16.8	109/720	15.1	12.6	18.0
6	158/1606	9.8	8.4	11.4	83/714	11.6	9.4	14.2
7*	102/852	12.0	9.9	14.3	102/586	17.4	14.4	20.7
34*	101/696	14.5	12.0	17.3	105/1028	10.2	8.4	12.2
38*	45/652	6.9	5.1	9.1	17/451	3.8	2.2	6.0
41*	100/991	10.1	8.3	12.1	70/501	14.0	11.1	17.3
42	105/593	17.7	14.7	21.0	106/492	21.5	18.0	25.4
43	103/885	11.6	9.6	13.9	102/829	12.3	10.2	14.7
45	106/682	15.5	12.9	18.5	86/627	13.7	11.1	16.6
46	104/1163	8.9	7.3	10.7	103/1155	8.9	7.3	10.7
47	104/543	19.2	15.9	22.7	136/817	16.6	14.2	19.4
49*	142/1263	11.2	9.6	13.1	102/393	26.0	21.7	30.6
50*	124/659 100/848	18.8 11.8	15.9 9.7	22.0 14.2	127/600 76/866	21.2 8.8	18.0 7.0	24.6 10.9

* Incompatible replicates.

Table 2 (continued)

Sample No.	Analyst 1 Count [F/(P+F)]	Analyst 1 p (%)	Analyst 1 95% Confidence Intervals L (%)	Analyst 1 95% Confidence Intervals U (%)	Analyst 2 Count [F/(P+F)]	Analyst 2 p (%)	Analyst 2 95% Confidence Intervals L (%)	Analyst 2 95% Confidence Intervals U (%)
52*	100/919	10.9	8.9	13.1	54/767	7.0	5.3	9.1
56	72/900	8.0	6.3	10.0	54/581	9.3	7.1	11.9
58*	48/1863	2.6	1.9	3.4	29/503	5.8	3.9	8.2
60*	100/1285	7.8	6.4	9.4	60/1190	5.0	3.9	6.4
68	67/1901	3.5	2.7	4.4	79/1685	4.7	3.7	5.8
	87/2237	3.9	3.1	4.8				
75*	111/1474	7.5	6.2	9.0	109/686	15.9	13.2	18.8
	105/1245	8.4	7.0	10.1				
76*	100/759	13.2	10.8	15.8	100/522	19.2	15.9	22.8
87	48/471	10.2	7.6	13.3	55/527	10.4	8.0	13.4
92	101/329	30.7	25.8	36.0	111/314	35.4	30.1	40.9
94*	35/3276	1.1	0.7	1.5	105/906	11.6	9.6	13.8
					72/1674	4.3	3.4	5.4
95*	43/1947	2.2	1.6	3.0	37/2820	1.3	0.9	1.8
96*	55/1571	3.5	2.6	4.5	36/625	5.8	4.1	7.9
104*	69/959	7.2	5.6	9.0	101/773	13.1	10.8	15.6
109*	111/2674	4.2	3.4	5.0	104/1890	5.5	4.5	6.6

* Incompatible replicates.

NOTE: Confidence limits were calculated using an approximate formula and a few may be in error by one or two units in the last place.

Table 3

Probability of Finding No Fibers in Counts of Size N

True Fiber Content, in Percent	Size of Count			
	N=200	N=500	N=1000	N=2000
0.01	0.98	0.95	0.90	0.82
0.05	0.90	0.78	0.61	0.37
0.10	0.82	0.61	0.37	0.14
0.15	0.74	0.47	0.22	0.05
0.20	0.67	0.37	0.14	0.02
0.25	0.60	0.29	0.08	0.01
0.30	0.55	0.22	0.05	<0.01
0.40	0.45	0.13	0.02	
0.50	0.37	0.08	0.01	
0.60	0.30	0.05	<0.01	
0.80	0.20	0.02		
1.00	0.13	0.01		
1.50	0.05	<0.01		
2.00	0.02			
2.50	<0.01			

Table 4

Results of the 28 Samples Identified by Both Analysts as Not Containing Fibers on the Initial Scan

	Analyst 1				Analyst 2			
			95% Confidence Intervals				95% Confidence Intervals	
Sample No.	Count [F/(P+F)]	p (%)	L (%)	U (%)	Count [F/(P+F)]	p (%)	L (%)	U (%)
36	not counted							
37	not counted							
†54*	22/828	2.7	1.7	4.0	5/899	0.6	0.2	1.3
55	not counted							
61	not counted							
†62	4/1395	0.3	0.1	0.7	0/1410	0.0	0.0	0.2
†63	no fibers observed on second scan							
†65	no fibers observed on second scan							
†66	18/1545	1.2	0.7	1.8	10/1598	0.6	0.3	1.1
70	not counted							
71	not counted							
72	not counted							
73	not counted							
78	not counted							
80	not counted							
81	not counted							

* Incompatible replicates.

† Samples selected for second scan.

Table 4 (continued)

Sample No.	Analyst 1 Count [F/(P+F)]	Analyst 1 p (%)	Analyst 1 95% Confidence Intervals L (%)	Analyst 1 95% Confidence Intervals U (%)	Analyst 2 Count [F/(P+F)]	Analyst 2 p (%)	Analyst 2 95% Confidence Intervals L (%)	Analyst 2 95% Confidence Intervals U (%)
†82	no fibers observed on second scan							
84	not counted							
86	not counted							
†88	no fibers observed on second scan							
89	not counted							
91	not counted							
93	not counted							
†98	18/746	2.4	1.4	3.8	9/522	1.7	0.8	3.2
†101	2/200	1.0	0.1	3.6	6/230	2.6	1.0	5.6
102	not counted							
107	not counted							
†108*	56/833	6.7	5.1	8.6	18/1165	1.5	0.9	2.4

* Incompatible replicates.

† Samples selected for second scan.

Table 5

Results on the 21 Samples Identified as Containing Fibers by Only One Analyst on the Initial Scan

Sample No.	Analyst 1 Count [F/(P+F)]	Analyst 1 p (%)	Analyst 1 95% Confidence Intervals L (%)	Analyst 1 95% Confidence Intervals U (%)	Analyst 2 Count [F/(P+F)]	Analyst 2 p (%)	Analyst 2 95% Confidence Intervals L (%)	Analyst 2 95% Confidence Intervals U (%)
35	not counted							
39	29/813	3.6	2.4	5.1	9/550	1.6	0.7	3.1
40	not counted							
44*	100/905	11.0	9.1	13.3	81/418	19.4	15.7	23.5
48	not counted							
51*	45/833	5.4	4.0	7.2	39/1354	2.9	2.0	3.9
53*	50/1091	4.6	3.4	6.0	42/1771	2.4	1.7	3.2
57	not counted							
59	not counted							
64	not counted							
67	not counted							
69	not counted							
74	not counted							
79	not counted							
83*	18/430	4.2	2.5	6.5	3/630	0.5	0.1	1.4
97*	36/1861	1.9	1.4	2.7	18/1693	1.1	0.6	1.7
99*	30/1212	2.5	1.7	3.5	1/739	0.1	0.0	0.8

* Incompatible replicates.

Table 5 (continued)

Sample No.	Analyst 1 Count [F/(P+F)]	Analyst 1 p (%)	Analyst 1 95% Confidence Intervals L (%)	Analyst 1 95% Confidence Intervals U (%)	Analyst 2 Count [F/(P+F)]	Analyst 2 p (%)	Analyst 2 95% Confidence Intervals L (%)	Analyst 2 95% Confidence Intervals U (%)
100	not counted							
103	not counted							
105*	86/1498	5.7	4.6	7.0	27/777	3.5	2.3	5.0
106	not counted							

* Incompatible replicates.

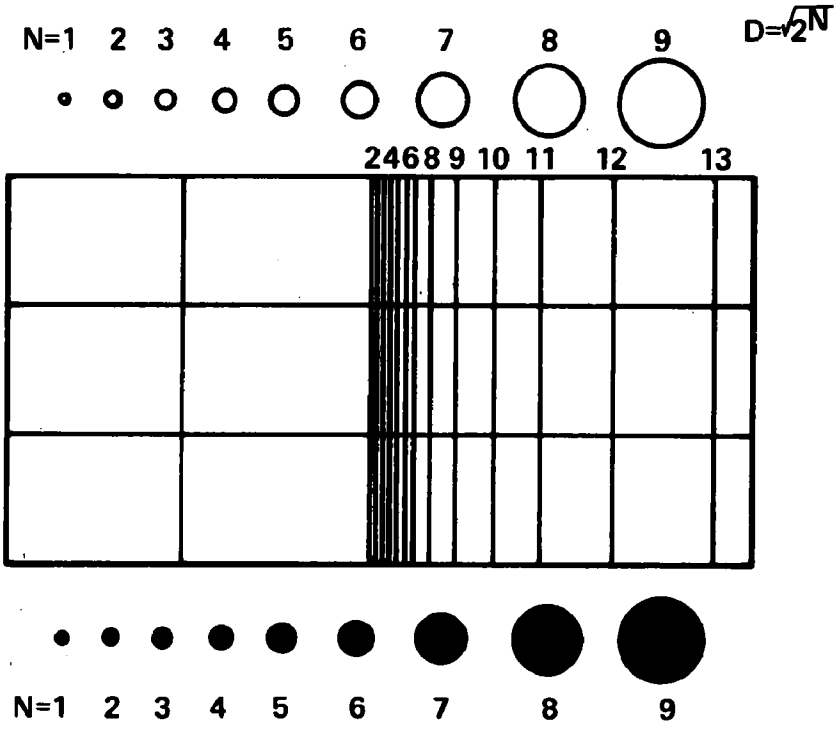


Figure 1. Porton Reticle

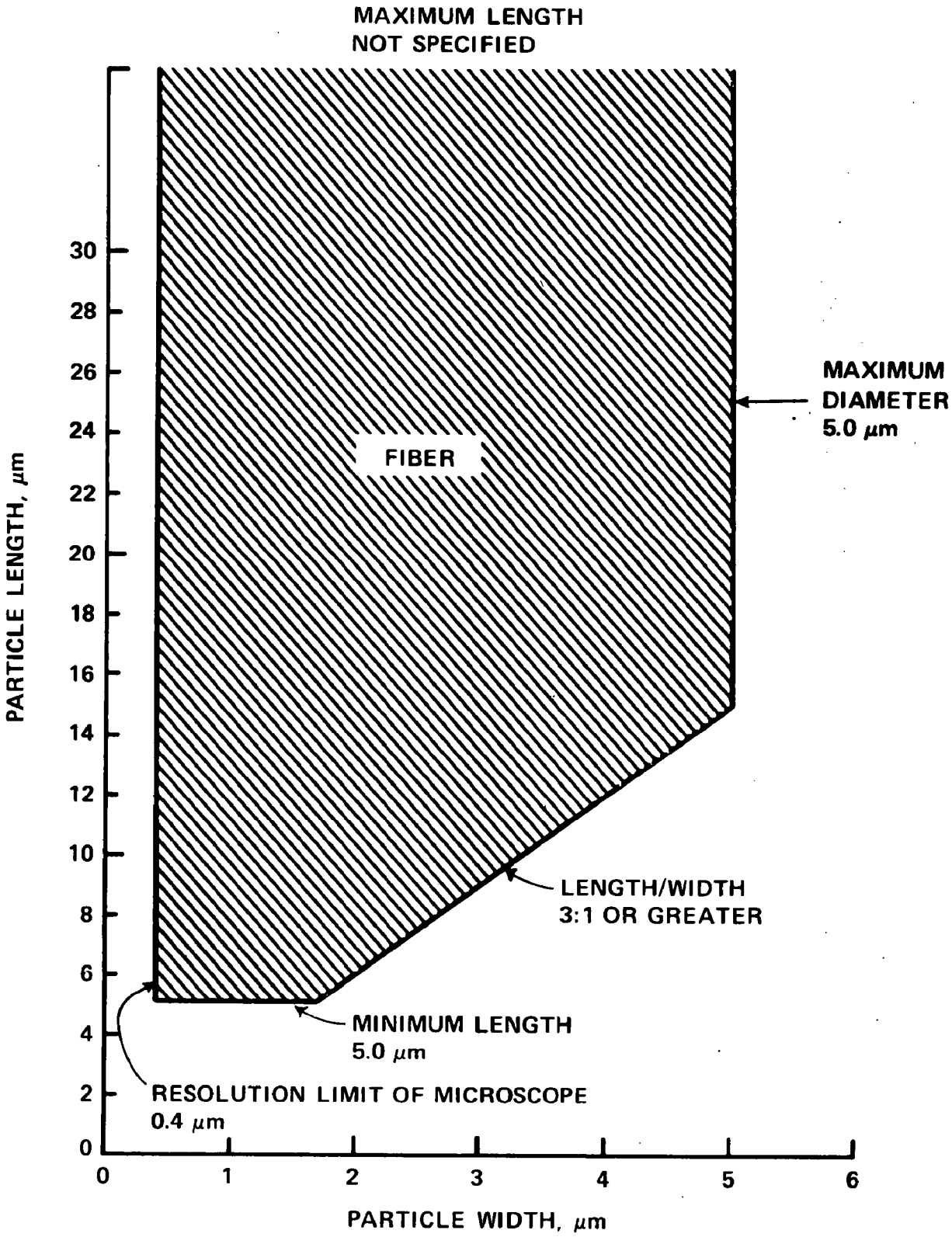
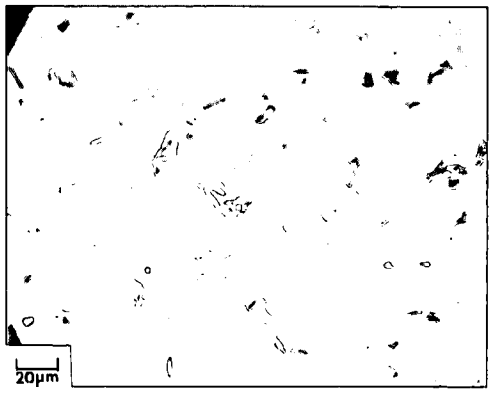


Figure 2. Fiber dimensions that would be observed as per OSHA definitions and resolution limit of microscope.

Sample 92

Light Microscope

Scanning Electron Microscope



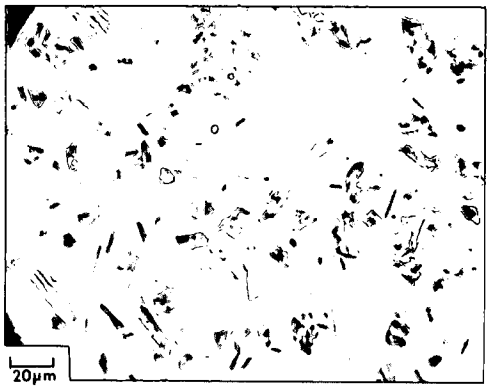
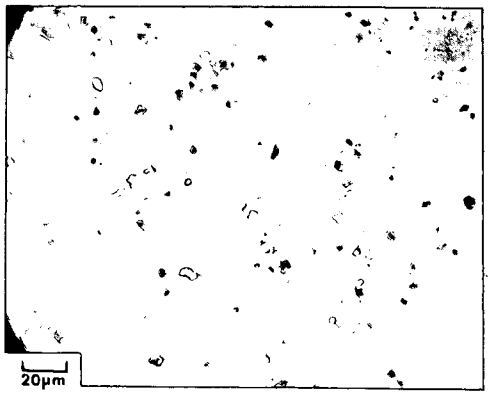
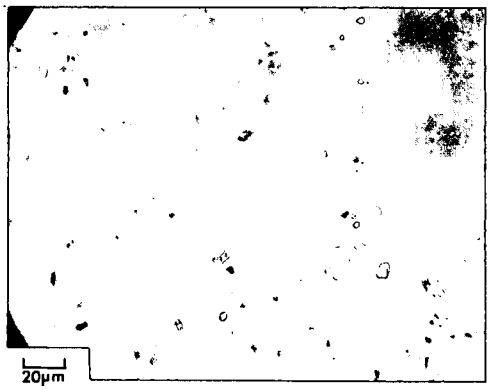


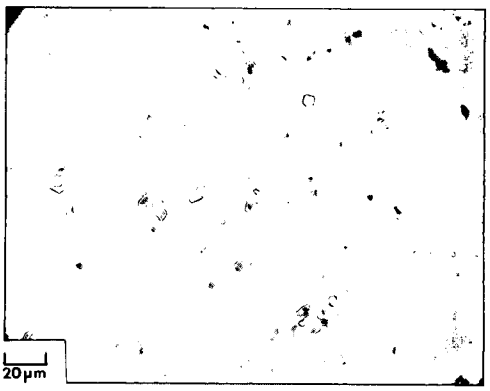
Figure 3a

Sample 81

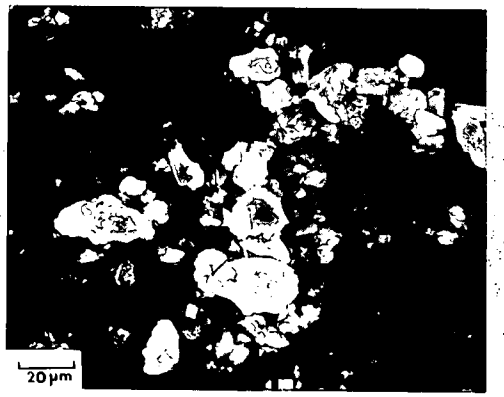
Light Microscope







Scanning Electron Microscope



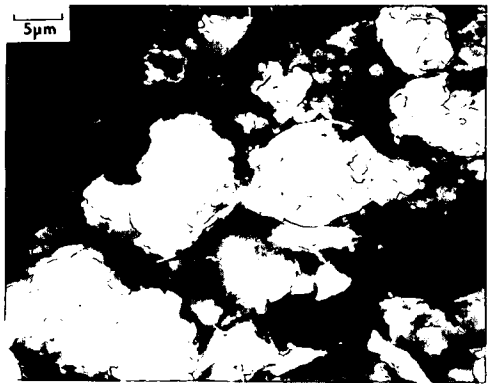
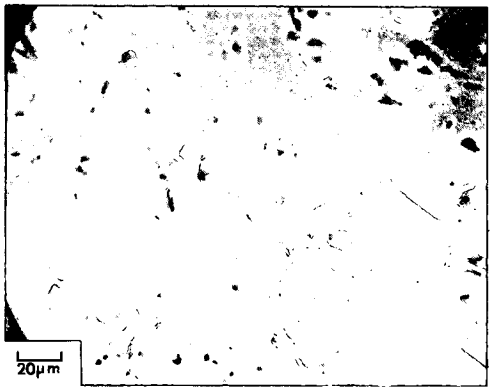
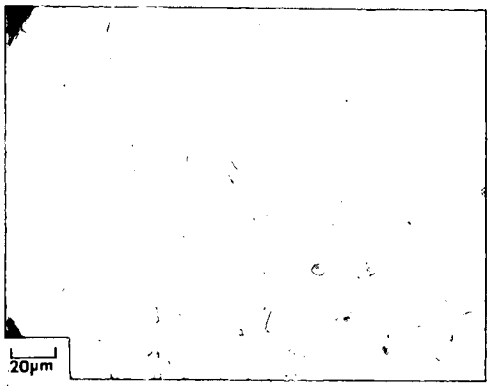


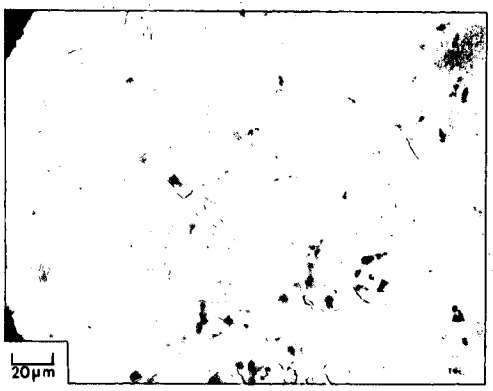
Figure 3b

Sample 108

Light Microscope







Scanning Electron Microscope

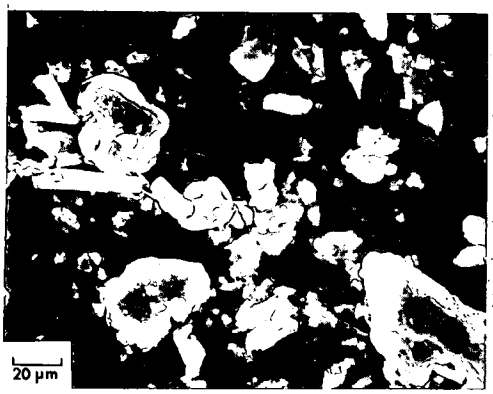


Figure 3c