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

Prepared by the Staff of the
Analytical Chemistry Division, P. D. LaFleur, Chief
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National Bureau of Standards
Washington, D. C. 20234

May 1977

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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.,

<u>Mineral</u>	<u>Refractive Index</u>
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 Zauszman, 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) $n^{25\text{ }^\circ\text{C}} = 1.546$
 - 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

* 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 $n^{25\text{ }^\circ\text{C}}$ 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

* 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.

* 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

<u>Sample</u>	<u>Description</u>
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)

<u>Sample</u>	<u>Description</u>
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)

<u>Sample</u>	<u>Description</u>
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 aggre- gation (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)

<u>Sample</u>	<u>Description</u>
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)

<u>Sample</u>	<u>Description</u>
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				Analyst 2			
	Count [F/(P+F)]	p (%)	95% Confidence Intervals		Count [F/(P+F)]	p (%)	95% Confidence Intervals	
			L (%)	U (%)			L (%)	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	18.8	15.9	22.0	127/600	21.2	18.0	24.6
	100/848	11.8	9.7	14.2	76/866	8.8	7.0	10.9

* Incompatible replicates.

Table 2 (continued)

Sample No.	Analyst 1				Analyst 2			
	Count [F/(P+F)]	p (%)	95% Confidence Intervals		Count [F/(P+F)]	p (%)	95% Confidence Intervals	
			L (%)	U (%)			L (%)	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 87/2237	3.5 3.9	2.7 3.1	4.4 4.8	79/1685	4.7	3.7	5.8
75*	111/1474 105/1245	7.5 8.4	6.2 7.0	9.0 10.1	109/686	15.9	13.2	18.8
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 72/1674	11.6 4.3	9.6 3.4	13.8 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

Sample No.	Analyst 1				Analyst 2			
	Count [F/(P+F)]	p (%)	95% Confidence Intervals		Count [F/(P+F)]	p (%)	95% Confidence Intervals	
			L (%)	U (%)			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				Analyst 2			
	Count [F/(P+F)]	p (%)	95% Confidence Intervals		Count [F/(P+F)]	p (%)	95% Confidence Intervals	
			L (%)	U (%)			L (%)	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				Analyst 2			
	Count [F/(P+F)]	p (%)	95% Confidence Intervals		Count [F/(P+F)]	p (%)	95% Confidence Intervals	
			L (%)	U (%)			L (%)	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				Analyst 2			
	Count [F/(P+F)]	<i>p</i> (%)	95% Confidence Intervals		Count [F/(P+F)]	<i>p</i> (%)	95% Confidence Intervals	
			L (%)	U (%)			L (%)	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.

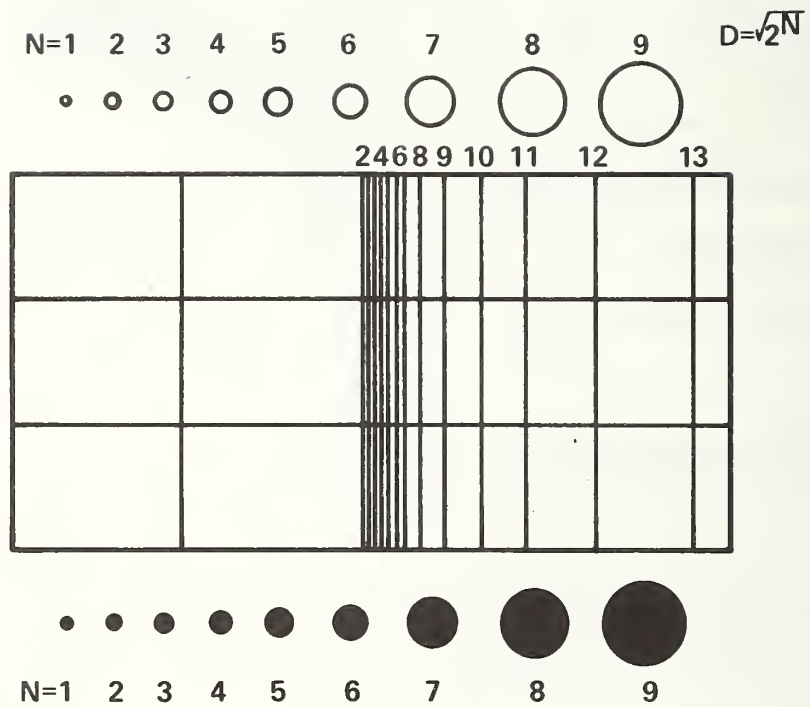


Figure 1. Porton Reticule

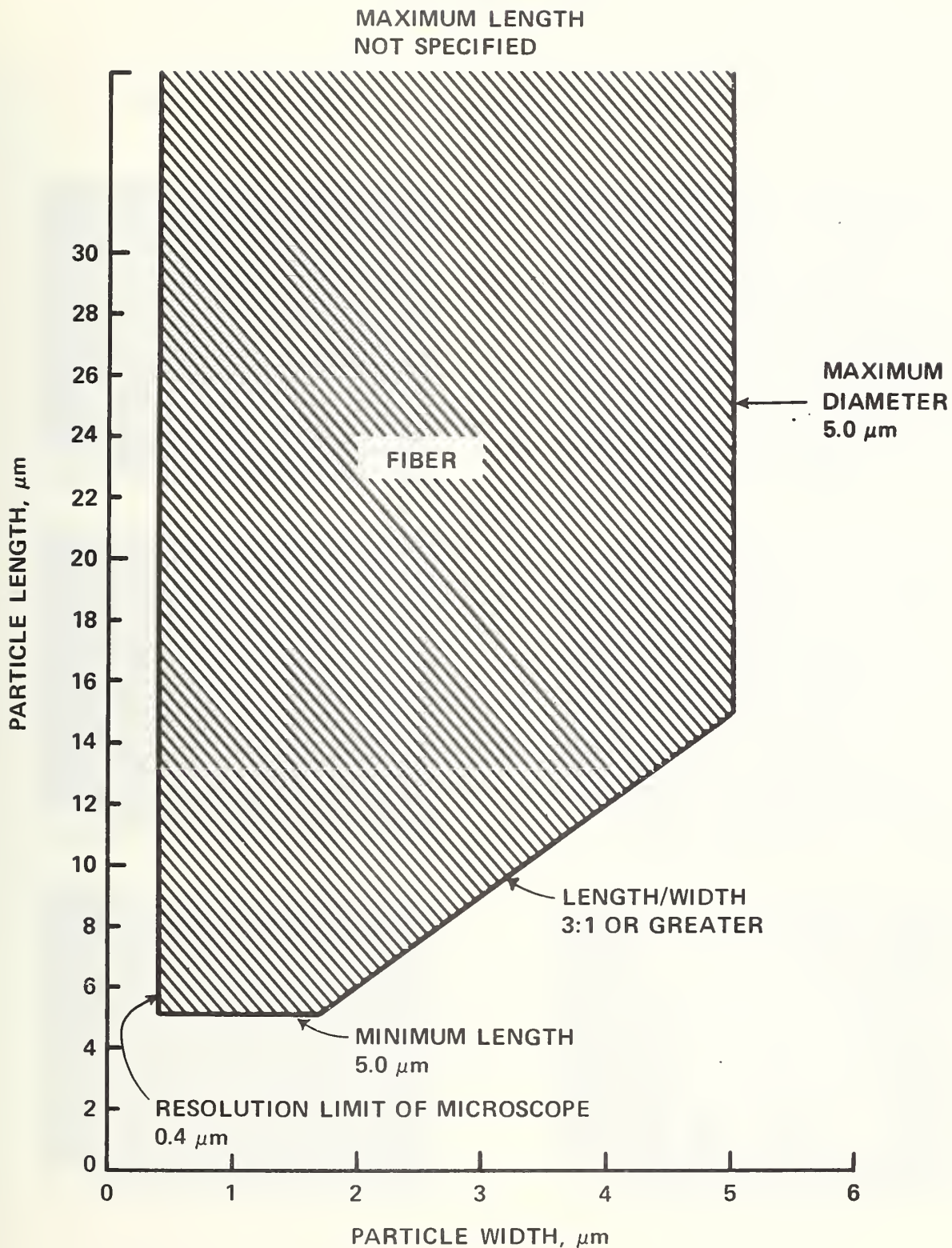


Figure 2. Fiber dimensions that would be observed as per OSHA definitions and resolution limit of microscope.



Sample 92

Light Microscope

Scanning Electron Microscope

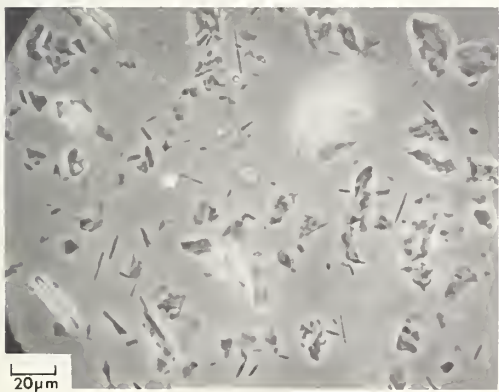
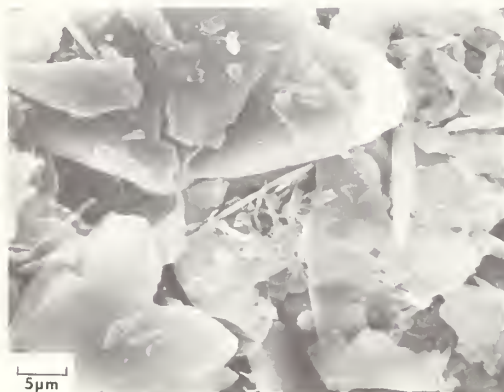
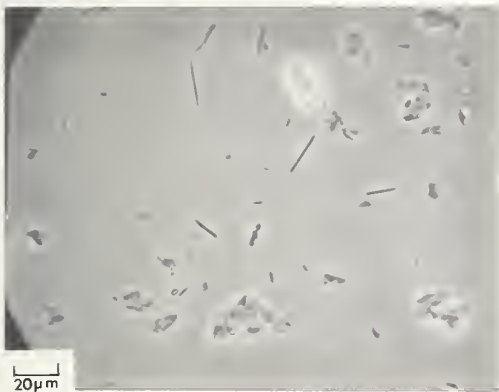
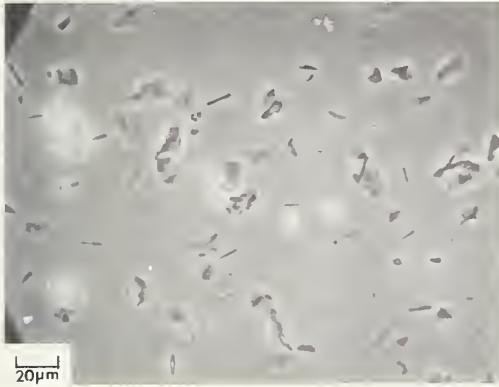


Figure 3a



Sample 81

Light Microscope

Scanning Electron Microscope

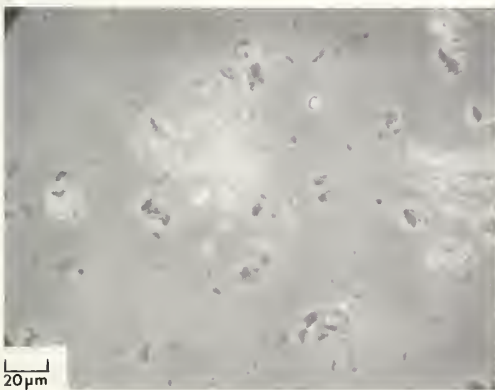
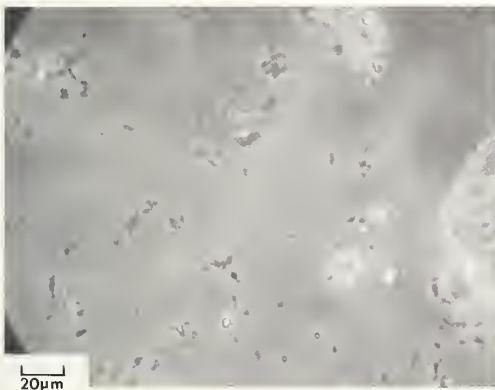
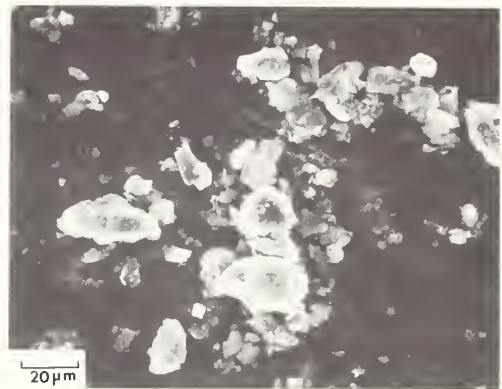
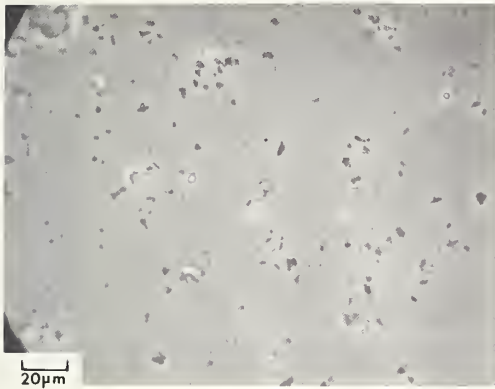


Figure 3b

[Faint, illegible text in the top left quadrant]

[Faint, illegible text in the top right quadrant]

[Faint, illegible text in the middle left quadrant]

[Faint, illegible text in the middle right quadrant]

[Faint, illegible text in the bottom left quadrant]

[Faint, illegible text in the bottom right quadrant]

Sample 108

Light Microscope

Scanning Electron Microscope

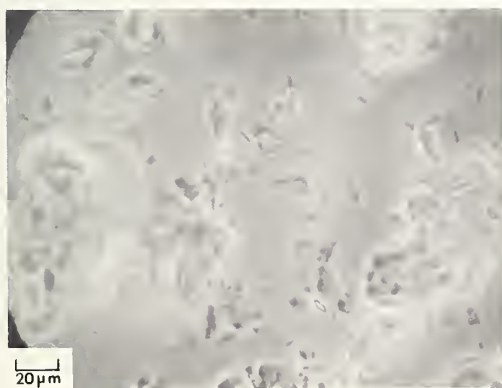
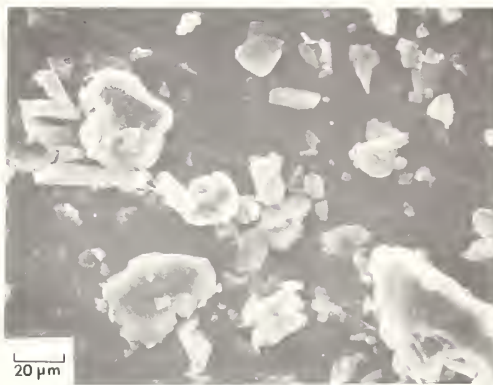
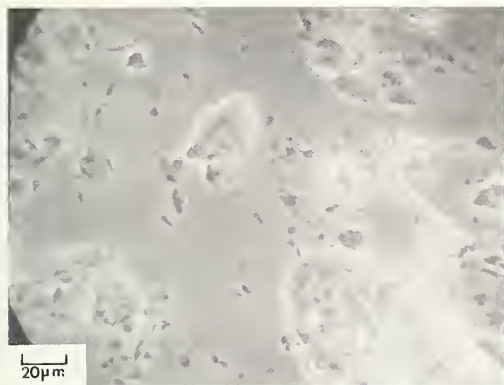


Figure 3c



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



Office of the Assistant Secretary

September 1, 1976

Dr. John Dehoffman
National Bureau of Standards
Building 223, Room B-368
Washington, D. C. 20234

Dear Dr. Dehoffman:

The purpose of this letter is to confirm the request for analysis of talc samples, as discussed by you and Dr. B. K. Kwon of OSHA. We ask that you determine the asbestos content of these samples.

The analysis of talc samples for asbestos is performed by OSHA and NIOSH, but the methodology is being challenged by those regulated by OSHA. Thus, I request that you investigate the asbestos content of these samples independently of OSHA, NIOSH, MESA, the Bureau of Mines or the private sector.

In a real sense, we look to you to resolve, (1) the variability in the definition of asbestos fibers in talc and (2) the asbestos content of these samples, expressed as number of fibers per unit weight or unit volume. Also, the asbestos content as percent of total weight, per the definition in (1) above, would be useful. We recognize it will be necessary to estimate these parameter from a sample of the materials forwarded to you.

Copies of the OSHA standards on asbestos, the proposed revised asbestos standard and two relevant articles are enclosed. Also, I request that you maintain close contact with Dr. B. K. Kwon of OSHA. As we previously discussed, this is an urgent matter and we hope the work can be completed as soon as possible.

Your assistance in resolving this difficult and lingering controversial issue is greatly appreciated. It is our desire to continue to maintain a good working relationship between our two agencies.

Sincerely yours,

A handwritten signature in cursive script that reads "Morton Corn".

MORTON CORN
Assistant Secretary of Labor

Enclosures

23 SEP 1976



UNITED STATES DEPARTMENT OF COMMERCE
National Bureau of Standards
Washington, D.C. 20234

Dr. Morton Corn
Assistant Secretary of Labor
Occupational Safety and
Health Administration
U. S. Department of Labor
Washington, D. C. 20210

Dear Dr. Corn:

I am writing in response to your letter of September 1, 1976, in which you requested that the National Bureau of Standards perform two tasks:

- (1) resolve the variability in the definition of asbestos fibers in talc, and
- (2) determine the asbestos content of a series of talc specimens.

We have briefly investigated in a general way the complex problems and arguments associated with the detection and measurement of asbestos. Although we are not currently performing asbestos analyses, we have several staff members who are experts in the chemical and physical characterization of particulates and who are informed on the talc fiber question as a result of discussions with people from federal agencies and private industry. We have based this reply on their knowledge and the literature available on measurement techniques for asbestos type materials.

NBS is quite often called upon by federal agencies to resolve questions in measurement methods and standards. It is always our practice to discuss these problems with all available experts in federal laboratories, regulatory agencies, private industry and universities to develop an understanding of the current practices, points of controversy and specific measurement problems. The conclusions reached in these studies are developed independently and are a result of NBS technical expertise and judgement. If this type of operation is acceptable to you, we would be willing to participate in a program to develop definition methods and standards for asbestos analysis, which may lead to the resolution of the variability in the definition of asbestos and the question concerning the fiber in talc.

Your first question, resolving the variability in the definition of asbestos fibers, seems to involve a large program that requires the identification of the important variables of the definition as they relate to health effects and then the development of methods to measure these variables effectively. Questions which involve health effects are outside the mission and competence of NBS and in our view, may not be resolvable through chemical and physical analysis alone. However, as a component of a larger program, NBS could compare the OSHA Federal Register methods of analysis with other proposed methods, such as scanning electron microscopy, electron probe microanalysis, selected-area electron diffraction, x-ray diffraction or differential thermal analyses. It is our opinion that this project would make a significant contribution to answering your first question but would have to be performed in cooperation with other federal agencies involved in the development of the important health effect variables. As an output of this project, NBS would probably develop Reference Methods of Analysis and Standard Reference Materials for asbestos. We would estimate that the NBS component of this project would require approximately three man years and cost \$225,000.

In response to your second request, the determinations of the asbestos content of talc samples, we would be in a position to perform this analysis duplicating the methods used by OSHA, NIOSH and MESA as specified in the Federal Register (29 CFR Part 1910). Assuming that there would be 80 samples involved, we estimate that this analysis, which would be billed on a reimbursable basis, would require no more than four man months and not exceed \$25,000. Since this analysis would be based on the definition of asbestos as specified in the Federal Register, it is unlikely that this project would serve to answer your first question. Furthermore, our initial investigation has indicated that there does not appear to be an agreement in the scientific community as to whether a specific fiber of a particular chemical composition is asbestos or not. For example, neither Dana's Manual of Mineralogy (18th ed.) nor Deer et al.'s Rockforming Minerals give a concise definition of either a fiber, or of asbestos per se. Deer et al. say (Vol. 2, p. 223): "The habits of the anthophyllite mineral vary from fibrous and asbestiform to bladed and prismatic". Any number of similar descriptions can be found in the literature.

If you would like us to perform the analysis of the talc samples or if you choose to establish a larger inter-agency program involved in defining asbestos variables, I suggest that members of your staff contact Dr. C. C. Gravatt, Deputy Chief, Analytical Chemistry Division, 921-2852, to work out the details of our involvement. If I can be of any further assistance in this matter, please let me know.

Sincerely,

Original
JOHN D.

John D. Hoffman
Director
Institute for Materials Research

bc: Signer
Dr. E. Ambler
E. Horowitz
G. Sinnott
B. Morrissey
J. Wachtman
R. Hayes
P. D. LaFleur
C. C. Gravatt
I. R. Barkty
K. F. J. Heinrich
J. Taylor
A. Farrar

310:00:CCGravatt:gbh 9-21-76

Retyped:

310.00:CCGravatt:gbh 9-23-76

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



Office of the Assistant Secretary

8 OCT 1976

Dr. John D. Hoffman
Director
Institute for Materials Research
National Bureau of Standards
U.S. Department of Commerce
Washington, D.C. 20234

Dear Dr. Hoffman:

I am writing in reply to your letter of September 23, 1976, in which you presented an approach by the National Bureau of Standards to the performance of two tasks which I previously requested in my letter of September 1, 1976. The tasks are as follows:

- (1) resolve the variability in the definition of asbestos fibers in talc, and
- (2) determine the asbestos content of a series of talc specimens.

I am in total agreement with your statement that the first task encompasses efforts by others than staff of the National Bureau of Standards in order to resolve current uncertainties in the definition of asbestos fibers in talc. It is also clear that for a most meaningful resolution of this question the properties of fibers in talc as they relate to epidemiological studies in the talc industry should be examined. I fear that the formulation of the study plan to approach this task will require a period of time of at least six months. However, I agree with you that this long range project would have a great deal of meaning for the assessment of both the occupational and environmental exposures of individuals to asbestos fibers. Therefore I would like to approach with you the establishment of a study to address Task One.

The second task also requires resolution and I was pleased to learn that the Bureau can undertake this during a period of time represented by approximately four-man months of effort at a cost of \$25,000. This is recognized to not be a research task. However, current disagreements in the occupational health field concerning the results of analyses performed according to procedures in the Federal Register (29 CFR Part 1910) are causing great difficulties to those of us concerned with implementation of the Occupational Safety and Health Act of 1970. Therefore, analyses by your laboratory of approximately 80 samples would

enable us to proceed with enforcement on the basis of the current methodology. Even after conclusion of your analyses there are those who may challenge the current methodology, but we will be in a better position to provide relief to those being exposed to asbestos if there is at least consistency of analysis with respect to current recommended analytical methodology. Therefore, please consider this letter an agreement on my part to proceed with Task Two.

For purposes of discussing both Task One developmental efforts and Task Two procedures, I request that you remain in communication with Dr. Byung Kwon of this agency, who will act as the project officer for both endeavors.

I wish to thank you for your prompt and encouraging response to the needs of the Occupational Safety and Health Administration on this occasion. I hope that we can continue to work with you in the future.

Sincerely,

A handwritten signature in black ink, appearing to read "Morton Corn". The signature is written in a cursive, flowing style.

Morton Corn
Assistant Secretary of Labor



UNITED STATES DEPARTMENT OF COMMERCE
National Bureau of Standards
Washington, D.C. 20234

3 NOV 1976,

Dr. Morton Corn
Assistant Secretary of Labor
Occupational Safety and
Health Administration
U. S. Department of Labor
Washington, D. C. 20210

Dear Dr. Corn:

I am writing in reply to your letter of October 8, 1976. The National Bureau of Standards is pleased to be able to participate in both of the following tasks as described in your letter:

- (1) resolve the variability in the definition of asbestos fibers in talc, and
- (2) determine the asbestos content of a series of talc specimens.

With respect to task number 1, Dr. C. C. Gravatt, Deputy Chief, Analytical Chemistry Division, will work with Dr. Byung Kwon of your staff to plan the NBS component of this interagency program. It is my understanding that they will be meeting in the near future, possibly with representatives of other Federal agencies to begin the development of this program.

The samples to be analyzed by NBS for the second task were picked up from Dr. Kwon by Dr. Gravatt on October 29. The samples were contained in a sealed package sent to Dr. Kwon by NIOSH and were thought to be blind samples i.e., the identification of the source of each sample was not to be in the package. When Dr. Gravatt opened the package on November 1, he found 80 samples indicated by the numbers on the attached sheet but also a document which identified the source of these 80 samples. Dr. Gravatt is the only person at the National Bureau of Standards who had read the document and after checking the sample numbers against the list, the document was placed in a security safe at the National Bureau of Standards. We will not read the document again and will await your recommendations as to its deposition.

2

Dr. Gravatt informed a Mr. Darrell Mathas of Dr. Kwon's Office of the existence of this document. We will initiate analysis of these 80 samples in the near future and expect to complete the work on or before March 1, 1977. We will be in contact with Floyd Matson and Willard Dixon of your Salt Lake City Laboratory concerning the specific procedure to be followed in this analysis. It is our understanding that Dr. Kwon will be forwarding a purchase order to us in the near future to cover the cost of this analysis.

We are glad to be of assistance in both of these projects.

Sincerely,

Original signed by
JOHN D. HOFFMAN
John D. Hoffman
Director
Institute for Materials Research

bc: Signer
Dr. E. Ambler
E. Horowitz
A. Farrar
G. Sinnott
B. Morrissey

310:00:CCGravatt:ghb 11-1-76

Samples Received by NBS from OSHA

October 29, 1976

001	053	082
002	054	083
003	055	084
004	056	086
005	057	087
006 cap open & some loss of material	058	088
	059	089
007	060	091
034	061	092
035	062	093
036	063	094
037	064	095
038	065	096
039	066	097
040	067	098
041	068	099
042	069	100
043	070	101
044	071	102
045	072	103
046	073	104
047	074	105
048	075	106
049	076	107
050	078	108
051	079	109
052	080	
	081	

The following set of notes were taken during a meeting with Mr. Willard C. Dixon of the OSHA Analytical Laboratory, 390 Wakara Way, Salt Lake City, Utah.

Willard Dixon (WD) is in charge of the microscopic analysis of samples for "asbestos" fiber content. As a result of a recent contract NBS has been requested to analyze several samples for "asbestos fibers" according to the procedures used at the OSHA laboratory. The purpose of the visit to Salt Lake City was to obtain information on their methods of microscopic analysis.

Meeting Date: 12/13/76

Sample Preparation

The preparation of bulk talc samples by the OSHA labs in Salt Lake City is done by the following procedure: Two drops of a mounting medium are placed on a clean microscope slide. Next, the end of a paper clip is dipped first into the two drops of solution and then into the bulk sample. The amount of material sticking to the end of the paper clip is mixed with the mounting solution on the slide. Finally, a cover slip is placed over the top of the preparation. The OSHA labs assumed that the talc samples were homogeneous when received. They do, however, draw two "representative" samples from different areas of each bulk talc sample.

Fiber Counting

The fiber counts on the different samples were done according to the procedure outlined in the draft of the article "Asbestos Fibers in Air" which was sent to us by WD on November 4, 1976. In addition, after November 21, 1974 they also used the revisions as proposed in the Department of Labor field information memorandum #74-92 dated November 21, 1974, when analyzing asbestos in talc. This memorandum, among other things, suggests an aspect ratio of five to one rather than three to one as the definition of a fiber. WD commented that the change to the larger aspect ratio did not have a major influence over the results, for tremolite asbestos in Vanderbilt Nylall Talc.

The "bundle of sticks" criterion mentioned in the memorandum is not strictly implemented because of the ambiguous nature of the statement.

The OSHA labs move the slide coverslip when necessary to determine if a given particle is a fiber or a talc plate standing on end.

Analytical Procedure

WD considers light microscopy tests to be of qualitative value only. Quantitative analysis of a mineral would be done by x-ray diffraction, if the results of light microscopy testing are not sufficiently conclusive.

The OSHA labs followed two different analytical procedures for the analysis of talc samples by light microscopy.

The first method was used prior to June 2, 1976. (On or about June 2, 1976 the OSHA labs were visited by a representative of the Vanderbilt Talc Co.) The first method used phase contrast microscopy. The mounting solution was a Cargille liquid with a refractive index of 1.546; 1.47 media was also used. The index of 1.546 was used because this is the index where quartz and chrysotile asbestos dispersion stains blue in bright field using a Zeiss phase contrast microscope. All fibers in the samples were counted according to the procedure outline in the article "Asbestos Fibers in Air," except as modified by memorandum #74-92 for asbestos in talc. When the sample is not on a membrane, this will require modification of the procedure. The procedure doesn't specify the use of auxiliary methods of examination such as polarized light, or retardation plates, however WD will use any auxiliary method he finds helpful in asbestos identification. The study of polarized light color patterns with a first order red retardation plate and crossed polars was an important technique in fiber identification before June 2, 1976.

The second method of sample analysis which was used after June 2, 1976 was dark field dispersion staining. The dark-field effect was produced by mismatching the phase contrast objective and the stop on the annulus in the condenser. The samples were mounted in a Cargille high dispersion liquid with a refractive index of 1.600. The results were reported as two separate fiber counts based on the dispersion staining color. One count reported the fibers which dispersion stained orange. These fibers were considered to be tremolite asbestos. The second count reported fibers which dispersion stained blue. These fibers were not considered to be asbestos.

After careful study of fiber characteristics, morphology, and colors in both bright field and dark field the fibers were counted in bright field at 400 X. If a fiber could not be identified in bright field the counted switched to dark field for identification then back to bright field to continue counting.

According to WD, there is a question concerning the validity of reporting the fibers which dispersion stain blue as not being asbestos. He said the shift in analytical procedure on June 2, 1976 was induced by the discussion with technologists from the Vanderbilt Co. At that time, the Vanderbilt Co. convinced him of the merits of 1.59 or 1.60 media to distinguish between Tremolite asbestos and talc fibers.

WD had learned about Dispersion staining from Walter C. McCrone associates literature and then began using dispersion staining to assist in differentiation using 1.60 media. Just recently, however, WD has received a sample from the California Department of Health which is called "White Tremolite." This material, which is fibrous, dark field dispersion stains blue in a liquid with refractive index of 1.600. If this material is in fact tremolite, then the fiber counts done by dispersion staining would under estimate the tremolite asbestos fiber content of the samples. The OSHA lab is currently studying the "White tremolite to determine if it is in fact tremolite. WD has given us a sample of the material which we are now studying using micro-Raman and x-ray micro-analysis. The sample of tremolite along with a second sample called "purple tremolite" were obtained by the California Department of Health from

Wards Natural Science Establishment, Inc.
P.O. Box 1712
Rochester, New York 14603
phone 716/467-8400

Other Procedures

Routinely the samples of talc are also analyzed by polarized light microscopy. This procedure would separate materials such as fiber glass from asbestos and with a first order red retardation plate helps to identify talc, asbestos fiber, plant and other fibers by the pattern of colors produced and the color changes with rotation of the stage.

Miscellaneous Information

Mounting Liquids:

WD commented that the liquids made by Cargille with refractive indices less than 1.4 may deteriorate, but the liquids with refractive index above this value are very stable.

Sample Grinding:

The OSHA lab assumes that the talc samples sent to them are representative of the bulk product or of airborne talc dust. The material is not further ground in the lab since in further grinding the fiber count would change.

Mounting Liquid:

WD said that if the talc samples were mounted in the mounting solution (see membrane filter method mentioned in the article "Asbestos Fiber in Air"), which has a refractive index of approximately 1.47, then it would not be possible to tell asbestos from talc in the dispersion staining method.

Filter Standards:

The filter samples sent to us by WD were the NIOSH Proficiency Analytical testing samples (PAT). These samples are used for inter-laboratory checks on counting. The fibers on these samples are extremely fine chrysotile fibers and are considerably more difficult to count than the fibers in the talc samples.

Problems with Inexperienced Counters:

WD said there are two problems with new counters:

1. They may not use the fine focus enough to detect fine fibers.
2. They may count membrane filter structure as fibers.

The second problem applies specifically to air samples so that it will not affect the counts on the talc samples. Dixon commented that the fibers in the talc samples are fairly large and are easily seen at 400X so that the first problem should not be severe.

Asbestos Material in Talc Samples:

The main asbestos material detected in the talc samples was tremolite. WD has learned since talking to John Small that some of the fibers he had thought to be talc fibers in Nyttall may be anthophyllite asbestos fibers.

Mismatching Phase Contrast Objective and Annulus:

When the operator mismatches the phase contrast microscope, the magnification must be recalibrated.

Method of Identifying Wollastonite in the Presence of Amphiboles:

The sample is treated with hot concentrated HCl for 20-30 minutes. The silicon dissolves leaving a Wollastonite shell behind. This shell is white and spongy and retains the original shape of the mineral. The amphiboles are unaffected by the HCl. The Wollastonite can also be distinguished from the amphiboles by its shape. It has a feathering appearance on the ends while the amphiboles usually have a prismatic to right-angle cleavage. (i.e., 56°, 124° and some cleavage closer to 90), but cleavage faces are straight rather than tapering.

Plant Fibers:

Several varieties of vegetal fibers, especially under low magnification, may be identified as asbestos fibers. One way to eliminate the interference is to ignite the sample. It should be noted that the sample must not be heated above about 500°C since chrysotile converts to forsterite $\text{Mg}_2(\text{SiO}_4)$ a part of the olivine series at approximately 650°C.

Nytall 99:

The Vanderbilt sample Nytall 99 is believed by WD to be the sample in contention. The material does contain tremolite which was determined by x-ray diffraction. This fact has been confirmed by Vanderbilt. The main question is the identification of the fibers which dispersion-stain blue, and the definitions of a fiber.

Bulk Standards:

WD said that there would be no way to prepare bulk talc samples doped with asbestos. The only way they could be calibrated was to have a multi-lab round robin. He has never had any standards for bulk talc made.

Sample Size:

WD said that many of the light tests would fail if the fibers are much less than 1 μm in diameter.

"Bundle of Sticks" Effect

For interpretation of the "bundle of sticks" effect, WD usually used his own judgment. One solution where there may be a problem is in a sample with a palisading effect, i.e., tremolite (see attached figure).

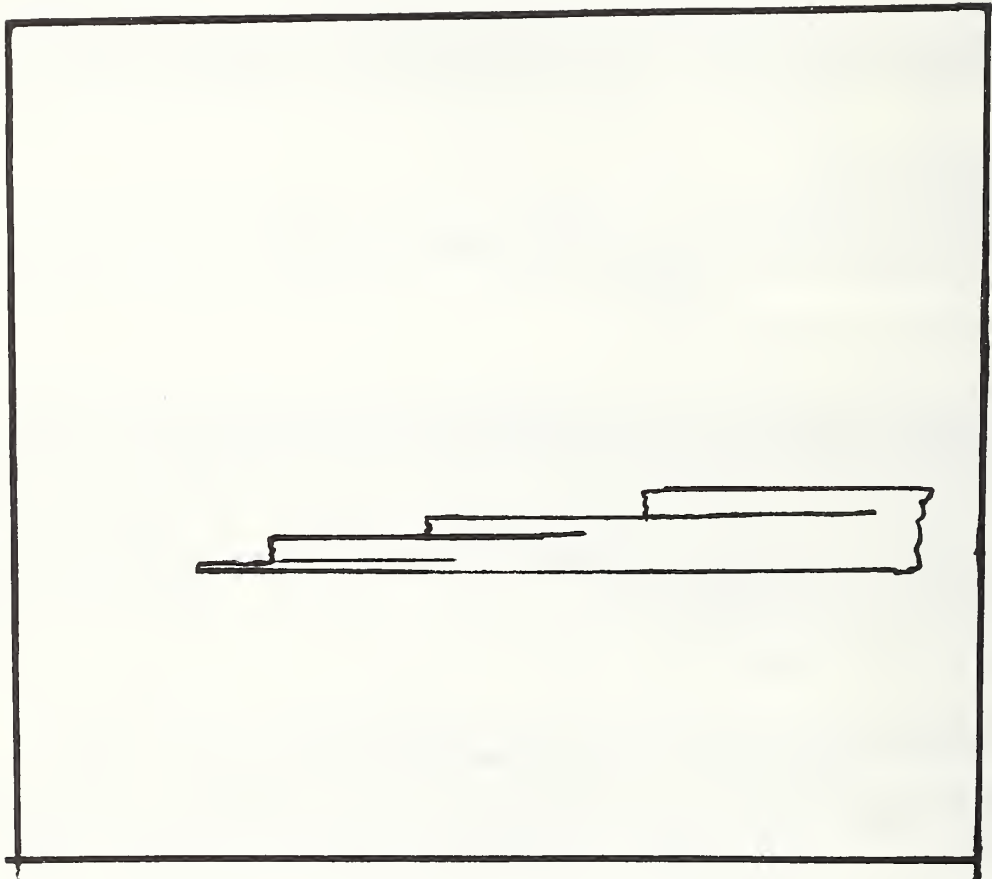
WD says he would call this a fiber, but Vanderbilt may not. He says he employs the 3 μm width and 5:1 aspect ratio to determine what is a fiber in talc samples.

Interference Samples:

WD showed me several talc samples and materials which can be confused with either talc or one of the asbestos minerals. These materials were mounted in both 1.546 and 1.600 liquids and studied with polarized, phase contrast and dispersion staining microscopy. They included:

CaSO ₄	Talc
Mica	Soap stone talc
Wollastonite	Nytall
Fiberglass	
Plant fibers	
Diatomaceous earth	
Vermiculite	
"Asbestos imitation"	
Natural gypsum	

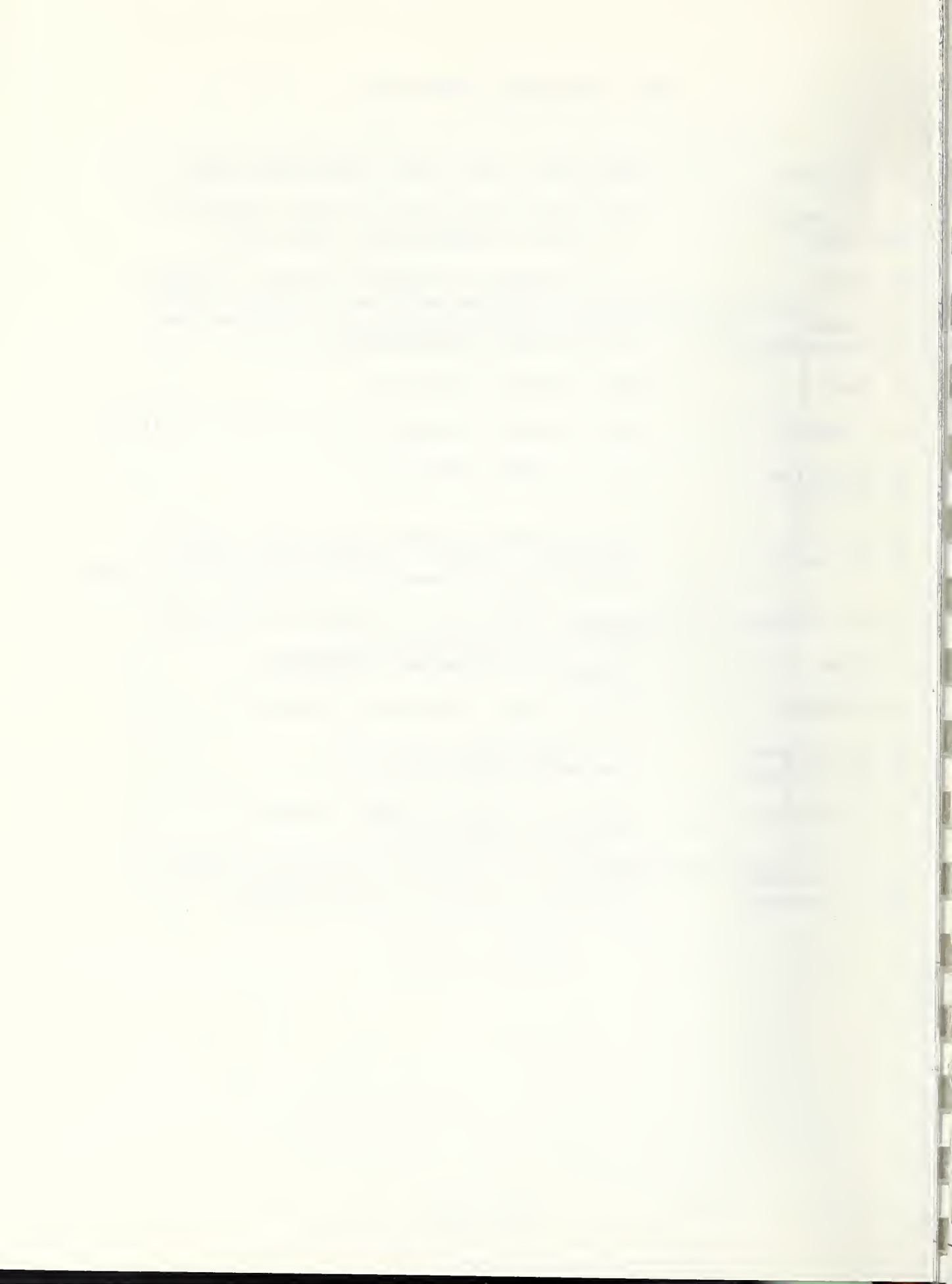
A sample of each of these materials except the talcs was obtained for study at NBS.



PALISADING EFFECT

List of Persons Consulted

W. C. Dixon	OSHA Anal. Lab., Salt Lake City, Utah
W. J. Campbell, and staff	Particulate Mineralogy Unit, Bureau of of Mines, College Park, Maryland
M. Ross	U.S. Geological Survey, Reston, Virginia
B. Mason E. Jarosewitch	Museum of Natural History, Smithsonian Institution, Washington, D. C.
W. Bank	MESA, Denver, Colorado
R. Thompson	EPA, Research Triangle Park, North Carolina
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C. Ruud	University of Denver, Colorado
W. McCrone	McCrone Labs., Chicago, Illinois
A. M. Harvey C. S. Thompson	R. T. Vanderbilt Co., Inc. Norwalk, Connecticut
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H. D. Stanley R. E. Norwood	Pfizer Inc., Minerals, Pigments & Metals Division, Easton, Pennsylvania



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5. SUPPLEMENTARY NOTES

6. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.)

The fiber content of 80 talc samples supplied to NBS by the Occupational Safety and Health Administration of the Department of Labor have been determined. A statistical analysis of a portion of the samples has been made.

17. KEY WORDS (six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons)

Asbestos; fiber; microscopy; talc

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