PLAINTIFF'S EXHIBIT
CHV-595

Chevron

Standard Oil Company of California

San Francisco, California
July 19, 1973

API ASBESTOS COUNTING STUDY
File 110.1

MR. J. A. SPENCE:

After reviewing Mr. R. S. Brief's report on the "Collaborative Study of the Microscopic Counting of Asbestos Samples" and the letters to Mr. Renes from the Subcommittee members, I feel that the study should be repeated. It should be noted that, although the results were discouraging enough, the variation among results did not even include the additional variation that would result from individual handling and mounting of the samples.

The NIOSH Chemical Reference Laboratory (CRL) tests the proficiency of governmental laboratories which analyze samples for OSHA compliance. They have developed a method of preparing artificial asbestos samples by suspending asbestos fibers in toluene and filtering the suspension on 0.8 micron membrane filters. I believe that samples thus prepared would provide a better collaborative study. Details of the CRL method should be available from Mr. Frederick H. Colen, Assistant Chief, Chemical Reference Laboratory, 944 Chestnut Ridge Road, Room 122, Morgantown, West Virginia 26505.

I have requested a set of asbestos samples from CRL and received a promise that it would be sent. Should API repeat the collaborative study, either as before or as recommended above, we should request to be included.

In his report (Page 3), Mr. Brief reports medical data obtained in a personal communication from Dr. N. Weaver on over one hundred petroleum industry insulators which reveal "no lung impairment of any type." Since we have been getting scattered reports of pulmonary function decrement from our operating companies, it would be worthwhile to obtain the specifics of the study in a quotable form. I would appreciate your requesting this information through your personal contacts.

S. L. DRYDEN

SLD

SLD:ll

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HUMBLE OIL & REFINING COMPANY

BATON ROUGE, LOUISIANA 70821

MANUFACTURING DIVISION
BATON ROUGE REFINERY
NEILL K. WEAVER, M.D., F.A.C.P.
MEDICAL DIRECTOR

April 6, 1966

POST OFFICE BOX 221

Mr. Allan E. Dooley
Health Division
Texaco, Inc.
135 East 42nd Street
New York, New York 10017

Re: Possible Health Effects
of Asbestos

Dear Mr. Dooley:

Workers with potential exposure to asbestos have been studied at two Humble Manufacturing Plants, and findings to date with regard to neoplasia are summarized on the attachment. (No identification is shown on this page, so that the data can be included in your A. P. I. survey without revealing the source.)

While cases of asthma, emphysema, Hamman-Rich syndrome (one case), pneumonitis, pleuritis, and focal fibrosis have been found in the exposed population, no cases of diffuse, progressive pulmonary fibrosis compatible with the clinical and radiologic diagnosis of classical asbestosis have been identified. Detailed clinical, epidemiological and environmental studies are continuing in an effort to define the significance, if any, of the disorders found in the exposed workers.

Yours very truly,

Neill K. Weaver

Neill K. Weaver, M. D.

NKW:pgd
Attachment

cc: Dr. V. C. Baird
Attention: Mr. J. W. Hammond

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Workers with Potential Exposure to Asbestos
(Population defined as of 1955, with retrospective and prospective studies from that point in time):

Insulators (active employees):	75
Insulators (inactive employees):	70
Grease Plant (active employees):	6
Grease Plant (inactive employees):	1
	152

Neoplasms Encountered:

Case 1: Bronchogenic Carcinoma, diagnosed 1956, age 48 (died, 1958).

Case 2: Adenocarcinoma of colon, grade 2, diagnosed 1956, age 60.

Case 3: Basal Cell Carcinoma, lower eyelid, diagnosed 1948, age 49.

Case 4: Squamous Cell Carcinoma larynx, diagnosed 1959, age 64.

Case 5: Cancer of lower lip, diagnosed 1963, age 57.

Case 6: Myelogenous Leukemia, diagnosed 1963, age 52.

Case 7: Transitional Cell Carcinoma of the Bladder, diagnosed 1961, age 70 (died 1962).

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First Order Analysis: Asbestos Workers cf. Controls

CA	E (97)	C (97)
	Skin 3	Skin 3
	Colon 1	Rectum 1
	Larynx 1	Squamous cell lung 1
	Bladder 1	Prostate 1
	Total 6	Leiomyosarcoma jejunum 1
		Total 7

Emphysema	5	5
Asthma	3	1
Fibrosis	1	2
Total	9	8

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AMERICAN PETROLEUM INSTITUTE
1801 K STREET, NORTHWEST
WASHINGTON, D.C. 20006
(202) 833-5742

JAMES M. McNERNEY, M.P.H.
Staff Toxicologist
Environmental Affairs-Medical

August 9, 1973

Neill K. Weaver, M.D.
Exxon Company, USA
P.O. Box 2180
Houston, TX 77001

Dear Neill:

With respect to the attached correspondence, what is the status of the continuing study of insulators which you discussed with Dick Brief and he referred to on page 3 in his report, "Collaborative Study of the Microscopic Counting of Asbestos Samples"?

Please let me know how you would like me to handle Jack Spence's inquiry? If data or reports are available, I personally would like to review them.

As you are probably aware, the workers in one of our member company refineries are being studied by Dr. Selikoff and I understand he is obtaining quite interesting results. The Exxon study may prove to be invaluable in the near future.

Looking forward to seeing you on September 4th, if not sooner.

Sincerely yours,

James M. McNerney

JMM/tmd

Attachments

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Standard Oil Company of California

San Francisco, California
July 19, 1973

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File 110.1

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S. L. DRYDEN

SLD

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Standard Oil Company of California
225 Bush Street, San Francisco, CA 94104

D. H. Barber
Manager
Safety Division
Personnel Department

July 25, 1973

James M. McNerney, M.P.H.
Staff Toxicologist
American Petroleum Institute
1801 K Street, NW
Washington, D.C. 20006

Dear Jim:

You will be interested in the attached comments by Mr. S. L. Dryden concerning the asbestos counting study. We would be interested in participating in future studies.

I concur with Stan's belief that it would be most helpful if we could obtain more information on the details and results of the medical study discussed by Dr. N. Weaver. Are these data to be published? If not, can they be obtained on a confidential basis?

Sincerely yours,

J. [illegible] Greence

Attachment
cc: Mr. L. E. Renes

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COLLABORATIVE STUDY OF THE MICROSCOPIC COUNTING OF ASBESTOS SAMPLES

By

Richard S. Brief
Esso Research and Engineering Company
Medical Research Division
P.O. Box 45
Linden, New Jersey 07036

May 15, 1973

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COLLABORATIVE STUDY OF THE MICROSCOPIC COUNTING OF ASBESTOS SAMPLES

By
Richard S. Brief
Esso Research and Engineering Company
Medical Research Division
P. O. Box 45
Linden, New Jersey 07036

Background Information[1]

The term "asbestos" refers to any of six naturally occurring crystalline mineral hydrated silicates: actinolite, amosite, anthophyllite, chrysotile, crocidolite, and tremolite. The degree of hydration varies from approximately 1.5 percent in some deposits of crocidolite to approximately 14.5 percent in the majority of the deposits of chrysotile. These minerals display a wide range of chemical compositions, as is indicated in Table 1.

The several types of asbestos were formed by the metamorphosis of serpentine and amphibole minerals, both classes of which contain silica. Chrysotile, which is a hydrated silicate of magnesia, is the principal crystalline form of serpentine. Over 90% of the world's asbestos production comes from chrysotile and it is the asbestos material used almost exclusively in the United States. The remaining five types of asbestos are crystalline forms of amphibole minerals. Crocidolite, frequently called blue asbestos, is associated with riebeckite. Amosite is the only asbestos of grunerite that is of commercial value. Anthophyllite is thought to be evolved from the metamorphosis of olivine. Tremolite occurs in crystalline, dolomitic limestone and is called actinolite when iron is present in amounts greater than 2 percent.

Electron microscopy reveals that the smallest fibrous subdivision of a chrysotile fiber, called a fibril, has an average outside diameter of 0.034 micrometer (μm). Further, it has been shown that the chrysotile fibril is a hollow tube, rather than a solid cylinder, with an average inside diameter of 0.018 μm. A suggested model views the chrysotile fiber as a tightly packed collection of fibrils, the interiors and interstices of which are filled with crystal fragments or amorphous material of the same chemical composition; the interfibril binding forces are relatively weak so that each "fiber" can usually be subdivided into large numbers of "fibers" of the original length. The elementary crystal structure, or fibril, of the amphibole asbestoses forms a solid cylinder considerably larger in outside diameter than the chrysotile fibril; the average outside diameter ranges from 0.1 to 0.2 μm. Although the majority of dry-milled asbestos fibers each contain many fibrils, smaller numbers of fibers composed of only one or two fibrils are always present; a considerable number of these fibers of smaller diameter are found in asbestos dust.

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TABLE 1

Properties of Asbestos Varieties

Property	Chrysotile	Crocidolite	Amosite	Anthophyllite	Tremolite	Actinolite
Chemical Formula	$Mg_3Si_2O_5(OH)_4$	$Na_2Fe_5Si_8O_{22}(OH)_2$	$(FeMg)_6Si_8O_{22}(OH)_2$	$(FeMg)_7Si_8O_{22}(OH)_2$	$Ca_2Mg_5Si_8O_{22}(OH)_2$	$(CaMgFe)_6Si_8O_{22}(OH)_2$
Essential Composition	Hydrous silicate of magnesia	Silicate of sodium and iron with some water	Silicate of iron and magnesium; higher iron than anthophyllite	Magnesium silicate with iron	Calcium and magnesium silicate with some water	Calcium-magnesium-iron silicate; water up to 5%
Percentage Chemical Composition						
SiO_2	37. to 44.	49. to 53.	49. to 53.	56. to 58.	51. to 62.	--
MgO	39. to 44.	0. to 3.	1. to 7.	28. to 34.	0. to 30.	--
FeO	0.0 to 6.0	13. to 20.	34. to 44.	3. to 12.	1.5 to 5.0	--
Fe_2O_3	0.1 to 5.0	17. to 20.	--	--	--	--
Al_2O_3	0.2 to 1.5	--	2. to 9.	0.5 to 1.5	1.0 to 4.0	--
H_2O	12.0 to 15.0	2.5 to 4.5	2. to 5.	1.0 to 6.0	0. to 5.0	--
CaO	trace to 5.0	--	--	--	0. to 18.	--
Na_2O	--	4.0 to 8.5	--	--	0. to 9.	--
CaO and Na_2O	--	--	0.5 to 2.5	--	--	--
Color	Green, gray, amber to white	Blue	Gray, yellow to dark brown	Yellowish, brown, grayish white	Gray-white, greenish, yellowish, bluish	Greenish
Texture	Soft to harsh, also silky	Soft to harsh	Coarse but somewhat pliable	Harsh	Generally harsh, sometimes soft	Harsh
Luster	Silky	Silky to dull	Vitreous, somewhat pearly	Vitreous to pearly	Silky	Silky
Refractive Index	1.50 to 1.55	1.7 pleochroic	1.64±	1.61±	1.61±	1.63± weakly pleochroi

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Workmen handling or working with asbestos and asbestos-containing materials have experienced adverse health effects. Inhalation of the asbestos fibers has been found to be etiology of disease. Thus, to minimize the effects resulting from exposure, it is essential to control the release of fibers into the breathing zone. Assessment of airborne concentrations of asbestos fibers is necessary to assure that controls are effective and workmen are not excessively exposed.

Of the various ways to sample and analyze suspended asbestos fibers in air, with particular relation to the respiratory hazard they create, the generally recognized best method today is collection on membrane filters and analysis by microscopic counting at 400-450X magnifications under phase-contrast illumination. This procedure was collaboratively tested and is the subject of this report.

Health Experience and Exposure Limits

Asbestos fibers can cause asbestosis (a form of lung fibrosis), lung cancer, and mesothelioma of the pleura and peritoneum (a form of cancer of the lining of the lung or abdominal cavity). This knowledge has arisen from studies of asbestos miners in South Africa and from asbestos fabricators in Great Britain. Epidemiologic studies of unionized asbestos workers in the United States also reveal an increased incidence of these diseases among those workers. It is important to recognize, however, that working with asbestos-containing materials per se does not necessarily produce the conditions noted. For example, in the petroleum industry a medical evaluation of over one hundred insulators, with 20 or more years experience, revealed that they showed no lung impairment of any type. This continuing study covers approximately 3000 man-years of exposure to asbestos fibers and suggests that there must be a threshold dose for producing asbestos created disease[2].

As a result of the untoward experience with asbestos, limits for airborne concentration have been set by various groups. In the United States, the present limit established by the Occupational Safety and Health Administration[3] (OSHA) is related to fibers longer than 5 um (a fiber traditionally relates to a structure which has a length to width ratio greater than 3:1). According to OSHA, the permissible exposure to airborne concentrations of asbestos fibers are shown in Table 2.

TABLE 2

OSHA EXPOSURE LIMITS

Effective Date	Fibers Greater Than 5 μm Per Cubic Centimeter of Air	
	8-Hour Average	Ceiling
Until June 30, 1976	5	10
After July 1, 1976	2	10

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The method of measurement requires that air samples be collected on membrane filters and that the fiber counts be done at 400-450X magnifications (with a 4 mm objective) using phase contrast illumination. The Asbestos Textile Institute[4] and the National Institute for Occupational Safety and Health (NIOSH)[5] have described this optical method in detail. The NIOSH procedure is appended.

Evaluation of the Method

Early efforts to standardize this method among government agencies suggested wide variation between results obtained by operators in different laboratories[6]. This was disquieting since the OSHA standard could be violated by the variability of the measurement scheme This could create a health problem if the asbestos count was in error on the low side and an excessive requirement for controls if the counts erred on the high side. Methodology which is required as part of regulations should properly be tested to understand the variance to be expected so that this information could be incorporated in implementation plans.

A review of the possible reasons for the variation suggests that the errors could reflect operator capabilities with microscopic counting procedures. In addition, different microscopes and optics, including the filters and magnification, could affect results. The operator is required to make judgments during the counting procedure to define the fibers to be included in the count. The rules of counting are not clearly defined by the regulations; but we offer some below, based on comments made by Scheinbaum[7].

1. The fibers must fit inside or pass through two predesignated sides of the viewing area formed by a counting reticle. If the fibers pass through either of the undesignated sides they are not included in the count. When the fiber count is very low a full field count is also acceptable provided a flat field objective is used so that aberrations at the edges are minimized.

2. Fibers which just meet at the end or along one length so that they appear to be "L", "T" or "Y" shaped are counted as one fiber. However, fibers which cross so that four ends can be seen are counted as two separate fibers.

3. A clump or mass from which several fibers emanate are counted as one fiber.

During counting, when it is possible to separate non-asbestos fibers from the count this should be done. Glass, mineral wool, synthetics, and natural fibers if included would bias the overall count upward. Typically, these other fibers have a different appearance than asbestos and where it is obvious that they are not asbestos fibers, they should not be included. For

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example, fiberglass fibers are long and straight with a uniform diameter, typically larger than asbestos, and with ends which appear to be fire-polished glass tubing. Mineral wool fibers show an uneven diameter along their length and often have a bulbous center from which numerous fibers emanate.

Because the regulations refer to 5 μm or greater in size, the selection of this length requires another judgment. For the Porton reticle used in our laboratory, a size greater than Porton Reticle number 6 was used to determine those fibers greater than 5 μm.

Background counts must be considered because the membrane filters can contain fibers which may be subsequently counted. However, this is usually a minor problem.

It has been mentioned that the materials once mounted, should be promptly counted to avoid changes in the sample with time resulting from such things as crystal growth, particle migration, evaporation of mounting material, and the like. Sometimes it is difficult to make prompt counts and here is where further work was indicated.

Finally, the calculation relating the count to the air concentration involves definitive measurements of the counting area related to the filtering area and a knowledge of the total air volume of the sample. A stage micrometer is usually used to accurately define the optical counting area and the filter area must be carefully measured. The accuracy of the rotometer used during sampling must also be checked by suitable calibration methods.

Because of the uncertainties described above, it was deemed worthwhile to establish a collaborative test among several industrial laboratories who were actively involved in assessment of asbestos fiber concentrations in air. The technique involved mounting 9 actual asbestos samples plus one blank membrane filter on slides and distributing them to the participants on a random schedule. The mounting media contained 0.05 grams of membrane filter per ml of 1:1 solution of dimethyl phthalate and diethyl oxalate.

The samples were prepared by cutting a wedge-shaped piece of the membrane filter (Millipore Filter Paper Type AA) while in the filter holder (a three-piece plastic container obtained from the Millipore Corporation, Bedford, Massachusetts). The piece was removed from the filter with a scalpel and forceps and placed dust-side up on a drop of the mounting media, which in turn, had been placed on a freshly cleaned, standard (35 mm x 75 mm), microscopic slide. A number 1-1/2 cover slip, carefully cleaned with lens tissue, was placed over the filter wedge. Slight pressure on the cover slip achieved contact between it and the mounting media. The specimen was rendered transparent by absorption and transfer of the mounting media into the filter. The ten specimens were prepared and counted in our laboratory within one day after preparation. The technique used was that described in the Appendix.

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The specimens were then sent by mail to each of six other participants for their count of these samples, A repeat count was made in our laboratory at the completion of the round-robin test. Air filtration data was provided and each laboratory was requested to calculate the respective number of fibers greater than 5 μm per cubic centimeter of air. Details on the microscope and optics used was also requested. Elapsed time between measurements was obtained. The results were statistically analyzed at the Esso Research and Engineering Company.

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STATISTICAL EVALUATION

The fiber concentrations obtained by the seven laboratories on the ten samples are shown in Table 3 in terms of the number of the fibers greater than 5 μm per cubic centimeter of air. As shown in the bottom of Table 3, the standard deviation appears to be a function of the mean value. A direct analysis of the data would, therefore, be overwhelmed by the greater magnitude of variation for the higher concentrations. To minimize this effect, a logarithmic transformation of the data was made with the following adjustments: 1) data from sample 2 were eliminated (this was the blank sample); and 2) the data for sample number 3 from laboratory number 5 was changed from zero to the equivalent concentration for one counted fiber (viz. 0.48 fibers/cc). Zero values are not permissible if a log transform is used and these adjustments were considered reasonable. Table 4 shows the transformed data (natural logarithms were used).

TABLE 3

Overall Results

	Fibers > 5 μm/cc air									
	Sample Number									
	1	2	3	4	5	6	7	8	9	10
Laboratory 1	53.7	.48	.95	33.7	.95	43.2	8.6	21.9	28.5	50.7
Number 2	29.3	.07	.66	19.3	.84	17.6	3.5	8.2	18.4	25.8
3	28.4	2.54	3.0	17.9	3.0	33.3	5.1	13.5	19.2	26.4
4	28.0	.00	2.1	19.0	1.6	39.0	4.7	9.5	19.0	28.0
5	43.2	.00	.00	23.1	.95	22.8	2.4	20.4	18.1	22.3
6	45.9	.39	.39	18.7	.20	26.5	1.6	11.0	23.6	26.5
7	42.4	.19	.38	33.0	.66	54.1	4.6	9.6	29.7	53.1
Mean	38.7	.55	1.1	23.5	1.2	33.8	4.4	13.4	22.4	33.3
Standard Deviation	10.2	.90	1.1	6.9	.9	12.7	2.7	5.5	5.0	12.9

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TABLE 4

Log Transformed Data

		Sample Number								
		1	3	4	5	6	7	8	9	10
Laboratory Number	1	3.98341	-.05129	3.51750	-.05129	3.76584	2.15176	3.08649	3.34990	3.92593
	2	3.37759	-.41552	2.96011	-.17435	2.86790	1.25276	2.10413	2.91235	3.25037
	3	3.34639	1.09861	2.88480	1.09861	3.50556	1.62924	2.60269	2.95491	3.27336
	4	3.33220	.74194	2.94444	.47000	3.66356	1.54756	2.25129	2.94444	3.33220
	5	3.76584	-.73397*	3.13983	-.05129	3.12676	.87547	3.01553	2.89591	3.10459
	6	3.82647	-.94161	2.92852	-1.60944	3.27714	.47000	2.39790	3.16125	3.27714
	7	3.74715	-.96768	3.49651	-.41552	3.99083	1.52606	2.26176	3.39115	3.97218
Mean		3.62558	-.18135	3.12453	-.10475	3.45680	1.35041	2.53140	3.08713	3.44797
Standard Deviation		.26720	.82301	.27337	.83163	.39026	.54770	.38711	.21289	.34959

* ln 0.48

In Table 4, the standard deviation is now less affected by the value of the mean so that the analysis of variance can properly include all the data tabulated.

Table 5 shows the analysis of variance (2-way classification) for the data in Table 4. Note that the total degrees of freedom were diminished by one to account for the substitution of 0.48 fibers/cc in place of 0.00 for sample 3, laboratory 5. The conclusions that can be drawn from Table 5 are as follows:

1. Different laboratories give significantly different results on the same sample.

2. Gross differences in sample concentration are observed with high probability.

3. A 95% confidence interval for fiber concentrations can be derived from the error mean square (.20655) as follows:

$$1.96\ \sqrt{.20655} = .89078$$

$$e^{.89078} = 2.437$$

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The 95% confidence interval for results is, therefore,

$$\text{Lower Limit} = \frac{X}{2.437}$$

$$\text{Upper Limit} = 2.437\ X$$

where X = fibers > 5 um/cc.

For example, a fiber concentration of 5 fibers > 5 um/cc measured in one laboratory could be reported (95% of the time) as 2.1 to 12.2 fibers > 5 µm/cc in other laboratories; and a fiber count of 2 fibers > 5 µm/cc could be reported as 0.8 to 4.8 fibers > 5 um/cc. To assure that the ceiling value of 10 fibers > 5 µm/cc is not exceeded, these data also suggest that no value should exceed 10/2.437 or 4.1 fibers > 5 um/cc.

TABLE 5

Analysis of Variance
On Log Transformed Data

Without Sample 2 and Adjustment of Sample 3 (Laboratory 5)

Source of Variation	Sum of Squares	Degrees of Freedom	Mean Square	F
Laboratories	3.93353	6	.65559	3.17*
Samples	130.29369	8	16.28671	78.8**
Error	9.70773	47	.20655	
Total	143.93495	61	--	

*Significant at > 95% Probability.
**Significant at > 99% Probability.

In an attempt to separate the random error within the laboratory, repeat counts were made in Laboratory 1 on the ten samples after 149 days had elapsed. The replicate results taken by the same operator in Laboratory 1 are shown in Table 6. A non-parametric sign-rank test of these results showed that the amount of time between analyses did not influence results. In addition, a parametric analysis of these paired results was done after a logarithmic transformation of the data and rejection of sample number 2.

TABLE 6

Repeat Results in Laboratory 1

Sample	Fibers > 5 μm/cc On Day 1	On Day 149
1	53.8	49.8
2	.48	.48
3	.95	1.9
4	33.7	31.7
5	.95	2.9
6	43.2	50.3
7	8.6	5.0
8	21.9	20.9
9	28.5	35.4
10	50.7	37.6

The analysis of variance is shown in Table 7. As anticipated the effect of time is seen to be nonsignificant. (The sample differences are significant, as expected.) The error term is related to one operator in one laboratory and, therefore, does not constitute a uniform result for all laboratories. However, using the same interpretive method described previously, the results in this laboratory, at least, are considered to fall within a 95% confidence interval when results are multiplied and divided by 2.0. The geometric methodology for determining acceptable limits of fiber concentrations seems reasonable, although disheartening, because of its magnitude.

TABLE 7

Analysis of Variance on Replicate Samples

Source of Variation	Sum of Squares	Degrees of Freedom	Mean Square	F
Time	.07371	1	.07371	.58
Samples	33.21484	8	4.15186	32.50**
Error	1.02202	8	.12775	
Total	34.31057	17		

**Significant at > 99% Probability

The blank filter reported as sample number 2 (if one excludes the results from laboratory number 3) would be, on average, about 0.2 fibers per cc. It follows, then, that the sealed filters from Millipore do not contribute significantly to the evaluation of asbestos fiber concentrations in air.

Conclusions

Microscopic examination of asbestos fibers > 5 μm at 400-450X magnifications with phase contrast illumination was collaboratively tested in 7 industrial laboratories using mounted specimens of 9 asbestos samples collected on membrane filter paper and one blank filter.

An analysis of variance shows that different laboratories can be expected to get significantly different results on the same sample.

A geometric methodology for determining confidence limits of fiber concentration suggest that 95% of the time a result in one laboratory multiplied and divided by 2.437 would be the expected range of results reported on the same sample in other laboratories. Even within one laboratory, although this factor was not extensively tested, the 95% confidence interval would be expected to fall within the measurement result multiplied and divided by 2.0.

The blank filter (Millipore Type AA three-piece filter assembly) contributes little to the overall count, averaging about 0.2 fibers greater than 5 μm per cubic centimeter of air.

CHEV
BB 011116

REFERENCES

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2. Weaver, N., M.D. Personal Communication. May 14, 1973.

3. Occupational Safety and Health Administration. Federal Register. 37(202):22102-22356. CFR Title 29-Chapter XVII - Part 1910.93a Asbestos. October 18, 1972.

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5. National Institute for Occupational Safety and Health. Criteria for a Recommended Standard -- Occupational Exposure to Asbestos. HSM 72-10267. 1972.

6. Lynch, J. R. Personal Communication. October 31, 1972.

7. Scheinbaum, M. Asbestos Measurement. Presentation at the American Industrial Hygiene Association Quad Section Meeting. New York, N. Y. December 1, 1972.

RSB:ecb
5/15/73

CHEV
BB 011117

ACKNOWLEDGMENT

The assistance and cooperation of the participating laboratories is acknowledged with sincere appreciation for the voluntary efforts of the staff members who represented the following organizations.

Name	Organization
R. G. Confer	Esso Research and Engineering Company Linden, New Jersey
R. M. Curtis	Shell Development Company Houston, Texas
E. K. Daniels	Mobil Oil Corporation New York, New York
E. N. Davis	Atlantic-Richfield Company Harvey, Illinois
R. L. Stoffer	Standard Oil Company (Indiana) Naperville, Illinois
F. M. Toca	Gulf Oil Corporation Pittsburgh, Pennsylvania
R. W. Veit	Phillips Petroleum Company Bartlesville, Oklahoma

Thanks are also due to Messrs. H. T. Oakley, Esso Research and Engineering Company, and R. Spirtas, University of North Carolina for their assistance in evaluating the data.

CHEV
BB 011118

APPENDIX

Sampling and Evaluation Procedure[5]

Principles of Sampling

A dust sampling procedure must be designed so that samples of actual dust concentrations are collected accurately and consistently. The results of the analysis of these samples will reflect, realistically, the concentrations of dust at the place and time of sampling.

In order to collect a sample representative of airborne dust, which is likely to enter the subject's respiratory system, it is necessary to position a collection apparatus near the nose and mouth of the subject or in his "breathing zone".

The concentration of dust in the air to which a worker is exposed will vary, depending upon the nature of the operation and upon the type of work performed by the operator and the position of the operator relative to the source of the dust. The amount of dust inhaled by a worker can vary daily, seasonally, and with the weather. In order to obtain representative samples of workers' exposures, it is necessary to collect samples under varying conditions of weather, on different days, and at different times during a shift.

The percentage of working time spent on different tasks will affect the concentration of dust the worker inhales since the different tasks usually result in exposure to different concentrations. The percentage can be determined from work schedules and by observation of work routines.

The daily average weighted exposure can be determined by using the following formula:

$$\frac{(\text{Hours} \times \text{conc. task A}) + (\text{Hours} \times \text{conc. task B}) + \text{etc.}}{8 \text{ Hours (or actual hours worked)}}$$

The concentration of any air contaminant resulting from an industrial operation also varies with time. Therefore, a longer sampling time will better approximate the actual average.

With the following recommended sampling procedure, it is possible to collect samples at the workers' breathing zones for periods from 4 to 8 hours, thus permitting the evaluation of average exposures for a half or full 8-hour shift--a desirable and recommended procedure. Furthermore, dust exposures of a more normal work pattern result from the use of personal samplers. In evaluating daily exposures, samples should be collected as near as possible to workers' breathing zones.

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Collecting Sample

The sample should be collected on a 37-millimeter Millipore Type AA filter mounted in an open-face filter holder. The holder should be fastened to the worker's lapel and air drawn through the filter by means of a battery-powered personal sampler pump. The filters are contained in plastic filter holders and are supported on pads which also aid in controlling the distribution of air through the filter. To yield a more uniform sample deposit, the filter-holder face-caps should be removed. Sampling flow rates from 1.0 liter per minute (lpm) up to the maximum flow rate of the personal sampler pump (usually not over 2.5 lpm) and sampling time from 15 minutes to eight hours are acceptable provided the following restraints are considered:

(a) In order to obtain an accurate estimate of the number of fibers the statistical error resulting from the random distribution of the fibers must be kept to an acceptably low level. Since fiber counts follow a Poisson distribution, a count of 100 fibers in a sample would have a standard deviation of $\sqrt{100}$ or 10 fibers or $\pm$ 10%. Thus the 95% confidence limits would be approximately 2 standard deviations or $\pm$ 20%. Since the 37 mm filter has an effective collecting area of 855 mm^2 and if the projected field area of the Porton reticle is 0.005 mm^2, then each field represents 1/171000 of the sample. Based on this ratio the following number of fields must be counted to measure the various limits in various sampling times:

Sampling Time Minutes	Flow Rate lpm	Number of Fields for 100 Fibers 0.2 fibers/ml	2.0 fibers/ml	10 fibers/ml
10	2	4350	435	91
15	2	2860	286	58
30	2	1430	143	29
90	1	1000	100	20
90	2	500	50	10
240	1	260	26	7
240	2	180	18	4
480	1	180	18	4

(b) Do not count a field containing over 20 fibers because in addition to the fibers being counted, there are also present a number of grains, which interfere with the accuracy of the count.

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Based on these restraints, i.e., number of fields to be counted and maximum number of fibers per field, acceptable sampling parameters for the various limits are underlined in the above table.

The following conclusions may be drawn from this analysis:

(1). The short-term limit should be for a period of at least 15 minutes and preferably 30 minutes.

(2). The 2.0 fiber/cc limit may be evaluated over periods of from 90 to 480 minutes.

As many fields as required to yield at least 100 fibers should be counted. In general the minimum number of fields should be 20 and the maximum 100.

Mounting Sample

The mounting medium used in this method is prepared by dissolving 0.05 g of membrane filter per ml of 1:1 solution of dimethyl phthalate and diethyl oxalate. The index of refraction of the medium thus prepared is ND = 1.47.

To prepare a sample for microscopic examination, a drop of the mounting medium is placed on a freshly cleaned, standard (25 mm x 75 mm), microscopic slide. A wedge-shaped piece with arc length of about 1 cm is excised from the filter with a scalpel and forceps and placed dust-side-up on the drop of mounting solution. A No. 1-1/2 coverslip, carefully cleaned with lens tissue, is placed over the filter wedge. Slight pressure on the coverslip achieves contact between it and the mounting medium. The sample may be examined as soon as the mount is transparent. The optical homogeneity of the resulting mount is nearly perfect, with only a slight background granularity under phase contrast, which disappears within one day. The sample should be counted within two days after mounting.

Evaluation

The filter samples mounted in the manner previously described are evaluated in terms of the concentration of asbestos fibers greater than 5 μm in length. A microscope equipped with phase-contrast optics and a 4-mm "high-dry" achromatic objective is suitable for this determination. 10X eyepices, one of which contains a Porton or other suitable reticle at the level of the field-limiting diaphragm, should be used. The left half of the Porton reticle field serves to define the counting area of the field. Twenty fields located at random on the sample are counted and total asbestos fibers longer than 5 μm are recorded. Any particle having an aspect ratio of three or greater is considered a fiber.

The following formulae are used to determine the number of fibers/ml:

$$(1)\quad \frac{\text{Filter area } (mm^2)}{\text{Field area } (mm^2)} = K$$

$$(2)\quad \frac{\text{Average net count} \times K}{\text{Air volume sampled (ml)}} = \text{fibers/ml}$$

For example, assume the following: area of the filter used was 855 mm^2, counting area of one field under the Porton reticle was 0.005 mm^2; average net count per field of 20 fields was 10 fibers; and sample was collected at 2 liters per minute for 90 minutes: Then:

$$\frac{855\ mm^2}{0.005\ mm^2} = 171{,}000\ (K)$$

$$\frac{10 \text{ fibers} \times 171{,}000}{2{,}000 \text{ ml/min} \times 90 \text{ min}} = 9.5 \text{ fibers/ml}$$

Calibration of Personal Sampler

The accuracy of an analysis can be no greater than the accuracy of the volume of air which is measured. Therefore, the accurate calibration of a sampling device is essential to the correct interpretation of an instrument's indication. The frequency of calibration is somewhat dependent on the use, care, and handling to which the pump is subjected. Pumps should be calibrated if they have been subjected to misuse or if they have just been repaired or received from a manufacturer. If hard usage is given the instrument, more frequent calibration may be necessary.

Ordinarily, pumps should be calibrated in the laboratory both before they are used in the field and after they have been used to collect a large number of field samples. The accuracy of calibration is dependent on the type of instrument used as a reference. The choice of calibration instrument will depend largely upon where the calibration is to be performed. For laboratory testing, a 1-liter burette or wet-test meter should be used. In the field, a rotameter is the most convenient instrument used. The actual set-up will be the same for all of these instruments. The calibration instrument will be connected in sequence to the filter unit which will be followed by the personal sampler pump. In this way, the calibration instrument will be at atmospheric pressure. Connections between units can be made using the same type of tubing used in the personal sampling unit. Each pump must be calibrated separately for each type of filter used, if, for example, it has been decided to use a filter with a different pore size. The burette should be set up so that the flow is toward the narrow end of the unit.

Care must be exercised in the assembly procedure to insure adequate seals at the joints and that the length of connecting tubing be kept at a minimum. Calibration should be done under the same conditions of pressure, temperature and density as will be encountered. The rotameter should be used only in the field as a check if the diaphragm or piston pumps are not equipped with pulsation dampeners. The pulsating flow resulting from these type pumps causes the rotameter to give results which are not as accurate as that obtained with a burette or wet-test meter. Calibration can be accomplished with any of the other standard calibrating instruments, such as spirometer, Marriott's bottle, or dry-gas meter. The burette and wet-test meter were selected because of their accuracy, availability, and ease of operation.

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