C3058
JMEA 4614
W IE
JOHNS-MANVILLE CORPORATION
HEALTH, SAFETY & ENVIRONMENTAL DEPT.
LIBRARY

Energy, Mines and Resources Canada / Énergie, Mines et Ressources Canada

CANMET
Canada Centre for Mineral and Energy Technology / Centre canadien de la technologie des minéraux et de l'énergie

THE EFFECT OF ASPECT RATIO ON FIBER COUNTS; A PRELIMINARY STUDY

A. A. WINER
INDUSTRIAL MINERAL LABORATORIES

M. COSSETTE
UNIVERSITY OF SHERBROOKE, QUEBEC

JUNE 1978

For presentation at the New York Academy of Sciences on the occasion of Science Week on "The Scientific Basis for the Public Control of Environmental Health Hazards in Workshop No. 1, June 26, 1978 on the Significance of aspect ratios in regulation of asbestos fiber exposure".

Project No. - 4.3.6.3.01
Wet Processing of Asbestos

MINERALS RESEARCH PROGRAM

MINERAL SCIENCES LABORATORY
REPORT MRP/MSL 78-139(OP)

JMEA 4614
02
03
76
81

THE EFFECT OF ASPECT RATIO ON FIBER COUNTS;
A PRELIMINARY STUDY

by

A. A. Winer* and M. Cossette**

- - -

ABSTRACT

The results of a study of the inter-laboratory precision obtained by the NIOSH counting method are reported. Three different aspect ratios including (a) 3:1, (b) 5:1, and (c) 10:1, were applied as fiber counting criteria. Likewise, maximum and minimum lengths covering the following ranges were also applied as criteria for the choice of particles to be counted: (a) 5 to 10 µm (b) 10 to 50 µm (c) >50 µm (d) >10 µm. Twelve laboratories (6 industry, 3 government and 3 research) counted two samples from each of 6 plants (1 asbestos-cement, 1 brake linings, 2 ore treating mills, 2 textiles).

Results obtained are summarized hereunder:

Counting Criteria		Mean Count	Coefficient of
Aspect Ratio	Min. Length	f	Variation
3:1	5 µm	100	0.43
5:1	5 µm	80	0.38
10:1	5 µm	53	0.54
3:1	10 µm	38	0.49

It appears that a 5:1 aspect ratio is better than 3:1 and 5 µm leads to better precision than 10 µm minimum length. However, a parallel study at one laboratory (ORF) of the same samples at high resolution indicated that correlation was excellent between TEM fibre count vs. visual microscope for the count using fibres >5 µm in length. In addition, most of the non asbestos particles are thereby eliminated. The majority of fibres are classed as >10:1 aspect ratio, a much smaller amount are in the >5:1<10:1 aspect ratio; the number of fibres with aspect ratio less than 5:1 aspect ratio are insignificant.

*Research Scientist, Industrial Minerals Laboratory, Department of Energy, Mines and Resources ** Director, Centre for Technology of the Environment, University of Sherbrooke, Sherbrooke, P.Q., Canada J1K 2R1.

Published by AsbestosClaims.Law
asbestosclaims.law/library/25493/1

INTRODUCTION

The "naturally occurring inorganic fibre task group" in ASTM Committee E34 (Occupational Health and Safety) have been developing an ASTM standard for these fibres which include asbestos. The method of counting fibres, the NIOSH technique, was determined to have a high degree of variance and this was confirmed at a recent workshop of the National Bureau of Standards. The method however is the best available for the purpose at this time. It has a number of advantages; it is relatively simple and can be easily learned. If the precision of the method were to be increased and simultaneously if the "fibres" counted were truly asbestos then the disadvantages of this relatively simple method would be overcome to a large degree.

The NIOSH technique has adopted the criteria of a 3:1 aspect ratio and greater than 5 μm in length for identifying and counting of fibres. Those familiar with grinding techniques had shown, that with some minerals, particulates of 3:1 aspect ratio could be created. As well, non asbestos fibrous materials were known to occur in association with asbestos materials, e.g., niomalite and brucite with chrysotile.

These suggestions are supported by electron microscopic studies by the U.S. Bureau of Mines, which showed that non-asbestiform varieties such as anthophyllite, tremolite and hornblende produced cleavage fragments of which 95 per cent had an aspect ratio smaller than 10:1 and 70 per cent smaller than 3:1. On the other hand, the majority of commercial milled chrysotile asbestos had an

aspect ratio greater than 10:1. This would suggest than an increase of the aspect ratio from 3:1, to say, 10:1 could significantly improve the discrimination between true fibres and non-fibrous particulates for counting purposes.

In order to overcome these problems the thought of increasing the aspect ratio was suggested. This would allow, it was hoped, greater discrimination between true fibres and non fibrous particulates thus increasing precision as well as being more certain that only asbestos fibres were counted.

A round robin series was begun to test this hypothesis. Objectives of the initial round robin were: to indicate problem areas and also to point the way to a more refined round robin series that would be statistically designed and allow for an analysis of variance. A parallel study of fibre count and identification on the same samples, was included in this initial series.

Twelve laboratories consisting of 3 government, 3 research and 6 industrial laboratories, participated in this initial study.

COUNTING CRITERIA AND EXPERIMENTAL METHOD

The technique for counting generally followed the NIOSH method using a membrane filter. Two samples from each of six plants were obtained on membrane filters. The type of plants are shown in Table 1. The two filters were taken near different machines.

TABLE 1

Origin of Filter Samples by Industry

1.	Asbestos-Cement	7.	Mining (mill)
2.	Asbestos-Cement	8.	Mining (mill)
3.	Brake linings (carding)	9.	Textile (twister)
4.	Brake linings (press 0416)	10.	Textile (twister)
5.	Mining (mill)	11.	Textile (blender)
6.	Mining (mill)	12.	Textile (blender)

Each filter was complete or had had one or two 1/8th wedges removed from it. One 1/8th wedge from each of the twelve membrane filters was sent to each of 5 participating laboratories and the sixth wedge was counted by all six mining laboratories. Each laboratory mounted and counted the sample, using its standard technique and criteria, except in so far as it was asked to count "respirable" fibres, that is less than 3 µm diameter, and to allocate them to the seven regions given on Table 2, (defined by both length and aspect ratio). Each laboratory was asked to count each sample with two counters. The laboratory microscopes are described in Table 3.

TABLE 2

Group Classifications

Length \ Aspect ratio	1:3 1:5	1:5 1:10	>1:10	Total
5 - 10 µm	1	2	3	8
10 - 50 µm	4	5	6	9
> - 50 µm			7	7
Total	10	11	12	13

TABLE 3

Microscope Type and Related Data Used in Counting

Counter Code [1]	Microscope	Magnification	Graticule size type	Graticule size µm²	Diameters Defined
I A1	Bausch & Lomb	400	Porton	3000	
B2, 3	"	400	"	3000	
C4, 5	"	400	"	3000	
D6, 7	"	430	"	2500	
E8	"	400	"	3000	
F9	"	430	"	2500	
G G10, 11, 12	Petro Zeiss	500	Circular	4500	
H13, 14	Olympus		Porton	3300	
I15, 16, 17	Dynoscope 70	400X	Porton	4000	
R J18		400X		5000	as seen
19	Zeiss	500X	Patterson	3400	as stipulated
K20	Leitz	500X	Porton	3300	

Footnotes:

1 Industry, Government, Research - Labs A to K - Counters 1-20.

Published by AsbestosClaims.Law
asbestosclaims.law/library/25493/3

One group of samples similar to those used for the optical microscope count, was also sent to the Ontario Research Foundation. The particulates were counted and identified by electron microscope (TEM) and compared to the count obtained by phase contrast optical measurement.

RESULTS AND DISCUSSION

A. Fibre Count by Phase Contrast Microscopy

The results were received with fibres classified into the first 7 groups (Table 4) as well as a field count. Also shown is a summary of the results on all 12 slides as means and standard deviations expressed as per cent of total count. The variations (as standard deviations) on fibre/unit area is about 50 per cent greater than on fibres as parts per total; this is presumably due to variations in fibre density and/or visual acuity of counters.

Effect of Length

The analysis of fibres into groups by length is shown in Table 5.

TABLE 4

Summary of Results

mean as % of total count from analysis of fibre/sq mm.

Length \ Aspect ratio	3:1 5:1	5:1 10:1	> 10:1	Total
5-10 µm	13.8	18.7	24.1	56.6
10-50 µm	2.9	7.9	26.6	37.4
>50 µm			5.9	5.9
Totals	16.7	26.6	56.7	100.0
		83.3		

standard deviation as % of total count from analysis of fibres/sq mm.

Length \ Aspect ratio	3:1 5:1	5:1 10:1	> 10:1	Total
5-10 µm	14.4	11.3	19.2	24.6
10-50 µm	7.7	9.8	19.2	18.3
>50 µm			6.0	6.0
Totals	20.8	16.5	34.0	34.3
		33.7		

Published by AsbestosClaims.Law
asbestosclaims.law/library/25493/4

TABLE 5

Analysis of Length

	Fibres by % of Total Length		
	5-10 µm	10-50 µm	>50 µm
Analysis by proportion of total			
Mean Overall	61.6	34.0	4.4
Standard Deviation	10.8	10.7	3.3
S.D./Mean	0.17	0.31	0.75
Analysis by Fibres/Unit Area			
Mean Overall	56.6	37.4	5.9
Standard Deviation	24.6	18.3	6.0
S.D/Mean	0.47	0.49	1.0
Range of Slide Means	28-77	21-51	1-12

Effect on Aspect Ratio

The analysis of fibres into groups by aspect ratio is shown in Table 6.

TABLE 6

Analysis of Aspect Ratios

	Fibres by % of Total Aspect Ratio				
	3:1 5:1	5:1 10:1	> 10:1	> 5:1	Total
Analysis by Proportion of Total					
Mean Overall	18.2	27.4	54.4	81.8	100
Standard Deviation	16.5	11.2	22.1	16.5	100
S.D/Mean	0.91	0.41	0.41	.20	0
Analysis by Fibres/Unit Area					
Mean Overall	16.7	26.6	56.7	83.3	100
Standard Deviation	20.8	16.5	34.0	33.7	34.3
S.D./Mean	1.25	0.62	0.60	.40	.34
Range of Slide Means	9-37	21-34	38-68	63-91	

It can be seen that the variability of the aspect ratio groups is much greater than the length groups (Table 5).

The interpretation of the results are discussed for each length region below:

(a) Long fibres: >50 μm

All fibres less than 3 μm diameter and longer than 50 μm had an aspect ratio greater than 10:1 so that there was only one group, No. 7, in this study. On average 1 to 2 per cent of the fibres were longer than 50 μm on the mining and asbestos cement samples and up to 12 per cent on the textile mill slides. Nearly all the single fibres seen on the slides had a diameter of less than 3 μm, although some of the bundles had apparent diameters greater than this.

(b) Medium length fibres: 10-50 μm

The fibres in this region formed 20 to 30 per cent of the total number for the mining and asbestos cement slides and 40 to 50 per cent for the brake linings and textile slides. Overall the average was 34 per cent.

The counters disagreed considerably on the allocation of fibres across the three aspect ratio regions.

(c) Short fibres: 5-10 μm

The fibres in this region formed 70 to 76 per cent of the total number for the mining and asbestos cement slides and 28 to 50 per cent for the brake linings and textile slides. The overall mean was 62 per cent with a standard deviation of 11 per cent.

Some difficulties were encountered by the counters. These comments are important in ascertaining that the new major study avoid some of the ambiguities and problems encountered in this preliminary study. The comments are listed below:

a) Assigning aspect ratios for fibres 500 10 μm in length where the fibre diameter is small.

b) Some of the slides were too dense (i.e., too many fibres/unit area) and some too light.

c) Many of the particulates in 3:1 and 5:1 aspect ratio groups were not chrysotile based on their prismatic appearance.

It was found to be much more difficult to judge the aspect ratio of a fibre than its length. The major problem in judging length was the failure to adjust the dimensions of the marks on the graticule to the exact length required. This can lead to a bias dependent on both counter and microscope. The difficulty of judging and the effect of the aspect ratio appears to depend on the length range.

IDENFITICATION AND COUNTING BY TRANSMISSION ELECTRON MICROSCOPY (TEM)

Problems were encountered with the slides from which the solvent had evaporated. This problem was overcome.

Up to 10 grid squares were counted, if required, to locate a total of 100 fibres. Chrysotile was identified by morphology (tubular structure) and occassional checks were made by selected area electron diffraction and energy dispersive X-ray analysis of the fibres. Other mineral fibres greater than 3:1 aspect ratio were also counted. Fibres longer than 5 μm were counted and tabulated separately.

RESULTS AND DISCUSSION

A summary of the optical fibre count data are shown in Table 7 and a summary of the relevant fibre counts and identification are shown in Table 8.

Practically all the fibres (Table 8) were chrysotile and therefore, the other fibres will not be considered further in this discussion.

It is interesting to note, that in the higher size ranges, large variations occurred between the different samples.

The larger fibres, >5 µm, which had been counted separately produced the results shown in Table 9. A comparison of the fibre count by optical microscope vs. that by TEM is graphically shown in Figure 1. A relationship definitely appears to exist between the two methods. The correlation coefficient of 0.978 is highly significant.

No such relationship exists between the two methods for fibres less than 5 µm in length, Figure 2. This is partially attributed to the origin of the samples, some fibres being more dispersed than others because of their industrial origin.

The vast majority of fibres counted by TEM have an aspect ratio exceeding 10:1. The results of fibre count analysis for two samples are shown in Tables 10 and 11, 2K326 and CCM16-02 respectively. Each table contains a total count of approximately 100 fibres. In Table 11, there are about 55 per cent fibres greater than 10:1 aspect ratio, 41 per cent between 5:1 and 10:1 aspect ratio and 4 per cent less than 5:1 aspect ratio. In

TABLE 7

Summary of Optical Fibre Count Data

Sample	Fibres Counted	Fields Scanned	Area Scanned, mm²	Fibre Density, Fibres/mm²
Asbest 1	208	155	0.513	405
Asbest 2	45	350	1.159	39
Lake JCP	225	350	1.159	194
Lake 2FF	194	350	1.159	167
2K258 (T1)	204	26	0.086	2372
2K215 (T2)	202	52	0.172	1174
2K326 (T2)	214	40	0.132	1621
CCM 16.0.2	200	155	0.513	390
CCM 16.0.3	203	69	0.228	890
Atlas 1	207	193	0.639	324
Atlas 2	119	350	1.159	103

Published by AsbestosClaims.Law
asbestosclaims.law/library/25493/7

TABLE 8

Summary of Routine Fibre Counts for All Fibres

Sample	** Fibre Concentration (Fibres/ml) Mean	95% Confidence Interval	Estimated Mass Concentration (Nanograms/m^3)	Concentration Equivalent to 1 fibre detected (Fibres/ml)	Fibre Type *
Asbest 1	230 <1.4	160 - 300	3800	1.37 1.37	C OF
Asbest 2	150 1.4	110 - 190	4700	1.40 1.40	C OF
Lake 3CP	320 9.4	240 - 390	45000	1.57 1.57	C OF
Lake 2FF	200 1.3	130 - 270	2700	1.29 1.29	C OF
2K258 (T1)	937 <7.4	700 - 1100	22000	7.38 7.38	C OF
2K215 (T2)	860 7.7	510 - 1200	36000	7.72 7.72	C OF
2K326 (T2)	870 17	500 - 1200	20000	8.28 8.28	C OF
CCM 16.0.2	750 <7.3	600 - 840	26000	7.25 7.25	C OF
CCM 16.0.3	840 <7.7	380 - 1300	16000	7.63 7.63	C OF
Atlas 1	150 <1.3	100 - 190	16000	1.28 1.28	C OF
Atlas 2	96 <0.98	57 - 120	1000	0.98 0.98	C OF

** Concentration assuming 1 m^3 sampling volume and 1/6th filter supplied.

* C = Chrysotile, OF = Other Fibres (Mineral Particulate >3:1 aspect ratio).

TABLE 9

Comparative Summary of Fibre Counts

Sample	Fibre Count in Fibres/mm^2 on Original Membrane Filter: Optical >5 um	TEM >5 um *	TEM all Fibres	TEM all Fibres, 95% Confidence
Asbest 1	405	2.4×10^4	3.8×10^5	$(2.70 - 5.00) \times 10^5$
Asbest 2	39	9.7×10^3	2.5×10^5	$(1.80 - 3.20) \times 10^5$
Lake 3CP	194	6.9×10^3	5.3×10^5	$(4.00 - 6.60) \times 10^5$
Lake 2FF	167	9.5×10^3	3.3×10^5	$(2.20 - 4.50) \times 10^5$
2K258 (T1)	2372	2.0×10^5	1.6×10^6	$(1.33 - 1.88) \times 10^5$
2K215 (T2)	1174	7.9×10^4	1.4×10^6	$(0.85 - 2.00) \times 10^6$
2K326 (T2)	1621	9.4×10^4	1.5×10^6	$(0.86 - 2.06) \times 10^5$
CCM 16.0.2	390	1.8×10^5	1.3×10^6	$(1.14 - 1.45) \times 10^6$
CCM 16.0.3	890	3.5×10^5	1.4×10^6	$(0.63 - 2.20) \times 10^5$
Atlas 1	324	3.8×10^3	2.5×10^5	$(1.70 - 3.20) \times 10^5$
Atlas 2	103	2.3×10^3	1.6×10^5	$(0.95 - 2.20) \times 10^5$

* Large area of grid separately scanned for fibres >5 um only, in order to increas statistical validity.

Published by AsbestosClaims.Law
asbestosclaims.law/library/25493/8

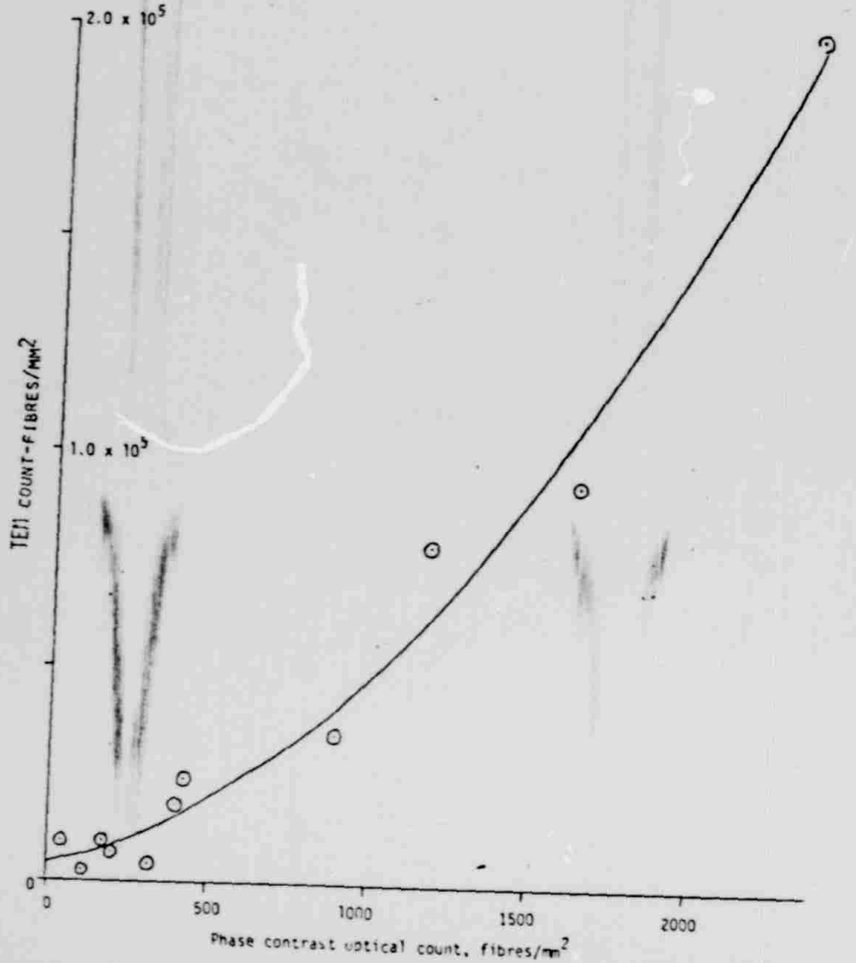


FIGURE 1. RELATIONSHIP BETWEEN TEM COUNT AND PHASE CONTRAST OPTICAL COUNT FOR FIBRES LONGER THAN 5/ M.

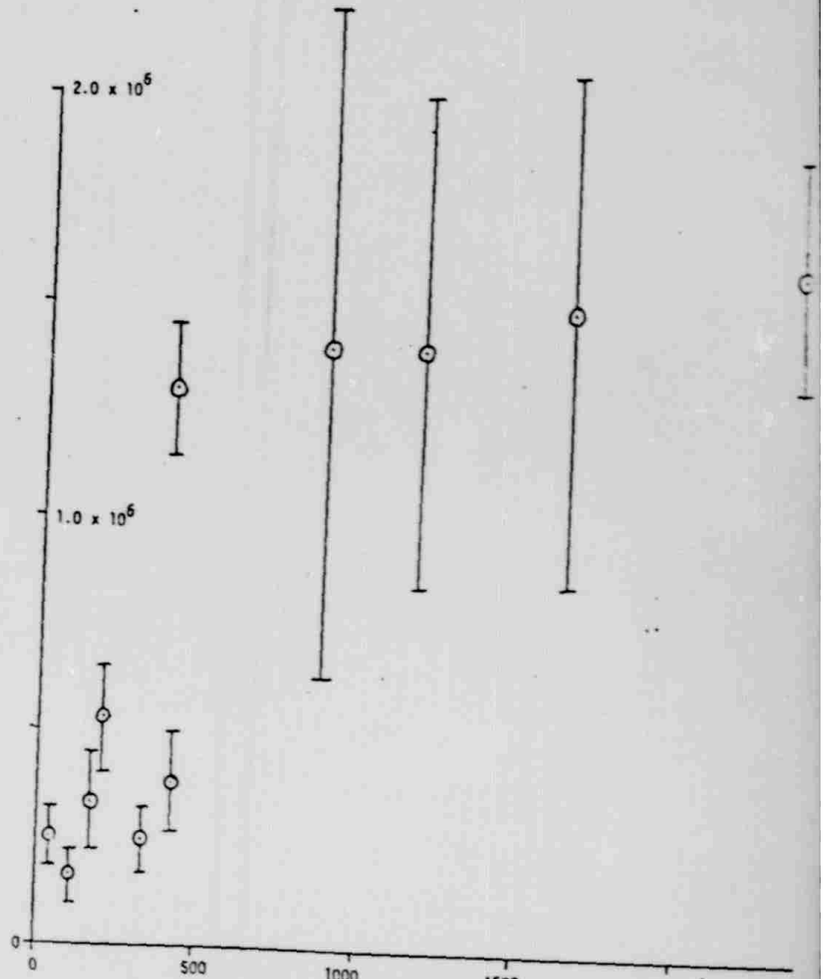


FIGURE 2 RELATIONSHIP BETWEEN TEM COUNT OF ALL FIBRE LENGTHS

Table 10, fibres greater than 10:1 aspect ratio amount to about 76 per cent of the total fibres, between 10:1 and 5:1 aspect ratio the percentage fibres amounts to about 24 per cent and there is less than one per cent fibres with an aspect ratio of less than 5:1.

TABLE 10

Asbestos Fibre Count Analysis:
Chrysotile Raw Sizing Data

SAMPLE: 2K326 (T2)

Length um	Width um	Aspect Ratio	Length um	Width um	Aspect Ratio	Length um	Width um	Aspect Ratio
0.82	0.045	18.0	1.41	0.136	10.3	0.59	0.091	6.5
0.27	0.045	6.0	0.36	0.045	8.0	1.18	0.091	13.0
2.09	0.136	15.3	2.68	0.136	19.7	0.73	0.045	16.0
2.50	0.045	56.0	0.68	0.045	15.0	0.50	0.045	11.0
0.41	0.045	9.0	0.55	0.045	12.0	1.14	0.091	12.5
1.18	0.136	8.7	0.95	0.091	10.5	0.54	0.045	12.0
3.27	0.045	72.0	1.36	0.273	5.0	0.95	0.045	21.0
1.41	0.136	10.3	0.73	0.045	16.0	0.45	0.091	5.0
1.86	0.091	20.5	2.86	0.045	63.0	0.27	0.045	6.0
1.45	0.136	10.7	0.45	0.045	10.0	0.32	0.045	7.0
2.77	0.136	20.3	0.27	0.045	6.0	0.73	0.045	16.0
1.18	0.045	26.0	1.45	0.045	41.0	1.59	0.183	8.7
2.05	0.136	15.0	3.27	0.091	36.0	1.18	0.045	26.0
2.09	0.136	15.3	1.32	0.045	29.0	1.45	0.045	32.0
0.59	0.045	13.0	5.59	0.045	123.0	6.23	0.045	137.0
5.73	0.091	63.0	6.23	0.136	46.7	4.64	0.091	51.0
1.45	0.045	32.0	1.68	0.045	36.0	2.95	0.045	66.0
0.73	0.091	8.0	0.73	0.045	16.0	2.05	0.091	22.5
20.59	0.045	452.0	0.95	0.045	12.5	0.27	0.045	6.0
0.95	0.045	21.0	0.36	0.045	8.0	0.32	0.045	7.0
1.64	0.091	14.0	7.26	0.045	162.0	2.91	0.045	64.0
0.73	0.045	16.0	0.91	0.045	20.0	18.18	0.045	400.0
0.32	0.045	7.0	1.00	0.045	22.0	0.73	0.045	16.0
0.36	0.045	8.0	0.73	0.045	16.0	1.14	0.136	8.3
4.14	0.045	91.0	2.09	0.045	46.0	0.64	0.091	7.0
1.18	0.142	8.3	1.36	0.091	15.0	0.32	0.045	7.0
0.73	0.045	16.0	7.45	0.045	164.0	0.59	0.091	6.5
1.18	0.045	26.0	5.64	0.045	124.0	1.41	0.136	10.3
0.64	0.045	14.0	0.32	0.045	7.0	0.73	0.045	16.0
0.55	0.045	12.0	0.36	0.045	8.0	1.00	0.091	11.0
0.36	0.045	8.0	1.23	0.045	27.0	1.50	0.045	33.0
1.00	0.091	11.0	0.82	0.045	18.0	1.09	0.045	24.0
2.09	0.045	46.0	1.09	0.045	35.0	0.91	0.045	20.0
0.59	0.045	13.0	0.27	0.045	6.0	4.82	0.091	53.0
0.59	0.045		0.95	0.136	7.0	1.37	0.045	30.0

TABLE 11

Asbestos Fibre Count Analysis:
Chrysotile Raw Sizing Data

SAMPLE: CCM 16.0.2

Length um	Width um	Aspect Ratio	Length um	Width um	Aspect Ratio	Length um	Width um	Aspect Ratio
1.68	0.045	37.0	0.95	0.091	10.5	1.41	0.091	15.5
2.73	0.045	60.0	0.91	0.045	20.0	0.27	0.045	6.0
0.23	0.045	5.0	0.59	0.045	13.0	1.36	0.091	15.0
0.41	0.091	4.5	0.59	0.091	6.5	0.41	0.091	4.5
5.50	0.045	121.0	0.55	0.045	12.0	0.23	0.045	5.0
1.55	0.045	34.0	0.36	0.045	8.0	0.50	0.045	11.0
0.59	0.091	6.5	0.73	0.091	8.0	1.41	0.091	15.5
1.05	0.182	5.7	0.41	0.045	9.0	3.00	0.273	11.0
0.41	0.045	9.0	2.59	0.045	57.0	3.95	0.045	87.0
0.59	0.045	13.0	0.35	0.091	9.5	0.35	0.045	8.0
1.05	0.136	7.7	1.09	0.227	4.8	0.73	0.091	8.0
0.32	0.045	7.0	0.32	0.045	7.0	0.54	0.182	3.5
0.86	0.091	9.5	0.41	0.091	4.5	0.50	0.045	11.0
0.73	0.045	16.0	0.27	0.045	6.0	1.23	0.045	27.0
0.23	0.045	5.0	0.64	0.045	14.0	0.86	0.045	19.0
0.64	0.045	14.0	8.02	0.045	177.0	0.41	0.045	9.0
0.50	0.045	11.0	0.36	0.045	8.0	0.73	0.091	8.0
1.14	0.091	12.5	1.68	0.045	37.0	0.73	0.091	8.0
0.36	0.045	8.0	3.09	0.273	11.3	0.86	0.091	9.5
0.50	0.045	11.0	0.95	0.091	10.5	0.64	0.045	14.0
0.77	0.045	17.0	0.35	0.045	8.0	0.68	0.091	7.5
0.68	0.045	15.0	0.36	0.045	8.0	2.32	0.045	52.0
0.41	0.045	9.0	1.45	0.091	20.5	1.32	0.045	29.0
3.23	0.136	23.7	0.23	0.045	5.0	1.82	0.045	40.0
0.41	0.045	9.0	0.27	0.045	6.0	0.64	0.045	14.0
0.50	0.045	11.0	0.29	0.091	6.5	0.68	0.091	7.5
0.32	0.045	7.0	1.27	0.045	28.0	0.64	0.045	14.0
2.41	0.045	53.0	1.77	0.136	13.0	1.18	0.091	13.0
0.50	0.045	11.0	0.50	0.045	11.0	1.45	0.182	8.0
0.68	0.091	7.5	2.09	0.136	15.3	1.77	0.045	39.0
0.32	0.045	7.0	1.50	0.091	11.0	0.64	0.045	14.0
0.32	0.045	7.0	3.50	0.409	8.6	0.45	0.045	10.0
0.32	0.045	7.0	0.55	0.045	12.0	2.77	0.182	15.2
1.05	0.091	11.5	1.14	0.045	25.0	1.45	0.136	10.7
0.41	0.045	9.0						