Asb. Fibers

DISCUSSION

Background

The FDA reportedly planned publication of a Good Manufacturing Practices (GMP) regulation which would limit chrysotile and tremolitic asbestos fibers to specified numbers in talc for food, pharmaceutical, and food contact uses.[1] Such a GMP, if published, would severely restrict use of talc in paper, which is classified as a food contact application.

The FDA earlier in the year had asked for comment on a proposed GMP and in this connection suggested a method for determination of respective numbers of chrysotile and tremolitic fibers using a light microscope and two specific refractive index liquids.[2]

Subsequent discussion with FDA officials established that the GMP limits would be 100 fibers of chrysotile and/or 1000 fibers of tremolite per milligram (mg) of talc. Said fibers, based on OSHA criteria, must be at least 5 microns in length and have a 3:1 aspect ratio, i.e., be at least three times as long as they are wide. Based on published reports[1], the GMP might also include limits for percentages of chrysotile and tremolitic fiber content.*

This report is concerned (1) with the adequacy of the FDA-proposed asbestos fiber count method, and (2) with the number of fibers in a broad selection of J-M and competitive talc samples.

FDA Fiber Count Method

The detailed method as proposed by Schulze and Eisenberg is included in Appendix I. It apparently was devised after reviewing the included references, many of which are outdated by later developments in the field.

To a layman the procedure appears to be simple and straightforward, so that results should be easily obtained and reasonably reproduceable.

As a check on the suitability of the method, duplicate sets of the talc samples from various sources as listed in Appendix II, Table I, were prepared and submitted to experienced minerologist-petrographers in two different laboratories. Each was furnished with the FDA fiber count method as shown in Appendix I, and each was asked to count chrysotile and tremolitic fibers in accordance with the verbatum method. Since the method does not so specify, it was requested that fibers to be counted be defined as being more than 5 microns in length and having a 3:1 aspect ratio.

An outside laboratory, Hazen Research, Inc., which has two petrographers, worked with the method as written for two weeks virtually full time and were unable to complete the count on any of the samples. At the end of that time, at a meeting at J-M, they asked to submit a report which would characterize the method written as unworkable, detailing their reasons for so stating. Their report, attached as Appendix III, contains these details.

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* Since the GMP has not been published, it is not known whether fiber numbers or fiber weight would be given primary emphasis or whether both would govern.

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The method was also submitted for comment to W. T. Caneer of the Colorado School of Mines, a minerologist with recognized expertise in the field of talc and fiber identification. His comments are as follows:

> "I do not believe that the method outlined is suitable for determining the presence of asbestos minerals. In fact it would tend to lead to the misidentification of some talc grains as chrysotile. The methods state that one can distinguish chrysotile asbestos from talc particles in a 1.574 refractive index liquid. The reasoning is the beta and gamma refractive indices (talc plate edges) are greater than the 1.574 index oil. This ignores the fact that talc shards with an axial ratio greater than 3 to 1 could be in an orientation such that the alpha index of refraction is being obtained. Since the alpha index is less than 1.574, they would be mistaken for chrysotile.
>
> It is also very difficult to use Beeke lines to determine the relative index of very small or thin minerals. For example, one cannot rely on optical methods to distinguish between amphiboles such as tremolite-actimolite series and hornblende minerals. (Deer Howis & Zussman V-2 P 257).
>
> We do not recommend the use of the method as written."

The second set of samples was submitted to the J-M Research Microscopy Lab where they were examined by Mr. Wolkodoff, the minerologist-petrographer. He speedily reached the identical conclusion with respect to the feasibility of the method as written, i.e., it is not feasible. The reasons are technical, i.e., with respect to the refractive index liquids used, the exact weight of sample chosen which may be too great for ultrafinely ground talc samples, magnification chosen, and the optics of the light microscope itself. However, the largest problem is the need for the observer to differentiate between thousands of talc platelets on edge and few or no chrysotile asbestos fibers in a single field of view. In finely ground talc samples, literally tens of thousands of platelets on edge may be seen simultaneously. Some idea of the intensity of the problem can be observed in Appendix IV, Figures I and II. These photos are each approximately half of a single field, in which the observer must distinguish between edges of talc platelets and chrysotile fibers. Shown here at substantially greater magnification, identification is difficult. At 400X, which is about the lower limit for detection of thin particles 5 microns in length, the problem is much more difficult. These pictures were taken with polarized light which has extinguished about 50 percent of the particles to add detail. The petrographer must observe the entire field both ways.

Since the microscope stage must be rotated in a horizontal plane for each suspected "fiber" to determine if, and at what angle, extinction will occur, and the distance to the objective lens must be increased or decreased in an attempt to determine if the Beeke lines move in or out, the logistics of the method become unmanageable. Repeated for many fields and for two indices of refraction for a single sample, the method is impossible. It certainly is not as easy as the FDA authors indicate.

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Numbers of Chrysotile and Tremolitic Fibers in Talc

Since Mr. Wolkodoff is accustomed to identification of various asbestiform minerals, and since another phase of the evaluation program does involve a need for numbers of chrysotile and tremolitic fibers in various talc samples, apart from the FDA method, a procedure was derived by him which eventually arrived at some defensible numbers of fibers in various talc samples. Presence or absence of either type of fiber was then confirmed by transmission electron microscopy.

Optical Fiber Evaluation

It is not the purpose of this report to deal in detail with microscopic methods used. Observations were made at a different magnification and using different refractive indices to reduce the problems mentioned above to barely manageable proportions. Indeed with some samples the counts were made with great difficulty and only the skill of the minerologist-petrographer made such counts possible.

Validity of Numerical Values Reported

In all counting techniques it must be recognized that a single numerical value reported represents an estimate of the true value from some range of possible values which is based on such factors as how large a number of fibers might be expected, and number of optical fields counted. Once an appropriate number of fields have been examined and fiber content determined, the breadth of the range then can be used as a basis for determining the probability that the actual number of fibers present, if all could be counted, would not exceed some predetermined value, e.g., 100 chrysotile fibers or 1000 tremolitic fibers in the proposed FDA GMP.

How the number of fields observed and the number of fibers actually seen affects the reliability of the probable number of fibers in 1.0 mg of talc sample is illustrated by Appendix V, Figures I through III. Because of the small chance that a fiber will be observed in successive fields, these curves are based on a Poisson distribution rather than the Gaussian distribution which would be involved if large numbers of fibers were expected. The fiber number scales approximate the FDA proposed chrysotile numerical fiber limits. Note that these scales for both actual fibers per mg and number of fields required to see an equal number of fibers would be displaced by a factor of ten for tremolitic fibers, i.e., for ten times as many fibers expected, one-tenth as many fields would be required. The field area and total area shown are those being used by J-M.

Figure III shows that for 100 chrysotile fibers per mg examination of 100 microscopic fields will identify only three fibers or approximately one fiber in every thirty fields, and conversely every fiber seen represents about 30 potential fibers in the total sample. When fiber counts first began, lack of recognition of the importance of examining an adequate number of fields resulted in reported numbers based on only twenty fields.

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Based on Figure III, there would be less than two chances in three of seeing even a single fiber so that even zero values are suspect, but if seen, the multiplier would be 168 and three fibers seen in 20 fields is the basis for a reported value of 504 chrysotile fibers per mg of Reeves jet milled concentrate, for example.

Figure IV relates fields counted and calculated (actual) number of fibers per mg to standard deviation or the range above and below the actual number of fibers predicted. Using the first example above, 100 fibers derived from three fibers seen in 100 fields, the standard deviation is about 60 fibers per mg. Using a range of one standard deviation on either side of the mean says that for successive samples, the values have a reasonable chance of falling between 40 and 160 fibers per mg. Figure V puts this information in a different way by saying that there is a 50 percent chance that successive determinations will be greater than the reported value of 100. This becomes very important if some number, like 100 fibers, is chosen as an arbitrary limit.

Returning to numbers based on only 20 fields, the standard deviation for a reported value of 100 fibers is 130 fibers per mg and the range of -30 to 230 is statistically indefensible. For this reason all of the earlier low number chrysotile counts based on only 20 fields must be disregarded.

All recent work, where a low number of fibers has been observed, is based on 100 fields which is defensible provided it is recognized that such reported values are within a fairly broad range of possible values. As an illustration, a recount of the chrysotile fibers in Reeves jet milled concentrate, previously reported as 504 fibers per mg, was requested. In four separate 1.0 mg portions with 100 fields each, the following values were reported:

Sample No.	Chrysotile Fibers/mg
1	141
2	100
3	229
4	69
mean	135

The range of these values is surprisingly close to that which would be predicted from Figure IV.

As the number of fibers increases substantially, say to the order of 1000 per mg or more, the differences made by one or two fibers in a substantial number of fields counted carries much less weight. Because of this, 20 fields is quite adequate under such circumstances. It should be understood, however, that the calculated values are still estimates within a fairly broad range and to avoid misinterpretation of the precision of such numbers it is recommended that they be rounded off to the nearest hundred under 10,000 and the nearest thousand above that figure.

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Numbers of Fibers Optically Counted

Table II lists all of the talc samples for which there are valid fiber count data in accordance with the foregoing discussion. A number of other talc samples were examined using the 20 field procedure, and either no fibers or small numbers were reported and all such reports must be regarded as inconclusive until new determinations are made with at least 100 fields, and for no fiber samples 200 fields would be better.

Fiber Detection by Electron Microscopy

All of the samples listed in Table I, in addition to attempts to count fibers with optical methods, were studied with the transmission electron microscope (TEM). Such studies were used to confirm presence or absence of chrysotile and tremolitic fibers. No attempt was made to use the TEM as a means of counting fibers for two principal reasons:

1. The field of view is so small that the foregoing statistical implications have even greater significance.

2. The TEM routinely detects sub-optical fibers and fragments of fibers far smaller than the OSHA criteria.

For the purposes of this report, TEM data are used to record presence or absence of both kinds of fiber without regard for fiber length. Presumably, if there are very short fibers of chrysotile, for example, in a TEM sample, there will be some fibers meeting the OSHA criteria, if a large enough sample is evaluated.

Presence or Absence of Fibers

A series of talc samples from Research sources (reference standards, development materials, outside purchases, etc.) were evaluated with the results shown in Table III. Chrysotile was detected in several samples usually considered to be chrysotile free, but amounts were small to trace levels, and the possibility of contamination cannot be ruled out. However, there is no question that chrysotile fibers were present where so reported.

Table IV reports presence or absence of fibers in a series of competitive samples sent in directly from users. Unlike the previous series, some of which are several years old, these samples presumably represent current competitive production. This may be particularly significant. For example, the standard United Sierra Mistron Vapor sample (DMI C-104) had no chrysotile, but every one of the eleven field samples does have some and the chance that all could be contaminated is extremely unlikely. Further, there is some evidence, which requires additional confirmation, that some fibers are a part of the talc minerology. This would definitely preclude contamination.

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The evaluation of Pfizer Microtalcs is more difficult because they present a mixed picture. Of eight samples of Microtalc and WCD SF 399 from the same crude, two have chrysotile and the other six do not. One of the two has so much fiber as to suggest gross contamination or a mislabeled sample, which rules it out of serious consideration at this stage of the investigation. As a result, the Pfizer materials still appear to be clean.

The two tables do contain a couple of unexpected results. Val Chisone Italian talc has an appreciable chrysotile content confirming the data of Table II. Both J&J Baby Powder purchased on the market and J&J Green Mountain Standard have chrysotile present; and Yellowstone talc, widely regarded as being chrysotile free, had a trace. In the latter case, new samples should be obtained to be certain that contamination had not occurred.

Only one sample of Englehart material, Emtal 599 standard (DMI C-13), was checked and it was chrysotile free. Additional samples from this source should be obtained and checked before conclusions are drawn about current production.

Fiber Counts Versus Percent Talc Purity

It was noted that the proposed FDA GMP might include limits based on either numbers of fibers per mg or on percent talc purity with respect to fiber content, or possibly both.

To convert from fiber count to percent purity requires that certain assumptions be made which would be difficult to sustain technically, e.g., average fiber length and width as well as actual numbers of fibers, and numbers and weights of talc particles. Since a jet milled talc may contain as many as 2.5×10^9 particles per mg, technically defensible data will be difficult to sustain.

However, accepting for the minute that valid assumptions have been made, it should be pointed out that the two kinds of standard are not compatible. If fiber count governs, then talc purity with respect to chrysotile content would have to be of the order of 99.9997, and would have to be 99.995 with respect to tremolitic fiber content. Conversely, if the quoted 99.99 percent purity with respect to chrysotile were to govern, of the order of 2000 fibers of the prescribed size would be permitted, and the number of tremolitic fibers would be increased approximately proportionally.

Actually, so far as the author can ascertain, neither kind of standard has any technical basis for the two fiber levels proposed. The round number limits arbitrarily chosen definitely are not compatible. If limits must ultimately be set, there is also an absolute need for some defensible basis of their selection.

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CONCLUSIONS

The following conclusions are drawn:

1. The FDA proposed fiber counting technique as written is unworkable, and according to at least one authority may lead to erroneous conclusions.

2. Fibers can be counted but, particularly with respect to chrysotile, very special conditions are required and then an unusually high degree of skill on the part of the petrographer is essential.

3. Such determinations cannot be made by even a well qualified microscopist, and there are very few minerologist-pertrographers who could make such counts.

4. Reporting exact numerical fiber counts when dealing with small numbers of fibers is erroneous unless a very large number of fields are examined with an optical microscope, and even then the reported number is an estimate within a range related to the number of fields possible, the number of fields examined, and the number of fibers actually counted.

5. For this reason, a single numerical fiber limit per unit weight is unsatisfactory as a standard unless the statistical range related to the counting method is specifically taken into the standard.

6. Presence or absence of chrysotile and tremolitic fibers in talc can be confirmed by TEM.

7. However, both the statistical considerations of counting (a very large number of fields and/or successive samples would be necessary) and fiber length definitions preclude use of this method for defensible numerical fiber counts.

8. Chrysotile fiber presence was confirmed by TEM in a substantial number of products previously thought to be, and sold as chrysotile free.

9. This is particularly true of United Sierra Mistron products where current production, as represented by customer samples from across the U.S., did show chrysotile without exception when examined by TEM.

10. Pfizer and WCD products appear to come from the only major chrysotile-free source, but too little information is available to exclude Englehart from this category.

11. Proposed FDA GMP fiber count and talc purity limits with respect to both chrysotile and tremolitic fibers are technically incompatible with an adjustment of several orders of magnitude required to get from one kind of unit to the other at equivalent levels.

12. The proposed limits in either case appear to be arbitrarily chosen, round number values without adequate technical basis, and perhaps more importantly, without adequate means for determination.

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RECOMMENDATIONS

It is recommended on the basis of the information in this report that:

1. For the reasons detailed herein, the FDA be told its proposed fiber count method will not work, and that the suggestion be made that it be withdrawn.

2. The FDA be told that the proposed exact numerical fiber limits, particularly with respect to chrysotile, also are unworkable because of the statistical limits inherent in any fiber counting method set up for low numbers of fibers in large numbers of talc particles. Somehow, the idea should also be conveyed to FDA that the arbitrary 100 chrysotile fiber limit, and probably the 1000 tremolitic fiber limit as well, are so low as to be indefensible from the technical standpoint of end use evaluation (which has to be medical).

3. The idea be conveyed to FDA that the respective limits proposed in their two kinds of standards are not of the same order.

4. If limits on chrysotile and tremolitic content must be established, then means be found to set them at levels which are defensible from both end use and ability to determine actual fiber content bases.

5. Additional samples of Englehart, Pfizer, J&J and Val Chisone talcs be obtained for TEM examination to provide more information about the possible occurrance of chrysotile fiber therein.

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Microscopic Detection of Asbestos in Talc

by

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The six kinds of asbestos can be distinguished from talc and from each other by their refractive indices, other optical crystallographic properties, and morphology. Talc occurs mainly in the form of thin plates, which may appear to be fibrous when seen edgewise in microscopic view. Beta and gamma indices of talc vary from about 1.575 to 1.590, beta being very close to gamma. All of the principal refractive indices of chrysotile are less than 1.574, and those of the amphibole types of asbestos are greater than 1.590. One can, therefore, distinguish chrysotile asbestos from fibrous looking talc particles in a 1.574 refractive index liquid, and the amphibole types from talc in a 1.590 liquid. The accompanying table of optical crystallographic properties for talc and the asbestos minerals shows refractive indices which are usually encountered in these minerals, but occasional samples may have indices which are somewhat higher or lower.

Method

Weigh out 1 mg of a representative portion of talc on each of two microscope slides. Mix the talc on one slide with a drop of 1.574 refractive index liquid, and the other with 1.590 liquid, and place on each a cover glass sufficiently large so that the liquid will not run out from the edge (18 mm. in diameter or larger). Observe the material with a polarizing microscope at a magnification of about 400 X, using the optical crystallographic technique to detect the presence of asbestos. In the 1.574 liquid look for asbestiform fibers with indices less than 1.574 in both extinction positions, and in the 1.590 liquid look for fibers with indices exceeding 1.590. If any such particles are found, check type of extinction and sign of elongation to confirm tentative identification as asbestos. Count and record the number of asbestiform fibers found in 1 mg. as determined from a scan of either or both slides at a magnification of about 400 X. To determine specific identity of asbestiform fibers, make mounts in appropriate refractive index liquids, and refer to the optical crystallographic data in the accompanying table.

OPTICAL CRYSTALLOGRAPHIC CHARACTERISTICS OF ASBESTOS MINERALS AND TALC

Substance	Reference for n	nα	nβ	nγ	Extinction	Elongation
ctinolite	(2) p. 172	1.614	1.630	1.641	Inclined	Positive
variety of tremolite)	(8) p. 285	1.615-1.655	1.625-1.665	1.64-1.68		
mosite	(4) p. 261	1.663*	--	--		
	(4) p. 261	1.675*	--	--		
nthophyllite	(2) p. 170	1.598	1.623	--	Parallel	Positive
	(2) p. 222	1.598	--	1.623		
	(5) p. 298	1.598-1.674	1.605-1.685	1.615-1.697		
	(2) p. 222	1.608	--	1.631		
	(2) p. 172	1.619	1.630	1.640		
	(6) p. 236	1.619-1.633	1.630-1.642	1.640-1.657		
	(2) p. 173	1.629	1.635	1.640		
	(2) p. 116	1.633	1.638	1.652		
hrysotile	(2) p. 99	1.493	1.504	1.517	Parallel	Positive
	(2) p. 100	1.508	1.512	1.522		
	(3) p. 154	1.529-1.559	1.530-1.564	1.537-1.567		
	(8) p. 260	1.53-1.54	--	1.54-1.55		
	(6) p. 290	1.542	--	1.555		
	(8) p. 260	1.542	1.543 (calc.)	1.555		
	(2) p. 104	1.546	1.550	1.557		
	(1) p. 40	1.540	1.557	1.557		
	(1) p. 40	1.548	1.560	1.560		

$\frac{n\alpha + n\beta}{2}$

bstance	Reference for n	nα	nβ	nγ	Extinction	Elongation
ocidolite	(2) p. 125	--	1.687	--	Parallel	Negative
iebeckite)	(2) p. 187	--	1.695	--		
	(6) p. 241	1.693	1.695	1.697		
	(2) p. 226	1.695	--	1.703		
	(2) p. 226	1.695	1.698	--		
	(2) p. 127	1.697	1.700	1.703		
alc	(8) p. 259	1.538-1.545	--	1.575-1.590	Parallel	Positive
	(2) p. 164	1.539	1.589	1.589	or 2° or 3°	
	(3) p. 167	1.539	1.589	1.589		
	(6) p. 281	1.539	1.589	1.589		
	(8) p. 259	1.540	nearly = nγ	1.575		
	(1) p. 41	1.541	1.585	1.585		
	(1) p. 41	1.544	1.592	1.592		
	(1) p. 41	--	1.594	1.594		
	(7) p. 758	1.545	1.584	1.584		
remolite	(2) p. 222	1.599	1.613	1.625	Inclined	Positive
	(8) p. 285	1.599	1.613	1.625		
	(3) p. 169	1.599-1.612	1.613-1.626	1.625-1.637		
	(2) p. 169	1.600	1.616	1.627		
	(2) p. 222	1.602	1.614	1.635		
	(2) p. 222	1.602	1.618	1.631		
	(6) p. 237	1.602-1.623	1.613-1.638	1.624-1.650		
	(7) p. 766	1.604	1.612	1.628		
	(2) p. 222	1.604	1.617	1.630		
	(2) p. 222	1.609	1.622	1.636		
	(2) p. 222	1.609	1.623	1.636		
	(2) p. 222	1.613	1.621	1.634		

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APPENDIX II
Table I

TALC SAMPLES FOR FDA CHRYSOTILE AND TREMOLITIC FIBER COUNTS

Group I - Research Collected Samples

Sample Number	Grade Designation	Source
DMI Std 70-33	J-M Cyclo-Sorb	J-M California Crude
DMI Std 70-34	J-M Cyclo-Fil	J-M California Crude
RD73201-1	Experimental	Hazen Reeves Concentrate
DMI C-13	Emtal 599	Englehart Vermont Crude
DMI C-14	J&J Green Mountain	Johnson & Johnson Vermont Crude
RC73219-1	J&J Baby Powder	Johnson & Johnson Vermont Crude
DMI C-95	Asbestine 5X	International Talc, New York Crude
DMI C-109A	Nytal 300	R T Vanderbilt, New York Crude
RE73166-1B	-200 mesh Italian Cosmetic	Talco Grafite Val Chisone Italy
RE73234-2	-300 mesh Talc	Westside Mines Mt Sealrock Australia
DMI C-108	Fibrene C-400	United Sierra California Crude
DMI C-25	Mistron Monomix	United Sierra Montana Crude
DMI C-124	Mistron Super Forst	United Sierra Montana Crude
DMI C-104	Mistron Vapor	United Sierra Montana Crude
DMI C-38	Yellowstone Talc	United Sierra Montana Crude
DMI C-68	Microtalc MP 12-50	Pfizer, Inc. Montana Crude

Group II - Samples From Field
United Sierra Division - Cyprus Mines Products

RS73242-20	Mistron Vapor(Mont Yellowstone)	Wausau Paper Co. Wassau Wisconsin
RS73243-1	Mistron Vapor(Mont Yellowstone)	Laminex Inc. Minneapolis Minnesota
RS73243-2	Mistron Vapor(?)(Mont Yellowstone)[1]	Laminex Inc. Minneapolis Minnesota
RS73243-4	Mistron Vapor(Mont Yellowstone)	Flambeau Paper Co. Park Falls Wisconsin
RS73243-7	Mistron Vapor(Mont Yellowstone)	Plainwell Paper Co. Plainwell, Mich.
RS73243-8	Mistron Vapor(Mont Yellowstone)	Badger Paper Co. Peshtigo, Wisconsin
RS73243-10	Mistron Vapor(Mont Yellowstone)	Western Kraft Co. Albany Oregon
RS73243-11	Mistron Vapor(Mont Yellowstone)	Kimberly-Clark Co Beech Island S.C.
RS73243-12	Mistron Vapor(Mont Yellowstone)	Blandin Paper Co Grand Rapids Minn.
RS73248-2	Mistron Vapor(Mont Yellowstone)	Crown-Simpson Pulp Co (N/R)
RS73248-3B	Mistron Vapor(Mont Yellowstone)	Nekoosa-Edwards Paper Co. Port Edwards Wisconsin
RS73243-9	Mistron T-076(Calif Panamint)	Valspar Corp. Minneapolis Minnesota

1. Samples marked (?) either poorly identified or apparently misidentified in the case of MP 12-52 which is not a usual grade number for Microtalc.

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APPENDIX II - Table I (Cont'd)

Pfizer, Inc. Products

Sample Number	Grade Designation	Source
RS73243-3A	Microtalc MP 12-50 (Mont)	Thilmany Paper Co. Kaukauna Wisconsin
RS73243-3B	Microtalc MP 12-50 (Mont)	Thilmany Paper Co. Kaukauna Wisconsin
RS73243-5	Microtalc MP 12-50 (Mont)	Flambeau Paper Co. Park Falls Wisconsin
RS73248-3A	Microtalc MP 12-52 (?)[1]	Nakoosa-Edwards Paper Co. Port Edwards Wisconsin
RS73243-6	Microtalc MP 10-52	Flambeau Paper Co. Park Falls Wisconsin

Whittaker, Clark & Daniels Products (Manufactured by Pfizer)

RS73242-21	WCD SF 399 (Mont)	Viking Paint Co
RS73248-1	WCD SF 399 (Mont)	DAP, Inc. Chicago Heights Illinois
RS73248-4	WCD SF 399 (Mont)	John K. Bice Co. Los Angeles Calif.

R. T. Vanderbilt Co. Products

RS73248-5	Nytal 300 (New York State)	DAP, Inc. Chicago Heights Illinois

1. Samples marked (?) either poorly identified or apparently misidentified in the case of MP 12-52 which is not a usual grade number for Microtalc.

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APPENDIX III

HAZEN RESEARCH, INC.

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Project No. 1431
Copy No. 3

DETERMINATION OF CONTENT OF ASBESTOS TYPE MINERALS IN TALC SAMPLES

for

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INTRODUCTION

On August 28, 1973, Hazen Research, Inc., was requested by Johns-Manville Corporation to perform a determination of chrysotile asbestos and tremolite content in thirty-five talc samples. Instructions were given to Hazen Research by Johns-Manville Corp. to conduct this analysis in accordance with the procedure of the U. S. Food and Drug Administration entitled Microscopic Detection of Asbestos in Talc by Arnold E. Schulze and William V. Eisenberg. All chrysotile asbestos and amphibole asbestos fibers of 5 microns or longer with a length to width ratio of 3:1 or more were to be counted. The count was to be based on 1 milligram representative portions of the talc samples. The specified method is a microscopic procedure which makes use of differences in indices of refraction among asbestiform minerals and talc (which can appear to be fibrous when platelets are standing on edge) by means of appropriate refractive index liquids. The actual distinction is made by means of the "Becke Line," a commonly used optical effect. This effect is achieved by slightly increasing the distance between a given particle and the microscope objective until a white line or halo (the Becke line) moves either into the surrounding index liquid if the particle index is lower than the liquid or into the particle if the particle index is higher than the liquid. The substage iris disphragm must be closed down in order to obtain parallel light rays and increased contrast at the particle-liquid interface. The intensity of the Becke line is determined by various factors, however, the principal factors are particle size and the amount of difference between the index of the particle and the liquid. The smaller the particle size and the closer the indices of particle and liquid, the weaker the Becke line will be.

J-M Research Center Report 414-3 on the FDA Fiber Count Method and Chrysotile and Tremolite in Talc Samples... Published by AsbestosClaims.Law
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APPLICATION OF PROCEDURE

One milligram samples were weighed onto petrographic glass slides and mixed thoroughly with the index oils of N 1.574 and N 1.590 as specified in U. S. Food and Drug Administration procedure. The samples were covered with 18 mm cover glasses. Considerable superimposition and several layers of particles were observed; therefore, 22 mm cover glasses were subsequently used.

The samples were examined with a Leitz Ortholux polarizing microscope at 400X, with 10X oculars and a recently developed 40X objective which has a remarkable flat field. One ocular was provided with a calibrated scale and the other with a square reticle. A Chayes click stage was used to scan the prepared slide in such a manner that successive areas came into view without overlaps or gaps.

The first counts were made on the samples of the coarsest grain size. It soon became apparent that this method would be exceedingly time consuming because of the numbers of talc platelets on edge which made it necessary to check the Becke line for each one in order to distinguish these particles from chrysotile. Furthermore, after a relatively short period of observation, eye fatigue resulted in uncertainty as to which way the Becke line was moving. This uncertainty is especially severe with thin fibers in the five micron range. Because of these difficulties, it was considered justifiable to find some means of reducing the number of fields which would have to be counted in order to produce a correct count. Several approaches were considered in which the total surface area occupied by one milligram of sample was divided into a grid of fields of known dimensions. A reasonable number of these fields was then spread over the entire surface in a geometric pattern which would

help to minimize sample segregation effects. These fields, representing a known fraction of the total surface area, were then subjected to the counting procedure. From the number of fibers found per surface area viewed, the total number of fibers per total area (or per 1mg) could then be determined.

It was during the foregoing period of work that it was realized that there is a fundamental drawback to the specified procedure which makes it unworkable. When samples are composed of very fine particles (as when jet milled), a large fraction of the talc platelets will be standing on edge. Many of these in the 5 to 10 microns by 1 to 2 micron size range cannot be distinguished from chrysotile by means of the Becke line because the relief in N 1.574 oil when oriented to show the gamma and beta indices is entirely too faint. The direction of movement of the Becke line cannot be observed with certainty. Any count determined under such circumstances would not be reproducible and would be totally unreliable even as an approximation. In order to affirm this conclusion, three microscopists (two full time, one part time) each separately examined the same sample which contained particles of from 2 to 10 microns in length. There was not only disagreement on the count but even disagreement as regards the nature of specific particles.

The distinction of tremolite does not present as severe difficulties as does chrysotile. It is probable that tremolite could usually be distinguished from talc because of its typical lath shaped fragments, relatively high index, extinction angle, and typical amphibole cleavage angles.

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CONCLUSIONS

It is concluded that the distinction of chrysotile from talc by difference in refractive indices, as outlined in the U. S. Food and Drug Administration procedure referred to in the Introduction, does not offer a workable means for the determination of the content of chrysotile asbestos in talc samples, particularly in samples of especially fine particle size due either to natural fineness or to processing. Since coarser samples will also contain a certain amount of fine particles, even in these samples chrysotile content cannot be reliably determined. Because of the great difficulty in distinguishing the difference in indices of refraction by means of the Becke line, and the consequent subjectivity of interpretation by each or any observer, no positive value can be attached to any given sample.

It is possible that with respect to tremolite content in talc samples an approximate count could be made by optical microscopy; however, this would be very time consuming and tedious.

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APPENDIX IV

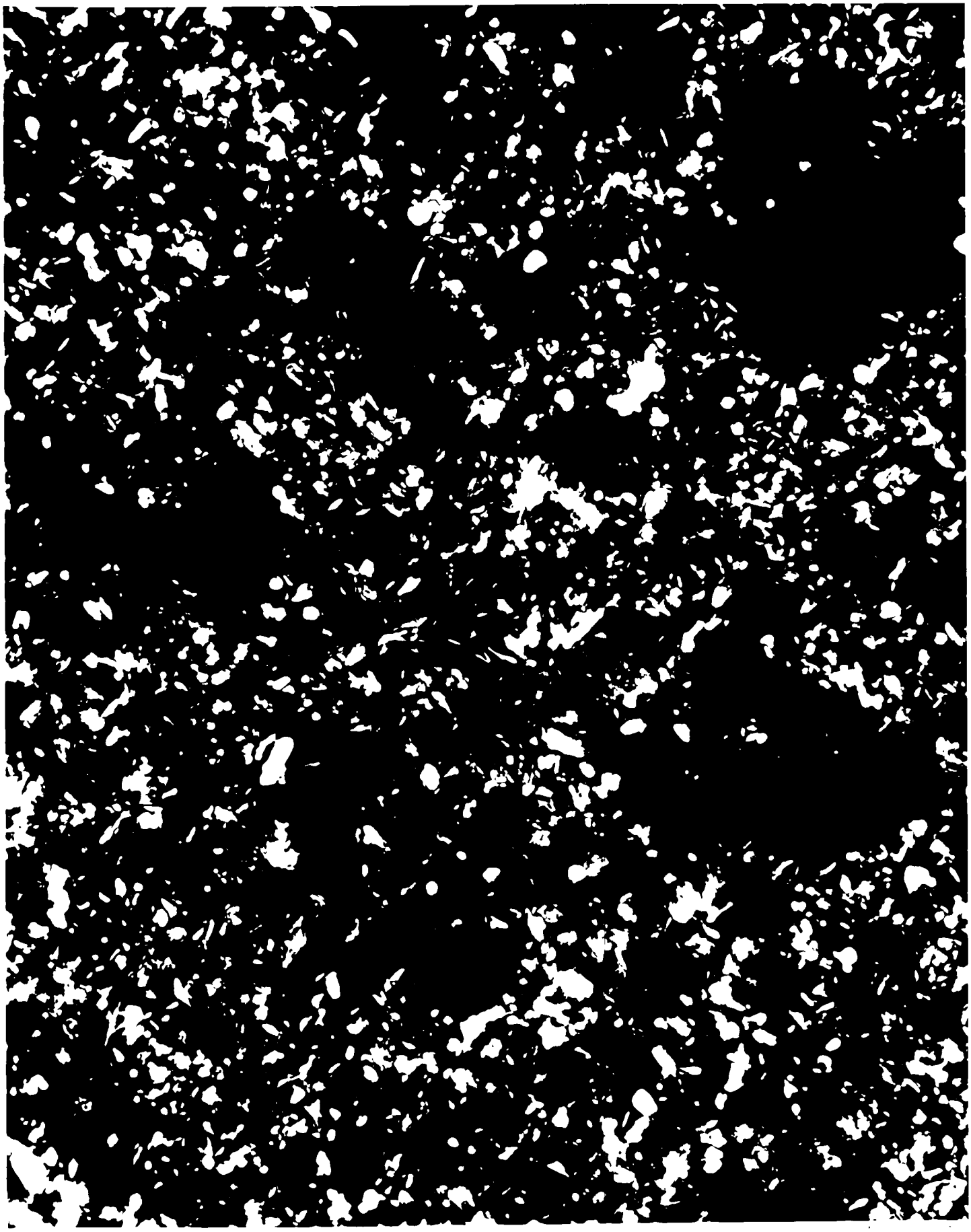
DESCRIPTION OF FIGURES I AND II

The following two (2) photos are each approximately 1/2 of a single field. These pictures were taken with polarized light which has extinguished about 50 percent of the talc particles. The magnification is 625X.

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APPENDIX V
Table II

J-M DETERMINED FIBER COUNTS FOR VARIOUS TALCS

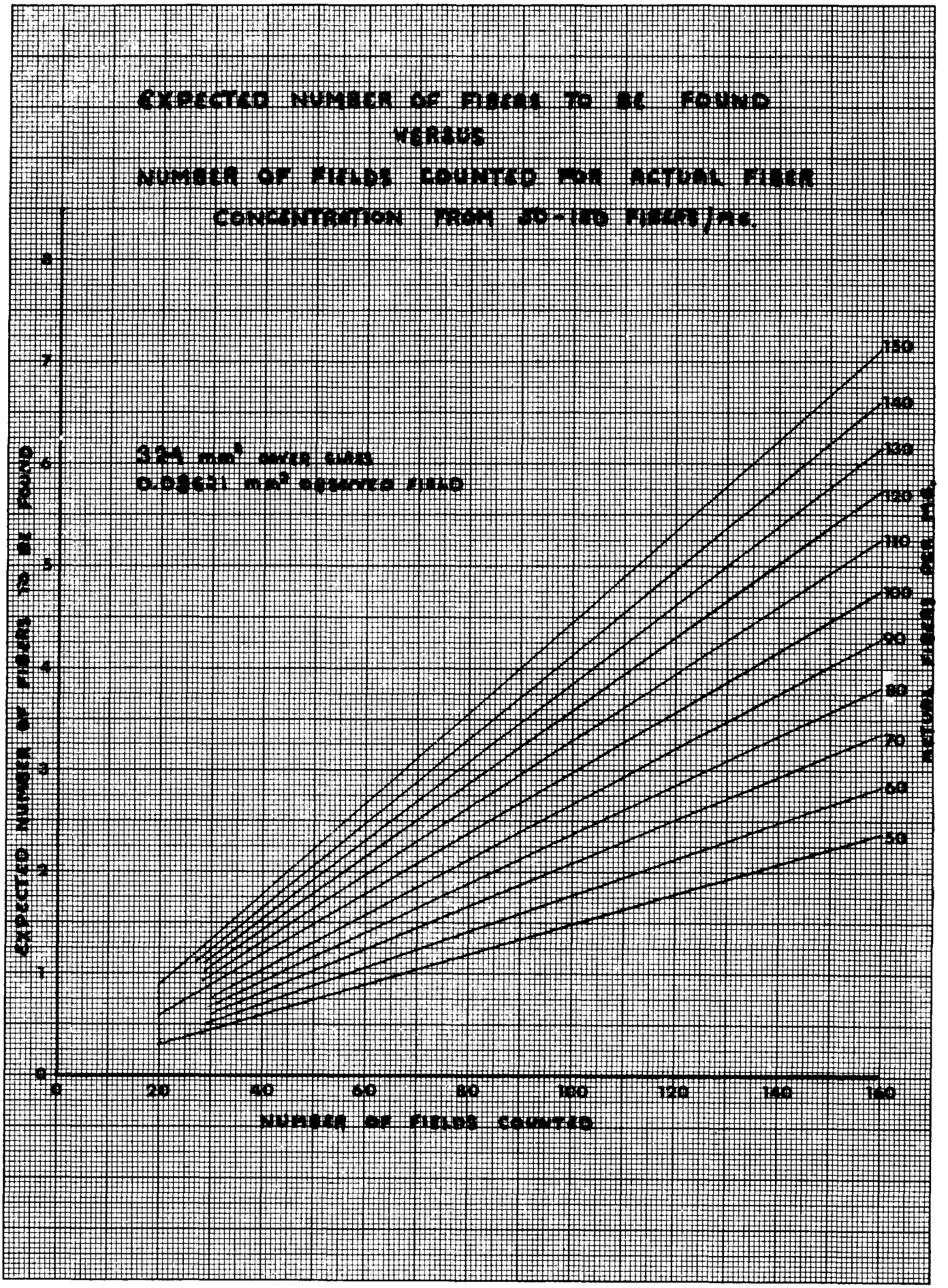
Talc	Ident. No.	Chrysotile/mg[1]	Tremolitic/mg[1]
J-M Cyclo-Sorb	DMI Std 70-33	N/D[2]	12,000
J-M Cyclo-Fil	DMI Std 70-34	N/D	35,000
United Sierra Fibrene C-400 (Death Valley)	C-108	1,400	6,500
International Talc Asbestine 5X (New York State)	C-95	17,000	17,000
R T Vanderbilt Nytal 300 (New York State)	C-190A	22,000	17,000
Talco Grafite Val Chisone (Italy)	RE73166-1B	1,900	N/D
J-M Reeves Jet Milled Concentrate (from Hazen)	RD73201-1	135	None

1. Fibers 5 microns ore more in length and 3:1 aspect ratio only.

2. Not determined. Too few fields to provide meaningful numbers.

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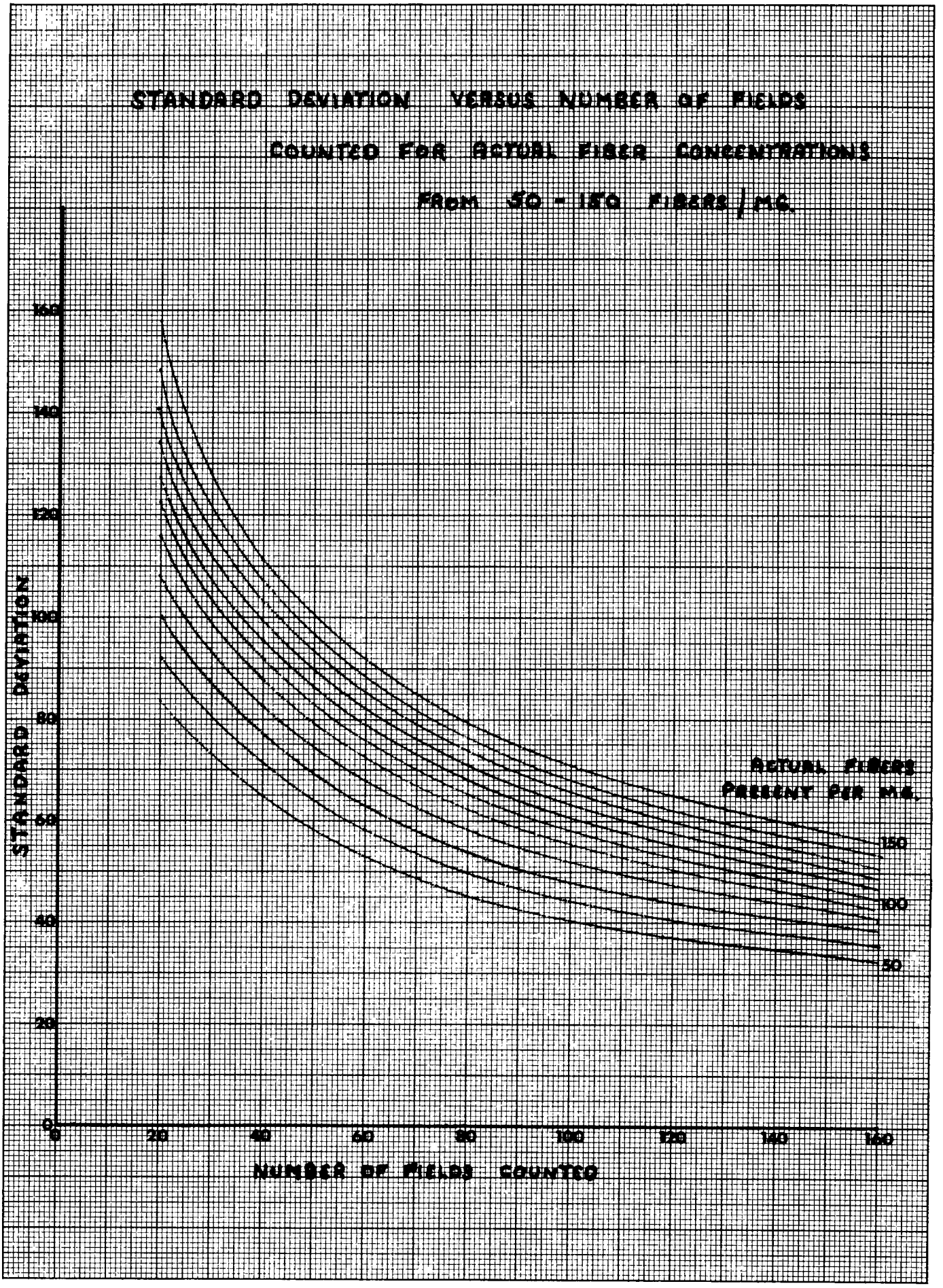
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K+E 10 X 10 TO THE CENTIMETER 46 1513
18 X 25 CM
KEUFFEL & ESSER CO.
MADE IN U.S.A.

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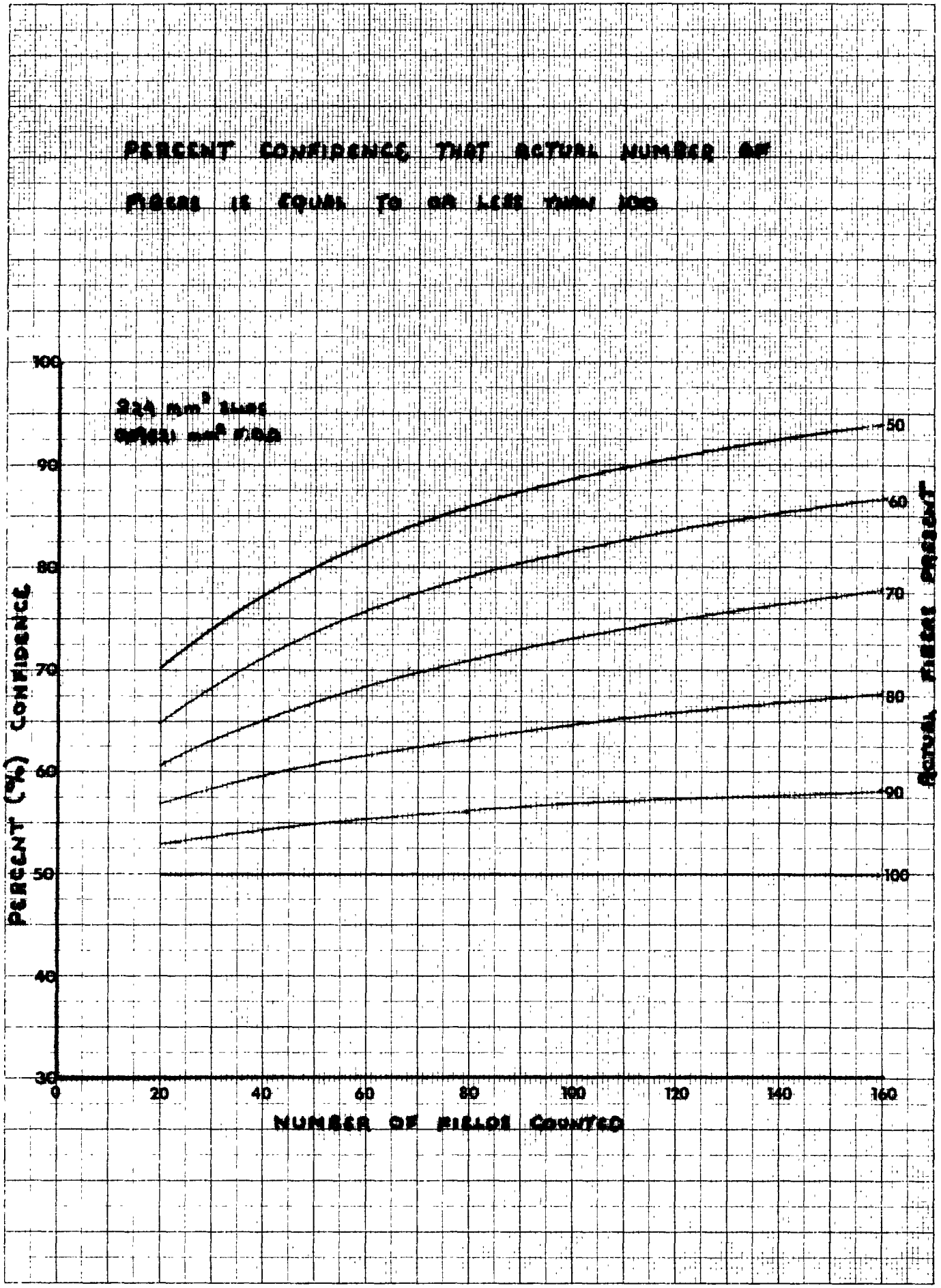
APPENDIX V
Figure IV



K+E 10 X 10 TO THE CENTIMETER 46 1513
18 X 25 CM MADE IN U.S.A.
KEUFFEL & ESSER CO

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APPENDIX V
Figure V



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APPENDIX VI
Table III

CONFIRMATION OF PRESENCE OR ABSENCE OF CHRYSOTILE AND TREMOLITIC FIBERS BY TEM

Research Collected Talc Samples, Sample No.	Grade and Crude	Chrysotile	Tremolitic
DMI Std 70-33	J-M Cyclo-Sorb (Calif)	Present(?)*	Present
DMI Std 70-34	J-M Cylco-Fil (Calif)	N.D.**	Present
RD73201-1	Reeves Jet Milled Concentrate	Present	N.D.
DMI C-13	Emtal 599 (Mont)	N.D.	N.D.
DMI C-14	J&J Green Mountain (Vt)	Present(?)	N.D.
RC73219-1	J&J Baby Powder (Vt)	Present(?)	N.D.
DMI C-95	Asbestine 5X (NY State)	Present	Present
DMI C-109A	Nytal 300 (NY State)	Present	Present
RE73166-1B	-200 Mesh Italian	Present	Present(?)
DMI C-108	Fibrene C-400 (Calif)	Present	Present
DMI C-25	Mistron Monomix (Mont)	N.D.	N.D.
DMI C-124	Mistron Super Frost (Mont)	Present(?)	Present(?)
DMI C-104	Mistron Vapor (Mont)	N.D.	N.D.
DMI C-38	Yellowstone (Mont)	Present(?)	N.D.
DMI C-68	Microtalc MP 12-50 (Mont)	N.D.	N.D.

* (?) indicates trace amounts which could be attributed to contamination. See text.

** Not detected or not positively identified.

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APPENDIX VI
Table IV

CONFIRMATION OF PRESENCE OR ABSENCE OF CHRYSOTILE AND TREMOLITIC FIBERS BY TEM

Talc Samples From Field, Sample No.	Grade and Crude	Chrysotile	Tremolitic
RS73242-20	Mistron Vapor (Mont)	Present(?)[1]	N.D.[2]
RS73243-1	Mistron Vapor (Mont)	Present(?)	N.D.
RS73243-2	Mistron Vapor (Mont)	Present(?)	N.D.
RS73243-4	Mistron Vapor (Mont)	Present[3]	N.D.
RS73243-7	Mistron Vapor (Mont)	Present	N.D.
RS73243-8	Mistron Vapor (Mont)	Present	N.D.
RS73243-10	Mistron Vapor (Mont)	Present	N.D.
RS73243-11	Mistron Vapor (Mont)	Present[4]	N.D.
RS73243-12	Mistron Vapor (Mont)	Present	N.D.
RS73248-2	Mistron Vapor (Mont)	Present[4]	N.D.
RS73248-3B	Mistron Vapor (Mont)	Present	N.D.
RS73243-9	Mistron T-076 (Calif)	Present	N.D.
RS73243-3A	Microtalc MP 12-50 (Mont)	N.D.	N.D.
RS73243-3B	Microtalc MP 12-50 (Mont)	Present[4]	N.D.
RS73243-5	Microtalc MP 12-50 (Mont)	N.D.	N.D.
RS73248-3A	Microtalc MP 12-52(?) (Mont)[6]	N.D.	N.D.
RS73243-6	Microtalc MP 10-52 (Mont)	N.D.	N.D.
RS73242-21	WCD SF 399 (Mont)	N.D.	N.D.
RS73248-1	WCD SF 399 (Mont)	Present[3]	N.D.
RS73248-4	WCD SF 399 (Mont)	N.D.	N.D.
RS73248-5	Nytal 300 (NY State)	Present[5]	Present[5]

1. (?) indicates presence of traces small enough to suggest they could be contaminated.

2. Not detected.

3. Present in trace quantities too great to be considered contamination.

4. More than trace quantities.

5. Appreciable amounts percentagewise.

6. Not a usual grade number, probably misidentified by source.

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