

PRODUCTS
DIVISION

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REQUEST FOR TECHNICAL SERVICE

DATE: 7/10/76
GROUP: Zambelli
DATE: June 25, 1976
CHARGE NO.: 71-048
REQUESTOR: R. H. Locke
MANUFACTURING or MANUFACTURING APPROVAL
NAME: H. A. Brown
APPROVED:

PROBLEM TITLE: Perform quantitative analysis of one-bag samples of MK-4 and MK-5 (already in Cambridge)

SIGNIFICANCE: To determine amount of tremolite present.



SPECIFIC OBJECTIVE: Approximation of analysis results which might be obtained by a laboratory facility other than ours.

SUGGESTED APPROACH: Perform analysis twice. The first approach utilize methods, equipment, and procedures which CPD laboratory personnel would use based on experience, etc. Second approach to be that (or those) which an outside facility would use (possibly same as CPD; if different from CPD, possibly more than a single alternate).

DEADLINE (Last day information will be of value): There is not now an identified deadline. However results are requested within a 30 to 45 day period if practical.

DETAILS OF PROBLEM: H. C. Duecker is familiar with all pertinent details.

ACCEPTED BY RESEARCH DEPT.:

DATE: 7/5/76

ASSIGNED TO:

J. C. Yang / Steven Vaughan

ADDITIONAL COPIES: Original to Library, B.A. Blessington, H.C. Duecker, F.W. Eaton, P.E. Korenberg, R.H. Locke, R.A. Merther, L.S. Shu, B.R. Williams, J.W. Wolter, E.S. Wood, R.M. Vining, CPD-T&A, File: 71-048

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15171792

REQUEST FOR TECHNICAL SERVICE:**CONFIDENTIAL**

NUMBER: 49189

GROUP: ZONOLITE

ACTUAL COST: \$2500.00

REPORTING DATE: May 26, 1977

SUMMARY:

Three bags of standard MK-4 product from plant locations in California from Los Angeles, Santa Ana, and Newark), and four MK-5 samples (from Los Angeles, Santa Ana, and Omaha) have been examined for their tremolite content.

All seven samples as received showed no detectable tremolite fiber content by x-ray determinations (our detection limit for tremolite is 0.2%). However, the materials were fractioned; glass fibers were mostly retained on a +6 mesh screen, vermiculite was floated off; most of the plaster of Paris was dissolved in water; and, CELIF fibers and organic matter were burnt off. The concentrated fines, collected on Millipore filter of 0.45 μ , showed the presence of trace amounts of tremolite fiber in two of the trace MK-4 samples (Santa Ana and Newark). By petrographic microscopic examination, this was estimated to be less than 0.015% of the total sample.

The concentrates were then submitted to Arthur D. Little, Inc., for transmission and scanning electron microscopic analysis (TEM and SEM), selected area electron diffraction (SAED) and energy dispersive x-ray analysis (EDAX).

By these sophisticated and time-consuming instrumental analyses, the amphibole fibers were positively identified and analyzed. On a mass basis, it was found to be less than 0.006% of the concentrates which corresponded to 1.7 ppm* (Santa Ana) and 4.1 ppm (Newark) of the total MONOKOTE® sample weight.

EXPERIMENTAL:Concentration

The concentration procedure of MONOKOTE is shown in Figure 1. The results are tabulated as follows:

* parts per million, or 0.00017%.

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Fraction	Description	Material Present	% by weight in each Fraction						
			MK-4			MK-5			
			L.A. (8/76)	S.A. (8/76)	Newark (8/76)	Omaha new (8/76)	Omaha old (10/76)	S.A. (10/76)	L.A. (10/76)
1	Soluble	plaster of Paris	28.5	40.2	46.0	33.3	37.2	43.6	40.4
2	+6 Mesh	glass fiber	57.8	56.9	47.1	56.0	49.2	49.1	55.3
3	-6 +50	glass fiber, expanded Vm. some insoluble plaster							
4	Fines	some insoluble plaster, fine Vm. and tremolite (?), gypsum	13.7	2.9	6.9	10.7	13.6	7.3	4.3
			100.0%	100.0	100.0	100.0	100.0	100.0	100.0

X-Ray Diffraction Analysis

No detectable tremolite found in any of the fractions of the seven samples.

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Petrographic Microscopic Examination

Based on the characteristic refractive indices and optical properties of vermiculite and tremolite fibers, using the liquid immersion technique, a trace of tremolite was found in the -50M +0.45 μ portion of Santa Ana MK-4, and Newark MK-4 samples.

Analysis by Arthur D. Little, Inc.

Even though the original request made by R. H. Locke was on one MK-4 and one MK-5 sample, we have decided to do several more since the product from each plant looked and behaved very differently. The MK-4 from Newark was very dense and the vermiculite present was poorly expanded in comparison with the others. Product from Santa Ana was very bulky and the plaster of Paris present in the composition dissolved more readily than the others.

The two concentrated samples suspected to have tremolite fibers were submitted to Arthur D. Little for fiber characterization and counting on transmission micrographs (Figures 2 and 3). Each fiber being counted was analyzed by SAED (selected area electron diffraction) to determine the structure of the fiber. It was found that 25-40% of fibers did not yield an SAED pattern indicating the fiber was amorphous, mostly organic and glass fibers. The breakdown of the fiber types and amounts is listed in Table 1.

Scanning electron micrographs were also taken on some of the fibers. They are shown in Figures 4 and 5, and energy dispersive x-ray analysis (EDAX) was employed to analyze the elements present in each fiber. The results are shown in Table 2.

CONCLUSIONS and COMMENTS:

The conclusion reached by A. D. Little, Inc. was that the amphibole fiber content, on a mass basis, corresponded to less than 0.006% of the supplied concentrated sample. Letter from Dr. E. Peters of ADL is attached. Computing the amphibole content in the MONOKOTE samples from Santa Ana and Newark, this corresponds to less than 1.7 ppm and 4.1 ppm, respectively. The level of tremolite fiber present was extremely low.

Julie C. Yang
Julie C. Yang

JCY:mlr

attachment

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NUMBER:	49189
GROUP:	ZONOLITE
ACTUAL COST:	\$2500.00
REPORTING DATE:	May 26, 1977

TABLE 1 - Fiber analysis by TEM (A.D.Little)

Fiber Observed	Sample 22281-1 Fines Fraction from Santa Ana, MK-4 Sample	Sample 22281-2 Fines Fraction from Newark, MK-4 Sample
Total fibers observed	104	54
% Amphibole	6	4
% Other Mineral (mostly gypsum)	33.5	35
% Ambiguous Mineral (with insufficient data for positive identification)	34.5	22
% Amorphous (organic, glass fiber)	<u>26</u> <u>100%</u>	<u>39</u> <u>100%</u>

TABLE 2 - EDAX Microchemical Analysis of Fibers
Observed by Scanning Electron Microscopy (A.D.Little)

Sample 22281-1		Relative X-ray Intensity			Probable I.D.
		Strong	Medium	Weak	
Fiber 1	Figure 6a	Al	S	Mg	
Fiber 2	Figure 6b	Si, Al	Mg, Ca, S	Fe, K	amphibole or glass
Fiber 3	Figure 4a	Al	-	Ca, S, Si	gypsum (?)
Fiber 4	Figure 4b	Si, Al, Mg, S	Ca, Fe	K	amphibole or glass
 <u>Sample 22281-2</u>					
Fiber 5	Figure 3	S, Ca, Al			gypsum

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Arthur D. Little, Inc. ACORN PARK • CAMBRIDGE MASSACHUSETTS 02140 • (617) 864-5770

April 5, 1977

Dr. Julie C. Yang
Manager, Research Technologies
Construction Products Division
W. R. Grace & Co.
62 Whittemore Avenue
Cambridge, Mass. 02140

Dear Julie:

C76494

As we discussed during your visit on March 11, 1977, low magnification transmission electron microscope photographs have been obtained from two representative grid pore openings of samples 22281-1 and 22281-2 to permit an estimate of the percentage of mass attributable to fibers, in particular, amphibole fibers. A previous analysis of these samples, reported on January 24, 1977, identified the presence of fibers, most of which were mineral. These results can be summarized as follows:

	<i>Santa Ana</i> <u>22281-1</u>	<i>Newark</i> <u>22281-2</u>
Fibers observed	104	54
Percent amphibole	6	4
Percent other mineral (mostly gypsum)	34	35
Percent ambiguous mineral	35	22
Percent amorphous (organic, glass fiber)	26	39

As some of the ambiguous mineral category may be amphibole, it is prudent to estimate a maximum amphibole fiber content of 10 percent. Due to a slightly larger fiber size, the amphibole fiber volume is about 15 percent of the total fiber volume, which corresponds to 1.6×10^{-12} cm³ per grid pore opening.

To estimate the relative amount of fibrous material present in the samples, low magnification TEM photographs were obtained from two representative pore openings of both samples. These were assembled into

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April 5, 1977

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Dr. Julie C. Yang
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montages, which covered entire pore openings. Particle volumes per pore opening were calculated for the two montages prepared for sample 22281-1A (exhibiting the heaviest particle loading) from the projected surface area and an estimated thickness of each particle, as follows:

0.2 μ m - particles showing electron beam penetration
over whole area

0.5 μ m - particles showing electron beam penetration
at edges

1-2 μ m - electron opaque particles

From these estimates, the ratio of fiber volume to total particle volume was estimated to be 0.04 percent (0.006 percent for amphibole fibers). For the assumption that the densities of all particles are equivalent, these percentages apply on a mass basis, as well.

From this analysis, we conclude that the amphibole fiber content, on a mass basis, corresponds to less than 0.006 percent of the supplied sample, which represented the insoluble residue fraction of a leached Monokote sample. This estimate should be reliable within a factor of two times.

Please contact me if you have any questions.

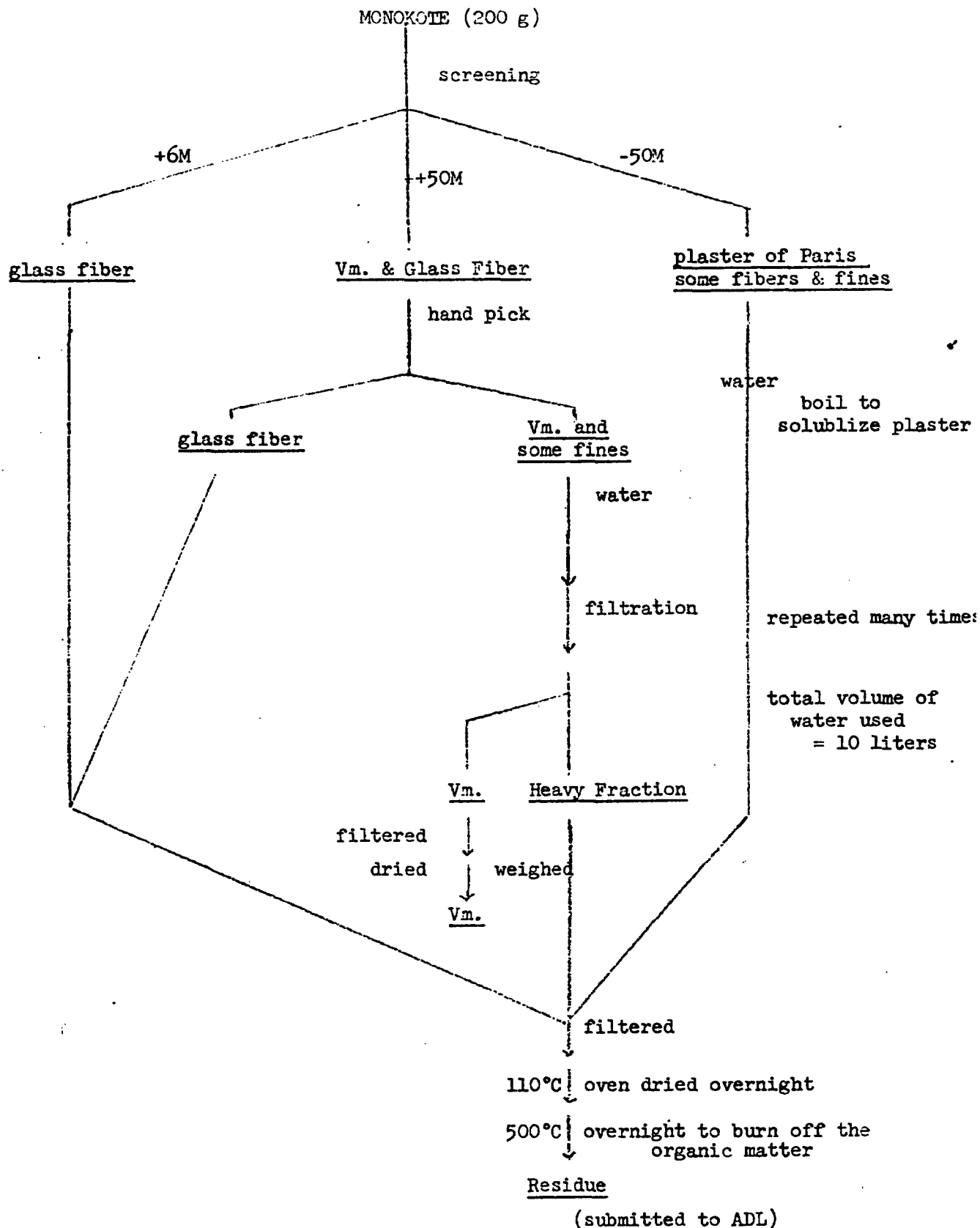
Very truly yours,



Edward T. Peters

/rdl

FIGURE 1 - CONCENTRATION OF FINES IN MONOKOTE®



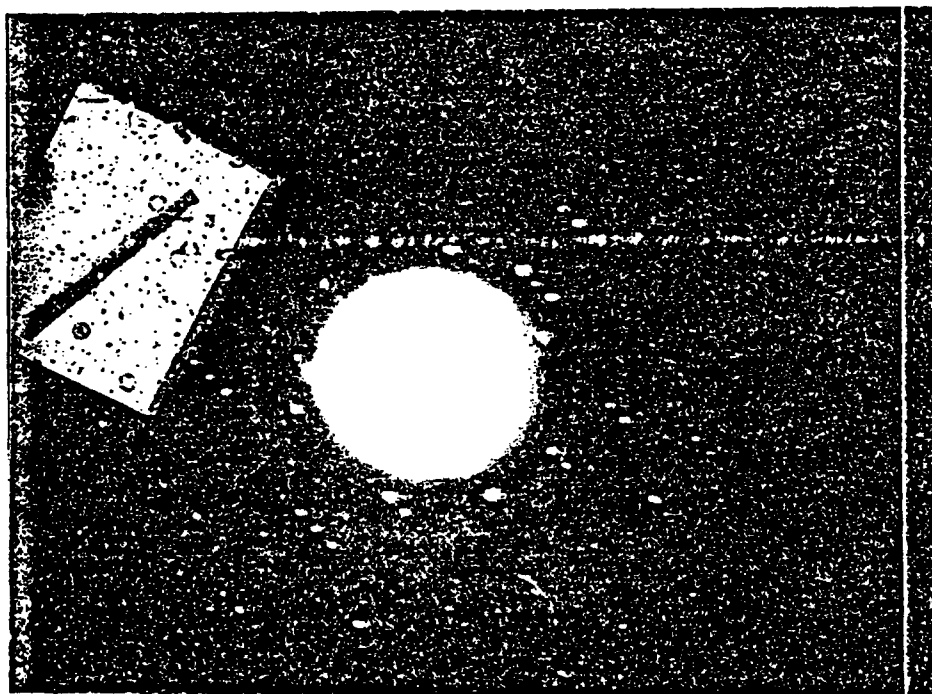
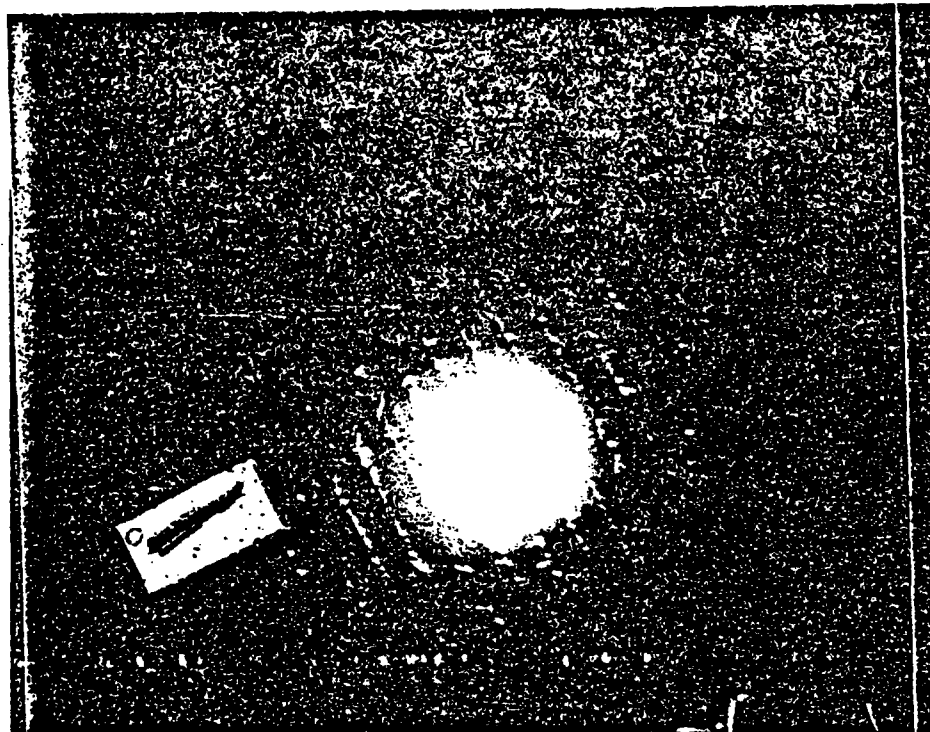


Figure 2. Transmission Electron Image of Fibrous Particles and Corresponding SAED Patterns, Sample 22281-1; 10,000x.

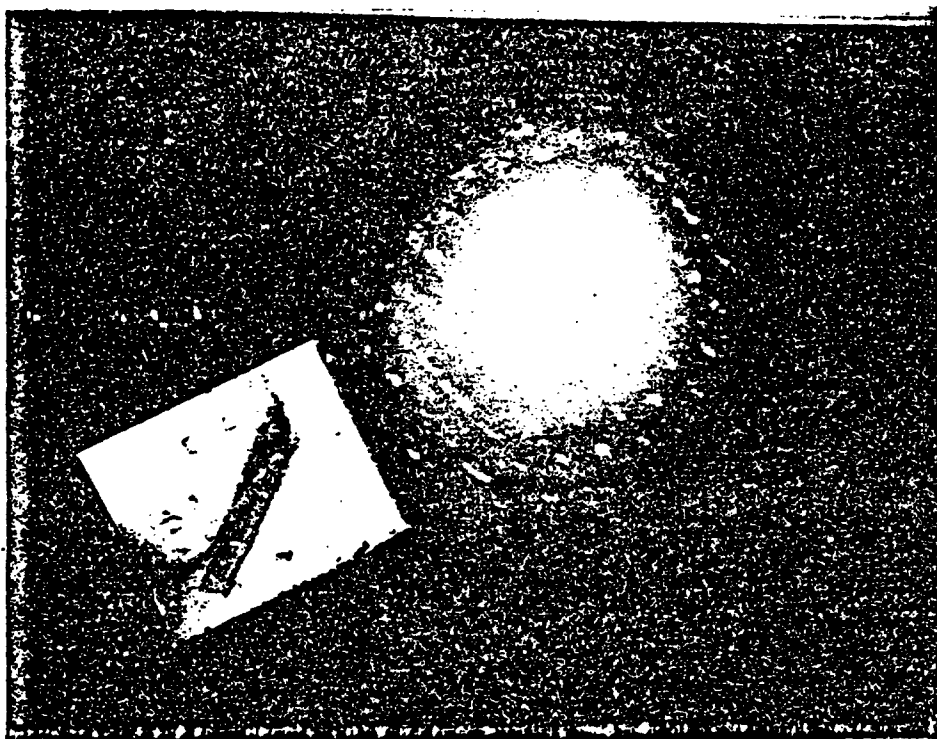


Figure 3. Transmission Electron Image of Fibrous Particles and Corresponding SAED Pattern, Sample 22281-2; 10,000x.

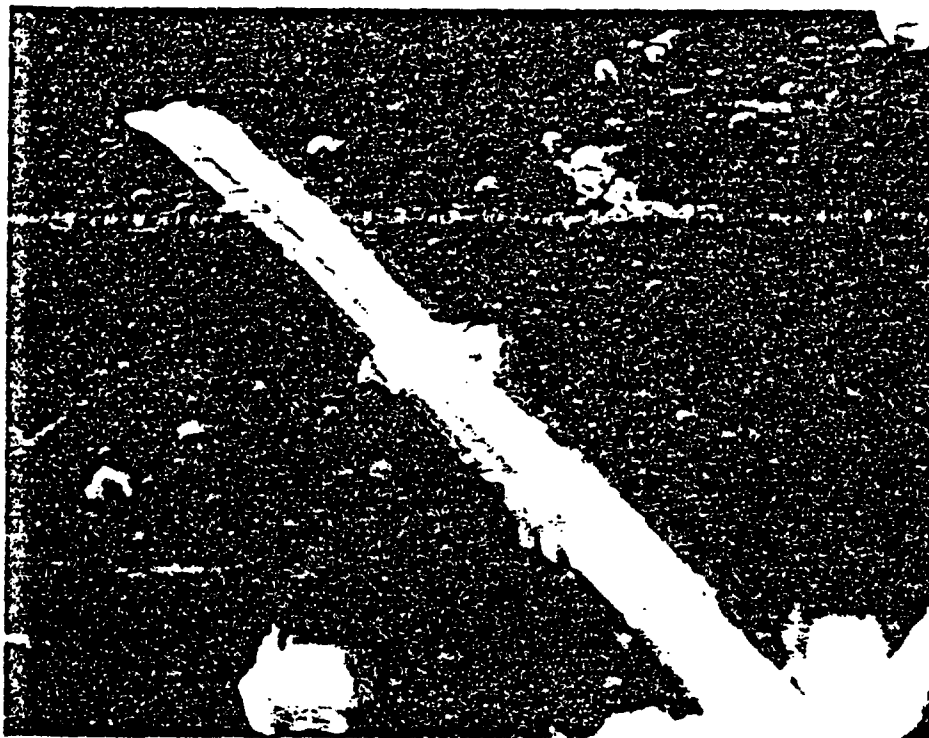


Figure 4. | Scanning Electron Micrographs of
Fibrous Particles in Sample 22281-1
a) 5500x, b) 5500x

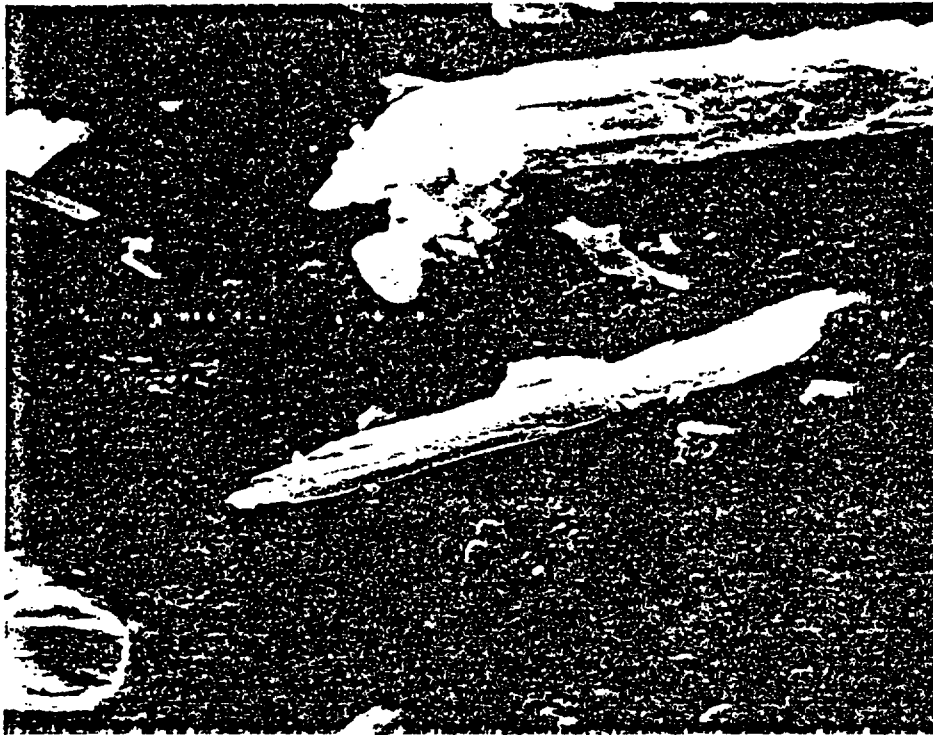


Figure 5 | Scanning Electron Micrograph of
a Fibrous Particle in Sample 22281-2,
5500x.

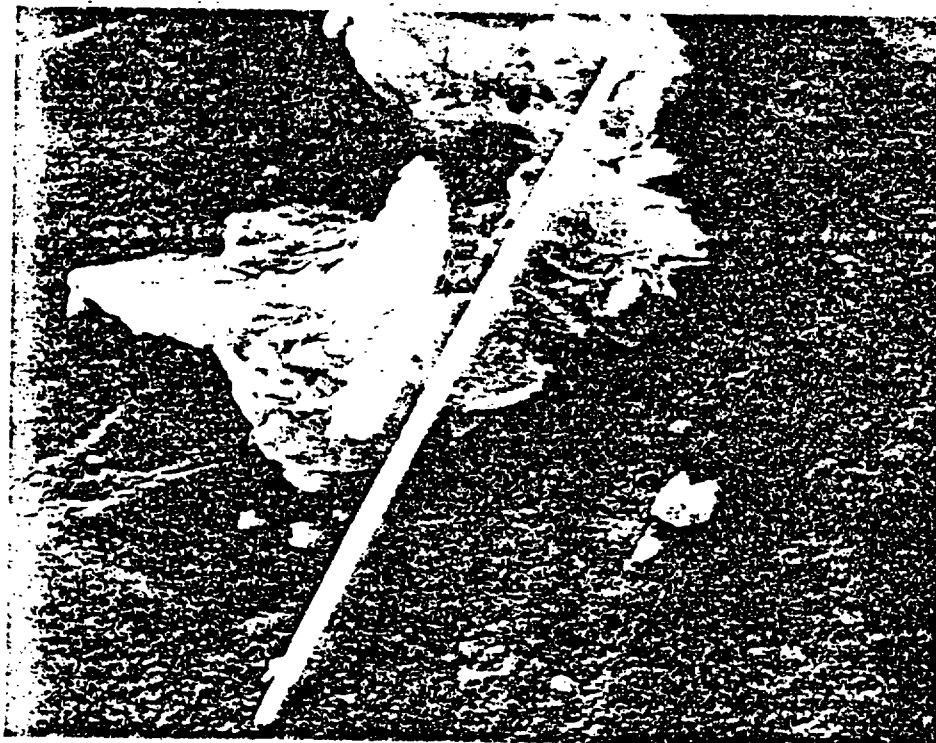
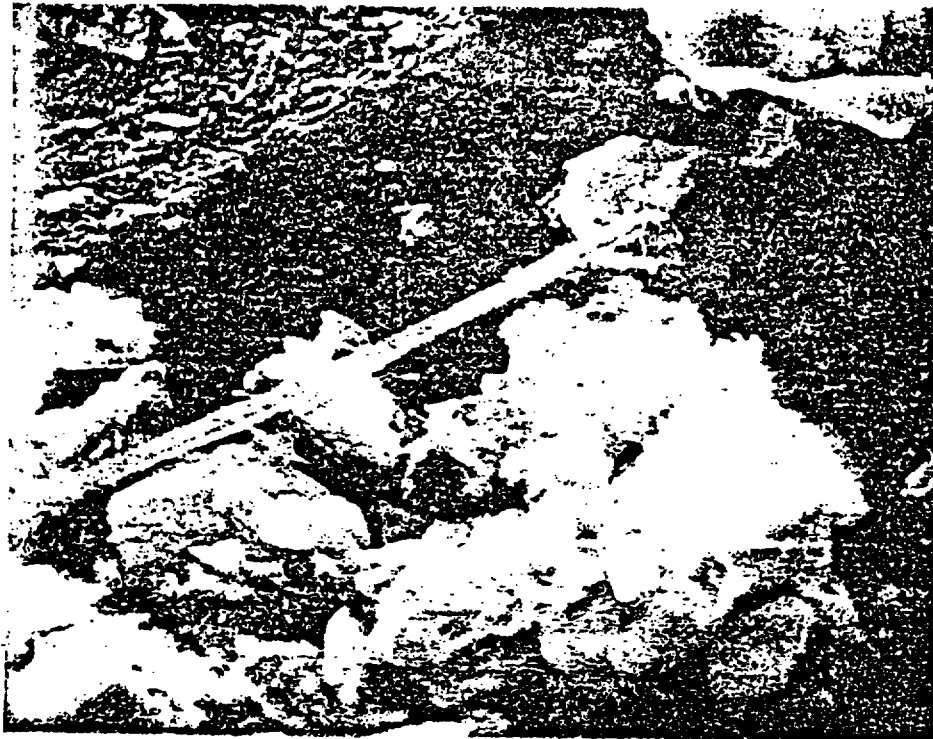


Figure 6) Scanning Electron Micrographs of
Fibrous Particles in Sample 22281-1.
a) 2400x, b) 1100x