SUMMARY REPORT

ISOPHORONE CATALYST DEVELOPMENT

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EVALUATION OF ASBESTOS FOR USE AS ISOPHORONE CATALYST

Authors: A. L. Maloy
R. W. Halstead

Date: March 24, 1965

Project No.: 113A10

File No.: 3854

SUMMARY A catalyst prepared from asbestos has been evaluated in the laboratory reactor for use as an isophorone catalyst. The reaction stabilized at a moderately high conversion level with a chemical efficiency to useful products above 90 per cent. The ratio of mesityl oxide to isophorone, however, was approximately 3:1. This catalyst, therefore, would not be suitable for the production of isophorone. The catalyst does have merit for use to produce mesityl oxide.

INTRODUCTION Mr. J. B. Johnson, R and D business team representative, (No. 13) requested the evaluation of asbestos upon the recommendation of Mr. H. D. Kinsey (NYO). Had the asbestos resulted in a suitable catalyst, the economics of using it would be favorable because of the low cost and plentiful supply.

DISCUSSION The asbestos, which was received as a dry powder, was mixed with enough water to form a paste and was then extruded with a caulking gun into long cylindrical pieces approximately 3/8-inch in diameter. The material was dried and then broken into small sections about 1/2-inch long. The finished catalyst had a surface area of 53.5M^2/g which is 2 to 5 times greater than the catalyst currently being used for the production of isophorone.

The laboratory reaction equipment used for this evaluation was described in Status Report 2915. The reaction parameters used for this investigation were as follows:

Reactor Temperature, °C	300-375
System Pressure, psig	30
Catalyst Volume, ml	1000
Contact Time, Seconds	64
Reactor Feed	Acetone

A reaction temperature of 355°C was maintained for the first 187 hours of operation to observe the decline in catalyst activity with catalyst age. A plot of conversion versus hours

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Chemicals Division
Union Carbide Corporation

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of operation, shown on Graph 1 in the attachments, indicates that the decline in activity after 187 hours of operation was less than 10 per cent. Also shown on this graph is the production of mesityl oxide and isophorone expressed in per cent of acetone conversion. As can be seen from the graph, the ratio of mesityl oxide to isophorone was approximately 3:1 throughout the 187 hours of operation. After 90 hours of operation, the chemical efficiency to useful products was 90 per cent or better for the duration of the run.

For the second phase of the investigation, the reaction was operated through a temperature range of 300-375°C to observe the influence of reaction temperature on catalyst performance. Graph 2, a plot of conversion versus reaction temperature, shows a significant increase in conversion when the temperature is increased from 300°C to 355°C. When the temperature was further increased to 375°C, the rate of increase in conversion was much lower. This can be attributed to the increased production of residues which deposited on the catalyst and masked the catalytic surface, thereby decreasing activity. The production of mesityl oxide and isophorone increased at about the same rate with increasing temperature. At reaction temperatures of 325°C and lower, the formation of residues was low and chemical efficiencies were in excess of 95 per cent.

CONCLUSIONS This material when considered for use as an isophorone catalyst, was quickly discredited because of the low conversion to the desired product. However, when operated at a reaction temperature of 300°C, asbestos shows considerable promise for use as a mesityl oxide catalyst. No attempt was made to thoroughly evaluate the catalyst for production of the latter material.

Attachments:
2 Figures

R.W. Halstead

whn

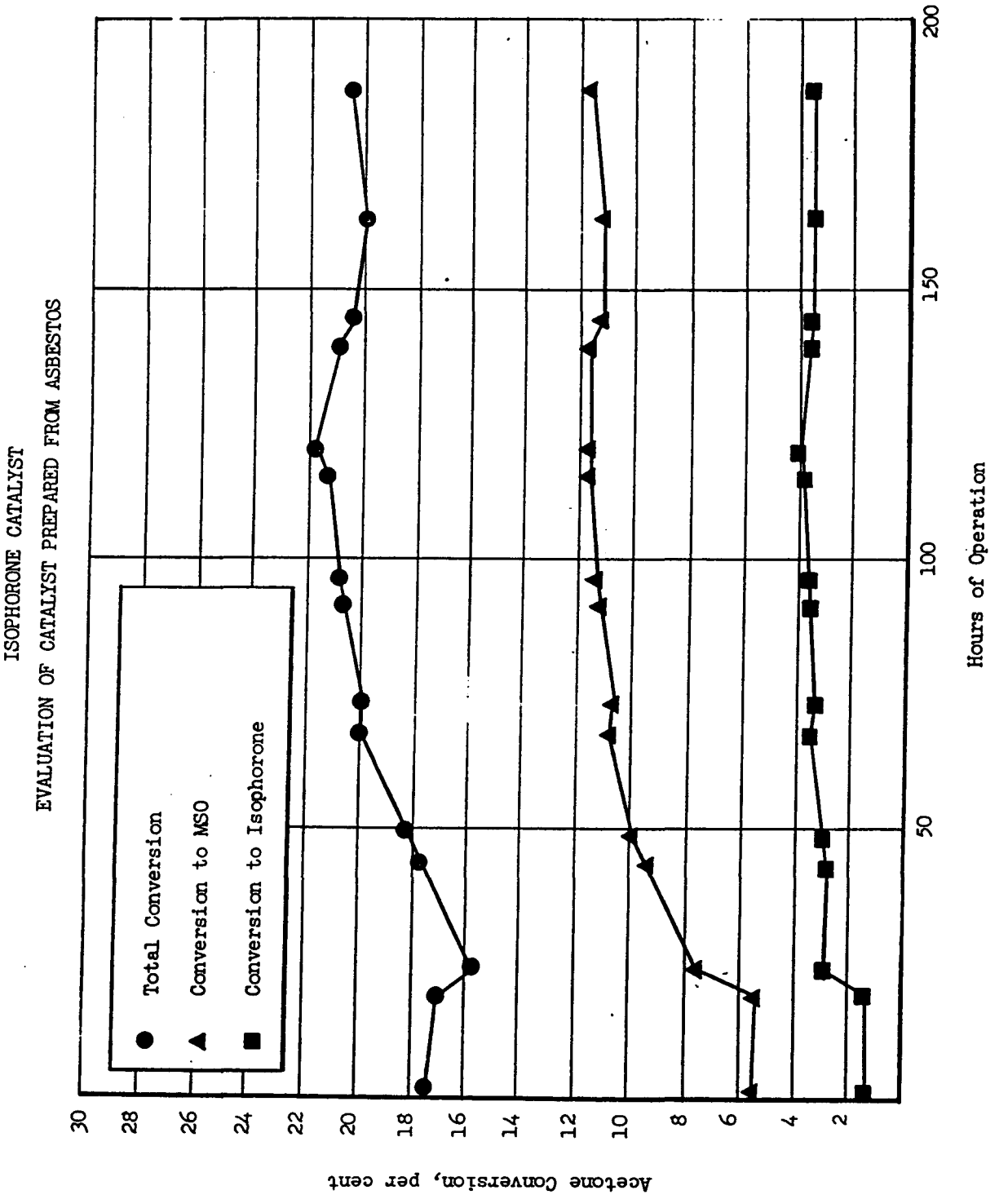
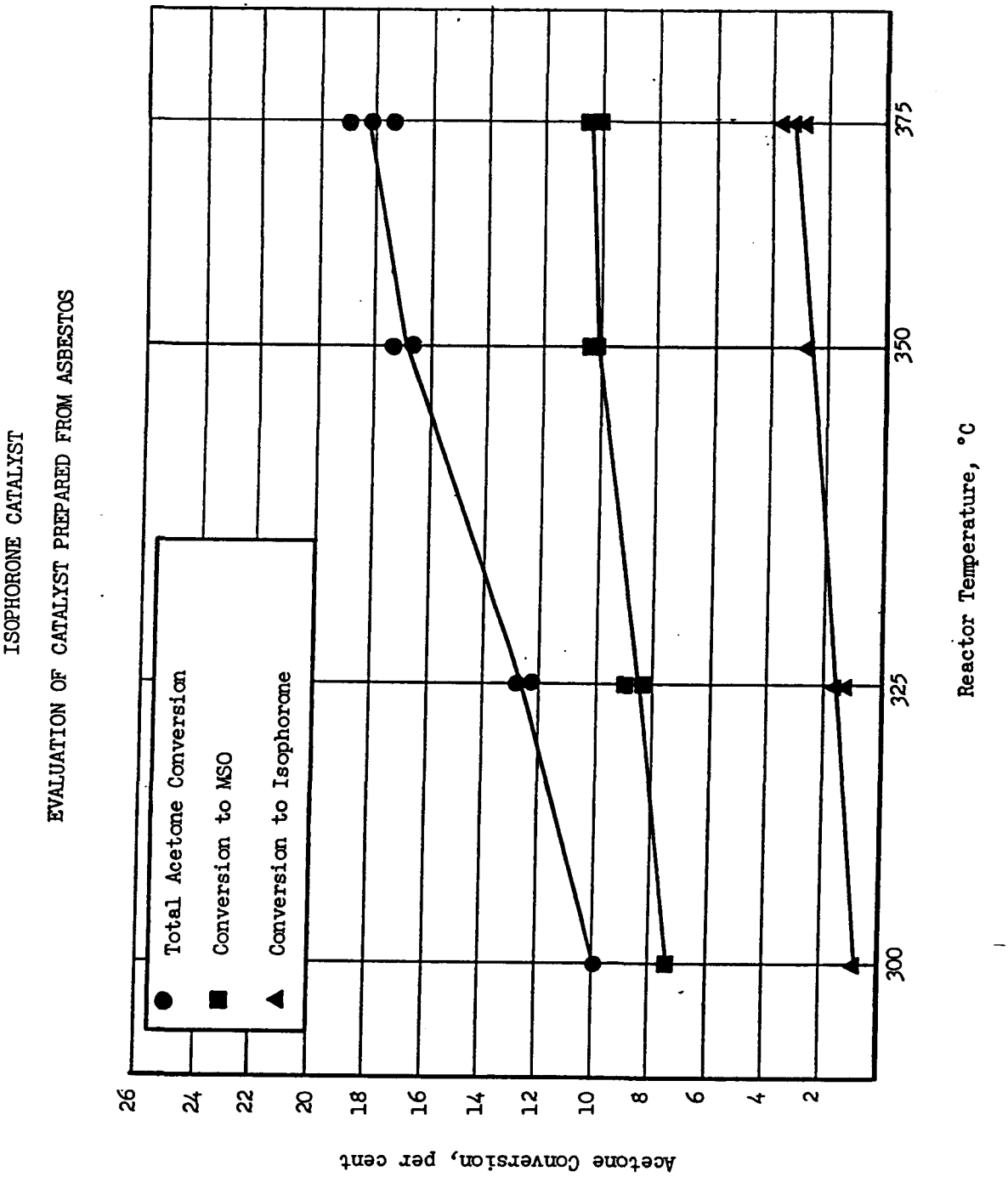

EVALUATION OF CATALYST PREPARED FROM ASBESTOS
ISOPHORONE CATALYST
Total Conversion
Conversion to MSO
Conversion to Isophorone
Acetone Conversion, per cent
Hours of Operation
30
28
26
24
22
20
18
16
14
12
10
8
6
4
2
50
100
150
200

FIGURE 2



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INTERIM REPORT

G

MODIFIED HIGH PURITY ASBESTOS:

THE EVALUATION OF SILICA-TREATED ASBESTOS AND ALUMINA-SILICA-TREATED ASBESTOS AS PAPERMAKING ADDITIVES

Author: G. L. Dickson

Date March 21, 1967

Study No. 61

Notebook No. 1691

SUMMARY

Previous work has shown that laboratory prepared silica-treated asbestos and alumina-silica-treated asbestos are suitable as papermaking additives, and that for this purpose they possess properties which make them superior to unmodified High Purity Asbestos. The present work was intended to evaluate pilot plant samples of similar materials by incorporating them in laboratory handsheets. The results again indicate that these materials can be adopted for use as papermaking fillers, and that their optical properties approach those of our present Asbestos "T" product. The favorable results obtained from this work suggest that a more complete program for evaluating these materials and their method of preparation should be initiated as soon as possible.

INTRODUCTION

1. Pilot Plant Production

The pilot plant production of four samples of alumina silicate-treated asbestos was carried out under the supervision of Dr. S. Chwastiak. For convenience, his Progress Report of December 1966, covering the method of preparation, is attached. Basically, it consisted of treating 13 pounds of High Purity Asbestos with sodium silicate, followed by four levels of addition of aluminum acetate. Also included in his report are the results of the zeta potential measurements made on the various samples.

2. Samples Identification

The five samples were received by the Paper Laboratory in opened form, i.e., the dried filter cakes had been opened dry in a Mikro-pulverizer. The samples were identified as follows:

Research and Development Department
Chemicals & Plastics
Operations Division
Union Carbide Corporation
Niagara Falls, New York

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(1) 1684-25-1 Sodium silicate treated (0.38% Al_2O_3)
(2) 1684-25-2 Aluminum acetate treated (1.30% Al_2O_3)
(3) 1684-25-3 " " " (2.72% Al_2O_3)
(4) 1684-25-4 " " " (5.25% Al_2O_3)
(5) 1684-25-5 " " " (7.35% Al_2O_3)

(Numbers in parenthesis denote Al_2O_3 content as established by chemical analyses)

3. Evaluation of Samples

A. Brightness

Compressed pellets were formed from the five samples and the brightness (reflectance) determined using the Photovolt brightness meter. The following results were obtained:

Sample	Brightness
25-1	82.0
25-2	82.5
25-3	82.5
25-4	83.0
25-5	82.5

These values appear quite favorable in contrast to that of 74-76 usually obtained on our High Purity Product. Brightness measurements were also made on similar pellets which had been heat treated at two temperatures. As can be seen from the following, there is considerable color degradation in all cases.

Sample	Untreated	2 hr. at 500°C.	2 hr. at 700°C.
25-1	82.0	73.0	66.5
25-2	82.5	73.5	65.5
25-3	82.5	74.5	67.5
25-4	83.0	73.5	68.5
25-5	82.5	74.5	68.0

B. Thixotropy

Viscosity measurements were obtained on styrene-paraplex systems containing modified asbestos, which is the present method for determining the acceptability of RG-244. These results indicate that the sodium silicate-treated sample (1685-25-1) was of marginal quality, since a viscosity of 20,000 cps at 25°C. is usually considered as being minimal. For academic reasons, the aluminum acetate-treated samples were also included and, as was expected, produced poor results.

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Sample	Viscosity Results (cps) 45°C.	25°C.
25-1	12,500	17,000
25-2	500	3,000
25-3	300	1,500
25-4	300	900
25-5	300	1,100

C. Laboratory Handsheets

The compatibility of the various samples in a papermaking system was determined by incorporating them at 3 addition levels in laboratory handsheets. The Noble and Wood equipment (8" x 8" sheet mold) was used, utilizing a 100% bleached sulfite pulp beaten to 200 CSf. For comparative purposes, High Purity Asbestos, Asbestos "T", and titanium dioxide (King City Japanese) were also included in this program. All fillers were added dry in the open form to the pulp furnish in a Hermann Standard Disintegrator. Sufficient alum was also added at this point to obtain a pH of about 4.8-5.0 in the sheet mold. The handsheets were conditioned and tested according to standard methods. These results, plus the analytical data, are presented in Tables I and II.

From this cursory examination, it would appear that all samples are suitable as additives for paper. The only exception might be Sample 1684-25-5, which produced specks in the finished handsheets. As this could be a determining factor governing the use of these materials in paper, their pulpability characteristics were determined according to our standard method.

1. Pulpability

Disintegrating time (min.)	+65 Fraction (%) 2	4	6	8
25-1	*	*	*	*
25-2	0.026	0.004	*	*
25-3	0.058	0.032	0.074	0.032
25-4	0.756	0.456	0.128	*
25-5	9.294	7.012	5.458	4.625
High Purity	0.936	0.430	0.344	0.374

*Too low to measure

These results show that only Sample 25-5 is more difficult to repulp than our High Purity material. Based on this, it can be assumed that the other samples are acceptable in this respect.

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2. Filler retention

No attempt was made at this time to evaluate the self-retention properties of the various samples. However, based on the amount of filler retained in the handsheets, the indications are that the modified asbestos products are comparable to High Purity and Asbestos "T".

3. Bulk - Porosity - Smoothness

Basis weight and caliper determinations tend to indicate that an increase in bulk is obtained at the higher addition levels. Sheffield porosity was lower in most cases where the handsheets contained the modified products, while Sheffield smoothness results showed the wire side of the sheet to be generally smoother. In no instance is there enough data available to make generalizations regarding these properties.

4. Optical Characteristics

The brightness and opacity characteristics of sheets containing the modified asbestos products are quite good and approximate those of sheets containing Asbestos "T". The calculated light scattering coefficients (s_p), as presented in Table III, show that surface treatment improves the optical characteristics considerably. It should be noted, however, that again there is not enough data available to predict the exact value for each material, and that the reported figures are only approximations. This is quite evident from the extremely low results obtained on Sample 1684-25-5, which are probably due to the poor filler distribution experienced with this sample. The values obtained for High Purity Asbestos, Asbestos "T", and titanium dioxide are in line with those obtained by other investigators.

CONCLUSIONS

The results obtained from this study are encouraging enough to prompt further work with these materials. The next program will duplicate in some respects the present work in order to ascertain whether or not these results are reproducible. Also to be included are several production variables such as the influence of slurry pH on filtration rates, and the effects of filtering variables on end product characteristics. The latter is believed to be important as there is some evidence available which indicates that these are important variables in the production of RG-244. Also to be considered are the self-retention characteristics of silicate and alumina silicate-treated asbestos and their effect on the retention of other paperloading materials.

Gordon L. Dickson

Gordon L. Dickson

GLD/bsn
Attachments

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ACL Doc 19284 · Page 9 of 20 asbestosclaims.law/library/19284/9

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ACL Doc 19284 · Page 10 of 20

TABLE I

Handsheet Preparation Data and Analytical Results

Set Number	% Filler Added	pH (Sheet Mold)	% Ash	% TiO_2	% Mg	% Filler
1	Control	4.8	0.22	-	0.01	-
2	5% High Purity Asbestos	4.9	2.70	-	0.76	3.04
3	10% " " "	4.8	5.07	-	1.36	5.44
4	15% " " "	4.8	7.39	-	1.91	7.64
5	5% Titanium Dioxide	4.8	1.17	1.18	-	1.18
6	10% " "	4.8	2.06	2.11	-	2.11
7	15% " "	4.8	2.67	2.70	-	2.70
8	5% Asbestos "T"	4.8	2.60	0.78	0.49	2.74
9	10% " "	4.9	5.65	2.05	1.07	6.33
10	15% " "	4.8	8.26	2.48	1.50	8.48
11	5% 1684-25-1	4.9	2.51	-	0.63	2.80
12	10% "	4.9	5.59	-	1.37	6.09
13	15% "	4.8	7.02	-	1.74	7.74
14	5% 1684-25-2	4.8	2.60	-	0.63	2.86
15	10% "	4.8	4.70	-	1.13	5.12
16	15% "	4.8	7.22	-	1.81	8.21
17	5% 1684-25-3	4.8	2.22	-	0.55	2.59
18	10% "	4.9	4.81	-	1.18	5.55
19	15% "	4.8	6.91	-	1.62	7.62
20	5% 1684-25-4	4.8	2.11	-	0.47	2.29
21	10% "	5.0	5.13	-	1.21	5.89
22	15% "	5.0	7.29	-	1.68	8.18
23	5% 1684-25-5	4.9	1.91	-	0.47	2.36
24	10% "	5.0	4.52	-	1.07	5.37
25	15% "	4.9	7.37	-	1.72	8.63
26	Control	4.8	0.09	-	0.01	-

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ACL Doc 19284 · Page 11 of 20

TABLE II

Handsheet Physical Test Data

Set Number	Basis Weight	Bulk	Porosity	Smoothness (Felt/Wire)	Brightness	Opacity	R_o	R_w
1	58.87	2.14	166	400/350	75.3	71.5	60.2	84.7
2	60.10	2.09	136	400/350	74.3	72.6	61.0	84.3
3	60.28	2.08	130	400/340	73.5	74.5	62.1	83.8
4	59.01	2.07	129	400/320	73.2	74.9	62.1	83.6
5	57.91	2.12	148	400/340	80.0	75.3	65.0	86.1
6	58.73	2.15	180	400/340	81.0	78.5	67.5	86.4
7	57.68	2.16	169	400/340	82.0	79.3	68.4	86.9
8	58.96	2.13	149	400/320	77.6	76.8	65.2	85.4
9	60.19	2.11	145	400/320	78.5	83.4	70.8	85.4
10	60.10	2.12	138	390/310	79.2	86.7	74.2	85.6
11	60.19	2.13	119	400/340	77.2	76.7	65.0	85.1
12	58.73	2.23	113	400/330	77.6	82.2	69.5	84.8
13	59.50	2.21	109	390/320	78.4	84.0	71.1	85.0
14	59.37	2.20	131	400/330	77.5	76.1	64.8	85.2
15	59.60	2.21	116	400/320	78.9	81.2	69.1	85.3
16	59.50	2.24	118	400/290	79.6	84.4	72.0	85.3
17	58.60	2.21	140	410/320	78.5	75.6	64.7	85.5
18	58.91	2.23	125	410/310	79.6	81.6	69.5	85.6
19	59.46	2.24	122	390/300	80.4	84.5	72.1	85.5
20	59.60	2.14	126	400/350	78.7	74.9	64.6	85.5
21	58.37	2.25	116	400/340	78.5	80.1	68.2	85.4
22	58.82	2.25	112	400/330	79.4	83.9	71.2	85.2
23	57.82	2.24	155	410/350	78.4	72.0	60.9	85.6
24	57.00	2.26	150	400/340	79.0	77.4	65.5	85.5
25	58.00	2.29	131	400/340	79.6	82.2	69.9	85.4
26	58.00	2.15	171	400/360	78.1	68.1	58.5	85.6

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ACL Doc 19284 · Page 12 of 20

TABLE III

Calculation of Optical Properties

Set Number	$C_{0.89}$	R_∞	s_W	s	k/s	k	k_p	s_p
1	71.1	0.80	1.65	280	0.02500	7.00	-	-
2	72.4	0.76	1.71	285	0.03789	2.57	84.2	395
3	74.1	0.79	1.83	304	0.02791	8.48	34.2	717
4	74.3	0.79	1.85	314	0.02791	8.76	30.1	720
5	75.5	0.83	2.01	347	0.01741	6.03	74.5	5932
6	78.1	0.85	2.22	378	0.01324	4.99	88.3	4929
7	78,7	0.85	2.35	407	0.01324	5.37	53.3	5000
8	76.3	0.81	2.06	349	0.02228	7.78	35.6	2810
9	82.9	0.83	2.72	452	0.01741	7.87	20.7	3002
10	86.7	0.84	3.13	521	0.01524	7.92	17.8	3125
11	76.4	0.80	2.07	344	0.02500	8.60	64.1	2571
12	82.0	0.82	2.57	438	0.01976	8.67	34.5	2874
13	83.6	0.83	2.82	474	0.01741	8.25	23.1	2791
14	76.1	0.81	2.00	337	0.02228	7.52	25.0	2273
15	81.0	0.83	2.52	423	0.01741	7.36	14.0	3066
16	84.4	0.83	2.95	496	0.01741	8.63	26.9	2911
17	75.7	0.82	1.99	340	0.01976	6.73	33.5	2587
18	81.2	0.83	2.56	434	0.01741	7.55	16.9	3063
19	84.3	0.83	2.95	496	0.01741	8.63	28.4	3110
20	75.6	0.82	1.98	332	0.01976	6.57	11.6	2533
21	79.9	0.82	2.38	408	0.01976	8.08	25.3	2445
22	83.6	0.83	2.83	481	0.01741	8.37	23.7	2738
23	71.1	0.81	1.66	287	0.02228	6.45	16.5	593
24	76.6	0.84	2.10	368	0.01524	5.59	19.2	1918
25	81.9	0.83	2.60	448	0.01741	7.80	16.2	2225
26	68.3	0.78	1.53	264	0.03103	8.18	-	-

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PROGRESS REPORT

SUBJECT		
	PROPERTIES OF ASBESTOS Job No. 845-70301	
WORK BY		PERIOD ENDING
	S. Chwastiak, A. M. Gonas, W. A. Beccue	December 31, 1966
DATA FILED IN		
	NB 1685, p. 67; NB 1684, pp. 25, 26	Page 1 of 6

SUMMARY

Alumina-silica-asbestos was prepared in the pilot plant. About 13 pounds of asbestos were treated with sodium silicate first, at a treatment level of 12.5 parts SiO_2 per 100 parts of asbestos. Aluminum acetate solution was added in stages, and large samples were taken after each addition. The final sample contained 7.35% Al_2O_3. Essentially, all of the aluminum added was recovered on the asbestos.

The silicate-treated asbestos surface was negatively charged above pH 3.5. A small addition of aluminum acetate made the surface even more negative in character, but further additions of aluminum acetate solutions resulted in products which had increasingly positively charged surfaces.

The various samples are being tested as paper additives by G. L. Dickson.

DISCUSSION

It was reported in the Progress Report dated July 15, 1966 that when silica-asbestos is treated with aluminum acetate, alumina is deposited on the surface. Electron microscope photographs were taken of the material from this test (1644-39). The silicate-treated asbestos is shown before treatment in Figure 1, and Figure 2 is a picture of the solid after the aluminum acetate treatment. The assay of this final product was 4.22% aluminum.

A large batch of aluminum acetate-treated material was made in the pilot plant. Twenty-five pounds of HPO asbestos in 200 gallons of water were opened further and then sized in a 30-mm. cyclone. The overflow was acidified with acetic acid and then further sized in a 10-mm. cyclone. Approximately 13 pounds of colloidal asbestos were obtained in 150 gallons of slurry. This suspension was treated with sodium silicate at a rate of 12.5 lb. of SiO_2 per 100 lb. of asbestos. Aluminum acetate was added in stages, and samples of suspension were taken after each addition. The samples were adjusted to pH 9 with sodium hydroxide before filtering in a small (1 ft. x 1 ft.) plate and frame filter press containing three plates, under approximately 40 psi. The remaining slurry at the end of the test was filtered in a large press under approximately 50 psi.

Both the dried filter cake and the filtrate from each sample were chemically analyzed. Rough mass balances were made from these data and the results are shown in Table I. It is seen that nearly all of the silica and all of the alumina were present in the filter cake. There is some error in the analyses or in the assumptions on which the calculations were based, since more alumina was found by assay of the solid than by calculation. However, the trend is clear; just about all of the aluminum acetate put into the suspension was abstracted by the silica-asbestos.

AUTHOR	DATE	WITNESSED BY	DATE
Kisoon Park/S. Chwastiak	1/6/67	J. E. Abrams	1/6/67

The various samples were opened dry in the Mikro-Pulverizer, and submitted to G. L. Dickson for testing as a paper additive. He will report on their effectiveness in paper later.

Portions of these samples were reslurried in water, and their surface properties were investigated by micro-electrophoresis. The data are shown in Figure 3. The silica-asbestos clearly is negatively charged above pH 3.5. For some unknown reason, adding a small amount of aluminum acetate causes the asbestos to become more negative. Further additions, however, cause the solid to become progressively more positively charged in nature. The electrophoretic data for asbestos and for the batch of treated asbestos prepared in the laboratory are shown for comparison.

AUTHOR	DATE	WITNESSED BY	DATE
Kieoon Park/S. Chwastiak	1/6/67	J. C. Edwards	1/6/67

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ACL Doc 19284 · Page 15 of 20

TABLE I

Aluminum Acetate Treatment of Silica-Asbestos in Pilot Plant, Run #25

Operation	Wt. Asbestos Lb. Add	Wt. Asbestos Lb. Cum.	Vol. Susp. Gal. Add	Vol. Susp. Gal. Cum.	pH	Sod. Silicate as SiO_2, lb. Add	Sod. Silicate as SiO_2, lb. Cum.	Al Acetate as Al_2O_3, lb. Add	Al Acetate as Al_2O_3, lb. Cum.	Chemical Analysis: Solution, g./l. Al_2O_3	Solution, g./l. MgO	Solution, g./l. SiO_2	Filter Cake, % Al_2O_3	Filter Cake, % MgO	Filter Cake, % SiO_2	Cal. Amount on Asb., Parts per 100 Parts Asb. Al_2O_3	Cal. Amount on Asb., Parts per 100 Parts Asb. SiO_2
Start, 10 mm. cyclone overflow		12.9		150	4.8												
Add 5.5 lb. sodium silicate solution (29.1% SiO_2)		12.9		150	9.1	1.60	1.60	none	none								
Take Sample 1684-25-1	-0.86	12.0	-10	140	9.1	-0.11	1.49	none	none	0.005	0.05	0.11	0.38	37.0	46.5	0	12.4
Add 8 l. Al acetate solution(1)		12.0	+ 2	142	7.1		1.49	+0.079	0.079								
Take Sample 1684-25-2	-0.68	11.3	- 8	136	7.1	-0.08	1.41	-0.004	0.075	0.005	0.02	0.11	1.30	36.3	45.4	0.665	12.5
Add 16 l. Al acetate		11.3	+ 4	140	6.2		1.41	+0.148	0.223								
Take Sample 1684-25-3	-0.81	10.5	-10	130	6.2	-0.10	1.31	-0.016	0.207	0.003	0.07	0.11	2.72	35.0	45.1	1.97	12.5
Add 24 l. Al acetate		10.5	+ 6	136	5.9		1.31	+0.238	0.445								
Take Sample 1684-25-4	-0.77	9.7	-10	126	5.9	-0.10	1.21	-0.033	0.412	0.007	0.05	0.09	5.25	33.8	43.4	4.25	12.5
Add 24 l. Al acetate		9.7	+ 6	132	5.7		1.21	+0.238	0.640								
Take Sample 1684-25-5	-0.73	9.0	-10	122	5.7	-0.09	1.12	-0.048	0.592	0.003	0.05	0.11	7.35	32.8	42.0	6.58	12.4
Add 2.6 l. of NaOH solution (4 M), then filter		9.0		122	9.6		1.12		0.592								

(1) Aluminum acetate solution contains 3 lb. salt in 20 gal. water

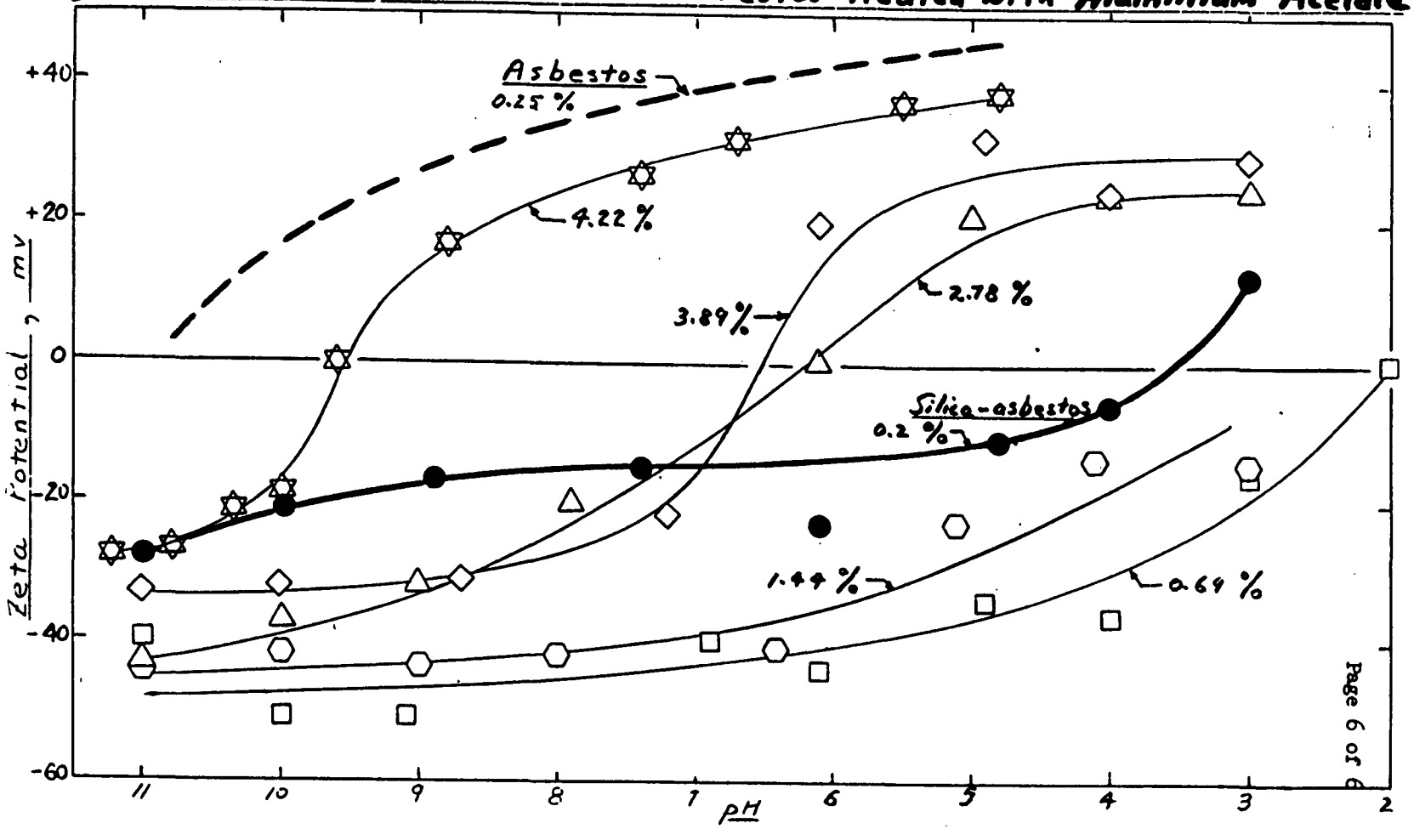
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Fig 3. Surface Properties of Silica-Asbestos Treated with Aluminium Acetate



Numbers on graph are Al assay of product

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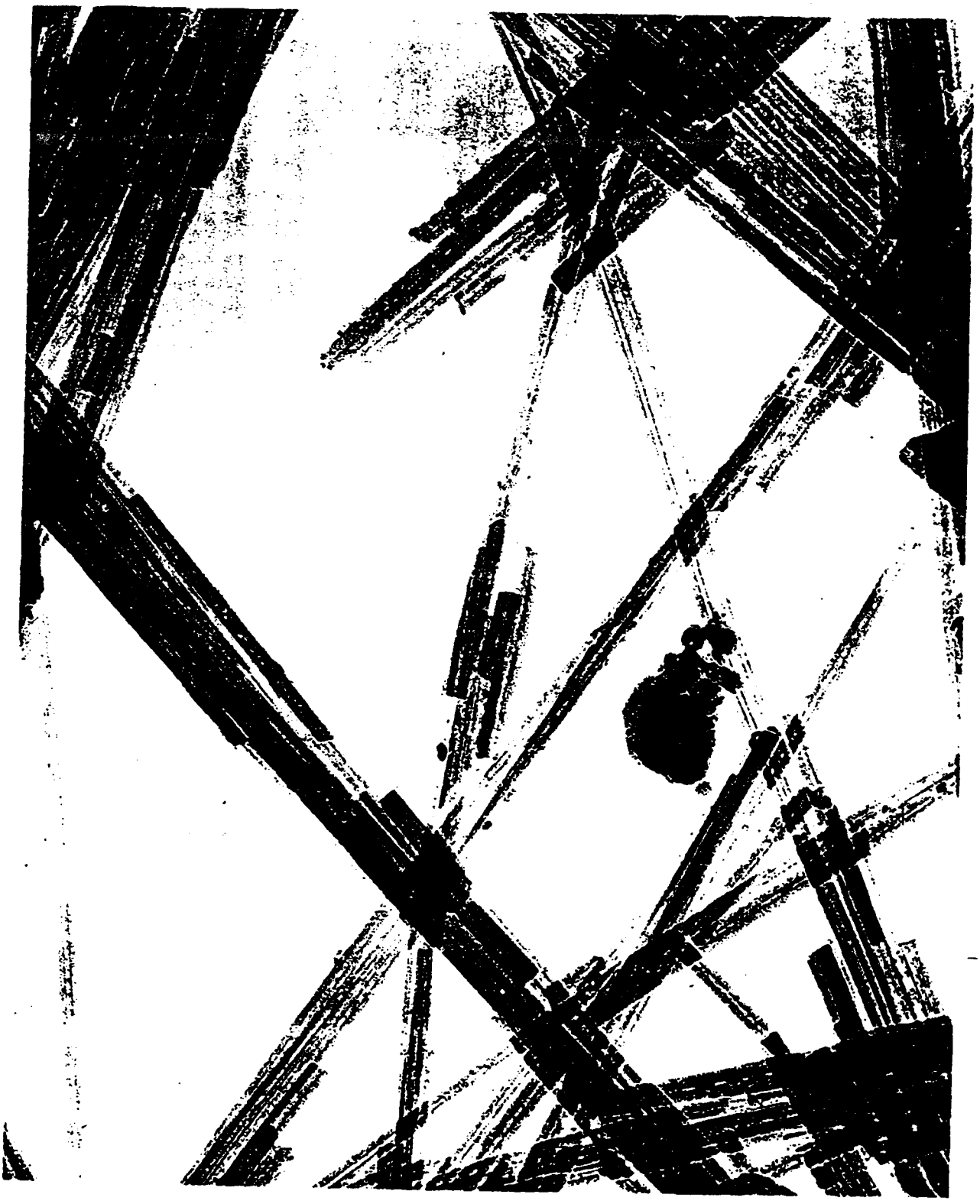


FIGURE 1 - Silicate-Treated Asbestos

Test No. 164h-71-1 - Photo No. 4605 - Magnification 105,000X

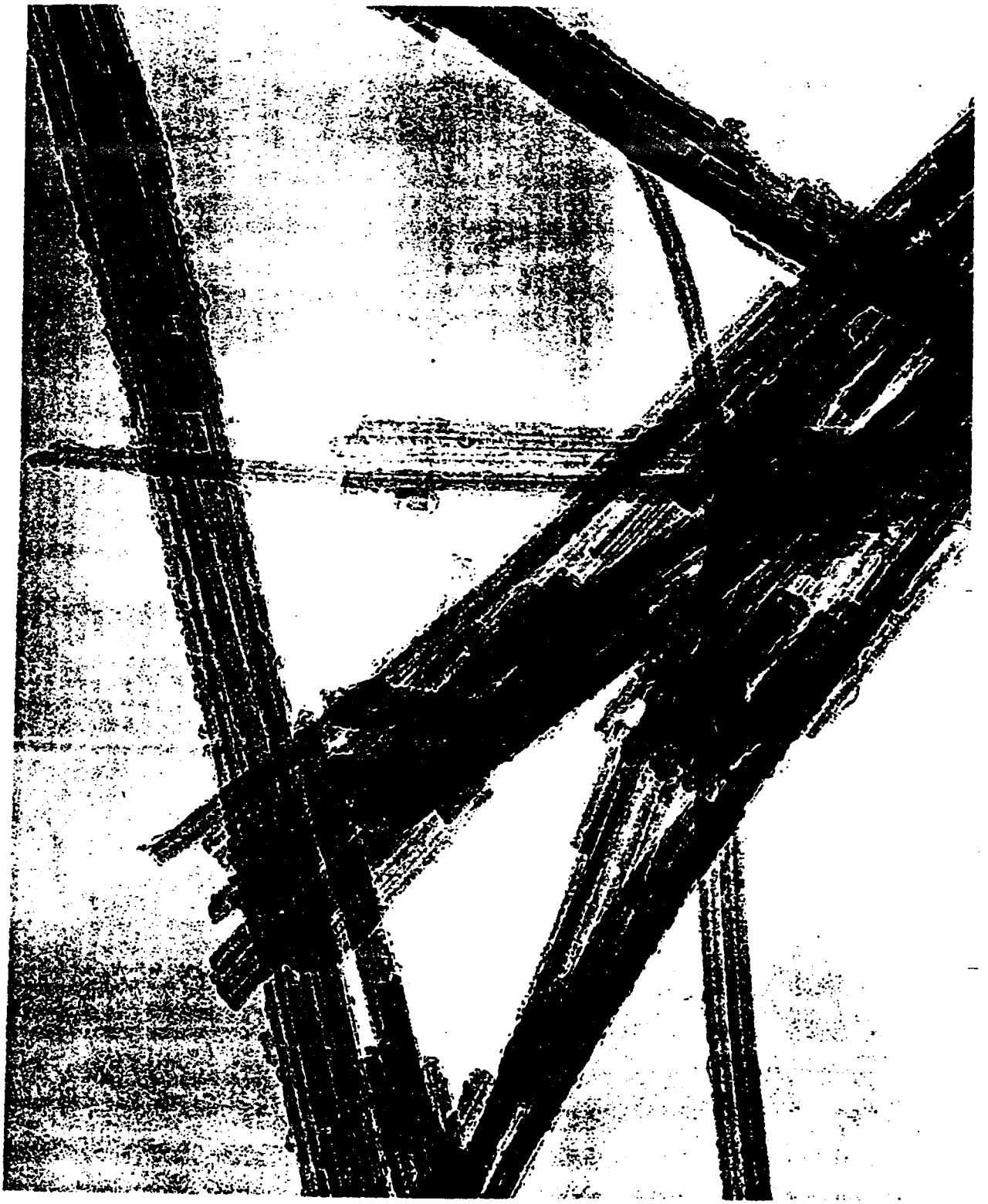


FIGURE 2 - Aluminum Acetate-Treated Silica-Asbestos
Test No. 1644-39-1 - Photo No. 4501 - Magnification 150,000X

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