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Date October 23, 1974
Originating Dept. "Calidria" Asbestos
Answering letter date
Subject Supervisbestos - A Review of
Product Quality Trends and
Competitive Market Position

Your copy of the report noted above is attached. Since the work was done the assessment of the competitive market position has been made obsolete by the introduction of a new, dustless Flosal. The review of quality trends and their possible relationship to changes in the nature of the ore being processed that is also covered are still relevant and important.

H. B. Rhodes
H. B. Rhodes

HBR:cjb
Attachment

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LOST IN CASE

SUPERVISBESTOS

A REVIEW OF PRODUCT QUALITY TRENDS AND COMPETITIVE MARKET POSITION

H. B. Rhodes

October 23, 1974

Union Carbide Corporation
Mining & Metals Division
Niagara Falls, New York

UCC 023482

SUMMARY AND CONCLUSIONS

Supervisbestos was introduced to the drilling industry through Montello in 1969 as a new premium-performance, premium-price mud additive. By 1971 it was a well established product with sales of 1671, 1170, and 1344 tons in 1971, 1972, and 1973, respectively.

In the last half of 1973 the principal competitor, Drilling Specialties Company, Inc., introduced a new version of their asbestos product, Flosal, that was alleged to be as good as Supervisbestos. Testing by Montello and reports from their customers' laboratories indicated that there was considerable merit to the claim. The performance gap formerly enjoyed by Supervisbestos seemed to have been closed in part by an improvement in Flosal but, more importantly, by a substantial drop in Supervisbestos performance. This study was undertaken to define more clearly what, if anything, had happened to Supervisbestos quality and the present competitive positions of Supervisbestos, Flosal, and Visbestos.

The performance of asbestos in well drilling depends on how strongly it is sheared as it is circulated with the mud. In this work, samples of Supervisbestos and Visbestos manufactured at different times are compared with Flosal and Atlas 1092 at shear conditions representative of those encountered in the field. Quality control data from both King City and Niagara Falls are used to fill in the overall picture.

In broad strokes, the results show that the Supervisbestos manufactured in 1971 and 1972 varied some from batch to batch but averaged about the same quality level throughout this time period. Using this 1971-72 period as a basis for comparison, there was a short time during the second quarter of 1973 where estimated field performance went up 10-15%. This was followed by a sharp drop of about 25% in August 1973 to an average level that was down a net of 10-15% from the 1971-72 base. This -10% to 15% condition prevailed through the last half of 1973 except for a 6-8 week period encompassing November where performance was down 25%. There was a moderate recovery in the first quarter of 1974 followed in June and July by a decline to near the low point (-25%) of November 1973. In late July, changes were made in the plant operation to give the product considerably more wet shearing. Product quality immediately returned to the 1971-1972 level.

The comparison between products showed that new Flosal had in fact been improved substantially. It was now about the same as the bottom quality Supervisbestos typified by the November 1973 and June-July 1974 product. Supervisbestos at this level was no longer a premium-performance product. Visbestos quality does not appear to have changed significantly over the time period studied. Atlas 1092 was the poorest product tested.

As a final point relative to product quality, it should be noted that a great majority of the product manufactured during this four-year period met the prescribed specifications. The problem has been that the specifications were set in 1971 at such a low level that product field performance could drop about 25% and still be within manufacturing limits.

This overall quality situation has been discussed in detail with our distributor, Montello. Their entire marketing position has been developed on the basis that Supervisbestos is a premium-performance material. They feel

that it is absolutely essential that product quality be returned to the 1971-1972 level if this can be done without manufacturing costs going right out of sight. Reasonable incremental costs will have to be passed on to the user. King City will provide an estimate of these costs when sufficient operating experience has been obtained at the new condition.

During the course of this study, it became apparent that there was a good correlation between overall plant recovery from the ore and the AEC quality control test Yield Point data. This is surprising since recovery depends on the rock-fiber circuit and the yield point depends on the wet opening provided by the Bauer and M-30 mills. This suggests that both the quantity of fiber in the ore and the ease of fiber liberation have changed significantly over the last several years. This appears to be the major cause of the trends in product quality observed.

RECOMMENDATIONS

This study leads to the following recommendations for future action:

1. The King City plant continues the manufacture of Super Standard Grade products using the specification of a maximum of 20% larger than 200 mesh in the M-30 discharge until the economics of this operation are established and a business decision is made.
2. The nature of the future ore supply should be investigated with regard to recovery and the potential effect on product quality.
3. The AEC Test #2 should be reviewed to establish a common procedure for both laboratories and to determine whether the accuracy can be improved. (No changes should be made at either laboratory until this is done.)

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INTRODUCTION

Union Carbide contracted with Montello on January 1, 1969 to distribute "Calidria" asbestos products to the oil well drilling industry. Two products were selected: Visbestos (SG-144) to compete with Flosal sold by Drilling Specialties and Supervisbestos (SG-200 pellets) as a new premium-performance, premium-priced product. Montello was to put their major selling emphasis on Supervisbestos.

As the market introduction of pelletized Supervisbestos progressed, it became evident that many of the drilling rigs could not open the pellets. Supervisbestos was then changed to a coarse ground material (attrition milled) at a packaged density of ~20 lb./ft.³. A smaller, one-eighth inch pellet version was later introduced in mid 1972, however, as a partial answer to meeting the OSHA regulations. Even these smaller pellets were difficult to open in certain common mud systems and so far have had limited market success.

In July of 1971, the Supervisbestos sales received a large boost in the form of a 600-ton spot order from the Atomic Energy Commission for use in drilling the pot-shot holes at the Amschitka atomic blast. Two specification tests were developed at that time to meet government requirements for product definition. Both were keyed to the low-shear, quick-opening characteristics of Supervisbestos. Product specification numbers for the government contract were selected which were high enough to exclude any other known asbestos from qualifying but were substantially lower than typical values for the Supervisbestos being manufactured at that time. The same specifications have been continued to the present time although very little of the product has been sold to the government.

About mid 1973, Drilling Specialties came out with a new version of Flosal that was alleged to be equal to Supervisbestos but sold at a considerably lower price. Atlas also had a new product. Considerable testing in the various mud laboratories resulted and, although the feedback was not unanimous, there were a substantial number of cases where Flosal looked as good as Supervisbestos. More important, however, there were a number of comments that Supervisbestos seemed to have dropped in quality and no longer provided the outstanding yield properties that it had shown previously. In view of this, the present study was undertaken to find answers to the following questions:

1. Have the properties of Supervisbestos changed from those it possessed in 1971?
2. What is the present competitive position between Supervisbestos, Flosal, Atlas 1092, and Visbestos?
3. Have the properties of Visbestos changed?

Two types of data have been assembled to answer these questions. The performance of asbestos in drilling a well depends on how strongly it is sheared as it is circulated in the mud. In the first part of this work, a series of samples of Supervisbestos and Visbestos manufactured at different times are compared with Flosal and Atlas #1092 at shear conditions ranging from 2-40 minutes, medium speed, in a Hamilton Beach mixer. Times of 10-20 minutes at these conditions are considered by industry to be representative of the kind of shear exposure the material receives in a well. The results thus show a comparison of products at a reasonable approximation of field performance conditions.

Supervisbestos is promoted for use alone and as a 50% by weight synergistic mixture with high-yield bentonite. Both types of systems were covered, therefore, in the laboratory mixing studies.

The second part of the work summarizes a variety of quality control test data by both King City and Niagara Falls going back as far as 1969. Results for the AEC Test #2, 2g./liter wet bulk, Available Fiber, and Ultimate Fiber are presented.

APPARATUS AND PROCEDURE

All mixing in the experimental portion of this study was done in a Hamilton Beach Model 936 single mixer with both agitators in place. Viscosities were measured at 600 and 300 rpm with a Model 35 Fann V.G. meter. The viscometer had been repaired and checked by the manufacturer at the start of the study.

All weighings were made with a Mettler balance with a scale reading to 0.1 g.

The test procedure was quite simple. A sample of the asbestos or asbestos-bentonite blend was weighed out, dry mixed in a covered beaker where appropriate, and added to 350 ml. of water in the mixer cup. The mixer was run at medium speed for the prescribed time. Times ranged, at set intervals, from 2 to 40 minutes. The mixer was shut off at the end of the mix and the temperature of the slurry was recorded. The slurry was poured into the Fann cup and viscosities were measured immediately. Three successive Fann 600-300 readings were taken in each case as soon as the needle stopped oscillating. Where the readings drifted, the initial reading was used to calculate properties. In each case, the 40-minute mix sample was also cooled to 25°C in one hour and the viscosities were checked.

Note that a separate sample was run for each time interval. This more time consuming procedure was necessary because handling losses each the viscosity was measured would have substantially depleted the sample as the test progressed.

All tests were run with distilled water. The synergistic systems were composed of 5g. of asbestos and 5g. of bentonite in 350 ml. of distilled water to give a total blend at 10 lbs./bbl. The tests where asbestos was used alone were run at 5g. per 350 ml. of water, i.e., at 5 lbs./bbl. No testing was done with such things as salt systems, fluid loss control additives, or with drilling solids added to the mud. These variables are important in the comparison of the field performance of the various products but were beyond the scope of the present study.

MATERIALS

The monthly composite samples provided by King City were used for the Visbestos and Supervisbestos mixing tests. They are identified by date of manufacture. The particular Supervisbestos samples tested were chosen to cover both a wide range of manufacturing dates and to typify well defined periods of quality level as identified by other quality control data. Wherever product was available, the Visbestos samples were selected from the same months as the Supervisbestos used.

The Flosal and Atlas products were purchased from field warehouses by Montello. They are identified by date. It is assumed, but not proven, that they represent typical production.

COMPARISON OF ASBESTOS PHYSICAL PROPERTIES

When the new Flosal first appeared in mid 1973, the standard UCC physical properties tests were run on a number of "Calidria" and competitive samples. These results, together with some comparable data obtained earlier on "old" Flosal and "old" Atlas (AZ-20), are summarized in Table I.

The most significant tests in the group are the flocculated and dispersed wet screening and the 2g./liter wet bulk measurement. The screening data show that Supervisbestos is slightly finer than Visbestos. The older version of Flosal was very coarse but the new version is now as fine or finer than Supervisbestos. The three products thus have approximately the same size distribution now.

The old Atlas product (AZ-20) had a flocculated wet screen analyses that was quite coarse and comparable with old Flosal. The addition of dispersant, however, showed a very large change in size distribution and the range became quite similar to the two UCC products. The new Atlas product #1092 is moderately finer but still quite coarse when flocculated, but is the finest material of the four by a good margin when measured when dispersed.

The 2g./liter wet bulk test gives the more clear-cut differences shown below:

<u>Product</u>	<u>2g./l. Wet Bulk (cc.)</u>
Supervisbestos	520-820
Visbestos	320-465
Old Flosal	195-200
New Flosal	280
Old Atlas (AZ-20)	185
New Atlas #1092	245

It was evident, therefore, that both the Atlas and Flosal products had in fact been changed considerably by finer grinding. Although substantial differences in physical properties between these and the UCC products still remained, testing under end use conditions was obviously necessary.

MIXING STUDY - DISCUSSION OF RESULTS

Introduction

This section of the report presents and discusses the data obtained to evaluate the field performance of the various asbestos products. A complete tabulation of data is listed in the series of tables at the end of the report. Each test sequence has been assigned a Data Reference Number which runs consecutively through the Tables. The effects of selected variables have been presented graphically in the Figures. All data used in each Figure has been identified by Data Reference Number so that the complete information can be located easily in the appropriate Table.

Supervisbestos - Bentonite Synergistic Blends

The data on the effect of mixing time on the various rheological properties of the Supervisbestos-bentonite blends are listed in Table II and are shown graphically in Figures 1 and 2. Each product tested is identified by the date of manufacture and the corresponding AEC Quality Control Test Yield Point as obtained at the Niagara Falls laboratory.

Figure 1 gives the Fann θ_{600} and θ_{300} data. These are direct readings and tend to give smooth, internally consistent curves. All of the products tested show a steady increase in Fann θ with mixing time with a gradual flattening out at the longer times. There is some scatter at times shorter than about ten minutes but thereafter the curves for the various samples run fairly well parallel. There is no tendency for the curves to converge as the shearing time increases. The shear intensity supplied by this mixer does not overcome differences in the Supervisbestos product.

Figure 2 gives the corresponding Yield Point and Plastic Viscosity results. These properties are calculated from the θ readings as shown below. Since the calculation compounds the experimental variations in both θ readings, the data tend to be more scattered.

$$\text{Yield Point} = 2 \theta_{300} - \theta_{600}$$

$$\text{Plastic Viscosity} = \theta_{600} - \theta_{300}$$

The Yield Point data in the figure have the same main trends as the Fann θ 's with some added minor tendency to converge at 40 minutes. The Plastic Viscosities are all quite low to mixing times of about 20 minutes and then tend to increase quite sharply. The products with the higher AEC Test Yield Points generally have the larger increases.

Examination of both figures shows that the properties of Supervisbestos have varied upwards and downwards since February 1972 with no obvious trend with date of manufacture evident in the limited number of samples tested. There appears to be a definite correlation with AEC Test Yield Point, however. In order to show this more clearly, the Yield Point for each product at 20-minutes mixing has been cross-plotted against the corresponding AEC Test Yield Point with the result given in Figure 3.

Industrial mud laboratories typically test products using Hamilton Beach or Multimixers operated at low or medium speed from 10 to 20 minutes. This is considered to give a reasonable approximation of the type of shear the product will encounter in the well. The 20-minute mixing selected for the product comparison can thus be taken as representative of field conditions in a good, well-operated drilling job.

Referring now to Figure 3, it can be seen that there is an excellent linear relationship between the AEC Quality Control Test and the performance expected from the product in the field. The slope of the curve is very close to 1.75 so that the test is actually a very sensitive tool to evaluate product quality. This correlation makes it valid to use the extensive quality control data that have been collected over the past four years to trace product quality variations in detail. This will be done in the next section.

It is also instructive to use Figure 3 to illustrate in a general way the magnitude of changes in product quality that have occurred, the possible reason why more complaints have not been received, and the inadequacy of the present AEC Test Yield Point specification. It will be shown subsequently that the results for February 1972 are representative of average product quality during the extended period covering the last half of 1971 and all of 1972 when the major market push was made to introduce Supervisbestos. The range of product quality manufactured at various times is compared with this base in the table below:

<u>Product Identification</u>	<u>AEC Test #2</u>		<u>Estimated Field Performance</u>	
	<u>Y.P.</u>	<u>% of Base</u>	<u>Y.P.</u>	<u>% of Base</u>
Best Ever Produced (June 73)	35.0	130	85	116
<u>Base</u> (Feb. 72, Typical of 2nd half 71 and 72)	27.0	100	75	100
Late 73, Early 74	22.5	83	65	89
Worse Produced* - Nov. 73	13	48	49	75

* Note that this is just slightly below the present specification of 15.

It appears that the field performance of the product in synergistic systems made in late 73 and early 74 was down around 10-15% from the typical material of 1971 and 1972. The occasional lots were down about 25%. In mud systems, a 10% change is hard to be certain of while a 25% change can be expected to show so field complaints were limited.

The present manufacturing specifications for Supervisbestos require a minimum value of 15 for both the Yield Point and Apparent Viscosity. These values were set in 1971 at the time of the AEC order at the lowest level that would keep any other asbestos from qualifying and to avoid having any of our product being out of specification. Product made at that time typically had values of 25-26 for Yield Point. The specification was thus set at a level that did not represent current product but at one which allowed a drop of about 25% in the efficiency of the product in its end use.

Supervisbestos (Only)

The mixing data for systems with 5 lb./bbl. of Supervisbestos only are summarized in Table III with the Fann θ values presented graphically in Figure 4. The θ_{600} data scatters without any clear pattern for the first five minutes and then generally "bunch" across the rest of the mixing. The θ_{300} data scatters out to about 20 minutes then bunches up. The November 1973 sample (AEC Yield Point 13) seems to be in the bottom of each band but it is doubtful if the difference could be proven statistically significant. Yield Point and Plastic Viscosity data showed the same kind of random band so results are not shown graphically.

Visbestos - Bentonite Synergistic Blends

The data on the effect of mixing time on the properties of Visbestos-bentonite blends are listed in Table IV and in Figures 5 and 6. Figure 5, the Fann θ data, shows April 1972, October 1973, and January 1974 grouped in a narrow band. The June 1973 data, where Supervisbestos also exhibited its highest properties, is well above the rest of the group. The March 1974 sample

was an experimental product ground in the attrition mill to approximate the density of Supervisbestos. In this synergistic system, it falls consistently below the usual Visbestos but by only a narrow margin.

The Yield Point curves in Figure 6 show the same pattern as the Fann θ 's. The Plastic Viscosities all group closely except that for the outstanding product made in June 1973 which increases substantially at mixing times greater than 20 minutes. With this exception, there does not appear to be any changes in the characteristics of Visbestos over the time period studied.

Visbestos (Only)

The data on the effect of mixing time on the properties of Visbestos only are listed in Table V. The Fann θ results are also shown graphically in Figure 7. In general, the results are very much like those for Supervisbestos alone (Figure 4) with considerable scatter but no clear-cut differences between products. The only exception here is the experimental coarse-ground material made in March 1974 which has considerably lower properties when used alone. This product was tested as a possible replacement for custom-packaged "Visbestos" for a large customer. It has since been abandoned.

Comparison of Various Asbestos Products - Synergistic Blends

The mixing data for old and new Flosal and Atlas 1092 both alone and in the synergistic 50/50 blend are summarized in Table VI. Appropriate portions of these data have been combined with those presented previously for the "Calidria" products to give the comparison for synergistic blends shown in Figures 8 and 9.

Referring to Figure 8, the Fann θ readings group quite well into three bands, i.e.,

1. Supervisbestos with AEC Test Yield Points of 22 and 28.
2. Supervisbestos with a Yield Point of 13, new Flosal, and Visbestos.
3. Old Flosal and Atlas 1092.

The Yield Point data in Figure 9 show the same pattern. In the region of 15-20 minutes mixing that approximates commercial use, Supervisbestos with an AEC Test Yield Point of 22-28 still shows a substantial margin in performance over the competing products. The Supervisbestos with an AEC Yield Point of 13, however, is indistinguishable from Visbestos and new Flosal. It is no longer a premium product. Atlas 1092 and old Flosal are not really competitive in performance.

Comparison of Various Asbestos Products (Only)

The mixing data for the same series of products used alone (except Atlas 1092) are presented in Figures 10 and 11. As discussed previously, these systems show much more scatter than the synergistic systems so it is more difficult to arrive at clear-cut conclusions.

The Fann θ data, Figure 10, show the two better Supervisbestos products on the top. The poorer Supervisbestos (YP=13) is moderately below them. This product builds viscosity faster than new Flosal to about 10 minutes mixing time and then the two products are quite similar and only a little below the better grades of Supervisbestos. Visbestos and old Flosal fall below in that order. There is also a pronounced tendency for all of the products to converge at the 40-minute mixing time where the extended shearing has overcome some of the differences.

The Yield Point data are given in Figure 11. The two better Supervisbestos products demonstrate some advantage in quick yield but after 15-20 minutes they are only marginally above new Flosal. Poor Supervisbestos (YP=13) and Visbestos fall noticeably lower and old Flosal is at the bottom. All of the products except old Flosal scatter in a broad band for mixing times longer than 20 minutes. It is thus clear that Flosal, in applications where it is used alone, shows a substantial improvement in properties. It is clearly better than Visbestos and may be hard to distinguish from good quality Supervisbestos.

SUPERVISBESTOS QUALITY CONTROL DATA - DISCUSSION OF RESULTS

Introduction

A series of selected samples were used in the previous section to show that the field performance of Supervisbestos since 1971 has ranged from up 10-15% to down 25%. It was also shown that there was an excellent correlation between estimated field performance and the AEC #2 Quality Control test so that this test can be used as a valid measure of product quality.

The AEC test is run on each 60-pallet lot by King City and on each monthly composite by Niagara Falls. It thus provides a very detailed picture of month-by-month variations in product quality.

In this section, the AEC test results for the two laboratories are first compared. The data are then used to trace Supervisbestos quality variations from 1971 to the present. Three other product characterization tests, 2 g./l. Wet Bulk, Available Fiber, and Ultimate Fiber which are only run on Supervisbestos at Niagara Falls are used to supplement the picture.

Interlaboratory Comparison - AEC Test #2

This test was developed to capitalize on the unique properties of Supervisbestos to build Yield Point quickly at low shear. The 50/50 synergistic mixture with bentonite which emphasizes this property and which gives a much more stable and reproducible mud for viscosity measurements was selected.

As originally proposed by Montello, a mixture of 5g. of asbestos and 5g. of high-yield bentonite were dry blended and added to 350 ml. of distilled water in the cup of a specially modified Hamilton Beach mixer. After mixing for 4 minutes at low speed ($\sim 13,000$ rpm), the Fann Viscosity was measured immediately. Specifications are set at 15 minimum for both Yield Point and Apparent Viscosity.

The Hamilton Beach mixer is manufactured with a "butterfly" flapper at the bottom of the shaft plus a rigid disc about 1-1/2" up the shaft. The official version of the test requires that both of these agitators be removed and be replaced with a single serrated disc at the bottom of the shaft.

It has turned out that this precise requirement on mixer configuration was not followed by either the King City or Niagara Falls laboratories. King City used a Multimixer with three solid discs on the shaft rotating at about 11,000 rp. Niagara Falls used the Hamilton Beach as supplied by the manufacturer. The Multimixer will shear the system somewhat less than prescribed mixing and the unmodified Hamilton Beach somewhat more. The consequences of this difference with a product having a Yield Point in the low 20's is illustrated in the following table. Duplicate determinations made on the same lot of product are shown.

Lot No.	Date	Lab.	Fann Data					Yield Point Difference NF-KC
			600	300	AV	PV	YP	
822-(6-60)	4/17/74	K.C.	35	28	17.5	7	21	+3
		N.F.	28	26	14	2	24	
846-(1-60)	4/18/74	K.C.	30	25	15	5	20	+6
		N.F.	32	29	16	3	26	
847-(1-25)	4/18/74	K.C.	33	27	16.5	6	21	+5
		N.F.	32	29	16	3	26	
847-(26-60)	5/28/74	K.C.	28.5	23.0	14.3	5.5	17.5	-0.5
		N.F.	33	30	27	3	17	

(1) N.B. Reference 2019-38

The Niagara Falls results with the higher shear tend to fall 3-6 points higher, although there is one case where this does not happen. It would also be expected that this spread will decrease for lower Yield Point products and increase for those with higher Yield Points.

A summary of all of the AEC test results from both laboratories from 1971 through August of 1974 is given in Table VII. The King City data are listed by batch number and an average for each month is also included. The Niagara Falls results are for the monthly composite supplied from King City. The data table has also been divided according to Mill Pile number for use in subsequent discussions of this variable.

The results for the monthly average by King City and the monthly composite by Niagara Falls are compared in Figure 12 coded by year. Considering the conditions under which the test is run, the agreement is surprisingly good. The results scatter on both sides of the "exact agreement" line with Niagara Falls higher 12 times and King City higher 10 times. The Niagara Falls results tend to run higher with the better products as would be expected from the duplicate comparison by lot just shown. There is no obvious bias by the year the test was run although the 1974 data have less scatter than previous years. There is only one case where the difference was more than 6. Overall, the test is working well but there is room for improvement.

Quality History

AEC Test No. 2: The individual lot data from Table VII is shown as a function of the date of product manufacture in Figure 13. The lot data points are connected by solid lines to show trends more clearly. Granular

Supervisbestos data are also shown and are included in the line connecting the points. Niagara Falls data for the monthly composites are given as individual, unconnected points at the middle of each month. The mill ore pile number and the recovery from each pile are shown along the bottom of the graph. The average Yield Point and Apparent Viscosity for the time period covered by each ore pile is also included for future reference.

The AEC test was not used until July of 1971 so only Niagara Falls data run recently on retain samples are available for 1970. In 1970, the Supervisbestos averaged a Yield Point of 35 and an Apparent Viscosity of 21. If a Yield Point correction of 6 is assumed to correct to the King City version of the procedure, the Yield Point will be around 29.

In 1971, the majority of the Supervisbestos was produced in July, August, September, and October to fill the AEC order. Average properties in this time period were Yield Point = 26.2 and Apparent Viscosity = 16.2. This was the general quality level at which the product had been introduced widely into the industry as a premium performance material. It is obviously well above the 15/15 values written into the AEC contract specification at that time.

Throughout 1972 and the first quarter of 1973 the product quality fluctuated somewhat but continued to average a very respectable 25.8/15.9 even when the low values for January, February, and March are included. In April, May, June, and July of 1973 product quality jumped sharply to an average of 33/19.6, and then the bottom dropped out. The last 5 months of 1973 averaged only 19.3/13.2 with November an all-time low of 14.4/10.9. It was in this time period and in early 1974 that questions and field complaints about the product being different began to come in.

Things seemed to improve some in February, March, and April 1974 but went downhill again in May and June. During this time period, a number of discussions were held with the plant as to what caused the problem and what needed to be done to get the Yield Point back around 25 as it had been over much of the life of the product. Review of wet screen analysis of the finished product showed no systematic changes so the evidence pointed to inadequate wet shearing.

For several years the plant has operated for super standard grade production with a process control point of a maximum of 25% + 200 mesh in the M-30 mill discharge. Review of the records showed that the 25% point was probably held in the average but was quite frequently exceeded for considerable periods. On June 6, 1974, a program was instituted to hold the 200 mesh consistently below 25%. This initially appeared to help but quality dropped again in the latter part of the month and through most of July so that a considerable portion of the product made was below specification. On July 18, 1974 the control point was lowered to 20% maximum. Product quality jumped immediately to a Yield Point of 25 or better and seems to be holding very well. It is intended that the plant continue to operate in this way to verify that consistent product can be made and to obtain sufficient data to establish the increase in cost.

Wet Bulk Test (2 grams/liter): In this test, 2 g. of asbestos are sheared for 10 seconds at high speed in a Waring Blender or Osterizer. The slurry is diluted to 1000 ml. in a graduated cylinder and allowed to stand for one hour. The settled bulk is reported in cubic centimeters. The results, even on the same sample, tend to be erratic so the test is normally run in duplicate and averaged.

Data obtained on the Niagara Falls composites are shown in Figure 14A. In 1970 and 1971 the data are fairly constant with some indication of a downward trend. In 1972 the results drop sharply in the first quarter and level out around 700 cc. 1973 shows the initial increase and then the sharp drop over the second half of the year and on into the first four months of 1974. There is some but not spectacular recovery after the increase in wet grinding in mid July. The correspondence between these test results and those for the AEC Test #2 shown previously in Figure 13 are not exact but the main trends are certainly quite similar.

Available and Ultimate Fiber Tests: These tests were developed to give a measure of the amount of fiber the user obtains when our product is mixed at low and at very high shear. In the Available Fiber test the slurry is mixed for 4 minutes at 2% solids in a Hermann Disintegrator. This level of shear is reputed to reopen asbestos "snowballs" but not to generate any new, liberated fiber. A measured aliquot of the mixed slurry is dispersed with Tergitol Anionic-7 and allowed to stand in a graduate for a 15-minute period. The non-dispersible fiber bundles settle. A measured portion of the slurry is then removed, flocculated, dried, and weighed to give the proportion of dispersible fiber. The Ultimate Fiber test uses basically the same procedure but the shearing is done for 5 minutes at high speed in a Waring Blender. These tests are always run in duplicate at Niagara Falls and the results are reported as the average. The difference between duplicate determinations is usually less than 5%.

The Ultimate Fiber data (top curve, Figure 14B) scatters somewhat but on the average holds fairly constant at 82-83% for 1970 through 1972. There is the usual sharp increase in the second quarter of 1973 followed by the sharp drop and lowest values in the last half of 1973. 1974 continues to show a downtrend into the upper 70% level. The current product contains around 5% more "rock" and other nonfibrous material than it did in 1971 and 1972. This property is related mainly to the operation of the rock-fiber circuit and would not be influenced by changes in the wet shearing in the Bauer and M-30 mills.

The Available Fiber data (lower curve) also scatter some but show a steady, clean-cut trend downward. It is worth noting that the change from the second half of 1971 to late 1973 is approximately 49/58 or 84%, a drop of 16%. This is very close to the ~15% change shown in the mixing studies for the estimated change in field performance of Supervisbestos over the same time period.

The very low shear used in the Available Fiber test makes it less sensitive than the AEC Test #2 and it does not reflect some of the up and down variations shown in Figure 13. It should be noted, however, that if the very high data for the second quarter of 1973 are omitted, the Yield Point and the Apparent Viscosity show the same gradual downward trend as the Available Fiber data. This is particularly true of the Niagara Falls composite samples.

Effect of Mill Ore Pile

As mentioned previously, Figure 13 showed the time periods over which the various mill ore piles were used and corresponding King City AEC Test #2 results (average) for the product manufactured during the corresponding time periods (Table VII). These average data are assembled for convenience in Table VIII and are shown graphically in Figure 15.

Considering the batch-to-batch fluctuations that went into each average the degree of correlation is quite remarkable. This is particularly true since the AEC test is run on finished product while the correlating variable "Recovery for Ore" is dependent on the operation of the rock-fiber circuit.

In the rock-fiber circuit, the ore is subjected to one stage of shearing in the fiber mill in combination with a series of separations. In going to Super Standard Grade, the slurry receives additional shearing in the Bauer and M-30 mills but gets no further beneficiation or refining. According to plant records, except for the adjustment in June and July of 1974, there have been no basic changes in the operation of either circuit over the entire period covered by these data. The correlation in Figure 15 together with the other data presented in this section thus suggests that the ore has changed in two important ways:

1. It contains more "rock" and less fiber.
2. The fiber it contains requires more wet shearing to arrives at the same degree of liberation.

There have obviously been some wide day-to-day fluctuations in the mill operation and some of the observed trends may be in part due to a gradual increase in throughput over the years. It seems clear, however, that a major contribution has been made by changes in the quality of the ore being processed. Since this effect is felt across the entire product line, and since recovery from the ore has important economic effects, it is suggested that it may be appropriate to investigate this problem further on a broader base.

SUMMARY, CONCLUSIONS, AND RECOMMENDATIONS

These sections have been placed at the beginning of the report. The reader is referred to that location.

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