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SEVENTH PROGRESS REPORT

ON

THE FUNDAMENTAL STUDY OF H-LO

TO

OWENS ILLINOIS GLASS COMPANY

C-57324

901

ROLL	81
CODE	67

November 30, 1944.

P 30

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Arthur D. Little, Inc.

CHEMISTS-ENGINEERS



CAMBRIDGE, MASSACHUSETTS

MR:GD

November 30, 1944.

Owens Illinois Glass Company
Toledo
Ohio

Attention: Mr. U. P. Brown

Gentlemen: C-57331

We present herewith a Seventh Progress Report on The Fundamental Study of E-10 and a proposed new suspending agent.

Recent tests indicate that 1.5 to 2.0% of either sulfate wood pulp or of newspaper in the form of slurry is superior to the 5 to 6% of beaten asbestos previously used as a suspending agent. This small amount of organic matter does not smoke when the E-10 is heated up to 1800°F. The use of woodpulp in place of asbestos represents a considerable saving in the cost of raw materials and also makes a better product.

The use of woodpulp as a suspending medium also opens up the possibility of correlations between chemical analyses and physical tests. When asbestos is used in the making of E-10 the chemical analysis of the product becomes so complicated that it is very time

consuming and the interpretation of results so difficult and questionable that we have not considered this method of investigation worth the time involved. Most of our previous chemical analyses were made on very small batches where the settling during induration was very slight.

We now have a procedure by which we can make blocks large enough for any desired physical tests, but containing no asbestos and therefore suitable for chemical analyses.

We are also including in this report data that has been collected in our study of induration time, and many other variables concerned with the making of E-10. Not enough information is as yet available on the making of E-10 with woodpulp to determine whether these variables as fixed for E-10 made with beaten asbestos, will also hold for E-10 made with paper pulp.

Respectfully submitted,

Arthur D. Little, Jr.

INTRODUCTION

Our work on E-LO for nearly a year was concerned with the making of batches by varying formulas, varying raw materials and varying time and temperature of induration, and evaluation of the extent of reaction by chemical analysis of the product. The past six months has been devoted to making larger batches and evaluation of various formulas, times and temperatures by physical tests on the product.

Although the investigations by chemical analysis gave information that greatly assisted in planning of formulas for larger batches which as noted in our sixth progress report are greatly superior in strength to any similar previous product, we have never been able to correlate our chemical analyses with quantitative tests of strength and heat conductivity. The difficulty that prevented such correlation was the finding of a proper suspending medium. When asbestos is used for this purpose the complexity of the chemical analyses made the results worthless for determining the extent of the reaction or calculating types of hydrate formed. Blocks large enough for physical tests had not been successfully produced without asbestos until September of this year.

The ideal suspending medium is one that will hold the lime and silica in uniform suspension until the reaction has progressed far enough to prevent settling and yet not interfere with the lime to silica reaction. Precooking should accomplish the desired result but is very difficult to control. Too little precooking will permit settling, making the block too heavy at bottom and too light and weak at top. If precooking is continued too long the mix will be too thick to pour uniformly producing planes of cleavage and the breaking up of structures already formed will prevent the development of maximum strength. Whether there is a happy medium between these two extremes was not determined in the ten batches 890-99 which we made with precooking. A relatively long research would be necessary to clear up this point and it is doubtful whether such a procedure would be commercially useful even if possible.

Another method that was investigated was the use of emulsifiers. These agents increase the volume by inclusion of air and permit the use of a much smaller volume of water which would represent a considerable saving in the drying operation. Here again the problem of control is very difficult. The weight of the final product could not easily be held at the desired amount

and in many cases the top of the block was lighter than the bottom because of larger air pores. Some blocks were made with reasonably uniform pore structure but they were either too heavy or too light. This line of investigation was discontinued when it was found that the emulsifiers or suspending agents used were incompatible with asbestos. Further work along this line might be justified if the saving in evaporation of $1/3$ to $1/2$ of the water would justify the extra cost of more rigid control and possible greater number of rejects.

Beaten asbestos was the medium chosen for our development of K-L0 in blocks large enough for physical test of strength and heat conductivity. Formulas and methods of mixing have been developed that produce a superior grade of K-L0 as detailed in our sixth report and continued in this report.

However within the last two months a new suspending agent, paper pulp, has been proven to be fully equal to beaten asbestos and $1\ 1/2$ to 2% is as effective as is 5 to 6% of beaten asbestos. The use of paper pulp would represent a considerable saving in cost of raw materials and has no disadvantages that we can foresee. In addition it opens up the possibility of making blocks of microporite large enough for physical tests but containing no asbestos and thus making practical a

correlation of chemical analyses with physical tests. This line of investigation should increase our knowledge of the types of hydrates that are present in the strongest products and possibly permit the setting up of standards whereby chemical analysis would show whether the product was over or underinsulated.

New Suspending Medium-Paper Pulp Slurry

Sulfate Pulp - The first batches made with pulp slurry were C112, C113 (Aug. 16-17). Sulfate pulp was beaten at 1% consistency for 15 hours. The freeness was then 37. Batches C112, C113 were made up using amounts of slurry equivalent to 5% dry pulp on total solids of the mix. The mixes were too thick to pour uniformly and 12 1/2% extra water was added. Pressure on the autoclave was released in the usual manner but C113 had exploded so that over half of the batch had been blown out of the pan. Batch 112 was not noticeably cracked but may have been weakened as the modulus was very low. It is believed that the consistency of the mix was so high that the top of the block held in the pressure until the pressure differential was great enough to tear the block open with explosive effect.

Batches C114, 115, and 116 were made with 2.9%, 2.1% and 1.4% pulp and C117, 118, and 119 were made with the same percentages of pulp but the pulp slurry was neutralized with lime during the beating. All of these batches exploded when the pressure was released except C119. C119 was 10.6 lbs. per cu. ft. and had a modulus of 80.1 air dry and 105.5 when tested immediately after 24 hours drying at 110°C.

D

Batches C126, 127, 128 (Sept. 9) made with 2.9%, 2.1% and 1.4% pulp were 10.2 to 10.5 lbs. per cu. ft. and had moduli above 80 air dry and above 90 when redried. Thus it has been proved that 1.4% of sulfate pulp can be used to replace the 5% to 6% beaten asbestos previously used.

Newsprint Pulp - Since chemical pulp (sulfate pulp) had been so successful as a suspending medium it was decided to try newsprint. A slurry was made up using 150 grams of shredded newspaper (Boston Post dated Sept. 11, 1944) to 15 liters of water. Considerable trouble was experienced with foaming and 25 grams of lime hydrate were added. After 3 hours beating and standing overnight the slurry had a freeness of 85 to 87. Batches C129 to C134 were made using from 5.0% to 1.4% of pulp. The newsprint pulp proved as satisfactory as the sulfate pulp although about 2% on weight of total solids is necessary. B133 containing 2.1% had considerably higher modulus than those containing higher or lower percentages of pulp. This is diagrammatically shown in Chart II where modulus is plotted against per cent of beaten pulp. B 119 with sulfate pulp and B 120, 121, and 122 with asbestos slurry are also shown for comparison.

We consider the use of wood pulp as a suspending medium as a very important development in this study of the making of K-10. We believe that more uniform slurry can be obtained since with asbestos of the grade used there is considerable variation in the amount of fiber and of non fibrous mineral matter in different lots.

Heat Tests - Microsporite made with 1.4% to 2.9% of wood pulp can be heated 24 hours at 1000°F. without any noticeable effect. Quantitative physical tests have not yet been made on heated samples. Test specimens 2" x 1" when heated red hot along one edge by use of gas burner and allowed to cool without any precautions as to slow cooling, did not crack or change shape. No smoke was noticed and only a slight grey color developed between the heated and unheated portions.

When heated in a muffle to 1800°F. the pieces curled, probably due to more rapid heating on side touching bottom of muffle, and shrank about 25% in all directions. The heated samples appeared stronger than before heating and showed no tendency to disintegrate or shatter, although cracks did open up on the long side of the curve. No intermediate temperatures have been tried.

Formula and Method used in mixing ingredients for H-10

Paper Pulp as Suspending Medium

Example:

	<u>Formula</u> <u>C119</u>	
Made 9-6-44	Indurated 10 hours	
Line Hydrate	330	47.1%
Celite	255	36.5
Clay	35	5.0
White Asbestos AM (not processed)	70	10.0
Sulfate Pulp in Slurry	10	1.4
	700	100.0
Total Water	3990	
Slurry 1% Sulfate (19 hrs.)	1000	
Extra Water	3000	
Water-Solids Ratio		5.70

Mixing Procedure

1. 2000 cc. slurry and 2000 cc. water poured into mixer.
2. Line added
3. Celite added
4. Clay added
5. Mixed until uniform (2 to 3 minutes)
6. Crude asbestos added slowly
7. Mixing stopped and blades cleaned
8. Mixed 15 minutes
9. Rest of water, 4000 cc., added gradually
10. Mixing stopped and blades cleaned
11. Mixed 15 minutes
12. Dumped into bucket
13. Stirred with paddle and poured in pans, 1 to 2 hrs. later
14. Autoclaved 10 hrs. at 150 lbs. steam pressure.
15. Dried at 110°C.

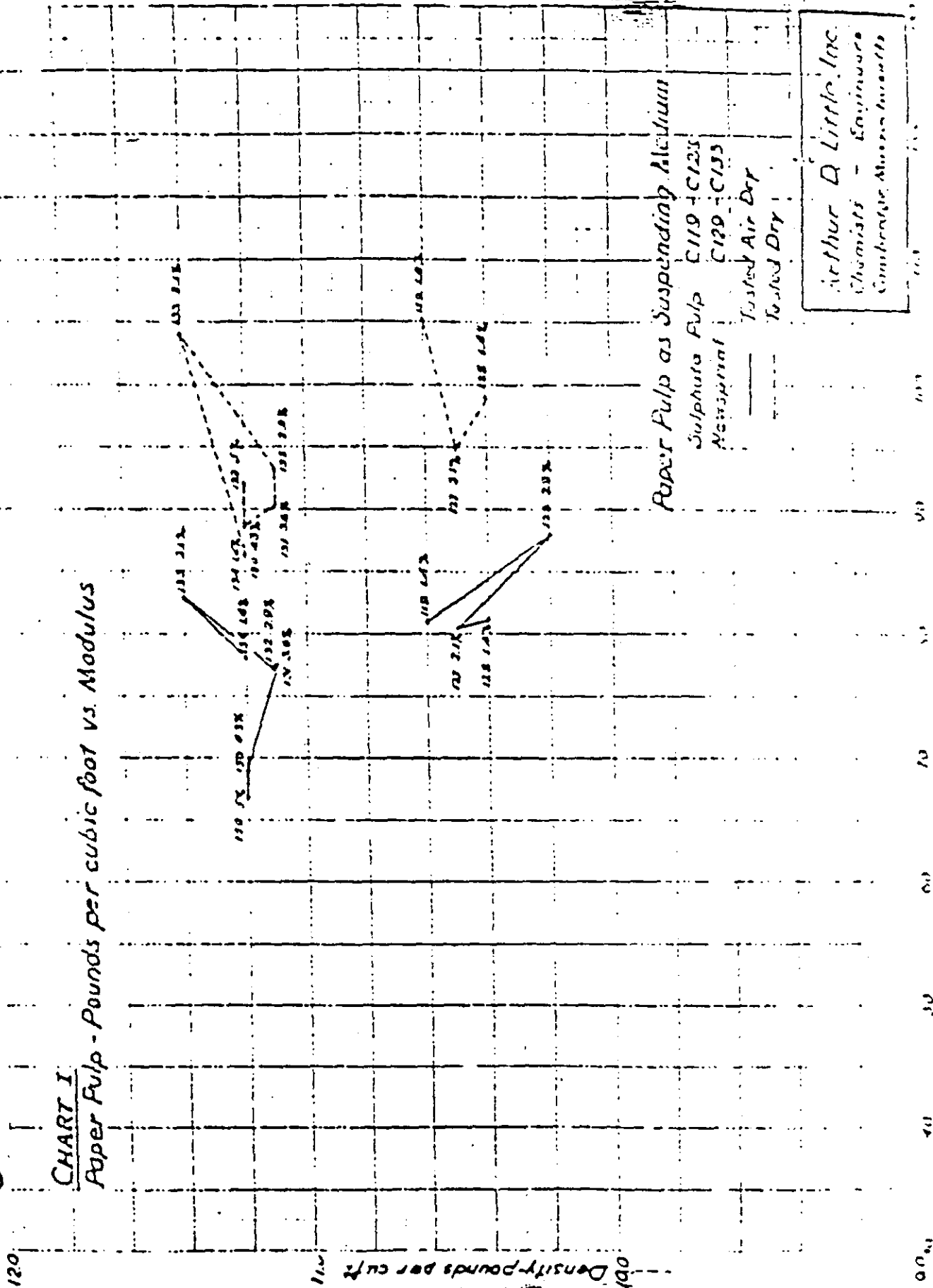
TABLE I

Paper Pulp as Suspending Medium

CaO:3102 Ratio 1:1 Induration 9 1/2 to 10 hours

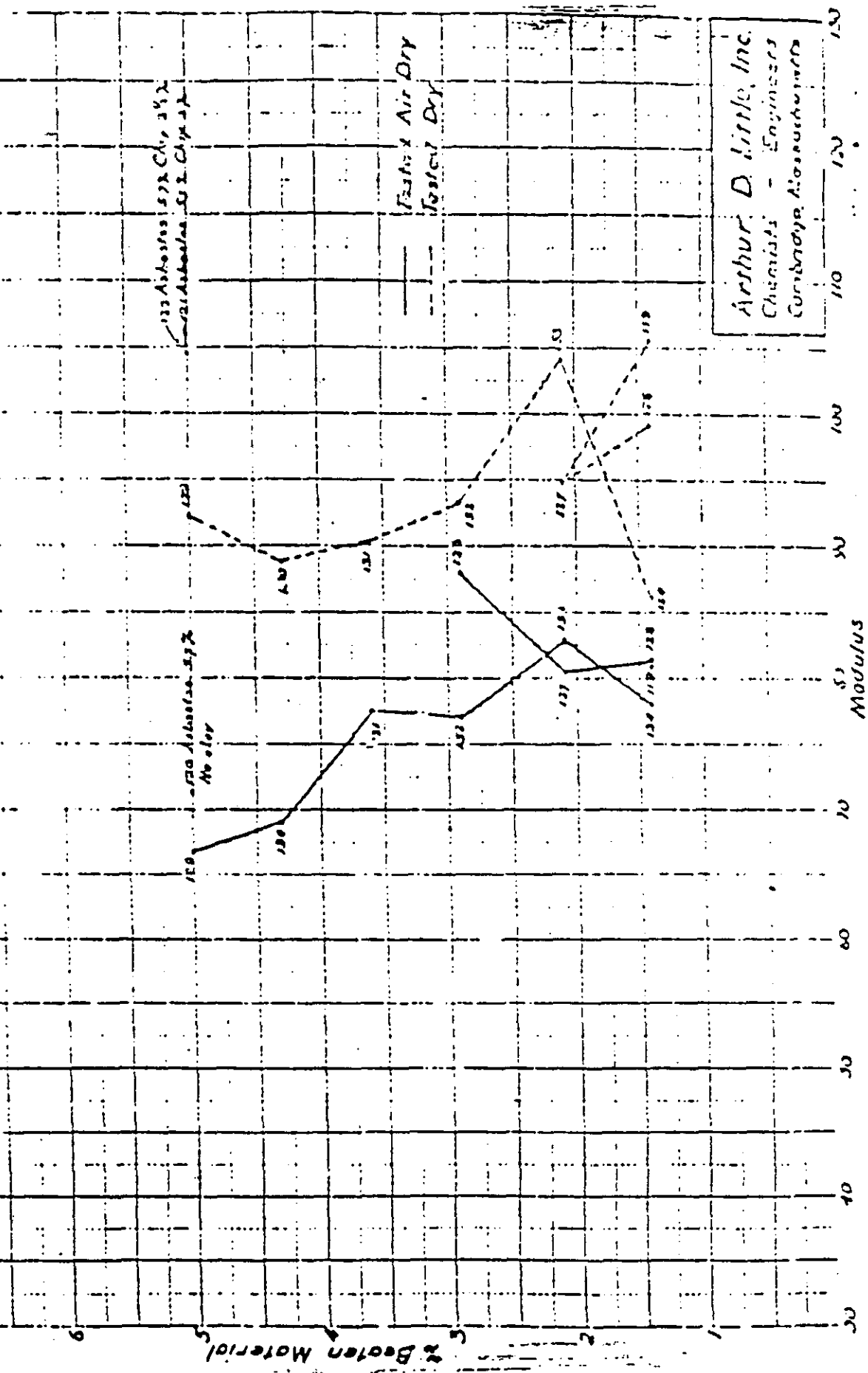
Batch Number	Clay	Added Materials-Percent			Physical Tests			Remarks
		Sulfate Pulp Beaten 15 hrs.	Newsprint Beaten 3 hrs.	Asbestos Crude 4M	Density lbs./cu.ft.	Modulus Air Dry	Modulus 100% Dry	
C112	10.0	5.7	-	10.0	9.6	16.6	13.6	Poor
C113	5.0	5.7	-	10.0	-	-	-	-
C119	5.0	1.4	-	10.0	10.6	81.0	105.5	Good
C126	5.0	2.9	-	10.0	10.2	83.0	-	Poor
C127	5.0	2.1	-	10.0	10.5	80.5	94.8	-
C128	5.0	1.4	-	10.0	10.4	81.3	99.1	-
C129	5.0	-	5.0	10.0	11.2	55.7	92.2	Poor
C130	"	-	4.3	"	11.2	59.0	83.8	Good
C131	"	-	3.6	"	11.1	77.5	90.3	Poor
C132	"	-	2.9	"	11.1	77.2	93.3	Poor
C133	"	-	2.1	"	11.4	82.9	101.1	Good
C134	"	-	1.4	"	11.2	78.0	85.3	-

CHART I
Paper Pulp - Pounds per cubic foot vs. Modulus



Arthur D. Little, Inc.
 Chemists - Engineers
 Cambridge, Massachusetts

CHART II.
Paper Pulp - Percent Pulp vs Modulus



Arthur D. Little, Inc.
Chemists - Engineers
Consulting Laboratories

General Comments on Batches C59 to C60

Table II lists all the batches made by our regular asbestos slurry formula since our sixth progress report. Batches using quicklime, high lime or high silica, yellow amosite in place of crude chrysotile, shale in place of clay and woodchip in place of beaten asbestos are covered in other sections of this report.

Batches C49-C52 included in last report were made with celite that had not been ignited. Batches C59 to C61 were mixed three days before induration and show very low strength. Previous work has indicated that ageing of mix up to 24 hours may be beneficial to strength of the final product but in the three days ageing the cold reaction had probably proceeded so far that the structure already formed was broken up in stirring and pouring into pans, preventing maximum strength in the finished product.

Batches C62 to C65 were made with hydrate for comparison with the quicklime batches C66 to C69 and while they show slightly better strength than the quicklime batches they are weaker than any previous series with 3 hour beaten asbestos and 16 to 17 hour induration.

Discussion of Low Strengths - Because of the low strengths of batches C62 to C65 it was thought that the lime used might have lost some of its activity but we now believe it was due to variation in the 4M asbestos. It is known that the 4M asbestos contains considerable fine non-fibrous material and as we were using the last quarter of the bag it is probable that the reduced proportion of fiber may have caused the lower moduli. This is illustrated by the progressive decrease in modulus of our 11 lb. per cu. ft., 3 hour slurry, 16 hour infiltrated batches.

		<u>Average</u>
C21-C25	5 Batches	110.5
C26-C32	4 "	92.1
C62-C78	2 "	81.2
C80	1 "	77.4

Similar decreases are apparent in 9 and 10 pound H-10 made during this period but not enough batches of the lighter weights were made to show so extensive a range.

Variation of Lime & Silica - When it was first realized that we were getting a progressive decrease in strength, the reactivity of the lime was considered the probable source. To check this C77 was made with about 6% excess lime, C78 by regular formula and C79 with excess silica.

were made in the wet pan using 10% yellow amosite the fibers were so pulverized that they could not be distinguished in the final product. It was thought that yellow amosite might still be used beneficially if the rollers were raised enough to reduce some of the grinding action. Raising the rollers was first tested on our regular formula H-20 the clearance being .045" on C86, C87, C88 and .005" on C89, C90, C91. The rollers did not turn in either case and the strengths were not quite as good as on other batches during this period. Some device should be made so that rollers would turn without so great friction on bottom of pan as is now necessary, if we are to experiment with yellow amosite.

Variation in time of beating asbestos - This subject was discussed in our sixth progress report and the conclusion given that beating for three hours was better than for two hours. The three hour beaten batches however were made before our average modulus began slipping and we can see now that they were not strictly comparable with the batches listed in that report made with two hour beaten asbestos. Repeated trials using three hour beaten asbestos have not shown any superiority over using two hour beaten asbestos.

Variation of Induration Time - One series of batches C45-C48 indurated 10 hours was given in our last progress report. Since then we have made three series (C83-85, C93-C100, C107-109) of batches indurated 10 to 11 hours and two series (C95-97, C104-106) indurated eight hours.

Ten Hour Induration - C83-C85 at 10 hrs. was not quite as good as C80-C82 at 17 hours, but C93-C100 at 11 hrs. was better than C92-C94 at 17 hours, and C107-C109 at 10 hours was better than C101-C103 at 16 hrs.

Eight Hour Induration - Batches C95-C97 at 8 hours was better than C92-C94 at 17 hours but not as good as C93-C100 at 11 hours. C104-C106 at 8 hours were about the same as C101-C103 at 16 hours but not as good as C107-C109 at 11 hrs.

These results indicate that sixteen to eighteen hour induration, the period used throughout previous work, is too long for the production of maximum strength or in other words E-10 indurated 16 hours is over indurated. As good results can be secured in eight hours as in sixteen, the ideal period of induration lying somewhere between these points.

Variations in Clay and Crude Asbestos - Batches C101 to C109 were made to recheck the 8, 10 and 16 hour induration periods, the advisability of 5% or 10% clay and the efficiency of 5% or 10% of crude asbestos (asbestos as fiber for hinging). These batches supported our

conclusion that 10 hour infiltration gave better strength than either 8 or 16 hours. For the addition of crude asbestos fiber, 10% gave definitely better strength with 5% clay than 5% asbestos did with either 10% or 5% clay. Whether 10% crude asbestos is necessary for hinging depends on how much hinging is required. With 5% asbestos the hinging is fair; with 10% the hinging is good.

This series of nine batches gives rather strong indication that 10% clay is too much. Batches C120 to C122 were made for comparison with shale mentioned elsewhere in this report, but they show that some clay is necessary for development of good strength. They also indicate that the optimum amount is somewhere between 0 and 5%.

Modulus after redrying at 110°C. - Although no tests are given for light weight material such as E-20, the A.S.T.M. Standards give tests for porous brick and insulating materials similar in composition to E-20 and in all cases specify 24 hours drying at 110°C. just previous to modulus test. Starting with batch C104 we have made part of our tests of each batch on air dry strips and part on oven dry strips. The latter give moduli 20 to 30% higher than the air dry strips. We understand that the Berlin Laboratory now conditions test strips at 50% relative humidity and 75°F.

TABLE II

(20)

Batches C59-C122 C60:S102 1:1

Batch Number	Induration Time	Added Materials-Percent				Physical Tests		
		Asbestos	Clay	Grass	Total Asbestos	Density lbs/cu.ft.	Modulus lbs/cu.ft.	Reaction
C59	16 hrs.	9.9	6.4	9.9	16.3	10.4	52.7	Fair
C60	"	9.8	6.9	9.8	16.7	9.9	52.4	"
C61	"	9.8	7.3	9.8	17.1	9.4	51.0	"
C62	"	9.9	5.9	9.9	15.8	11.1	52.9	"
C63	"	9.9	6.4	9.9	15.3	10.3	64.7	"
C64	"	9.8	6.9	9.8	16.7	9.9	61.0	"
C65	"	9.8	7.3	9.8	17.1	9.1	52.2	Poor
C78	"	9.9	5.9	9.9	15.3	11.1	59.5	Fair
C80	17 hrs.	10.0	5.7	10.0	15.7	11.3	77.4	"
C81	"	"	6.4	"	16.4	10.2	62.2	"
C82	"	"	7.3	"	17.3	9.2	48.5	Fair to Good
C83	10 hrs.	10.0	5.7	10.0	15.7	11.4	70.4	Good
C84	"	"	6.4	"	16.4	10.2	59.9	"
C85	"	"	7.3	"	17.3	9.4	44.9	"
C86	16 hrs.	10.0	5.7	10.0	15.7	12.0	69.2	Fair to Good
C87	"	"	6.4	"	16.4	10.8	53.8	Good
C88	"	"	7.3	"	17.3	9.2	40.3	Good
C89	"	"	6.4	"	16.4	10.7	52.7	"
C90	"	"	5.7	"	15.7	11.6	57.7	Fair
C91	"	"	7.3	"	17.3	9.5	43.3	Fair to Good
C92	"	"	5.7	"	15.7	11.2	58.1	Good
C93	"	"	6.4	"	16.4	10.1	46.8	Poor to Fair
C94	"	"	7.3	"	17.3	9.2	35.9	Fair
C95	8 hrs.	10.0	5.7	10.0	15.7	11.5	65.1	Fair to Good
C96	"	"	6.4	"	16.4	10.5	55.8	Fair
C97	"	"	7.3	"	17.3	9.4	43.0	"
C98	11 hrs.	10.0	5.7	10.0	15.7	11.1	65.9	Fair
C99	"	"	6.4	"	16.4	10.3	51.3	"
C100	"	"	7.3	"	17.3	9.1	45.2	"
C101	16 hrs.	10.0	5.7	5.0	10.7	11.1	66.0	Poor to Fair
C102	"	5.0	5.7	5.0	10.7	11.0	71.0	Fair
C103	"	5.0	5.7	10.0	15.7	11.1	65.9	Fair
C104	8 hrs.	10.0	5.7	5.0	10.7	11.6	67.2	Poor
C105	"	5.0	5.7	5.0	10.7	11.9	65.2	Fair
C106	"	5.0	5.7	10.0	15.7	11.3	79.1	Good
C107	10 hrs.	10.0	5.7	5.0	10.7	11.6	67.5	Fair
C108	"	5.0	5.7	5.0	10.7	11.2	91.9	Fair
C109	"	5.0	5.7	10.0	15.7	11.3	100.4	Good

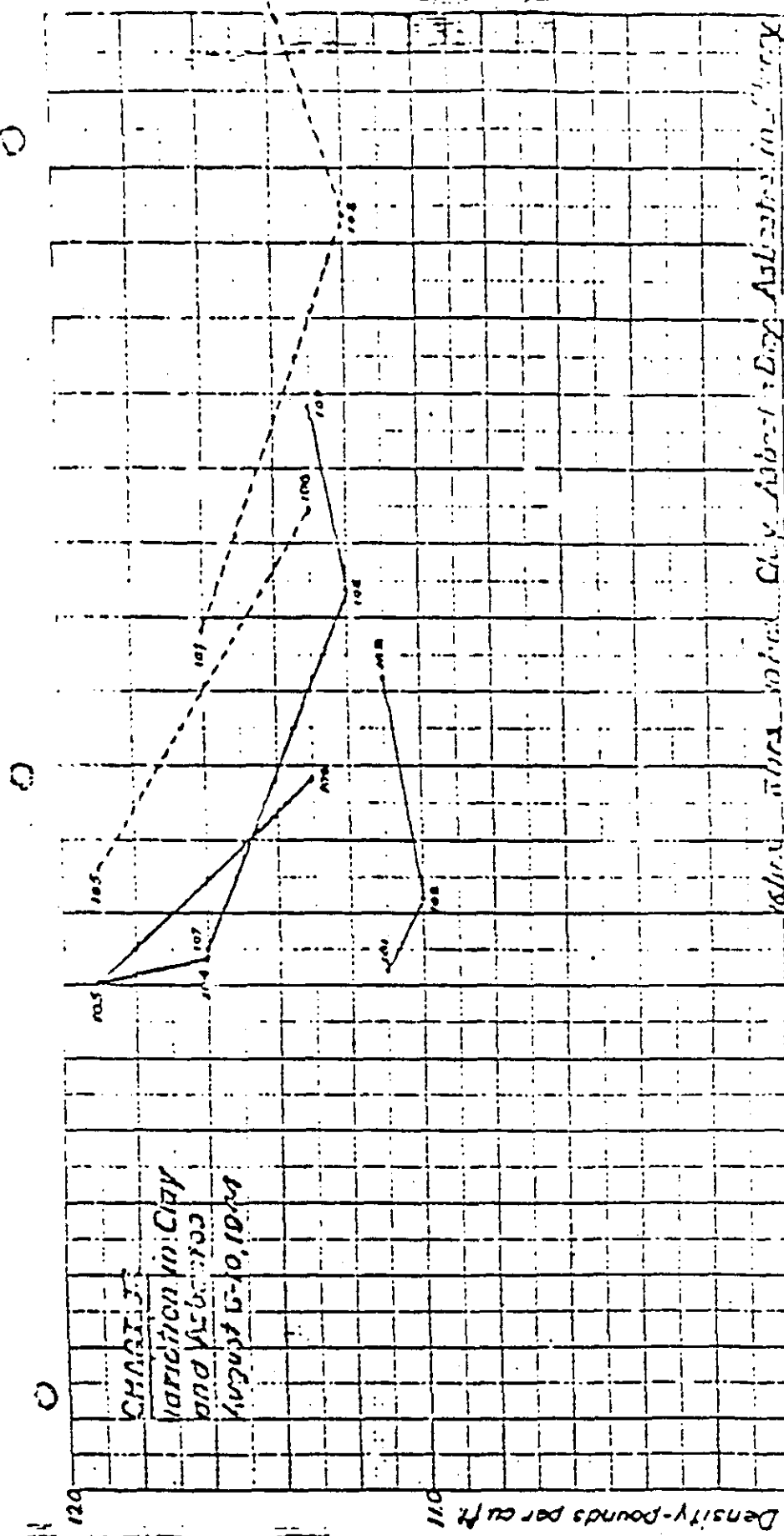
TABLE II (Continued)

Batch Number	Curing Time	Added Materials-Percent				Physical Tests		
		Clay	Asbestos	Crude Asbestos	Total Asbestos	Density lbs/cu.ft.	Modulus lbs/cu.ft.	Rating
C120	16 hrs.	-	5.0	10.0	15.0	11.2	51.2	Fair
C121	"	5.0	"	"	"	11.0	85.7	Good
C122	"	2.5	"	"	"	11.3	90.8	Good

Tested after 24 hours drying at 110°C.

C105	8 hrs.	5.0	5.7	5.0	10.7		73.5	
C106	"	5.0	"	10.0	15.7		97.4	
C107	10 hrs.	10.0	"	5.0	10.7		69.2	
C108	"	5.0	"	5.0	10.7		117.7	
C109	"	5.0	"	10.0	15.7		132.1	
C120	15 hrs.	-	5.0	10.0	15.0		71.4	
C121	"	5.0	"	"	"		104.9	
C122	"	2.5	"	"	"		104.6	
C123	"	10.0*	"	"	"		114.4	
C124	"	5.0*	"	"	"		65.0	
C125	"	2.5*	"	"	"		69.4	

* Hostone Shale used instead of Clay



Test	Clay Substituted in Clay (%)	Density-Pounds per cu ft (Air Dry)	Density-Pounds per cu ft (Dry)
C101	10%	107	105
C102	5%	107	104
C103	5%	109	108

Lime Hydrate 2 to 3 months old

— Tested Air Dry
 - - - Tested Dry

Arthur D Little Inc.
 Chemists - Engineers
 Cambridge, Massachusetts

TABLE III

QUICKLIME INSTEAD OF HYDRATE

CaO:SiO₂ Ratio 1:1

Batch Number	Indu- cation Time	Added Materials-Percent				Density lbs/cu.ft.	1
		Clay	Asbestos	Asbestos	Total Asbestos		
C66	17 hrs.	9.9	5.9	9.9	15.8	11.0	58
C67	"	9.9	6.4	9.9	16.3	10.2	61
C68	"	9.9	6.9	9.9	16.8	9.4	61
C69	"	9.8	7.3	9.8	17.1	9.1	50.5
C74	15 1/2 "	9.9	5.9	9.9	15.8	11.0	58.4
C75	"	9.9	6.9	9.9	16.8	9.6	57.3
C76	"	9.8	7.3	9.8	17.1	9.2	48.7

Quicklime vs. Hydrate

Two series of batches C66 to C69 and C74 to C76 Table III and Chart IV were made using quicklime instead of lime hydrate. The lime was slaked in Simpson wet pan, the other ingredients added and process continued in the usual manner. In some of these batches the clay, celite and lime were mixed dry, then water added sufficient to wet powder and to slake the lime, with the idea that the excess heat from slaking would start the lime-silica reaction. Too much heat was absorbed by the other ingredients so that the temperature did not rise high enough for test slaking of the lime. In the other batches a pocket was made in the pile of celite, the lime slaked in this pocket and as soon as the temperature of reaction started to drop the mixer was started up. Asbestos slurry was used instead of water for slaking the lime.

These batches were no better than the batches made with lime hydrate. They averaged in strength about the same as batches C37 to C40 made with 3/4 hour beaten slurry.

Shale as Substitute for Clay

Shale (from Eastone Co.) additions of 2.5%, 5%, and 10% were made to batches C125, 124 and 123 instead of using clay. This shale is an aluminum silicate with high affinity for lime. C123 with 10% shale when tested after relaying had nearly twice the modulus of the batches containing 2.5% and 5%; however, it was only slightly better than batches with 2.5% and 5% clay made in the same autoclave run. The clay batches were considerably stronger than the 10% shale batch when tested air dry. The 10% shale sample increased remarkably in the relaying. This possibly indicates that a long induration period is indicated for shale batches.

A series of batches was made with 10% to 60% shale using additional lime equivalent to 22% of the weight of shale used. C135 with 10% shale had slightly higher modulus (air dry) than C123 but did not show the remarkable increase on relaying that C123 did. C136 is irregular as considerable excess lime was added by error, however the air dry and dry moduli were 65.1 and 50.5 respectively. C137 with 30% shale also had a dry modulus of over 50 but batches with higher percentages of shale had moduli between 20 and 30.

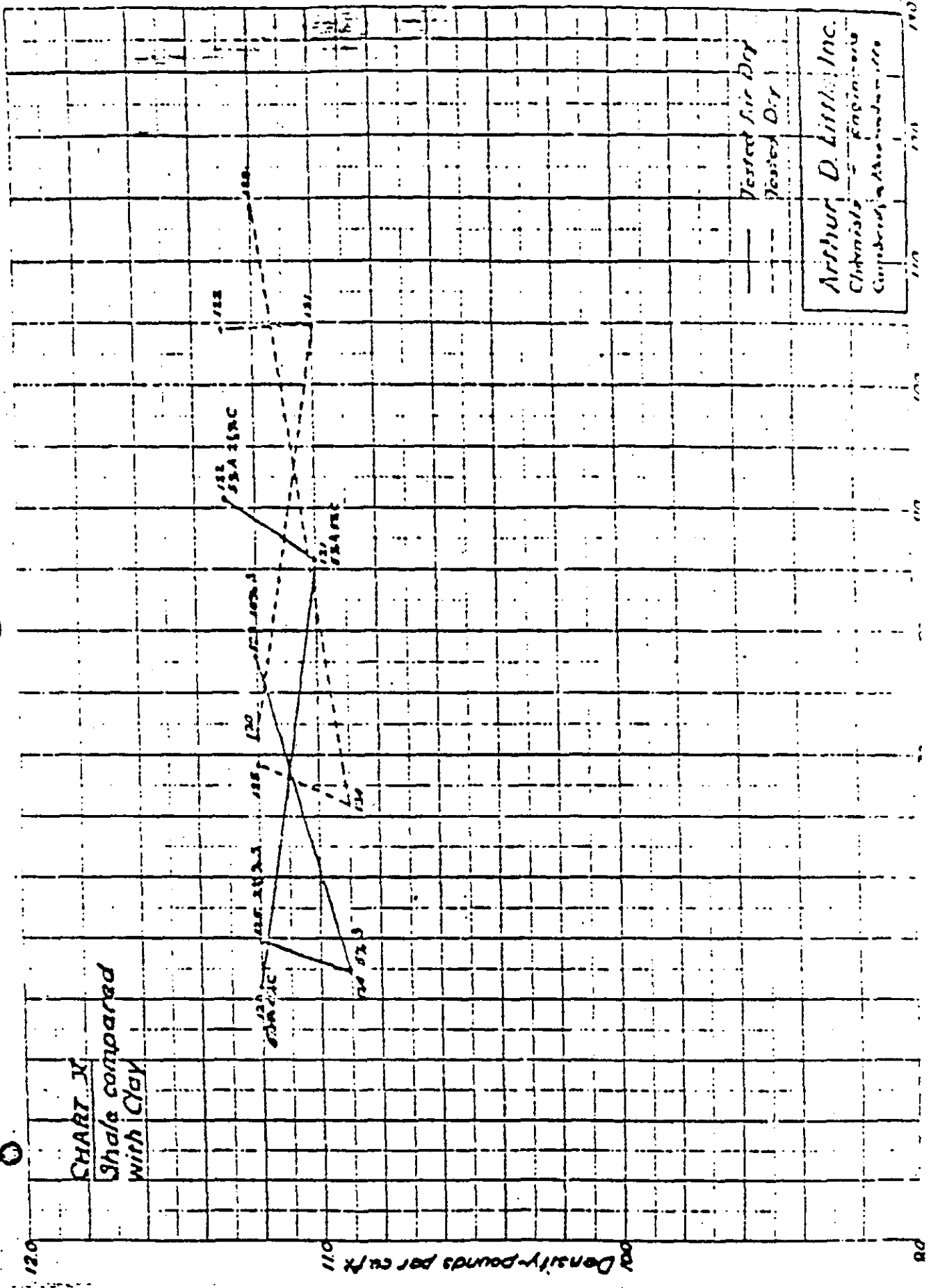
Although the above quoted experiments do not look favorable to the use of shale in K-LO, shale does represent a cheap source of aluminum silicate and with the proper adjustment of lime and of induration time might be a valuable addition to K-LO.

TABLE IV

Shale instead of Clay

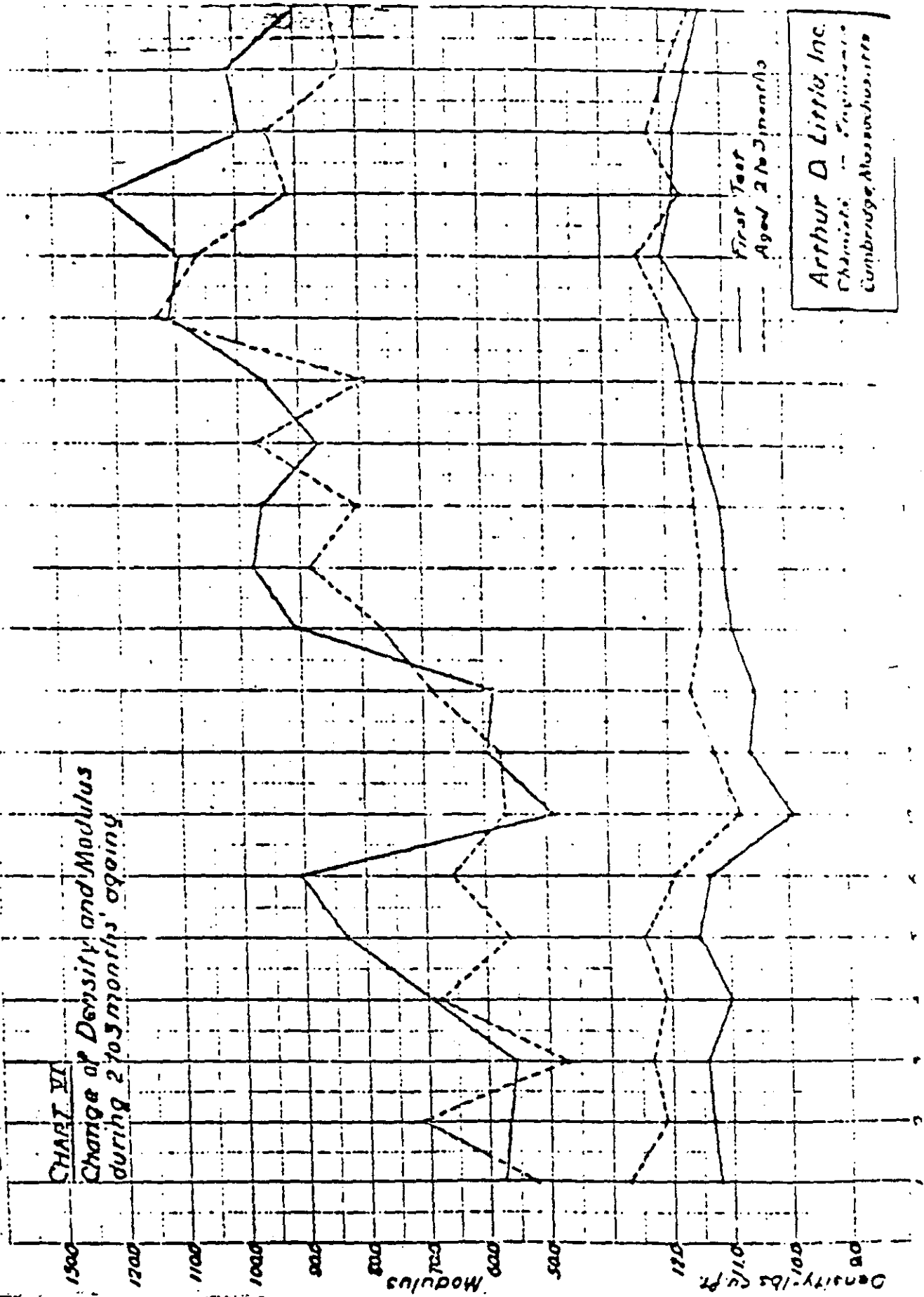
Batch Number	Added Materials - Percent				Physical Tests		
	Portland Cement	Asbestos	Crude Oil	Total	Density	Modulus*	Appearance
Shale Additions to 1:1 E-LO							
C123	10.0	5.0	10.0	15.0	11.2	114.4	Fair
C124	5.0	•	•	•	10.9	65.0	•
C125	2.5	•	•	•	11.2	69.4	•
Additional Lime (22% of Shale) added to 1:1 E-LO							
C135	10.0	5.0	10.0	15.0	11.2	93.9	Fair
C136	17.5	4.4	8.7	13.1	12.3	80.5	•
C137	30.0	5.0	10.0	15.0	11.1	52.0	Poor
C138	40.0	•	•	•	11.2	28.7	•
C139	50.0	•	•	•	11.5	22.7	•
C140	60.0	•	•	•	12.0	20.0	•

* Modulus after 24 hours redrying at 110°C.



Arthur D. Little, Inc.
 Chemical Engineers
 Consulting Engineers

CHART VI
Change of Density and Modulus
during 2703 months' ageing



Arthur D Little, Inc.
Chemists - Engineers
Cambridge, Massachusetts

FIGURE 7

LAYER-EFFECT

JOHN'S-MANVILLE
THERMOBLASTOS
1.31

UN
LINE

DIRECTION
OF ST.

