16

E. I. DU PONT DE NEMOURS & COMPANY
PIGMENTS DEPARTMENT
NEWPORT PLANT

Serial No. KN-67-1

Copy to: ~~[illegible]~~
#16

NEWPORT PLANT
PIGMENT COLORS RESEARCH REPORT

SUBJECT: LONG RANGE MODERNIZATION PROGRAM - ALTERNATE EQUIPMENT STUDIES - SIZE REDUCTION WITH VIBRATING BALL MILL

PERIOD COVERED: MARCH, 1966 - JANUARY, 1967

SERIAL NO. KN-67-1

DATE: 3/1/67

KN-67-1

COPY NO. 16

DISTRIBUTION:

1 - Research Numerical File
2 - Research Office File
3 - M. Hunt/E. Gonick
4 - W. S. Struve/A. A. Brizzolara
5 - J. H. Cooper/B.H. Perkins/H.R. Linton/F.F. Ehrich
6 - W. J. McClure/R. A. Hageman/H. Arnoul/Q.C. File
7 - P. J. Monahan (Vital Records)
8 - E. L. Rodowskas/W. A. Jenkins
9 - G. M. Loughran/F. N. Odell/Q.C. File
10 - R. H. Wetzel
11 - J. W. Minnich
12 - H. H. Gyorgy
13 - J. F. Maurer
14 - R. E. Stuber - Louviers
15 - N. R. Levins - Louviers
16 - C. E. Rick
17 - Research File - Colors - Newport
18 - Extra
19 - Extra
20 - Extra
21 - Extra
22 - Extra
23 - Extra
24 - Extra
25 - Extra

NEWPORT PLANT

PIGMENT COLORS RESEARCH REPORT

SUBJECT: LONG RANGE MODERNIZATION PROGRAM - ALTERNATE EQUIPMENT STUDIES - SIZE REDUCTION WITH VIBRATING BALL MILL

PERIOD COVERED: MARCH, 1966 - JANUARY, 1967

SUBMITTED BY: D. H. EASTHAM   Date Submitted: 1/9/67

APPROVED BY: J. W. MINNICH JWM   Date Released: 3/1/67

I. INTRODUCTION

As part of a program to modernize the CPC operation, reduce costs and improve productivity, an improved method for size reducing CPC was desired. This study was conducted to provide information for future planning and expansion of CPC facilities. Activity centered on a vibrating ball mill as a prime candidate for the following reasons:

A. Easy to charge and discharge.

B. Smaller and less expensive than rotating mills.

C. Easily adapted to pressure relief system.

More work is recommended on premilling and solvent milling to optimize the operation. Attention should be directed toward optimum charge of media and pigment, respectively.

II. CONCLUSIONS

A. A vibrating ball mill will provide a practical, effective means of producing premilled CPC at an economical production rate.

B. A vibrating ball mill does an excellent job of solvent milling; one vibrating mill has capacity of two existing rotating mills.

C. A vibrating solvent mill can be practically equipped with a safety relief system.

D. Dispersion milling capacity of a vibrating ball mill is not great enough with commercially available mills to justify replacement of existing mills or use for additional expansion.

E. Temperature control would be a difficult problem with a commercial vibrating dispersion mill.

III. SUMMARY

A vibrating ball mill is essentially the same as a conventional ball mill except the grinding energy is supplied to the grinding media via vibration instead of rotation. At the vibration frequency and amplitude normally used, the rate of energy input per unit volume grinding media is to the order of 10 times as great as a rotating mill which results in a much higher grinding rate.

Effectiveness of a VBM (vibrating ball mill) was determined for use in existing milling operations for equipment replacement or as a means to expansion.

III. SUMMARY (Cont'd)

Premilling

A commercial scale mill (30 gal.) has demonstrated a high rate of strength development as evaluated by solvent breaching. A Schutz-O'Neill, Model 250 L (210 gal.) operating under similar conditions is capable of producing premilled crude at an instantaneous rate equal to existing plant premills. Where longer premilling cycles are required for solvent breached products, the vibrating mill offers even more advantage. Production rates were compared on an instantaneous basis assuming existing premill loading is 1,200 lb. with 6 hr. grinding time. Excellent operability has been experienced in both lab and commercial scale mills.

Solvent Milling

The Schutz'O'Neill mill was also effective as a solvent mill (acetone milling) but most favorable candidate is a "Sweco Vibro-Energy Wet Grinding Mill". The "Sweco", Model M80 (182 lb. CPC/batch) mill offers larger batch size than Schutz-O'Neill mill with instantaneous grinding rate equivalent to two conventional solvent mills. With the relatively stationary machine, a pressure relief system could easily be provided.

Dispersion Milling

Dispersion or "salt" milling proved to be the most difficult. A combination of milling variables was found that produced a satisfactory product but the instantaneous grinding rate was not sufficiently high to justify a commercial installation. The same problems that exist with conventional rotating mills are also present with the vibrating mill. From a heat transfer standpoint, a vibrating mill would be more difficult to operate because of the higher rate of energy input per unit surface of heat transfer making temperature control more difficult.

IV. DISCUSSION

General

The most comprehensive theory available on vibrating mills is developed in the publication: Vibration Mills and Vibration Milling by H. E. Rose and R. M. E. Sullivan (Constable-London). The following equation was developed by the authors to predict the power input to the charge:

IV. DISCUSSION (Cont'd)

General (Cont'd)

$$Pc = 3.0 \times 10^{-8} \omega^3 \mathcal{L}^2 Mc$$

Where: Pc = Power dissipated in charge of VBM, KW
ω = Frequency of rotation, radian/sec.
$\mathcal{L}$ = Amplitude of vibration, cm
Mc = Mass of charge, kg.

During tests at vendor's lab, mill temperatures were measured before start-up and during milling. Neglecting the effects of conduction and radiation, the equation above was found to agree within reason with the temperature measurements. The temperature rise per unit time and total mass was known which was assumed to be the energy input to the charge; this energy was 50% below that predicted by the equation which is a reasonable agreement considering the assumption that ignored all heat losses.

Scale-up of VBM lab data does not appear to be a problem. Tests conducted in a commercial scale mill agreed with lab data. This is not surprising when the mechanism is examined; a small mill is just a section of a large mill.

Premilling

A Schutz-O'Neill, Model 250L, VBM mill produces premilled crude at an instantaneous rate equal to existing premills. Caking tendency, an anticipated problem with a vibrating mill, is not serious when using grinding media composed 80% cylpebs and 20% 1/2" steel balls.

A lab scale VBM, acquired for development purposes, can be utilized to more rigorouly optimize premilling. Figure 1 exhibits the rate of strength development in lab and large scale tests. Figure 2 shows the approximate relationship of 2 of the controlling variables in VBM size reduction (because of scant data, a linear relationship was assumed). From Figure 2, the amplitude of vibration has the greatest effect on rate of strength development. The loading (100% loading assumes filling voids with crude pigment) has an effect but not as pronounced.

Capacity calculation was made on following basis:

	Vol. %
Grinding Media	80%
Crude CPC	150% (Based on void volume of grinding media)

IV. DISCUSSION (Cont'd)

Premilling (Cont'd)

Physically, a loading of 160% is maximum that can be physically added with a media charge of 80%. According to Figure 2, at the end of one hour a loss of 2 points of strength would result from 150% loading but strength is still equal to standard. Obviously, the premilling operation was not optimized and the opportunity is available to probably exceed the capacity of existing mills.

All comparisons were made to N-930 standard which now requires 15 hour premilling. The same grinding times are assumed to apply to premilled crude for solvent milling but it is possible that shorter millings may be satisfactory for use in solvent mills. A commercial premill would produce more but smaller batches probably necessitating the use of automated bulk handling equipment to keep labor costs to a minimum.

Allis-Chalmers manufactures a VBM with a 90 gal. grinding chamber powered by two 75 HP motors (Schutz-O'Neill has one 30 HP motor) which provide an extremely high rate of energy input. The largest A-C mill sells for about $22,000 compared to the cost of a Schutz-O'Neill mill of $11,000. A-C is working on larger vibrating mills but short bearing life has prevented full-scale marketing. Another contact should be made at a later date to determine A-C's progress in this area.

Solvent Milling

The solvent milling (acetone milling) responds very favorably to vibratory milling. Two types of mills were tested, the Schutz-O'Neill mill and a "Sweco Vibro-Energy Wet Grinding Mill". The Sweco (Southwestern Engineering Co.) mill was the most attractive from capacity standpoint because of its relatively large size (182 gallons batch size). A smaller Sweco mill was tested at the Experimental Station and strength development rates indicated that one Model M80 Sweco mill has the capacity of 2 existing solvent mills. Rate of strength development is shown in Figure 3. Capacity calculations are included in Appendix.

If additional dispersion milling capacity is required at Newport in the future, one VBM solvent mill could be installed to replace 2 existing solvent mills and free one mill location for installation of a dispersion mill.

IV. DISCUSSION (Cont'd)

Solvent Milling (Cont'd)

A "Sweco" Model M80 sells for about $24,300 and could replace two solvent mills with replacement (equipment cost) value of about $30,000 each.

One obvious advantage over existing rotating mill, would be the ease of providing a pressure relief system because of the stationary nature of the mill. Mechanical considerations preclude the use of a relief system with our existing solvent mills.

The solvent milling operation was not optimized and the opportunity to achieve even greater production from a given mill is open. For example, during testing it was noted that circulation of slurry through the grinding media was excellent which could make possible smaller grinding media charges and more CPC-solvent slurry.

Dispersion Milling

Dispersion milling responds favorably to vibratory milling but not to the extent of premilling and solvent milling. In general, premilling and solvent milling is about 600% faster than rotating mill; dispersion milling is about 250% faster - not enough to justify a plant installation.

The principle that makes vibratory milling more rapid, high rate of energy input, would make dispersion milling on commercial scale difficult. The ratio of energy input to heat transfer area in a VBM is much higher than existing rotating mills which would create a temperature control problem. Temperature control in existing dispersion mills is near marginal. Above 75-80°C experience has shown that caking of the charge is almost certain. Maintaining a plant scale VBM below 80°C would be extremely difficult plus the incentive is not great enough to develop a suitable technique.

Satisfactory products were made with half the standard amount of alum; this would increase milling capacity but still not enough. To be attractive, the milling time would have to be to the order of 1 hour. Equal strength (to standard N-797) was achieved in a minimum of 2-1/2 hours.

IV. DISCUSSION (Cont'd)

Dispersion Milling (Cont'd)

Various grinding media were tried until an acceptable combination was found to be a mixture of cylpebs and steel balls (same mixture as for premilling). Balls were included because of higher grinding rates than cylinders. The sharp edges of the cylpebs (1/2" x 1" steel cylinders) seem to keep the mill walls clean and prevent caking.

The test results are shown in Figure 4 with the effects of alum concentration and amplitude of vibration shown in Figure 5. The curves in Figure 5 are only approximations and would require additional testing for precision. However, the role of both amplitude of vibration and alum concentration is apparent. At practical levels of amplitude the effect of reducing the alum/CPC ratio from 6 to 3 added about 1-hour of additional grinding. This doubles the amount of CPC present and increases grinding capacity about 17%.

The most notable result was achievement of a BGD (N-797) product that was 5-7% stronger than a lab extracted in-process standard.

V. REFERENCES

Research Notebook No. 5098

Correspondence: R.E. Stuber to D.H. Eastham, 3/17/66; CPC Size Reduction Via Vibrating Ball Mill

R.E. Stuber to D. H. Eastham, 6/24/66; Acetone Milled CPC Analyses

R.E. Stuber to D.H. Eastham, 7/6/66; Vibrating Ball Mill Tests on CPC Pigments

Literature: Allis-Chalmers literature, operating instructions
Allis-Chalmers Manufacturing Co.
Milwaukee 1, Wisconsin

Schutz-O'Neill literature
Schutz-O'Neill Co.
307 Portland Ave, South
Minneapolis, Minnesota 55415

Sweco - Vibro-Energy Wet Grinding Mill
Southwestern Engineering Co.
6111 East Bandini Blvd.
Los Angeles, California 90054

/mc

APPENDIX

TABLE I

PREMILLING TESTS
EXPERIMENTAL DATA

Date	Mill Vol. Gal.	Milling Time, HR.	Speed, RPM	Amplitude, MM	Loading, %	Strength	Hue	Chroma
4/14	0.275	0.125	1620	1.75	100	122	-	-
		0.25			100	111	-	-
		0.50			100	108	R_{15}	D_{17}
		1.00			100	108	R_{10}	D_{10}
		0.125			100	116	-	-
		0.25			100	108	-	-
		0.50			100	104	sR_9	sD_8
		1.00			.00	102	vsR_5	vsD_4
6/20	30	0.25	1760	4.75	85	107	-	-
		0.50			85	102	-	-
		1.00			85	98	-	-
		0.25			336	130	-	-
		0.50			336	115	-	-
		1.00			336	105	-	-
		0.25		2.35	360	140	-	-
		0.50		2.35	360	125	-	-

Newport, Delaware
February 3, 1967

LONG RANGE MODERNIZATION

CAPACITY OF VBM - PREMILLING

Commercial scale Schutz-O'Neill mill Model 30L, demonstrated essentially equal strength after 0.75 hr. grinding time at 85% loading. Assume this rate applies in Model 250L at 150% loading.

250L - 29.5 $Ft.^3$ - Total Volume

Grinding media will occupy 80% of total volume; Porosity of charge is 45%; use 150% fillage of voids, so:

$(29.5\ Ft.^3)\ (0.80)\ (0.45)\ (1.5) = 15.9$

Crude CPC = 10 $lb./ft.^3$, (15.9) (10) = 159 lb. CPC

Grinding time - 0.75 Hr.

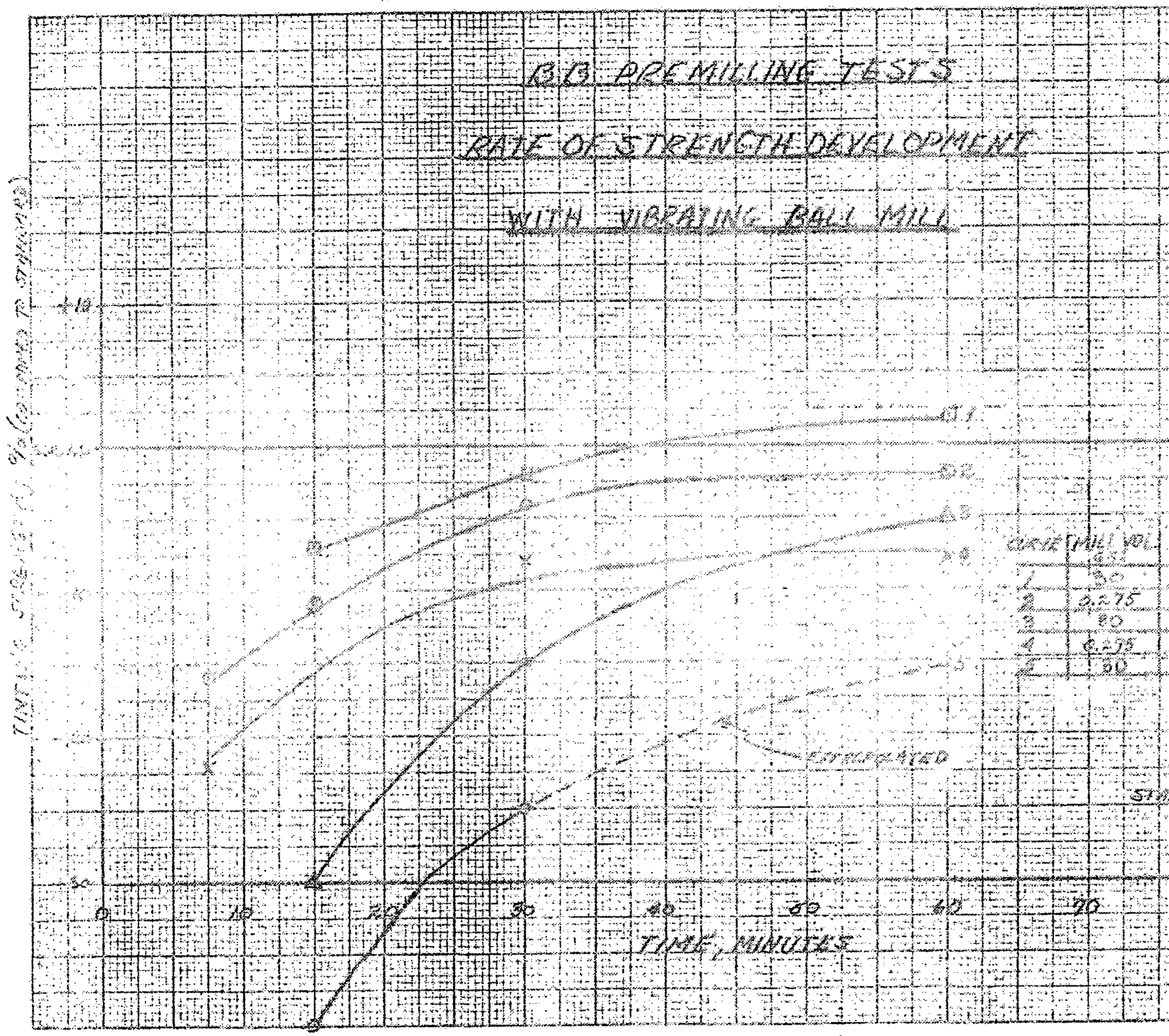
$$\boxed{\frac{159}{0.75} = 212\ \text{lb./hr. Instantaneous Rate for VBM}}$$

Existing Mill:

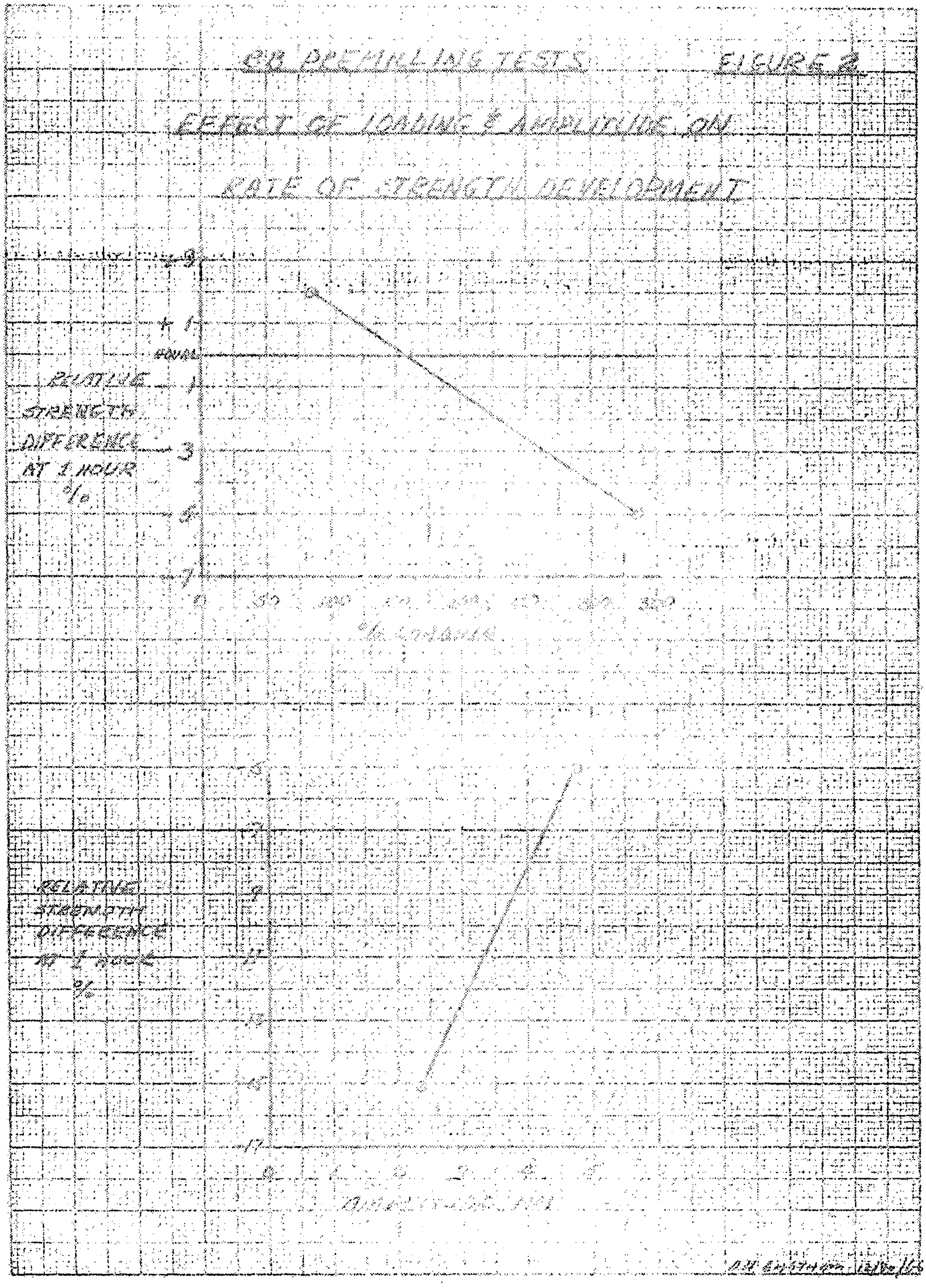
Charge 1200 lb.
Grinding time - 6 Hr.

$$\boxed{\frac{1200}{6} = 200\ \text{lb./hr. for Existing Mill}}$$

D. H. Eastham 12/30/66



B.B. PREMILLING TESTS
RATE OF STRENGTH DEVELOPMENT
WITH VIBRATING BALL MILL
TINTING STRENGTH % (COMPARED TO STANDARD)
+10
TIME, MINUTES
0
10
20
30
40
50
60
70
EXTRAPOLATED
CURVE
MILL VOL.
1
30
2
0.275
3
90
4
0.275
5
50


CB PREMILLING TESTS
FIGURE 2
EFFECT OF LOADING & AMPLITUDE ON
RATE OF STRENGTH DEVELOPMENT
RELATIVE
STRENGTH
DIFFERENCE
AT 1 HOUR
%
+1
1
3
5
7
0
% LOADING
RELATIVE
STRENGTH
DIFFERENCE
AT 1 HOUR
%
9
17
0

Newport, Delaware
February 3, 1967

LONG RANGE MODERNIZATION

ESTIMATE OF POWER INPUT

Mill Charge 4000 g
1300 g
5300 g Total weight (Grinding Media Mill)
66 g Charge
5300
5366 g Charge and Mill, etc.

1620 RPM 3.5 mm Displacement

57.3 °/Rad.

$\frac{1620}{60} = 27$ RPS $\qquad \frac{27(360)}{57.3} = 169.5$ Rad./Sec.

$Pc = 3 \times 10^{-8}\, \omega^3 \alpha^2\, Mc$

$\alpha = 1.75$ mm Amp. 0.175 cm

$Pc = 3 \times 10^{-8}\, (169.5)^3\, (0.175)^2\, (5.366)$

$Pc = 0.0243$ KW

Temperature Rise 6°F in 10 Min.
7°F in 10 Min.

Avg. 6.5°F in 10 Min.
0.65°/Min.

c_pSteel = 0.12 Btu/lb.°F

$Q = Mc_p\, \Delta T$

$m = \frac{5366}{454} = 11.82$ lb.

$Q = (11.82)\,(0.12)\,(0.65)$

$Q = 0.923$ Btu/Min.

1 KW Hr. = $\frac{3412.19 \text{ Btu}}{\text{KWH}}$

$(0.923)\,(60) = 55.4$ Btu/Hr.

$\frac{55.4 \text{ Btu/Hr.}}{3412.19 \text{ Btu/KW-Hr.}} = 0.016$ KW

$\frac{0.0243}{0.016} = 1.5$
$\pm 50\%$

D. H. Eastham
12/29/66

TABLE II

SOLVENT MILLING TESTS (BBS)

EXPERIMENTAL DATA

Date	% Charge	Premill Time, Hr.	Solvent Mill Time, Hr.	Speed RPM	Amplitude MM	Mill Vol, Gal.	Strength	Hue	Chroma	Comments
4/21	100	6	1	1750	2	0.275	106	sR_7	vsD_5	1/8"Shot for Media
			3	1750	2	0.275	98	sG_7	vsI_5	
5/17			2	1200	-	2.6	115	-	-	Used Plant cylpebs for grinding media
			4		-		107	-	-	
			6				104			1/8" Shot for Media
5/17			2		-		103	vsG_3	vsI_2	
			4		-		100	vsG_6	vsI_5	
			6		-		98	sG_8	sI_8	
9/9			2	1725	2	0.275	108	R_{17}	D_{15}	
			3	1725	2	0.275	101	sR_7	vsD_5	
9/15		Crude	Up to 6 Hr.	1725	2	0.275	-	-	-	By visual inspection no significant size reduction accomplished

Newport, Delaware
February 3, 1967

LONG RANGE MODERNIZATION

SOLVENT MILLING - VBM - CAPACITY

Assume use of Model M80 - Slurry volume 182 gal: (182 lb. dry CPC). Tests at Experimental Station were not conducted at maximum amplitude $\therefore$ higher rate of strength development can be expected. At demonstrated grinding time of 4 HR. capacity on instantaneous basis would be:

$$\frac{182 \text{ lb. CPC}}{4 \text{ HR.}} = 45.5 \ \frac{\text{Lb./CPC}}{\text{HR.}}$$

$$\frac{45.5}{27.6} = 1.65 \times \text{Existing Solvent Mill}$$

Existing Plant Rate:

$$\frac{550 \text{ Lb.}}{20 \text{ HR.}} = 27.6 \text{ Lb./HR.}$$

For 3 HR. Grinding Time:

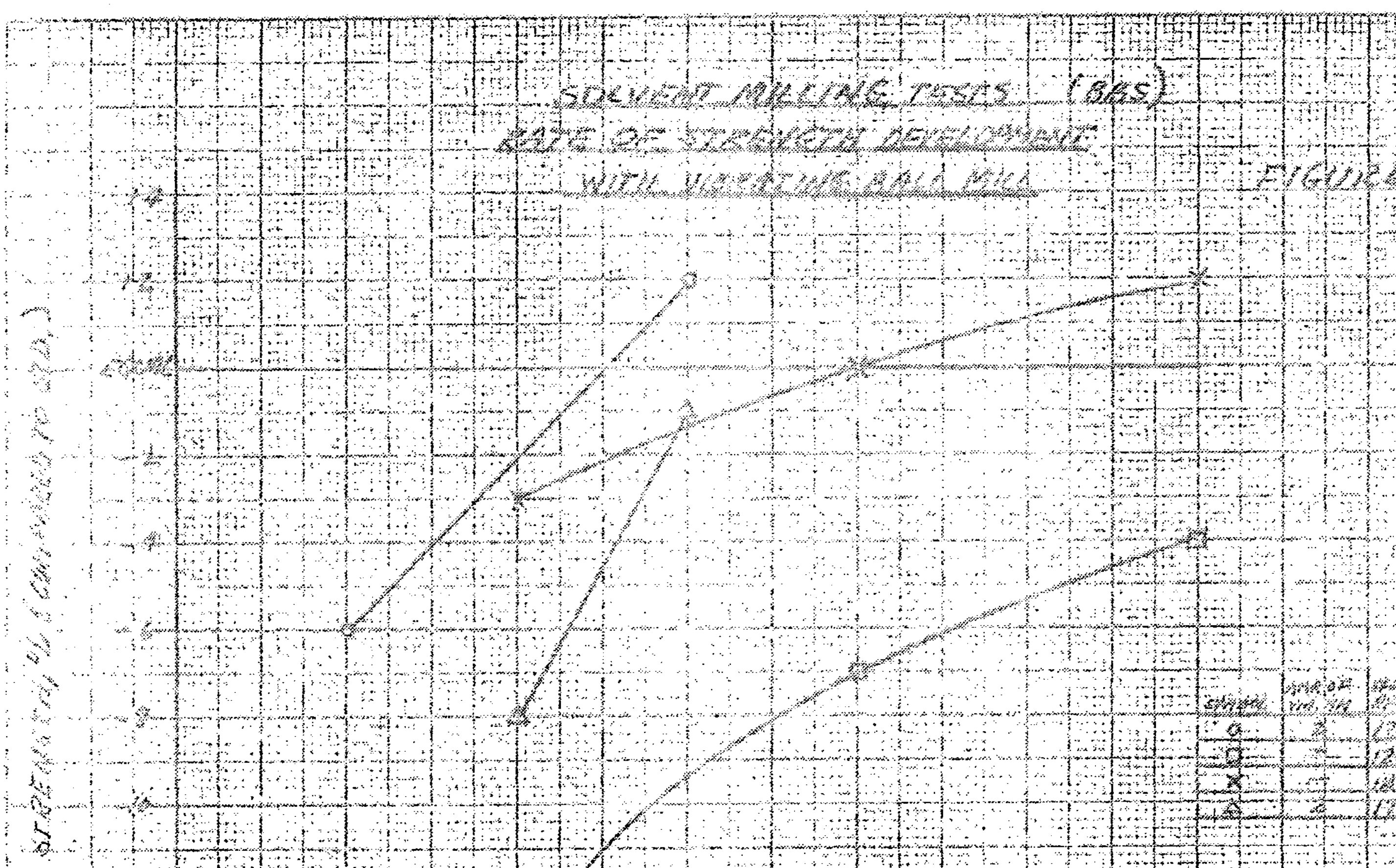
$$\frac{182 \text{ Lb. CPC}}{3 \text{ HR.}} = 60.7 \ \frac{\text{Lb. CPC}}{\text{HR.}}$$

To Equal 2 Plant mills need:

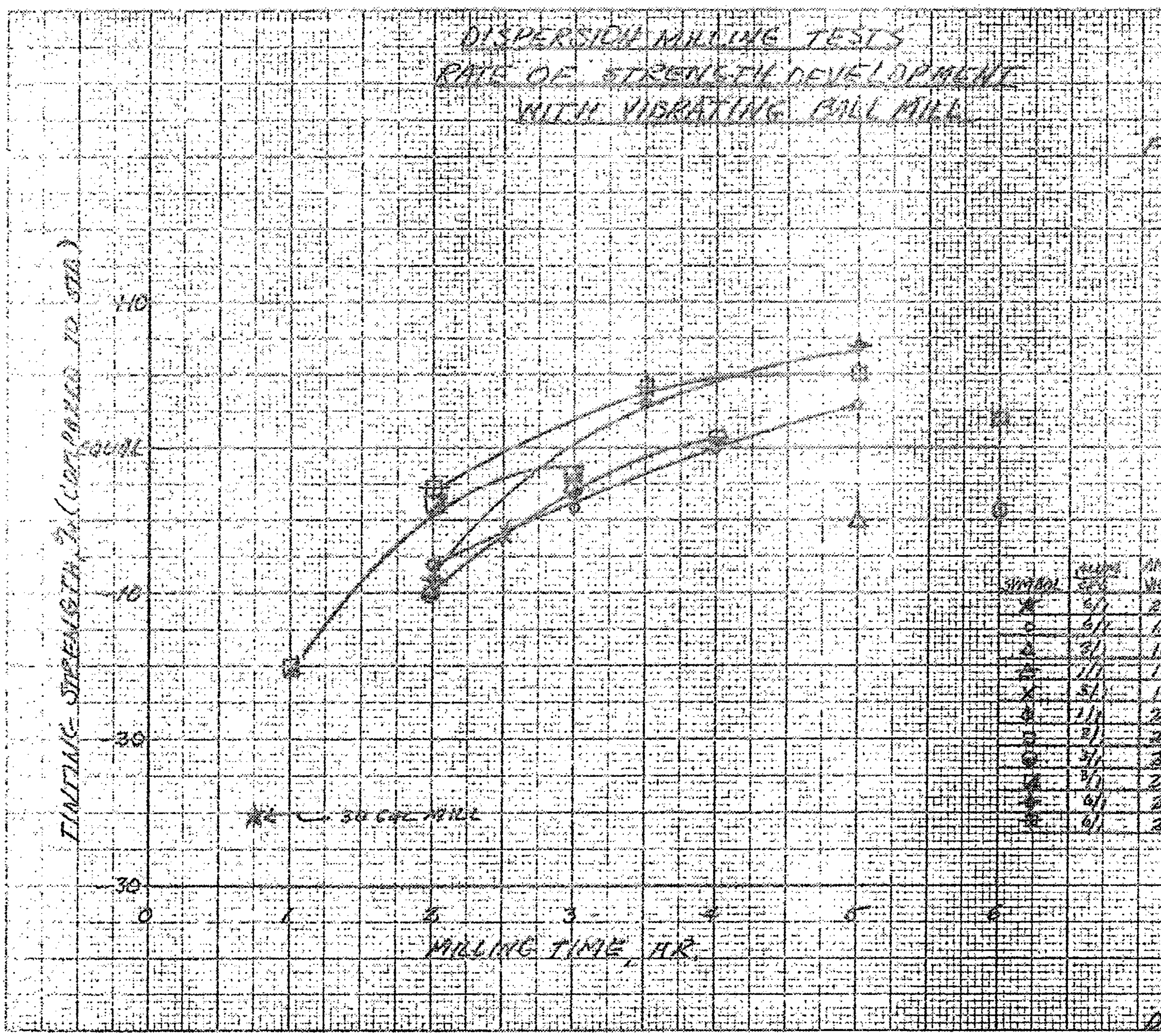
$$\frac{182}{2 \times 27.6} = 3.32 \text{ Hr. Grind Time}$$

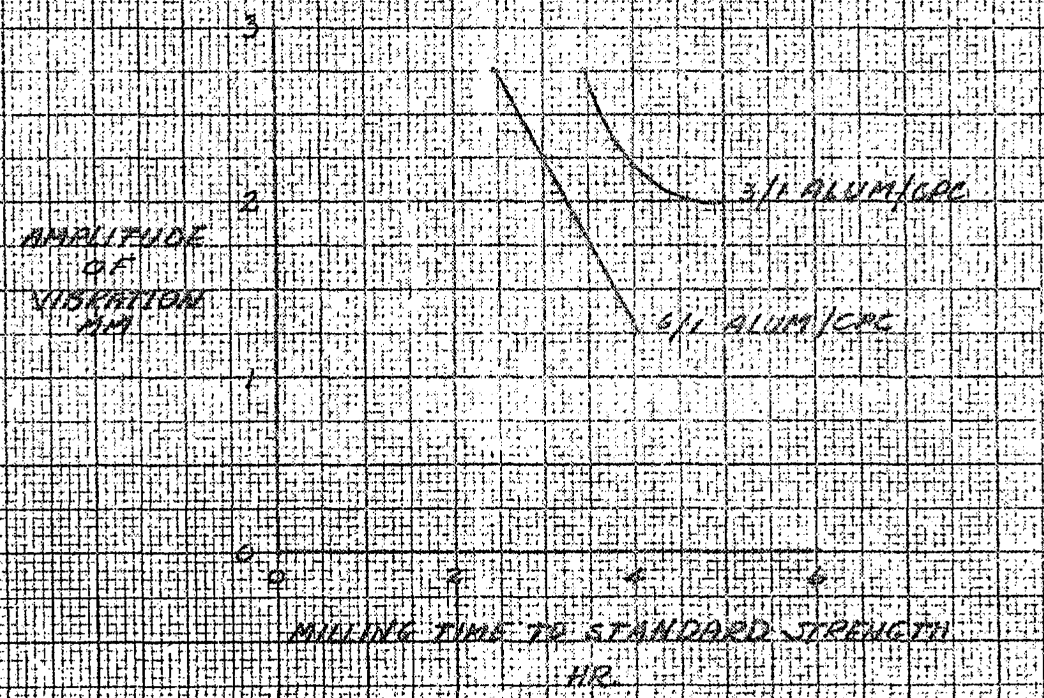
Strength deficiency was less than 1% at 3.32 Hr. from Figure 3. I am assuming that using maximum amplitude will more than compensate for the strength deficiency.

K+E 10 X 10 TO THE 1/2 INCH 359-11
KEUFFEL & ESSER CO. MADE IN U.S.A.



KEUFFEL & ESSER CO. 10 X 10 TO THE 1/2 INCH 359-11 MADE IN U.S.A.





NOTE: ABOVE CURVES DEVELOPED FROM CURVES OF FIGURE

TABLE III

DISPERSION MILLING TESTS

EXPERIMENTAL DATA

Date	Alum CPC	Ampli-Tude MM	Speed RPM	Loading %	Milling Time, Hr.	Strength	Hue	Chroma	Comments
6/30/66	6/1	2.35	1760	340	0.25	150:100	-	-	Rubber lined mill - Stopped at 0.75 Hr. because of mill temperature
					0.50	145:100	-	-	
					0.75	125:100	-	-	
8/30/66	6/1	1.25	1725	100	2.0	108:100	vG_{30}	vD_{30}	
					3.0	104:100	vG_{20}	vD_{18}	Good performance
					4.0	100:100	G_{14}	D_{12}	
					5.0	97:100	sG_8	sD_7	
9/12/66	1/1				5.0	-	-	-	Caked severely
	3/1				5.0	105:100	R_{25}	D_{25}	
9/30/66	1/1	1.25/1.5			3.5	-	-	-	Mill caked - raised amplitude and mill cleaned up
	1/1	1.50			3.5+2.5	106:100	-	I_8	
	3/1	1.25/1.5			6.0	106:100	-	vD_{18}	
	1/1	2.0			6.0	104:100	R_6	D_{12}	
	3/1	2.0			6.0	98:100	=	I_{12}	
10/21/66	3/1	2.0			2.0	110:100	G	-	Mild caking - dumped and extracted uncaked portion
	3/1	2.0			3.0	103:100	G	-	
	3/1	2.25			4.0	100:100	vsG	vsI	No caking - raised amplitude
10/25/66	3/1	2.75			1.0	115:100	-	-	Caked on cylpebs in one chamber
	3/1	2.75			2.0	104:100	-	-	Did not cake - 74°C mill temperature
	3/1	2.75			3.0	102:100	-	-	Did not cake
1/4/67	6/1	2.25			2.0	109:100	-	-	Did not cake
					3.5	97:100	-	-	
					5.0	93:100	-	-	
1/7/67		2.75			2.0	103:100	G18	D_{29}	
		2.75			3.5	96:100	sG_8	sI_6	Did not cake
		2.75			5.0	95:100	G_{15}	sD_6	