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E. I. du Pont de Nemours & Company

F & F, Research Division

Experimental Station Laboratory

Research Report

c-1

IMPROVED PIGMENT GRINDING

1. Vibration Assisted Mill Base Preparation
2. SoniCo's Orboresonance Technology

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INTRODUCTION

The purpose of pigment grinding is to incorporate pigment into paint vehicle to obtain a fine particle dispersion. According to Patton (Ref. 1), this process is carried out in three overlapping steps. They are wetting, grinding and dispersion. Wetting refers to the displacement of air and moisture in the pigment by other constituents of the mill bases. Grinding refers to the mechanical break-up of flocculates and agglomerates. Finally, dispersion refers to the permanent particle separation. This work is concerned with the second step whose successful execution is a prerequisite to a well dispersed mill base.

OBJECTIVE

Our commercial objective is to develop grinding techniques that yield ball mill, two-roll mill and flushed pigment qualities at lower cost by adding resonance vibration to the present 47 process.

Our immediate technical objectives are to establish the effect of (1) adding vibrational energy to sand mills and (2) using denser, harder media than sand in the 47 process.

SUMMARY AND CONCLUSIONS

The scope of vibration is normally defined by stating amplitude and frequency. Another way is to specify acceleration and frequency. The experiments described in this report (Figure 4) were carried out at relatively low G* (13-18G) acceleration and average frequency (40-60 cps). This is in contrast to the SoniCo's capability (Ref. 2) which is 150-480G at 60-350 cps. Presently, SoniCo is fabricating a modified sand mill at our request. The purpose of our experiments, therefore, was to obtain some evidence of improvement via vibration assisted milling using available equipment.

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*1G = Gravitational acceleration = 386 (in./sec.^{2}) in vibration terminology.

The available equipment consisted of a wide frequency range oscillator-amplifier-electromagnetic driver we have borrowed from the Engineering Technology Laboratory. With this set-up, we have examined five different mill base systems. Of these, the Harmon® Blue system was chosen for a detailed analysis.

<u>913-560 Harmon® Blue</u>

The experimental results with 913-560 copper phthalocyanine blue mill base show a marked decrease in seeding as measured by gloss measurements (Figure 1). To substantiate this finding, electron micrographs of the mill base cross sections were prepared (Figure 2). It is apparent that the unbaked mill base surface prepared with vibration is smooth while the surface of the control mill base without vibration is rough. The degree of flocculation determined by electron micrographs of the cross-sections (Figure 2) were about the same.

In addition, we determined tinting strength and L, a, b Adam's color coordinates. The results show increasing tinting strength with vibration (Figure 5). The Adam's coordinate changes calculated from the "Colormaster" reflectance measurements also indicate that the mill base prepared with vibration is bluer than the control sample (Tables 1, 2).

The scanning of important variables for this system was done by performing a factorial experiment. The Yates' method of analysis (Ref. 12) showed that, in addition to vibration, the effect of (sand/mill base) ratio and % solvent in mill base are important. The (sand/mill base) = 1.5 was found to be optimum for this system (Figure 6).

In every electron micrograph including Flint's let-down sample, we found dense agglomerates that are not completely wet (Figures 2, 3). Similar particles were observed in the case of 901-550 Monastral® Blue G (Figure 7).

High spot experiments to assess the effect of grinding media were also performed. Of the three different media used, only steel shot showed signs of improved gloss (Table 3, Figure 9).

Other Mill Bases

Four other systems examined are 907-686 Chrome Yellow, 907-582 "Milori" Blue, 901-6253 Black and 868-550 Monastral® Blue. A reasonably detailed examination for each of the above systems was necessary to select a proper system since either too easy or too complex a system might not reflect the effects of the low G orboresonance. The results are summarized in Table 4.

Mill base samples of Chrome Yellow in particular were sent to G. Orvis of Flint Development Laboratory. He concluded that the mill bases with vibration (Figure 4) possess higher tinting strength and cleanliness (Ref. 3). These two properties, however, were not reflected in the electron micrographs (Fig. 8) which showed excellent dispersion in both cases, indicating the difficulty of defining such properties as cleanliness and transparency.

Orboresonance

A working knowledge of orboresonance vibration was gained by visiting SoniCo laboratory (Ref. 2). This knowledge was essential in calculating the performance of MB Electronics' electromagnetic, variable frequency oscillator system (Ref. 4). Required mathematics with worked out sample calculations were carried out to analyze our experiments. Detailed description of the hardware and the measuring techniques will be reported later along with actual experimental results at SoniCo.

PATENT STATUS

Most of the patents on orboresonance belong to Bodine Soundrive Company. SoniCo, Inc., which is the subsidiary of Shell Oil, also owns some patents and has an agreement with Bodine Soundrive to use its technology freely. Du Pont in turn has a working contract with SoniCo and Bodine Soundrive to utilize the art. A detailed agreement can be obtained from B. M. Sturgis of Orchem. A voluminous patent study on the subject has been made by R. D. Morse of Engineering Department (Ref. 5).

A Notice of Invention on the vibration assisted milling based on our findings up to date will be written by 11/1/68.

ACTION TAKEN OR PROPOSED

Based on the findings contained in this report, the SoniCo's proposal to fabricate a orboresonance assisted model sand mill was adopted. The proposed fabrication time is four weeks.

Chrome Yellow mill base prepared by our early crude experiments were evaluated by G. Orvis of Flint. We will send another sample which is ground with our improved equipment by 9/15/68. "Harmon" mill bases will be sent to W. Moon and S. Arora for their evaluation. We expect to obtain their appraisal within a month.

ACKNOWLEDGMENTS

I would like to thank N. Komodromos, W. Bullock, W. Balloon, S. Arora, G. Orvis, W. Moon and J. King of Flint for their help. The assistance of W. Callahan of Philadelphia Process Development, D. Troy of the Experimental Station, and J. Lamiet of Pigments Dept. is appreciated. L. Lenhart of Marshall Laboratory helped to expedite the computer calculation.

DISCUSSION

1. Application of Vibration to Various Mill Base Grinding

Breakdown of F & F mill base production indicates that approximately 70% of the total production is processed via 47, 48 and 49 processes (Ref. 6). The remaining 25% is ball milled while about 1% of very difficult to grind pigments go through two roll mills. Cost-wise, typical milling cost ratios of these different treatments for automotive grade lacquer show: 47 process/ball milling/2-roll milling//1/1.75/6.5. We can, therefore, cut down the mill cost considerably by enlarging the scope of sand mill usage.

Another incentive for this project was pointed out by FitzGerald who recognized the importance of deagglomeration in controlling the degree of flocculation by means of AB type dispersant (Ref. 7). More specifically, he suggested the use of orboresonance technology and harder grinding media. Du Pont has a detailed agreement with SoniCo to exploit this technology (Ref. 8). Harder grinding media such as "Zircoa" and steel shot are available from Quackenbush Company.

A few exploratory experiments, however, immediately uncovered the need to:

- find proper candidate mill base system for testing which can reflect the effect of superposed vibration on normal sand mill operation and

- select proper methods of measuring the mill base quality.

Hegman gage, for example, was too crude for all the systems we have examined. Observation of a Brownian motion in a dilute system has also been used extensively, but its relationship to the dispersion stability of the real, concentrated system is not defined clearly. To select a proper system, we consulted the Flint dispersion group who supplied the following list (Ref. 9).

Mill Base Systems	Pigment/Vehicle	Need For Improvement
Group I (47 Process)		
907-686 (2 pass)	Chrome Yellow// MMA/DEAM	One pass desired.
907-808 (2 pass)	Monastral® violet	" " "
907-804 (2 pass)	"Thiofast" red lake	Improved tinting strength
907-131 (1 pass)	Ti-Pure® white	" " "
907-630 (1 pass)	Ferrite yellow	Less grit, lower fineness
907-393 (1 pass)	Monastral® yellow	" " " "
Group II (Ball Mill)		
907-582	"Milori" Blue	Improved seed/stability
901-6253	Peptized carbon black/MMA	Improved tinting strength
Group III (2-Roll Mill)		
901-3393	Monastral® maroon/ MMA	Extreme grit
901-5804	"Thiofast" red lake	" "
Group IV (under development)		
W-560	"Harmon" blue// MMA/AN	Develop as 47 process
W-816	Monastral® Maroon B	" " " "
W-678	3RLT "Irgazin" yellow	" " " "
W-684	Monastral® Orange	" " " "

The above list indicates areas where research is needed in terms of technical difficulty. I have also consulted a list which contains information on usage volume and per pound price (Ref. 10). The results of reasonably detailed experiments (Tables 1, 3 & 4) indicated that the "Harmon" blue system of Group IV is most suited for our study.

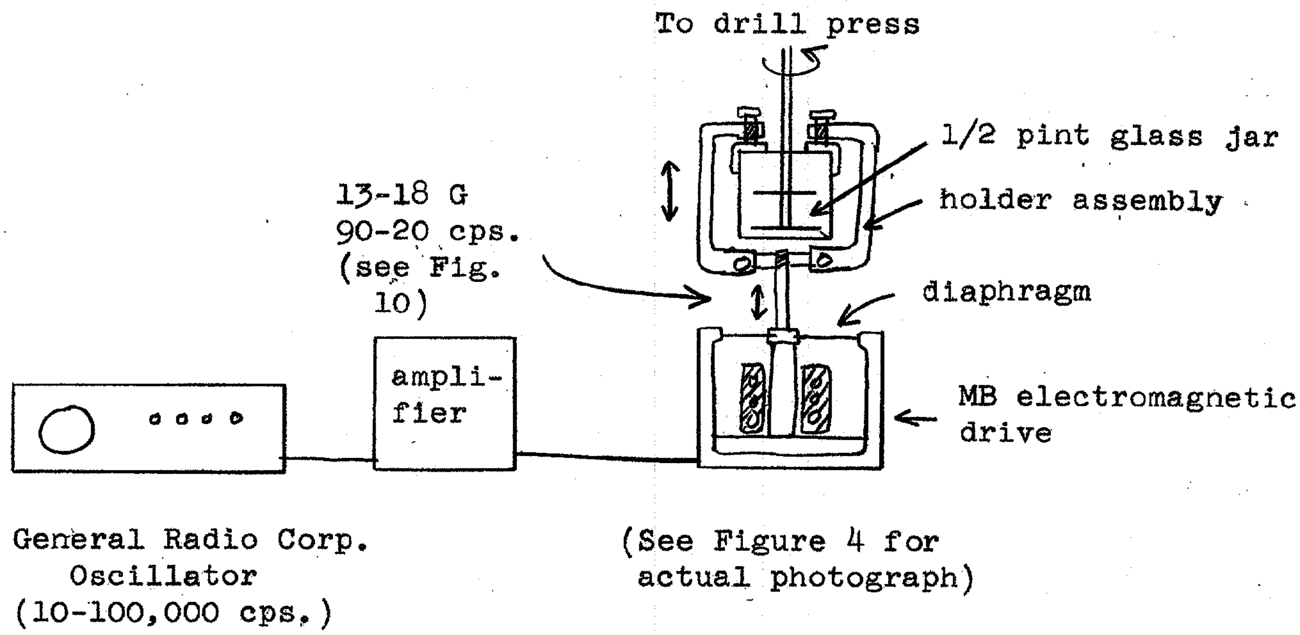
a. <u>913-560 "Harmon" Blue Mill Base</u>

This 913 line dispersion based on Allied Chemical's "Harmon" blue chlorinated copper phthalocyanine pigment is produced for 944-, 945- line automotive refinish. The sprayed panel differs from the regular copper phthalocyanine blue (W-550, for example) pigment in its greenish flip-flop when viewed from the side. As the electron micrographs show (Fig. 2), the system exhibits large areas of flocculation plus some foreign material (Fig. 3) which is not wet completely by the liquid portion of the mill base. Our initial guess on the identity of these dark particles was that they might be sand fragments. However, the presence of this material in the mill base ground with steel shot (Fig. 9) seems to contradict the supposition. Similar particles were observed in a system where we used Du Pont's Monastral® blue phthalocyanine blue (Fig. 7). Pigments Department thinks that they might be small impurities of aluminum sulfate, sodium sulfate or sodium carbonate (Ref. 11) which are used to grind the dried pigment filtrate.

The 913-560 mill base prepared according to the 47 process exhibits a large number of seeds. These seeds are readily perceivable visually as small dots when viewed from the side. Under optical microscope (100X), the seeds appear as small mounds. Currently, we are attempting to take electron micrographs of the cross-section to see what is underneath. These mounds, of course, tend to lower the gloss due to the decreased specular reflection. And, since the gloss measurement is relatively quick and reproducible, this method was ideal to check the effect of vibration.

As will be discussed in detail later, the level of vibrational energy used in experiments described in this report is an order of magnitude less than what is realizable in SoniCo's orboresonance equipment. Therefore, until SoniCo gets ready to perform more meaningful experiments, we decided to examine other variables as well. More specifically, we examined the effects of vertical vibration (Fig.4, see section on orboresonance), (sand/mill base) ratio, quantity of solvent, blade geometry.

A schematic diagram of the vibration assisted 47 process set-up is sketched below.



The mill bases thus obtained were doctor-bladed onto glass slides and examined for gloss and "Colormaster" readings (Table I). The measured values were then analyzed by Yates' method (Ref. 12) to define important variables. It was found that the following variables, shown in the order of effect, had marked influence on mill base quality.

(More sand) > (Vibration + more sand) > (Propeller type blade) > (Vibration) > (More solvent)

Based on this finding, we decided to examine the effect of batch grinding time when the 47 process is carried out with vibration and with more sand (Figures 1 & 5). In Figure 1, ten minutes grinding time is nominally equivalent to a single pass in continuous operation. It can be seen that the 20° gloss of the test case is nearly twice that of the control. This finding is also reflected in the surface roughness depicted in the electron micrographs (Figure 2). The pictures, however, do not reflect marked reduction in the flocculated area. We hope this will be the case when an orboresonance of large magnitude is applied.

In addition, we also examined tinting strength by mixing 5 parts of mill base with 95 parts of 926-93774 white. The mixtures were doctor bladed onto the black ceramic panels. The tinting strength then was compared in terms of the green flectance (Fig. 4, Table 2, Appendix). Yates' analysis again showed that vibration alone or vibration with higher (sand/mill base) ratio gives better quality mill base. The effect of grinding time is shown in Figure 4.

b. <u>901-550 Monastral® Blue Mill Base</u>

Chronologically, this was the first mill base we have tried. This copper phthalocyanine blue differs from the "Harmon" blue in that its sky blue color does not contain the greenish tint. Unlike "Harmon" blue, it also did not have any seeding problem, and the degree of flocculation was much less pronounced (Fig. 7).

In this case, the vibrational energy was also introduced by shaking the can horizontally by using the "Vibromixer" which was available from PLE. Attempts were made to analyze the result by viewing the diluted mill bases with 560X optical microscope. But with this method, the difference between the mill bases prepared with and without vibration could not be differentiated.

We also letdown this mill base according to 868-0550 formulation to determine hiding power by preparing wedge sprays. Again, the mass tone color was rather deep and did not show the effect of vibration.

c. 907-686 Chrome Yellow Mill Base

Of all the systems tried, this lead chromate mill base was by far the easiest to grind. In fact, even the raw mill base without any sand grinding exhibited Brownian motion under optical microscope. This is reflected in the electron micrograph of the mill base cross-section (Figure 8).

The mill bases prepared with horizontal vibration, however, were sent to G. Orvis of Flint for his evaluation. He concluded that vibration was beneficial in obtaining higher tinting strength and cleaner color. Our analysis verified his findings as shown below. Reflectance readings are shown in Table 4.

Panel	Adam's Color Coordinates L	a	b
Control 1	93.69	-8.02	56.02
Control 2	91.19	2.98	51.54
With Vib. at 60 cps.	90.21	2.94	54.55
With Vib. at 150 cps.	90.04	2.96	54.79

We also subjected this mill base to ultrasonic vibration (20,000 cps.), using R. Prugh's equipment at Jackson Lab., Orchem. Again, the optical microscope observation showed little difference although the settling rate of the sample with ultrasonic vibration was markedly slower than the control.

d. 907-582 "Milori" Blue Mill Base

This is the ferric-ferrocyanide mill base which is normally ground in ball mills. In this study, however, we used 47 process with and without vertical vibration. The effect of different kinds of grinding media was also studied (Table 4). It can be seen that change of sand to steel shot, "Ceramedia" glass slags and "Zircoa" ZrO_2 gave pooer results. Vibration at 50 cps., however, seemed to improve the situation.

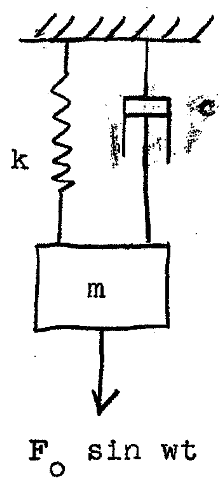
In general, however, the seeds observed in all the runs were so severe that ~~none of the~~ variables seemed to have any effect. We, therefore, changed our emphasis to the "Harmon" blue system which showed much less degree of seeding.

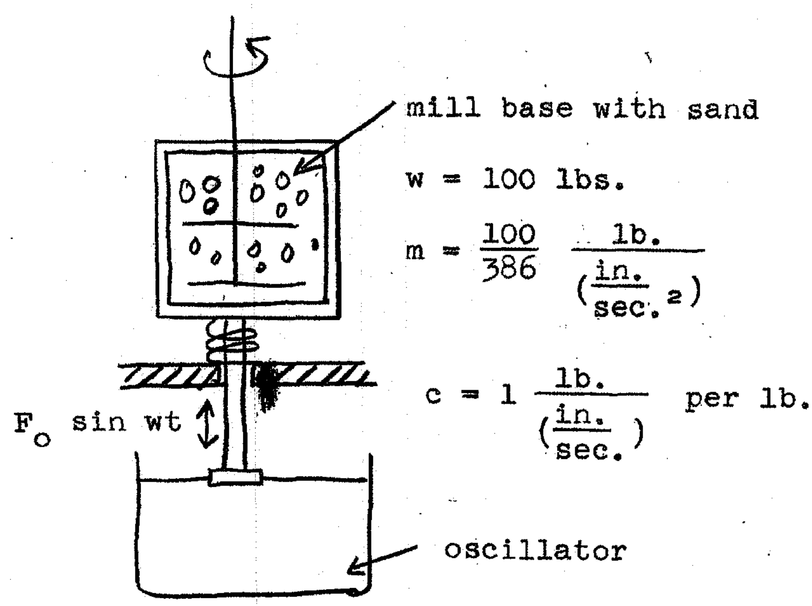
2. <u>Orboresonance Technology</u>

The word, orboresonance, describes the response of a system with elastic element to a cyclic, oscillatory force. The response is characterized by large amplitude at high frequency. In the case of nonresonating system, the amplitude decreases considerably as we increase the frequency (Fig. 10).

The starting point to deduce the above statement is the well known governing equation of the mass-spring-damper system shown below subjected to sinusoidal forcing function (Ref. 13).

$$m \frac{d^2x}{dt^2} + c \frac{dx}{dt} + kx = F_o \sin wt \qquad (1)$$



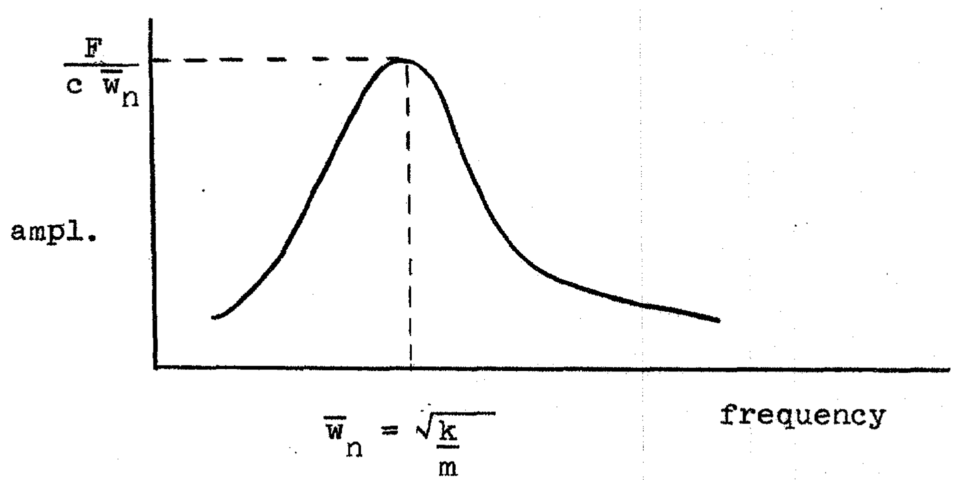


t = time
x = displacement
w = oscillation frequency in radians
c = dampening coefficient
k = spring constant of the elastic element
m = mass of the system in lb./(in./sec.^{2}) units
F_o = maximum amplitude of forcing function

The right-hand diagram shown above depicts the model of our sand grinder with some hypothetical numbers for the purpose of illustration. An elementary solution of the governing equation gives:

$$\left(\begin{array}{l}\text{Amplitude of}\\ \text{responding}\\ \text{system}\end{array}\right) = X = \frac{F_o/k}{\sqrt{\left(1-\frac{mw^2}{k}\right)^2 + \left(\frac{cw}{k}\right)^2}} \qquad (2)$$

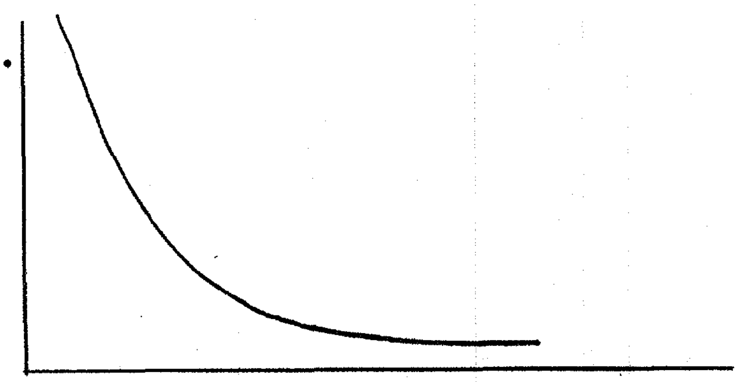
Plot of equation 2 is shown below.



If we do not have an elastic element, (k = o), the above equation becomes:

$$X = \frac{F_o}{w\sqrt{m^2w^2 + c^2}} \qquad (3)$$

Therefore, if m is large, X becomes small unless F_o is very large.



The actual measurements of our experimental unit are shown in Figure 10. It can be seen that the natural frequency of the system was found to be at 30 cps. which is too low. One way to get around this is to increase k which means a stiffer spring, which in turn requires a stronger oscillator to drive the system.

The acceleration, measured in terms of G unit which is 386 (in./sec.^{2}), can be obtained from the following relation which can be easily derived.

$$(\text{acceleration}) = \frac{d^2x}{dt^2} = -(2\pi f)^2 x \qquad (4)$$

where f = frequency in cps.

By measuring amplitude and frequency, therefore, the G forces can be computed. The calculated G forces for our system showed that our unit can generate 18 G at best (Fig. 10).

The present unit, therefore, is a rather feeble device compared to the SoniCo's 100-500 G, 50-350 cps. unit. All arrangements have been made to run the experiment at SoniCo within two months.

To describe the resonance vibration more fully, one also needs to know the method of determining dampening coefficient, power consumption and Q calculation which determines average energy stored per energy throughput per cycle. I have learned these techniques during my visit to SoniCo. Details of these calculations will be described in the next report along with the actual orboresonance experimental results.

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TABLE 1

FACTORIAL DESIGN WITH 913-560 SYSTEM TO DETERMINE IMPORTANT VARIABLES

Order of Exp.	Design. 407E-33-	Vibration	(Sand / Mill Base)	Solvent Quant.	Blade Geom.	Final Temp.	Solid %	Gloss 20°	Gloss 60°	"Colormaster" Reflectance Blue	Green	Red
1	ab	Yes	1.3	Normal	Flat	--	35.0	31.9	71	64.52	38.65	28.12
2	abd	Yes	1.3	N	Propeller[2]	72°C	37.9	33.1	75	65.06	39.79	29.15
3	bc	No	1.3	20% more	F	45	28.3	20.8	64	65.17	40.44	30.29
4	a	Yes	1.0	N	F	--	33.8	25.2	67	65.40	40.34	29.95
5	acd	Yes	1.0	20%	P	50	29.3	17.1	58	65.03	40.02	29.72
6	ad	Yes	1.0	N	P	50	36.8	23.5	63	65.56	40.94	30.70
7	b	No	1.3	N	F	52	35.4	33.0	71	65.00	39.70	29.26
8	d	No	1.0	N	P	53	33.6	25.4	66	65.11	40.11	29.76
9	bcd	No	1.3	20%	P	47	29.5	26.1	67	65.20	39.95	29.46
10	abc	Yes	1.3	20%	F	--	28.9	23.6	66	64.97	39.54	29.07
11	c	No	1.0	20%	F	--	28.6	16.8	58	64.73	39.79	29.58
12	abcd	Yes	1.3	20%	P	51	29.0	23.2	64	64.66	39.28	28.85
13	cd	No	1.0	20%	P	50	28.5	16.6	59	66.04	42.10	31.97
14	(1)[1]	No	1.0	N	F	--	33.3	25.5	68	64.90	39.46	29.04
15	bd	No	1.3	N	P	51	33.6	30.2	73	64.66	39.68	29.35
16	ac	Yes	1.0	20%	F	--	28.6	13.7	56	65.22	40.50	30.30

[1] Control

[2] See 407E-34 for dimensions.

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TABLE 2

EFFECTS OF VIBRATION AND (SAND/MILL BASE) RATIO ON TINTING STRENGTH AND COLOR PARAMETERS FOR 913-560 BLUE MILL BASE[1]

Experiment	Grind. Time	Sand/Mill Base	Adams Coordinate Changes[3]			
			ΔL[2]	Δa	Δb	ΔE
407E-50-a	5 min.	1.0	-0.35	-0.18	-0.45	0.60
"	10 min.	1.0	-0.23	+0.05	-0.47	0.53
"	15 min.	1.0	+0.17	+0.58	+0.18	0.63
407E-50-ab	5 min.	1.3	-0.6	-0.82	-1.03	1.45
"	10 min.	1.3	-0.38	-0.19	-0.73	0.85
"	15 min.	1.3	-0.59	+0.12	-0.63	0.87
407E-50-abb	15 min.	1.5	-1.25	+0.20	-1.34	1.84
407E-50-abb	15 min.	2.0	-0.83	+0.43	-0.75	1.21

NOTES:

[1] Marshall Lab computer program ML-1104 was used.
[2] Negative changes in ΔL, Δa and Δb means higher tinting strength, greener and bluer color respectively. ΔE = total color difference in NBS units. According to ASTM D2244-64T, repeatability of these measurements are within 0.1 unit.
[3] In every run, the sand mill was vertically vibrated at 60 cps., 15 G acceleration. The control runs on the other hand were carried out without vibration.

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TABLE 3

EVALUATION OF 913-560 "HARMON" BLUE MILL BASE. EFFECT OF VIBRATION AND GRINDING MEDIA[1]

Run	Grinding Media	Vib. Freq.	Solid %	20° Gloss	"Colormaster" Reading Blue	Red	Green[3]	Comments
407E-6 Sd	Sand	No vib.	--	22.8	65.37	31.76	20.24	Control
			35.7%[2]	21.7				
407E-6 St	Steel Shot	" "	--	27.7	64.20	32.02	20.74	
			35.7%	24.7				
407E-6Z	"Zircoa"	" "	--	17.6	---	---	---	
407E-6C	"Ceramedia"	" "	--	17.6	---	---	---	
407E-6V40	Sand	40 cps.	45.3	33.5	66.24	32.42	20.85	
		" "	35.7	30.2				
407E-6V40AB	"	" "	42.4	34.9	66.23	32.24	20.64	5% to the pigment of PCl-Dteta added.
			35.7	33.0				
407E-6V50	"	50 cps.	43.6	34.4	65.92	32.35	20.94	
			35.7	27.0				

NOTES:

[1]The mill bases were ground for 30 min. at 2000 rpm.

[2]Samples adjusted to 35.7% by adding Lucite® solvent.

[3]Smaller value indicate high tinting strength.

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TABLE 4

EVALUATION OF VARIOUS MILL BASES TO FIND A GOOD CANDIDATE FOR VIBRATION ASSISTED 47 PROCESS GRINDING

Run	System	Pigment	Grind Media	Vib. Freq. (cps.)	Ampl. (mm)	Grind. Time (min.)	Gloss 20°	Gloss 60°	"Colormaster" Reading[1] Blue	Green[2]	Red	Optical Observat. Brown. Mot.	Gloss	Comments
379E-198	(907-686) (926-0686)	Chrome Yellow	Sand	No vib. (control)		20	66	82	30.04	81.04	95.81	Yes	No	Letdown blended with white. Sprayed, baked.
			"	60	2.9	20	64	81	27.15	79.18	96.11	"	"	
			"	150	Not det.	20	65	81	26.82	78.81	93.75	"	"	
407E-1	907-582	"Milori" Blue	"	No vib. (control)		30	46.4	76.5				No	Yes	Many small seeds.
			Steel Shot	" "	--	30	39.4	70				"	"	V. many large seeds.
			"Ceramedia"[3]	" "	--	30	39.9	58.5				"	"	V. many med. seeds.
			"Zircoa"	" "	--	30	35.7	68				"	"	Same as steel shot case.
				30	4.5	30	51.2	75				"	"	Many small seeds.
			"	40	2.5	30	47.4	74				Yes	"	Many large seeds.
			"	50	1.5	30	53.2	76				"	"	V. small seeds, not many.
407E-4	901-6253	Black	Sand	No vib. (control)		30			30.66	28.40	27.27	Slight	"	
			Steel Shot			30			30.53	28.44	30.53	"	"	V. Slow in settling when diluted.
			Sand	40		30			32.54	29.94	28.72	"	"	
			"	50		30			31.17	28.96	27.84	"	"	
379E-177	868-0550	Monastral® Blue	No Brownian motion. Flocs were noticeable. See notebook for details.											
379E-186	868-0550 901-550	" "	Four mill bases with horizontal vibration of 50, 100, 150, 1500 cps. showed no improvement in gloss, degree of flocculation as observed by optical microscope, G reflectance, and hiding power as determined by wedge spray.											

NOTES:

[1] Mill bases were mixed with suitable white paints such as 926-0131 in 1/9 or 5/95 proportion for reflect. measurement.

[2] Smaller value indicate high tinting strength.

[3] Trademark of Quackenbush Co. "Zircoa" = ZrO_2, "Ceramedia" = spherical glasses and glass like slags.

YA/rsh
9/24/68

FIGURE 1

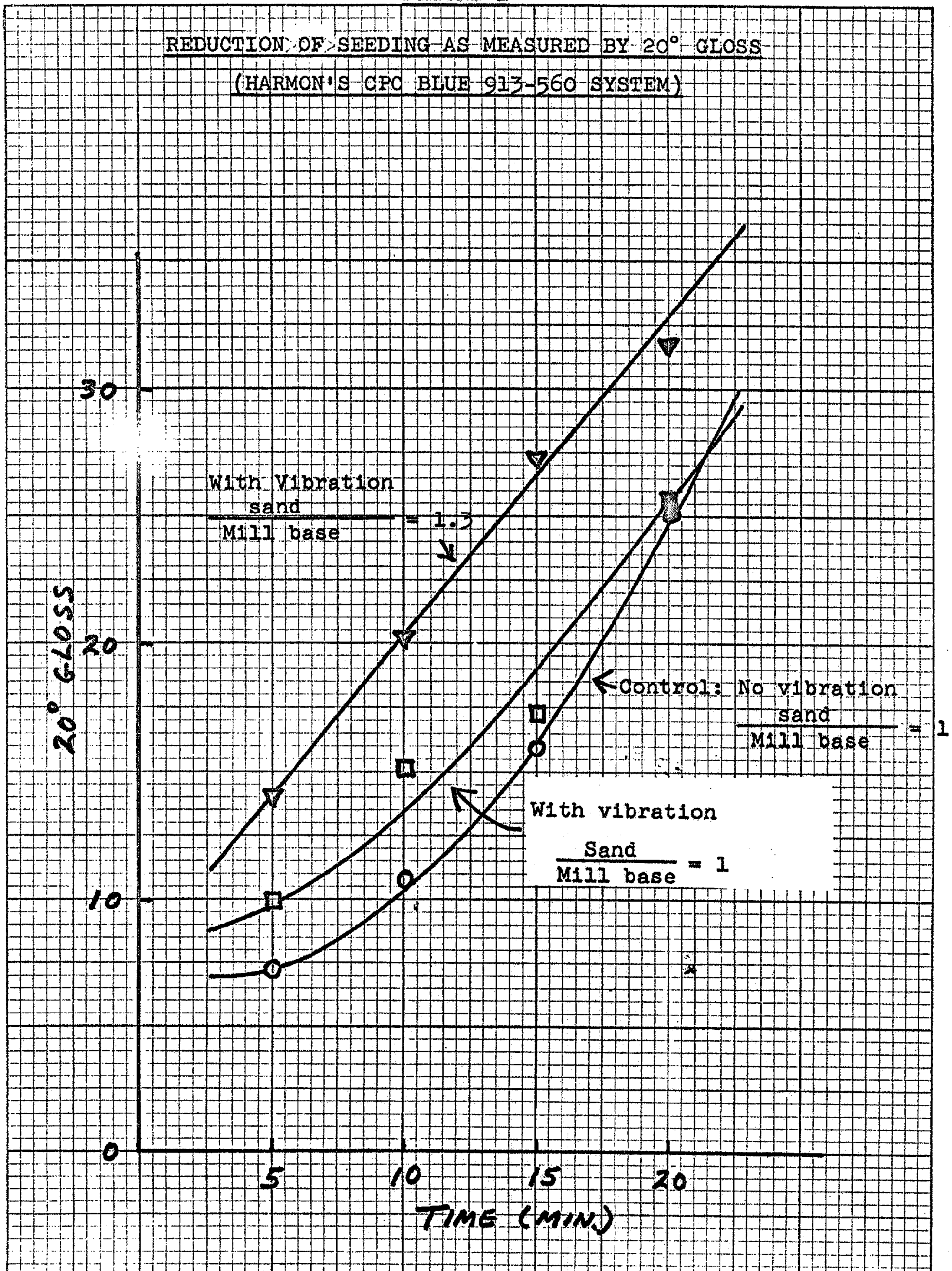

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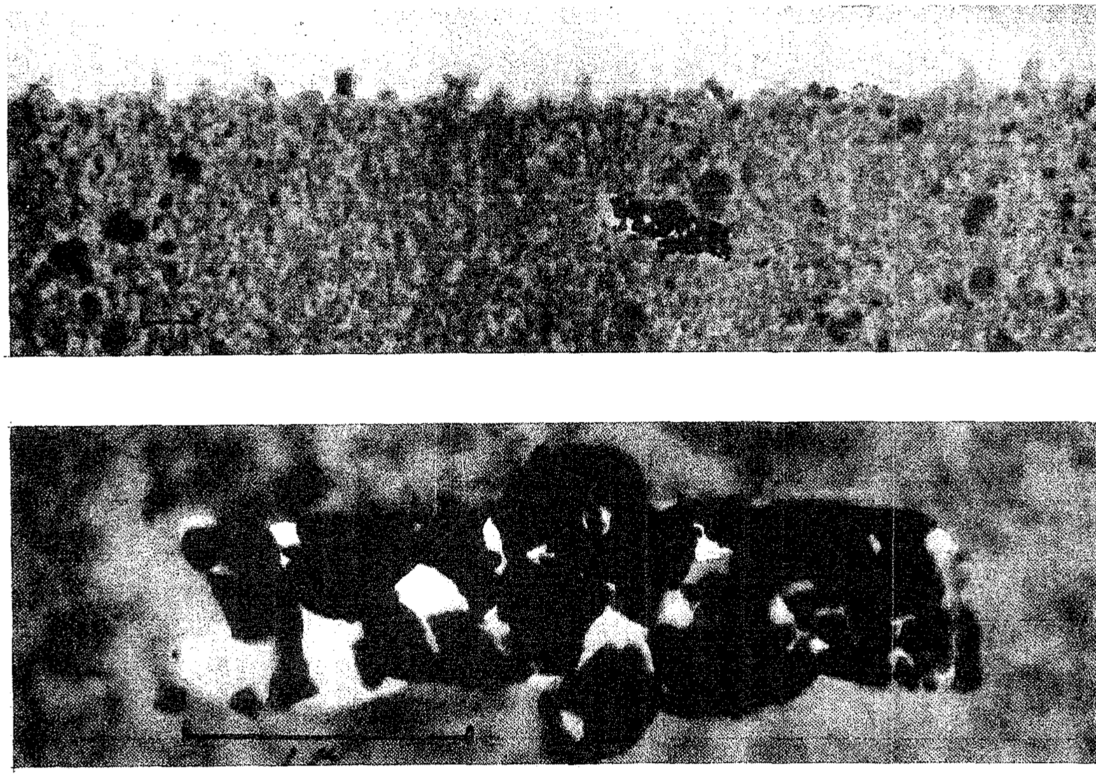
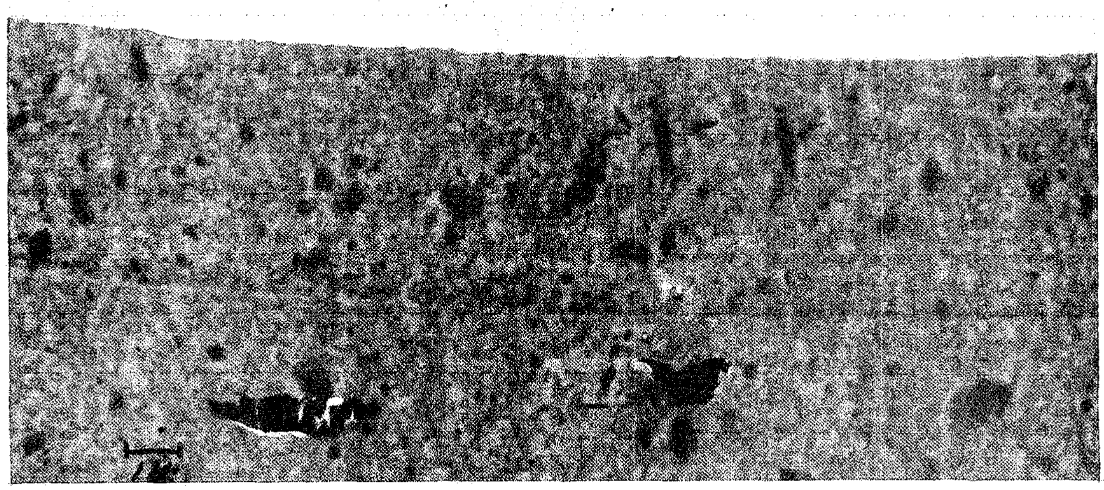
FIGURE 2

ELECTRON MICROGRAPHS OF 913-560 CPC HARMON® BLUE UNBAKED MILL BASES FILM CROSS SECTION

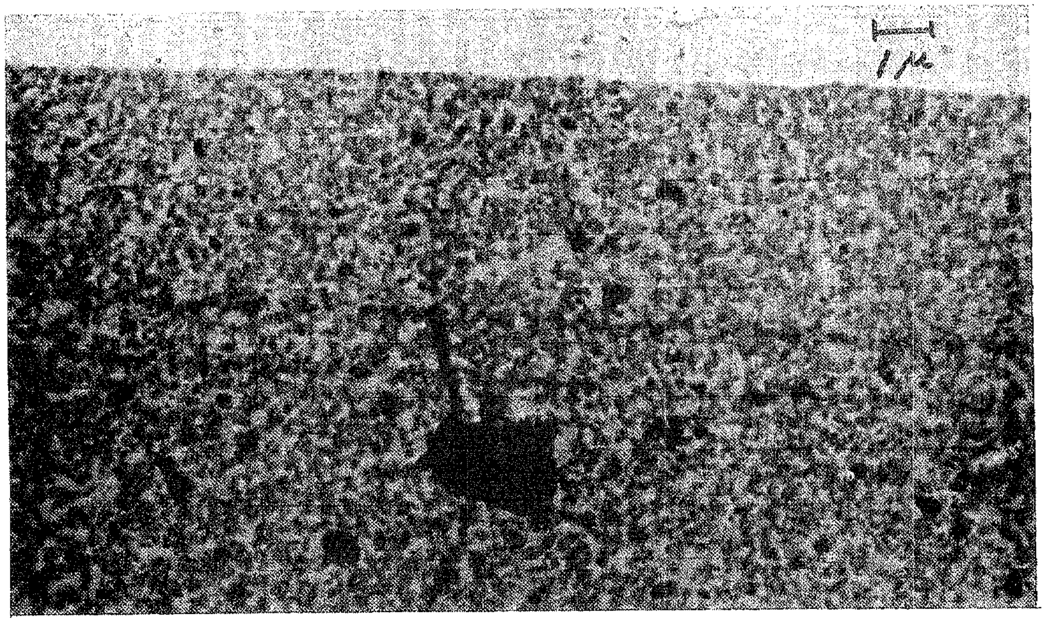


Mill base prepared without vibration.
(Run 407E-38-(1), Control, 20° Gloss = 25.5)

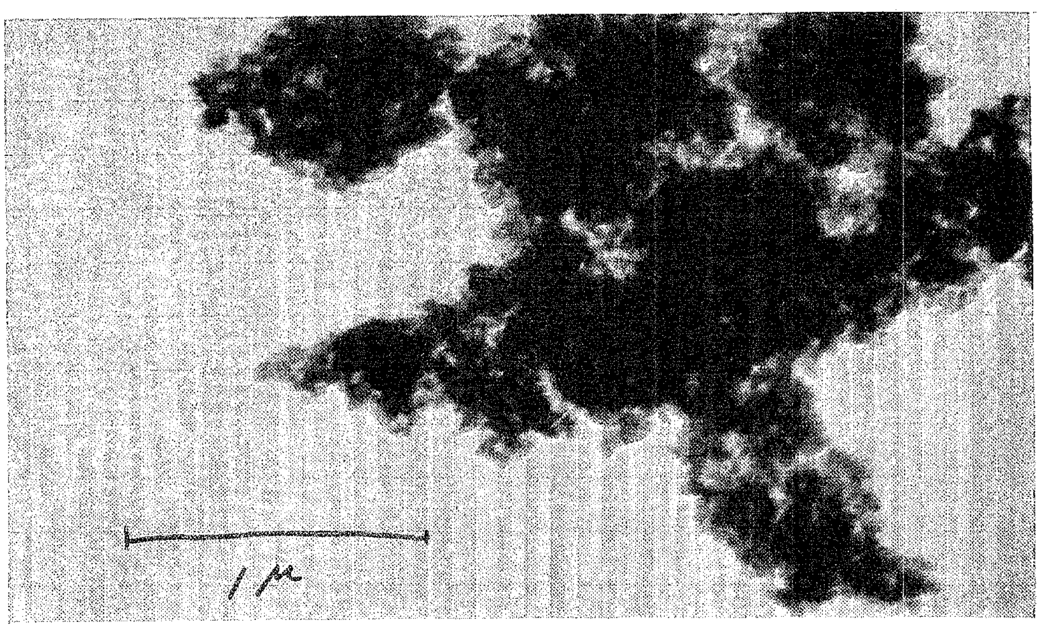


Mill base prepared with vibration and more sand.
(Run 407E-38-ab, 20° Gloss = 31.9)

FIGURE 3



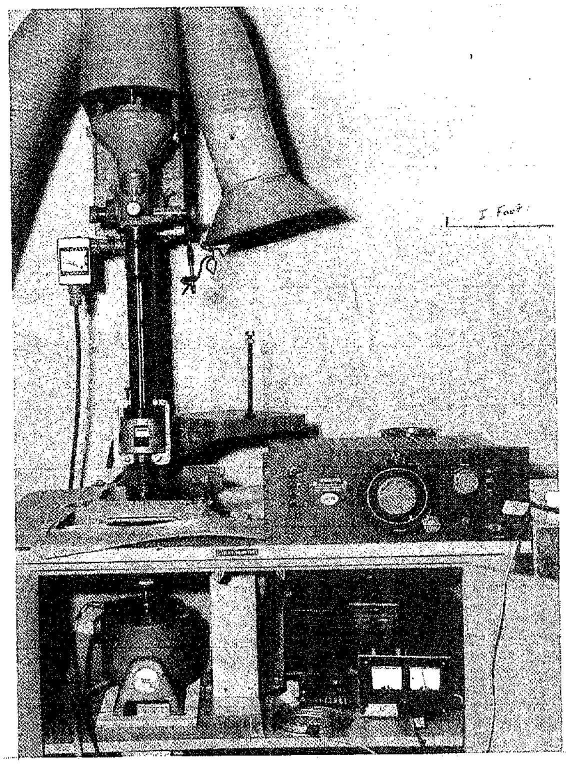
Harmon's blue letdown (901-G-24039) of Flint Dev. Lab. Note the large dark agglomerate.



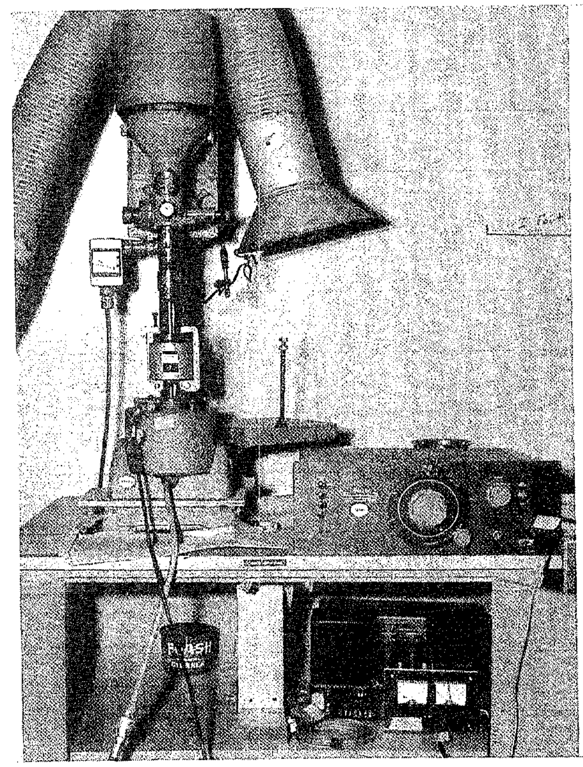
Electronmicrograph of the dry Harmon's blue pigment. Note the darker particle.

FIGURE 4

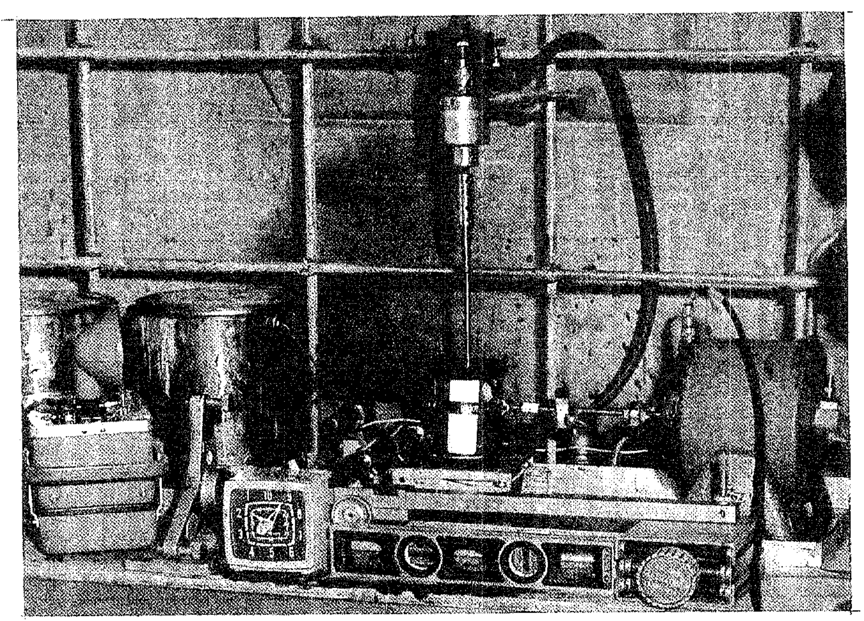
VIBRATION ASSISTED MILL BASE GRINDING



Vertical vibration with
elastic element (steel spring)



Vertical vibration without
elastic element



Horizontal vibration (Earlier runs)

FIGURE 5

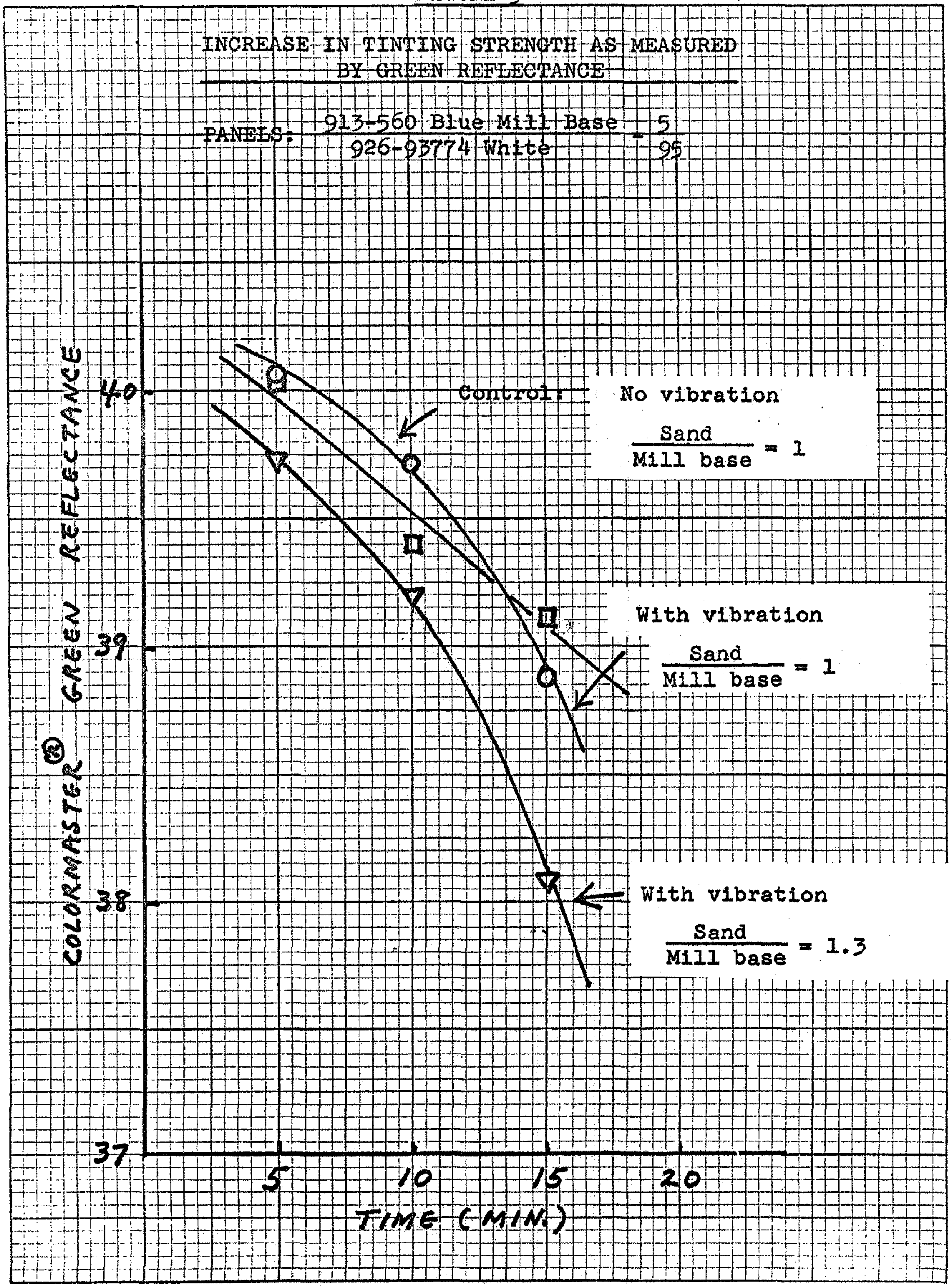

INCREASE IN TINTING STRENGTH AS MEASURED
BY GREEN REFLECTANCE
PANELS: 913-560 Blue Mill Base - 5
926-93774 White - 95
Control: No vibration
Sand / Mill base = 1
With vibration
Sand / Mill base = 1
With vibration
Sand / Mill base = 1.3
COLORMASTER® GREEN REFLECTANCE
40
39
38
37
5
10
15
20
TIME (MIN.)

FIGURE 6

EFFECT OF MORE SAND IN VIBRATION ASSISTED 47 PROCESS

(HARMON'S CPC BLUE 913-560 SYSTEM GROUND FOR 15 MIN.)

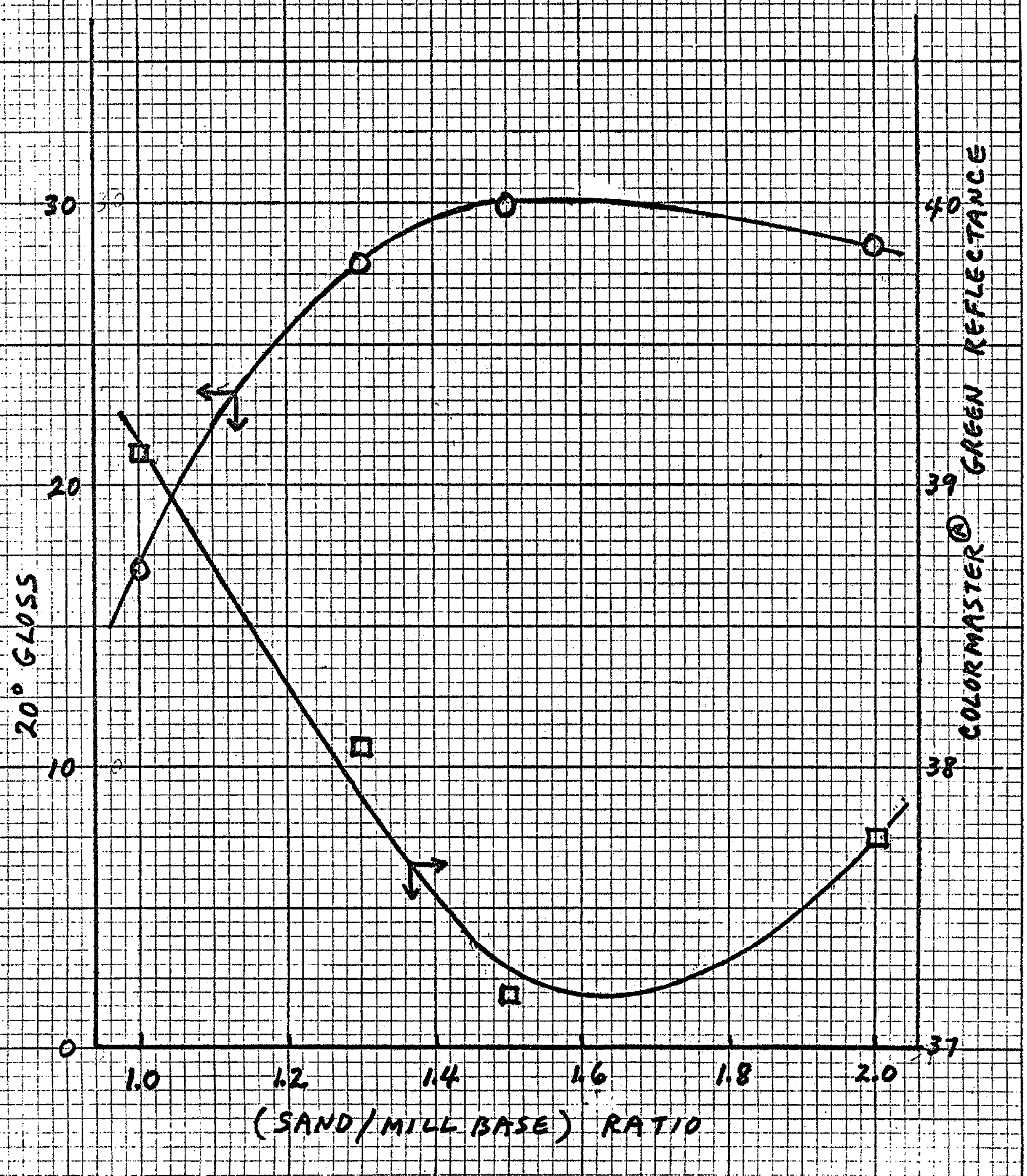
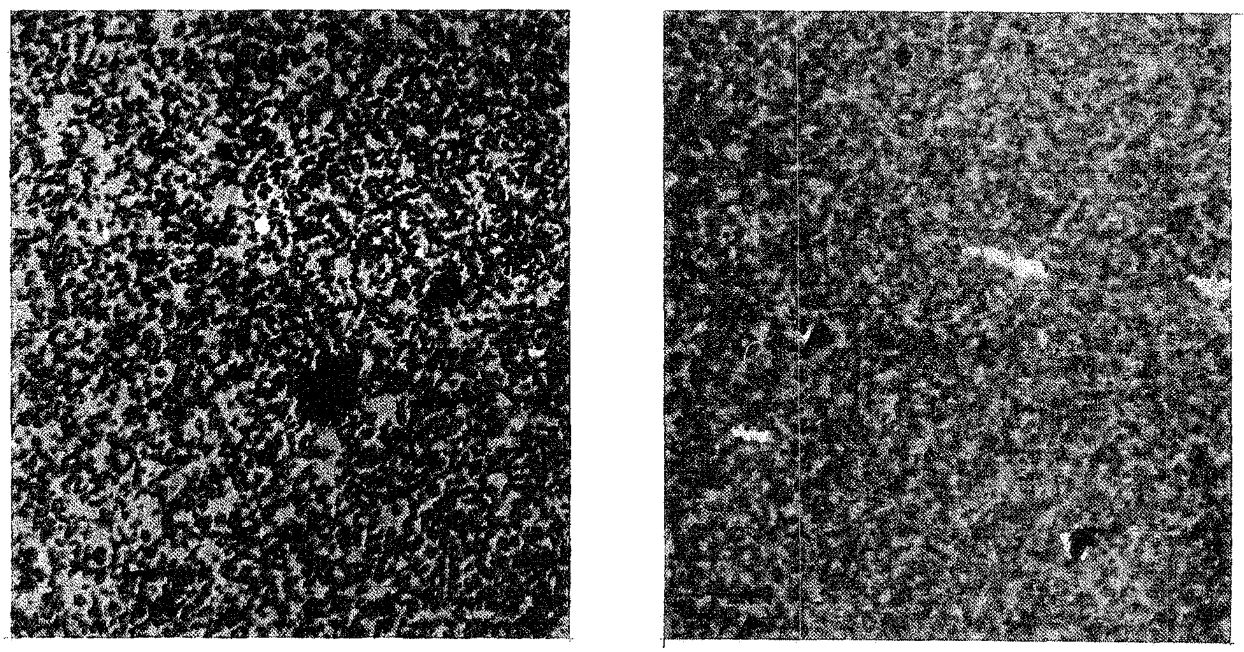


FIGURE 7

AIR-DRIED, MILL BASE FILM CROSS SECTION OF 901-550 MONASTRAL® BLUE (RUN 379E-190). THE DEGREE OF DISPERSION IS BETTER THAN THAT OF FIGURE 2



<u>FIGURE 8</u>

MILL BASE CROSS SECTION OF 907-686 CHROME YELLOW (RUN 379E-198). ONLY ONE LARGE PARTICLE SHOWN ABOVE WAS FOUND IN 30 X 50μ^2 AREA, INDICATING GOOD DISPERSION

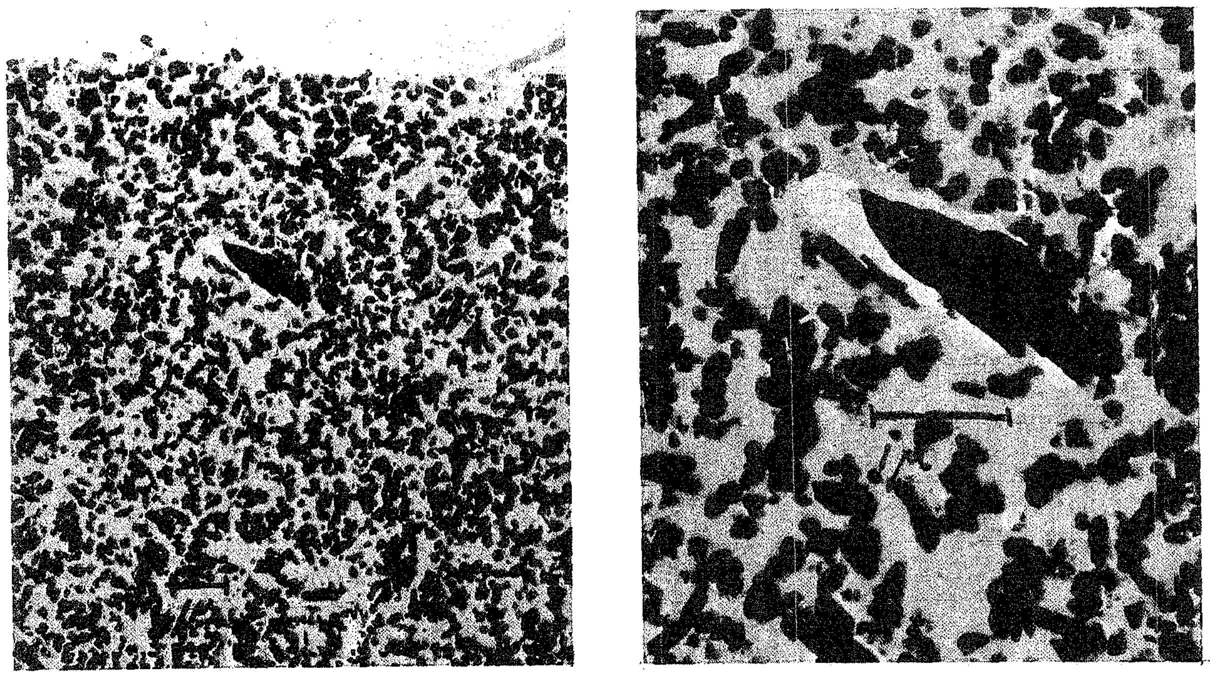
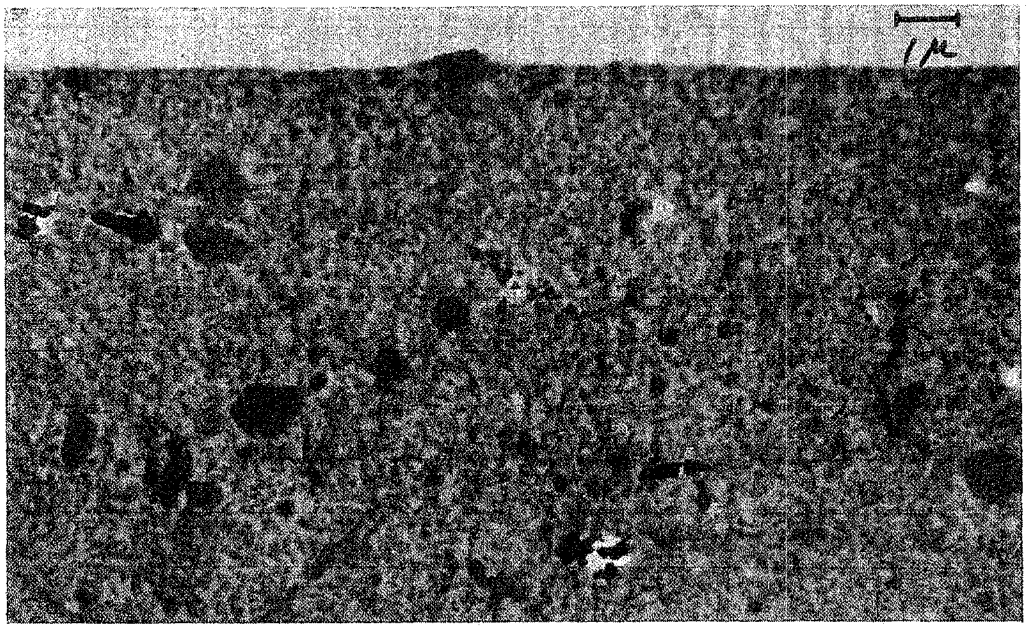
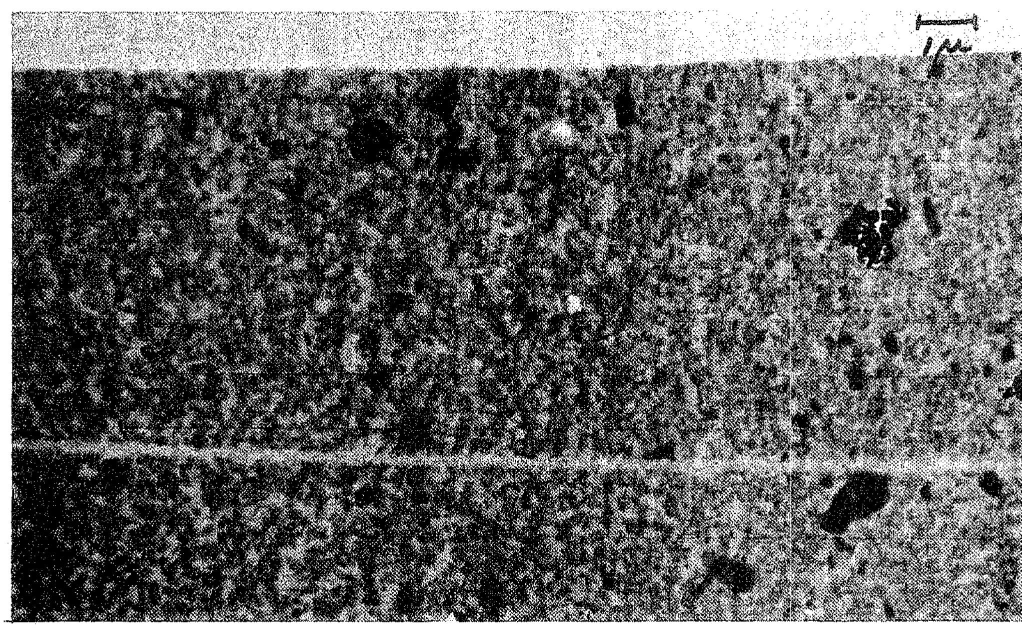


FIGURE 9



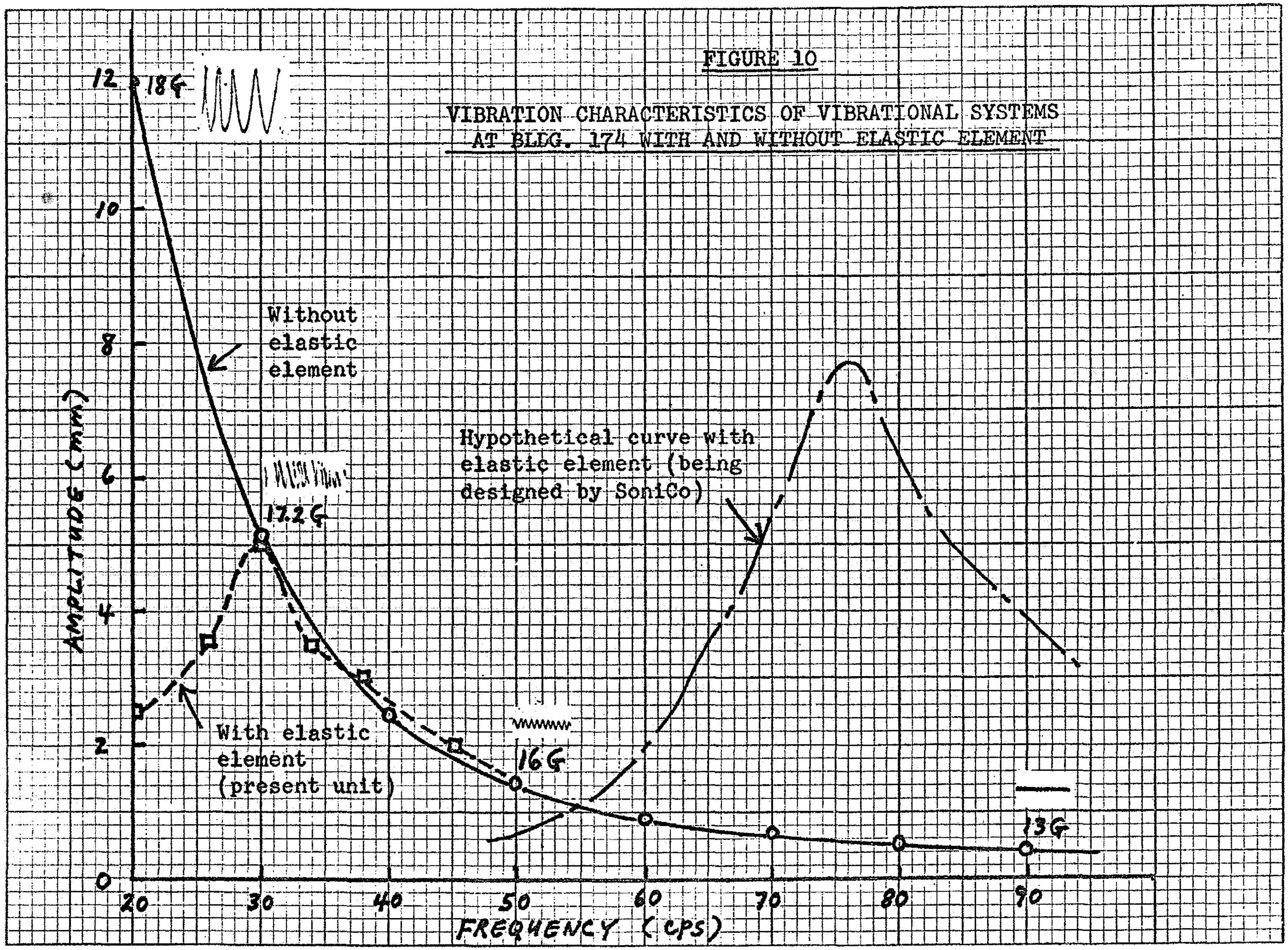
Harmon's blue mill base ground with -20 + 25 mesh steel shot without vibration (407E-6 Steel)



Harmon's blue mill base ground with PC1-D teta A-B dispersant with vibration (407E-6 V40AB)

FIGURE 10

VIBRATION CHARACTERISTICS OF VIBRATIONAL SYSTEMS AT BLDG. 174 WITH AND WITHOUT ELASTIC ELEMENT



APPENDIX

The following equations relating G, R, B values of the "Colormaster" differential colorimeter to the a, b, L values were supplied by N. Komodromos of Flint Laboratory. Computer program ML1104 was used by L. Lenhart of Marshall Laboratory to perform the conversion.

$$\text{Lightness} = L = 25.29\ G^{1/3} - 18.38$$

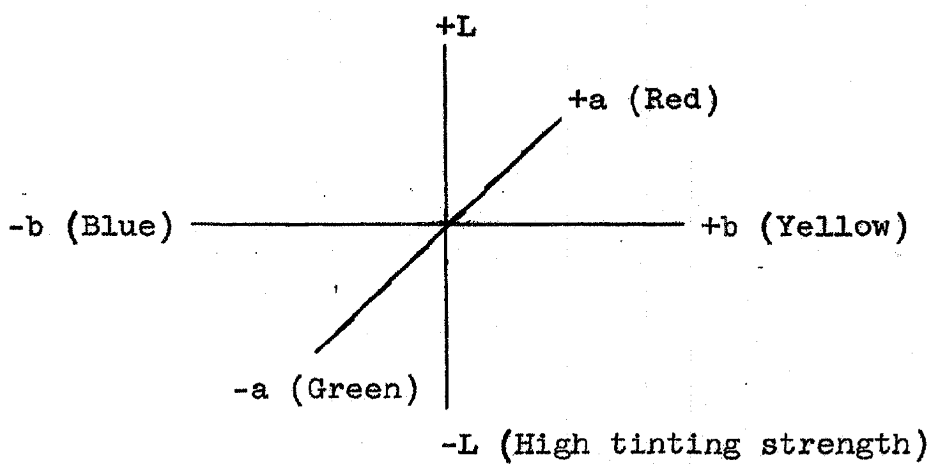
$$R^1 = 0.8\ R + 0.2\ B$$

$$a = 106.0\ (R^{1\ 1/3} - G^{1/3})$$

$$b = 42.34\ (G^{1/3} - B^{1/3})$$

$$\Delta a = a_{sample} - a_{std.}$$

$$\Delta E = (\text{Total color difference} = (\Delta L^2 + \Delta a^2 + \Delta b^2)^{1/2}\ \text{in NBS units})$$



ABSTRACT

Vibration assisted "47 Process" sand milling is described. The range of vibration imposed was 13-18 G at 40-60 cps. In particular, a mill base with "Harmon" copper phthalocyanine blue was investigated in detail. The results showed significant increase in gloss and tinting strength. The effect of denser grinding media also was briefly studied.

This study was conducted to obtain preliminary evidence of the beneficial effects of orboresonance assisted pigment milling. With orboresonance, one can generate 150-480 G at 60-350 cps. The principle of orboresonance is discussed briefly in the report.

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