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FABRICS & FINISHES DEPARTMENT

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PARLIN LABORATORY

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SUBJECT: INVESTIGATION OF "47 PROCESS" AS APPLIED TO "DUCO" MANUFACTURE

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CONFIDENTIAL

PARLIN LABORATORY REPORT NO. 977

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INTRODUCTION

The "47 Process" was developed at the Philadelphia Laboratory during the latter part of 1946. The term "47 Process" is used to designate the grinding and dispersion of pigments in vehicles involving the use of very small grinding media such as sand in an agitated vessel. Data obtained at Philadelphia on their work with "DULUX" (1) have indicated the following advantages of this process over the standard ball grind: improved quality, improved service through decreased cycles, lower initial investment required, and lower power costs.

The late Mr. John Marshall in his letter of April 1, 1948 to Mr. G. E. Conde asked that a brief preliminary study of the "47 Process" be conducted by the Parlin Laboratory with a view to exploring its possibilities particularly as a means of supplementing W & P capacity. As outlined by Mr. Marshall, the primary purpose of the laboratory work would be to acquire data which could subsequently be used by the Parlin Process and Methods Group to apply "47 Process" techniques to the grinding of "Duco".

A rather extensive study was made to investigate "47 Process" as a possible substitute for current vacuum W & P dispersions. Pigments currently dispersed by a ball mill were studied to a lesser extent. The subject process does not appear to offer economic advantages over the "49 Process"; therefore, enamels which can easily be adapted to the latter method of manufacture were not included in this study.

The experimental work in connection with this study is divided into eight sections.

A. Dispersion of Maroon Toner (W-896).

B. Dispersion of Milori Blue (W-582).

C. Evaluation of 60-80 Mesh Sand.

D. Dispersion of highly pigmented products.

E. Manufacture of single pigment tinting enamels currently dispersed in the ball mill.

F. Application of "47 Process" technique to pigmented EPON finishes.

G. Dispersion of Santocel "C" (G-222).

H. Evaluation of Teflon grinding discs.

SUMMARY AND CONCLUSIONS

1. "47 Process" dispersions of W-582 Milori Blue and W-896 Maroon Toner into "Duco" formulations compare favorably in gloss and color characteristics with corresponding ball mill dispersed enamels. However, attempts to obtain the jetness of tone and freedom from haze, which are possible by pulp process dispersion were unsuccessful.

2. In the grinding of Milori Blue and Maroon Toner pigments, faster grinding rates and slightly improved color were obtained by using 60-80 mesh sand in place of 20-30 mesh sand commonly used in "47 Process" work.

3. Steel shot appear to be more effective than sand in grinding certain highly pigmented products.

4. Laboratory work to date indicates that single pigment "Duco" formulations currently dispersed in the ball mill can be dispersed by the "47 Process" to give favorable gloss, color, and durability characteristics.

5. The "47 Process" appears to give satisfactory dispersion of pigments into EPON Resin finishes.

6. Satisfactory laboratory scale dispersion of Santocel "C", a silica aerogel, were made by the "47 Process" using glass equipment.

7. On the basis of limited tests, grinding discs made from Teflon appear to give favorable resistance to wear in the subject process.

ACTION TAKEN

Parlin Laboratory Release Letter No. 380 was written suggesting that the factory purchase and install a semi-works unit. The Parlin Plant now has a 23 gallon "47 Process" unit in which the Process and Methods Group are applying laboratory findings on a plant production scale.

PATENT INFORMATION

The "47 Process" is embraced by patent application Hochberg FFD-1509. One Office Action has been received to date and the cited reference art was the same as that known at the time of filing. The use of "Teflon" discs may represent a patentable improvement, but it appears premature to initiate patent action in this connection.

EXPERIMENTAL

A. Dispersion of W-896 Maroon

Maroon Toner is currently dispersed into lacquer vehicles by the pulp process. This process gives a very deep clean tone which in the past has not been duplicated by ball mill dispersion techniques. Considerable savings in the cost of manufacture could be effected if the "47 Process" could be utilized for the dispersion of this pigment.

The Maroon Toner pigment was ground in (1) dibutyl phthalate, blown castor oil, and solvents; (2) alkyd resin and solvents; (3) blown castor oil, dibutyl phthalate, nitrocellulose solution, and solvents; (4) alkyd resin, nitrocellulose, and solvents; and (5) the entire nitrocellulose, solvent, resin, and plasticizer content. Make-ups of the dispersions were compared with ball mill dispersed enamels and with pulp process enamels.

The mill bases which did not contain nitrocellulose gave light, dull, and dirty colors when made into 253-033M Maroon Tinting Enamel. Color and gloss improved as the nitrocellulose content of the mill base was increased. Also the heat generated by the sand grinding increased with the nitrocellulose content. Formulations in which the pigment was ground in solvent, nitrocellulose solution, and plasticizer, as well as those ground in solvent, nitrocellulose, and resin gave better gloss and color characteristics than did ball mill dispersions. However, the depth of tone which can be obtained by the pulp process could not be approached by "47 Process" dispersion.

A brief study of the effects of varying grinding cycles indicated that gloss improves and color becomes cleaner as grinding cycles are lengthened. The jetness of tone appears to reach a maximum with approximately 1 hour's grinding at 2350 r.p.m. using a 50/50 volume ratio of sand to mill base. Beyond this optimum grinding cycle a lighter and redder tone is produced.

At the request of the Parlin Plant, the subject process was compared with ball mill grinding as a method of reworking maroon enamels which were low in gloss. "47 Process" grinds were made using a 50/50 volume ratio of sand to the maroon tinting enamel. Although satisfactory gloss could eventually be obtained by both methods, neither process gave a color which would meet the requirements of 253-033M Maroon Tinting Enamel.

As shown in Figure 1 of the Appendix, the rate of grinding increased as the agitator speed was increased. Also a considerably higher rate of dispersion was obtained in the "47 Process" than in the ball mill.

B. Dispersion of W-582 Milori Blue

The current method of dispersing Milori Blue pigment by the pulp process involves the separation of water from the WX stock by vacuum distillation. This process affords a comparatively costly method of manufacture and introduces a slight fire hazard. If the "47 Process" could be used to disperse pigments of this type, savings in manufacturing costs could be realized and critical W & P equipment could be released for other uses.

The laboratory devoted a great deal of work to the determination of the mill base composition which appeared to give the best dispersion of Milori Blue pigment. The results are given in Table I of the Appendix.

Pronounced differences in the color and gloss of the finished products resulting from the various mill base compositions were noticed. As the dispersions all used a constant ratio of sand to mill base, and constant agitator speeds, it appears that the grinding vehicle composition greatly influences pigment dispersion. The formulation which appeared to give the best overall color and gloss characteristics consisted of blown castor oil, dibutyl phthalate, pigment, and solvent. This formulation was used in subsequent experiments to determine the optimum viscosity and grinding time for Milori Blue dispersions.

Samples were taken at intervals of grinding and made into finished product. Sprayouts of the resulting 253-051 Milori Blue lacquer showed that the color is a reddish blue at the beginning of the grind period. At the beginning, as grinding proceeds, the color becomes darker and bluer. If grinding, is extended for a long period of time, however, the color changes toward a light blue. With good mixing action, about 40-60 minutes appears to be the optimum grinding time in laboratory batch process equipment.

Particle size measurements of certain formulations were made by using an electron microscope. From Table I it can be seen that some relationship seems to exist between particle size and color and gloss characteristics. Particles near to or larger than 1 micron tend to impair the gloss of a Milori Blue lacquer. Agglomerates slightly smaller appear to scatter light and produce a lighter color. The deep jet blue tone which is desired in Milori Blue lacquers appears to be produced by unit particles. The tendency for these lacquers to become lighter on extended sand grinding might be explained partially on the basis that extended grinding gives a larger proportion of agglomerates of such size that they exert a pronounced light scattering effect.[3] Undoubtedly the contamination caused by fragments from the sand and the mixing equipment also contribute to the lighter color. Contamination was further studied in the evaluation of Teflon discs.

The first grinds made in castor oil, dibutyl phthalate, and solvent were of very low viscosity (about 19 centipoises). The Philadelphia Laboratory reported that their best results were obtained using viscosities, slightly less than 500 centipoises. Viscosities of grinds in the Parlin experiments were varied by eliminating solvents from the grind portion. The composition and viscosities of the mill bases and the color and gloss of the finished product are given in Table II appended.

On the basis of these results it would appear that the viscosities recommended by Philadelphia for dispersion in "DULUX"[2] also are about optimum for dispersing into "Duco" vehicles. A viscosity of 1200 centipoises appears to be near the maximum for good agitating action. Viscosities in the range from 50 to 1200 centipoises give comparable gloss of the finished product.

The type of solvent in the mill base appears to influence the color developed on grinding. Data on the effect of solvent are not complete or conclusive, however.

Generally speaking, satisfactory gloss, hiding and tinting strength could be obtained by "47 Process" dispersion of Milori Blue pigments. The best color produced was more desirable than that obtained by ball mill grinding. It would be satisfactory for use in certain lacquer formulations but was not within Parlin factory color limits. Parlin Laboratory shaders were not able to match 202-51568 Nightshade Blue, a metallic blue, using "47 Process" material because the color was somewhat light and milky as compared to pulp process dispersions.

The best results obtained were produced by grinding the pigment in castor oil, dibutyl phthalate, and hydrocarbon at viscosities from 100 to 1200 centipoises. The grinding time is fairly critical and the optimum cycle will vary with the equipment used. In laboratory batch processes, grinds of slightly less than 1 hour appear to give best results. Minor improvements in color were obtained by using 60-80 mesh sand and by using silicone oil as a wetting agent.

C. <u>Evaluation of 60-80 Mesh Sand</u>

Experience in the Philadelphia Laboratory and throughout the Finishes Division indicates 20-30 mesh Ottawa sand to be the most satisfactory general purpose grinding medium for "47 Process". However, it is a generally accepted theory that for maximum grinding efficiency an optimum ratio exists between the diameter of the grinding media and the diameter of the particles being ground. Therefore, it appears logical that finer sand might be more effective in dispersing pigments of very small particle size.

As in ball mill grinding, it is difficult to formulate a general theory of "47 Process" operation. Four types of grinding action probably take place: impact grinding, rolling or crushing, attrition grinding (whereby pigment particles are rubbed against themselves and against sand particles), and a kneading action or work against pigment agglomerates by the viscous pull of the grinding vehicle. The fineness of sand affects the latter action only insofar as it affects the viscosity of the grind.

As in ball mill grinding the grinding surface is inversely proportional to the cube of the diameter of the grinding media. Grinding by attrition would be expected to increase as the diameter of the grinding media decreases. Also within certain limits, the amount of rolling or crushing would become greater as the diameter of the grinding particle decreases. The amount of grinding by impact would be expected to decrease as the grinding media becomes smaller as will be discussed under Section D of the report.

Fahrenwald and Lee in their studies (4) found that a 17° angle of nip gave optimum grinding efficiency in a ball mill. The angle of nip can be calculated from the formula $\cos N/2 = (r + a) \div (r + b)$ where "N" is angle of nip, "r" represents the radius of grinding particles, "a" equals 1/2 the space between grinding media, (assumed to be zero) and "b" is the radius of the particle being ground. By this formula the grinding media should be approximately 3.5 microns in diameter to give maximum efficiency in grinding of agglomerates .1 micron in diameter, and approximately 1/6 inch for grinding 325 mesh pigments.

J. M. Dallavalle in his book on Micromeritics (5) states that for maximum grinding efficiency, the following condition should exist: particle diameter/ball diameter $^2 = 6 \times 10^{-6}$ where diameters are expressed in microns. According to this formula, 125 mesh particles or about 130 microns would give best results for grinding agglomerates near .1 micron in diameter, and about 1/4 inch particles would be preferred for grinding 325 mesh material.

Both the above formulas are based on ball mill grinding. The first formula is based on work with comparatively large particles. The latter formula is based on studies of small particles and is undoubtedly more applicable to the grinding of Milori Blue and Maroon pigments. Both formulas show that particles for dispersing pigments such as Milori Blue, which has a unit particle diameter of approximately .03 microns, should be appreciably smaller than particles used for grinding talc and other coarse pigments which may be as much as 40 microns in diameter. It can be readily understood, however, that the use of very fine grinding media creates an objectionable filtration problem.

It is possible that a mill base containing pigment particles of wide range in diameters may be ground more efficiently by (1) grinding in stages using coarse sand in the first stage and fine sand in the last stage or (2) by using sand of a wide range in diameters as a grind media. Both methods have proved effective in ball mill grinding; however, very little work in connection with this study was directed along these lines.

In order to evaluate the merits of the finer sand, W-582 Milori Blue and W-896 Maroon Toner were dispersed using 60-80 mesh sand as a grinding media. Sand which will go through an 80 mesh sieve was not used as it is very difficult to filter from the mill base and cannot be obtained in uniform spherical shape. Make-ups of the 60-80 mesh sand dispersions were compared with corresponding make-ups from dispersions made using 20-30 mesh sand.

253-033M Maroon made from a 1 hour 60-80 mesh sand grind gave a Gardner gloss reading of 93 as compared to a reading of 68 obtained from a corresponding make-up of a 20-30 mesh dispersion. A 1 hour 20-30 mesh sand dispersion of Milori Blue in a thin mill base (38 centipoises) composed of pigment, dibutyl phthalate, castor oil, and solvent gave a Gardner gloss reading of 76 while a corresponding 60-80 mesh sand dispersion gave a Gardner reading of 85. Subsequent 1 hour grinds of a mill base composed of castor oil, dibutyl phthalate, and petroleum naphtha (viscosity of 325 centipoises) gave a gloss of 85 for the finer sand and a gloss of 80 for the 20-30 mesh sand. Of interest is the fact that the observed gloss differential of the above dispersions is more pronounced than is the 60° angle gloss differences obtained by the Gardner Glossmeter. Also slight improvements in color were noted although even the finer sand did not give the desired color characteristics.

There is some indication that less abrasion of the container results from the use of the fine sand. This might be expected as the force of liquid friction opposing the force of the moving sand particles becomes more effective as the size of the sand decreases.(6) Therefore the finer sand particles have considerably less momentum by the time they reach the sides of the container.

The 60-80 mesh sand appears to "body" the mill base to a greater extent that does the coarser sand. Thus, either a higher ratio of mill base to sand, or a lower viscosity mill base is required to give an optimum grinding consistency.

Generally speaking, the use of 60-80 mesh sand increases filtration problems. However, on the basis of laboratory tests, faster grinding rates and slightly improved color can be obtained by using the finer sand for dispersing Milori Blue and Maroon pigments.

D. <u>Dispersion of Highly Pigmented Products</u>

PX undercoats and other highly pigmented products are usually dispersed in the ball mill. The "47 Process" should be convenient for use in the manufacture of big movers of this type as a unit could be used continuously on one product for a considerable period of time. Significant savings in labor costs could thus be effected.

Certain PX undercoats which can be ground at low viscosities apparently can be manufactured satisfactorily by the the "47 Process". Included in this category are flexible surfacers, sealers, and undercoat primers. 233-1567 Flexible PX Surfacer, 233-1538 PX Primer, and 233-9609 Dark Oxide Sealer have been satisfactorily dispersed on a laboratory scale by sand grinding. In all 233 line products the grind portion plus the take out portion appears to give a practical grinding consistency. The largest percentage of ball mill capacity is utilized in the manufacture of primer surfacers. Attempts to manufacture these products by "47 Process" have been discouraging. Satisfactory fineness, as judged from smoothness of surface and lacquer hold out tests could be obtained, but all sand ground primer surfacers which gave satisfactory hold out of topcoats presented poor dry-sanding characteristics.

All or part of the following factors may contribute to the poor sanding qualities of sand ground primer surfacers: (1) some of the sand may be dispersed into the primer surfacer, (2) some of the pigment may be retained in the sand, or (3) the pigment particles may be so ground during the "47 Process" operation that a sprayed film forms a closely packed, hard sanding surface.

As mentioned in the discussion of Teflon grinding discs (Section H of this report), a clear dispersed by "47 Process" definitely shows some contamination from sand which is of such small particle size that it cannot be removed by felt and nainsook filtration. In the case of primer surfacers, very hard pigments are being ground. There is a possibility that these pigments would increase the wear of the sand particles so that contamination by sand might be appreciable. In this connection it was also noticed that sanding qualities diminished as the grinding cycle increased. However, ash and total solids determinations on sand ground 233-1912 Light Gray Primer Surfacer and 233-1913 Dark Gray PX Primer Surfacer showed that solids increased on grinding but that the ratio of pigment to total solids did not change appreciably during the grinding procedure. These results would suggest that any contamination due to dispersed sand was offset by retention of other pigment in the grinding media.

Initially it was believed that the poor sanding qualities of batch "47 Process" dispersions might be explained on the basis that some of the sanding aid or pigment is retained in the sand. It was hoped that satisfactory sanding would result from the use of a continuous process grind. Actually, continuous process batches showed an improved over batch dispersions but they were not comparable to corresponding ball mill ground products.

The primer surfacer ingredients and thinner T-3614 were ground at spraying viscosity. The resulting material gave better sanding than did heavier mill base dispersions but was still inferior to the factory standards in this respect.

Slight changes in effective pigment to binder ratio of primer surfacer formulations could have an appreciable effect on sanding. Changes in grinding procedure probably would effect the average particle size and thereby the oil absorption characteristics of the pigment particles. It has been shown that variations in the grinding procedure can have a pronounced effect on the overall performance of a primer surfacer [7].

As mentioned in Section C of this report many types of grinding probably take place in "47 Process" dispersions. It is believed that here as in ball mill grinding, under optimum grinding conditions the large pigment particles are for the most part broken up by impact force. Some of the pigment particles present in primer surfacer formulations are approximately 40 microns in diameter and probably can be broken up best by this method.

The force of centrifugal settling [8] can be expressed by the formula:

$$F = \pi/6g \left[d_p^3 (C_p - C_l)\, r\, w^2 \right]$$

where F = force in grams, g = acceleration of gravity, d_p = diameter of particle, C_p and C_l = specific gravity of the particle and of the liquid respectively, r = the radius in centimeters through which the particle is traveling, and w = the angular velocity of the particle in radians per second.

The above formula is admittedly not directly applicable to the "47 Process"; however, it does show the magnitude of the factors which influence the impact force developed by sand particles traveling through a liquid. As in centrifugal settling, the impact force increases as the speed and diameter of the grinding particles are increased and as the difference in densities of the grinding media and the vehicle becomes larger. The impact force is decreased by the viscous resistance of the medium to the motion of particles hurling through it as expressed by Stoke's law. Therefore, a grinding media of a high specific gravity and a low viscosity mill base would be expected to promote a faster rate of impact grinding.

The composition of primer surfacers is such that mill bases of viscosities lower than 20 cup material cannot be easily obtained without altering the ultimate product composition. Consequently, mill bases cannot be formulated which will give a desirable viscosity for sand grinding. It is observed that very little turbulence and rolling is obtained. The attrition of sand and pigment particles which results from sand grinding of a viscous mill base might be expected to give preferential grinding of pigments and extenders which have low shear resistance, the same particles which promote easy sanding. A primer-surfacer film containing a mixture of pigments would then present tops of the larger pigment particles which might be expected to be of the harder sanding variety.

Some encouraging results were obtained by using grinding media of a higher specific gravity than sand. Steel shot 1/16" in diameter were used to replace sand in the "47 Process" grinding equipment. Primer surfacers ground with shot gave better dry sanding characteristics than did sand ground material. Also satisfactory fineness as measured by smoothness and lacquer hold out could be obtained in less time by this method. Laboratory batches of 233-1912 Light Gray Primer Surfacer which appeared to be satisfactory in all respects were produced by grinding with 1/16" diameter steel shot.

E. Manufacture of Single Pigment Enamels Currently Dispersed in the Ball Mill

Ball mill grinding is utilized at Parlin chiefly for the grinding and dispersion of pigments which are coarse and hard. Included in this category are iron oxide pigments, extender pigments, bone blacks, Turkey Burnt Umber, etc. Generally speaking, laboratory work indicates these pigments can be satisfactorily dispersed by the "47 Process". A brief investigation of "47 Process" grinding of these pigments indicated that the ball mill formula including the add to pull portion generally forms the most favorable grinding vehicle. On this basis MB-253-C895 Red Iron Oxide, MB-253-0373M Victoria Maroon, MB-253-059 Monastral Blue, MB-3475 Burnt Umber and MB-0284 Bone Black were dispersed by the "47 Process". Each was ground 1 hour at 2350 r.p.m. using approximately a 50/50 volume ratio of sand to mill base. When made into 253-line tinting enamels each of these dispersions produced gloss and color comparable to their corresponding standards.

Exposure tests of "47 Process" 253-020 Bone Black and

253-040 Burnt Umber were made in Florida and in Delaware. Results from 12 months exposure tests indicate that the sand ground enamels show slightly less chalking and slightly better polish back characteristics than do corresponding enamels dispersed in the ball mill.

The Parlin factory has installed a 23 gallon semi-works "47 Process" unit. The Process and Methods Group has attempted to disperse W-284 Bone Black in this equipment using laboratory developed formulas. Satisfactory gloss and color have been obtained but some objectionable grit formation was encountered. Further work is underway to overcome this difficulty by varying the mixing procedure used in incorporating the mill base into finished 253-020 Black.

It is expected that other single pigment enamels currently dispersed in the ball mill can also be adopted to factory-scale "47 Process" production. The subject process may find use as a quick method of producing certain small production items at hand mix. However, in this connection, cleaning the sand between batches presents a serious problem. It may prove to be more practical to replace the sand than to clean it between colors. If so, the sand would add considerably to the ultimate cost of the product.

F. <u>Application of "47 Process" Technique to Pigmented EPON Finishes</u>

In accordance with a request from Dr. A. C. Buck of the Parlin Laboratory, the "47 Process" was evaluated as a method of dispersing pigment into EPON resin finishes.

600 gms. of J-200-X-10129 White was premixed and ground with 900 gms. of 20-30 mesh sand. An agitator speed of 2350 r.p.m. was used and samples were taken at intervals. Fair dispersion was obtained after an 8 minute grind. A 1 hour grind gave visual gloss comparable to a 5 day pebble mill grind.

Dispersion in EPON resin solution generated a considerable amount of heat and temperatures as high as 75°C. were obtained. Both the white and the blue formulations were of satisfactory viscosity for "47 Process" dispersion. Further study of sand grinding technique as applied to EPON resin solutions is being conducted by the plant Process and Methods Group and by the Adhesives Research Section of the Parlin Laboratory.

G. <u>Dispersion of Santocel "C", G-222</u>

Santocel "C", a silica aerogel, is used quite extensively in both "Duco" and "DULUX" type furniture finishes as a flatting agent. Considerable difficulty has been encountered in producing a dispersion of G-222 which would give suitable clarity and flatting efficiency. Currently, however, a satisfactory product is made by mill grinding with glass beads.

"47 Process" dispersions of this flatting agent which were made in a quart can using stainless steel agitators resulted in material of very poor clarity. It was evident that fragments from the agitator and from the mixing vessel were contaminating the product. Accordingly, subsequent experiments were made using glass agitating discs in a glass container. Samples were taken at intervals and submitted to the Furniture Laboratory for evaluation.

The "Duco" flatting mill base samples were compared with a 7.5 hour glass bead grind by adding 20 parts of each to 80 parts of 14000 Clear, 28% solids type F lacquer. Results are given below:

Grind	Grit after Filtration	Sheen	Clarity
Glass Bead 7.5 hrs.	None	67	OK
"47 Process" - 2 min.	None	69	Equal to std.
"47 Process" - 10 min.	None	72	Better than std.
"47 Process" - 30 min.	Def.	77	" " "
"47 Process" - 60 min.	Def.	78	" " "

Travelometer tests indicated the settling of each of the "47 Process" samples to be comparable to the glass bead ground sample.

It can be seen from the above results that "47 Process" grinding gives a faster grinding rate than does glass bead grinding. Clarity improves with longer grinding but flatting efficiency decreases. Later tests indicate that an 8 minute sand grind appears to be comparable in all respects to a 7.5 hour glass bead grind.

The "DULUX" flatting mill base grinds also showed that higher clarity but poorer flatting efficiency results from extended grinding. Here a 10 minute grind period appears to be comparable in all respects to a 6 hour glass bead grind.

Santocel "C" dispersion by the "47 Process" shows considerable promise; however, special equipment appears to be necessary to the production of Santocel mill bases of suitable clarity by this method.

H. Evaluation of Teflon Grinding Discs

At the suggestion of Mr. J. W. Clough, grinding discs were made of Teflon. "47 Process" runs were made of a clear to compare the performance of Teflon grinding discs with stainless steel grinding discs. The clear used was composed of blown castor oil, dibutyl phthalate, and hydrocarbon. 1-1/2 hour grinds were made using a 50/50 volume ratio of sand to mill base. One grind was made using stainless steel agitator discs. The other grind used Teflon agitator discs. An aigtator speed of 2350 r.p.m. was used in both cases. The products from both grinds showed contamination as shown in Table III of the Appendix.

It is significant that sand accounted for approximately 78% of the contamination of the Teflon disc ground material and only 44.6% of the contamination of the metal disc ground material.

This would indicate that Teflon is considerably more resistant to abrasion than is the stainless steel used in the metal discs. The fact that metal discs give .50% contamination by sand as against .25% obtained by the Teflon discs indicates that more grinding is done by the metal discs. The gloss and color characteristics of Milori Blue dispersions made by the two types of discs also indicates this to be true.

It is believed that the amount of agitation could be increased by corrugating or chanelling the Teflon discs. On the basis of work to date, the use of Teflon grinding discs appears to merit further study.

CONFIDENTIAL INFORMATION

This subject is generally considered confidential and should receive restricted distribution.

The "47 Process" has been disclosed to foreign affiliates.

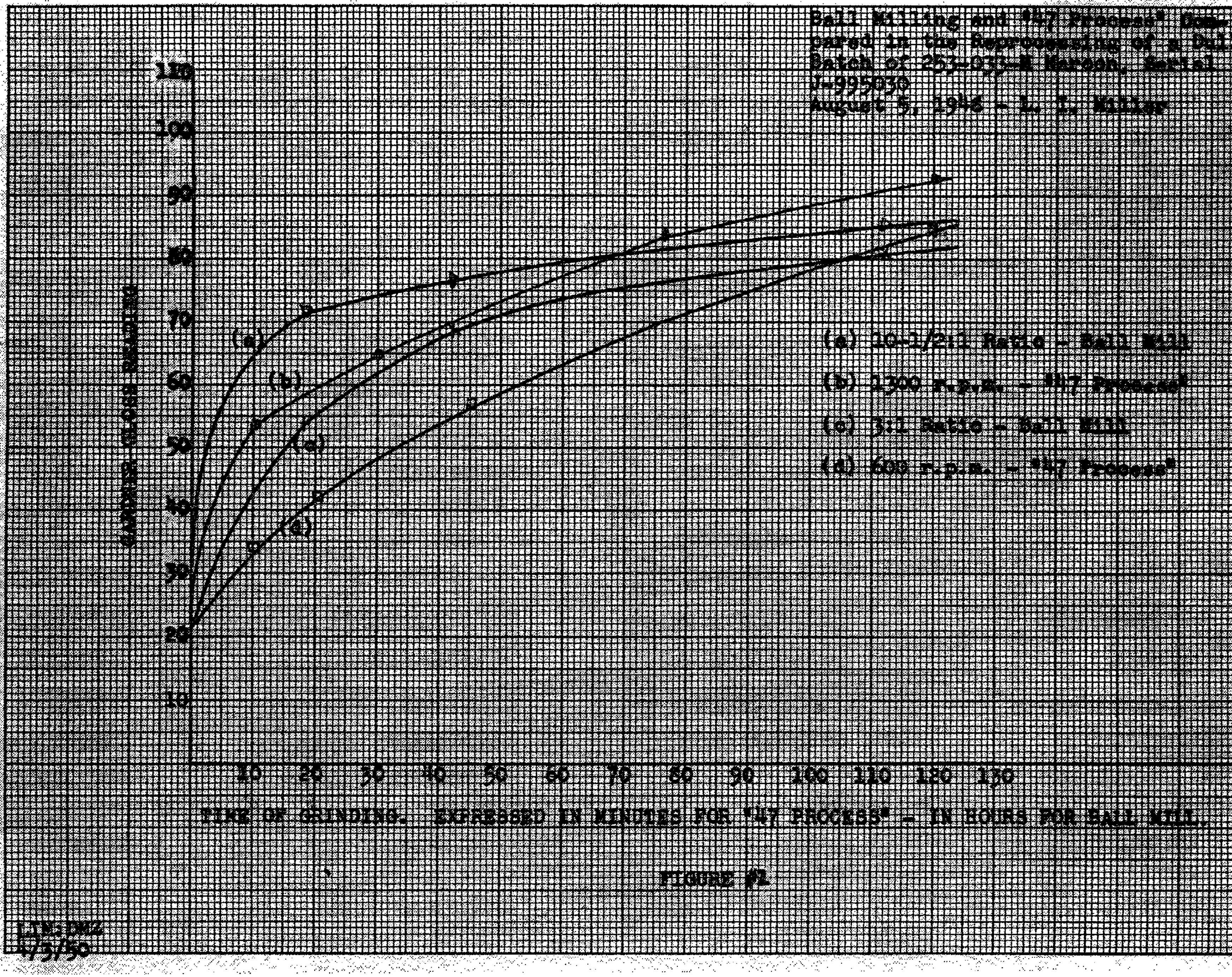
Ball Milling and "47 Process" Compared in the Reprocessing of a Ball Batch of 253-037-B Maroon, Serial J-995030
August 5, 1946 - L. L. Miller
GARDNER GLOSS READING
110
100
90
80
70
60
50
40
30
20
10
10 20 30 40 50 60 70 80 90 100 110 120 130
TIME OF GRINDING. EXPRESSED IN MINUTES FOR "47 PROCESS" - IN HOURS FOR BALL MILL.
(a) 10-1/2:1 Ratio - Ball Mill
(b) 1300 r.p.m. - "47 Process"
(c) 3:1 Ratio - Ball Mill
(d) 600 r.p.m. - "47 Process"
LTM:DMZ
4/3/50

FIGURE #2

TABLE I

VEHICLES USED FOR GRINDING MILORI BLUE PIGMENT

Notebook Number	Pigment Lot	Dispersion Process	Parts Nitro-cellulose	Parts Alkyd	Parts Dibutyl Phthalate	Parts Castor Oil	Parts Solvent	Parts Pigment	* Red to Blue	** Dark to Light	Gardner 60° Gloss	Maximum Particle Diameter in Microns	Average Particle Diameter in Microns	Remarks
2299-78	39838	"47 Process"	-	6.4	-	2.9	23.9	7.3	6	4	80	-	-	
2299-79	"	"47 Process"	-	-	-	2.9	21.2	7.3	2	3	76	-	-	
2299-80	"	"47 Process"	-	-	5.2	-	21.8	7.3	2	2	70	-	-	
2299-81	"	"47 Process"	-	-	5.2	2.9	17.1	7.3	3	1	81	-	-	
2299-82	"	"47 Process"	5.4	-	-	2.9	33.8	7.3	5	2	76	-	-	
2299-84	"	"47 Process	16.0	6.4	-	2.9	64.9	7.3	5	1	74	1.00	.14	
2299-85	51733	Mod. Pulp	14.7	-	4.1	2.4	-	7.3	3	1	76	0.6	.09	Best color.
2299-86	"	"47 Process"	5.4	6.4	-	2.9	39.5	7.3	2	2	72			
2299-88	"	"47 Process"	3.6	6.4	-	-	35.0	7.3	1	4	69			
2299-89	"	"47 Process"	-	6.4	-	-	26.6	7.3	4	4	71			
2299-90	"	"47 Process"	-	-	-	2.9	21.2	7.3	3	2	80	.75	.11	Silicone added.
2299-91	"	"47 Process"	16.0	6.4	5.2	2.9	64.9	7.3	6	4	76			
2299-92	"	"47 Process"	-	-	5.2	2.9	17.1	7.3	4	2	70			
2299-93	"	"47 Process"	-	6.4	-	-	26.6	7.3	4	4	76			
2299-94	"	"47 Process"	5.4	-	5.2	2.9	34.1	7.3	4	4	70			
2299-95	"	"47 Process	-	-	5.2	2.9	17.1	7.3	2	1	80	.90	.14	
2299-97	"	"47 Process"	16.0	6.4	-	2.9	64.9	7.3	5	1	74			
2299-99	"	Ball Mill	-	3.7	-	2.9	12.4	7.3	2	2	74	1.20	.18	

*1 is reddest in masstone.

**1 is darkest in masstone.

Note: 2299-85 is only formulation entirely satisfactory in color. Colors become less desirable as they become lighter than 1 and as they vary from 3 in red to blue scale.

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TABLE II

EFFECT OF VISCOSITY ON THE GRINDING OF MILORI BLUE

Formula	Parts H-111	Parts H-120	Parts Pigment	Parts Alcohol	Parts Ketone	Parts Hydro-carbon	Centipoises Viscosity	60° Gloss	Color
205-A	5.2	2.9	7.3	3.7	7.1	6.3	19	76	Reddest of group
207-A	5.2	2.9	7.3	3.7	7.1	4.4	40	76	Red and light
207-D	5.2	2.9	7.3	3.7	4.0	4.4	60	80	Red, but bluer and lighter
207-E	5.2	2.9	7.3	3.7	-	4.4	52	82	Lightest and bluest
207-F	5.2	2.9	7.3	-	-	4.4	105	78	Fair color. Light and blue.
207-G	5.2	2.9	7.3	-	-	3.2	325	80	Best color but a little light.
207-H	5.2	2.9	7.3	-	-	2.0	1200	80	Light and red.

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TABLE III

CONTAMINATION OBTAINED IN SAND GRINDING

	Metal Disc		Teflon Disc	
	Felt & Nainsook Filtration	Cheesecloth Filtration	Felt & Nainsook Filtration	Cheesecloth Filtration
% Ash	1.07	1.13	.239	.26
Siliceous Matter				
% of Sample	.44	.50	.236	.25
% of Ash	41.6	44.6	98.9	96.2
Matter Removed by Gooch Filtration				
Dried @ 105°C.			.27	.32
Ignited @ 700°C.			.24	.28
% Teflon			.03	.04

Note: The above figures are based on 1-1/2 hour grinds of a clear consisting of dibutyl phthalate, castor oil, and hydrocarbon. (325 centipoises)

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