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Serial Number 18802

Tent. S.W. Process, S.N. 18802-A
Prel. Cost Estimates, " "

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E. I. DuPont de Nemours & Company

Jackson Laboratory

March 27, 1945

The Removal of Grit from Wet "Monastral" Products

Progress Report

N. M. Bigelow

O. Stallmann

Division Head

The Removal of Grit from Wet "Monastral" Products

N. M. Bigelow

JLR-59-1, No. 54 Serial Number 18802

Objects of the Investigation:

Short Range: to reduce the grit content of "Monastral" products (in paste and powder form) to passable limits (0.1%).

Ultimate: to produce "Monastral" products which are completely free of grit.

Period Covered by the Report:

November 8, 1943 to March 1, 1945.

Historical Background:

The uses to which the various "Monastral" products are put make it highly essential that they be as free from grit as possible. The reasons for this are obvious, and need not be discussed here. At present, all of the phthalocyanine pigments on the market contain some grit, regardless of their type or source. DuPont's "Monastral" pigments are neither strikingly superior or inferior to competing types in this respect. It is certain that the trade requirements as to the grit content of phthalocyanine pigments will become increasingly stringent in the future; and it is also certain that in a competitive field, the fact that one brand of pigment contains relatively less grit than a competing brand may be a critical factor in sales acceptance.

When the program described in this report was initiated the specifications for various "Monastral" pigments called for a permissible maximum of 0.25% to 0.50% of grit retainable on a 200-mesh screen, on a dry colors basis. Considerable difficulty was being encountered in meeting this standard. Furthermore, it was realized that ultimately this standard would have to be raised. The objects of the investigation were framed with both of these factors in mind.

Conclusions:

The combined efforts made in laboratory and in the producing areas, and the installation of a settling box of improved design,

have resulted in a reduction of the grit content of the most important "Monastral" products to 0.15% of grit or less. This represents the best attainments in equipment or by methods now known to us.

No method of producing completely grit-free "Monastral" products has been found as yet.

Summary:

The settling box, the vibrating screen and the Dorr Hydro-separator have been investigated as means of removing grit from moist "Monastral" products. The settling box was developed to a fairly satisfactory performance, and is now being used in removing grit from CPC Paste Crudes HT, LT, D and SG. This piece of equipment will reduce the grit content of acid pasted types to about 0.10-0.15%, and of salt-milled types to 0.3%. These figures appear to represent the practical limits of this equipment. The vibrating screen removes grit from undispersed pigment suspensions fairly well, and is being used in the Ponsol Area to reduce the grit content of Polychloro CPC Paste Crude D. The Dorr Hydro-separator is not applicable to our problem.

Patent Situation:

Nothing of a patentable nature is disclosed in this report.

Plans for Future Work:

The Bird Solid Bowl Centrifuge is being studied as a means of removing grit from moist "Monastral" products.

Experimental:

The experimental portion of this report is arranged as follows:

Part One - The Nature of the Grit in "Monastral" Products
Part Two - Grit Removal from Wet "Monastral" Products

I. The Settling Box
II. The Vibrating Screen
III. The Dorr Hydro-separator.

Part One

The Nature of the Grit in "Monastral" Products

Theoretically, it should be possible to make and keep "Monastral products free of grit. In actual practice, however, the introduction of grit into the products is inevitable. Phthalic anhydride enters the phthalonitrile converter in gaseous form, free of grit. Even in passage through the catalyst, however, a trace of abraded catalyst is added to the phthalonitrile. The drying, premixing and baking operations all introduce grit to the crude product. In acid pasting, the sulfuric acid solution is screened before drowning; this removes the coarse grit only. From the drowning step on, all of the iron rust, sand and tramp impurities which are introduced accidentally in the finishing steps, remain in the product until it arrives at the last finishing step. If press cakes of semi-finished products are left in contact with the air, portions will dry to form hard particles which are as troublesome as any other type of grit.

Since the introduction of grit into "Monastral" pigments is inevitable, a positive means of removing it must be included in the finishing process. When the product is finished in the form of a dispersed paste, its grit content is removed fairly easily by passing the paste through bolting cloth on a vibrating screen immediately before the final packaging. The removal of grit from undispersed "Monastral" products, such as press cakes or undispersed pastes, is much more difficult. The press cakes are too thick to screen. The use of dispersing agents here is not permissible, because the dispersed product would no longer be suitable for use in flushing pastes or for drying to powder types.

Since grit may be introduced into "Monastral" pigments in every operation which is performed upon them, it is obvious the grit removal step should be placed as late as possible in the sequence of finishing operations. The last step common to all "Monastral" products is the ammonia slurry. This appears to be the most logical place to remove grit from all "Monastral" products. Additionally, dispersed "Monastral" pastes can be screened immediately before packaging (which is done at the present time), and the grit content of "Monastral" powders can be controlled by a suitable combination of fine grinding and size classification.

It was decided, therefore, to attack the problem of grit removal at the ammonia slurry stage for all products, and at the final grinding step in the case of powders.

At the time when this investigation was undertaken, the grit content of semi-finished "Monastral" products, defined as material retained on a 200-mesh screen by wet screening methods, and referred to a 100% color basis, was in the order of 0.3% to 0.6%. The grit content of Polychloro CPC Paste Crude Acid D, the worst offender at the time, occasionally exceeded 1%. The composition of the grit was as follows:

1. Sand and soft coal fly ash. The sand came both from dissolved mortar in tile-lined equipment and atmospheric contamination; the ash from the latter source.

2. Iron rust, mainly from iron equipment.

3. Dry pigment, formed by allowing press cakes to stand around in the air, or by the re-use of improperly cleaned filter presses. This material is properly classified as grit, since it can scar an engraved copper printing roll almost as badly as silica or iron.

4. Tramp materials, such as window glass, tile, nuts, bolts, etc. Such contamination was avoidable and occurred only occasionally.

The grit came in a complete range of particle sizes, as is shown by the following examinations of representative samples of grit. The first sample is grit separated from CPC Paste Crude HT by passage of the slurry through the settling box. (See JLNB 4259, p.42). To obtain this sample, the scrapings from an empty settling box were mixed on a Hamilton-Beach mixer with an excess of Compound 8 and then washed on a 325-mesh screen, to remove all of the dispersible pigment. The residue on the screen was then classified through graduated screens and dried.

Table I

Composition of Representative Grit from CPC Paste Crude HT

	Size on 60 mesh	Distribution - Screen Mesh Sizes Through 60 on 100	Through 100 on 200	Through 200 on 325
Percent of Total Grit	60.6	16.2	14.4	8.8
Percent Silica and Ash	25.2	7.46	16.7	2.6
Percent Ferric Oxides	61.0	41.1	40.7	28.1
Percent Organic (by Difference) Including Dried Pigment	13.8	51.4	42.6	69.3

The composition of another typical sample of grit, removed from an undispersed slurry of Polychloro CPC Paste Crude D by passage through 300-mesh bolting cloth, is tabulated below. In this case the isolated grit was slurried with an excess of Compound #8 and rinsed thoroughly on a 325-mesh screen to remove adhering wet pigment.

Table II

Composition of Representative Grit from Polychloro CPC Paste Crude D

	Size on 60 mesh	Distribution - Screen Mesh Size Through 60 on 100	Through 100 on 200	Through 200 on 325
Percent of Total Grit	40.6	38.0	9.3	2.0
Percent Silica and Ash	46.2	57.9	59.8	53.1
Percent Ferric Oxide	36.7	32.1	31.0	32.9
Percent Organic (by Difference)including Dried Pigment	7.1	10.0	9.2	14.0

It will be noted that the greatest part of the grit does not pass a 200 mesh sieve, and that most of the grit actually is coarser than 60 mesh. This is the dangerous grit, and, fortunately, the easiest to remove. Our present "Monastral" standards contain enough grit between 100-325 mesh particle size that the deliberate addition of this grit to "Monastral" Fast Blue B Powder Std. in amounts up to 10% does not alter materially its wedge-grit rating in printing ink. The inks so prepared "showed slightly inferior wedge grit results. However, for practical purposes the differences appear to be negligable" (See JLNB 4259, p.43, and Technical Laboratory Memo 2018, 11-19-43). It appears, therefore, that a pigment free of all grit retainable on a 200 mesh screen will be satisfactory for most present-day applications. However, new uses for "Monastral" pigments are being developed in which higher standard will be required. The pigmenting of plastics which are subsequently extruded into filaments is a case in point. Therefore, while the immediate grit problem is satisfied by the removal of grit retainable on a 200 mesh screen, solution of the ultimate problem will require the removal of all grit retainable on a 325 mesh screen.

Grit Determination Methods

The Technical Laboratory tests for grit by suspending a sample of paste or powder equivalent to 1/4 lb. of 100% pigment in a liquid medium (water for aqueous pastes; alcohol-water or pure alcohol for powders) and washing the slurry through a standard 200-mesh screen with a large excess of water. Undispersed lumps are crushed with the fingertips and then washed through the screen. The screen and its content of grit are then dried for a half-hour at 110°; the grit is then transferred to a sheet of paper and weighed. Grit is reported as milligrams per quarter pound of 100% pigment.

Jackson Laboratory felt that this test relied on too small a sample, and depended too much on the operator's thoroughness. Additionally, there was no provision for recovering the pigment subjected to the test. Accordingly, two tests were developed in Jackson Laboratory.

First Jackson Laboratory Grit Determination Method

A sample of press cake equivalent to 150 g. of 100% pigment is dispersed with an excess of Compound #8 in a Hamilton-Beach mixer ("soda mixer") in two or more installments. The dispersed

pastes are poured through a standard 200-mesh screen and rinsed through with an excess of water. The screen and its contents are dried in an oven at 110°; then the grit is transferred to a sheet of paper and weighed. Grit is reported as the percent of total solids, 100% basis.

Second Jackson Laboratory Grit Determination Method

A sample of press cake equivalent to 500 g. of 100% pigment is slurried with enough water to give a 5% suspension. This suspension is filtered through a specially-prepared screen made by cutting the bottom off a Hamilton-Beach mixer cup and soldering a disc of 200-mesh screen in its place. The 200-mesh screen is protected by a removable disc of coarse screen which lies on top of it. The mixing cup is mounted on a Hamilton-Beach mixer and high-speed agitation is employed during the screening; the agitator is placed close to the coarse screen but not in contact with it. The slurry is added to the cup through a large glass funnel. Another glass funnel is placed below the mixer cup and the screened slurry is run through a rubber hose into a series of buckets, for return to subsequent slurry operations. When the mixture has been screened, the slurry buckets, funnel and mixer cup are rinsed with water until the effluent from the slurry cup is colorless. Cup and contents are then dried at 110°. The dried grit is transferred to paper, weighed and reported as percent on a 100% pigment basis.

If the material to be tested for grit is already in slurry form, a suitably-sized weighed sample is screened as described above. The effluent is filtered on a large (13 in.) Neutsch. The filter cake is weighed and solids are determined; grit content is calculated on the bases of these results. The portion of the filter cake left after the determination of solids can be returned to later operations.

It must be admitted that the Technical Laboratory and Jackson Laboratory methods do not give closely comparable results. Probably neither method is very accurate, and will probably require more study in the future. Jackson Laboratory feels that its methods are more reliable, since they use a larger sample, and also reduce to a more constant degree the physical work done in forcing the pigment through the screen.

Part Two

Grit Removal from Wet "Monastral" Products

I. The Settling Box

The settling box is a long open wooden tub through which the grit-containing slurry flows, in certain cases over and under a series of baffles and in other cases without baffling. The grit settles to the bottom of the box, while the degritted suspension flows over a wier out of the box, to be pumped into a filter press.

The method removes coarse grit, down to 80-mesh material, very effectively. It is not effective in removing grit finer than 80-mesh in particle size; the highly flocculated pigment entraps fine grit and prevents its settling. It has the technical disadvantages that large amounts of pigment are held up in the settling box, and that the material which settles out contains much good pigment as well as grit. This material must be accumulated and reworked separately.

The principle of the settling box is old. During DuPont's early development of salt milling an arbitrarily designed settling box was used to remove grit from the acid suspension of CPC Paste Crude Acid SG (formerly CPC SG Wet). It proved capable of removing grit quite effectively, and has since been used for degritting CPC Paste Crude HT suspension, following the ammonia slurry.

It was felt that a settling box, designed by a competent hydraulics engineer on the basis of modern physical principles, might be a more effective means of degritting slurries of phthalocyanine pigments. Accordingly, Mr. T. W. Stricklin was requested to present this engineering problem to Mr. C. E. Lapple of the Technical Division. At Mr. Lapple's request, Mr. G. Barnhart of the Miscellaneous Dyes Division determined the viscosities of CPC slurries of various concentrations. (See letter from N. M. Bigelow to Mr. Lapple, dated 12-21-43, which is appended to this report for purposes of record). Mr. Berry's calculations indicated that the most suitable settling box would be a rectangular trough 10 feet in length, 3 feet in width and 18 inches in liquid level depth. The slurry was to be introduced through a 3-inch pipe into the bottom of a stilling chamber made by the insertion of a weir 6 inches from the inlet end of the box. The suspension

was to flow through the box without agitation or baffling. A baffle suspended from above dipped into the suspension to a depth of 6 inches, 14 inches from the outlet end of the box. The purpose of this baffle was to hold back foam and floating grit. The suspension flowed under this baffle and then over a 17-1/2 inch wier set into the box 6 inches from the outlet end. The effluent then was to be pumped from the outlet end of the box to a filter press.

The box was designed to handle a 1% pigment suspension prepared by diluting the usual 5% ammonia slurry suspension continuously with cold water just ahead of the centrifugal pump which transferred the slurry into the settling box. The calculated optimum rate was 30 gallons per min., equivalent to 150 lb. of 100% pigment per hour. (See letter from C. E. Lapple to N. M. Bigelow, dated 12-23-43, which is appended to this report for purposes of record.)

A settling box was constructed according to the above specifications and placed in #64 Building of the Semi-Works, adjacent to a 1500 gallon wooden slurry tank and a 30" wooden filter press.

1. Tests with Polychloro CPC

Shortly before the grit removal program was commenced, the grit content of Polychloro CPC Paste Crude D was excessively high, averaging over 0.5% (600 mg.), and occasionally exceeding 1%. This material was chosen as the most profitable subject for study. A Tentative Semi-Works Process for the Manufacture of Polychloro CPC Paste Crude D Special (Low Grit) was written (Serial Number 18802-A, approved 3-17-44), and a number of experimental charges were run. This process combined the standard ammonia slurry of Polychloro CPC Paste Crude Acid D and the passage of the slurry through the settling box. A preliminary series of experimental charges proved that under the best conditions possible (1% slurry, 15 gal. per min. throughput) the settling box could reduce the grit content of Polychloro CPC Paste Crude D to about 60 mg. (0.05%) by the Technical Laboratory test. Under conditions approaching normal operation, the settling box appeared to be capable of lowering the grit content to about 125 mg. (0.1%). The campaign was not well carried out, due to shortages of labor, Jackson Laboratory supervision and the mechanical short comings of the equipment. It was found impossible to discharge a 5% slurry from the tub by gravity; and

even when a pump was installed, great difficulty was found in introducing the slurry to the settling box at an even rate. Due to variations in feed water pressure, the dilution of the 5% slurry with water at an even rate was also impossible without constant attention. Ultimately, it was found necessary to make up the slurry at the concentration at which it was to be put through the settling box. Excluding orienting runs, and false runs, seven runs were made in January, 1944. Under part-time Jackson Laboratory supervision. The data obtained on these runs are tabulated below.

Table III

Grit Removal from Polychloro CPC Paste Crude Acid D
First Campaign

Charge No.	1	2	3	4	5	6	7
Slurry Concentration, Percent	2.83	3.04	0.92	0.95	0.88	1.65	1.50
Throughput, gal. per. min.	13.	7.	13.	17.	8.7	6.8	15.5
Percent Grit in Feed Method #1	1.29	0.72	2.50	1.52	1.62	2.06	1.27
Percent Grit in Effluent, J.L. Method	1.08	0.39	0.54	0.60	1.46	1.13	1.09
Percent Grit in Effluent, T.L. Method	0.17	0.055	0.044	-	0.11	0.13	0.12
Tech. Lab. Grit Rating, Mg. per Quarter Pound	190.	62.	50.	-	125.	152.	140.

This experimental lot was used in the preparation of "Monastral" Fast Green GF Paste; this particular material had to be used because of an acute shortage of Polychloro CPC Paste Crude D. Since the finished paste received the usual screening, no report can be made as to the grit content of the lot as a whole.

Later in the spring the Sales Division placed an order for the production of 2000 lb. of low grit "Monastral" Fast Green G Powder, to be prepared by use of the settling box. Before this material could be produced, the grit content of Polychloro CPC Paste Crude Acid D was reduced sharply. A lead-lined steel drowning tank had been used in the acid pasting of the crude pigment. Seams had developed in the lead lining of this tub, and

the exposed steel was contributing heavily to the grit in the product. Ultimately the tub was replaced by a tile-lined drowning tub, and the grit content of the Polychloro CPC Paste Crude Acid D dropped at once to about 120 mg. (Technical Laboratory method). Thus, the quality of the starting material was already equal in quality to the anticipated product. However, the paste was ammonia slurried and passed through the settling box. As might be expected, there was no appreciable reduction of its grit content. The actual performances on individual runs are tabulated below.

Table IV

Grit Removal From Polychloro CPC Paste Crude Acid D
Second Campaign

Charge	Grit Content*	Charge	Grit Content
10	110	18	268**
11	62	19	100
12	80	20	150
13	70	21	128
14	60	22	130
15	50	23	100
16	110	24	105
17	170**		

*Milligrams per quarter pound, Tech. Lab. method.
**The "hold up" in the settling box was recirculated in these charges, accounting for the abnormally high grit content.

The Polychloro CPC Paste Crude D so prepared was dried and standardized as "Monastral" Fast Green G Powder, Lot 101. Two grit determinations on this lot at the Technical Laboratory indicated total grit contents of 230 and 280 mg., and magnetic grit contents of 60 and 20 mg. Since this lot was no higher in quality than other lots prepared at the same time, it was released to the trade without special treatment, and accepted without comment.

2. Tests with CPC Paste Crude SG

In connection with the study of the Bird Centrifuge which is now in progress, the grit content of CPC Paste Crude Acid SG, which is regularly passed through the new settling box, has been determined. The grit content of three representative charges, made after a regular ammonia slurry, were 0.32%, 0.32% and 0.34%.

This probably represents the optimum performance of the settling box with this material under normal operating conditions.

Tests on the Original Settling Box with CPC Paste Crude HT

The Technical Laboratory and the Semi-Works have tested the performance of the older settling box on CPC Paste Crude HT, by measuring the grit contents of each individual charge and of each lot of "Monastral" Fast Blue BF Press Cake into which they were composited. Preliminary grit figures were predicted for each lot of press cake by calculating weighted average grit figures for every component charge. The results indicate first, that the original settling box may be keeping gross contamination in check, but is not making a particularly satisfactory product; and second, that the grit detection test now used by the Technical Laboratory is not accurate.

In the table below, grit is reported as milligrams per quarter pound, dry basis.

Table V

Grit Content of "Monastral" Fast Blue BF Press Cake After Passage Through the Settling Box

	Grit Rating			Grit Rating	
Charge	Prelim.	Final	Charge	Prelim.	Final
98	324	93	112	305	190
99	300	90	113	475	170
100	280	165	114	470	360
101	254	400	115	407	220
102	158	285	116	120	245
103	280	250	117	-	250
104	423	260	118	-	230
105	196	250	119*		130
106	156	200	120		185
107	134	222	121		72
108	120	150	122		172
109	550	175	123		185
110	283	169	124		307
111	124	155	125		164

*In charges 119 to 125, the solids content of the ammonia slurry was lowered from 5%-6% to 3%, with the object of determining whether this would increase the effectiveness of the settling box. No marked effect was apparent, and the change in process was dropped.

Cost of Grit Removal in the Settling Box

The added cost of removing grit from Polychloro CPC Paste Crude D by means of a settling box in the Semi-Works at a rate of 3,700 lb. per month, 100% basis has been estimated at $10.85/cwt., 100% basis. Details may be found in Jackson Laboratory Cost Estimate, S.N. 18802-A, and in a letter from Dr. O. Stallmann to Dr. H. A. Lubs, dated 3-29-44. It is believed that in suitable equipment this cost could be reduced to about $5.00/cwt. 100%.

II. The Vibrating Screen

For some years the Ponsol Area has passed at least part of its Polychloro CPC Paste Crude D slurry through a 50-mesh vibrating screen prior to filtration. According to their experience this was not a successful operation; the grit either wore holes in the screen or plugged it, causing later additions of slurry to flood over the screen, rather than to filter through it. Moreover, the useful effect of a 50-mesh screen would not be very pronounced, even if its use were not connected with so much difficulty. A 100-mesh vibrating screen would be far more effective in grit removal, and it appeared desirable to determine exactly what sort of pigment could be produced by such a treatment. The Ponsol Area was unwilling to undertake the problem; their main concern was to maintain the production of Green, and they felt that passing the Green slurry through a 100-mesh vibrating screen would slow up production excessively. The Basic Colors Area, however, had suitable equipment, consisting of a mild steel tank, a screen and a filter press, which was not in constant use, and arrangements were made to carry out the tests. (See JLNB 4259, p. 75.) A typical ammonia slurry was prepared (7.4% solids concentration) and passed through the vibrating screen. The screen plugged badly, and after two hours' operation water was added to the slurry, reducing its solids content to 5.2%. This slurry passed through the vibrating screen without difficulty, at the estimated rate of about 150 lb. of 100% pigment per hour. The material was filtered and washed without difficulty.

The material remaining on the screen was accidentally discarded by the operator.

An attempt was made to pass a separate dilute ammonia slurry of green (3.7% solids) through 300-mesh bolting cloth on a vibrating screen. This proved to be impracticably slow, and

the charge was finally passed through the 100-mesh vibrating screen. Again no difficulty was encountered.

Samples of the press cake, dried in the laboratory, contained 20-40 mg. of grit per quarter pound by the Technical Laboratory test. This is an excellent rating. Inspection of the grit recovered from the screen indicated that 0.37 lb. of grit was removed from 100 lb. of 100% pigment. The particle size distribution, expressed as percent of pigment, dry basis, was as follows: over 60-mesh 0.36%; 60-100 mesh, 0.013%. The grit contained 25% Fe_2O_3 and 59% silica, the remainder being dried pigment, wooden splinters, etc.

These two special charges of Polychloro CPC Paste Crude D were dried and standardized as "Monastral" Fast Green G Powder Lot 96. The Technical Laboratory reported (letter from Dr. K. C. Johnson to Mr. C. F. Schaumann, dated 1-11-44) that "the lot, which was dried in the Semi-Works and micropulverized at G&M, shows somewhat higher grit (85 mg.) than the laboratory dried material, but lower than standard and very much lower than recent production."

The Basic Colors Area estimated that the process outlined above would add $12.26/cwt. 100% to the current cost of Polychloro CPC Paste Crude (see letter from Mr. C. J. Darling to Dr. O. Stallmann, dated 3-8-44).

No commercial use has been made of this method for producing low-grit material. The abrupt improvement in the quality of Ponsol Area Paste Crude made a separate de-gritting operation less imperative. In addition, Jackson Laboratory found that the ammonia-ammonium chloride slurry was definitely corrosive to mild steel, which made it unadvisable to use the Basic Colors equipment for this operation.

III. The Dorr Hydroseparator

The Dorr Company has recently developed a machine for the continuous and semi-automatic classification of solids in aqueous suspension which is based on the differential settling rates of its various components through an ascending column of water. Mr. C. E. Berry of the Technical Division arranged a test program at the Dorr Company's laboratories, and a sample of Polychloro CPC Paste Crude D was sent there under the code designation MDD-869. The visit was made on September 21, 1944. A few settling experi-

ments convinced Mr. G. M. Darby of the Dorr Company that Polychloro CPC Paste Crude D was too highly flocculated to be amenable to grit separation in the Dorr Hydroseparator. For details, see the letter from Mr. T. W. Stricklin to Dr. H. A. Lubs, dated 9-27-44.

Submitted for typing - May 2, 1945
Typed - May 7, 1945

AES

APPENDIX

COPY

CC: Dr. I. Gubelmann, Dr. E. C. Humphery — Orchem
Dr. H. A. Lubs, Orchem
Mr. C. F. Schaumann, Mr. T. A. Martone — Dyestuffs
Dr. Wm. Kirk, Mgr., Dye Works
Att: Mr. J. K. Reed, Dr. E. F. Hitch
Mr. G. H. Schuler, Dir., T. L.
Att: Dr. C. K. Black, Dr. K. C. Johnson
Mr. J. M. Tinker, Dir., J. L.
Att: Mr. T. W. Stricklin, Jr., Mr. C. J. Darlington

December 21, 1943

DR. A. P. TANBERG, DIRECTOR
EXPERIMENTAL STATION

ATT: MR. C. E. LAPPLE

GRIT REMOVAL FROM "MONASTRAL" PRODUCTS

As promised in our conference of November 19 at the Experimental Station, and confirming yesterday's telephone conversation, we, herewith, submit the experimental data which you have requested in order to make the necessary calculations for designing an improved settling box. These data were obtained by determining the rate of fall of spherical glass beads through suspensions of CPC Paste Crude D (drip drowned) and CPC Paste Crude HT (drowned at high turbulence). The suspensions were taken from the ammonia slurry tub and are representative of the material that will be passed through the proposed settling box. The determinations were made by Mr. G. Barnhart.

The glass beads which were used had the following dimensions: specific gravity 2.334; diameter 3.50-3.63 mm.; average weight 0.0571 g.

CPC Paste Crude D Slurry; specific gravity at 3.6% solids concentration, 1.028.

CPC Content of slurry, %	Rate of Settling, Feet per Min.
nil (water)	57
2.27	43
2.60	39
2.92	35
3.25	29.5
3.60	14.3

CPC Paste Crude HT Slurry; specific gravity at 6.5% solids concentration, 1.030.

CPC Content of slurry, %	Rate of Settling, Feet per Min.
nil (water)	57
1.0	57
2.0	57
3.0	57
4.0	55
5.0	50
6.0	34
6.5	27

CPC SG Slurry; (suspension formed by extracting a salt-milled mixture of 1.0 part of CPC and 4 parts of sodium chloride with 25 parts of 1% sulfuric acid).

CPC Content of slurry, %	Rate of Settling, Feet per Min.
nil	57
5.0	42

At present we have no data on the ammonia slurry of Polychloro CPC Paste Crude D; but preliminary observations indicate that it will resemble CPC Paste Crude D in its properties.

We trust that the information given above will enable you to carry out the calculations you have offered to make in order to arrive at recommendations as to the designing of an improved settling box. We should appreciate it if you would give this problem your earliest possible consideration.

If for any reason you should be unable to take care of this matter promptly, please notify us, since the removal of grit from current production of "Monastral" colors is an emergency problem of such urgency that we shall have to go ahead with the construction of a settling box not later than a week from now.

JACKSON LABORATORY

N. M. Bigelow

NMB:mnr

COPY

CC: Mr. S. I. Winde, I. E. D., D. W.
Att: Mr. W. O. Jewett
Mr. J. M. Tinker, Jackson Lab.
Att: Mr. T. W. Stricklin, Jr.

December 23, 1943

DR. N. M. BIGELOW (3)
JACKSON LABORATORY
DYE WORKS

GRIT REMOVAL FROM C.P.C. COLORS

Confirming our telephone conversation, attached is a sketch giving the suggested size of a settling box to handle a C.P.C. Paste Crude D slurry at the rate of 150 lb./hr. of dry color.

Based on the settling rate data for 3.6 mm. glass beads in a C.P.C. Paste Crude D slurry at 25°C., we have estimated the apparent viscosity as follows:

Slurry Concentration Weight %	Settling Rate ft./min.	Apparent Viscosity Antipoises
0.00	57	0.9
2.27	43	4.0
2.60	39	5.5
2.92	35	7.8
3.25	29.5	12.9
3.60	14.3	37.4

These values of apparent viscosity should be regarded as giving only the relative order of magnitude and may be in considerable error for the following reasons:

(1) For the relatively large beads, settling velocity is not very sensitive to viscosity, whereas for the fine particles (under 100 mesh), the settling velocity is directly proportional to the fluid viscosity.

(2) If the slurry shows plastic properties, the apparent viscosity will vary depending on the conditions so that it may be impossible to settle very fine particles under the action of gravity depending on the yield stress of the slurry.

(3) The settling tests were conducted at 25°C. At the higher operating temperatures, the viscosity may be much different (either higher or lower) depending on the relative effect of temperature on degree of solids flocculation and fluid viscosity.

Despite their very approximate character, however, these data (when plotted) show that for solids contents of over 1% the slurry shows a very rapid increase in apparent viscosity. It is therefore suggested that the settling chamber be operated with a 1% rather than the usual 2 - 5% slurries by introducing the required amount of water ahead of the centrifugal pump leading from the mixing tank to the settling box. On this basis, the settling box shown on the attached sheet is estimated to remove particles larger than 325 mesh and having a specific gravity greater than 210 when handling 30 gal./min. of a 1% slurry by weight. This estimate assumes the absence of any plastic properties, such as a yield point, in the case of a 1% slurry.

It will be noted that, with the exception of the baffles, the suggested box is not much different than the existing box. We feel that the dilution of the slurry is the critical factor in securing satisfactory settling and the increased liquor volumes to be handled are far more than compensated for by the reduced viscosity resulting from dilution. It is furthermore suggested that the present box be equipped with a water feed at the pump intake to determine the effect on pigment quality when diluting the slurry from the mixing tank to 1% with hot and with cold water.

In preparation for any further required improvements in degritting, it is suggested that the settling tests be conducted with the 90 micron sand which we shipped to you yesterday using water and various concentrations of each slurry. We would suggest that a known wet volume of this sand be fed to the settling tube. After a specific time, the sand that has passed through the bottom trap can be separated from the slurry by washing through a 325 mesh screen and then measuring the sand thus settled out volumetrically. Since the sand is not entirely uniform in size, this will give a means of estimating the mean settling velocity. The settling velocity of the sand is estimated as 1 to 4 ft./min. in water at room temperature.

We trust this meets your immediate needs and will be pleased to offer any further assistance in the solution of this problem.

TECHNICAL DIVISION
/s/ C. E. Lapple