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NEWARK PLANT

PIGMENT COLOR RESEARCH REPORT

Progress Report

Title: Coalesced Phthalocyanine Pigments II
Evaluation of Pigment BX-CR and other Coalesced
Pigments. Texture and Analytical Study.

Period Covered: November 1, 1943 - June 1, 1944.

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I. INTRODUCTION:

This report, continued from KN-43-178, summarizes the work done at Newark on evaluation of coalesced phthalocyanine pigments in the period from November 1, 1943 to June 1, 1944. All evaluations of recent and earlier products of this type carried out in this period are listed in the Tables, Part V, which are continued from those of the previous report.

Violet DX, of interest in prior work as a shading color for phthalocyanine blues, was found to be unstable to baking in alkyd enamels, which defect was considered a serious handicap to its promotion as a paint pigment. The properties of Violet DX (and other DX colors) have been described in a separate report, KN-44-25.

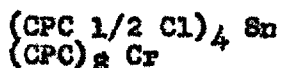
Results from outdoor exposures of subject pigments in durable vehicles will be summarized later after 12 to 18 months exposure on the fences. Details of physical studies of these products will be included in a separate report.

II. PATENT SITUATION

This aspect of the problem has been summarized in the earlier report. Considerable attention has been given to assessing the novelty of the cobaking coalescence process on various substrates at Jackson Laboratory. Extensive analytical and property studies on these pigments have been made at Newark towards the same end.

It has been shown at Jackson Laboratory that Pigment BX-CR can be made by the process of Example 3 in Br. Pat. 453,767 (I.G.). There is no U.S. equivalent of this patent.

In addition to coalesced phthalocyanine pigments two other types of products have been found of interest as non-flocculating phthalocyanines, viz., mixtures of CPC toners with other metallic phthalocyanines toners, and complex "two metal" phthalocyanine toners. Examples of the first type are mechanical mixtures of CPC and iron phthalocyanine, and of CPC and titanium phthalocyanine. The second type of product, previously unknown, has been isolated from various coalesced phthalocyanine pigments at Jackson Laboratory, and can also be prepared in toner form. Two types of "two metal" phthalocyanines are illustrated by the following empirical formulae:



Both of these latter types of products appear novel and patentable, but more data is necessary for patent purposes.

No complete prior art search on the phthalocyanines has been made either at Jackson Laboratory or Newark.

The patent aspects of the above three types of products are discussed in considerable detail in recent letters of A. J. Stratton and the writer to E. R. Allen, and of O. Stallmann to H. A. Lubs.

III. SUMMARY AND CONCLUSIONS:

1. This report continues the study started in KN-43-178, Coalesced Phthalocyanine I. Laboratory work has been concentrated in the past seven months on investigation of Pigment BX-CR and related products: analyses, patent aspects, and texture improvement. Some 26 new products, not listed in the earlier report, have been evaluated.

2. Pigment BX-CR is the best non-flocculating phthalocyanine pigment prepared to date, and appears to be a desirable addition to the "Monastral" line.

3. Crude Pigment BX-CR analyzed, as an average, $\pm 20\%$ monochloro CPC, $\pm 70\%$ TiO_2 , and $\pm 10\%$ water and ammonia extractable materials. Extraction of the pigment did not alter its tinctorial properties noticeably, and the extracted pigment was equal to crude in its flocculation resistance. The extractable portion consisted chiefly of phthalimide and inorganic copper and ammonium salts. Crude Pigment BX-CR was acidic, an aqueous slurry having pH of 4.65; and a neutralization equivalent of 0.1 Eq. $\text{NaOH}/100\text{g}$.

4. Steam micronization of crude Pigment BX-CR has produced acceptable texture. The micronized pigment gave satisfactory enamel fineness by mixing in alkyd vehicle over a 3 roll-paint mill. Other grinding methods, or treating agents tested to date failed to give satisfactory texture improvement. These are results from one set of micronized samples. Further study of micronization is planned with this and other coalesced phthalocyanine pigments.

5. Micronized samples of Pigment BX-CR were equal in tinting strength to equivalent extensions of Monochloro CPC toner with "Ti Pure" R-610, and in some cases appeared stronger than the latter (ink mill evaluations).

6. The non-flocculating characteristics of Pigment BX-CR do not appear to be due to the action of known or probable by-products or unreacted intermediates in the crude pigment. None of these materials had any beneficial effect on the flocculation of CPC toners in lacquers tinted with TiO_2 .

7. Titanium phthalocyanine has been found to be a non-flocculating phthalocyanine toner, which has prevented the flocculation of CPC toner in one test when a mixture of 20%

"TiPC": 80% CPC was extended with TiO_2 in lacquer. Other tests failed to check the first result. This effect of "TiPC" appeared similar to that of iron phthalocyanine discovered several years ago. Other metallic phthalocyanines remain to be investigated for their effect on the flocculation of CPC toners.

8. The non-flocculating properties of Pigment BX-CR cannot be explained by formation of "TiPC" during the bake. The toner pigment isolated from BX-CR by acid pasting flocculated in the usual way and contained only traces of TiO_2 . Titanium phthalocyanine was stable to acid pasting.

9. A fraction of toner phthalocyanine which was non-flocculating in TiO_2 lacquer tint has been extracted with alpha chlor naphthalene from Pigment BX-CR. The nature of this toner has not been determined to date, but it is suspected to be a complex of TiO_2 or Ti and CPC similar to those recently isolated at Jackson Laboratory from coalesced pigments containing chromium and tin, respectively.

10. Co-baked phthalocyanine/ultramarine pigments were non-flocculating when extended with TiO_2 in lacquer. This is the only colored pigment used to date as substrate in the co-baking process which gave this property. Some of the metal free/ultramarine products containing 5% or less of phthalocyanine were interesting tinctorially, and are being evaluated as paint pigments.

11. "Monastral" Fast Green G toner (polychloro CPC), known for some time to be a non-flocculating phthalocyanine, was found to inhibit the flocculation in lacquer of CPC toner (Blue B) in mixtures containing 40% or more by weight of the former. Various non-flocculating toner pigments other than phthalocyanines such as azo red, iron blues, chrome yellows, etc. had no noticeable effect on the flocculation of CPC toners when mixed with the latter in TiO_2 lacquer tints.

12. The easy dispersion of crude coalesced phthalocyanine pigments of all types in linoleum, as discussed in the preceding report, has been confirmed.

13. Green G-CR on blanc fixe (polychlor green), prepared by dry chlorination of the cobaked monochloro CPC/blanc fixe counterpart of Pigment BX-CR, continues to be of interest as a potentially cheap extended polychloro green. The crude product had poor texture, but was noticeably improved in this respect by salt milling. Steam micronization of this pigment has not been tried to date. Interesting, intense greens similar to standard chrome greens in full shade were obtained by tinting the salt milled pigment with greenish yellow, X-493-D.

14. Considerable time has been devoted to the preparation of additional exposure panels of the coalesced pigments in durable vehicles. To date Pigment BX-CR, the blanc fixe counterpart of BX-CR, and Green G-CR on blanc fixe have appeared fully equal in lightfastness, chalk resistance, and durability to dry mix controls of standard phthalocyanine toners and "Ti PURE" R-610. Green G-CR on "Ti PURE" R-LL for R-610 has been found to be inferior to "Monastral" Green G/"Ti PURE" R-610 controls in chalk resistance and color retention.

IV. DISCUSSION OF EXPERIMENTAL RESULTS

Most of the laboratory work in the past six months has been devoted to an intensive study of Pigment BX-CR, its composition and analysis, texture and methods of improving same. The analytical study had as its primary aim the elucidation of the nature and kinds of products in Pigment BX-CR, and related coalesced pigments with the ultimate purpose of obtaining data on these pigments for patent purposes. In this connection, some study has been made of the effect of various phthalocyanine toners, and organic and inorganic pigments on the flocculation of "Monastral" Fast Blue B (BT-172-D) in lacquers tinted with TiO_2 . A number of new coalesced pigments have been examined, the most interesting being those on tin oxides and ultramarine, respectively.

A detailed summary of these evaluations is given in the tables, Section V. The most important results for each of the pigments studied in any detail are discussed briefly below.

A. Pigment BX-CR

Five semi works runs of $\pm 200\%$ each have been made to date at Jackson Laboratory. Of these, lot 3 gave the highest yield of CPC, and was the best of the runs tinctorially and strength-wise. Lot 5 was made with cuprous chloride in place of cupric chloride, and gave the lowest yield of CPC in the series. Tinctorially, lot 5 is greener and duller than lot 1, and is also weaker.

Experimental work in the Newark laboratories has been carried out with lots 1 and 3, only.

1. Analysis and Patent Aspects. Flocculation Studies.

The composition of Pigment BX-CR was of interest for two purposes, viz., (1) determination of the true CPC content of the pigment, and (2), identification of by-products from the reaction and determination of their effects on the flocculation of Blue B toner (BT-172-D) extended with TiO_2 in lacquer.

Four general theories have been postulated to explain the non-flocculating and non-separating characteristics of Pigment BX-CR.

1. Coating of the TiO_2 substrate with the phthalocyanine pigment resulting in an integral tint.
2. Formation of a covalent compound between TiO_2 and CPC. This condition has been termed chemisorption. The resultant compound need not be of the classical type, i.e., formed from definite and multiple proportions of reactants.
3. Inhibition or prevention of flocculation of the CPC in the coalesced pigment with by-products formed in the reaction.
4. Formation of non-flocculating metallic phthalocyanines other than CPC, or metallic complexes of CPC, by reaction of phthalonitrile or phthalocyanine with Ti, TiO_2 , or some other titanium compound formed during reaction.

Physical methods appeared best adapted to a study of the pigment from the viewpoint of theories 1 and 2. Attempts are being made at present to obtain good electron diffraction patterns of Pigment BX-CR in the hopes of obtaining evidence bearing on these postulates. Electron micrographs at 25,000 magnification have been obtained with a number of coalesced pigments. In one of these, Pigment BX-CR appeared as a mixture of free phthalocyanine, coated titanium dioxide particles, aggregates of CPC and TiO_2 , and perhaps free TiO_2 (uncoated). A 1:20 CPC/ TiO_2 tint looked like straight TiO_2 , i.e. no free phthalocyanine particles could be detected in the field chosen for photographing. This indicated that all the phthalocyanine in this product had been deposited on the surface of the TiO_2 .

Pigment BX-CR has been extracted in a Soxhlet extractor with benzene, ethyl acetate, glacial acetic acid, 95% ethanol, acetone, and water, respectively. Ethanol, acetone and water are the most efficient extractants; water being the preferred solvent for removal of inorganic and organic by-products as well as any unreacted starting materials, other than TiO_2 . Insoluble copper compounds were removed by washing the water-extracted pigment with NH_4OH-NH_4Cl solution, and after this extraction the residue apparently contained only titanium dioxide and phthalocyanine.

The aqueous extracts were separated by standard methods, and the chief products isolated and identified. The extracted pigment, after drying, was analyzed for copper, and the CPC content calculated as monochloro CPC. Results from these analyses on lots 1 and 3 of Pigment BX-CR follow.

lot No.	% Composition H ₂ O Extractable	Pigment BX-CR		
		NH_4OH-NH_4Cl Extractable	Monochloro CPC	TiO_2
1	9.33	0.93	20.18	69.56
3	7.0	0.67	21.26	71.07

lot No.	Cu in Extracted Pigment	Monochloro CPC in Crude (calculated)	Monochloro CPC* (Jackson Lab. Results)
1	2.35	20.18	18.5
3	2.4	21.26	20.35

Composition of Extracted Material (lot 1)

	%	% in lot 1
Phthalimide	28.55	2.93
H ₂ O Soluble Copper Salts**	15.60	1.82
Iron, as Fe ₂ O ₃	2.43	0.25
Copper oxides	9.06	0.93
Unidentified	45.64	4.55

*Obtained by acid pasting, isolating toner, and analysis of the toner for copper and chlorine.

**Calculated as Cu Cl₂.

The material designated above as unidentified consisted mostly of ammonium salts (from the water extraction only), and small amounts of amorphous organic materials. In addition, some free HCl is known to be present in the crude pigment.

Crude Pigment BX-CR was found to be acidic, an aqueous slurry having a pH of 4.65. A saturated solution of phthalimide had a pH of 4.35. BX-CR had a neutralization value of about 0.001 equiv. NaOH/gram. Most of the caustic was used to react with the phthalimide and soluble inorganic metallic salts as well as any free HCl which may have been present.

The extracted Pigment BX-CR samples were tinctorially equal to crude, and all (except one extracted with hot dilute HCl) equal to the latter in resistance to flocculation in lacquer. The HCl extracted pigment was non-flocculating "as is", but flocculated slightly when tinted with TiO₂. Despite the fact that removal of by-products did not harm the paint properties of the crude pigment, it appeared expedient

to determine the effect of known and probable by-products from the BX-CR process on the flocculation of CPC toner. Towards this end, the following materials were tested in composite CPC (BT-172-D)/TiO₂ grinds in lacquer using amounts of reagents ranging from 10% to 50% by weight of the CPC toner in the lacquer*.

NaCl
CuCl₂
Cu₂Cl₂
Titanium phthalate
Phthalimide
Copper phthalimide
Titanium "
Aluminum "
Ferric "
Crude product from water extraction of Pigment BX-CR

None of these agents had any beneficial effect on the flocculation of the CPC toner.

Copper phthalocyanine was "coated" in aqueous slurry with copper phthalimide (cf. 1085-17) but the treated pigment showed no improvement in flocculation resistance.

* See Experiments: 1085-1, 1085-2, 1085-3, 1085-13, 1045-28, 1002-33, 1002-34, 1002-43

TiO₂ (both "Ti PURES" FF, and R-610, respectively) was "coated" in aqueous slurry with phthalimide, copper, iron, aluminum, and titanium salts, respectively, of phthalimide (cf. 1085-14). Finally, TiO₂ (FF) was baked with phthalonitrile, and phthalimide, respectively, in amounts ranging from 10% to 50% by weight of the TiO₂ at 240-250°C. (cf. 1085-15). The products baked with phthalonitrile were light blue apparently due to some phthalocyanine formation. When these treated TiO₂ pigments were used to tint CPC toner in lacquer, none of the systems showed any less flocculation than controls made from untreated TiO₂.

Since none of the known or probable by-products in Pigment BX-CR had any beneficial effect on the flocculation of CPC toner, it appeared that theory 3 above should be abandoned.

A study of the nature of the toner content of Pigment BX-CR was started to determine whether products of the type postulated in theory 4 above could be isolated and characterized. It was felt that two types of phthalocyanines were most likely to occur in the coalesced pigment, viz., titanium or titanyl phthalocyanine, and/or a complex of CPC or monochloro CPC with titanium or an oxide or salt of this metal. Compounds of the latter type have been isolated recently at Jackson Laboratory from coalesced monochloro CPC/TiO₂

pigments containing stannous oxide, and chromous oxide, respectively.

A sample of crude titanium phthalocyanine obtained from Jackson Laboratory was found to be a non-flocculating toner when tinted with TiO_2 in lacquer. Furthermore, in one test, a mixture of 20% of this crude "TiPC" and 80% CPC toner was non-flocculating when extended 90% with TiO_2 in lacquer. This result has not been confirmed; three subsequent tests with composite grinds of "TiPC"/CPC/ TiO_2 failed to give the complete non-flocculation observed in the first trial. This result recalled a similar effect of crude iron phthalocyanine, discovered at Newark several years ago.* A mixture of 15% "FePC"/85% CPC was found to be non-flocculating in TiO_2 lacquer tints. The titanium phthalocyanine/CPC blend is considerably more intense than the iron phthalocyanine mixture.

The phthalocyanine in Pigment BX-CR can be completely removed by acid pasting the coalesced pigment, filtering the sulfuric acid solution of phthalocyanine from the undissolved TiO_2 , and drowning the filtrate in water. The toner recovered in this way was tinctorially about the same as monochloro CPC toner, and no better in its resistance to flocculation than the latter. These toners analyzed 0.03% to 0.05% TiO_2 . Therefore, it can be concluded that no titanium phthalocyanine existed in Pigment BX-CR since "TiPC" is stable to acid pasting. Furthermore, if a "two metal" complex of TiO_2 or other titanium derivative with CPC or monochloro CPC existed, it was broken down by solution in concentrated sulfuric acid.

Alpha methyl naphthalene, and alpha chlor naphthalene have been used as solvents to extract some of the toner from Pigment BX-CR, alpha chlor naphthalene being preferred. A sample of the crude pigment was extracted three times successively with this solvent at 230-250°C, and the crystallized toner from each extraction kept separately. The first fraction contained a large amount of phthalimide, which was removed by washing with hot acetone. The concentrated toner of this first fraction was a greenish blue which was non-flocculating in lacquer extended 80% with TiO_2 . The sample was too small for both lacquer test and analysis. The second toner fraction analyzed only 0.04% TiO_2 , and was not tested in lacquer. The third fraction was ground in lacquer extended 80% with TiO_2 when it flocculated almost as severely as regular CPC toner. These extractions are being repeated.

* See KN-41-172

One other explanation for the non-flocculating property of Pigment BX-CR was postulated, namely, formation of some non-flocculating chlorinated CPC in the process which could prevent the flocculation of the CPC formed by the normal reaction. It has been known for some time that Polychloro CPC green is non-flocculating when tinted with TiO_2 in lacquer. This toner when mixed with CPC (BT-172-D) in 40:60 ratio prevented the flocculation of the latter in lacquer tints (cf. 1085-8), but smaller amounts than 40% green toner were ineffective. A partially chlorinated (octa chloro CPC) toner, obtained from Jackson Laboratory, was found to flocculate severely and had no beneficial effect on the flocculation of regular CPC toners. Therefore, the above theory does not appear tenable on the basis of this evidence.

Only one non-phthalocyanine pigment has been found to inhibit flocculation of CPC toner in lacquer tints (cf. 1061-2). This is a co-acid pasted Violet DX/ TiO_2 lake which is non-flocculating per se (Violet DX toner flocculates severely). Following this lead, 50/50 mixtures of CPC toner and various precipitated and pigment dyestuff azo pigments, chrome yellows, and iron blue were tested in lacquers tinted with TiO_2 (cf. 1061-33). None of these prevented the flocculation of the CPC toner.

2. Texture

Crude Pigment BX-CR was found to be harsh textured (gritty), and poor in rate of strength development (ink mill evaluation) compared to equivalent TiO_2 extensions of standard "Monastral" Fast Blue B or BX. It was too gritty to disperse to satisfactory enamel fineness in a paint vehicle over a roller mill, but the crude pigment ball milled in paint vehicles to satisfactory enamel fineness.

The following grinding and treating methods have been studied in an effort to improve texture. Details can be found in the tables.

- Steam Micronization
- Air Micronization
- Salt Milling (1 qt. cans, and 50 gal. mill)
- Dry Ball Milling "as is"
- Dry Ball Milling with various agents
- Edge runner Milling (small mill at Chambers Works;
large mill at Baltimore)
- Micropulverization, various conditions with and
without agents
- Aqueous slurry treatments with various agents
- Extraction with water, and organic solvents
- Wet ball milling

Only steam micronization has been effective in improving the texture without harming the flexibility of the pigment in its resistance to flocculation. Salt milling also improved texture

but at the expense of flocculation resistance when further extended with TiO_2 . Steam micronization gave pigments of excellent wedge grit rating compared to controls of standard CPC or Ramapo Blue toners extended with "Ti PURE" R-610, but the rate of strength development of the micronized samples was inferior to the latter (ink mill evaluations). The micronized pigment ground in alkyd enamel over a 3-roll paint mill gave satisfactory enamel fineness. Thus, micronization appears to give a pigment suitable for general paint use. Micronization will make possible a totally dry process for Pigment BX-CR.

Wedge grit and strength development values for representative micronized samples, compared to those from other grinding or treating processes, and to controls are tabulated below.

Sample	Texture Pigment BX-CR #1 Transparent Varnish					
	Wedge Grit			Str. Development		
	2L	4L	6T	2L	4L	6T
Control: 25% BT-172-D/75% "TiPure" R-610	17-	17+	18	155	125	100
Control: 25% BP-173-D/75% " " "	17+	18	19-	108	103	100
Control: 25% BX Toner/75% " " "	13+	14+	16	230	190	110
Control: Salt Milled 25% BX toner - 75% "Ti Pure" R-610	15+	16	17+	140	125	103
BX-CR, Edge runnered & micronized	17-	17+	19-	160	140	110
BX-CR + TEA oleate, micronized	18-	18	19	200	175	115
BX-CR + Nuc zinc wetter, "	16+	17-	18	160	140	120
BX-CR, salt milled, 72 hrs.	15	16-	17+	155	145	110
BX-CR, edge runnered	13	13+	15+	240	200	115
BX-CR, wet ball milled	16+	17	17+	175	155	115
BX-CR, air micronized	14-	14-	14+	260	205	117
BX-CR, micropulverized	13-	13	14	300	250	125
BX-CR, dry ball milled	13	14-	15	275	200	130
BX-CR, water extracted	12	12+	17+	265	260	120
BX-CR, crude	13-	13	14-	300	250	130

The tinting strength of two micronized samples at 10T passes in No.1 litho varnish compared to 25% "Monastral" Fast BlueB, soft powder, (BT-172-D)/75% "T1 PURE" R-610, and 25% "Monastral" Fast Blue BX, lot 4/75% "T1 PURE" R-610, respectively, as controls was as follows:

Sample	Blue B Control = 100	Blue BX Control = 100
Micropulverized, lot 3	140	114
Steam micronized, 1051-18a ₂ , (lot 3)	117	95
Steam Micronized with Nuo Zinc Wetter (lot 3)	120	98

Lot 3 contained 21.26% Blue BX and 71.07% TiO₂. On this basis lot 3, micropulverized, appeared only slightly weak against TiO₂ extensions of standard toners, and when micronized was stronger than equivalent mechanical extensions of Blue BX toner.

A few grinds were made on the ink mill with heavier bodied varnishes than No.1 transparent. In #3 and #5 varnishes, crude BX-CR developed somewhat greater strength, but was too poor in wedge grit for practical application. A further slight improvement in wedge grit and strength development was obtained by grinding in #7 varnish on a heated mill. Grinding in these and #1 varnishes at high pigment/vehicle ratios also gave some slight ink improvements in these respects. The best strength development rating for the crude pigment was obtained by grinding in an air bodied linseed oil (Colloxin), but the inks livered too rapidly for wedge grit determination. This last result was of interest in view of the ease with which the crude coalesced pigments dispersed in linoleum. Also, Pigment BX-CR ball milled in raw linseed oil gave a very high gloss finish. These results suggest that the pigment is wetted better by linseed oil than with the regular lithographic varnishes.

A sample of BX-CR, lot 3, was air classified at the Experimental Station. About 20% of this pigment (micropulverized at Jackson Lab.) was above 80 microns in size. The small particle size fractions were ground on the ink mill, and rated for wedge grit. None of them, including the finest 0-10 micron portion, was any better in wedge grit than the starting pigment. These results suggest that the poor texture of the pigment is inherent, and not due to extraneous matter or small quantities of grit.

3. Other Evaluations

The easy dispersion of crude coalesced phthalocyanine pigments in linoleum noted in previous work has been confirmed. Crude Pigment BX-CR dispersed just as readily in the linoleum composition as the micropulverized product, and salt milled

Pigment BX-CR appeared to offer no advantages for this use over crude. However, the salt milled form was tinctorially somewhat brighter than crude. Monochrome CPC co-baked on whiting, 1:3, D-5419 (MDD-616) dispersed satisfactorily in linoleum but gave dull, very green composition compared to crude Pigment BX-CR.

The effect of Pigment BX-CR in preventing flocculation of CPC toner extended with TiO_2 has been demonstrated previously. This property has been evaluated for systems containing smaller quantities of BX-CR, and results are summarized below for lacquers pigmented as follows:

<u>% Pigment BX-CR</u>	<u>% Blue B Toner BT-172-D</u>	<u>% TiO_2</u>	<u>Flocculation</u>
75	6.25	18.75	none
50	12.5	37.5	none
25	18.75	56.25	slight
10	22.5	60.0	severe

Pigment BX-CR was superior in this property to Harmon's non-flocculating CPC/aluminum benzoate lake* (U.S. Pat. 2,327,472), i.e., Pigment BX-CR will prevent the flocculation of greater amounts of CPC toner than the latter lake. In general, flocculation resistance of Pigment BX-CR is superior to the Harmon lake.

Pigment BX-CR has been compared to 75% TiO_2 extensions of iron blue pigments for strength by rubout in varnish drier. The crude coalesced pigment was weaker than the TiO_2 extension of B-197-D, but about equal to that of B-216-D. Salt milled BX-CR showed a $\pm 10\%$ strength advantage over a 25% B-216-D/75% "Ti PURE" R-610 extension.

Pigment BX-CR has been evaluated in non-slip deck coating, Spec. 52-D-9, and appeared satisfactory in lightfastness and working properties for this use. These results have not been confirmed to date.

Considerable exposure experience has been obtained on Pigment BX-CR and related coalesced phthalocyanine/ TiO_2 pigments in alkyd and lacquer vehicles during the past six months. Those pigments made on "Ti PURE" R-610 appeared satisfactory in chalk resistance, color retention, and durability, and were equal in these properties, in most cases, to corresponding dry mix controls made with BT-172-D or BP-173-D (Blue B Toner, and rosinated Blue B) extended with "Ti PURE" R-610 in lacquer. Six months lacquer

* See KN-44-22 for evaluation of this product

exposure* in Florida of Pigment BX-CR, lot 1, made by the Parlin Laboratories of the Finishes Department both "as is" and in metallic finishes have checked these results.

In alkyd vehicles, Pigment BX-CR and related products on "Ti PURE" R-610 appeared somewhat less chalk resistant than corresponding dry mix controls of BT-172-D/"Ti PURE" R-610 after 6 months to 1 year Florida exposure, but were otherwise equal to the latter. However, this difference in chalk resistance was not very great and appeared to be within the limits of experimental error. Recently, the Philadelphia Laboratories of R&F have observed poor chalk resistance very much inferior to controls for Pigment BX-CR, lot 1, in glossy "Dulux". This behavior has never been observed in Newark's series. Independent exposures of this pigment by the Newport Laboratory have agreed with Newark's results. A sample of Pigment BX-CR, lot 3, has been submitted to Philadelphia for evaluation.

B. New Products of the Pigment BX-CR Type

One new product of this type has been prepared at Jackson Laboratory, CPC cobaked on "Ti PURE" R-LL (R-610) made with phthalonitrile and cupric sulfate in the presence of small amounts of calcium oxide. This product, of interest from theoretical and patent viewpoints, was a very greenish blue similar tinctorially to Pigment G-CR, and totally unlike similar dry mix extensions of Blue B toner with TiO_2 . Jackson Laboratory reported that the green shade pigment was not obtained from similar bakes in which blanc fixe or other inert extenders were substituted for TiO_2 substrate. Instead, these latter bakes gave the expected blue shade of CPC extensions.

This green shade Pigment BX-CR, D-5614 (MOD-747) analyzed 17% phthalocyanine, and 16.75% of the crude pigment was extractable with water and ammonia. An aqueous slurry of this pigment had a pH of 8.1, and titratable "acidity" about half that of crude Pigment BX-CR.

This product showed the same non-flocculating characteristics as Pigment BX-CR in lacquer vehicles, and is equal to the latter in flexibility in use, i.e. it can be extended with TiO_2 or fortified with CPC toners without flocculation occurring. The crude pigment had poor texture similar to that of crude Pigment BX-CR.

* Parlin Series J-2936

C. Blanc Fixe Counterpart of Pigment BX-CR (Cobaked Monochloro CPC/Blanc Fixe, 1:3)

This product has been of interest for some time as a "full shade" coalesced pigment for tinting Pigment BX-CR and other tint pigments, as well as a cheap phthalocyanine pigment for use where the non-flocculating properties of BX-CR are not essential. However, much less study has been given to this pigment either at Jackson Laboratory or at Newark than to its TiO_2 counterparts. To date only laboratory preparations have been made.

The crude pigment was harsh textured and appeared weaker in tinting strength than Pigment BX-CR crude. It dispersed satisfactorily in linoleum, but was weaker, duller and greener than equivalent extension of Blue B toner (BT-172-D). Micronization of this pigment is recommended to improve its tinctorial properties and texture.

In lacquer this pigment was somewhat duller "as is" than equivalent blanc fixe extension of BT-172-D, but weaker and greener in tint than the latter. It was non-flocculating "as is", but almost invariably showed flocculation and separation in paint vehicles when tinted with TiO_2 . However, a recent sample (D-5611, MDD-744) flocculated much less in a TiO_2 lacquer tint than equivalent blanc fixe/ TiO_2 extension of BT-172-D.

The crude blanc fixe pigment was more acidic than Pigment BX-CR. An aqueous slurry had a pH of 2.7 and a neutralization equivalent of 0.078 eq./100 g.

Outdoor exposures in durable vehicles are in progress in several series. To date, subject pigment both "as is" and tinted with "Ti PURE" R-610 has appeared equal in weathering characteristics to corresponding dry mix controls made with BT-172-D (Blue B toner) in both alkyd and lacquer vehicles after 6 to 9 months exposure in Florida and New Jersey, respectively.

D. Green G-CR Pigments (Polychloro Green CPC on Substrates)

Two forms of this pigment have been evaluated, viz., Green G-CR on "Ti PURE" R-LL for R-610, 1:2; and its counterpart on Blanc Fixe. These pigments, made by dry chlorination of their Pigment BX-CR counterparts, have been studied much less extensively than the latter. No semi-works manufacture has been attempted, and the process is still in the laboratory stage. However, the proposed process for preparation of Green G-CR pigment appears to be potentially cheaper than the present toner process. Since flocculation is no problem with polychloro CPC (the toner is non-flocculating) the preferred product for development is the blanc fixe pigment.

The properties of Green G-CR on "Ti PURE" R-LL were reported in KN-43-178 (cf. pp 81-84). The crude pigment was harsh textured, but its ease of dispersion in linoleum has been confirmed. Linoleums and paint films pigmented with it were darker and bluer "as is" than equivalent dry mix extensions of polychloro green toner, GT-486-D, or its resinated counterpart, GP-5014-D. Salt milling the crude pigment improved texture and tinting strength, but the salt milled pigment was considerably bluer than crude, or GT-486-D/TiO₂ tints. Green G-CR on TiO₂ was not as effective as GT-486-D in inhibiting the flocculation of BT-172-D/TiO₂ blends in lacquer.

Green G-CR on "Ti PURE" R-LL has shown poor chalk resistance and lightfastness as judged by its behavior after 6 to 8 months Florida exposure in durable vehicles. This is the expected behavior of pigments containing this type TiO₂.

Crude Green G-CR on blanc fixe was darker and slightly bluer in both linoleum and lacquer than dry mix extensions of GT-486-D/Blanc Fixe. It had harsh texture, but dispersed satisfactorily in linoleum to give intense green shade compositions. The tinting strength of the crude pigment was inferior to that of GT-486-D/blanc fixe mix in varnish drier. Salt milled G-CR on blanc fixe was found to have softer texture than crude and tinctorially equal to the latter. This salt milled pigment tinted with shading yellow, Y-493-D, approximated chrome greens G-550-D (light), and G-553-D (dark) in full shade but were more intense than the latter. The masstone-tint relationships of Green G-CR/Y-493-D blends were not the same as for the chrome greens, i.e., a masstone match with the coalesced pigment for a chrome green did not represent a tinctorial match to the latter in tint.* The salt milled pigment used in these evaluations was made at Jackson Laboratory. It did not give entirely satisfactory enamel fineness by mixing in alkyd enamel over a 3-roll paint mill being markedly inferior in this respect to GT-486-D/blanc fixe mix.

Green G-CR on blanc fixe has appeared to be fully equal to dry mix controls of GT-486-D both "as is" and extended with "Ti PURE" R-610 in chalk resistance, color retention, and durability after 6 months Florida exposure in durable vehicles.

* See KN-44-26 for a more detailed discussion of this phase of the problem

E. Pigment G-CR

This pigment has continued to be of interest because of its unusual green shade, and for technical reasons.

The crude pigment contained about 7% water extractable material, mostly organic in nature. The extracted pigment was equal tinctorially and in its paint properties to crude. An aqueous slurry had a pH of 10.5, and a neutralization equivalent of 0.065 eq. NaOH/100 g.

The crude pigment was harsh textured, but dispersed satisfactorily in linoleum giving interesting greenish blue compositions stable to heat cure and soap-fast. Salt milled Pigment G-CR had better texture, and was very red, intense and strong vs. crude. The salt milled pigment was redder than a salt milled mix of metal free phthalocyanine toner and "Ti PURE" LG, and even redder than Pigment BX-CR. Salt milled metal free phthalocyanine toner was a very dull bluish gray of no interest. Salt milled Pigment G-CR flocculated in lacquer, but may be of interest as a linoleum color.

Crude Pigment G-CR has proved less lightfast, and slightly less chalk resistant than dry mix BT-198-D (metal free phthalocyanine)/"Ti PURE" LG controls in durable vehicles after 6-8 months Florida exposure. Exposure in lacquer by Parlin Laboratory* has confirmed these results; Pigment G-CR showed too poor color retention after 6 months Florida exposure to be of interest to Parlin.

F. Green Z Pigments

Only one new product of this type has been made at Jackson Laboratory since November of last year. This was a "1:100" cobake on Kadox.

One crude Z (1:3) pigment contained about 16% of water extractable material. An aqueous slurry of another similar Green Z had pH 6.4. Its neutralization equivalent was 0.12 eq./100 g. The extracted pigment was equal tinctorially and in its paint properties to crude.

The crude Green Z pigments were harsh textured. Salt milling improved texture and radically altered their tinctorial properties. A salt milled Green Z was very intense, red and strong vs. crude.

* Parlin Series J-2936

Considerable exposure experience has been obtained on some of these products. All appeared to darken "as is", and fade more when extended with TiO_2 than corresponding dry mix controls of BT-172-D or Monochloro CPC and the appropriate zinc oxide. Some of the Green Z pigments have micro checked after 6-8 months Florida exposure in durable vehicles. Parlin exposure in lacquer*, six months Florida, indicated that a 1:3 Green Z changed too much in color to be acceptable. In addition metallies of this pigment showed the so called metallic effect being darker when viewed at one angle, but lighter at another.

G. Other Products

1. Coalesced Pigments on Ultramarine

About 15 products of this type have been made at Jackson Laboratory, the majority during 1944. Cobaked and cosalt milled pigments from metal free phthalocyanine, CPC, and Monochloro CPC were also prepared. The most interesting tinctorially were the cobaked pigments containing metal free phthalocyanine in amounts of 5% or less which were intense transparent blues somewhat similar to ultramarine, and superior tinctorially to dry mixes of BT-198-D and ultramarine. The most interesting property of all the cobaked phthalocyanine/ultramarine products was their non-flocculation in lacquer/ TiO_2 tints. Mechanical mixtures of phthalocyanine toner, and salt milled phthalocyanine/ultramarine combinations, all flocculated in the usual way when extended with TiO_2 in lacquer.

More complete details on these evaluations are given in the tables.

2. Coalesced Pigments on Stannous and Stannic Oxides, and Mixtures of These with Blanc Fixe.

A monochloro CPC pigment made on stannic oxide, 1:3, was non-flocculating "as is", but flocculated when extended with TiO_2 in lacquer. A similar product on stannous oxide showed much less flocculation when extended with TiO_2 in lacquer, but was not the equal of Pigment BX-CR in this respect. Tinctorially, the stannous oxide pigment was a dull, greenish blue of little interest. Both the stannic and stannous oxide pigments were practically non-flocculating when tinted with TiO_2 in alkyd enamels, but dry mix controls of the tin oxide/ TiO_2 /Fast Blue BX toner appeared about equal to them in this respect.

A coalesced monochloro CPC pigment on a mixture of blanc fixe and stannous oxide as substrate has been of interest as a non-flocculating CPC product containing blanc fixe. This product was composed of 1 BX-CPC:2.34 Blanc Fixe:0.66 SnO.

* Series J-2936

Similar products on mixtures of blanc fixe and prime white pigment oxides are discussed below. The stannous oxide containing product was found to be non-flocculating in lacquer when extended with TiO_2 in tests made at Tech. Lab. However, flocculation tests run at Newark showed that this pigment exhibited considerable flocculation when tested in this way in lacquer, but was non-flocculating in "Dulux"/ TiO_2 systems. Stannous oxide appears to have some effect in preventing the flocculation of CPC when the oxide is present as substrate in a coalesced phthalocyanine pigment, but is less effective than TiO_2 or the other preferred substrates.

3. Coalesced Pigments on Mixtures of Other Metallic Oxides and Blanc Fixe.

Two products of this type have been studied at Newark, viz., 1 CPC-BX:2.6 Blanc Fixe:0.4 ZnO , and 1 CPC-BX:2.2 Blanc Fixe:0.4 ZnO :0.4 TiO_2 . The first named pigment was better in its resistance to flocculation in TiO_2 lacquer systems than a straight blanc fixe pigment, and about equal to the similar blanc fixe/stannous oxide pigment described above. The blanc fixe pigment containing both ZnO and TiO_2 was the best of these types showing only slight flocculation when tinted with TiO_2 in lacquer. None of these blanc fixe-metallic oxide pigments were equal to Pigment BX-CR in flocculation resistance. From a practical viewpoint, blends of Pigment BX-CR and its blanc fixe counterpart are suggested as equivalents to these cobaked products made on mixtures of blanc fixe and an oxide pigment.

V. EXPERIMENTAL DATA

All evaluations with coalesced phthalocyanine pigments made since November 1, 1943 are listed in the following tables. New products submitted since this date, and evaluations made on earlier products since KN-43-178 issued are included. The following tables are continued from the above mentioned report. The tables are divided into sections according to the method used for coalescence as follows:

- A. Cobaking Method
- B. Salt Milling Method
- C. Ball Milling without Salt
- D. Bonding or Coating Processes
- E. Experimental Organic Toners and Inorganic Pigments
Used as Controls in Evaluation Studies.

Section E was not included in KN-43-178, and includes all special or experimental pigments employed as controls in paint and other evaluations of the coalesced pigments.

The reader is referred to the preceding report, KN-43-178, for further explanation of the system of classification of the experimental products, nomenclature, and abbreviations used in these tables.

* The asterisk denotes new products prepared since November 1943.

TABLE A.

PRODUCTS PREPARED BY COBAKING

Al. Copper Phthalocyanine

b. Zinc Oxide

Proportions PC/Substrate	Substrate ZnO	D-Number	J.L. Code No.	Treat- ment	Evaluation
1:3	U.S.P.ppt.	5410	MDD-614		Exposure "as is" and extended 50% with "Ti PURE" R-610. Rezyl 807-1.
1:3	U.S.P.ppt.	5411	MDD-615		Exposure "as is" and extended 50% with "Ti PURE" R-610 Rezyl 807-1.

Notebook or File Record	Properties	Suggested Applications
1061-16(Series 6821)	See KN-43-178, p.35	
1061-16(Series 6821)	See KN-43-178, p.35	

Al. Copper Phthalocyanine

c. Inorganic Blues

1. Ultramarine

Proportions CPC/Sub- strate	Substrate Ultramarine	D- Number	J.L.Code No.	Treat- ment	Evaluation
1:19	#511	5564 ^B	MDD-690	Micro- pulverized	1. Tintorial proper- ties and flocculation in lacquer "as is" and extended with TiO₂. 2. Exposure "as is" and extended with TiO₂ in Rexyl 807-1 3. Lacquer properties. Exposure in lacquer. Weatherometer exposure. 4. Alkyd properties. Exposure outdoors and in weatherometer. 5. In Navy Ship Paints 52-P-44, 52-P-45, 52-P-47. Exposure.
1:19	#511	5646 ^B	MDD-786		1. Flocculation proper- ties in lacquer "as is" and extended with TiO₂. Exposure outdoors. 2. Alkyd properties. Exposure outdoors and in weatherometer. 3. In Navy Ship Paints 52-P-44, 52-P-45, 52-P-47. Exposure.

Notebook or File Record	Properties	Suggested Applications
1. 1061-13	Darker and duller than ultramarine, and more opaque in lacquer and alkyd. Non-flocculating in paints when ex- tended with TiO_2 . About equal in strength to ultramarine. Intense and very red vs. corresponding dry mix of CFC toner and ultramarine.	Dependent on outdoor durability and light- fastness. Cheap paint pigment of ultra- marine type.
2. 1061-15(Series 6820)		
3. 1061-25(Series 6840)		
4. 1061-31(Series 6856)		
5. 1061-32(Series 6857)		
1. 1061-25(Series 6840)	Tintorially equal to above sample, D-5564. Non-flocculating in paints when extended with TiO_2 .	As above for D-5564.
2. 1061-31(Series 6856)		
3. 1061-32(Series 6857)		

A2. Monochloro GFE

a. Titanium Dioxide
1. Pigment BK-CR

Proportions PG/Substrate	Substrate "Ti Pure"	D- Number	J.L.Code No.	Treat- ment	Evaluation
1:3	R-610	5181	MDD-477	Screened crude	1. Evaluation as a linoleum pigment. 2. Evaluation in Spec. 52-D-9, non-slip deck coverings.
1:3	R-610	5179	MDD-479	Micro- pulverized	1. As Linoleum Pigments
Semi Works lot 1		5246	MDD-520 J-59-F-64A		2. Extraction with Benzene 3. Neutralization equivalent. 4. Lacquer Properties of C ₆ H ₆ extracted pigment. 5. Extraction with water and ammonia-ammonium chloride. 6. Lacquer properties of water extracted pigment. 7. Copper analysis of H ₂ O extracted pigment. 8. Extraction with 95% ethanol, glacial acetic acid, and ethyl acetate. Lacquer properties of these extracted pigments. 9. Extraction with 2% HCl; and 2% NH ₄ OH. Lacquer properties of these extracted pigments.
1:3	R-610	5179 5246			10. Extraction with acetone 11. Lacquer Properties of acetone extracted pigment. 12. Texture Acetone Extracted Pigment. 13. Texture water extracted pigment. 14. Texture study in #1 litho varnish at various propor- tions; in #3 litho varnish, and #5 litho varnish. 15. Treatment in water slurry with various resins, naphthenates, poly- dioxolane, celloxin, TRA esters.
Semi Works lot 1					

Notebook or File Record	Properties	Suggested Applications
1. 1045-24,40	Dispersed readily in linoleum to give attractive, light blue composition.	See KN-43-178, p. 37,39.
2. 1051-15,16	Weatherometer exposure in non-clip deck covering showed crude EX-CR to be more permanent than ultramarine which fades badly.	
1. 1045-24,40	See KN-43-178, p.39 Extraction and Analysis EX-CR, lot 1, contained:	See KN-43-178,p.39
2. 1045-25	20.18% Monochloro CPC	
3. 1045-28	69.56% TiO ₂	
4. 1045-25	10.26% extractable material.	
5. 1045-26-28 1045-45	Water was most efficient extractant, and all by products, except CuO, can be removed with it.	
6. 1045-26,28,45	CuO is extracted with NH ₄ OH, NH ₄ Cl solution. The extractable product consisted of:	
7. 1045-28	Phthalimide	
8. 1045-27	Copper salts	
	Ammonium salts	
	Small amounts of unidentified materials.	
9. 1045-32,39	Conc. H ₂ SO ₄ removed all toner pigment from Pigment EX-CR. Alpha-methyl naphthalene and alpha-chloronaphthalene dissolve small amounts of CPC toner, and sufficient quantities of toner have been recovered from Pig. EX-CR with these solvents for analysis.	
10. 1045-45,35,23	The toner removed with conc. H ₂ SO ₄ flocculated in same way in TiO ₂ extension in lacquer as regular Blue B toner. The acid pasted toner contained only negligible amount of titanium, and no titanium phthalocyanine.	
11. 1045-45	Toner CPC removed from Pigment EX-CR with α-chloro naphthalene varied depending on the fraction studied. The first fraction was contaminated with phthalimide, but was non-flocculating in lacquer-TiO ₂ tints. A second fraction, from another extraction of the same sample, contained negligible quantities of Ti or TiO ₂ . A third fraction from the same sample flocculated when extended with TiO ₂ in lacquer in the same way as regular Blue B toner (BT-172-D). Results must be repeated,	

Proportions PG/Substrate	Substrate "Ti Pure"	D- Number	J.L.Code No.	Treat- ment	Evaluation
					16. Texture of preceding wet treated products.
					17. Lacquer properties of above products.
					18. Extraction of phthalocya- nine component with alpha-methyl naphthalene. Analysis of extracted toner.
					19. Salt milling (1 qt. can) 1 to 24 hrs.
					20. Texture of above salt milled products.
					21. Lacquer properties (flocculation, etc) of above salt milled pigments.
					22. Flocculations: Lacquer BK-CR + CPC toner + TiO ₂ .
					23. Tintorial and strength comparisons to iron blue by rubout.
					24. Conc. H ₂ SO ₄ extraction of toner. Paint properties, analysis of extracted toner.
					25. Extraction with alpha chloro naphthalene.

Notebook or File Record	Properties	Suggested Applications
	and larger samples of the toner pigments obtained.	
15. 1085-5	<u>Extracted Pigment BX-CR</u>	
	The organic solvents used to remove by-products, water, and dilute ammonia did not harm the non-flocculating properties of the pigment in lacquer when tinted further with TiO_2 . Dilute HCl when used for extraction gave an extracted pigment non-flocculating "as is" in lacquer, but showing slight flocculation when tinted with TiO_2 . However, the extent of this flocculation was much less than mechanically mixed control of Blue BX toner/ TiO_2 .	
16. 1085-34		
17. 1061-26		
18. 1085-41		
19. 1068-26	Tinctorially, all the extracted samples were slightly more intense than crude, but otherwise equal.	
20. 1068-30		
21. 1061-18	<u>Neutralization Equivalent and Acidity</u>	
	pH, aqueous slurry: 4.65	
	Neutralization equivalent: 0.095 eq. NaOH/100 grams.	
22. 1045-11		
1061-19		
23. 1051-14	<u>Texture and Methods for Improving Texture</u>	
	Crude micropulverized Pigment BX-CR is harsh and gritty and inferior in rate of strength development vs. controls of standard CPC toners extended with "Ti Pure" R-610 ground on a 3-roll ink mill in #1 litho varnish. Slightly better wedge grit and strength development resulted when Pigment BX-CR was ground at high F/S ratios in #1 varnish, or in heavier litho varnishes (#3, #6) but the improvement was of no practicable value.	
24. 1085-42	Extraction of BX-CR with water or acetone did not improve its texture. Micropulverization with polydiexolanes was also ineffective. Aqueous slurry treatment with resinsates, naphthenates, etc., did not improve texture. These latter products all showed slight flocculation when tinted with TiO_2 in lacquer.	
25. 1085-45	Salt milling BX-CR (laboratory) for 1 to 6 hrs. greatly improved wedge grit and strength development, but the salt milled products were inferior in both these respects to standard CPC/ TiO_2 mix controls. Salt milling for 6 hrs. did not affect the resistance of BX-CR to flocculation, or harm	

Proportions PC/Substrate	Substrate "Ti Pure"	D- Number	J.L. Code No.	Treat- ment	Evaluation
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1:3
Semi Works
lot 3

R-610

5629^M

MDD-750

Micropul-
verized

1. Tintorial comparison to lot 1 in lacquer
2. Extraction with water, and $\text{NH}_4\text{OH}-\text{NH}_4\text{Cl}$. Copper analysis of extracted pigment. Lacquer properties.
3. Micropulverized with polydioxolanes. Texture.
4. Texture in #1 litho varnish, #7 litho varnish, and colloxin (air bodied linseed oil).
5. Ball milled, dry "as is"; with "Dupanol" WA; with Alkanol B; with Picolyte Resin; with Gum Dammar; with Stabellite Resin; with Galex Resin; with Barium Rosinate; with

Notebook or File Record	Properties	Suggested Applications
	its flexibility in use. Longer periods of milling resulted in a pigment which flocculated when tinted with TiO_2 in lacquer, but appeared O.K. at same depth in alkyl enamels. Salt milling was not as effective in improving texture as steam micronization discussed under lot 3, B-5629, (following entry). <u>Flocculation Properties</u> A mixture: 25% Pigment BX-CR 18-3/4% CPC toner 56-1/4% "Ti Pure" R-610 in lacquer showed only slight flocculation. Increasing the proportion of Pigment BX-CR in such a mix gave non-flocculating lacquer systems. <u>Strength vs. Iron Blue</u> Crude Micropulverized Pig. BX-CR weak vs. 25% B-197-D/75% "Ti Pure" R-610 Crude micropulverized Pig. BX-CR ±10% strong vs. 25% B-216-D/75% "Ti Pure" R-610.	
1. 1085-14	<u>Tinctorial Properties</u> More intense, more redder, stronger than lot 1, B-5179 (5246).	Same as that listed in KN-43-178, p.39
2. 1085-20	<u>Composition</u> 21.26% CPC (monochlore) 71.07% TiO_2 7.0% H_2O Extractable 0.67% $\text{NH}_4\text{OH-NH}_4\text{Cl}$ extractable	
3. 1085-28	<u>Air classifications</u> 0.10 microns: 18.2% 10.20 " : 16.2% 20-40 " : 23% 40.80 " : 27.6%	
4. 1085-36	Residue, over 80: 15%	
5. 1051-17	<u>Texture</u> Steam micronization, with or without agents, gave satisfactory wedge grit and improved strength development (inferior in this latter property to Blue B/R-610 dry mix control).	

Proportions PG/Substrate	Substrate	D- Number	J.L. Code Treat- No. ment	Evaluation
1:3	R-610	5629	MDD-750	Trisethanolamine Oleate; with celloxin; with Nuade; with Nuodex Aluminum; with Nuc sine wetter. Texture evaluation of above samples. 6. Steam micronization of ball milled samples containing Alkanol B; Barium Resinate; Trisethanolamine Oleate; Celloxin; Nuade; Nuodex Aluminum; Nuc sine wetter. Texture evaluation of above. 7. Edge runner milling "as is"; with Staybellite Resin; with Nuade. Steam micronization of edge runner milled sample "as is" (15 min.) 8. Texture of Products of #7. 9. Micropulverization "as is" and with same agents as in #5 above. Texture of these products. 10. Flocculation properties of products from #3, #5, #6, #7, and #9 above in lacquer "as is" and extended 50% with "Ti Pure" R-610. 11. Air classification (Experimental Station). Texture. 12. Paint Texture Evaluation of micronized sample vs. BT-172-D/TiO₂ control, and micropulverized BX-GR. 13. Specially micropulverized samples. Texture.
1:3 Semi Works lot 3	"Ti Pure" R-610	5629	MDD-750	

Notebook or File
 Record

Properties

Suggested
 Applications

6. 1051-17
 1051-21

Ball milling (dry) or micropulverization with the various agents listed did not improve texture. Edge runner milling with and without agents listed did not improve texture. Samples ball milled with Duponol, Copol resin, Staybellite, Colloxin, and Munde flocculated slightly when extended with T10 in lacquer.

7. 1051-18

Grinding the crude micropulverized pigment in #1 litho varnish gave slightly better strength development and wedge grit than in #2 varnish. Colloxin (air bodied linseed oil) as vehicle produced better strength development than in any of the litho varnishes, but the ink bedded too rapidly for wedge grit evaluation.

8. 1051-17,21

Strength of Steam Micronized Samples
 117 pts = 100 pts. 25% BT-172-B/75% "Ti Pure" R-610
 95 pts = 100 pts. 25% BK toner, lot 4/75% "Ti Pure" R-610
 102 pts = 100 pts. 25% BK toner, lot 4 (soft powder)
 75% "Ti Pure" R-610

9. 1051-20

above comparison at 10T passes.
 Roller Mill Enamel Compounding with Micronized
 Pigment BK-CN

10. 1061-26

Micronized sample was equal to BT-172-B control in enamel fineness, and appeared superior in gloss. The micronized pigment was greener than Blue B control, or Blue BK control.

11. Letter
 C.R. Berry to
 V. Chalupski,
 4-7-44 Roller
 Particle Size
 Analyzer Test
 No. 488.

1085-46

12. Letter: D.H. Stubbs
 to A. Siegel,
 4-4-44

13. 1051-22

A2. Monochlore CPG

A. Titanium Dioxide
1. Pigment BX-CR

Proportions PG/Substrate	Substrate "Ti Pure"	D- Number	J.L. Code No.	Treat- ment	Evaluation
1:3 Semi Works lot 1	R-610	5199	MDD-486	Dry Ball Milled (BX-CR)	Flocculation in lacquer "as is" and extended 50% with "Ti Pure" R-610
1:3 Semi Works lot 1	R-610	5357	J-59-F-71	Salt Milled	1. As linoleum pigment 2. Texture in #1 litho varnish. 3. Flocculation in lacquer "as is" and extended with TiO ₂ . 4. Comparison by rubout to iron blues strength and tinctorial properties.
1:3 Semi Works lot 1	R-610	5585 [*]	MDD-732	Edge Runner Milled (Jackson Lab.)	1. Texture in #1 litho varnish
<u>3. "Ti PURE" R-LL for R-610</u>					
1:3	R-LL	5414 [*]	MDD-747	Made from PN+CuSO ₄ + CaO+TiO ₂	1. Rubout in V.D. compared Pigment G-CR 2. Flocculation properties in lacquer "as is", ex- tended with TiO ₂ ; forbi- fied with PG toners. Tinctorial comparisons to Pigment BX-CR, Fig. G-CR; Green 2; and Green G-CR. 3. Extraction with water. Analysis for CPG. 4. Neutralization equiva- lent. 5. Texture in #1 litho varnish. 6. Exposure in alkyd GE-2458.

Notesbook or File Record	Properties	Suggested applications
1061-26	O.K. in flocculation "as is" and extended with TiO_2 in lacquer.	See KH-43-178, p.43
1. 1045-24 2. (1068-30 (1045-36 3. 1061-18,26 4. 1051-14	Crude Pig. BK-CR equal to salt milled pigment in linoleum. Better strength development than crude, but only a better wedge grit. Stronger than 75% R-610 extensions of iron blues (B-197-D, B-216-D). Flocculates when extended with TiO_2 in lacquer.	See KH-43-178, p.43
1. 1045-43	No improvement in texture over micropulverized.	No advantages over crude micro-pulverized.
1. 1051-19 2. 1085-7 3. 1085-10 4. 1085-16 5. 1085-39 6. 1061-22(Series 6830)	Reported by J.L. to contain 17% colored pigment. Found by Newark analytical method: 16% CFC. Green, dull and weak vs. Pigment BK-CR. Non-flocculating coalesced CFC pigment in lacquer "as is", fortified with CFC or metal free PC toner; and extended to 1:19 with TiO_2 . Tintorially like Pigment G-CR in lacquer. pH, aq. slurry, 8.1 contains about half titratable "acidity" of BK-CR. 16.75% of crude pigment is extractable with H_2O ; NH_4OH . Hard, gritty pigment inferior in strength development to standard BK toners extruded with TiO_2 .	Similar to Pigment G-CR and Pigment BK-CR. No apparent advantages compared to these pigments.

Proportions PG/Substrate	Substrate	R- Number	J.L.Code No.	Treat- ment	Evaluation
1:2	R-LL	5352	J-59-F-68		Exposure "as is" and ex- tended with "Ti Pure" R-610 in Resyl 807-1
6. "Ti TINTS"					
1:20	"Ti Tint" Yellow, MTP-53	5164	MDD-473		Exposure Resyl 807-1
B. Zinc Oxide Green & Pigments					
	Substrate ZnO				
1:3	U.S.F.ppt.	5355	J-59-F-77		Exposure "as is" and ex- tended with "Ti Pure" R-610, Resyl 807-1
1:3	U.S.F.ppt	5408	MDD-612		Exposure "as is" and ex- tended with "Ti Pure" R-610. Resyl 807-1
1:3	U.S.F.ppt.	5500	MDD-645		Exposure "as is" and ex- tended 50% with "Ti Pure" R-610. Resyl 807-1.
1:3	ZEE-55	5414	MDD-619		1.Extraction with Benzene. Lacquer properties of extracted pigment. 2.Extraction with water. 3.Lacquer properties of water extracted pigment. 4.Exposure "as is" and ex- tended 50% with "Ti Pure" R-610 in Resyl 807-1.
1:3	Horsehead XI-3 (N-525)	5430	MDD-620		Exposure "as is" and ex- tended 50% with "Ti Pure" R-610. Resyl 807-1.
1:3	XI-503 Horsehead	5431	MDD-621		Exposure "as is" and ex- tended 50% with "Ti Pure" R-610. Resyl 807-1.

Notebook or File Record	Properties	Suggested Application
1061-15 (Series 6820)	See KN-43-178, p.47	
1061-15 (Series 6820)	See KN-43-178, p.51	
1061-16 (Series 6821)	See KN-43-178, p.53	
1061-16 (Series 6821)	See KN-43-178, p.53	
1061-16 (Series 6821)	See KN-43-178, p.53	
1. 1045-25,25A 2. 1045-30 3. 1045-32 4. 1061-16 (Series 6821)	12.7% pigment extractable with benzene. 15.7% pigment extractable with water. Extracted pigments similar tinctorially and in fluorescence properties to crude.	See KN-43-178, p.53
1061-16 (Series 6821)	See KN-43-178, p.55	
1061-16 (Series 6821)	See KN-43-178, p.55	

Proportions PC/Substrate	Substrate ZnO	D- Number	J.L.Code No.	Treat- ment	Evaluation
1:3	Horshead XX-601	5432	MDD-622		Exposure "as is" and extended 50% with "Ti Pure" R-610, Rezyl 807-1
1:3	Florence Green Seal 8	5433	MDD-623		Exposure "as is" and extended 50% with "Ti Pure" R-610, Rezyl 807-1.
1:3	Kadox Black Label 15	5434	MDD-624		1. Exposure "as is" and extended 50% with "Ti Pure" R-610, Rezyl 807-1. 2. Salt milled (1 qt.can) for 1 to 24 hours.
1:3	Kadox Black Label 15	5612 ^m	MDD-745	None	1. Flocculation lacquer: "as is" and extended with TiO ₂ . 2. Neutralization equiva- lent. 3. Texture in #1 litho varnish. 4. Rubout evaluation in VD.
1:3	35% Loaded ZnO-Lehigh 6	5435	MDD-625		Exposure "as is" and extended 50% with "Ti Pure" R-610 in Rezyl 807-1.
1:6	U.S.P.ppt.	5498	MDD-643		Exposure "as is" and extended 50% with "Ti Pure" R-610. Rezyl 807-1.
1:15	U.S.P.ppt.	5497	MDD-642		Exposure "as is" and extended 50% with "Ti Pure" R-610. Rezyl 807-1.
1:20	U.S.P.ppt.	5499	MDD-644		Exposure "as is" and extended 50% with "Ti Pure" R-610. Rezyl 807-1

Notebook or File Record	Properties	Suggested Application
1061-16 (Series 6821)	See KN-43-178, p. 55	
1061-16 (Series 6821)	See KN-43-178, p. 55	
1. 1061-16 (Series 6821)	See KN-43-178, p. 55	
2. 1068-28	Salt milled pigment v. strong, and v. red va. crude.	See KN-43-178, p. 55
1. 1085-7	Similar tinctorially to other Green 2 pigments. Non- floculating "as is" and extend- ed with TiO ₂ in lacquer. pH in H ₂ O: 6.4 Neutralization eq: 0.12 eq. NaOH/100g. Poor texture.	
2. 1085-16		
3. 1085-37		
4. 1051-19		
1061-16 (Series 6821)	See KN-43-178, p. 55	
1061-16 (Series 6821)	See KN-43-178, p. 55.	
1061-16 (Series 6821)	See KN-43-178, p. 55	
1061-16 (Series 6821)	See KN-43-178, P. 55	

Proportions PG/Substrate	Substrate	D- Number	J.L.Code No.	Treat- ment	Evaluation
1:100	Kadox Black Label 15	5613 ^M	MDD-746	None	1. Rubout in V.D. 2. Exposure "as is", extended with "Ti Pure" R-610, and tinted with "Monstral" Fast Blue B toner (BT-172-D)
<u>f. Whiting</u>					
1:3	Whiting	5419	MDD-616		As a linoleum pigment
<u>g. Blanc Fixe and Barites</u>					
1:3	Blanc Fixe 5303		J-59-F-67	None ⁸	Exposure "as is" and extended with "Ti Pure" R-610 in Rexyl 807-1.
1:3	Blanc Fixe 5611 ^M		MDD-744	Replenish- ment sample for 5303	1. Neutralization equiva- lent. 2. As linoleum pigment 3. Flocculation in lacquer "as is" and extended with TiO ₂
<u>h. "Ti Bar", "Ti Cal", "Ti Sil", and TiO₂/Blanc Fixe, and other mixtures with Blanc Fixe</u>					
1:1.5:1.5	TiO ₂ :Blanc Fixe 5353		J-59-F-69		Exposure "as is" and extended with TiO ₂ in Rexyl 807-1.
1:2.6:0.4	Blanc Fixe Zinc Oxide	5654 ^M	MDD-793	None	Paint properties (alkyd & lacquer) Flocculation. Exposure.
1:2.2:0.4: 0.4	Blanc Fixe	5655 ^M	MDD-794	None	Paint properties (alkyd and lacquer) Flocculation. Exposure.
1:2.34:0.66	Blanc Fixe SnO	5653 ^M	MDD-792	None	Paint properties (alkyd & lacquer) Flocculation. Exposure

Notebook or File Record	Properties	Suggested Application
1. 1051-19 2. 1061-22 (Series 6830)	V.weak, light green pigment	Possible use as low-chalking zinc oxide of "Ti Blue" shade range.
1045-24	V.green and dull in linoleum vs. Pigment BK-CR.	See KN-43-178, p. 57
1061-15(Series 6820)	See KN-43-178, p. 59	
1. 1085-16 2. 1085-44 3. 1061-34	pH, H ₂ O slurry: 2.7 Neutralization equiv: 0.078 eq.NaOH/100g Dispersed satisfactorily in linoleum. Showed only slight flocculation in lacquer tinted with TiO ₂ .	Same as D-5303 above.
1061-15(Series 6820)	See KN-43-178, p. 61	
1061-28(Series 6849)	Non-flocculating "as is" in lacquer and "Dulux". Showed cons.flocculation when extended with TiO ₂ in lacquer, but OK in alkyd. Greener product in tint than Pigment BK-CR.	No apparent applications that cannot be filled with BK on blanc fixe and Pigment BK-CR.
1061-28 (Series 6849)	Non-flocculating "as is" in lacquer and alkyd. Flocculated slightly when tinted with TiO ₂ in lacquer but less than D-5654 above. Greener in tint than Pig. BK-CR.	As above.
1061-28	Dull and green pigment vs. Pigment BK-CR. Flocculated considerably when tinted with TiO ₂ in lacquer, but otherwise OK both in lacquer and "Dulux".	As above.

m. Inorganic Blues

Proportions PC/Substrate	Substrate	D- Number	J.L.Code No.	Treat- ment	Evaluation
1:19	Iron Blue B-216-B	5252	MDD-526		Exposure "as is" and ex- tended with "Ti Pure" R-610 in Resyl 807-1
1:3	Ultramarine	5443	MDD-626		1. Exposure "as is" and ex- tended 50% with "Ti Pure" R-610 in Resyl 807-1. 2. Lacquer properties. Ex- posure outdoors and in weatherometer. 3. Alkyd properties. Expo- sure outdoors and in weatherometer.
1:19	Ultramarine 5647 ^M #511		MDD-787		1. Flocculation in lacquer: "as is" and extended with "Ti Pure" R-610. Exposure. 2. Alkyd properties. Exposure outdoors and in weather- ometer.
<u>n. Ferrite Yellow</u>					
1:4	Ferrite Yellow	5356	J-59-F-78		1. Exposure "as is" and ex- tended with "Ti Pure" R-610 in Resyl 807-1. 2. Exposure by Parlin in lacquer.
<u>q. Miscellaneous Substrates</u>					
<u>3' Tin Oxides</u>					
1:3	Stannic Oxide	5651 ^M	MDD-790	None	Paint properties in lacquer and alkyd both "as is" and extended with "Ti Pure" R-610. Exposure.
1:3	Stannic Oxide	5652 ^M	MDD-791	None	Paint properties in lacquer and alkyd both "as is" and "Ti Pure" R-610 extensions. Exposure.

Notebook or File Record	Properties	Suggested Applications
1061-15 (Series 6820)	See KN-43-178, p. 63	
1. 1061-15(Series 6820)	See KN-43-178, p. 65 Dark, dull, and green vs. ultra- marine. Non-flocculating when extended with TiO_2 in lacquer	None
2. 1061-25(Series 6840)		
3. 1061-31(Series 6856)		
1. 1061-25(Series 6840)	Dark, dull and greener than ultra- marine, and inferior tinctorially to corresponding metal free product. Non-flocculating when extended with TiO_2 in lacquer or alkyd enamel.	As a cheap paint pigment of ultra- marine shade de- pending on dura- bility and light- fastness.
2. 1061-31(Series 6856)		
1. 1061-15(Series 6820)	See KN-43-178, p. 67	
2. Series J-2942		
1061-28(Series 6849)	Fairly intense blue. Practically tenser depth in full shade. Floccu- lated when extended with TiO_2 in lacquer, but O.K. in alkyd enamel.	None
1061-28(Series 6849)	Dark, dull, green pigment. Non- flocculating "as is" in lacquer and alkyd, and when extended with TiO_2 in alkyd enamel. Showed cons. flocculation when extended with TiO_2 in lacquer.	None

A4. Metal Free Phthalocyanine
a. Titanium Dioxide
Pigment G-CR

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Proportions PG/Substrate	Substrate "Ti Pure"	D- Number	J.L.Code No.	Treat- ment	Evaluation
1:4	LG	5248 5304 5610 ^m	MDD-522 J-59-F-66 MDD-743	Micro- pulverised	1. Rubout in V.D. 2. As a linoleum Pigment 3. Extraction with Benzene. Lacquer properties of ext. pigment. 4. Extraction with water. 5. Lacquer properties of water extracted pigment. 6. Exposure: Resyl 807-1. 7. Salt Milling (1 qt.cans) 1 to 24 hrs. Eval. in V.D. 8. pH and neutralization equivalent.
Semi Works lot 2					
1:4	LG	5376	J-59-F-80	Salt Milled	1. As linoleum pigment. 2. Exposure Resyl 807-1.
Semi Works lot 2					
1:4	LG	5656 ^m	MDD-795	Salt Milled	1. Rubouts in V.D. Floculation in lacquer.
Semi Works lot 2					
1:4	LG	5249	MDD-543	Dry Ball Milled	Exposure: Resyl 807-1
1:4	LG	5320	MDD-579	Chg.2 acid Pasted	Exposure: Resyl 807-1
1:4	LG	5319	MDD-578	Chg.1 acid pasted	Exposure: Resyl 807-1
<u>2. "Ti Pure" LG other than Pig. G-CR</u>					
1:4	Spec.BRC-190	5270	MDD-544		Exposure: Resyl 807-1
1:4	Spec.BRC-191	5271	MDD-545		Exposure: Resyl 807-1
1:4	Spec.BRC-192	5272	MDD-546		Exposure: Resyl 807-1
1:4	Spec.BRC-193	5273	MDD-547		Exposure: Resyl 807-1

Notabook or File Record	Properties	Suggested Applications
1. 1051-19 2. 1045-24,40 3. 1045-25,25A	Interesting, greenish blue, pigment readily dispersible in linoleum.	See KN-43-178, p. 71
4. 1045-29 5. 1045-32	7.15% extractable material in pigment. Extracted pigment equal tinctorially and in paint proper- ties to crude.	
6. 1061-14(Series 6819)	pH, H ₂ O slurry: 10.5 Neutralization eq: 0.065 eq.NaOH/100g	
7. 1068-27 8. 1085-16	Salt milled pigment is v.strong, intense, v.red vs. crude.	
1. 1045-24,40 2. 1061-14(Series 6819)	V.red in linoleum vs. crude. Redder pigment than Pigment BK-CR	See KN-43-178, p. 71
1. 1061-29	Greener, and duller than earlier salt milled sample, D-5376. Red strong, and intense vs. BT-198-D/ TiO ₂ mix in lacquer.	Same as for D-5376 above.
1061-14(Series 6819)	See KN-43-178, p. 73	
1061-14(Series 6819)	See KN-43-178, p. 73	
1061-14(Series 6819)	See KN-43-178, p. 73	
1061-14(Series 6819)	See KN-43-178, p. 75	
1061-14(Series 6819)	See KN-43-178, p. 75	
1061-14(Series 6819)	See KN-43-178, p. 75	
1061-14(Series 6819)	See KN-43-178, p. 75	

b. Zinc Oxide

Proportions PG/Substrate	Substrate ZnO	D- Number	Trade Name	Treat- ment	Evaluation
1:4	U.S.P. ppt	5261	MDD-535		Exposure: Resyl 807-1

g. Blanc Fixe and Barytes

1:4 Nickel Baker	Blanc Fixe	5264	MDD-538		Exposure: Resyl 807-1
1:4	Barytes	5265	MDD-539		Exposure: Resyl 807-1

h. Inorganic Blue Pigments

1:19	B-216-D Iron Blue	5253	MDD-527		Exposure: Resyl 807-1
1:3	Ultramarine #511	5445	MDD-628		1. Exposure: Resyl 807-1 2. Lacquer properties, and exposure:TiO ₂ extension 3. Alkyd properties. Exposure.
1:99 Nickel Baker	Ultra- marine #511	5640 ^M	MDD-780	Micro- pulver- ized.	1. Lacquer properties, and exposure:TiO ₂ extension. 2. Alkyd properties. Expo- sure. 3. In Navy Ship Paints. Expo- sure.
1:39 Nickel Baker	Ultra- marine #511	5641 ^M	MDD-781	Micropul-	1. Lacquer properties and exposure:TiO ₂ extension. 2. As above: alkyd 3. In Navy Ship Paints. Exposure.
1:19 Nickel Baker	Ultra- marine #511	5565 ^M	MDD-691	Micropul- verized	1. Lacquer properties, and exposure:TiO ₂ extension. 2. As above: alkyd 3. In Navy Ship Paints. Exposure.
1:19 Nickel Baker	Ultra- marine #511	5642	MDD-782	Micropul- verized	1. Lacquer properties, and exposure:TiO ₂ extension. 2. As above: alkyd 3. In Navy Ship Paints, exposure.

Notabook or File Records	Properties	Suggested Applications
1061-16(Series 6821)	See KN-43-178, p. 79	
1061-14(Series 6819)	See KN-43-178, p. 79	
1061-14(Series 6819)	See KN-43-178, p. 81	
1061-15(Series 6820)	See KN-43-178, p. 81	
1. 1061-18(Series 6820) 2. 1061-25(Series 6840) 3. 1061-31(Series 6856)	Darker, duller, and greener than 1:19 product. Non-flocculating in lacquer when extended with TiO ₂	None
1. 1061-25(Series 6840) 2. 1061-31(Series 6856) 3. 1061-32(Series 6857)	Similar tinctorially to ultramarine. Non-flocculating when extended with TiO ₂ in lacquer	None
1. 1061-25(Series 6840) 2. 1061-31(Series 6856) 3. 1061-32(Series 6857)	Similar to ultramarine tinctorially, non-flocculating when extended with TiO ₂ in lacquer.	Dependent on durability and lightfastness.
1. 1061-25(Series 6840) 2. 1061-31(Series 6856) 3. 1061-32(Series 6857)	Slightly darker and greener than ultramarine but more intense than dry mix control of Blue G Toner and ultramarine. Non-flocculating in lacquer extended with TiO ₂ .	As above.
1. 1061-25(Series 6840) 2. 1061-31(Series 6856) 3. 1061-32(Series 6857)	Same as D-5565	As above.

Proportions PG/Substrate	Substrate	D- Number	J.L.Code No.	Treat- ment	Evaluation
1:19 Iron Baker	Ultramarine #511	5643 ^m	MDD-783	Micropul- verized	1. Lacquer properties, and exposure: TiO ₂ extension. 2. As above: alkyd
1:19 Nickel Baker	Ultramarine #511	5644 ^m	MDD-784	Micropul- verized	1. Lacquer properties, and exposure: TiO ₂ extension. 2. As above: alkyd 3. In Navy Ship Paints. Exposure.
1:4 Nickel Baker	Ultramarine #511	5645 ^m	MDD-785	Micropul- verized	1. Lacquer properties, and exposure: TiO ₂ extension. 2. As above: alkyd
A5. Polychlore GFC (Green)					
a. Titanium Dioxide					
1. Green G-CR					
1:2	^{"Ti Pure"} R-LL for R-610	5477	J-59-P-83	H ₂ O ex- tracted	1. As linoleum pigment. 2. Extraction with benzene lacquer properties of ext. pigment. 3. Flocculation in lacquer in mixtures containing Green G-CR, ST-172-D ("Monastral" Fast Blue B) and TiO ₂ compared to mixtures of ST-486-D ("Monastral" Fast Green G) and TiO ₂ . 4. Salt milling (1 qt. cans) 1 to 24 hrs. 5. Exposure: Reyl 807-1.
g. Blanc Fixe and Barytes					
Green G-CR					
1:2	Blanc Fixe	5222	MDD-509	Extracted	Exposure: Reyl 807-1 "as is" and TiO ₂ extension.

Notebook or File Record	Properties	Suggested Applications
1. 1061-25(Series 6840) 2. 1061-31(Series 6856)	Much darker than products made in nickel baker	As above
1. 1061-25(Series 6840) 2. 1061-31(Series 6856) 3. 1061-32(Series 6857)	Dark, green, and opaque vs. ultramarine. Similar tinctorially to dry mix. Non-flocculating when extended with TiO_2 in lacquer	None
1. 1061-25(Series 6840) 2. 1061-31(Series 6856)	Stronger product than D-5644. Similar properties.	None
1. 1045-24,40 2. 1045-25,25A 3. 1085-8 4. 1068-29 5. 1061-15(Series 6820)	See KN-43-178, p. 81 Intense green tint pigment for linoleum. Dispersed satisfactorily. About 5% extractable with benzene. Extracted pigment tinctorially equal to crude. Salt milled pigment was stronger and bluer than crude. Green G-CR was not as effective as Polychloro green toner, GT-486-D, in preventing flocculation of Blue B toner/TiO_2 mixture in lacquer.	
1061-15(Series 6820)	See KN-43-178, p. 83	

Proportions PG/Substrate	Substrate	D- Number	J.L.Code No.	Treat- ment	Evaluation
1:2	Blanc Fixe	5609 ^M	MDD-742	Extracted	1. pH and neutralization equivalent. 2. Texture in #1 litho varnish. 3. Exposure: Lacquer, and GE-2458 alkyd. 4. As linoleum pigment
1:2	Blanc Fixe	5686 ^M	MDD-809	Salt milled	1. Blends with Y-493-D to match chrome greens; roller mill paint formulation.

Notebook or File Record	Properties	Suggested Applications
1. 1085-16	Harsh, gritty pigment. Can be salt milled to intense, strong pigment otherwise equal tinctorially to crude. pH, H ₂ O slurry: 7.55 Neutralization eq: 0.036 eq NaOH/100g Disperses satisfactorily in linoleum.	Same as for D-5222, above
2. 1085-38		
3. 1061-22(Series 6830)		
4. 1085-44		
1. 1051-23	Approx. matches in alkyd enamel for G-550-D and G-553-D can be obtained by blending with Y-493-D. Mixes with green G-GR were more intense than chrome greens. Texture of this salt milled Green G-GR inferior to GT-486-D. The salt milled pigment did not give good enamel fineness by mixing in alkyd enamel over a 3-roll paint mill.	Paint and linoleum pigment.

TABLE B
PRODUCTS PREPARED BY SALT MILLING

B1. Copper Phthalocyanine
m. Inorganic Blue Pigments

Proportions PC/Substrate	Substrate	D- Number	J.L. Code No.	Treat- ment	Evaluations
1:19	Ultramarine #511	5566 ^m	MDD-692	Salt Milled, H ₂ O extract- ed.	Flocculation: TiO ₂ ex- tension in lacquer. Exposure: "as is" and TiO ₂ extension in Resyl 807-1.

B2. Monochlore CPC
m. Inorganic Blue Pigments

1:3	Ultramarine #511	5444	MDD-627	Salt Milled	Exposure: "as is" and TiO ₂ extension in Resyl 807-1.
1:19	Ultramarine #511	5567 ^m	MDD-693	Salt Milled	Lacquer properties: "as is" and TiO ₂ extensions. Exposure: "as is" and TiO ₂ extension in Resyl 807-1.

B4. Metal Free Phthalocyanine
a. Titanium Dioxide

1:4	<u>"Ti Pure"</u> LG	5370	MDD-606		Exposure: Resyl 807-1
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B5. Polychlore CPC (Green)
a. Titanium Dioxide

1:3	R-610	5347	MDD-599		As linoleum pigment. Exposure: Resyl 807-1
1:3	FF	5348	MDD-600		Exposure: Resyl 807-1

Notebook or File Record	Properties	Suggested Applications
1061-13	Tintorially, duller and more opaque than similar cobaked product. Flocculated when ex- tended with TiO_2 in lacquer.	None
1061-15(Series 6820)	Tintorially, duller than simi- lar cobaked product. Flocculated when extended with TiO_2 in lacquer.	None
1061-13 1061-15(Series 6820)	Same as for D-5444	None
1061-14(Series 6819)	See KN-43-178, p. 89	
1045-24 1061-15(Series 6820)	Dispersed satisfactorily in linoleum. Greener pigment than salt milled Green G-CR.	See KN-43-178, p. 91
1061-15(Series 6820)	See KN-43-178, p. 91	

Notebook or File Record	Properties	Suggested Applications
1061-13	Tintorially, duller and more opaque than similar cobaked product. Flocculated when ex- tended with TiO_2 in lacquer.	None
1061-15(Series 6820)	Tintorially, duller than simi- lar cobaked product. Flocculated when extended with TiO_2 in lacquer.	None
1061-13	Same as for D-5444	None
1061-15(Series 6820)		
1061-14(Series 6819)	See KN-43-178, p. 89	
1045-24 1061-15(Series 6820)	Dispersed satisfactorily in linoleum. Greener pigment than salt milled Green G-CR.	See KN-43-178, p. 91
1061-15(Series 6820)	See KN-43-178, p. 91	

TABLE I

**SPECIAL C. P. INORGANIC PIGMENTS AND
ORGANIC TONERS USED AS CONTROLS IN
EVALUATIONS OF THE COALESCED PHTHALOY-
AMINE PIGMENTS.**

El. Phthalocyanine
u. Copper Phthalocyanine

Description	D-No.	J.L.-No.	Remarks
GFC Crude B. Extracted with hot NH_4OH	5315	MDP-574	Used as control in salt milling experiments.
Salt Milled GFC	5358	S-59-T-75	Used as control for co- baked, salt milled, and other GFC products in evaluations and exposures.
Titanium Phthalo- cyanine	5648	MDP-788	Flocculation of TiO_2 tints in lacquer, and effect of TIFC on flocculation of GFC (1061-24). Titanium phthalocyanine is a greenish blue pigment which is non-floccula- ting when extended with TiO_2 in lacquer. A mix of 20% TIFC and 80% Blue B Toner when extended with TiO_2 in lacquer was practically non-floccula- ting. Check runs of these grinds showed the usual severe flocculation (1061-34). Iron PC was found to behave similarly several years ago. The crude TIFC was acid pasted to yield a stronger toner also non-flocculating when extended with TiO_2 in lacquer (1085-43). % TiO_2 in crude: 6.5% % " " acid pasted toner: 9%

**E1. Phthalocyanine
e. Monochloro CPG**

<u>Description</u>	<u>D-No.</u>	<u>J.L.-No.</u>	<u>Remarks</u>
Crude E. Extracted with hot NaOH	5316	MDD-575	Used as control in salt milling experiments.
Salt Milled Blue BX	5377	J-59-F-81	Used as controls in evaluations in paints, etc., and in exposure series.
"Monastral" Fast Blue BX, lot 4 (Tech. Lab. Standard)	5505 4848	-	As D-5377
"Monastral" Fast Blue BX, soft powder, lot 3	5625	-	As D-5377

d. Chlorinated Phthalocyanines other than Monochlor

"Octachloro PC"	5667	MDD-800	Chlorinated CPG; 24% chlorine. Flocculation properties in lacquer, and effect of this chloro CPG on flocculation of CPG toner (1061-30). This toner flocculated more severely in lacquer than Blue B extended with TiO_2 . Mixes of this toner with Blue B showed no improvement in flocculation over regular Blue B/ TiO_2 tints in lacquer.
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e. Metal Free Phthalocyanine

Salt Milled Toner	5619	MDD-748	1. Texture, ink mill (1085-40) 2. Flocculation in lacquer when extended with TiO_2 (1061-29). Very dull, gray blue pigment of little interest. Flocculated in lacquer when extended with TiO_2 in same way as standard Blue G.
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E2. Other Organic Toners

Description	B.No.	J.L.No.	Remarks
Violet DK " "	5327 5621	J-59-F-72 J-50-F-1	Also used Violet DK/"Ti Pure" R-LL (R-610) lake, salt milled, D-5423, J-57-F-82. As shading color for OPC, coalesced OPC, and as salt color. Exposures and paint evaluations. See KN-43-178 KI-44-29
"Ponsol" Brilliant Violet 4RN Salt Milled	5375	J-53-F-83	As control vs. Violet DK pigments above. See above reports, and Exposure Series 6744.
"Lithocel" Fast Yellow 3 OD, paste	5471	-	As shading color for Pigment BK-OR, and Green G-OR, respectively, to produce interesting lightfast greens. See Series 6787.

E3. Chrome Yellows, G.P. - Treated

Y-232-D, Zinc Yellow, baked 2 hrs. at 190°C.	5326	MDD-585	Controls for cobaked PC pigments on Y-232-D. See KN-43-178, pp. 65, 81.
Y-469-D, Chrome Yellow, baked 2 hrs. at 190°C.	5325	MDD-584	Controls for cobaked PC pigments on Y-469-D. See KN-43-178, pp. 65, 81.
Y-538-D, Chrome Yellow, baked 2 hrs. at 190°C.	5324	MDD-583	Controls for cobaked PC pigments on Y-538-D. See KN-43-178, p. 65.

E4. Inorganic Blue Pigments.

Ultramarine #511	5478	-	As controls in paint work for coalesced phthalocyanines made on this ultramarine. See 1061-13, 1061-15 (Series 6820); 1061-25 (Series 6840); 1061-31 (Series 6856); 1061-32 (Series 6857).
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25. Titanium Dioxide

Description	D-No.	J.L.-Code	Remarks
"Ti Pure" FF, salt milled	5372	MDD-608	Control for salt milled "Ti Pure" FF tints. See Series 6764
Super Soft "Ti Pure" FF	5392	-	Control for products made on this grade of TiO ₂ . See Series 6764.
"Ti Pure" R-610, baked 2 hrs. at 190°C.	5407	MDD-611	Control for cobaked products on "Ti Pure" R-610
"Ti Pure" R-610, salt milled	5374	MDD-610	Control for salt milled products made with "Ti Pure" R-610. See Series 6764.
"Ti Pure" R-LL for R-610	5363	-	A sample of this TiO ₂ used at at Jackson Laboratory for baking and milling preparations. Used as control in exposure series.
"Ti Pure" R-LL for R-610, salt milled	5373	MDD-609	Control for salt milled "Ti Pure" R-LL tints. See Series 6764.

26. Zinc Oxide

U.S.P. ppt. ZnO Mallin Kreutz	5364	-	Sample of this brand of zinc oxide used as substrate for Green Z pigments. of KN-43-178, pp. 51,53.
Horsehead XK-3	N-525	-	Modular. Average particle size 0.35- 0.45 microns. Control for Green Z, KN-43-178, p.55.
Horsehead XK-503	5403		Modular. Average particle size 0.6 to 1.0 micron. Control for Green Z, KN-43-178, p.55.
Horsehead XK-601	5405		Acicular. Used as control for Green Z, KN-43-178, p.55.
Florence Green Seal 8	5404		Modular. Average particle size 0.25 to 0.4 micron. Used as control for Green Z, KN-43-178, p. 55.

E6. Zinc Oxide

Description	D-No.	J.L.-No.	Remarks
Kadox Black Label 15	N-149		Nodular. Average particle size 0.11 to 0.17 micron. Used as control for Green Z, KN-43-178, p. 55.
35% Leaded ZnO, Lehigh 6	5400		Average particle size 0.4 to 0.6 micron 62-66% ZnO, remainder basic sulfate white lead. Used as control for Green Z, KN-43-178, p. 55.

E7. Other Inorganic Oxides

Stannous Oxide	5662	MDD-797	Used as control in evaluation of coalesced pigments containing SnO.
Stannic Oxide	5663	MDD-798	Used as control in evaluation of coalesced pigments con- taining SnO ₂