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NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

PIGMENT STABILITY AT HIGH TEMPERATURES

Period Covered December, 1963 - Present (Part Time)

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NEWARK PLANT

PIGMENTS COLOR RESEARCH REPORT

SUBJECT: PIGMENT STABILITY AT HIGH TEMPERATURES

PERIOD COVERED: December, 1963 - Present (Part Time)

SUBMITTED BY: C. W. Manger C W Manger Date Submitted: 8/12/68

APPROVED BY: B. H. PERKINS B H Perkins Date Released: 9/11/68

ABSTRACT

Most organic pigments, including QA's and CPC's, which are reasonably stable to dry heat, show marked color changes in polystyrene and other plastics processed at high temperatures (> 400°F.). It has been shown that pigment solubility is the predominant cause of loss of tinting strength, hue change, and poor lightfastness. Solubility promotes crystal growth and phase conversion and also the pigment can remain in solution in the solid polymer. CPC-AF and QAAF markedly enhance stability of CPC Blue and QA, respectively, by inhibiting crystal growth and phase conversion through a solution-adsorption mechanism. A relatively few pigments are unstable to heat, per se, and lose color through decomposition, e.g., chrome yellow and several azo pigments.

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PIGMENT STABILITY AT HIGH TEMPERATURES

I. INTRODUCTION

Work at Chestnut Run on the injection molder in the latter part of 1963 emphasized that most of our colored pigments show marked color changes in polystyrene and other plastics as a function of molding temperature. The maximum satisfactory temperature for most pigments lies somewhere between 400° and 600°F. The objectives of this study were to determine causes of pigment failure, to establish the principles which govern pigment stability in molten resins, and to set up guides for pigment improvement in this application.

Most of this report was composed during January, 1965, however, its issuance was delayed for inclusion of results of additional studies which were postponed indefinitely.

II. SUMMARY AND CONCLUSIONS

1. Solubility of the pigment in the hot plastic is the primary cause of color change at high temperatures in our more important organic pigment, including those which are of "Monastral" quality in other applications. In many of the less stable pigments such as some of the azos and chrome pigments, decomposition predominates.

2. Solubility promotes crystal growth as well as phase conversion of less stable polymorphs. Pigment also remains in solution after molding, in many cases, resulting in loss of tinting strength, hue change, and poor lightfastness.

3. The loss of tinting strength as the temperature is increased is due primarily to crystal growth. Hue changes can be due to phase conversion to a more stable polymorph or to crystal growth alone. Poor lightfastness is primarily a result of pigment retained in solution after molding.

4. Where solubility is the principal cause of color change with temperature, the magnitude of the change varies considerably with colored pigment concentration in the plastic. At low concentrations, a greater percentage of the total colored pigment can go into solution and recrystallize or remain in solution after molding to give a greater change than the same pigment at relatively high concentrations.

5. Solubility can also effect phase conversion of less stable polymorphs. For example, alpha-phase CPC blue can convert to the beta phase, and gamma-phase quinacridone can convert to the beta phase, resulting in hue changes in both cases.

6. The most promising approaches to enhanced pigment stability include (a) use of less soluble pigment types, (b) insulation of the pigment from the resin by an inert coating, (c) use of crystal growth inhibitors. (Note: All three approaches have been explored since this study was made, e,g., dicarboxyQA, "Krolor", and CPC-AF.)

7. In the quinacridone family, there is a direct correlation of solubility in solvents, such as dimethylformamide and 1-chloronaphthalene, and color change with temperature in plastics. For example, the much less soluble 2,9-dichloroquinacridone shows much less color change than the parent quinacridones and the much more soluble 4,11-dichloroquinacridone shows a much greater color change.

8. Addition of a phase stabilizer/crystal growth inhibitor such as QAAF to a QA pigment markedly enhances stability in molten plastics. CPC-AF also has a marked effect on the stability of the meta-stable alpha-phase CPC Blue (BT-427-D). A study of the mode of action of the agents CPC-AF and QAAF has indicated a solution/adsorption mechanism where the agent is adsorbed on the pigment particles from solution. The agents QAAF and CPC-AF have no apparent stabilization effect on QA and CPC when exposed to high temperatures (dry, in air) indicating that a solution/adsorption mechanism is most likely.

9. CPC-AF is not effective in stabilizing the alpha-phase CPC without the presence of calcium Staybelite. With no calcium Staybelite present, there is essentially no stabilizing effect. Magnesium Staybelite is at least equally as effective as the calcium Staybelite, whereas other metal Staybelites and rosinates have much less effect. The role of the calcium Staybelite and magnesium Staybelite has not been definitely resolved. It may act as a dispersant for the CPC-AF.

10. Alpha-phase CPC can grow to larger crystals of the alpha phase before the eventual conversion to the beta phase when exposed to solvents.

11. Marked stabilization to crystal growth in hot solvents can be obtained by heating CPC blues and QA's as the dry pigments or as mill powders before extraction. Apparently crystal growth is inhibited by a reaction product which is on the crystal surfaces or is available through a solution/adsorption mechanism.

12. Many pigments show evidence of decomposition especially at temperatures approaching 600°F. There is some evidence of reduction in several pigments in polystyrene at 600°F., e.g., CPC green, chrome yellow, molybdate orange, and several azo pigments. Quinacridonequinone shows apparent reduction to a blue form.

13. Calcium Staybelite effects a marked improvement of the Fadeometer lightfastness of the TiO_2 pigment, R-101. After 100 hours exposure, R-101 molded in polystyrene at 1.0 phr (per hundred parts of resin) shows considerable yellowing. Calcium Staybelite /R-101 at 0.1/1.0 phr in polystyrene shows essentially no change on exposure.

14. A "valley parameter", obtained from measurements of the valleys between x-ray diffraction doublets, has been developed for use in indicating growth of the alpha-phase CPC before converting to beta phase.

III. PATENT SITUATION

The patentability of the following is being explored:

1. Stabilization of TiO_2 with calcium rosinates.
2. Crystal growth inhibition by dry heating of pigments.
3. Crystal growth inhibition by CPC-AF and QAAF.

IV. PROGRAM

Some of the information obtained in this study has contributed, in part, in the development of new products such as "Krolor" and 2,9-dicarboxyQA.

V. GENERAL DISCUSSION

The following principles of high temperature stability of colored pigments have been previously accepted or were suggested by this study:

A major cause of the instability of many colored organic pigments in plastics at high temperatures is solubility of the pigment in the molten plastic. In the case of pigments which are soluble, the pigment dissolves in the molten plastic as a function of temperature and time up to the limit of solubility. The overall effect of solubility is a function of the pigmentation level, i.e., at very low color concentration (e.g., less than 0.01 phr) a large percentage of the pigment would be in solution and would have a marked effect on the observed color and lightfastness. For example, RT-759-D, γ-QA, shows considerable solution in polystyrene at 0.01 phr. At higher pigmentation levels, the percentage of pigment in solution is less and there is a smaller effect on the observed color and lightfastness. The poor lightfastness is a result of the instability of the pigment solution to light.

Pigments which are soluble in the molten plastic can also dissolve and recrystallize from solution where the concentration is above that of a saturated solution. This would tend to give larger crystals and more stable polymorphs resulting in weak tints and possibly different hues. Pigments which are of the most stable polymorphic form can dissolve and recrystallize as larger crystals.

The pigment retained in solution after molding is usually fluorescent under UV radiation, its lightfastness is usually very poor, and its color may be quite different from that of the pigment out of solution, causing a change in hue.

In pigments which exist in more than one polymorphic form, such as the quinacridones and CPC's, phase conversion is possible at elevated temperatures in polystyrene and other plastics, e.g., gamma quinacridone can convert to beta phase, and alpha CPC can convert to beta phase. Large crystallite size gamma quinacridone is somewhat more resistant to phase conversion in polystyrene than smaller crystallite sized material but the effects of pigment solubility are comparable.

It is quite definite that polystyrene at 600°F. is a reducing environment. Evidence for this includes: blueness in polychloro CPC green, blueness in the yellow quinacridonequinone, greenness and dullness of chrome yellows, decolorization of several azo pigment dyestuffs, and slight yellowing of TiO_2. In cases where solubility of the pigment predominates, evidence for reduction is not detectable.

The crystal growth inhibitors, CPC-AF and QAAF, being somewhat more soluble than the parent pigments, actually add to the amount of pigment in solution after molding, and, at low pigment concentrations (ca. 0.01 phr), would actually have a deleterious effect on the pigmented plastic. However, at relatively high pigment concentration (ca. 0.1 phr) these agents have a very marked beneficial effect on color change due to inhibition of crystal growth. Their mode of action is primarily that of solution/adsorption where the agent dissolves and then adsorbs on the pigment particles thus preventing crystal growth. In polystyrene, at 0.1 phr, these agents increase the maximum satisfactory temperature from ca. 450°F. to ca. 500°F. At temperatures above 500°F., the relative solubilities of the agent and pigment are such that efficient absorption of the agent on the pigment is not likely and crystal growth can occur.

VI. EXPERIMENTAL DETAILS

A. Plastic Systems Studied NB 1762 12/26/63

Most of the information regarding plastics described in this report is based on observations of pigments in general purpose polystyrene (Dow Styron 666 K27 Resin) injection molded on the Van Dorn injection molder at Chestnut Run. In most cases, dry blends of pigment, resin and TiO_2 (R-101) were made in a can on a Red Devil paint shaker. This mix was then placed in the injection molding machine and molds were made at 400°F. as quickly as possible to serve as controls. Subsequent molds were made after holding the resin/pigment mixture at various higher temperatures. Limited observations were also made on pigments dispersed in linear polyethylene (Alathon 7040).

B. Pigment Solubility 1762-14

The solubility of various pigments in plastic systems has been demonstrated through the use of spectrophotometric reflectance and transmittance spectra of tints and masstone transparencies, respectively. The spectra in the visible region (400-700 nanometers) of pigments in solution are usually sufficiently different from those of the equivalent dispersions (no solution) as shown in Figure 1. Figure 1 shows reflectance spectra of polystyrene molds of γ-QA, RT-759-D, made after holding in the molding machine at 400°F. and at 600°F. The QA in solution absorbs at ca. 420 and 500 nm whereas the dispersion absorbs at ca. 495, 530, and 565 nm.

Other tests for pigment solubility are included in the appended Memorandum Report.

Evidence of pigment solubility has been obtained in many pigment/plastic systems including the following:

Pigment Type	Code	System
γ-QA	RT-759-D	Polystyrene
"	RT-790-D	"
"	RT-796-D	"
β-QA	RT-791-D	"
"	"	Vinyl
"	RT-796-D	Polystyrene
QA Scarlet	RT-786-D	"
"	RP-830-D	"
Toluidine Red	RT-386-D	"
Orange F	YT-565-D	"
CPC Blue R	BT-391-D	"
CPC Blue RF	BT-427-D	"
CPC Blue G	BT-383-D	"
" " "	"	Vinyl
4,11-Cl_2QA	1847-14C (EEJ)	"

C. Behavior of Clear Polystyrene and Common Extenders 1762-14

Clear polystyrene shows essentially no difference when molded at 400°F. and 600°F. in the molding machine. Unless the pigment reacts with the polystyrene, there would be substantially no color change due to the resin itself. There is a slight fluorescence under UV radiation of the 400° mold and somewhat more in the 600° mold. It should be noted that considerable yellowing occurs when polystyrene is heated in an excess of air (oxidation).

Titanium dioxide, R-101, at 1.0 phr (per 100 resin), shows very slight yellowing at 600° vs. 400°F. in the absence of air. This would have a minor effect on color change in tints.

Calcium Staybelite, N-865-A, as a 0.1/1.0 R-101 phr "tint", shows very slight yellowing at 600° vs. 400°F., but this is probably due to the yellowing of the R-101, since it is equal, if not better, to R-101 at 400° and at 600°F. This extender should have no effect on the color change in tints except where the pigment may interact with the extender either chemically or as a solvent for the pigment. This extender effects a marked improvement of the lightfastness of R-101 (NB 1762-25).

Calcium rosinate as a "tint" shows very slight yellowing at 600° vs. 400°F. which may be due primarily to the R-101. However, it is yellow vs. R-101 and calcium Staybelite at 400° and at 600°. This extender would have a small effect on the hue of tints at all temperatures. There is a possibility of interaction with the pigment.

Nickel carbonate, N-947-B, as a "tint" shows very slight yellowing at 600° vs. 400°F. which may be due to the R-101. It is greenish vs. R-101 at 400° and at 600°F. This extender would have a small effect on the hue of tints at all temperatures. There is also a possibility of interaction with the pigment.

In summary, the polystyrene resin, R-101 titanium dioxide, calcium Staybelite, calcium rosinate, and nickel carbonate, in the absence of any interaction with the colored pigment, would have at most only a minor effect on the observed color changes at 600° vs. 400° in polystyrene, however, the possibility of interaction has not been completely ruled out.

D. Effect of High Temperature per se 1762-17

Crystal growth and phase conversion of certain pigments such as the quinacridones and CPC's can occur at temperatures in the order of 300°C. (572°F.) and higher. For example, the dry gamma quinacridones, RT-790-D and RT-796-D, when heated at 315°C. under high vacuum for ten minutes, showed partial conversion to beta phase and noticeable crystal growth. Noticeable crystal growth also occurred when the beta quinacridones, RT-791-D and RT-845-D, were heated in air at 315°C. for ten minutes. This means that the unsubstituted QA's and CPC's, even if rendered insoluble, could give color changes at high temperatures in plastic systems due to the high temperature alone.

Certain pigments, such as chrome yellow and some azo pigments, decompose quite readily when exposed to temperatures $> 400°F$.

E. Application of Electron Microscopy Plate No. 3526, 3527

Electron micrographs were obtained on pigments extracted from polystyrene molds by dissolving the polystyrene in ethyl acetate, followed by centrifugation.

Electron micrographs showed marked crystal growth at 600°F. molding temperatures in all of the CPC blues examined, i.e., BT-297-D, BT-427-D, BT-380-D, BT-391-D, and BT-449-D. Even the ordinarily stable beta-phase pigment, BT-297-D, grew into needle-like crystals up to one micron long, in a polystyrene tint at 0.1/1.0 phr molded at 600°F. as seen in Figure 2. (The opaque particles are TiO_2.).

F. CPC-AF 1762-20

Meta-stable alpha-phase chlorine-free CPC such as Blue R, BT-391-D, is color stable in molded polystyrene up to about 450°F. at 0.1/1.0 phr. At higher temperatures, there is a marked crystal growth and phase conversion to the stable beta phase. However, BT-427-D (75% Blue R/15% CPC-AF/10% Ca Staybelite) is color stable at 0.1/1.0 phr up to about 550°F. The agent CPC-AF, in conjunction with Ca Staybelite, inhibits growth of the crystal and conversion to beta phase. At higher temperatures and/or lower colored pigment concentrations, the stabilizing effect is less pronounced. An accelerated test for beta conversion of the alpha phase Blue R, which involves exposing the pigment to boiling ethyl benzene (to simulate polystyrene) for ten minutes, was used to study the effect of the calcium Staybelite in conjunction with the CPC-AF. Various other calcium compounds and the solvent-soluble compounds of other metals were dry-mixed with Blue R and CPC-AF at the composition levels of BT-427-D, i.e., Blue R 75%/CPC-AF 15%/ CaSx 10% and exposed to boiling ethyl benzene for ten minutes. The extent of conversion to beta phase was as follows:

Compound	% Beta Phase
None	100
Ca Staybelite	10+
Staybelite resin	100
Mg Staybelite	10
Sr Staybelite	50
Ni Staybelite	50
Ba Staybelite	70
Ca rosinate	70
Ba rosinate	70
Ca oleate	60
Ca acetate	90
Cu naphthenate	80
Al stearate	100
Zn stearate	100

The MgSx appears to have a slight advantage over the CaSx whereas all other compounds examined were much less effective.

G. QAAF, AQD, DDQA 1762-24

The experimental beta quinacridone RT-845-D (6.5% QAAF) shows a marked improvement in color stability in molded polystyrene vs. the untreated RT-791-D at 0.1/1.0 phr. To study the effect of various agents on QA's, an accelerated testing method for crystal growth of the quinacridones was used where dry pigment was exposed to boiling DMF for 10 minutes. In the QA's examined so far, there is a general correlation of solubility in DMF and in polystyrene. The agents (QAAF (o-carboxybenzamidomethyl QA), AQD (Al quinacridone disulfonate) and DDQA (didodecyl QA), when dry mixed at 5% with standard QA pigments and exposed to boiling DMF for 10 minutes, had varying effects on crystal growth as evidenced by x-ray line broadening ($\beta_{1/2}$) as follows:

		$\beta_{1/2}$*	
Sample	Agent	As Is	10' in boiling DMF
RT-790-D, PS-82154	None	18.5	
"	"		11.0
"	5% QAAF		15.8
"	" AQD		12.2
"	" DDQA		14.0
RT-791-D, PS-97189	None	13.4	
"	"		9.4
"	5% QAAF		12.0
"	" AQD		9.5
"	" DDQA		11.6
RT-759-D, PS-63011	None	12.5	
"	"		11.7
"	+ 5% QAAF		11.9
RT-845-D, 27602-3	6.5% QAAF	19.0	
"	"		17.3
"	+ 10% CaSx		17.0

*So-called "$\beta_{1/2}$", as used at Newark, is empirically measured peak width at half maximum intensity.

Of the three agents, the QAAF shows the most beneficial effect; the more soluble DDQA shows somewhat less effect than does QAAF; whereas the relatively insoluble AQD shows little, if any, effect. It is interesting to see that calcium Staybelite has essentially no effect when used with QAAF/QA whereas it is essential in the case of CPC-AF/CPC.

H. βQA from Heated Mill Powder 1762-34

The product obtained by heating (>150°C.) an alum mill powder of βQA (by W.J.Marshall) shows marked stability when exposed to boiling dimethylformamide (10 min.) with essentially no change in x-ray line broadening ($\beta_{1/2}$ at 5.8°). This product, which is presumably sulfonated and/or oxidized on the crystal surfaces and contains Al, is stable apparently because the "coating" is present in sufficient quantity to prevent the hot solvent from dissolving the base pigment. Analysis also showed 3% QAQ.

I. Heated Quinacridones 1790-1, 2 (12/7/64)

Marked increases in stability of QA's to boiling DMF were observed when various pigment samples were heated in air (analogous to pigments from heated mill powders by W.J.M.). For example, the following tabulation shows x-ray line broadening data ($\beta_{1/2}$) "as is" and heated in air, and each after exposure to boiling DMF for 10 minutes.

		$\beta_{1/2}$		Phase	
		Before	After	Before	After
Sample	Treatment	DMF	DMF	DMF	DMF
RT-819-D, lot 31655	"as is"	19.0	β→γ	β+α/γ	All γ!
RT-819-D, lot 31655	10 min.at 340°C.	16.0	15.5	β+slγ	β+slγ
" "	3 hrs.at 250°C.	17.0	16.0	β+slγ	β+slγ
RT-790-D, PS-82154	"as is"	18.5	11.0	γ	γ
" "	10 min.at 330°C.	16.0	15.2	γ	γ
RT-845-D (βQA + 6.5% QAAF)	"as is"	19.0	17.3	β+vsl α/γ	β+vsl α/γ
" "	10 min.at 345°C.	16.8	16.8	β	β

J. Heated CPC's 1762-27,32

As in the case of the QA's, heated CPC Blue and pigment extracted from heated CPC mill powders show markedly increased stability to solvents (e.g., 10 minutes in boiling ethyl benzene for CPC) as compared to unheated counterparts.

For example, in 1762-27D, alum dispersion-milled, β-phase CPC Blue (BGD) was heated as mill powder for 3 hours at 250°C. and then extracted in the normal way. When its crystal stability was compared with an unheated control (27B) by exposure to boiling ethyl benzene for 10 minutes, the following results were obtained by x-ray line broadening ($\beta_{1/2}$):

	$\beta_{1/2}$	
Sample	7.0°	9.1°
27B, from unheated mill powder, "as is"	14.7	15.8
" " " " " " after EtBz	11.2	11.7
27D, from heated mill powder, "as is"	14.5	15.8
" " " " " after EtBz	14.0	15.2

An α-phase chlorine-free CPC Blue RF, which showed remarkable stability to boiling ethyl benzene, was prepared by heating BT-427-D (Blue R 75%/CPC-AF 15%/Ca Staybelite 10%) 10 minutes at 330°C. A comparison of % β phase conversion and "valley parameters" ($V_{27°}$) (higher number indicates higher crystallinity) of heated Blue R (no AF or CaSx) and heated BT-427-D with their controls is as follows:

Sample	$V_{27°}$	% β
23A, Blue R (N-872-A), "as is"	47	0
" " EtBz	-	100
32AB, Blue R 10 min./320°C., "as is"	77	0
" " EtBz	-	>50
BT-427-D, PS-00123 "as is"	48	0
" " EtBz	68	<5
32EF, BT-427-D 10 min./320°C., "as is"	70	0
" " EtBz	70	<5

The above data show that crystal growth occurs during the dry heating (valley parameter increases) of both the Blue R and the BT-427-D. However, on exposure to hot solvent, the dry heated BT-427-D shows no further growth or significant conversion to β phase, whereas the heated Blue R converts to >50%β. Although the unheated BT-427-D shows no significant phase conversion in the hot solvent, it does grow considerably as the α phase.

K. X-ray Diffraction "Valley Parameter" 1762-31 (9/21/64)

During the study of the use of CPC-AF in inhibiting crystal growth of α-phase CPC Blue, there were indications of increased crystallinity of the α phase before conversion to β phase on exposure to solvents. Since α-phase CPC has doublet x-ray diffraction peaks which are not suitable for measurement of line broadening as $\beta_{1/2}$, a technique for use with doublet peaks was needed. Such a technique is described in the paper (Photostat #4215, Newark Research Library) "Transformation and Growth of CPC Crystals in Organic Suspensions" by Suito and Uyeda in Koll. Zeitz. and Zeit Fur Polymere 193, 97-111 (1963) Dec. (English) where they use such a parameter on α-CPC.

To determine the "Valley Parameter" of doublet peaks which are separated by a "valley", one measures in chart divisions the average height above the baseline of the two peaks and then divides the depth of the valley by the average height and multiplies by 100. The number decreases with decreasing crystallinity, manifesting actual line broadening of the two peaks.

This technique has been used very effectively in studies of crystallinity of α-phase CPC's including CPC-AF in BT-427-D, BT-449-D, and HT Blue B, and Blue R.

CC: W.S.Struve
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File
Extra (3)

Newark, New Jersey
March 20, 1964

MEMORANDUM

SOLUBILITY OF ORGANIC PIGMENTS IN PLASTICS

This report summarizes several simple tests for indicating solubility of colored organic pigments in plastic systems. It is hoped that this information will be of some help to those who are developing and evaluating pigments for use in plastics at high temperatures and that it may evoke some additional thoughts on this subject.

A major cause of the instability of many colored organic pigments in plastics at high temperatures is solubility of the pigment in the plastic system. In the case of pigments which are soluble, the pigment dissolves in the molten plastic as a function of temperature and time up to the limit of solubility. The overall effect of solubility should be a direct function of the pigmentation level, i.e., at very low color concentrations (e.g. less than 0.01 phr), a large percentage of the pigment would be in solution and would have a marked effect on the observed color and lightfastness, whereas, at higher pigmentation levels the percentage of pigment in solution would be less and there would be a smaller effect on the observed color and lightfastness.

Pigments which are soluble in the molten plastic can also dissolve and recrystallize from solution where the concentration is above that of a saturated solution. This would tend to give larger crystals and more stable polymorphs, resulting in weak tints and possibly different hues. Pigments which are of the most stable polymorphic form can dissolve and recrystallize as larger crystals. Ostwald ripening can also occur.

The following simple tests can be used for indicating solubility of the colored pigment in the plastic.

1. Fluorescence - Observe pigmented plastic, processed at different temperatures, under ultraviolet radiation to detect differences in fluorescence. Solutions of many colored organic pigments in organic solvents are fluorescent and the intensity increases with the concentration. However, it must be kept in mind that the unpigmented plastic may also be fluorescent, e.g., polystyrene molded after 10 minutes at 600°F. is much more fluorescent than a 400°F. control.

2. Lightfastness - Expose pigmented plastic in Fadeometer for 24 or 48 hours. Plastics containing pigment in solution will show a change proportionate to the percentage of the total pigment in solution. Where a large percentage of the pigment is in solution, the lightfastness will be poor, even in pigments which are normally excellent. In cases where there is pigment in solution, the exposed portion will not show fluorescence.

3. Spot Test - Place one drop of solvent (for the plastic and not the pigment) on the pigmented plastic and observe color change after solvent evaporates. Any pigment dissolved in the plastic will "kick out" as the plastic dissolves in the solvent. There is evidence that the pigment which kicks out is of a very small crystallite size and may be smaller than the starting pigment (especially crudes). For general purpose polystyrene, ethyl acetate is a suitable solvent. Solvents which can be used with polyethylene, polypropylene, vinyl, etc., are being sought.

4. Solution of Plastic - Dissolve plastic in a solvent which will dissolve the plastic and not the pigment. Any pigment which is in solution in the plastic will kick out. Evaporate the solvent from this solution to obtain pigmented plastic again. The resulting colored plastic will be representative of the pigment which was not in solution in the plastic plus the pigment which came out of solution and will be dependent upon the relative amounts of each. For general purpose polystyrene, ethyl acetate is a suitable solvent.

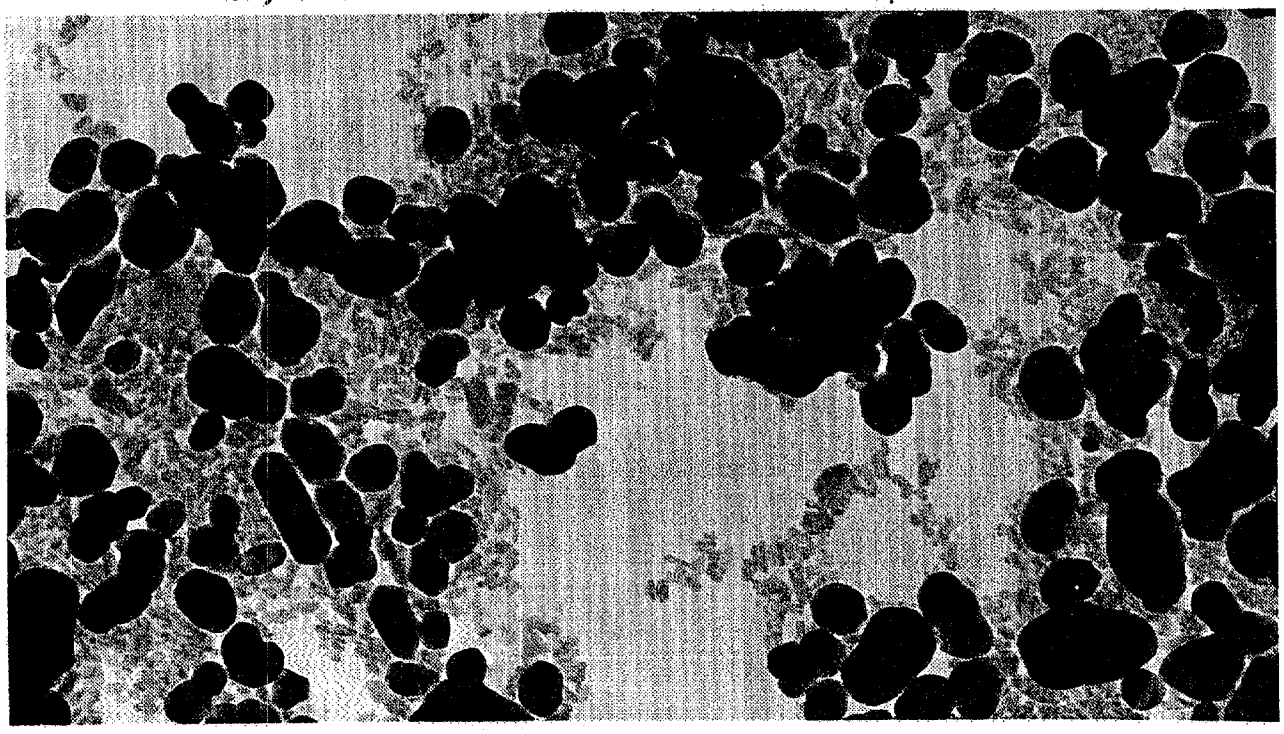
5. Vinyls at High Temperatures - Heat pigmented vinyl (e.g., for 10 minutes at 400°F.) between polished metal plates. Two hot plates top to top may be used for heating. Compare versus same vinyl sheet press polished at a lower temperature (e.g., 330°F.). Pigments which are soluble in the vinyl will intensify in color and fluorescence at the higher temperature. Unpigmented vinyl under these conditions will increase in fluorescence but will show little color change. R-101 pigmented vinyl shows only a very slight yellowing but there is more fluorescence because of the vinyl itself. The lightfastness of the pigmented vinyl exposed to the higher temperature will be worse than that at the lower temperature if solution occurs. Light absorption spectra of 0.1 phr masstones may also indicate solubility at the higher temperatures.

Figure 2

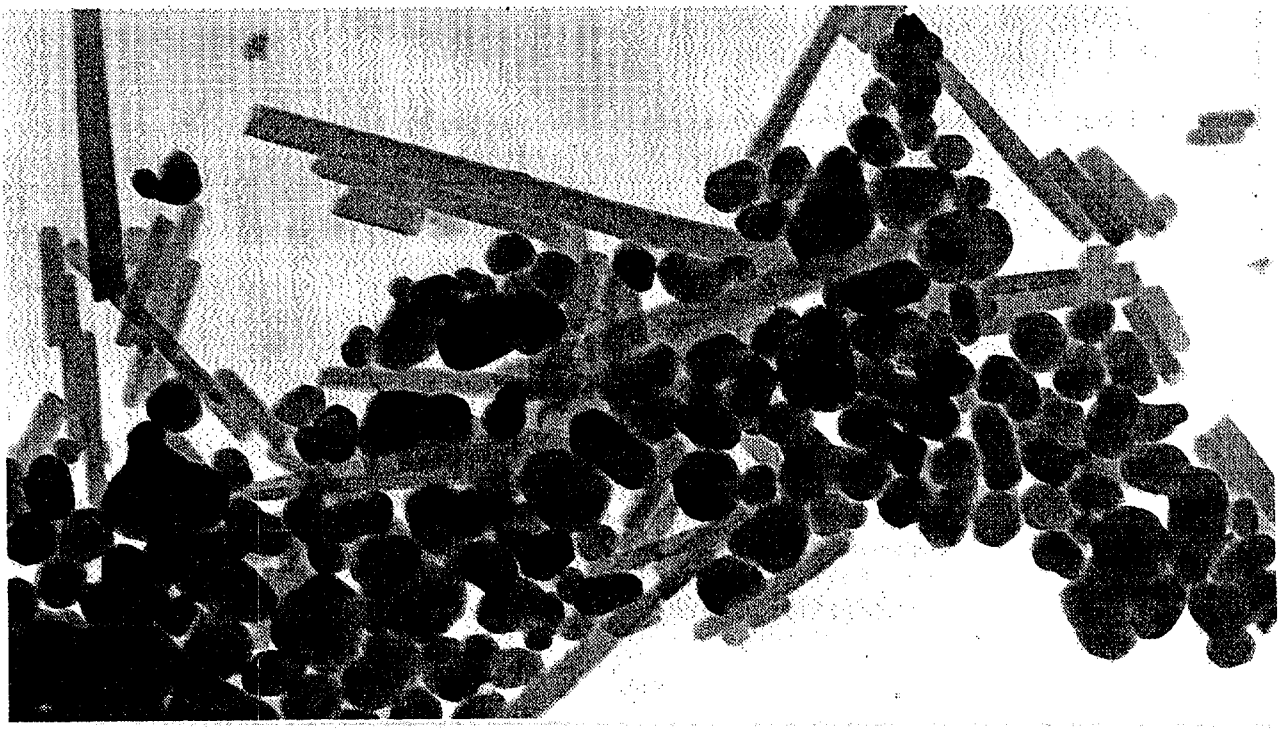
BT-297-D/TiO_2 extracted from polystyrene molds.

40,000X

1μ



Molded at 400°F.



Molded at 600°F.

References: Chestnut Run Work Request Numbers

G-63-67 "Heat Stability of Pigment Colors in General Purpose Polystyrene"
NC-63-43 "Heat Stability of Experimental Phthalocyanine Blues in Polystyrene"
NC-64-7 "Heat Stability Studies in Polystyrene"

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