Report No. R-65-22

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F. & F. Research Division

Marshall Laboratory

Research Report



# HIDING POWER OF EMULSION PAINTS

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| Date Issued; | April 9, 1965 |
| Period Covered: | 6/64 - 3/65 (Part Time) |
| Project No.: | Formerly P-1224, Now P-28101 |
| Previous Reports: | R-64-33 |
| Notebook Nos.: | 8247, 8289, 8373 |

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4/8/65

## INTRODUCTION:

Flat emulsion paints are replacing flat solution paints. Thus in 1963 F. & F. produced over 5 million gallons of "Lucite®" Wall Paint, "Lucite®" House Paint and "Flow Kote®".

However the Marketing Research Section have concluded (Ref. 22) that "Lucite" Wall Paint ranks second in frequency of complaints among all major brands both with regard to "coverage, hiding and streaking" and "lack of washability". They also conclude (Ref. 23) that customers' intent to repurchase should be higher and that "this will require better initial application properties, improved customer awareness of product benefits, as well as improved availability in outlets."

There is a large incentive therefore to develop a better understanding of those physical phenomena which affect the marketability of emulsion paints.

The writer emphasized in the last report (Ref. 8) that the hiding power perceived by the customer was especially poor for our important dripless paints partly because of their poor leveling and partly because of their large loss of hiding on drying.

The reasons for the loss of hiding have been studied by W. T. M. Johnson (Ref. 14), M. P. Morse (Ref. 15) and the writer (Ref. 8).

## OBJECTIVES:

The objective was to develop an understanding of the factors which contribute to loss of hiding power on drying and to apply the knowledge to improving the cost/property balance of emulsion finishes.

The discovery of leads to improved leveling and lap uniformity led to another objective to develop an interior semi-gloss emulsion paint. Work on the latter objective will be described in a separate report.

## SUMMARY AND CONCLUSIONS:

Loss of hiding power on drying is common to white emulsion paints except those formulated at such high pigment concentrations that there is insufficient polymer to fill all the interstices. Increased light scattering by pigment/air interfaces compared to pigment/vehicle interfaces can compensate in highly pigmented paints for the normal decrease in hiding power on drying.

Emulsion paints can lose opacity on drying due to the following factors:

(1) The refractive index of the vehicle increases when the water evaporates and this decreases the light scattering.

(2) The refractive index change leads to increased diffuse internal and external reflectance at the paint/air interface.

(3) The titanium dioxide particles are too crowded for most efficient light scattering after the film shrinks on drying from about 3 mils to 1 mil.

(4) This same shrinkage causes large extender particles and sometimes air to displace titanium dioxide from parts of the dry film. Some extender particles are thicker than the dry film thickness.

(5) The light scattering by the emulsion particles is lost when the water evaporates and particles coalesce.

The losses from these factors can be counterbalanced by a gain in hiding power due to increased light scattering from pigment/air interfaces.

This improved knowledge of the phenomena which cause loss of paint opacity on drying has provided leads for increasing dry hiding and reducing cost.

Techniques are now available for separating the contributions to hiding power from light scattering at pigment-polymer and pigment-air interfaces and from light adsorption due to tinting to off-white shades. In "Lucite" Ceiling White, tinting provides 40% of the hiding power, while light scattering from air interfaces accounts for 21% of the hiding and light scattering from polymer interfaces accounts for all the remaining 39%.

Most Trade Sales white paints are now tinted. They are too yellowish gray initially to be rated very high for whiteness. The apparent whiteness and the hiding power can be improved by lowering the green reflectance and increasing the blue reflectance.

Experimental low cost paints have been formulated using the functional extender Hi Sil with the present "Lucite" vehicle, with the "Flow-Kote" vehicle and also with the new BP (methyl methacrylate/butadiene/methacrylic acid) emulsion. These formulations have a smoother texture than "Lucite" and show a gain in hiding on drying instead of the 25% and 40% losses for "Lucite" and "Flow-Kote" respectively. The advantages in cost and hiding must be weighed against a slightly higher 85° sheen than "Lucite" and a small sacrifice in resistance to stain penetration and scrubbing.

Electronmicrographs have confirmed that the Zeolex in the Development Laboratorys test paint was poorly dispersed. Hi Sil 422 or other 0.1-0.2 micron extenders could be used to avoid excessive water and surfactant demand and the poor reproducibility of Zeolex 80.

The Development Laboratory has reduced the hiding power loss on drying of the BP emulsion paint by using diatomaceous earth instead of the ultra coarse and ultra fine extenders which were in the Zeolex "Lucite" paints.

#50 "Lucite" House Paint loses significant hiding after 5 months shelf storage. The Development Laboratory has shown that a new dispersion system and/or the elimination of the solution of phenylmercury oleate in mineral spirits gives paints which have not lost hiding power on ageing. (Ref. 27)

No technique has been found for causing a gain in hiding in gloss and semi-gloss paints. "Duco®" Satin Sheen and automotive acrylic lacquers lose 35% and 25% respectively of their hiding power on drying. Electronmicrographs of cross sections of the dried films suggest opacity and other properties could be improved if we could eliminate titanium dioxide aggregates.

The writer believes that at present the best ways to improve hiding power as judged by a consumer are to improve the flow and leveling, to eliminate $TiO_2$ aggregates, to use a 0.1-0.2 micron extender, to use low refractive index binders and to tint to new Dry Plate Standards in which the green reflectance is slightly lower and the blue reflectance slightly higher than at present. Increasing the PVC (pigment volume concentration) or decreasing the critical PVC is an excellent low cost way to increase hiding power but other properties sometimes suffer.

Another long range route to higher hiding power would be to develop high solids vehicles which dry to a low refractive index.

While trying to improve the apparent hiding power by improving the leveling, leads for gloss paints were obtained. Previously the only route to good leveling in semi-gloss emulsion paints had been the use of casein as a thickener. Another report will describe the work in semi-gloss paints.

ACTION TAKEN OR PROPOSED:

Information from this project is being communicated to the Development Laboratory and Marketing as it is developed. The difficulties which stem from excessive use of Zeolex 80 have been described. Less Zeolex 80 and less large calcium carbonate are being used in HG Super Ceiling Paint. Neither Zeolex 80 nor calcium carbonate are used in the new BP experimental "Lucite". Hi Sil 422 will be evaluated by J. J. Gallagher in "Flow-Kote". Marketing have been informed (Ref. 20) that the apparent whiteness and hiding power of our "white" paints can be improved without increasing cost by more blue and black colorant additions and less red/yellow/green colorant additions. The water clean-up trim candidate has been reviewed with Marketing who have provided a list of Essential Properties, Desired Properties, etc. (Ref. 24)

Electronmicrographs of cross sections of 868 line automotive acrylic lacquer paint films and "Corfam®" primers and basecoats have been forwarded to Flint (Ref. 17) and Newburgh (Ref. 18) to provide them with visual evidence of $TiO_2$, Monastral Red or Blue aggregation of flocculation in the dry films.

The research on semi-gloss emulsion paints will be described in another Research Report.

Emphasis will continue to be placed on developing a semi-gloss emulsion paint. Leads for improved hiding power in "Lucite" Wall Paint will also be sought. If other deficiencies are uncovered by Marketing in the Consumer Panel Test of the BP paints, these will also be investigated.

PATENT SITUATION:

Competitive Patents

None have been found.

Patent Protection

A. R. Krueger's FFD-1236 Butadiene Copolymer Latex Paints has been forwarded to Legal Department for preparation. It will cover the BP paint with its titanium dioxide/diatomaceous earth pigmentation.

A lead for improving apparent hiding power by improving levelling and lapping is incorporated into the writers' recent patent proposal (Ref. 32).

PUBLICATION PROPOSAL:

No publication is being proposed until information in this report has been exploited.

DISCUSSION:

Hiding power is one of the most important property of paints. In 1963, F. & F. used 32 million pounds of titanium dioxide for opacity. In some of our most important products, e.g. "Lucite" Wall Paint, the cost of the titanium dioxide (60 cents/gallon) represents over half the ingredient cost.

"Lucite" Wall Paint because of its driplessness and its high viscosity at low shear rates can be applied in a thick enough coat to give complete wet hiding. Unfortunately when it dries it loses about 30% of its wet hiding power. This loss is more noticeable in "Lucite" because unlike most other paints it does not level out on drying. The brushmarks, roller patterns, and lap marks all become more conspicuous because of the poor leveling and because the reflectance is so dependent on film thickness.

CAUSES OF LOSS OF HIDING ON DRYING

In an earlier report (Ref. 8) it was postulated that the loss of hiding power* on drying was due to the following five factors:

1. The $TiO_2$ scatters more light when it is surrounded by water which has a low refractive index (1.33) than when it is embedded in dry polymers (1.5 is a typical refractive index).

2. Excessive crowding of the $TiO_2$ occurs when the wet film shrinks from about 3 mils wet to 1 mil dry for "Lucite" (36% solids by volume). If $TiO_2$ particles are closer together than about 0.2 microns their light scattering efficiency is reduced. The following table from Ref. 4 shows the effect of pigment volume concentration on the distance apart of particles in a dry film.

DISTANCE APART OF PIGMENT PARTICLES
(ASSUMED TO BE UNIFORM & SPHERICAL)

| Pigment Volume Concentration | Edge to Edge Separation (as a percent of diameter) | Edge to Edge Separation for 0.3 micron particles |
|---|---|---|
| 10 | 0.949 | 0.28 microns |
| 15 | 0.703 | 0.21 " |
| 20 | 0.547 | 0.16 " |
| 25 | 0.436 | 0.13 " |
| 30 | 0.351 | 0.11 " |
| 35 | 0.284 | 0.09 " |
| 40 | 0.228 | 0.07 " |
| 45 | 0.180 | 0.05 " |
| 50 | 0.140 | 0.04 " |

*Hiding power is the spreading rate in square feet/gallon which would give a contrast ratio of 0.98 between the reflectance over a black and a white substrate. The white substrate should have a reflectance of 80%.

In most white paints we are already using so much titanium dioxide that if the particles were evenly distributed in the paint binder, they would be closer together than 0.2 microns. This means that the titanium dioxide particles are separated by less than half a wavelength of visible light, and that they are scattering light less efficiently. The decrease in light scattering efficiency with increasing titanium dioxide concentration is illustrated by figures 6, 7, 8 and 9 taken from Ref. 3. Mitton and Vejnoska[1] showed that the scattering coefficient increased linearly with $TiO_2$ pigment volume concentration up to the concentration at which the edge-to-edge separation became equal to half the wavelength of light incident on the film. Gonick[2] emphasized that maximum hiding efficiency is obtained at low pigment volume concentration and with finer particle size pigments.

In the wet film of a typical low solids (30%) emulsion paint the pigment particles are much further apart and consequently more efficient scatterers of light.

3. There is an increase in diffuse internal and external reflectance (Ref. 5) at the paint/air interfaces when an emulsion paint dries. This is due to the increase in refractive index on evaporation of water.

4. A "window" effect decreases the dry hiding power. This is due to $TiO_2$ being displaced from areas of the film by extender particles which are often thicker than the dry film thickness.

5. Loss of the light scattering by polymer emulsion particles when they coalesce also contributes to the hiding power loss.

ANALYSIS OF HIDING POWER DATA

In order to analyze hiding power results it is important to separate the hiding power into that due to light absorption and that due to light scattering.

The hiding power can be calculated in several ways from measurements of reflectance. The old way of plotting contrast ratio versus spreading rate is illustrated in Figure 4. This does not allow the hiding power to be calculated for various reflectivity or brightness levels. It does not correct for the fact that most paper or glass backgrounds do not have an 80% reflectance. A better way is to use Computer Program ML-1015 which corrects for reflectance of the uncoated paper or glass, determines the best straight line through the experimental points, and gives the hiding power. An even better way is to use ML-1005 which calculates the Kubelka-Munk Scattering and Absorption Coefficients and therefore enables a formulator to calculate what the hiding power would be after toning to different reflectance levels.

The agreement between these methods is illustrated by the following data on variations of #50 "Lucite" made with 4 different emulsions.

#50 "LUCITE" FROM VARIOUS EMULSIONS
HIDING POWER CALCULATED BY IBM 1620
(ML-1005 & ML-1015 PROGRAMS)

| EMULSION | COMPUTER PROGRAM | HIDING POWER SQ. FT./GAL. WET | DRY | PERCENT LOSS |
|---|---|---|---|---|
| Rhoplex AC-34 | ML-1005 | 517 | 346 | 33 |
| | ML-1015 | 519 | 355 | 32 |
| Rhoplex AC-33 | ML-1005 | 514 | 330 | 36 |
| | ML-1015 | 516 | 350 | 32 |
| RL-11005 (IO) | ML-1005 | 524 | 330 | 37 |
| | ML-1015 | 498 | 342 | 31 |
| RC-W-13384 (HG) | ML-1005 | 519 | 335 | 35 |
| | ML-1015 | 513 | 351 | 32 |
| Confidence Limit (mean of 2; ML-1005) | | ± 16 | ± 16 | |
| Least Significant Difference | | 22 | 22 | |

An even better program ML-1020 is being written by L. Lenhart. This will compute the mean hiding power, the standard deviation of the mean and also show what the hiding power would be at various reflectivity levels.

EFFECT OF TINTING TO OFF-WHITES

Tinting from a reflectivity of 95% to 81% increases hiding power by 61%. This is calculated as follows. At a reflectivity ($R_{oo}$ or G value) of 81% the hiding power equals the scattering coefficient (S) multiplied by the "Factor A" (Ref. 6, 7) for a reflectivity of 81%. The ratio of "Factor A" for G = 81% to "Factor A" for G = 95% is 239/148 = 161/100. In most cases tinting produces little change in the scattering coefficient so that to a good approximation the hiding power for off-whites can be estimated from S and the "Factor A" for the measured reflectivity.

There seems to be a large incentive for improving the hiding power of many of our white paints by tinting to an off-white with lower green reflectance. The writer has recommended (Ref. 20) that consideration be given by Marketing to rebalancing our whites so that the blue reflectance will be slightly greater than the green reflectance. This can be done by using slightly more blue and black colorants and less red, yellow and green when tinting our "whites". This would increase the apparent whiteness and the hiding power.

Most of our white paints in the following table score poorly for whiteness on the Hunter or Taube formula (Ref. 28)* which takes into consideration the finding that the average person weights yellowness as 4 times as undesirable as grayness.

i.e. W = green reflectance - 4 times
( " " - blue reflectance)

= G - 4 (G-B)
= 4B-3G

APPARENT WHITENESS

| | |
|---|---|
| Magnesium Oxide White | 100 |
| "Flow-Kote" White | 62 |
| "Lucite" Wall White (Step 2 in Release Letter R-1863-T) | 67 |
| "Lucite" Ceiling White | 78 |
| Superwhite | 91 |

In Table I of Ref. 30 "Flow-Kote" was the worst for reflectivity of 8 Flat Wall Paints. This deficiency is made more obvious to the consumer by "Flow-kote's" yellowishness both initially and especially on ageing.

ILLUSTRATION OF HOW THE HIDING POWER OF A WHITE CAN BE IMPROVED WITHOUT SACRIFICING THE APPARENT WHITENESS

| GREEN REFLECTANCE G | BLUE REFLECTANCE B | APPARENT WHITENESS | RELATIVE HIDING POWER |
|---|---|---|---|
| 95 | 91 | 80 | 100 |
| 90 | 87.5 | 80 | 118 |
| 85 | 84 | 80 | 141 |
| 80 | 80 | 80 | 161 |
| 75 | 76 | 80 | 194 |

ILLUSTRATION OF HOW THE HIDING POWER OF "LUCITE" WALL WHITE WAS IMPROVED (RL-1863-T) WITH A SACRIFICE IN APPARENT WHITENESS

| CODE | G | B | APPARENT WHITENESS | RELATIVE HIDING POWER |
|---|---|---|---|---|
| 392-101 (1/1/64) | 90.27 | 86.25 | 74 | 100 |
| " Step 1 | 88.62 | 84.08 | 70 | 106 |
| " Step 2 | 87.00 | 81.91 | 67 | 112 |

*Better formulas are available (Ref. 31) but they must be kept confidential until Eastman Kodak have presented a paper at the next Tappi Testing Conference.

## LEADS FOR ELIMINATING LOSS OF HIDING ON DRYING

If our understanding of the factors which affect loss of hiding on drying is valid, we should reduce hiding loss in "Lucite" by:

1. Replacing the excessively coarse calcium carbonate (W-90) with a somewhat finer grade (W-1004).

2. Reducing $TiO_2$ concentration from the present 2.2 lbs./gal. (Only 1.55 lbs./gal. are used in the Hi Sil formulations).

3. Adding an extender (Hi Sil 422) having the appropriate size for spacing $TiO_2$ particles about half a wavelength apart.

Each of the above changes when made singly in "Lucite" Ceiling White reduced the loss of hiding. The loss of hiding was completely eliminated when all three changes were made in either the 392-029 quality (Rhoplex AC-33/Styrene-butadiene/Piccopale) or the BP quality (methyl methacrylate/butadiene/methacrylic acid = 65/33/2). Improved dry hiding is also evident in the following data:

| CODE | DRY HIDING POWER (SQ. FT./GAL.) WHEN REFLECTANCE = 81% | CHANGE ON DRYING |
|---|---|---|
| 392-029 "Lucite" Ceiling White | 2.35 x 239 = 562 | 29% loss |
| 8247-181 Hi Sil "Lucite" | 2.55 x 239 = 610 | 9% gain |
| 8116-140 Hi Sil BP | 2.52 x 239 = 602 | 6% gain |
| 392-B-39053 BP "Lucite" Semi-works batch used in panel tests | 2.43 x 239 = 580 | 10% loss |

The Hi Sil "Lucite" formulation was 20 cents lower in ingredient cost than the present commercial product 392-029. Possible deficiencies of this formulation are a slightly higher sheen (3 instead of 1.5 @ 85°) and reduced stain removability and scrubbability. The sheen can be reduced without causing much loss of hiding by adding some W-90 in place of W-1004.

## ZEOLEX 80 FORMULATION

By the time the Hi Sil 422 lead was developed the Development Laboratory was unfortunately committed to the Zeolex 80 approach (Ref. 25). Zeolex causes several difficulties when used in sufficient quantity to eliminate loss of hiding power on drying. These were pointed out. For example on pages 15 and 16 of Research Report R-64-33, the writer suggested that "special precautions will be necessary with Zeolex paints to ensure reproducibility and paint stability."

The semi-works batches later were found to vary considerably in resistance to synaresis and in smoothness at the lap. On page 16 attention was drawn to the "sacrifice in brushing, lap appearance, cracking resistance, scrub resistance, porosity and resistance to staining" in the Zeolex formulation. In a subsequent Consumer Panel Test consumers detected some of these deficiencies. (Ref. 9). Consumers detected no improvement in hiding power versus a control that had been panel tested in previous years.

The poor dispersion of the ultra fine Zeolex 80 in the Consumer Panel Test paint (392-W-99397) is visible in Figure III. This is an electronmicrograph taken by Dr. D. Jordan of Pigments Department of a one eighth micron cross section of the dried paint magnified 3500 times. Zeolex 80 could improve dry flat hiding even if it was not completely dispersed but such a system would tend to have poor reproducibility, stability, etc.

No difficulties have been experienced with any emulsion in eliminating the loss of hiding on drying via the Hi Sil 422 approach. Negligible hiding loss or even hiding gains have been obtained with the present mixed "Lucite" vehicle, with the new BP emulsion, with the styrene-butadiene/Piccopale combination used in "Flow Kote" and with straight acrylics.

Hi Sil 422 has also been tested in other modifications of "Lucite" in which the usual 2.2 lbs. of Ti-Pure R-505 are replaced by 1.5-1.6 lbs. of other $TiO_2$'s and W-90 is replaced by W-1004. The following table illustrates the negligible hiding power loss and the high scattering efficiency of the $TiO_2$ in these formulations compared to the "Lucite" control 392-029. Ti Pure R-901-C gave highest scattering efficiency and should be lower in mill cost to Pigments Department than R-505.

EFFECT OF VARIOUS $TiO_2$'S IN HI SIL FORMULATION

| CODE | HI SIL | HIDING POWER LOSS ON DRYING | SCATTERING COEFFICIENT | SCATTERING EFFICIENCY* | $TiO_2$ TYPE |
|---|---|---|---|---|---|
| 8116-5B | Yes | None (13% gain) | 2.33 | 1.50 | Raw R-901C** from Cyclone Discharge |
| 8116-45A | " | " (4% gain) | 2.57 | 1.61 | Filter cake** from R-901C process. |
| 8116-45B | " | " (Dry=wet) | 2.68 | 1.74 | Lab batch of micronized RC-901C** |
| 8249-181 | " | " (9% gain) | 2.55 | 1.65 | R-505(W-56) |
| Control 392-029 | No | 29% loss | 2.35 | 1.07 | R-505(W-56) |

*Scattering Efficiency is the Scattering Coefficient per unit concentration of $TiO_2$($mils^{-1}$ divided by lbs. $TiO_2$ per gallon)

**These samples were kindly provided by J. Bishop (Pigments Dept., New Johnsonville)

## VARIOUS $TiO_2$'S IN "LUCITE" CEILING WHITE

The following table shows that in the present 392-029 formulation the $TiO_2$ which is now being used is superior to the other types of $TiO_2$ tested*. These included two chloride process $TiO_2$'s (one was an unprocessed, uncalcined sample from the cyclone discharge) and an experimental glass encased $TiO_2$. The later eliminated the loss of hiding power. However, not enough $TiO_2$ could be put into the paint for practical hiding because of the low concentration of $TiO_2$ in the glass, the low volume solids of 392-029 and the relatively low pigment volume concentration (50%). Dr. Willcox of Pigments Department is correcting this deficiency by increasing the $TiO_2$ level and by classifying the glass-encased-$TiO_2$ into fractions which pack more densely and can be used at higher PVC's.

Our results on combinations of $TiO_2$ and glass encased $TiO_2$ are available in Ref. 26. Further work is planned. Dr. Willcox's latest approach gives higher hiding power at very high pigment volume concentrations (70%) of mixtures of well packed glass-encased-$TiO_2$ and conventional $TiO_2$. The cost of a gallon of this type of paint will be high.

### VARIOUS $TiO_2$'S IN 392-029

| | SULFATE W-56 CONTROL | CHLORIDE W-123 | RAW CHLORIDE CYCLONE DIS. | GLASS ENCASED |
|---|---|---|---|---|
| Measured | | | | |
| Hiding Power | | | | |
| Wet | 473 | 478 | 511 | 160 |
| Dry | 334 | 328 | 345 | 161 |
| Oiled | 280 | 278 | 304 | 146 |
| % Loss | 29 | 31 | 32 | 0 |
| Hiding Power | | | | |
| Dry ($R_{\infty}$ =81%) | 561 | 510 | 405 | 224 |
| Scattering Coefficient | | | | |
| S (Dry) mils$^{-1}$ | 2.35 | 2.13 | 1.70 | .94 |
| Lbs. $TiO_2$/Gallon | 2.2 | 2.12 | 2.09 | 0.78 |
| $TiO_2$ Efficiency (=S/lbs. $TiO_2$) | 1.07 | 1.0 | 0.81 | 1.2 |

*Ti-Pure R-901-C was not tested. In another table in a different formulation it was superior to Ti-Pure R-505 (W-56).

"FLOW KOTE"

The following data show the effect in "Flow Kote" (389-231) of increasing the pigment volume concentration from the normal 45% to 65%. The $TiO_2$/gallon and the ratio of $TiO_2$/clay/diatomaceous earth were kept constant by adjusting the solids with water and methylcellulose.

EFFECT OF INCREASING THE PVC OF "FLOW KOTE"

| PIGMENT VOLUME CONCENTRATION | LOSS OF HIDING POWER ON DRYING | DRY HIDING POWER IF TINTED TO $R_\infty = 81\%$ | $TiO_2$ EFFICIENCY TOTAL | $TiO_2$ EFFICIENCY EXCLUDING AIR INTERFACES | PERCENT HIDING POWER DUE TO - AIR INTERFACES | POLYMER INTERFACES | ABSORPTION OF LIGHT |
|---|---|---|---|---|---|---|---|
| 45 | 43% | 425 | 0.87 | 0.72 | 11 | 57 | 32 |
| 50 | 37% | 471 | 0.96 | 0.66 | 20 | 43 | 37 |
| 55 | 31% | 542 | 1.11 | 0.66 | 25 | 37 | 38 |
| 60 | 21% | 596 | 1.22 | 0.63 | 30 | 33 | 37 |

The above data illustrate that increasing the PVC of this pigmentation will reduce the hiding loss on drying but the ratio of light scattered by air interfaces/polymer interfaces becomes quite high. Stain removability would therefore be poor.

A better approach for pigmenting the "Flow Kote" vehicle (styrene-butadiene + Piccopale) is to use a modification of the "Lucite" Ceiling White pigmentation in which Hi Sill 422 and a medium size $CaCO_3$ (W-1004) replace the very large $CaCO_3$ (W-90) and some of the $TiO_2$. As shown in the following table this results in a negligible (4%) loss of hiding on drying, higher $TiO_2$ efficiency, and lower cost without an excessive amount of scattering due to air interfaces.

| | LBS. $TiO_2$ PER GAL. | PERCENT LOSS OF HIDING POWER ON DRYING | DRY HIDING POWER IF TINTED TO $R_\infty = 81\%$ | $TiO_2$ EFFICIENCY TOTAL |
|---|---|---|---|---|
| "Lucite" Ceiling White | 2.2 | 29 | 562 sq. ft. | 1.07 |
| Hi Sil "Lucite" | 1.54 | 9(Gain) | 610 | 1.65 |
| Sears Ceiling White | 3.37 | 22 | 985 | 1.22 |
| | | | 581($R_\infty = 93$) | 1.22 |
| "Flow Kote" 45% PVC | 2.05 | 43 | 425 | 0.87 |
| Expt."Flow Kote" with Hi Sil "Lucite" Pigmentation (45% PVC) | 1.58 | 4 | 514 | 1.36 |
| BP(392-B-39053) | 1.9 | 10 | 580 | 1.28 |
| Hi Sil BP | 1.6 | 6(Gain) | 602 | 1.57 |
| Satin Sheen 60-160 (Alkyd Solution) | 2.88 | 35 | 496 | 0.72 |

BP PAINT

Also included in the above table are data for the BP Paint (392-B-39053) which was panel tested recently. It is based on a pigmentation developed by A. R. Krueger consisting of W-43/W-1011 (Ti Pure R-510/Celite 365 = diatomaceous earth). The advantage of this pigment/vehicle combination is its good stain removability, spachtle cracking resistance, smoother texture and lower cost.

It is instructive to calculate the packing indices for blends of W-43/W-1011. At weight ratios of 49/51, 57/43 and 64/36 the packing indices are 0.48, 0.58 and 0.74 respectively (Ref. 11). The more W-1011 the lower the packing index. At the 57/43 ratio selected for the BP paint the packing index is 0.58. This means that 58 volumes of the mixture of W-43/W-1011 require 100 volumes of liquid for dynamic packing.

The "dynamic critical PVC" is therefore 58/158 or only 37%. This is lower than the "dynamic critical PVC" for the "Flow Kote" pigmentation (55%) and for "Lucite" Ceiling White pigmentation (55%).

The dynamic critical PVC's are calculated on the Bendix computer. They provide a good estimate of the interstitial volume in the base under dynamic conditions and a very rough estimate of the space between the pigments to be filled with polymer and air.

EFFECT OF LEVELING AND LAPPING DEFICIENCIES ON HIDING POWER

In an earlier report the writer recommended that "ultra fine extenders like Zeolex and ultra-coarse extenders like W-90 calcium carbonate should be avoided whenever possible in our longer range work" (Ref. 8). However a paint containing a relatively large concentration of Zeolex 80 had already been developed by the Development Laboratory. A consumer panel test showed that consumers could detect the deficiencies of this low cost Zeolex paint (Ref. 9). On page 16 of the marketing research section report it is stated that the only "significantly different disliked feature" of the Zeolex paint was that it dried unevenly and did not leave a smooth surface. This leveling and lapping defect probably made it difficult for consumers to see the small hiding power advantage.

It is worth repeating that "dripless" emulsion paints which have poor leveling and lapping show poorer apparent hiding power when applied with a brush or roller than when applied with a doctor blade. Laboratory hiding power measurements which are made on very uniform drawdowns do not correct for the thick areas of paint which the painter cannot avoid when ease of spreading or leveling are poor. The leveling is so poor in "Lucite" Ceiling White (392-029) that the average film thickness in the valleys (as measured with the slit microscope for a film brushed on glass) was only 59 ± 5 percent of the thickness on the ridges. The following table suggests how this film thickness variation affects the contrast ratio.

| | Film Thickness | Spreading Rate | Contrast Ratio |
|---|---|---|---|
| Valleys | 1.03 ± 0.25 | 560 sq. ft./gal | 0.99(wet) 0.97(dry) |
| Ridges | 1.73 ± 0.34 | 334 " " " | 0.99(wet) 0.99(dry) |

## EFFECT OF EXCESSIVE CROWDING OF $TiO_2$ IN SEMI GLOSS PAINTS

In the following table the loss of hiding power on drying increases from 23% to 54% as the PVC of $TiO_2$ (W-123) is increased from 10 to 35%. The scattering efficiency which equals the scattering coefficient divided by the lbs. of $TiO_2$/gallon decreases from 1.39 mils$^{-1}$ at 10% PVC to 0.57 mils$^{-1}$ at 35%. This is similar to that observed by Gonick and coworkers (Ref. 2 & 3). The decrease is due to the excessive crowding of $TiO_2$.

EFFECT OF PVC IN SEMI GLOSS EMULSION PAINTS

| VEHICLE | | PERCENT PVC | HIDING POWER % LOSS | SCATTERING EFFICIENCY |
|---|---|---|---|---|
| RC-11103 and | H-654 | 10 $TiO_2$ | 23 | 1.39 |
| " | " | 20 " | 48 | 1.02 |
| " | " | 25 " | 49 | 0.96 |
| " | " | 30 " | 55 | 0.75 |
| " | " | 35 " | 54 | 0.57 |

The data on the 20% PVC (All $TiO_2$) paints in the following table show that the scattering efficiency in the dry film is higher for the aqueous systems than for the automotive solution paints. The aqueous systems show a larger loss of hiding on drying partly because the lower refractive index of the water gives very high wet hiding power.

SCATTERING EFFICIENCY AND LOSS OF HIDING ON DRYING (EMULSION VS. SOLUTION)

| | 20% PVC $TiO_2$ | | |
|---|---|---|---|
| | $TiO_2$ | % LOSS | SCATTERING EFFICIENCY |
| RC-11103/H-654 acrylic emulsion | W-123 | 48 | 1.02 |
| RC-11005 (IO) acrylic emulsion | W-123 | 43 | 0.90 |
| Itaconic Acid water soluble Copolymer in water/isopropyl alcohol | W-123 | 29 | 1.14 |
| RC-909/CAB/H-654 solution acrylic | W-124 | 22 | 0.78 |

In the next table the effect of varying the type of $TiO_2$ in 27% pigment volume concentration acrylic emulsion paints is shown. Ti Pure R-900 and Ti Pure R-902 gave highest light scattering efficiency, highest gloss and highest loss of hiding power on drying. The finer Ti Pure 990 and the SW-467 grades ($TiO_2$ coated with 15% silica for improved resistance to chalking) were poorer in hiding power.

EFFECT OF TYPE OF $TiO_2$ IN SEMI GLOSS EMULSION PAINTS

| $TiO_2$ Type | Kubelka Munk Scattering Coefficient(s) | Percent Loss of Hiding Power on Drying | Light Scattering Efficiency | 60° Gloss |
|---|---|---|---|---|
| Ti Pure R-900 | 2.38 $mils^{-1}$ | 51 | 0.92 | 66 |
| " R-990 (Extra fine grade) | 2.17 | 52 | 0.84 | 29 |
| SW-467 | 2.31 | 36 | 0.89 | 20 |
| SW-467 Calcined | 2.21 | 32 | 0.85 | 19 |
| Ti Pure R-909-(J666) | 2.31 | 48 | 0.89 | 42 |
| Ti Pure R-902-(4915) | 2.34 | 46 | 0.90 | 60 |

(The emulsion in the above paints was an iminated IO except MMA/2EHA/MAA - 45/51/4. All paints contained 2.6 lbs. "$TiO_2$"/gallon.)

EFFECT OF EXTENDER IN SEMI GLOSS EMULSION PAINTS

Replacing one third of the $TiO_2$ by an equal volume of a fine extender such as Hi Sil 422 or W-1008 reduces the hiding power loss and increases the scattering efficiency as shown below.

| TYPE OF EXTENDER | PIGMENT VOLUME CONCENTRATION $TiO_2$ | PIGMENT VOLUME CONCENTRATION EXTENDER | PERCENT LOSS OF HIDING POWER | SCATTERING EFFICIENCY |
|---|---|---|---|---|
| None | 30 | 0 | 55 | 0.75 |
| Hi Sil 422 | 20 | 10 | 38 | 1.15 |
| W-1008 | 20 | 10 | 50 | 1.05 |
| None | 20 | 0 | 48 | 1.02 |

However replacing one third of the $TiO_2$ by an equal volume of emulsion solids (i.e. reducing PVC from 30 to 20%) also improved scattering efficiency.

Lithopone costs about one third as much as $TiO_2$ and gives about one third as much hiding power. $TiO_2$/lithopone mixtures give slightly less hiding power loss on drying and in some gloss emulsions the lithopone reduced the wet crocking.

## LOSS OF HIDING OF "LUCITE" HOUSE PAINTS ON AGEING

The following data shows that lab batches of #50 "Lucite" House Paint lose hiding on drying and on ageing (5 months at room temperature) whether they are based on Rhoplex AC-34, Rhoplex AC-33, Polymer IO (RC-11005), or Polymer HG (RC-W-13384). The probability limits of this conclusion are above 99% according to an Analysis of Variance conducted by Mr. H. Hirschhorn.

### THE EFFECT OF ROOM TEMPERATURE AGEING ON THE WET AND DRY HIDING POWER OF #50 "LUCITE" MODIFICATIONS

| | HIDING POWER (sq. ft./gal) | | | | SCATTERING COEFFICIENT (mils$^{-1}$) | | | |
|---|---|---|---|---|---|---|---|---|
| | Initial | | Aged 5 Months | | Initial | | Aged 5 Months | |
| | Wet | Dry | Wet | Dry | Wet | Dry | Wet | Dry |
| AC-34 | 517 | 346 | 347 | 252 | 3.38 | 2.07 | 2.12 | 1.48 |
| AC-33 | 514 | 330 | 408 | 292 | 3.30 | 2.00 | 2.58 | 1.69 |
| IO | 524 | 330 | 429 | 295 | 3.30 | 1.95 | 2.68 | 1.72 |
| HG | 519 | 335 | 408 | 278 | 3.58 | 2.02 | 2.74 | 1.62 |
| Confidence Limit (95%) | ± 16 | ± 16 | ± 22 | ± 17 | ±0.07 | ±0.06 | ±0.16 | ±0.05 |
| Least Significant Difference | 22 | 22 | 32 | 23 | 0.11 | 0.08 | 0.22 | 0.07 |

For several years an experimental modification of #50 "Lucite" House Paint has been on exposure. In this formulation Tamol 731 dispersant, methylcellulose thickener and phenylmercury oleate mildewicide (in mineral spirits) are replaced by Tamol 850, hydroxyethylcellulose and dry phenylmercury propionate. Dr. D. R. Strehlau has shown that this formulation does not lose hiding power on ageing. The Development Laboratory may propose release of this new formulation in IO and/or AC-34 qualities if the spring house test inspection reveals no sacrifice in cleanliness retention or durability.

## $TiO_2$ EFFICIENCY

The following statement (Ref. 10) was made by J. S. Long, President of Paint Research Institute.

"At the PRI workshop session at the 41st Annual Meeting, Drs. Bobalek and Zettlemoyer gave talks on their work.

In both cases they brought out a very vital point; namely, that electronmicroscope pictures and other evidence showed rather clearly that, in even the best dispersion (grind) we get, we do not disperse down to the individual pigment particles or even close to this, but in most cases the smallest particles we get are at least quadruple; that is, four particles of $TiO_2$ clustered together. There are some single particles so we know it can be done but a high percentage of the final particles we achieve have four $TiO_2$ particles or in some cases many more than this. It would be, perhaps, a fair statement that at no time do we achieve 25% efficiency in dispersion of pigments. This is our bread and butter. This is what we do more than anything else. And yet it looks as though we are putting into the can something like two times as much pigment as is needed to get the same hiding power had we succeeded in dispersing pigment particles perfectly. In other words, these papers pointed out something that you ought to know but didn't. Chances are that it would have been years before you did find out if it were not for the work of these two professors under the Paint Research Institute. This kind of work is not normally done in individual company laboratories. I think most formulators would feel very comfortable if the Hegman reading was between 7 and 8 and would not realize that if proper wetting agents, formulations, and dispersion techniques were carried out, the total quantity of pigment per gallon could well be from 1/3 to 1/2 of what we now use. This now presents great opportunities and indicates that considerable research is needed at high level to find ways and means of getting the high degree of dispersion that we thought we had."

The above quotation is an exaggeration according to several Pigments Department researchers. 30-40% is probably a good estimate of the hiding power which is being lost due to $TiO_2$ aggregates which we are unable to break down into individual crystals. Dr. E. D. Lewis (Pigments Department) informed the writer that his group have increased hiding power by 20% by fractionating out $TiO_2$ aggregates.

## ELECTRONMICROGRAPHS OF PAINT FILMS

Some excellent transmission electronmicrographs have been taken by Dr. Don Jordan (Pigments Department) of one eighth micron cross sections of paint films. Figures 1 and 2 show that a large fraction of the $TiO_2$ is present as aggregates or flocculates in the dried film for both acrylic emulsion and acrylic solution (automotive) paints.

It is interesting that the $TiO_2$ efficiency (scattering coefficient divided by the pounds of $TiO_2$/gal) is higher for the emulsion paint than for the solution paint.* Since the refractive indexes are quite similar for the dry vehicles, the difference may be due to the better dispersion of $TiO_2$ in aqueous surfactants.

*See table on Page 14.

These electronmicrographs of $TiO_2$ in dried paint films show much less clumping of $TiO_2$ than electronmicrographs of $TiO_2$ obtained by diluting wet samples. It is extremely difficult to dilute and collect $TiO_2$ without causing clumping.

Cross sections of the emulsion primer and basecoat sprayed on "Corfam®" also show $TiO_2$ aggregates and Monastral® Red and Blue flocculation (Ref. 12). Some of the "Corfam" finishing difficulties may be due to this flocculation* and to the silicone antifoam agent in Nopco NDW (Ref. 13), used in 548-938, the white base for the "Corfam" primers and basecoats.

REFRACTIVE INDEX CHANGES

The hiding power increases about 3.5% for a decrease of 0.01 in the refractive index of the binder (Ref. 3). At good hiding power levels it may require about 10% more $TiO_2$ to raise the hiding power by 3.5%. Some paints can actually decrease in hiding power when the $TiO_2$ level is increased (See Fig. 9). Therefore changing to a low refractive index binder is often better than increasing $TiO_2$ level.

Unfortunately there are no low cost, coalescible emulsion polymers with refractive indices as low as water or fluorinated polymers (Ref. 16). Ethylene copolymers are relatively high.

The refractive index of a polymer can be predicted from the equation (Ref. 21):

$$(R.I.)^2 = \frac{1 + 2\frac{A}{m}d}{1 - \frac{A}{m}d}$$

where d = density, A = molar refractivity of the monomer units

m = molecular weight of the monomer.

EXPERIMENTAL:

The wet and dry hiding power were measured by the method described on page 20 of Ref. 8.

Estimate of Scattering Due to Air Interfaces

Techniques are now available for estimating how much of the scattering is due to pigment/polymer interfaces as distinct from air interfaces. This is important for under-

*The amount of flocculation will vary depending on dryness of spray and shear. The very low viscosity makes $TiO_2$ settling and flocculation of colors difficult to prevent.

standing the mechanism by which ultra fine (Zeolex 80), fine (Hi-Sil 422), or poor packing porous extenders (Celite) operate and for helping to predict the susceptibility to staining, stain removability, scrub resistance and cracking.

The scattering coefficients are calculated by the IBM-1620 computer using the ML-1005 program (Ref. 29). The measurements required are the weight of paint, the area covered, the reflectances of the white substrate before and after coating and the reflectance of the black substrate after coating. The reflectances are measured for the wet coating and for the dry coating before and after oiling with Nujol.

On drying white paints usually show a decrease in scattering coefficient and in reflectivity. If it were not for the decrease in reflectivity the loss of hiding power would be even greater on drying.

This is illustrated by the following data on "Flow Kote":

CHANGES IN UNTINTED "FLOW KOTE" ON DRYING AND OILING

| | Wet | Dry | Oiled | % Loss on Drying | % Loss on Oiling |
|---|---|---|---|---|---|
| Hiding Power | 511 sq. ft./gal. | 290 sq. ft./gal. | 278 sq. ft./gal. | 43 | 4 |
| Scattering Coefficient | 3.40 $mils^{-1}$ | 1.78 $mils^{-1}$ | 1.48 $mils^{-1}$ | 48 | 17** |
| Reflectivity | 94.5% | 92.1% | 88% | 2.5 | 4* |

*This loss <u>increases</u> hiding power

**This loss <u>decreases</u> hiding power

/jp

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## APPENDIX I

## SUGGESTED PROCEDURE FOR ANALYZING HIDING POWER DATA

The following calculations illustrate the procedure used for estimating the contributions to hiding power from the light scattering due to pigment/polymer interfaces and air interfaces and from light absorption due to tinting to an off-white shade. Computer program ML-1005 gives the following data for a laboratory batch (8247-8C) of untinted "Lucite" Ceiling White*or Wall White*.

| Untinted Hiding Power (sq.ft./gal.) | | | Scattering Coefficient(s) mils$^{-1}$ ** | | | $R_\infty$ |
|---|---|---|---|---|---|---|
| Wet | Dry | Oiled | Wet | Dry | Oiled | Dry |
| 473 | 334 | 280 | 3.25 | 2.35 | 1.53 | 96.6% |

To obtain the dry hiding power of this paint after it has been tinted to $R_\infty$ = 81% one multiplies the scattering coefficient for the dry film by the Factor A (Ref. 7) for $R_\infty$ = 81% i.e. Hiding Power (for $R_\infty$ = 81) = S times Factor A = 2.35 x 239 = 562 sq. ft./gal. The increase in hiding power due to the increased light absorption by the traces of tinting colors = 562 - 334 = 228 or 68%. The hiding power due to light scattering by polymer/pigment interfaces = S (oiled) times Factor A = 1.53 x 142.4 = 218. The hiding power due to light scattering by air interfaces = 334 - 218 = 116. Therefore the ratio is 228/218/116, or 40% of the total hiding power is due to tinting, 39% is due to scattering by polymer/pigment interfaces and 21% to scattering by air interfaces.

The total dry scattering power efficiency of $TiO_2$ can be compared in formulations by dividing the scattering coefficient (mils$^{-1}$) by the concentration of $TiO_2$ (lbs./gallon).

*These products are now the same except they are tinted to different dry plate standards.

**The units are mils$^{-1}$ of wet film thickness.

## GLOSSARY

DEFINITIONS:

Contrast Ratio, C. - Ratio of the reflectance of a paint film over a black substrate of 5 per cent or less reflectance, to the reflectance of the same paint, equivalently applied and dried, over a substrate of 80 per cent reflectance.

Hiding Power. - Number of square feet over which a gallon of paint can be uniformly spread to produce the specified contrast ratio, $C = 0.98$.

Reflectivity, $R_{\infty}$. - The reflectance of a film so thick that a further increase in thickness does not change the reflectance.

The Kubelka-Munk theory relates the reflectance, R, of a paint film at any thickness, by means of two coefficients: a scattering coefficient S, and an absorption coefficient, K. The absorption coefficient is defined as the limiting value of the rate of decrease of transmittance per unit colorant layer thickness as the thickness approaches zero.

$$K = (dT_i/dX)_{X \to 0}$$

The scattering coefficient is defined as the limiting value of the rate of increase of reflectance per unit thickness as the thickness approaches zero, the layer being backed by an ideal black backing.

$$S = (dR_o/dX)_{X \to 0}$$

The scattering is due primarily to the pigment in the paint whereas the absorption is due to both the pigment and the vehicle. When a paint film is so thick that a further increase in thickness does not change its reflectance, then this reflectance is known as its reflectivity, $R_{\infty}$. For such a thick film, S, K, and $R_{\infty}$ are related by

$$K/S = (1-R_{\infty})^2/2R_{\infty}$$

RC-13384 is HG emulsion = methyl methacrylate/2-ethylhexyl acrylate/methacrylic acid = 44/54/2.

RC-11005 is IO emulsion = iminated methyl methacrylate/2-ethylhexyl acrylate/methacrylic acid = 45/53/2.

RC-11103 is a 2 stage emulsion = methyl methacrylate/ethyl acrylate/methacrylic acid = 39/57/4.

BP Emulsion = methyl methacrylate/butadiene/methacrylic acid = 65/33/7.

RC-909 = methyl methacrylate solution resin.

CAB = celluloseacetate butyrate

Itaconic acid "water soluble" copolymer RU-5031 = low molecular weight terpolymer of methyl methacrylate/ethyl acrylate/ itaconic acid (20/70/10) in isopropyl alcohol.

Zeolex 80 is sodium silicoaluminate (0.022 microns)

Hi Sil 422 is a "precipitated hydrated silica" pigment containing 80.2% $SiO_2$, 6.3% CaO (0.12 microns)

H-654 benzylbutyl phthalate.

PVC = pigment volume concentration = volume of pigment as a percent of total solids volume.

## ABSTRACT

Leads are presented for reducing the loss of opacity which occurs when paints dry. Among the factors discussed are refractive index change, titanium dioxide crowding, tinting to improve the apparent whiteness and the hiding power, and the effect of poor levelling on the apparent hiding power.

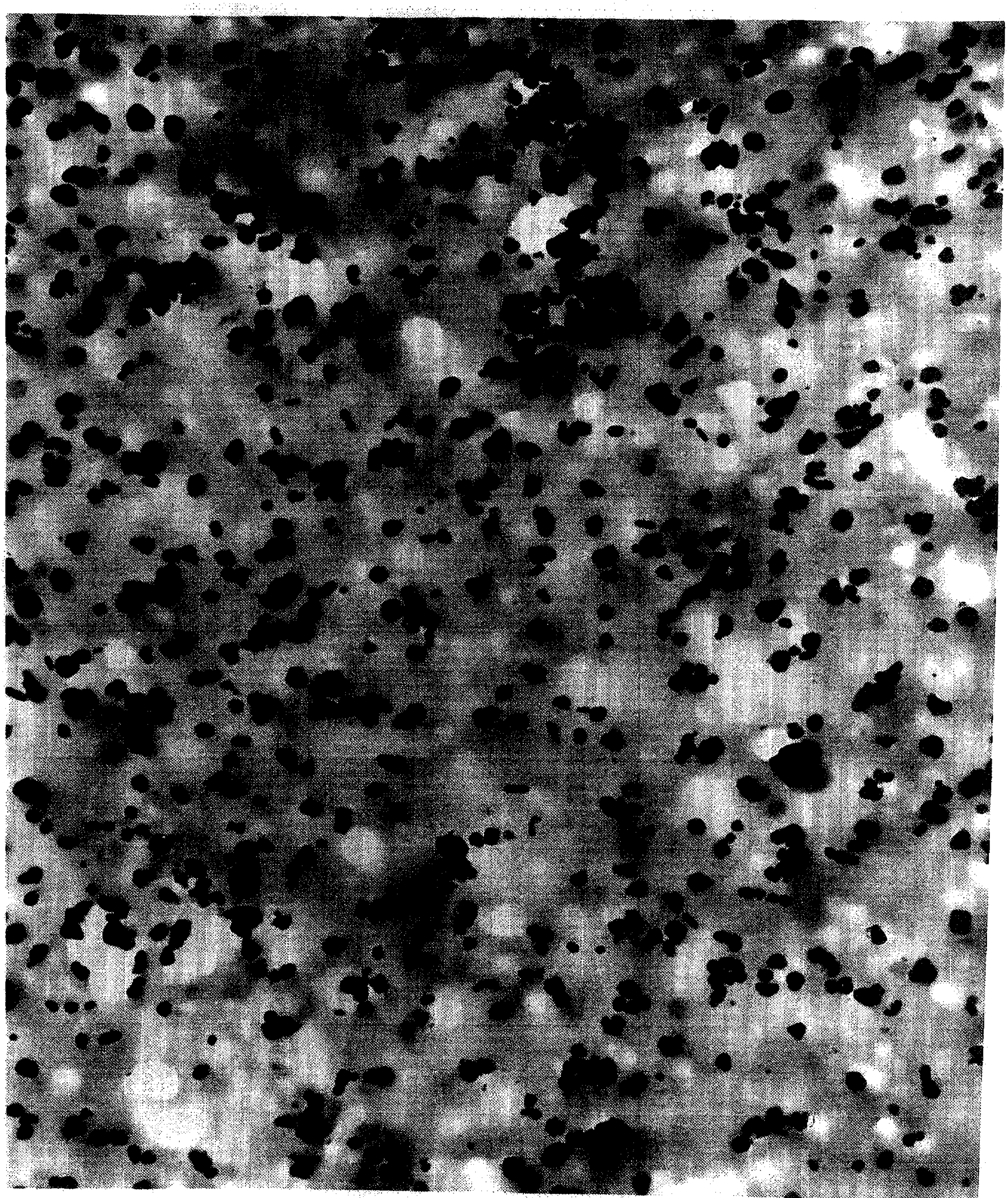


FIGURE I
CROSS SECTION OF AN EMULSION PAINT (8116-200)
SHOWING DISPERSION OF TITANIUM DIOXIDE
1 MICRON = 0.875 CMS


FIGURE II
CROSS SECTION OF AUTOMOTIVE "LUCITE®" ACRYLIC LACQUER (868-93774)
SHOWING POOR DISPERSION OF TITANIUM DIOXIDE
1 MICRON = 1.7 CMS

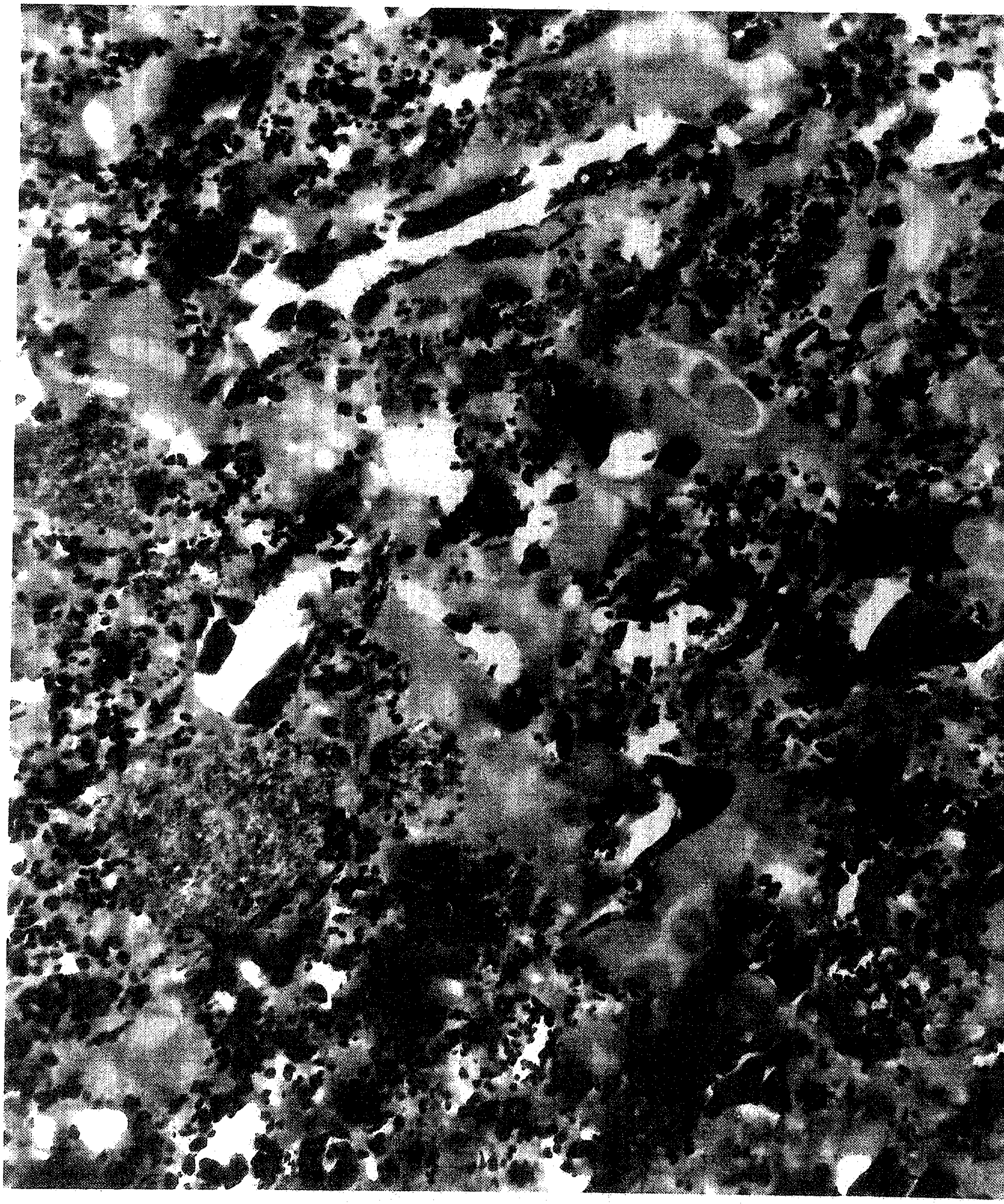


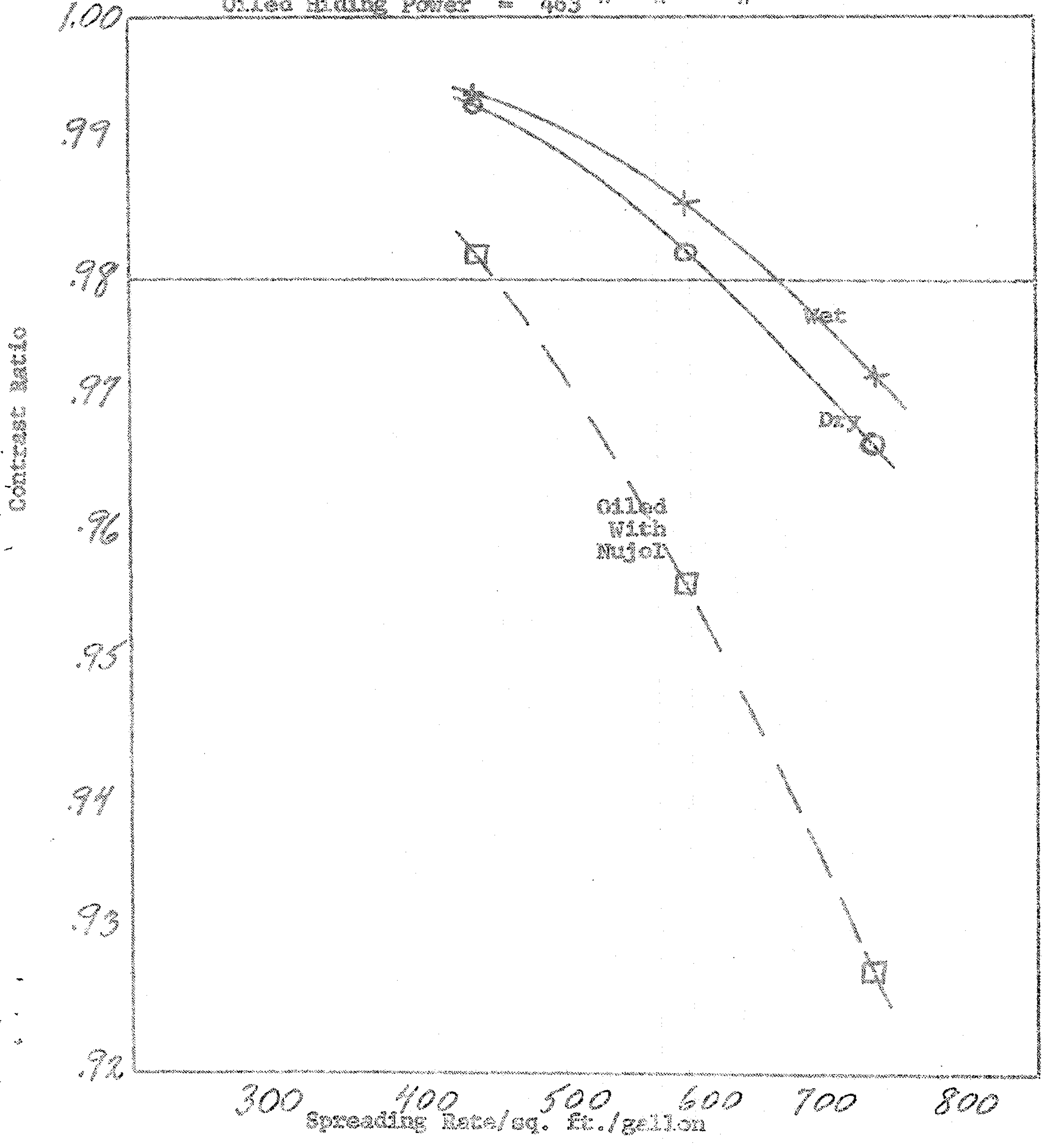
FIGURE III

CROSS SECTION OF ZEOLEX-80 PAINT (PANEL TEST PAINT 392-W-99397)
SHOWS POORLY DISPERSED ZEOLEX-80
1 MICRON = 0.35 CMS.

FIGURE 4

HIDING POWER OF A LAB. BATCH (392-B-39005)
OF A BUTADIENE/METHYLMETHACRYLATE/
METHACRYLIC ACID "LUCITE" WALL PAINT

Wet Hiding Power = 670 sq. ft./gallon
Dry Hiding Power = 625 " " "
Oiled Hiding Power = 463 " " "



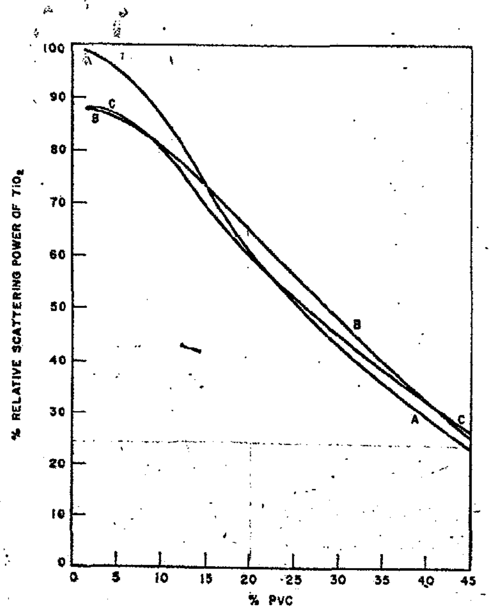



*Figure 6 — Scattering power comparison of $TiO_2$ in an automotive enamel*

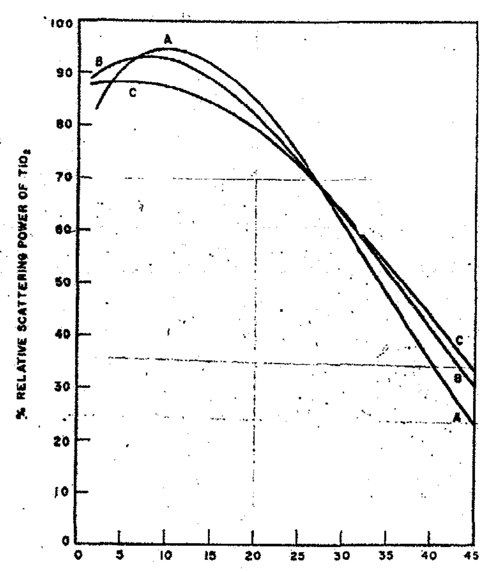



*Figure 7 — Scattering power comparison of $TiO_2$ in an architectural enamel*



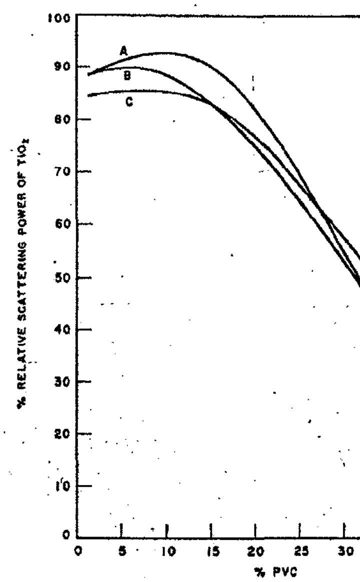



*Figure 8 — Scattering power*
*of $TiO_2$ in a can coating*

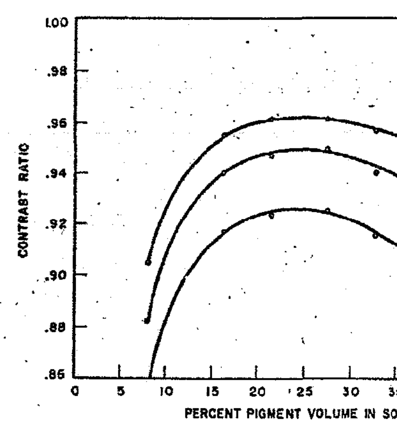



*Figure 9 — Hiding of rutile*
*dioxide in a urea-alkyd ename*
*dry film thicknesses*

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