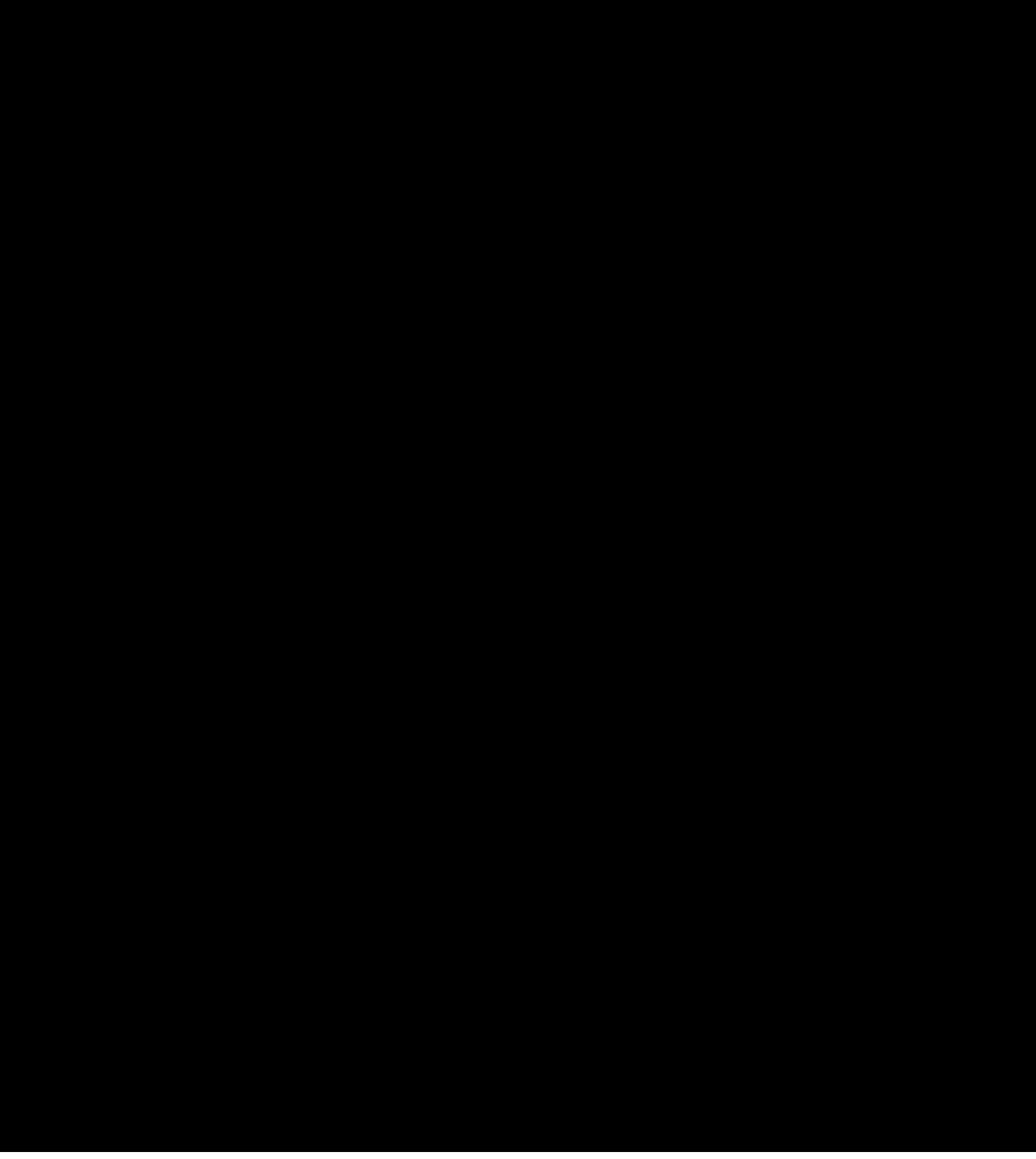




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#162 - Studies in Flat Wall Paint Formulation

The Resin Research Department has submitted the following variations of (4113) for evaluation in Flat-Tone White.

- RD 5177 P.E.-glycerol-phthalic-rosin-soya varnish.
Body - G. A.V. - 1.7. Color - 8.
Solids - 30%. Solvent - Shell Sol 72.
- RD 5156 P.E.-glycerol-phthalic-Abitol-dehydrated
castor varnish. Body - F. A.V. - 3.7. Color - 8.
Solids - 30%. Solvent - Shell Sol 72.
- RD 5178 P.E.-glycerol-phthalic-rosin-adipic-dehydrated
castor varnish. Body - A. A.V. - 15.8.
Solids - 30%. Solvent - Shell Sol 72
- RD 5163 P.E.-phthalic-Abitol-dehydrated castor varnish.
Body - F. A.V. - 5.0. Solids - 30%.
Solvent - Shell Sol 72.
- RD 5164 P.E.-phthalic-Abitol-dehydrated castor varnish.
Body - E. A.V. - 3.1. Solids - 30%.
Solvent - Shell Sol 72.
- RD 3674 Glyceryl-ethylene glycol-isophthalate-soya varnish.

In all cases where Shell's Sol 72 was used in the varnish it was also used as the solvent in the flat wall paint. The experimental batches of Flat-Tone White were checked for general performance as indicated by the data below. Quart samples were placed in storage to study package condition and stability of viscosity over a period of several months.

Our initial tests indicated that flats made with RD 5178 are poor in uniformity of tint over various surfaces. The Resin Research Department has agreed to repeat RD 5156 in order to check our initial tests which appear to be in error. Each paint was reduced with extra thinner to the point where the vehicle broke to check each odorless type of varnish for thinner tolerance.

(Chart Follows)

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0007-SWP-000114945

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Experimental Data on Flat-Tone

Paint No.	OJ 469 R	OJ 470 R	OJ 471 R	OJ 472 R	OJ 473 R	OJ 474 R	OJ 442 R
Varnish Tested (4115)		RD 5177	RD 5156	RD 5178	RD 5163	RD 5164	RD 3674
Thinner Used (5027)		Shell Sol 72	Shell Sol 72	Shell Sol 72	Shell Sol 72	Shell Sol 72	(5027)
Viscosity #5							
Fresh	12.6"	15.2"	9"	6.6"	17.8"	17.0"	19.0"
3 Days	16.2"	18.4"	9"	6.0"	25.0"	17.8"	(24 hrs.) 20.0"
6 Days	15.2"	17.4"	50.0"	6.8"	23.6"	20.4"	(31 days) 22.0"
Brushing	Good	Good	Good -	Too free	Good	Good	Sl. more feel than regular.
Flow	Good	Good -	Good	Sl. short	Good	Good	Good
Uniformity of Tint on Lap	Very good	Very good	Very good	Very good	Very good	Very good	Very good
Uniformity of Tint Over Spackle	Very good	Good -	Good	Very poor	Good	Good	Very good
Uniformity of Sheen	Good -	Good -	Good -	Good -	Good	Good -	Good -
Drying Over Night	Very good	Fair -	Very good	Good	Very good	Very good	Very good
Washability Over Glue	750 cycles	770 cycles	1500 cycles	900 cycles	1000 cycles	1260 cycles	1500 cycles
% Reduced With Sol 72 or (5027)	100% OK	100% OK	25% Broke	100% OK	100% OK	100% OK	100% OK
Reduced Paint Clear Liquid (24 hours)	7/8"	1-1/8"	-	1-1/4"	1-1/2"	1-1/2"	1"

0007-SWP-045574

0007-SWP-000114946

#187 - Physical Properties of Pigments

Work on the electrokinetic properties of pigment dispersions has indicated that the surface potential and its sign are important criteria of the dispersion characteristics of that pigment. Although electrophoretic behavior indicates whether the sign of the charge on a given pigment is positive or negative, the magnitude of the surface potential is not indicated directly. This is due to the fact that the electrokinetic migration velocity of pigments suspended in paint vehicles is a function of the dispersive capacity and general nature of the vehicle/thinner combination as well as the actual surface potential or ion capturing capability of the pigment surface. Titanium dioxide, for instance, may migrate rapidly in an electrical field if suspended in a bodied linseed oil or an alkyd vehicle, whereas the same pigment will show negligible migration when suspended in raw linseed oil, particularly if the pigment is slightly moist.

A new approach to this problem of surface potentials of pigment suspensions is indicated, based on the following general considerations. As is well known, a whole group of colorimetric pH indicators is available which change color at a given hydrogen ion concentration. These color changes cover substantially the complete pH range from 0 to 14. Each indicator changes color at a very narrow pH region and is consequently characteristic of a very precise conductometric potential equivalent to a precise capacity to either capture or release protons.

Under normal circumstances, i.e., when pigments are suspended in ionic media, the addition of an indicator of the above type to the suspension is ineffective. This is due to the fact that an ionic liquid is in itself capable of acting as a proton donor or acceptor, so that the surface can be "satisfied" by the mere presence of such a liquid. However, in an aprotic medium like mineral spirits (neither proton donor nor acceptor, i.e., non ionic) neither an acid nor base can function alone. If a base is added to an acid under these circumstances the proton from the acid can pass to the base so that a neutralization equilibrium is established and the law of mass action is applicable.

As a consequence, the equilibrium constant in the neutralization reaction in an aprotic solvent is a measure of the relative strengths of the neutralized acid and of the conjugate acid corresponding to the base used. The value of K can thus be ascertained if the color of the base differs from that of its conjugate acid. As this is the case with the above colorimetric pH indicators, it appears that a series of indicators would be capable of bracketing the surface potential of pigment particles.

0007-SWP-045575

0007-SWP-000114947

#187 - Cont.

The indicators, however, must fill one further prerequisite before they can be employed. They must be adsorbed to the pigment surfaces before proton exchange can take place. This means that they must be at least partly soluble in the aprotic medium chosen. Screening of a large number of indicators available for this characteristic has yielded only a few so far which are usable and these lie in the pK range below 7. It is expected that further work will yield a broader range of indicators for this work. It is thought that this type of approach will yield valuable information such as the after-bodying tendencies of some pigment combinations in certain vehicles, seeding tendencies and other characteristics associated with the degree of dispersion and agglomeration of pigment systems suspended in hydrophylic as well as hydrophobic media.

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#86 - Leaded Zinc Pigments

Continued carbon arc exposures on this series of yellow leaded zinc pigments have now reached 620 hours. At this stage the panels are showing chalking and are rated as follows:

<u>Panel</u>	<u>Pigment</u>	<u>White Chalk Mask</u>	<u>Degree of Chalk</u>
A	AURL 2298 Light Yellow	5	7
B	AURL 2263 " "	7	6
C	AURL 2267 " "	7	5
D	AURL 2297 Dark Yellow	7	9
E	412 Shaded to match D	4	5
F	SWP Tint Base Shaded to match D	8	7
G	AURL 2264 Dark Yellow	9	8
H	412 Shaded to match G	6	4
I	SWP Tint base shaded to match G	8	7

The above data indicate that the darker yellow leaded zinc pigments are more chalk resistant and produce a chalk that is more nearly self-colored than the other leaded zincs of this series. The poor performance of 412 dry shaded would indicate that there probably is a good possibility of formulating an even more permanent tint base by using colored leaded zinc pigments. Work is under way on this problem and will be reported on at a later date.

0007-SWP-045577

0007-SWP-000114949

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162 - Studies in Flat Wall Paint Formulation

The Resin Research Department has submitted an isophthalic (4113) type vehicle coded RD 3694 for evaluation in Flat-Tone White Rex 810. The T. F. Washburn Company and McWhorter Chemical Company have also submitted samples of flat wall paint vehicles for our inspection and test. These vehicles have the following properties.

RD 3694 Glyceryl-Abitol-isophthalate-soya
plus Antarox A-400.
Body - V +. A.V. - 16. Solids - 40%.

No. 3540 O. S. Alkyd
T. F. Washburn Company

S 2415 Odorless Alkyd
Viscosity - thixotropic. Color - 8 Max.
A. V. - 6 Max. Solids - 39 to 41%.
From McWhorter Chemical Company

The three alkyds were compared with (4113) in Flat-Tone White Rex 810 for brushing, flow, drying, stain removal, washability, etc. The T. F. Washburn alkyd was very short in flow and poor in uniformity over various surfaces. The S 2415 Odorless Alkyd was similar to (4113) in all tests and slightly better than (4113) in stain removal properties. The S 2415 Alkyd, however, gave slightly high viscosities for Flat-Tone White which detracted slightly from the brushing properties of the flat wall paint. RD 3694 is too low in body for Flat-Tone White. The experimental flat based on RD 3694 has poor uniformity of tint over various surfaces. Complete test data are presented in the following table.

Paint No.	<u>498 R</u>	<u>506 R</u>	<u>499 R</u>	<u>500 R</u>
Vehicle Under Test	(4113)	#3540 O.S. Resin	S 2415 Alkyd	RD 3694 Alkyd
Viscosity #5, Fresh	11.0"	7.2"	17.2"	5.0"
O'White	11.0	10.0	22.2	7.0
25 Days	13.2	13.0	21.4	5.0
Brushing	Good	Good	Good (-)	Too free
Flow	Good (-)	Poor	Good (-)	Good
Drying	Good	Good	Good	Good

(Cont.)

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<u>Paint No.</u>	<u>498 R</u>	<u>506 R</u>	<u>499 R</u>	<u>500 R</u>
Vehicle Under Test	(4113)	#3540 U.S. Resin	S 2415 Alkyd	RD 3694 Alkyd
Uniformity of Sheen	Fair +	Good	Fair +	Good
Uniformity of Tint	Good (-)	Very good	Fair +	Very good
Uniformity of Tint Over Various Surfaces	Good (-)	Poor	Good (-)	Poor
Washability over Glue Size	830	819	688	857
Stain Removal				
Film Dried 1 Week				
Dust	105 cycles	31 cycles	26 cycles	33 cycles
Blue Ink	604 "	703 "	456 "	486 "
Grease	141 "	76 "	44 "	70 "

0007-SWP-045579

0007-SWP-000114951

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#187 - Physical Properties of Pigments

Further work and literature search has been undertaken to ascertain the surface potential of pigment surfaces. A new indicator, Bromophthalein Magenta E (tetrabromophenolphthalein ethyl ester) is used by the National Bureau of Standards for assessing the acidity or basicity of organic molecules in aprotic media by colorimetric changes. It appears that this material might be of particular value for pigment work because of its ability to indicate color change at various values of proton exchange capacity. If the material is not available commercially, an attempt will be made to synthesize it in the laboratory.

0007-SWP-045580

0007-SWP-000114952

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#177 - Pigment Studies

A series of rutile titanium pigments are under evaluation for chalk resistance. Included in the series are minimum and maximum chalking standards of Titanium Pigment Company grade Titanox RA NC 1004 dry. Minimum and maximum chalking standards of Calco Unitane OR 640 are also included. Calco has also submitted a sample of their experimental grade ML 177D4 claimed to be a super chalk resistant material. These pigments are being evaluated in the carbon arc Damar vehicle test as well as in exterior gray and Colonial Yellow tints. Results are not sufficiently far advanced to merit report at this time.

0007-SWP-045581

0007-SWP-000114953

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#162 - Studies in Flat Wall Paint Formulation

The Resin Research Department has submitted three (4113) type vehicles coded RD 5195, RD 5203 and RD 5208 for evaluation as flat wall paint vehicles:

- RD 5195 P.E.-glycerol-phthalate-Rosin Amine D-soya varnish. Body X -. Acid value - 4.4. Color - 7. Solids - 30%. Solvent - (5027).
- RD 5203 P.E.-glycerol-phthalate-rosin-soya, varnish. Body - 0 to P. Acid value - 3.3. Color - 5 +.
- RD 5208 P.E.-glycerol-phthalate-rosin-soya, varnish (steam blow). Body - U +. Acid value - 6.0. Color - 5 +.

The three alkyls were compared with (4113) in Flat-Tone White Rex 810 for brushing, flow, drying, washability, etc. The varnishes were formulated to obtain improved stability and compatibility. Tests show that these varnishes are deficient in hold-out and in one coat properties as indicated by the data in the following table.

Test Data - Rex 810 With Special Vehicles

Paint No.	OJ 547 R	OJ 548 R	OJ 549 R	OJ 550 R
Vehicle tested	(4113)	RD 5195	RD 5203	RD 5208
Viscosity - #5 orifice				
3 days	10.0"	16.2"	11.0"	10.0"
19 "	13.6"	24.0"	15.0"	12.0"
27 "	11.8"	18.0"	11.0"	9.0"
Brushing	Very good	Very good	Very good	Very good
Flow	Good -	Fair -	Fair -	Fair -
Drying	O.K.	O.K.	O.K.	O.K.
Uniformity of Sheen	Good -	Good -	Good -	Good -
" " Tint	Good -	Good -	Very good	Good
Uniformity over various surfaces	Good -	Fair	Fair -	Fair -
Washability over glue size	480 cycles	560 cycles	482 cycles	460 cycles

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162 - Cont.

During the latter part of January a meeting was held with representatives from Paint Research, Resin Research, Varnish-Resin and Technical Service Laboratories present, to discuss the settling and gelation problems encountered with Flat-Tone production. In connection with this problem the Paint Research Department started a series of storage tests to check the effect of various agents such as Bentone 34 - 62 Y 4, high and low amounts of calcium linoleate BI 336, calcium soyate, Acto 450 (4081), etc. The test paints had been in storage for approximately three months at 80 to 90° F. when the initial inspection was made.

The first examination indicated that 16 pounds BI 336 and 4 pounds 1826 dry are not sufficient to prevent settling in the Flat-Tone formulations. The increase in BI 336 from 16 to 24 pounds made a definite improvement in package condition but further improvement is needed. We have found that one-half gallon of lecithin (5094) in addition to the regular suspending agents produced a soft paste after three months' storage. This is also true in the case where two pounds Bentone 34 was added to the BI 336 and 1826 calcium stearate as used in the Flat-Tone formulation. From our examination of these storage samples, we have recommended the continuation of 24 pounds of BI 336 and 4 pounds 1826 dry plus one-half gallon of lecithin (5094) and/or two pounds Bentone 34. The storage tests on Flat-Tone Dawn Blue Rex 808 indicated that 312 dry Nytal 300 has better suspension properties than 20 dry Asbestine and should be used in all Flat-Tone rexes.

The ratings for package condition as listed in the following table are based on the numerical values assigned to the description of paint condition as covered by Sherwin-Williams Test Method 414.42 (10 - perfect suspension, 0 - very poor).

Package Condition at Approximately 3 Months' Storage

Batch	Rex	Suspending Agent/100 Gals.						Package Condition
		BI 336	1826	(5094)	(4081)	62 Y 4	Inert	
400 R	Rex 810							
408 R	Card 21	16#	4#	-	-	-	200# 20 Dry	4
409 R	" "	16#	4#	-	-	-	200# 20 Dry	4
416 R	Special	16#	4#	1/2 gal.	-	-	200# 20 Dry	8
417 R	"	16#	6#	-	-	-	200# 20 Dry	6
424 R	"	16#	4#	-	5#	-	200# 20 Dry	5
425 R	"	16#	4#	-	-	2#	200# 20 Dry	8
429 R	"	16#	4#	-	-	4#	200# 20 Dry	5
430 R	"	24#	4#	-	-	-	200# 20 Dry	6
431 R	"	24# *	4#	-	-	-	200# 20 Dry	6
431 R	"	16# *	4#	-	-	-	200# 20 Dry	4

*Varnish partly gelled.

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Package Condition at Approximately 3 Months' Storage									
Batch	Rex	Suspending Agent /100 Gals.					Inert		Package Condition
		BI 336	1826	(5094)	(4081)	62 Y 4			
435 R	808	16#	4#	-	-	-	200# 20 Dry 150# 2247		6
436 R	"	16#	4#	-	-	-	200# 20 Dry 150# 6300		6
437 R	"	16#	4#	-	-	-	200# 312 Dry 150# 2247		7
438 R	"	16#	4#	-	-	-	200# 2201 Dry 150# 2247		4 *
439 R	"	16#	4#	-	-	-	300# 312 Dry		7
440 R	"	16#	4#	-	-	-	300# 2304 Dry		2
441 R	"	16#	4#	-	-	-	450# 2247 Dry		7

*Varnish partly gelled.

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0007-SWP-000114956

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187 - Physical Properties of Pigments

Work on an alternative method for determining CPVC of a pigment/binder system has continued. The purpose of the study is to eliminate the filtration step in the normal CPVC testing method, as this is frequently time consuming and sometimes practically impossible for certain specific pigment and vehicle combinations. The system, as developed so far, requires somewhat more manipulation and its accuracy is no better than the filtration method. It has the advantage, however, of being able to determine CPVC of difficultly filterable pigment/binder systems.

Briefly, the method consists of the following steps. The PVC of the paint to be tested is brought above the CPVC by the conventional method of vehicle substitution. If the PVC is already above the CPVC, this step may be eliminated. An accurately known volume of the paint (between 0.5 to 1 cc) is flowed out on the surface of a three inch square of plate glass, the "puddle" of paint being regulated so as not to exceed 1.5 to 2.0 inches in diameter. The thinner is then allowed to evaporate out of this film, which may require from about 10 minutes to an hour.

Since there is a deficiency of vehicle in the system due to the fact that the PVC has been brought above the CPVC, the pigment particles can pack into the closest system commensurate with their degree of dispersion, similar to that accomplished by the filtration step in the conventional CPVC testing method. Consequently, there remains only to determine the volume of this pigment cake, including all the voids. This volume, divided into the true volume of the pigment present (known from the formulation and the accurate volume of paint originally deposited on the glass plate), will be the equivalent of the CPVC of that pigment/binder system.

Unfortunately, some difficulties are encountered in determining this small volume with sufficient accuracy. These stem mostly from the fact that since it is necessary to have the PVC of the paint system to be tested above the CPVC, a substantial volume of the dried pigment cake will consist of interstitial air voids. The most accurate method of measuring the small volume of the cake appears to be the employment of specific gravity measuring techniques. This involves submerging the pigment cake into a liquid such as water for weighing. Since the water has a tendency to penetrate very slowly into the cake, displacing some of the air which is lost as bubbles rising to the top of the water, the results obtained are somewhat inaccurate.

This has been circumvented for the most part by sealing the pigment cake surface with a 3:1 dilution of a latex film such as Rhoplex AC-33. The plate with the film is dipped into the latex and the excess latex is

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Several other variations in the technique of determining the volume of the pigment cake by the same general principles are being considered. A complete report and a recommended method will be issued as soon as these systems have been investigated.

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0007-SWP-000114958

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177 - Pigment Studies

Damar chalking tests covering several grades of titanium oxide have now been completed in duplicate. To obtain more conclusive data, exterior exposures are under way which relate simple chalking to the more inclusive study of chalking, erosion, and white chalk mask of light tints. Chalk ratings at various intervals in the Atlas Twin Arc Weather-Ometer appear in the following table.

Panel	Pigment	Chalk Ratings					
		40 Hr.	75 Hr.	175 Hr.	270 Hr.	350 Hr.	410 Hr.
1.	2174 dry, Ti Pure R 610	10	10	8	7	7	7
2.	2228 dry, Ti Pure R 110	8	7	6	6	5	5
3.	2229 dry, Ti Pure R 510	9	10	8	7	7	7
4.	2269 dry, Titanox RA	9	8	6	6	5	5
5.	Unitane Sample ML 177 D 4	10	10	9	7	7	8
6.	Unitane OR 640, Lot 118013	10	10	9	8	7	8
7.	Unitane OR 640, Lot 118072	10	10	9	7	7	8
8.	1004 dry, Titanox RANC D52 H 21 #7	10	10	7	6	6	6
9.	1004 dry, Titanox RANC D52 H24 #6	10	10	8	7	6	6
10.	1403 dry, Ti Pure FF	4	4	4	1	0	0
11.	Ti Pure R 110 Z	9	10	6	7	5	5

A review of the above data indicates that the various pigments are performing in much the manner that would be expected. Of particular interest is the performance of Ti Pure 110 Z.

During the early stages of this exposure it performs much like Ti Pure R 510 but as the exposure progresses the degree of chalking more closely resembles the Ti Pure R 110 type.

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177 - Cont.

In these tests we could not differentiate between the high and low chalking standards of Unitane OR-640. Neither could we illustrate the super chalk resistant claims made for the experimental pigment sample ML 177 D 4. It is possible that as this exposure continues it may be possible to differentiate between them by their final failure.

The results on Titanox RANC verify their reported difference, sample D52 H21 #7 being less chalk resistant. It is of interest that Unitane OR 640 appears to be more chalk resistant than Ti Pure R 610 or Titanox RANC. More extensive experience with multiple samples is required to prove or disprove this point.

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162 - Studies in Flat Wall Paint Formulation

The Resin Research Department has submitted two isophthalate (4113) type varnishes for evaluation as flat wall paint vehicles:

RD 6311: Glycerol-Abitol-isophthalate-soya
with Antarox 400. Body - I.
A. V. - 11.1. Solids - 40%.

RD 3714: Glycerol-ethylene glycol-isophthalate-soya varnish.
Body - T +. A. V. - 10.5.

Both vehicles were formulated for better stability and compatibility with mineral spirits and other vehicles on storage. Various production batches of Flat-Tone White and tints made with (4113) have gelled on storage due to poor compatibility. RD 6311 and RD 3714 are somewhat low in body and gave rather poor uniformity of tint over tight and porous surfaces, when used in Flat-Tone White. Test results appear in the following table.

Experimental Vehicles in Flat-Tone

Paint No.	OJ 658 R	OJ 659 R	OJ 660 R
Vehicle Tested	(4113)	RD 6311	RD 3714
Viscosity - S-W Cup #5 Orifice			
Fresh	7.8"	8.8"	8.4"
24 Hr.	8.6"	9.0"	8.8"
7 days	6.8"	4.4"	6.2"
Brushing	Good	Good	Good -
Flow	Good	Good	Good
Drying	Good	Good	Good
Uniformity of Sheen	Good	Fair	Good -
" " Tint	Good	Fair -	Fair
" over various surfaces	Good	Fair -	Fair -
Washability	570 cycles	735 cycles	568 cycles

0007-SWP-045589

0007-SWP-000114961

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162 - Cont.

The Resin Research Department has also submitted five odorless type (4113) vehicles for evaluation in flat wall paint formulations:

RD 5193 Glycerol-P.E.-phthalate-Abitol-Dehydrol.
Body - J +. A. V. - 6.0. Solids - 30%
Solvent - Sol 72

RD 5196 Glycerol-P.E.-phthalate-Abitol-Dehydrol.
Body - F +. A. V. - 2.1. Solids - 30%
Solvent - Sol 72

RD 5201 P.E.-phthalate-Abitol-Dehydrol.
Body J - K. A. V. - 14.8. Solids - 30%
Solvent - Sol 72

RD 5205 P.E.-phthalate-rosin-soya.
Body E -. A. V. - 12.2. Solids - 30%
Solvent - Sol 72

RD 5223 Glycerol-P.E.-phthalate-rosin-maleic anhydride-soya.

Laboratory tests have indicated that RD 5223 is much superior to RD 5193, RD 5196, RD 5201 and RD 5205 in uniformity over various surfaces, when used in Flat-Tone White. This varnish will be checked further for odor characteristics with Syntex 141 Alkyd (Jones-Dabney) and McWhorter Alkyd S 2415 (McWhorter Chemical, Inc.) These two competitive alkyds have exhibited good uniformity over surfaces that vary in porosity. Test data covering the various experimental vehicles appear in the following table:

Experimental Data on Flat-Tone White
Using Odorless (4113) Type Vehicles

Paint No.	OJ 601 R	OJ 602 R	OJ 603 R	OJ 604 R	OJ 605 R	OJ 606 R
Vehicle Under Test (4113)	RD 5193	RD 5196	RD 5201	RD 5205	RD 5223	
Viscosity - S-W Cup #5 Orifice						
Fresh	9.2"	11.0"	11.0"	11.4"	10.2"	13.0"
5 days	13.2"	14.0"	13.8"	20.0"	16.8"	10.2"
12 "	10.2"	12.4"	12.0"	25.0"	17.0"	9.4"
25 "	11.4"	18.4"	16.2"	20.8"	10.0"	13.8"
Brushing	Good -	Good -	Fair	Fair	Good -	Good
Flow	Fair	Fair	Fair	Fair	Fair	Sl. short

(Cont.)

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Experimental Data on Flat-Tone White
Using Odorless (4113) Type Vehicles
(Cont.)

Paint No.	OJ 601 R	OJ 602 R	OJ 603 R	OJ 604 R	OJ 605 R	OJ 606 R
Drying	Good	Good	Good	Good -	Good -	Very good
Uniformity of Sheen	Fair	Good -	Good -	Good -	Fair +	Fair
" " Tint	Good	Good	Fair	Very good	Very good	Very good
" over various surfaces	Good -	Poor	Poor	Good -	Fair	Very good
Washability over glue size (cycles)	490	560	949	577	591	358

The Chicago Technical Service Department reported on December 10 that the Chicago Flat-Tone production showed definite settling of the pigment and some gelling of the vehicle on storage. This resulted in an investigation of the production records on (4113) for 1952. The Varnish-Resin Laboratory issued a report on their production of (4113) for this period on January 7.

It was decided after a joint meeting with the Chicago Technical Service, Varnish-Resin, Resin Research and Paint Research Laboratories that further work should be undertaken to improve the (4113) alkyd vehicle for reproducibility in the factory as well as for compatibility and stability on storage. The Paint Research Department has started various tests to evaluate suspending agents, inerts, pigmentation, etc.

In connection with this investigation the Varnish-Resin Laboratory has submitted several off-standard production batches of (4113) for evaluation.

Off-Standard Batches (4113)

Standard
Characteristics

Varnish	(4113)	Batch 90	Batch 91	Batch 92	Batch 97	Batch 106
Body (Fresh)	U - W	O -	V	Z	V +	K -
Body (40 Days)	-	U	Z ₁ - Z ₂	Z ₃	Z	O
Cure	10 - 20	15"	17"	20"	20"	25"

(Cont.)

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Off-Standard Batches (4113)
(Cont.)

	Standard Characteristics		Batch 90	Batch 91	Batch 92	Batch 97	Batch 106
Varnish	(4113)						
A. V.	3.6 - 5.4	6.3	5.8	5.4	5.8	4.5	
Color	9 Max.	6	6	6	5-6	5	
Solids	29.5 - 30.5	28.7	31.8	27.9	28.9	28.8	
Wt./Gal.	7.12 - 7.29	7.25	7.21	7.15	7.13	7.13	

Laboratory batches of Flat-Tone White made with these varnishes were checked for brushing, flow, drying, uniformity of tint and sheen. As indicated above, these varnishes had very poor stability over a period of 40 days. However, the flat wall paints made at the 40 day period have shown good stability over a period of 20 days' storage.

It is interesting to note that batches 90, 91 and 97 with viscosities ranging from U to Z₁ gave almost identical viscosities in the Flat-Tone White formulation. The (4113) Batch 92 (Z₃ body) gave high consistencies, while (4113) Batch 106 gave low viscosities in Flat-Tone White. In other characteristics such as brushing, flow, drying, uniformity and washability, we could detect only minor variations.

The paints will be held in storage for further observations on settling, gel formation, consistency changes, etc. Test data are presented in the following table.

Experimental Data on Flat-Tone White
Using Various Production Batches (4113)

Paint No.	OJ 608 R	OJ 609 R	OJ 610 R	OJ 611 R	OJ 612 R
Vehicle Tested	Batch 92	Batch 97	Batch 91	Batch 90	Batch 106
* Body (Fresh)	Z	V-W	V	O -	K
* " (40 Days)	Z ₃	Z	Z ₁ - Z ₂	U	O

(Cont.)

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#162 - Cont.

Experimental Data on Flat-Tone White
Using Various Production Batches (4113)
(Cont.)

Paint No.	OJ 608 R	OJ 609 R	OJ 610 R	OJ 611 R	OJ 612 R
<u>Paint Characteristics</u>					
Viscosity - S-W Cup #5 Orifice					
Fresh	35.0"	13.0"	13.2"	13.8"	8.0"
3 Days	43.2"	14.6"	13.8"	15.0"	7.0"
20 "	44.6"	13.0"	12.8"	13.0"	7.2"
Brushing	Good	Good	Good	Good	Good
Flow	Short	Fair -	Fair	Fair	Fair
Drying	Good	Good	Good	Good	Good
Uniformity of Sheen	Good -	Good -	Good -	Good -	Fair
" Tint	Very good	Good -	Good -	Good -	Good -
" over various surfaces	Good	Good	Good	Good	Good
Washability over Glue Size (cycles)	600	457	570	590	593

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0007-SWP-000114965

1187 - Physical Properties of Pigments

Work on the evaluation of viscosity data obtained on the high shear viscometer has continued. An interesting empirical relationship correlating the viscosity of a suspension at infinite shear velocity with the concentration of pigment by volume present in the system has been developed. Thus the plot of the hyperbolic function of the logarithm of the ratio of the viscosity of the pigmented system to the viscosity of the vehicle, when plotted against the volume percentage of pigment present, becomes a straight line. The advantage of this type of presentation over previous methods, based primarily on the Vand derivations, is the ease of determining the two constants of the system. One is the volume percentage of the densest degree of packing to which the system can be subjected. This value seems to correspond closely with the conversion of the oil absorption by the spatula method to volumetric terms. The other constant may be obtained from the knowledge of a single viscosity measurement of the system.

A nomograph has been constructed which correlates these functions in a very simple way. With this, the changes in viscosity at infinite shear velocity of a given pigmented system with changes in pigment concentration and viscosity of the vehicle phase can be obtained readily. Data from the above correlates very well with three different pigmented systems studied so far. More data will be required before the universal application of the method to all paint systems will be confirmed.

Progress is also being made in correlating the viscosity and structure of paints at given shear velocities with fundamental constants of the paint system. More work is required in this field before definite predictions of viscosity behavior of given systems can be made.

A talk on viscosity relationships was prepared for a research meeting, and a paper on a similar subject is being written for presentation at the fall meeting of the American Chemical Society.

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177 - Pigment Studies

Chrome yellow pigments were evaluated in the Weather-Ometer for degree of darkening and were rated visually as follows after 210 hours' exposure.

<u>Pigment</u>	<u>Appearance</u>	<u>Rating</u>
12074	Very dark	2
12150	Considerable darkening	5
12096	Less than considerable	6
12147	Slight darkening	8
D 3060	Slight darkening	8

The good performance of D 3060 affords an additional lemon yellow that is somewhat less red in mass tone than the 12147 standard.

0007-SWP-045595

0007-SWP-000114967

187 - Physical Properties of Pigments

The preparation of a paper on "Solid-Solid Interactions in Liquid Media" has been started. This paper will be presented at the American Chemical Society meeting to be held in Chicago during September.

A paper on "A Method of Measuring Brushability, Sagging, and Flow-out of Paints" by R. J. Blackinton, of the Los Angeles Club, appeared in the April issue of the Official Digest, pages 205-215. The author indicates that a shear velocity of 3000 reciprocal seconds is sufficient to reproduce the conditions obtained by the average painter in applying a paint on a substrate by brush. From the experiences gained with the high shear viscometer developed and used in this laboratory and described in the paper "A High Shear Method of Rating Brushability of Paints" published in the Journal of Colloid Science in June, 1952, p. 306-315, it appears that these values are not high enough and should be at least 10,000 reciprocal seconds or even higher. At 3,000 reciprocal seconds some paints, possessing a considerable degree of structure, may be classified as having a greater resistance in brushing than others having less structure when actually average painter opinion would reverse the two ratings.

The flow rating method proposed in the Blackinton paper has the disadvantage of eliminating one of the important variables encountered in the actual flow-out mechanism found in paint application. In the viscometer described, no provision is made for the fact that in a paint film thinner is constantly being lost at a rate which is undeterminable from a rheological standpoint. This has two effects which are not accounted for in the derivations as stated in the paper; (1) The viscosity of the film increases with loss of thinner, and (2) the film thickness decreases.

Furthermore, the rheological flow curve as produced at low and very low shear velocities is invariably a function of the instrument used for the measurement. This stems from the fact that at low shear velocities, structure plays the major role in determining the flow curve of the material being tested. Unfortunately, this structure can be reduced by two different, and apparently independent physical mechanisms; (1) increased shear, and (2) increased time at constant shear. No provisions are made in the described instrument to include these variables in a reproducible fashion which even approximates the conditions found in actual practice. Therefore, the flow ratings determined by this method may approach the same relative rating found in practice, but the variables not accounted for would make the ratings somewhat unreliable. The closer the instrument measuring flow comes to the actual conditions found in paint application, the more nearly accurate the ratings of the flow characteristics would be expected to be.

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7187 - Cont.

A modification of a system for measuring flow suggested by H. Skylie, of Cleveland Technical Service Department, is being evaluated and promises to give consistent ratings with field tests of the same paints.

A special chromic oxide green house paint having poor flow properties was prepared for the purpose of checking the performance of a series of flow control agents. The test paint formula follows.

350	pounds	Magnesium silicate	-	312	dry
100	"	Titanium dioxide	-	2174	dry
400	"	Leaded zinc oxide	-	412	dry
120	"	Chromic oxide	-	478	dry
34.2	gal.	Raw linseed oil	-	(2)	
17.0	"	Bodied linseed oil	-	(2538)	
9.8	"	Bodied linseed oil	-	(2538)	
1.2	"	Lead naphthenate	-	(4230)	
.8	"	Mn	"	(4231)	
10.0	"	Mineral spirits	-	(348)	

Rheotol failed to show the expected performance in the first test paint prepared from regular laboratory stocks of raw materials. We then repeated a part of the series using control paint prepared from the same materials after dehydration for 18 hours at 200° F. Rheotol converted this material from one that brush marked badly to one of objectionably high flow. The possible sensitivity of Rheotol to small amounts of moisture makes it appear unsafe for general factory use. Its application apparently should be limited to use in special cases requiring correction of such properties as gloss, flooding and flow. Rheotol is an efficient grind aid for oil type systems but it might produce surprising side reactions because of its moisture sensitivity. It promoted fast settling to a hard cake in the green test paint, but has not exhibited this effect in SWP Rex 471.

The original batch of green test paint showed a 1 H grind. To obtain a paint with better dispersion, a second batch was prepared with dehydrated pigments which had a 3 H grind. Improved dispersion produced a paint with lower consistency but with no apparent effect on flow properties.

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186 - Leaded Zinc Pigments

Series 18380 - 7 F, C were exposed during the month, testing tinted leaded zinc paints RB 4141 - 4144. The special leaded zincs involved are AUFL 2319 - 2322. SWP Tint Base is shaded to match the four special shades, as RB 4256 - 4259.

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186 - Leaded Zinc Pigments

Exposures 18625-33 CS, CN, F, K were prepared during the month, all leaded bevel siding exposed vertical south at Florida, Coffeyville and Chicago and also north at Chicago. These tests evaluate Ozlo 18 M leaded zinc in 978 W 9 Painter Craft Exterior Semi-Paste Paint #684. The paints may be described as follows:

<u>Q No.</u>	<u>Pigment Cost</u>	<u>Leaded Zinc</u>	<u>Zinc Oxide</u>	<u>Available Lead</u>
485 R	\$111.00	1424 Dry	352 lb.	20.8 lb.
486 R	112.70	Ozlo 18 M	352 "	31.
487 R	110.44	" "	355 "	23.4
488 R	110.25	" "	352 "	24.6

A regular Rex 450 SWP Undercoater - Rex 471 SWP Gloss White is used as control. The paints are applied as 3-coat work with reductions as per the 978 W 9 label, and also as 3-coat work starting with Rex 450 and following with two coats as per label.

0007-SWP-045599

0007-SWP-000114971

1187 - Physical Properties of Pigments

A number of model systems of pigment dispersions have been prepared to study further the relationships correlating the pigment concentration by volume in a paint with its viscosity at infinite shear velocity. The equation is closely related to the Vand derivation and is of the form:

$$\log \frac{\eta_{\infty}}{\eta_0} = \frac{kC}{U-C}$$

where η_{∞} is the viscosity of the paint at infinite shear velocity, η_0 is the viscosity of the vehicle, C is the volume percentage of pigment in the paint, and U and k are constants of the system. U is the ultimate pigment volume concentration of the system, and k is a function of the shape factor of the pigment.

The high shear viscosities of the pigments anatase titanium dioxide 1403 dry, magnesium silicate 20 dry and a mixture of equal parts of these pigments dispersed in raw linseed oil (2), bodied linseed oil (310), and an SAE #10 motor oil, respectively, are being studied. After dispersion of a 30% pigment concentration of each of these nine pastes on the three mill mill, the respective systems were diluted to 25, 20, 15, 10 and 5% pigment volume content. Viscosity at infinite shear velocities of these systems is being obtained.

The above systems are sufficiently diversified as to pigment particle shape and size distribution, and the vehicles induce such widely varying structural effects that it is felt that these systems are sufficiently comprehensive to prove or disprove the relationships thus far established. It is hoped that the data obtained from the viscosity measurements may also yield interesting relationships of the structure effects induced by various concentrations of the same pigments in various vehicles.

0007-SWP-045600

0007-SWP-000114972

#187 - Physical Properties of Pigments

High shear viscosities of a number of pigment and vehicle combinations have been determined at various concentrations of pigment. The pigment concentration/viscosity values at infinite shear velocities check the hyperbolic modification of the Vand viscosity equation well within experimental error. This states that the logarithm of the specific viscosity of a pigment/binder system at infinitely high shear velocity is a hyperbolic function of the volumetric concentration of pigment present, V , and two constants that characterize the system - U , the viscosimetric ultimate pigment volume concentration, and K , the shape factor of the pigment, or:

$$\log \eta_s = \log \frac{\eta_\infty}{\eta_0} = \frac{KV}{U-V}$$

η_0 is the viscosity of the supernatant liquid of the system and η_∞ the viscosity of the system at infinite shear velocity.

The systems studied so far include 0, 5, 10, 15, 20, 25, 30 and 35 per cent by volume of 1403 dry titanium dioxide, 20 dry magnesium silicate and a combination of a 1:1 ratio of 1403 and 20 dry in (310) bodied linseed oil, (2) raw linseed oil and SAE #10 mineral oil combinations diluted to about 0.45 poise viscosities with kerosene. The pigment dispersions, except those using the SAE #10 oil, were first made up at 35% pigment volume concentration and diluted to the lower values to insure equal grind of all the pigment in each series. The SAE #10 oil dispersions at high values of pigmentation showed such extreme structure that concentrations of pigment lower than 35% were required.

Since these model systems check the equation so well, several commercial multi-pigment, multi-vehicle paints will be tested to confirm completely the relationships for all viscosimetric systems of the pigment in oleophilic vehicle systems.

The vehicles employed in the model systems were purposely chosen to give as high a divergence of structure in the dispersions as practicable. Bodied linseed oil is a vehicle of relatively high dispersive capacity, while raw linseed oil and mineral oils are rather poor in this respect. With these dispersions another empirical relationship has been established between structure and concentration of pigment. This is to the effect that the dynamic modulus due to the structure of a pigment dispersion, $\eta_s S$, is a hyperbolic function of the concentration of pigment, V , and two constants which differ from the previous ones. These constants are C , a value which seems to be related to, but not necessarily identical to the CPVC of the system, and k , the physical meaning of which is still questionable. The equation

$$\eta_s S = \frac{kV}{C-V} \quad \text{fits the data obtained on the viscosity}$$

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187 - Cont.

measurements on the model systems surprisingly well. S in the equation is the slope of the viscosity curve in the shear velocity/viscosity relationship:

$$\eta = \eta_{\infty} e^{SD^{-1/2}} \quad \text{used to find the value of } \eta_{\infty}$$

the viscosity of a system at infinite shear velocity from measurements at several shear velocities.

Attempts to establish the physical meaning of U in the first equation have led to a possible method for determining the ultimate pigment volume concentration of a pigment system. This is the point at which a pigment system is packed into its tightest configuration. It is independent of the vehicle employed originally for the dispersion. The method for obtaining this point may eventually lead to a system of obtaining the equivalent of the "ideal" oil absorption of a system independent of operator errors. More work is required before the validity of the endpoint obtained and its relationship to the viscosity of the system involved can be established definitely.

0007-SWP-045602

0007-SWP-000114974

#187 - Physical Properties of Pigments

The pigment concentration/viscosity characteristics of two commercial paints, Rex 471 SWP Gloss White and Rex 810 Flat-Tone White were determined to check the conformance of multi-component pigment-binder systems with the viscosity characteristics found for model dispersions. Vehicle was removed from a portion of each paint by centrifuge. The original paints were then diluted to 80, 60, 40, and 20% of their original pigment content with the resulting supernatant vehicle. The high shear viscosities of these systems and the supernatant vehicles were determined, and the viscosities extrapolated to infinite shear velocities.

The resulting values for viscosities at infinite shear velocity fit well the logarithmic hyperbolic functions found for the model systems. This indicates that mixtures of pigments and vehicles follow the modified Vand and Brailley derivations. This was found to be true despite the fact that the Rex 810 vehicle showed considerable divergence from Newtonian flow properties for measurements at finite shear velocities.

The UPVC* factors for the Rex 471 and Rex 810 paints are 0.56 and 0.53, respectively, while the k factors are 0.88 and 0.89. Knowing these factors, the viscosity at infinite shear velocity of any concentration of a specific pigment combination, V, in a specific combination of non-volatile and volatile vehicles can be calculated for that system by the simple equation

$$\log \eta_s = \log \frac{\eta_{\infty}}{\eta_0} = \frac{kV}{\text{UPVC} \cdot V}$$

The effect of increasing or decreasing the PVC in a given paint on the viscosity characteristics of that paint can thus be assessed readily. Work on determining the effect of pigment concentration on the structure of multi-component pigment/binder systems is being continued.

*UPVC denotes the "ultimate" pigment volume concentration, i.e., at closest packing.

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#177 - Pigment Studies

Panels 12349 - 12358 C were examined after 4-1/2 years' exposure at 45° south in Chicago. This is a set of 6" x 12" cold rolled steel panels on which an evaluation of a stainless steel powder has been conducted. Other test conditions on this pigment have been reported previously. In general, these earlier tests showed that stainless steel is not a good pigment in paints that are used in contact with metal substrates. Under these conditions they tend to accelerate corrosion. These Chicago exposures serve to confirm the earlier findings. The only advantage for stainless steel over aluminum in surface coatings was observed where severe chemical fumes are prevalent. Even here a good chemically resistant vehicle such as vinyl copolymers and a primer are necessary to realize any benefit in appearance from the stainless steel pigment. Under normal atmospheric conditions aluminum pigmented top coats retain better appearance than stainless steel pigmented top coats.

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187 - Physical Properties of Pigments

The evaluation of the high shear velocity viscosity data is being continued. It has been found that when the liquid phase involved in the suspension of a pigment/binder system is not strictly Newtonian, a correction factor accounting for this divergence from linearity is required. The vehicle system of Rex 810 Flat-Tone has been found to be somewhat non-Newtonian, while most latex vehicles show wide departure from Newtonian behavior due to the incorporation of such materials as thickening agents into the system.

Although insufficient high shear viscosity data is presently available for latex paint systems, the general laws obtained for normal oil paint systems containing Newtonian binders are followed by the Rex 810 system with non-Newtonian binders when the appropriate corrections are made.

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177 - Pigment Studies

The color stability and flooding characteristics of a series of improved du Pont copper phthalocyanine blue pigments are being compared in a mineral spirits soluble alkyd enamel. Each of the pigments under study was dispersed in the bodied linseed (310), zinc resinate (3071) combination used in BL 44 Tint Base. Bases prepared in this manner were used to shade factory produced Rex 710 White Enameloid to approximately equal color densities. Samples of each enamel tint are being stored at room temperature and at 135° F. to study color and flooding stability.

Initial characteristics and ratings of the pigments under study follow:

Pigment		<u>Flooding</u>	<u>Tint Factor</u>
CP 1037	S-W Toner	Very bad	97
BT 373-D	du Pont xylene stable toner	Very bad	98
BT 366-D	du Pont resin coated toner	Moderate	93
BL 282-D	du Pont aluminum benzoate lake	Very slight	126
CG 5960	du Pont xylene stable toner	Very bad	94
P 125	du Pont xylene stable toner	Very bad	105

Tint factors represent the grams of tint base used to shade 50 ounces of Rex 710 enamel to the depth of color at which the series is being evaluated. Flooding is rated by observing the extent to which a disturbed area of wet film contrasts with adjacent undisturbed film surfaces. The fresh film is disturbed at regular intervals by wiping the finger tip gently across the surface during the setting up period.

0007-SWP-045606

0007-SWP-000114978

1187 - Physical Properties of Pigments

From purely theoretical considerations, Vand was able to expand the derived equations of Einstein relating the pigment volume concentration of paint-like suspensions of fine particles in liquids to include values of up to about 35% pigment. Somewhat later, Brailley, with the use of additional correction factors was able to extend these equations to include up to 45 - 50% of pigment. The Vand equation is of the general form:

$$\log \frac{\eta_{\infty}}{\eta_0} = K \log \frac{1}{(1-c-qc^2)}$$

where η_{∞} is the viscosity of the system at infinite shear velocity, or where no structure remains in the system, η_0 is the viscosity of the supernatant vehicle, c is the volume percentage of pigment in the system and K and q are constants of the system. K represents the shape factor and q the "immobilization factor," or that quantity of vehicle immobilized when two particles of the system collide.

One of the major difficulties in the use of the above equation with given pigment concentration/viscosity data, is the fact that the constants K and q are very difficult to evaluate. This can be done presently only by the use of a series of tediously solved simultaneous equations, and thus far no usable graphical analysis has been evolved. Consequently the use of the Vand equation becomes very cumbersome for practical evaluation of paint systems. However, the equation is very nearly proportional to a hyperbolic form of the type:

$$\log \frac{\eta_{\infty}}{\eta_0} = \frac{kc}{U-c}$$

where η_{∞} , η_0 and c have the same significance as before and k is very nearly proportional to K while U is very nearly a linear function of q . U in this case represents the pigment concentration of a particular dispersion at which the viscosity becomes infinitely great.

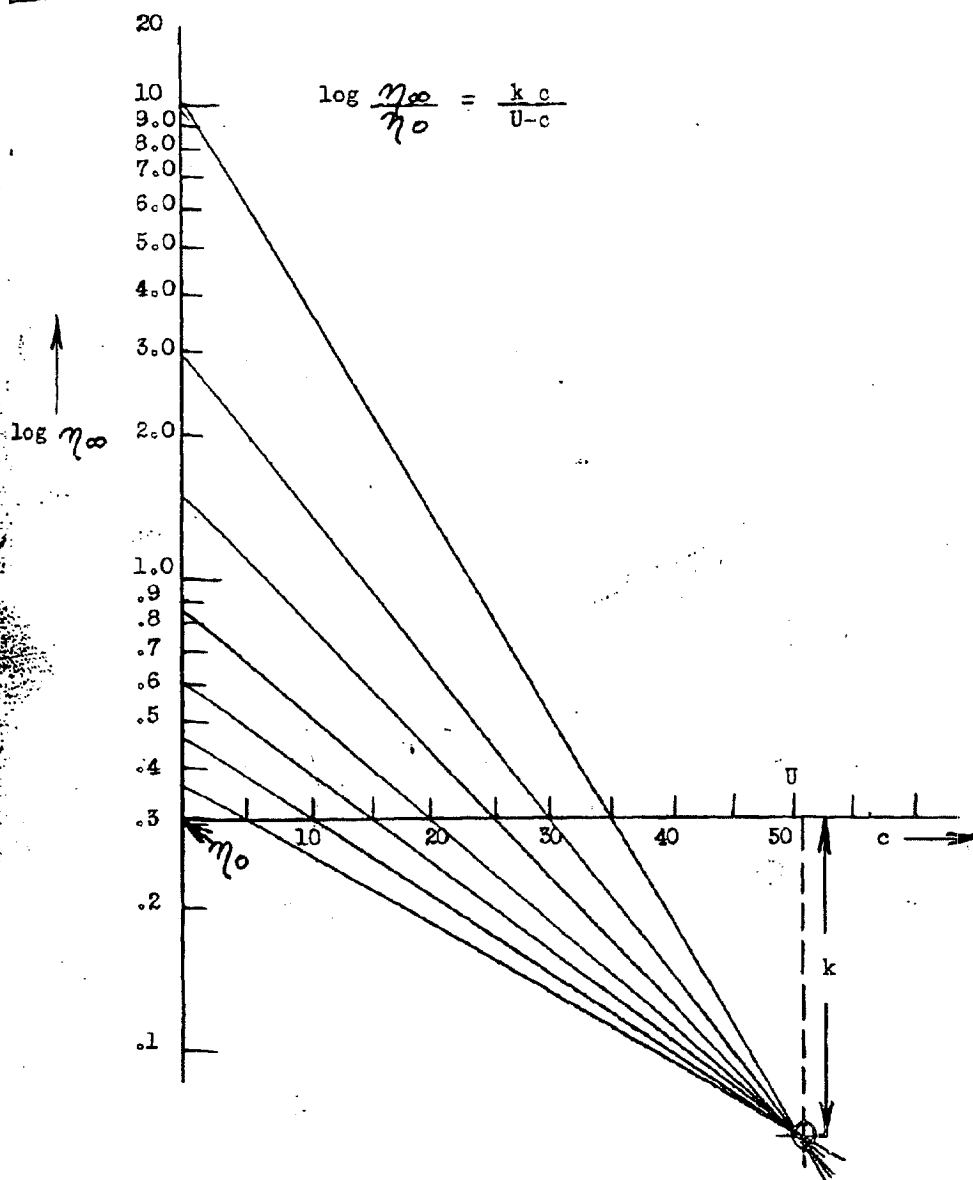
The advantage of working with the hyperbolic equation is the fact that it can be solved very readily by the use of a simple nomograph on which the constants k and U can be evaluated easily. This takes the well known Z form. One leg of the Z is a plot of $\log \frac{\eta_{\infty}}{\eta_0}$, the other k , and the diagonal is c . The plot takes on the following characteristics:

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Fig 187 - Cont.



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#187 - Cont.

The above plot represents the pigment concentration/viscosity data obtained by determining the viscosity at infinite shear velocity of 35, 30, 25, 20, 15, 10, 5 and 0 per cent of titanium oxide, 1403 dry or 6600 dry, dispersed in raw linseed oil - thinner vehicle. By connecting the experimental values of $\log \eta_{\infty}$ as the ordinate with the pigment concentrations as the abscissa, as in the above diagram, the lines all converge at one point. This point very simply designates the values of k and U , the former being the linear distance below the abscissa expressed as a function of a logarithmic decade employed in the $\log \eta_{\infty}$ plot, and U is the projection of the point parallel to the ordinate and read off as a percentage of pigment on the c axis. Knowing k and U , then for any combination of pigment and binder, the viscosity of any arbitrary percentage of pigment can be determined readily by the above simple steps. Similarly the increase or decrease in viscosity by the addition or subtraction of any arbitrary quantity of the combination of pigments used in the dispersion can be determined, as well as the effect of changing the viscosity of the vehicle on the viscosity of the pigmented system. At this time no simple means of determining k or U has been worked out except through the viscosity measurement of at least two concentrations of the given pigments in the given vehicle. It is hoped, however, that at least U can be measured by other means in the future.

One of the added advantages of the above system of obtaining k and U is the fact that the nomograph can be constructed easily on the same semi-logarithmic paper on which the extrapolations for η_{∞} are made in the plotting of

$$\log \eta^* = \log \eta_{\infty} + S/\sqrt{D}$$

for determining η_{∞} from viscosity data at finite shear velocities.

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#187 - Physical Properties of Pigments

The possibility of using a centrifuge for a rapid and simple pigment characterization test is being investigated. To date several pigments have been studied by dispersing in raw linseed oil, bodied linseed oil, and an alkyd vehicle with the rapid ball milling technique and subsequently centrifuging a portion to observe the volume of the pigment cake. Comparable results have been found with the use of either the Sharples super-centrifuge or the Size 1 Type 1 International centrifuge. The volume of pigment cake is reproducible with samples from the same batch while different results are obtained with different batches. Also, the volumes check quite closely when the various vehicles are used for a given pigment, with raw linseed oil giving a slightly larger volume pigment cake. When a minute amount of water is added to the raw linseed oil dispersion of TiO_2 to cause agglomeration, the resulting cake is noticeably larger and softer. However, when the pigment cakes are stirred and again centrifuged, additional liquid can be drawn off and it is found that volumes are in closer agreement.

While this endpoint has not the rheological significance of PPF, CPVC and UPVC, it is reproduced accurately, being entirely mechanical. It therefore may be as useful as present oil absorption tests. A preliminary paper has been prepared for eventual publication.

Paint Research Bulletin RC-93 "Prediction of Paint Viscosities in Relation to Ultimate Pigment Volume Concentration" is being distributed with this report.

0007-SWF-045610

0007-SWP-000114982

187 - Physical Properties of Pigments

One of the objections to determining the CPVC of some paint systems has been the fact that on occasion excessively long filtration times are required. The majority of paint systems can be filtered, subsequent to vehicle substitution, in the relatively short periods of one to two hours. However, some systems containing highly dispersed, very fine pigment particles may require five hours or more for the completion of the filtration step. Consequently, any technique which would shorten or eliminate the filtration step would be of considerable interest.

A method has now been developed which dispenses with the filtration step and substitutes a drying step at about 120° F., which need only be long enough to eliminate volatiles. For the great majority of paints, this should require no more than 10 to 15 minutes for even those containing very fine and highly dispersed pigments. The general technique is very similar to the previous CPVC methods employing the filtration cell. The paint is first put through the vehicle substitution step. Whereas with the filter cell method, this was an optional step, it is a necessary link in the new technique. This is because the dried cake subsequently measured must have a composition above the CPVC. This is accomplished most easily by a preliminary vehicle substitution. Only a single substitution is required for the new method, while several might be required for the filtration method for paint samples which filtered with considerable difficulty.

An exact small volume (.5 to 1.0 ml) of the substituted paint is placed upon a flat glass plate of about 4 x 4 inch size. After drying either at room temperature or at slightly elevated temperatures long enough to remove volatile thinners it is clamped into a special mercury dilatometer and the exact volume of the paint film is determined. From this volume and the volume of the total pigment in the wet film, the CPVC can be calculated in a manner similar to the original method. The whole determination, from the substituted paint forward, requires little more than 20 minutes, irrespective of the dispersion or fineness of the pigment involved.

Considerable difficulty was encountered in developing a method for determining the volume of the dried pigment cake. Because of the fact that the physical composition of this cake must be above the CPVC for the method to function, a considerable amount of air is usually present within the cake. The original attempts to measure the cake volume were made using water as the measuring liquid. The water has a tendency to penetrate the cake to a slight degree and force out a number of air bubbles. To obtain the true volume of the cake, including voids, these air bubbles must also be measured as part of the cake volume.

A cell was designed where these free air bubbles could be measured simultaneously with the remainder of the pigment cake. It was found, however, that, depending on the temperature of the water used for measurement, these bubbles would partially dissolve in the water if this were too cold, or other bubbles plate out if it were too warm, so that false results were obtained. An attempt was made to prevent water penetration by use of a very thin silicone coating over the cake surface, but this too did not meet with success.

0007-SWP-045612

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187 - Cont.

The method now employed, and which seems entirely satisfactory, utilizes a mercury cell with a very thin film of live rubber stretched across its face. The rubber conforms readily to the exact contour of the dried pigment cake, and when its glass substrate is pressed against the cell, the exact volume of the cake is measured. With this technique considerable time can be saved in future CPVC determinations.

0007-SWP-045613

0007-SWP-000114985

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#177 - Pigment Studies

Accelerated chalking tests on various titanium dioxide pigments in damar show the following results at 120 hours' Weather-Ometer exposure:

<u>Code</u>	<u>Description</u>	<u>Chalk Rate</u>
1403	Ti Pure FF	4
1901 R	Titanox 168 L0	5
2030	Titanox AA	7
2173	Ti Pure R 200	8
2228	Ti Pure R 110	8
2174	Ti Pure R 610	8
1004	Titanox RA NC	8
2132	Titanox RC HT	9
Ti Pure R-110*	80,000 series Chlorine Process	8
Unital OR 340	Unital CR 340	9
2140	Titanox RC	8
66 A 1*	Ti Pure LW	6
Ti Cal R 27	Ti Cal R 27	8

10 = Absent

0 = V. V. free chalk

*Of particular interest due to supply situation.

Phthalocyanine blue lake 24 R supplied by the C. P. Department retains greater flood resistance than du Pont's BL 282 D and Harmon's Palomar #73 after two months' storage at normal and at elevated temperatures.

Reflectance Data

<u>Pigment</u>	<u>Age</u>	<u>Blue</u>		<u>Green</u>		<u>Amber</u>	
		75° F.	135° F.	75° F.	135° F.	75° F.	135° F.
24 R	Fresh	58.2	-	24.9	-	16.6	-
	1 Mo.	59.2	59.5	24.6	24.2	16.2	15.9
	2 Mo.	58.3	58.9	23.9	24.3	15.8	16.1
BL 282 D	Fresh	56.8	-	25.0	-	16.8	-
	1 Mo.	58.0	57.5	25.0	24.8	16.7	16.5
	2 Mo.	56.8	56.7	24.6	24.9	16.4	16.7
Palomar #73	Fresh	61.3	-	25.5	-	16.8	-
	1 Mo.	62.8	62.8	25.6	25.2	16.8	16.4
	2 Mo.	62.0	62.0	25.1	25.0	16.3	16.4

Films cast from enamels aged for two months show a greater change in the color values of the 24 R tinted specimens than those containing BL 282 D or Palomar #73 pigmentation.

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177 - Cont.

Crystal stability in strong solvents has been checked for two new phthalocyanine blue toners, SK 61 A and SK 61 B, prepared by the Chemical Products Laboratory. After one month storage at 130° F. as dispersed in a vinyl vehicle, these two pigments show more fading than a competitive phthalocyanine blue pigment. This fading presumably is the result of crystal growth resulting from a slight solubility of the pigment in the solvents. The competitive pigment has the disadvantage of greater agglomeration than the S-W samples. A fourth phthalocyanine blue toner, 474 E-A, was also included in these tests as a control. Failures due to crystallization of 474 E-A were not as severe in this latest test as they were in previous tests, so the indications are that the tests should be repeated.

0007-SWP-045615

0007-SWP-000114987

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March, 1952

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187 - Physical Properties of Pigments

Work on the viscosity of paints at very high shear velocities was continued. A series of experiments was planned to prove or disprove the validity of the concept that at infinitely high shear velocities all structure in a paint has been effectively eliminated. Unfortunately, infinite shear velocity cannot be achieved by physical measurement, so that we must resort to a mathematical or graphical approach.

By plotting the reciprocal of the shear velocity, D , against the viscosity, η , of the paint at a particular shear velocity, a graph is obtained which, when interpolated to $1/D = 0$ gives the value of the viscosity of that material at infinite shear velocity. A linear plot of these values results in a curve, which it is difficult to extrapolate. By plotting these values as the logarithm of the viscosity against the reciprocal square root of the shear velocity, straight line relationships are observed for most paints, provided the shear velocity does not go materially below about 300 reciprocal seconds. This is illustrated in the accompanying graph.

If two paint systems having exactly the same composition with the exception that one has a greater degree of structure than the other have their viscosities determined and the values plotted as outlined above, the intercept at infinite shear velocity should meet at the same point. Such systems can be prepared by employing the bodying effect of water on such pigment/binder combinations as TiO_2 with raw linseed oil. If such a system is prepared by first drying the TiO_2 carefully, a paint of relatively low structure is obtained. By adding 1% of water to this system, it can be bodied up considerably. If the viscosities of these two systems, which differ only negligibly in composition but materially in structure, are determined and plotted in the manner suggested above they will be found to have the same viscosity at infinite shear velocity. This is illustrated on the graph.

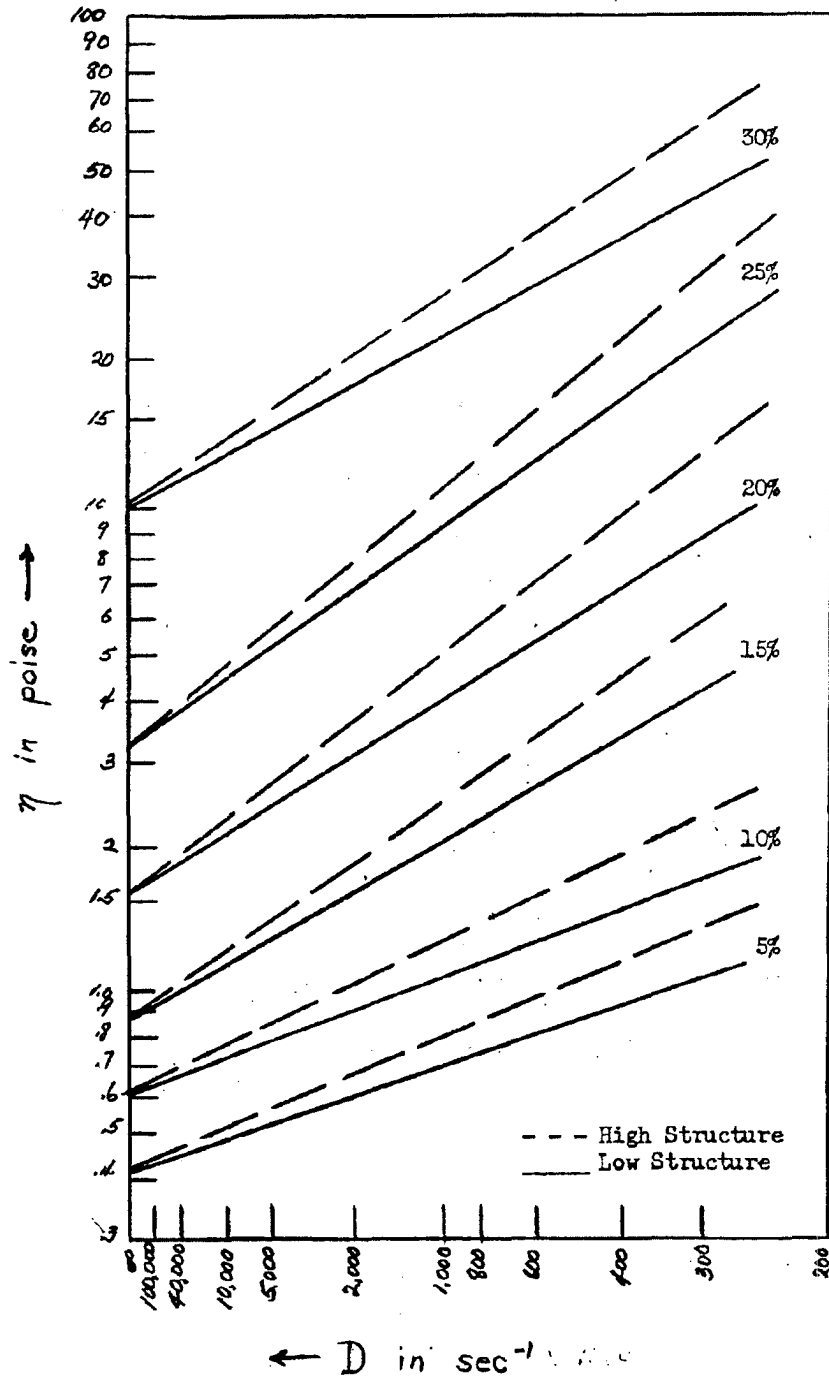
A series of paints was run in this fashion with 5, 10, 15, 20, 25 and 30% pigment. All of these materials were found to conform to the above, indicating the original assumptions to be correct for this pigment/binder combination. Further tests will be carried out on other combinations as well as on paints chosen arbitrarily.

It is hoped that considerable information can be obtained by this type of analysis about production batches of paints.

0007-SWP-045616

0007-SWP-000114988

7187 - Cont.



0007-SWP-045617

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March, 1952

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177 - Pigment Studies

Continued exposure of the TiO_2 paints in the accelerated chalking tests show the following results at 375 hours in the Weather-Ometer.

<u>Code</u>	<u>Description</u>	<u>Chalk Rate</u>
1403	Ti Pure FF	2
1901 R	Titanox A 168 LO	2
2030	Titanox AA	2
2173	Ti Pure R 200	4
2228	Ti Pure R 110	2
2174	Ti Pure R 610	3
1004	Titanox RA NC	2
2132	Titanox RC HT	2
Ti Pure R 110	80,000 series Chlorine Process	2
Unitane OR 340	Unitane OR 340	2
2140	Titanox RC	2
66 A 1	Ti Pure LW	1
Ti Cal R 27	Ti Cal R 27	2

10 - absent

0 - very, very free chalk

These results would indicate that all TiO_2 pigments eventually tend to arrive at the same constant chalk rate in this accelerated test and that their differences lie in the length of period before chalking starts. It will be recalled that ratings at 120 hours reported in February listed the pigments in the expected order.

Titanium Pigment Corporation has submitted a sample of Titanox C 50, a 50% TiO_2 calcium base pigment. It was compared with Titanox RC HT, the 30% TiO_2 calcium base pigment, in Flat-Tone White, Semi-Lustre White and in SWP Gloss White on an equal TiO_2 basis, balancing extender content. The tests showed Titanox C 50 to have good grinding properties and to be useful where it would be desirable to increase hiding within the same volume presently occupied by the 30% TiO_2 pigment, or where more selectivity of inert content is preferred. However, its hiding power per pound of TiO_2 is somewhat lower. Data obtained in substitution in Flat-Tone White Rax 810 are as follows:

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	OJ 645 Q	OJ 655 Q	OJ 656 Q
	565# Titanox RC HT 200# 20 dry Magnesium Silicate	340# Titanox C 50 416# 20 dry	340# Titanox C 50 200# 20 dry 200# 2247 dry - Atomite
Viscosity #5 Orifice			
Fresh	9.8 sec.	18.3 sec.	8.4 sec.
Over night	11.4 "	26.1 "	9.6 "
Brushing	Good	Good -	Good +
Flow	Slightly short	Short	Slightly short
Drying (over night)	Very good	Good	Good -
Uniformity of Sheen	Good	Slightly less uniform than 645 Q	Good
Uniformity of Tint			
(a) Over lap	Good	Good	Good
(b) Over various surfaces	Good	Good	Good
Scrub resistance			
over glue size	1200 cycles	1158 cycles	1240 cycles
Hiding - Contrast Ratio			
at 400 sq.ft./gal.	.987	.975	.977

Substitution of Titanox C 50 in Semi-Lustre White Rex 210 for Titanox RC HT on a TiO_2 basis results in a reduction of opacity from 478 square feet per gallon to 452 square feet per gallon at 95% contrast ratio. Substitution in SWP Gloss White for the Titanox RC HT content on a TiO_2 basis shows only minor loss of hiding. Panels 16874-89 were placed on exposure during the month to check the weathering properties of Titanox C 50 in SWP Gloss White and yellow and gray tints.

Continued storage of two new phthalocyanine blue toners in vinyl vehicles SK 61 A and SK 61 B, prepared by the Chemical Products Laboratory has shown some promise for these pigments. As reported last month these pigments showed considerable fading during the first month of storage. However, this fading does not appear to be due to crystal size growth as microscopic examination shows no crystals whereas the control, du Pont's standard, shows

0007-SWP-045619

0007-SWP-000114991

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crystals. In addition, the 474 E-A sample which was reported to have shown better performance than expected at one month has now failed very badly, re-stating confidence in the test procedure.

The special phthalocyanine blue lake 24 R supplied by the C. P. Department shows acceptable color stability after a three month aging test in an enameloid White Rex 710 paint system. Samples were stored at room temperature at 135° F. This material compared very favorably with control samples of du Pont's BL 282-D and Harmon's Palomar #73. It retains superior flood resistance during the first hour of drying of the wet film, after which it takes an favorable position with respect to the reference materials in this respect.

Hunter Multipurpose reflectance values show a slight increase in strength of color for all samples through the over-all test period. Data for this inspection follow. Refer to page 10 of the February report for previous data.

Pigment		Reflectance Data		
		Blue	Green	Amber
24 R	75° F.	57.4	24.1	15.9
24 R	135° F.	57.8	24.5	16.3
BL 282 D	75° F.	55.5	24.8	16.4
BL 282 D	135° F.	55.2	24.9	16.7
Palomar #73	75° F.	59.9	25.2	16.3
Palomar #73	135° F.	60.2	25.1	16.4

Exposures 15380-91 F were examined after 12 months' exposure at Florida vertical south. These panels, all cedar bevel siding primed with 450 Undercoater, were retired because only fading developments were requested. The tests consisted of five phthalocyanine blue pigments submitted by the Organic Research Laboratory. They were made up into: (a) Rex 412 SWP Castilian Blue, full color depth, and (b) let-downs of same with one part each to 7 parts of SWP Tint Base by volume. The pigments are:

- #474E-A — S-W, untreated.
- 474E-B — S-W, treated.
- 474E-C — General Dyestuff, Heliogen Blue, ENC.
- 474E-D — du Pont Monastral Fast Blue, ENC, Lot 62 special.
- 474E-E — " " " " " Lot 50 regular.

No film failure or gloss or dirt differences were noted. The routine reports showed greater chalking on "C" and "D," but this was only barely noticeable at 12 months. All the full colors looked fairly good, allowing for some chalk-masking. The let-downs were much poorer in appearance, due to excessive chalk-masking and dirt. A few very minor differences in general fading were noted, but not enough to warrant rating any sample definitely better than the others.

0007-SWP-045620

0007-SWP-000114992

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April, 1952

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Physical Properties of Pigments

A paper on the "Flow Properties of Pigmented Systems" is being prepared for presentation at the Gordon Research Conference on Organic Coatings on June 19. A research bulletin will be issued on this subject in the near future.

0007-SWP-045621

0007-SWP-000114993

Paint Research
May, 1952

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Physical Properties of Pigments

The determination of the CPVC for a number of asphaltic materials containing coarse fillers was attempted. The filler particles were so large, however, that the usual CPVC technique was not applicable and modification of the method must be employed in order to obtain checking results.

Further work on the "drying method" for CPVC, which eliminates the filtration step, has been carried out. A simple method for sealing the test cell has been worked out so that the former difficulties with air leakage appears to have been eliminated.

A research bulletin, RC-91, will be distributed. This covers the subject of "Flow Properties of Pigmented Systems" and is to be presented at the Gordon Research Conference on Organic Coatings.

0007-SWP-045622

0007-SWP-000114994

Paint Research
May, 1952

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Studies in Flat Wall Paint Formulation

At the Trade Sales Technical Board meeting held in Chicago on May 13 we were requested to evaluate the performance of the Lowe Brothers (39-A-14) and the S-W alkyd (4113) in several Mellotone and Flat-Tone colors. The following rexes were checked: Mellotone Crown Blue 601, Persian Brown 603, Persian Green 607; Flat-Tone Highland Green 841, Wild Blue 842 and Winslow Brown 837.

In general we found (39-A-14) to be superior to (4113) in uniformity over various surfaces and on our regular 15 minute lap test when used above rexes.

The Mellotone formulas made with (4113) are thin in initial body the Flat-Tone colors made with (39-A-14) are abnormally high in consistency. The tests indicate that a pigmentation based on pure titanium dioxide is preferred to all titanium calcium for the hiding pigment in order to obtain better washability and uniformity over tight and rough surfaces. We have found that a combination of pure TiO_2 and added calcium is preferred to titanium calcium for improving washability and uniformity of tint over such surfaces. However, this type of pigmentation would be somewhat higher in cost based on equal opacity. The improvements that can be made in washability and in uniformity of tint through pigmentation are more than can be obtained through the use of (39-A-14).

Experimental Data on (39 A 14) and (4113) in Flat Wall Paints

Rex	Vehicle Tested	Viscosity		Brushing	Flow	Uni- formity on 15 min. lap	Wash- ability over glue size (cycles)	Uni- formity over various surfaces
603	(39 A 14) Lowe Bros.	11"	15.8"	Good	Good	Good	552	Good
603	(4113)	5.4	5.8	Rather thin	Good -	Good -	920	Fair
607	(39 A 14) Lowe Bros.	15	24.4	Good	Good	Good -	857	Good -
607	(4113)	6.6	6.5	Too thin	Good -	Fair	1203	Good -
601	(39 A 14) Lowe Bros.	18	33.8	Slightly sticky	Good	Good	351	Good

(Cont.)

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Cont.

Experimental Data on (39 A 14) and (4113) in Flat Wall Paints
(Cont.)

	Vehicle	Viscosity		Brushing	Flow	Uni- formity on 15 min. lap	Wash- ability over glue size (cycles)	Uni- formity over various surfaces
Rex	Tested	Fresh	O'Nite					
Q 601	(4113)	10.2*	15*	Good	Good -	Good -	1110	Fair
Q 841	(39 A 14) Lowe Bros.	20	25	Slightly sticky	Good	Fair -	412	Good
Q 841	(4113)	8.8	9.4	Good	Fair	Poor	601	Fair
Q 842	(39 A 14) Lowe Bros.	20	24	Slightly sticky	Good	Good -	559	Good
Q 842	(4113)	8	8.2	Good	Fair	Poor	503	Fair
Q 837	(39 A 14) Lowe Bros.	15	17	Good	Good	Fair -	203	Good
Q 837	(4113)	6.6	7	Too thin	Fair	Poor	874	Fair
Q 841	(39 A 14) Chicago	17.2	18.6	Good	Good	Very good	1160	Good*
Q 841	(4113)	7.0	8.5	Good	Good	Good	1280	Fair*
Q 841	(4113)	5.7	5.8	Too thin	Good	Good	1940	Very good**

Pigmentation: 1403 titanium oxide, 2132 rutile titanium calcium,
566 calcium carbonate, 6300 Calwhite.

Rutile TiO₂ and calcium carbonate pigmentation.

Frederick A. Stresen-Reuter, Inc. has submitted a sample of No. 205 Calcium Pulp for our inspection. This material is a calcium pulp made from fatty acids. The calcium soyate pulp was compared with calcium linoleate dry in EX 677. Experimental batches of Flat-Tone White were prepared with these bases and the paints have been placed in storage to check suspension properties.

The Oakland laboratory has submitted a sample of Flat-Tone White with Lasher's L 238 OM low odor vehicle from Specialty Resins. The flat paint sample is somewhat better than regular Flat-Tone White in odor but was inferior in uniformity of tint over tight and porous surfaces.

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Paint Research
May, 1952

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Leaded Zinc Pigments

Panels 13146-9 F, C were examined after approximately 3 years' exposure at Florida vertical south and at Chicago north and south, all cedar bevel primed with Rex 450 Undercoater. (a) Regular SWP Tint Base of early 1940 lbs. of 412 dry co-fumed leaded zinc was compared with (b) our batch of Lead Company's Dutch Boy Buff, a formula using a mechanical blend of lead and zinc, with all lead as 18 dry basic carbonate white lead and a lower inert content than our formula. An added formula (c) was one the same as (a) but a mechanical blend using 9 dry basic sulfate lead and 18 dry to match the composition of the 412 dry.

The results on these tests had varied considerably in the past, serious failure had shown up. At present there is serious microscopic failure at Florida on both mechanical-mix formulas, none on (a). During most of the first year, Dutch Boy looked good due to superior color-retention; at the end of the year it looked poor due to chalk-masking and dirt (as did the (c) formula); by the end of the second year it is back to a better appearance than the others due to more thorough elimination of chalk-mask and dirt. The panels are being continued in test because of these past variations.

0007-SWP-045625

0007-SWP-000114997

Paint Research
December, 1952

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#86 - Leaded Zinc Pigments

Laboratory produced samples of yellow leaded zinc pigments have been placed on exposure at the Chicago farm as well as in Miami, Florida. For these exposures the pigments were dispersed in the SWP tint base vehicle substituting the yellow leaded zinc pigments for the entire pigment volume normally contained in Rex 473 Tinting White. In comparison with this series of leaded zinc paints two control paints were used. Control paint A was standard Rex 473. Control paint B was made using Ozlo 412 leaded zinc oxide in the same manner as used on the experimental pigments. Portions of these control paints were shaded to match the lightest of the leaded zinc yellows while other portions were shaded to match the deepest of the leaded zinc yellows.

Carbon arc exposures on this series of paints have completed 535 hours. At this stage chalking is just beginning on the control leaded zinc paints. This chalking is only slight but there is evidence of some chalk face. The mixed pigment tint base is not chalking but it has flatted almost completely. The colored leaded zinc paints are quite chalk resistant at this stage of exposure. This chalk resistance would be of considerable importance if it carries through on exterior exposure and in more practical formulating approaches. Panels 18148-59 C, F were prepared for exposure in Chicago and Florida south vertical of the initial experimental paints. Work is under way on the more complete evaluation of pilot plant batches of similar pigments.

0007-SWP-045626

0007-SWP-000114998

Paint Research
March, 1951

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#177 - Pigment Studies

The phthalocyanine blue color stability test that is being run on a series of five unidentified samples supplied by the Chemical Products Department continues. The samples show excellent color stability for all samples in alkyd and oil type vehicles as represented by Enameloid and SWP formulations, respectively. Samples of these rexes prepared from the separate pigment specimens have been observed after two months' aging at normal room and 135° F. temperatures. Similar tests in vinyl formulations show weakness in certain samples after one month's storage.

None of the full color dispersions in Enameloid Blue - Rex 708 show any color difference between the room and oven stored samples. A slight fading is apparent in all the oven stored let down tints of Enameloid and SWP. The full color SWP Rex 412 Castilian Blue samples show a slight deepening of color at the 135° F. storage level.

It is now apparent that the poor wetting and severe agglomeration and/or solubility reported for these pigments on page 12 of the February report is not a general characteristic, but it does apply to some of the samples in vinyl systems. Accumulated data will be released after the 3-month inspection.

0007-SWP-045628

0007-SWP-000115000

Paint Research
March, 1951

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#86 - Leaded Zinc Pigments

Exposures 12510-44 C, F were examined after two years' exposure at Florida, vertical south and at Chicago south and north. The panels are primed with regular Rex 450 Undercoater except for a few cases in which one of the test variations is made in the undercoater as well as in the top coat. The series was put out in cooperation with the National Lead Company, to the extent that they received samples of our paints and exposed them in their regular manner at Sayville, Long Island. We have received no recent information from them except as observed on our last annual inspection trip to their farm.

The broad purpose was to evaluate various replacements of 412 dry 35/65 leaded zinc with 414 dry 12/88 leaded zinc and National's 45 X basic silicate white lead in Rex 471 SWP Gloss White and in SWP tint base. The substitution, less extensive in the tints than in the whites, involved three methods:

- A. Zinc oxide replacement by equal poundage, lead by equal volume.
- B. Maintain 35/65 lead/zinc ratio, replace on total volume.
- C. Same, but replace on total weight, adjust volume with extender.

(In certain cases involving only Method A, (1) the zinc oxide level was lowered from the normal 32% to 25% and 20%, and (2) zinc oxide was introduced as Azo 222-33 instead of leaded zinc.)

A previous report at approximately one year showed no significant differences in performance, at least none which were confirmed at all locations. The present routine study of Florida and Chicago tests indicates that no new differences of consequence have developed in the second year. The main tendency on dirt collection seems to be that the regular guides are cleaner than the specials, except that those the same as the regular but lower in zinc oxide seem cleaner.

The SWP tint base tests, buff and gray, are all well within a satisfactory range of dirt collection and fading. The special formulas run about the same as the regulars in both respects. One sample carrying 414 dry and 45 X at a 25% ZnO level is definitely cleaner than the average, and one similar but at 20% ZnO is somewhat cleaner and noticeably better in color. This is based on Florida results, which also show some incipient checking. Chicago tests show no noticeable differences. In general, the use of 45 X and 414 dry seems fully satisfactory through two years' exposure, and essentially the same can be said of the reductions of zinc oxide level, which would have advantages from the standpoint of cost and supply. (The behavior at lower zinc oxide levels is rather unexpected and no ready explanation suggests itself.)

0007-SWP-045629

0007-SWP-000115001

Paint Research
April, 1951

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#177 - Pigment Studies

The phthalocyanine blue color stability test has been carried through the 3-month aging period in Enameloid Blue - Rex 708 and SWP Castilian Blue - Rex 412. All samples continue to show good stability at 135° F. storage temperature as well as at 75° F. However, with vinyls marked failures are apparent after two months' storage. One pigment showed early failure in the form of agglomeration but this failure has not progressed. Two of the pigments showed bad fading as a result of large crystal growth. The other two pigments showed some agglomeration and some crystal growth but in general gave the best performance. There are indications that the phthalocyanine blues in a urea-alkyd enamel are going to duplicate the performance in vinyls.

0007-SWP-045630

0007-SWP-000115002

Paint Research
June, 1951

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#177 - Pigment Studies

Phthalocyanine Blue Lakes

The color stability of two phthalocyanine blue lake pigment samples is being determined in standard Enameloid White - Rex 710 shaded to the approximate depth of color of Enameloid Pastel Blue. The pigment specimens were selected for this study because of their superior performance in flooding characteristics tests run on 12 preparations submitted by the C. P. Department. du Pont's phthalocyanine blue lake HL 282 D is the control pigment in these tests.

The pigment sample WSF 59 shows a slight weakness after aging one month at 135° F., while WSF 70 appears to possess excellent stability. Additional inspections will be made after two and three month aging periods.

0007-SWP-045631

0007-SWP-000115003

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July, 1951

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177 - Pigment Studies

The Chemical Products Department experimental phthalocyanine blue pigment sample WSF 70 continues to show good color stability in the alkyl type Enameloid formulation after two months aging. Samples of Enameloid White Rex 710 tinted with BL 44 type bases, containing WSF 59, WSF 70, and du Pont's BL 282 D laked phthalocyanine blue pigments, have been stored at room temperature and at 135° F.

The BL 282 D sample appears to have bleached very slightly during the 135° F storage, but the Hunter reflectance values of this film show excellent stability in comparison with the reflectance values of the original fresh film. A loss of color strength is readily apparent in the 135° F stored sample of WSF 59 tinted enamel and is confirmed by the increase in reflectance shown by this chip. WSF 70 shows continued stability along with a dispersing action in the 135° F storage condition which has developed an increase in the color strength at this inspection.

Reflectance Values

		<u>Fresh Enamel Film</u>			<u>2 Month Aged Enamel Film</u>		
		<u>Green</u>	<u>Blue</u>	<u>Amber</u>	<u>Green</u>	<u>Blue</u>	<u>Amber</u>
BL 282 D	75° F	28.8	62.0	20.2	28.6	61.4	20.0
	135° F				28.8	61.6	20.2
WSF 59	75° F	29.4	63.8	20.7	29.0	63.1	20.6
	135° F				30.8	63.0	22.6
WSF 70	75° F	28.6	61.2	20.3	28.4	60.7	20.0
	135° F				27.5	60.5	19.6

0007-SWP-045632

0007-SWP-000115004

Paint Research
August, 1951

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#187 - Physical Properties of Pigments

The first phase of the work on correlating the brushability of paints with their viscosities at high shear velocities has been completed. Very good check results are obtained when the values are compared at a shear velocity of about 15,000 reciprocal seconds, which corresponds to the shear velocities obtained in practical brushing. Because of the physical limits of the present viscometer, the viscosities of the more viscous paints at the above mentioned shear velocities had to be obtained by extrapolation of a curve. To do this, a number of readings for each paint are required. The methods and procedures for doing this are set forth in a research bulletin to be circulated in the near future.

The project is to be continued with the construction of an apparatus on which the brushability of a paint can be determined with a single reading. This should make the device very adaptable to routine testing of paints.

Further work is also contemplated to establish the relationship between viscosity values obtained at high shear velocities and the flow characteristics of paints. It is hoped that a complete rheological system can be established with which the viscosity behavior of given paints can be accurately predicted over relatively large ranges.

Experimental work on establishing a gravimetric method for CPVC is being continued. The method shows promise of extending the scope of CPVC work, making the determination faster for some pigment/binder systems than the present filter cell method. At the present time, however, the gravimetric method has not been perfected to the point where it can be employed with certainty, but some of the results obtained checked well with the present method.

A paper on the "Electrokinetic Properties of Pigment Dispersions" will be presented at the Diamond Jubilee Meeting of the American Chemical Society at New York.

0007-SWP-045633

0007-SWP-000115005

Paint Research
August, 1951

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#177 - Pigment Studies

The experimental phthalocyanine blue laked pigment, WSF 70, prepared by the C. P. Department, shows good color stability after aging 3 months at 75 and 135° F. in the form of Enameloid alkyd type pastel blue tints. This material has shown excellent stability under 75° F. storage and has increased slightly in color strength at 135° F. The moderate loss of reflectance across the board by the 135° F. 3-month aged sample confirms this gain in color strength.

WSF 59, a second C. P. phthalocyanine blue lake, exhibits excellent stability at the 75° F. level but it faded observably in the 135° F. test. This material showed a slight weakness at 135° F. at the 2-month inspection. The extent of color change is indicated by the variation of reflectance values with respect to those established by films cast when the enamel was fresh.

Both of these lakes retain their effective recovery of color after their films are disturbed during the first 15 minutes of their setting up time. They are equal to the control sample, du Pont's blue lake EL 282 D, at this point, but show increasing flooding characteristics when disturbed after this time. Flooding is apparent in the EL 282 D tinted sample at this point also, but it increases less with time than in the WSF 59 and WSF 70 tinted films.

Hunter Multipurpose Reflectometer test data follows:

Reflectance Data

		<u>Fresh Enamel</u>			<u>Aged 2 Months</u>			<u>Aged 3 Months</u>		
		<u>Green</u>	<u>Blue</u>	<u>Amber</u>	<u>Green</u>	<u>Blue</u>	<u>Amber</u>	<u>Green</u>	<u>Blue</u>	<u>Amber</u>
Control	75° F.	28.8	62.0	20.2	28.6	61.4	20.0	28.7	61.2	19.9
	135° F.				28.8	61.6	20.2	29.1	61.8	20.4
WSF 59	75° F.	29.4	63.8	20.7	29.0	63.1	20.6	29.4	63.2	20.7
	135° F.				30.8	63.0	22.6	31.8	62.6	23.4
WSF 70	75° F.	28.6	61.2	20.3	28.4	60.7	20.0	28.4	60.6	20.2
	135° F.				27.5	60.5	19.6	27.8	60.4	19.8

0007-SWP-045634

0007-SWP-000115006

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November, 1951

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187 - Physical Properties of Pigments

The paper on "Oil Absorption and Critical Pigment Volume Concentration" has been revised to include the latest concepts on the subject. It will be presented at the Louisville Production Club meeting on December 12.

0007-SWP-045635

0007-SWP-000115007

Paint Research
December, 1951

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A77 - Pigment Studies

A new and improved phthalocyanine blue laked pigment 24-R supplied by the C. P. Department has been placed under practical test for flooding characteristics and color stability. An Enameloid White Rex 710 sample has been tinted to the approximate shade of Enameloid Pastel Blue with a HL 44 type base prepared from 24-R. This is being aged in package at room and at 135° F. temperatures along with reference samples of Rex 710 that have been shaded to equal depths of blue with du Pont's HL 282-D and Harmon's Palomar Blue #73.

Films tinted with 24-R exhibited superior flood resistance in comparison with HL 282-D and #73 in draw-downs of the freshly prepared materials. Further examinations will be made after 2 weeks and 1, 2 and 3 month intervals.

0007-SWP-045636

0007-SWP-000115008