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#148 - Examination of White Lead

As a portion of our cooperative work with the Lead Industries Association, a study was made of the ease of mixing, dispersion, and grinding of various white pigments normally used in house paints, in a mixture of 60% (2) and 40% (48) (as used in the SWP vehicle).

For the first comparison, pastes were prepared with equal volumes of oil per volume of pigment. The pigment was added to the oil in the plastograph and allowed to mix for 20 minutes. The plasticity curves thus produced showed a peak plasticity reading at the time of final pigment addition, followed by a lowering to a nearly constant value at the end. The area under the curve should be proportional to the work done in mixing the paste, and was determined by counting the number of unit squares.

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Plastograph Mixed Pastes With Equal Volumes of Oil per Volume of Pigment

Pigment	Gallons Pigment	Gallons Oil	Plastograph at 20 Min.	Reading Peak	Curve Area
S-W Q. P. Basic Carbonate - 18 dry	100	175	55	55	80
Dutch Boy Dutch Basic Carbonate	"	"	65	160	150
Dutch Boy HTS Basic Carbonate	"	"	50	90	98
Dutch Boy 111 Basic Carbonate	"	"	85	1000	383
Sublimed Basic Sulphate - 9 dry	"	"	110	350	275
Ozlo 412 Leaded Zinc	"	"	105	215	127
Anatase Titanium Dioxide - 1403 dry	50	"	Too dry to mix	1000	-
Rutile Titanium Dioxide - 2173 dry	100	"	" " " "	1000	-
Magnesium Silicate 20 F dry	100	"	40	125	52

Precipitated basic carbonate white lead Dutch Boy 111 shows its characteristic poorer mixing properties compared with the usual types of carbonate. This is recognized by the producer. Anatase TiO_2 shows considerable resistance to mixing, as does rutile. There are indications, however, that rutile 2173 dry has better mixing qualities than anatase 1403 dry.

The next step was to compare the pigments on an equal plasticity basis, in preparation for grinding tests. By varying the amount of oil the plasticity was adjusted so that in all cases a reading of 100 units was obtained on the plastograph after 20 minutes. Before addition to the plastograph the pigment and oil were mixed together with a spatula, using

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the minimum amount of work necessary to obtain a homogeneous paste. These pastes of equal plasticity were passed through the 3-roll 8" Kent mill and the plasticity again determined. The production rate was noted, attempting to grind all pastes to the same fineness. The magnesium silicate was ground as fine as possible, while still maintaining a reasonable production rate.

As an approach to an estimate of the portion of the binder coming under the influence of the surface forces of the pigments, oil absorption values were determined using the standard A.S.T.M. method, using the 60/40 blend of raw linseed and Z_2-Z_3 bodied linseed. The gallons of "bound" oil were determined from these oil absorption values. "Total" oil is the total amount in the mixture. "Excess" oil is the total oil minus bound oil.

An assumption may be made that the bound oil clings to the pigment particles and does not contribute to a lowering of consistency as does the free oil. On this basis the volume of "free" oil was calculated per volume of pigment plus bound oil.

Example:

128.3	gallons	total oil	per	100	gallons	pigment
51.9	"	bound oil	"	"	"	"
76.4	"	free oil	"	"	"	"

$$\frac{76.4}{100.0 + 51.9} = 50.3 \text{ gallons free oil per 100 gallons pigment plus bound oil.}$$

See chart on next page.

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Pastes of Equal Pre-Grind Plasticity

Pigment	Plasticity: Before milling	Plasticity: After milling	Production Rate Gal. Pig. per Hr.	Fineness	Total Oil per 100 Gallons Pigment	Oil Absorption: Pounds per 100 Lbs.	Oil Absorption: Gals. per 100 Gals.	Free Oil: Gals. per 100 Gals. Pigment	Free Oil: Gals. per 100 Gals. Pig. plus Bound Oil
S-W Q. P. B.C.W.L.	105	40	.43	5-1/2	128.3	7.2	51.9	76.4	50.3
Dutch Boy Dutch B.C.W.L.	100	30	.30	4-3/4	138.8	6.9	49.8	89.0	59.4
Dutch Boy HTS B.C.W.L.	105	40	.39	4-3/4	127.7	6.6	47.6	80.1	54.3
Dutch Boy 111 B.C.W.L.	99	20	.15	4-3/4	268.8	11.9	75.0	193.8	110.7
Sublimed B.S.W.L.	100	23	.37	6-1/4	177.5	9.8	66.5	111.0	66.6
Ozlo 412 Leaded Zinc	101	25	.79	6-1/2	171.8	10.5	65.0	106.8	64.7
Anatase TiO_2	98	57	.25	6	300.7	30.8	127.1	173.6	76.2
Rutile TiO_2	100	40	.20	5-1/2	305.1	27.0	120.5	184.6	83.5
Magnesium Silicate	110	107	.45	2	131.2	20.9	63.5	67.7	41.4

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Two mixed pigment pastes were prepared to compare the calculated plasticity of mixtures of two or more pigments with the actual plasticity. The oil requirement of the mix was calculated on the oil required by the individual pigments to produce a 100 unit plasticity mix. Actual plasticities were found to be slightly lower than the predicted 100.

Paste Content	Plasticity Before Milling	Plasticity After Milling	Production Rate Gals. Pig./Hr.	Fineness
50 gal. 1403 dry 50 " 18 dry 214 " oil	90	39	0.20	5
460 lb. 412 dry 140 " 1403 200 " 18 200 " 20 F 45 " Oil	73	53	0.37	4

Pastes with Equal Volumes of Oil per Volume of Pigment

A further comparison was made using equal volumes of oil per volume of pigment. The paste was mixed on the pony mixer for 20 minutes, measured on the plastograph, milled, and measured again. In each case 100 gallons of pigment were used with 175 gallons of oil. Three zinc oxide samples are to be added to this list.

Pigment	Plasticity Before Milling	Plasticity After Milling
18 dry B.C.W.L.	35	22
Dutch Boy Dutch	43	20
" " HTS	35	22
" " 111	182	55
9 dry B.S.W.L.	70	33
412 Leaded Zinc	72	25
1403 TiO_2	650	205
2173 TiO_2	255	48
20 F Mg Silicate	32	32

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Sharp decreases in plasticity on grinding indicate incomplete wetting in the mixer. Magnesium silicate was thoroughly wetted in the mixer since no change appeared after application of further mechanical energy by grinding. S-W Q.P. White Lead and National's H.T.S. lead are obviously easy wetting. Anatase TiO_2 is difficult to wet, more so than rutile.

Acto 450 added to the above pigments and oil was very effective in increasing wetting in the case of the high consistency titanium oxide pastes, but had a negligible and variable effect when the original paste consistencies were low. This may indicate where wetting agents may be expected to be effective.

Work will continue on this project and will include studies of surface areas, consistencies at complete wetting, etc., to compare the mixing and grinding properties of such pigments. We hope to arrive at a measure to predict mixing and grinding properties of multiple pigment combinations including both active and inactive pigments.

As a further step in the Lead Industries Association research, blistering tests were run on various types of house paints to determine whether lead carbonate content was of any benefit in regaining adhesion after blistering had occurred and the blisters eventually had subsided. A uniform white pine board in two sections was painted with the selected paints as two coat systems (48 hours between coats) on the face and edges with the back unpainted and allowed to dry for one week. The panel sections were then mounted on the blistering cabinet (unpainted back down) with the cabinet run at 125° F. and saturated humidity. After 72 hours the panels were removed to permit the blisters which had formed to subside if they would. After drying for one week, the paint films were raked severely with a rough steel blade to determine the adhesion.

The paint systems used included:

Blistering - Adhesion Tests

Test No.		Materials Applied	Reduction Used Per Gallon	P.V.C.
1.	1st coat	L.I.A. House Paint OJ 436 J	1 pint raw linseed oil 1 pint turpentine	31.3%
	2nd coat	"	No reduction	36.4%

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Blistering - Adhesion Tests
Cont.

Test No.		Materials Applied	Reduction Used per Gallon	P.V.C.
2.	1st coat	SWP 450 Undercoater	No reduction	37.2%
	2nd coat	L.I.A. House Paint OJ 436 J	" "	36.4%
3.	1st coat	Lead-in-Oil	100# Soft Paste W.L. 4 gal. Replacement Oil 1-3/4 gal. turpentine 1 pint drier	29.1%
	2nd coat	L.I.A. House Paint OJ 436 J	No reduction	36.4%
4.	1st coat	Ti-Sil House Paint OJ 427 J	1 pint raw linseed oil 1 pint turpentine	25.3%
	2nd coat	"	No reduction	28.9%
5.	1st coat	Ti-Sil House Paint OJ 435 J	1 pint raw linseed oil 1 pint turpentine	31.3%
	2nd coat	"	No reduction	36.4%
6.	1st coat	Lead-in-Oil	100# Soft Paste W.L. 4 gal. Replacement Oil 1-3/4 gal. turpentine 1 pint drier	29.1%
	2nd coat	Lead-in-Oil	100# Soft Paste W.L. 3 gal. Replacement Oil 1 pint drier	33.2%
7.	1st coat	Rex 436 - SWP Fume Resisting White	1 pint raw linseed oil 1 pint turpentine	30.7%
	2nd coat	"	No reduction	36.3%
8.	1st coat	du Pont Moisture Res. H.P. OJ 34 J	1 pint turpentine	39.7%
	2nd coat	"	No reduction	39.7%

Undercoats at 450 sq. ft./gal. Top coats at 550 sq. ft./gal.

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Blistering - Adhesion Tests
Cont.

Test No.	Permeability Mg./Hr. Based on 24 Hr. Test	Blistering 24 Hours	30 Hours	72 Hours	Adhesion
1. 1st coat	2.5	None	Very slight	Bad	Fair -
2nd "	2.7				
2. 1st "	2.3	"	" "	Def. +	Fair
2nd "	2.7				
3. 1st "	2.9	"	Trace	Definite	Fair +
2nd "	2.7				
4. 1st "	2.1	Definite	Definite +	Bad	Poor
2nd "	2.3				
5. 1st "	1.9	"	"	"	Fair -
2nd "	2.0				
6. 1st "	2.9	None	None	None	Very good
2nd "	3.0				
7. 1st "	2.0	Bad	Very bad	V.V. bad	Very poor
2nd "	2.0				
8. 1st "	2.1	None	None	Slight	Very good
2nd "	1.9				

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It will be noted that while the permeabilities of the films follow the blistering tendencies to a degree, all the permeabilities are low and in the zone which normally is definitely below the "critical" permeabilities. However, there is an apparent relation between lead carbonate content and blistering and adhesion, except for the du Pont Moisture Resisting Paint, which contains no lead whatsoever. It is pigmented with TiO_2, ZZZ-33 zinc oxide and York Whiting in the vacuum bodied OKO 2-1/2 M linseed oil, at a p.v.c. of 40%. It is very effective in resisting blistering, but it must be kept in mind that this is still not an acceptable paint from a brushing standpoint.

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Otherwise two coats of lead-in-oil or lead-in-oil under the Lead Industries top coat (46% leaded zinc, 14% TiO_2, 20% basic carbonate white lead, 20% magnesium silicate) or 450 Undercoater (with 47% basic carbonate) under the Lead Industries Association paint, showed considerable resistance to blistering and retain adhesion. The lead-free Rex 436 and the Ti-Sil formulas (no carbonate, 17-1/2% basic sulfate white lead) show low resistance to blistering, and low adhesion at the end of the test.

Further tests must be made to take care of certain necessary improvements in technique but even the data collected so far is sufficient to stimulate some interesting speculation.

#177 - Pigment Studies

Following a method suggested by the Newark Technical Service, a series of pigments were evaluated as to dispersion properties for the Lead Industries Association. The Hoover Automatic Muller is used to perform measured amounts of grinding on pigment in oil mixtures. The degree of dispersion accomplished is measured on the Hegman Gauge.

The method used is as follows:

Weigh out 3 gms. of white lead carbonate or its approximate volume equivalent of other pigments. To this add sufficient oil to give a paste consistency similar to that accomplished with 3 gms. BCWL and 0.55 cc acid refined linseed oil, 13 A.V.

Mull this mixture with a 100# weight interrupting at each 50 revolutions for a pick-up and mixing of the paste.

To make a fineness reading, reduce the above mixture with 1 cc mineral spirits and test in the usual manner on the Hegman gauge.

	Mixture		Hegman Fineness				
	Wt. Pig.	cc Oil	100 Rev.	200 Rev.	300 Rev.	400 Rev.	500 Rev.
S-W Q.P. Basic Carbonate	3 gms.	0.55	4-3/4	5-1/2	6-1/2		
Sublimed Basic Sulfate	3	0.65	4*	5*	6-1/2*		
Dutch Boy Dutch	3	0.55	5-1/2	5-1/2	5-3/4	6	
Dutch Boy HTS Basic Carb.	3	0.55	4-1/2	5-1/2	6		
Dutch Boy 111	3	1.00	-**	-**	-**	-**	-**
Ozlo 412 Leaded Zinc	2.25	0.55	7	(150)	(200)		
Anatase Titanium Dioxide	1.50	1.00	5	(6-1/2)	(7)		
Rutile Titanium Dioxide	1.50	1.00	5-1/4	5-3/4	5-3/4	5-1/2	6
Magnesium Silicate	1.10	0.40		5	5-1/4	5-1/4	5-1/2

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*Bulk of material better than this rating.

**Persistent large particles; however, bulk of material is fine.

A few remarks are pertinent on the above. As conceded by the producer National's Precipitated White Lead 111 has a few large particles which are hard to grind, the remainder being fine particles. Ozlo Leaded Zinc reduces to its ultimate fineness very rapidly, as does anatase TiO_2. Rutile TiO_2 (2173 dry) is less easy to disperse. The regular lead carbonates are easy to disperse. Magnesium silicate is still 5-1/2 after 500 revolutions.

#97 - SWP 471

Lead-Zinc Substitutions in SWP

The raw material situation is such that it has become necessary to provide alternate formulas to use mechanical mixtures of lead carbonate and zinc oxide to displace the 412 dry co-fumed leaded zinc oxide normally used in SWP. It is pointed out that this substitution will affect durability adversely. The adverse effect is minimized by careful selection of the zinc oxide. Exposure studies at Coffeyville, Florida and Chicago indicate the following zinc oxides to be preferred for durability.

2059	dry	New Jersey XX-503
2295	"	Azo ZZZ-33
2143	"	Azo ZZZ-11
-		Azo ZZZ-55
1707	"	New Jersey XX-50

Of the above group, 2143 dry - Azo ZZZ-11 gives too high a consistency for practical formulation and 2295 dry - Azo ZZZ-33 is on the borderline of high consistency after removing all water from the formula.

New Jersey's XX-503 and Azo ZZZ-55 appear the most likely substitution zinc oxides. 1707 dry, XX-50 should be used only as a last resort, since its durability is quite inferior to the other alternates given. All of the above substitutions are made in combination with basic carbonate white lead to replace the 412 dry on a chemical equivalent basis.