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Report No. R-62-33

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Research Report

LOW COST, FAST DRYING MAINTENANCE PRIMERS

Date Issued	:	4/23/62
Period Covered	:	6/6/61 - 3/13/62
Project No.	:	Unassigned
Previous Reports	:	R-62-16
Notebook Nos.	:	7444

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LOW COST, FAST DRYING MAINTENANCE PRIMERS

INTRODUCTION:

It is generally held that the vehicle in a good metal primer is one that is able to wet and penetrate rust and other surface fissures on the metal substrate and that this property is probably limited to low viscosity vehicles. For example, "Antoxide" 373-844, a low cost metal primer featuring barium lanolate as a sulfate scavenger, is based on slow drying, soft linseed oil. Here we have good penetration and low cost, but slow dry. V54 Primer 7067-5001 is based on low viscosity esters of vinyl dioxolane. With this product we have good penetration and fast dry, but high cost.

Our goal is good penetration and both fast dry and low cost. In a recent research report, R62-16, it was suggested that if the pigments in a primer system are flocculated, even a viscous vehicle may wet and penetrate a porous substrate sufficiently. On checking this hypothesis, we found several vehicle-pigment combinations which might provide a basis for low cost, fast drying, penetrating primers. Saleable products may have already evolved, but because so little time was expended on formulation and testing, additional work on formulation is likely to be fruitful. This report outlines the work already completed.

OBJECTIVE:

To formulate a line of low cost, fast drying, penetrating primers for thick film application over clean or slightly rusted steel. It is hoped that this paint will also be suitable for mill scaled plus partially rusty mill scaled steel with minimum preparation.

SUMMARY AND CONCLUSIONS:

1. A low cost pigment system - 5% lamp black, 20% zinc oxide and 75% talc - dispersed in several vehicle systems provided primers which penetrated porous surfaces and protected clean and lightly rusted steel substrates in accelerated corrosion tests.

2. The preferred vehicle system was 40% Aroplaz 6006 (ADM's short oil alkyd) and 60% V54 linoleate. Other systems meriting attention include a copolymer of V54 linoleate and vinyl toluene, a blend of V54 linoleate with a hydrocarbon resin and a blend of styrenated oil and linseed oil.

3. Assuming intermediate costs of 46¢ and 30¢ per lb. for V54 linoleate, the mill cost of a primer based on V54 linoleate and Aroplaz 6006 will be $3.20 and $2.84 per gallon, the mill cost of a primer based on V54 linoleate and a hydrocarbon resin will be $2.40 and $2.10 per gallon. On the same basis of calculation, the mill cost of Antoxide 373-844 is $2.43 per gallon and V54 Primer 7067-5001 is $5.07 per gallon assuming a cost of 44¢ a lb. for the mixed esters.

4. No advantage was found for utilizing barium lanolate or other barium salt in the above primer systems.

5. A limited number of experiments involving changes of colorants, pigment volume, choice of vehicle, additives and several studies of drying as a function of film thickness are reported. These may provide a basis for future studies.

ACTION TAKEN:

A Research Backlog Proposal was submitted. The information in this report was communicated to the V54 Marketing Specialists and to research personnel working on V54 maintenance primers. Several experimental formulations were exposed Oct., 1961 in a Florida paper mill by the V54 Marketing Group. Other formulations were exposed March, 1962 on the roof of the #231 Building in Philadelphia. Arrangements were made with J. Ansul to include two experimental primers in a field test scheduled at the Chambers Works.

PATENT SITUATION:

Patent proposal FFD-1732 relates to V54 type monoesters and their uses. The data mentioned in this report will be considered in preparing the patent application. Product patents will be considered if commercial or technical interests develop.

PUBLICATIONS:

None contemplated.

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PUBLICATIONS:

None contemplated.

DISCUSSION:

As part of Project P2700-D, an effort was made to improve the dry and hardness of "Antoxide"* 373-844 by modification with V54 diesters. This attempt was unsuccessful, but in the course of this work, a number of interesting observations on the interrelationship of penetration, pigment flocculation and corrosion resistance were made (1). As a result we redirected our efforts and attempted on an unassigned basis to develop low-cost, fast drying, penetrating primers. In so doing, we used the "Antoxide" as the control because (1) the "Antoxide" paint functions well over rusty steel with respect to penetration and corrosion resistance; (2) the inadequacy of "Antoxide" is primarily in its slow drying, soft vehicle combination (80:20 linseed oil: long oil alkyd RC-141); and (3) the pigmentation** is cheap and effective. An effort was therefore made to improve the dry and hardness of "Antoxide" primarily by vehicle modification.

Good primer performance has long been associated with an ability to wet and penetrate porous substrates. However, the minimum penetration required for a given paint on a given substrate has never been established. Red Lead Primer 67-800, "Antoxide" 373-844, V54 Primer 7067-5001 and V54 Shop Primer 7501-W5001, possessed equivalent wetting power, as measured by placing one drop of paint on #1 Whatman filter paper and observing the diameter of the diffused ring. When these paints were diluted (2 grams paint and 8 grams solvent), the pigments settled rapidly leaving a clear supernatant liquid. It was noted that those primers which failed to penetrate the filter paper remained suspended when diluted. In salt spray testing using similar primers, the primers that penetrated appeared to possess better corrosion resistance over slightly rusted steel than those which failed to penetrate. Our corrosion substrates consisted of lightly rusted polished SAE-1010 steel ("Q" panels), Kenneth Tator test panels*** or rusted autobody steel. The paints were air dried one week, cross cut, and exposed two weeks in salt spray.

*Registered Du Pont Trademark

**W-225	- lamp black	- 4.8 wt. %
W-78	- zinc oxide	-15.1 " "
G-383	- barium lanolate	- 5.0 " "
W-132	- Talc	-75.1 " "

***Hot rolled carbon steel panels simulating welded steel construction.

The initial work was described in Research Report R62-16. In that report, an assumption was made that pigments in penetrating primers were flocculated and that this condition allowed the vehicle to penetrate porous substrates. It was further assumed that the extent of spread is related to the fluidity of the vehicle and to the time that the vehicle remains fluid. Without extensive testing, we acted on these assumptions to formulate primers with fast setting and high viscosity components. Several vehicle combinations were found which with "Antoxide" pigmentation, exhibited in accelerated tests evidence of rust penetration and an encouraging degree of protection to rusted substrates. These involve (1) combinations of several vinyl toluene copolymers with linseed oil, (2) a combination of V54 linoleate and Aroplaz 6006*, and a (3) copolymer of V54 linoleate and vinyl toluene.

EXPERIMENTAL:

The experimental work is treated in three categories - pigment studies, vehicle studies and paint performance.

Because the program was carried on sporadically over an extended period of time, the paint tests were changed from time to time. As a consequence, results from series to series cannot always be rigorously correlated. Generally, paints were adjusted to a viscosity of 83 KU, tested for dry on the RCI drying time recorder at film drawdowns of 3 and 6 mils, checked for penetration and sedimentation rates, brushed on clean and rusted steel panels, air dried one week, scratched and exposed 2 weeks to salt spray. At all times, "Antoxide" 373-844 was used as control; and when a paint is rated 'corrosion-resistant', it means that it clearly outperformed the "Antoxide" control. As progress was made, other primers such as 67-800 Red Lead Primer and 7067-5001A V54 Primer, were used as controls, drying tests were made at low and high temperatures at various film thicknesses and intercoat adhesion studies were investigated.

A. Pigment Studies

The basic pigmentation of "Antoxide" is

		Wt. %
W-225	Lamp Black	4.8
W-78	Zinc Oxide	15.1
W-132	Talc	75.1
G-383	Barium Lanolate	5.0

In the course of this program, a number of changes were made.

*Aroplaz 6006, ADM's fast drying short oil soya alkyd, 50% in xylene.

1. Barium Salts

"Antoxide" is Du Pont's registered trademark for a class of low cost metal primer systems featuring barium lanolate as a sulfate scavenger (2). Several new barium salts were recently offered for sale by Food Machinery and Chemical Corp. - barium dihydrogen cyanurate, barium hydrogen cyanurate, barium benzene sulfonate, barium sulfostearate and barium phytate. Curious to determine the effect of replacing barium lanolate, these new salts were used in a group of paints based on "Antoxide" 373-861*. On the basis of drying, penetration, sedimentation and salt spray resistance, these formulations (3) appeared equivalent, with perhaps a slight edge in favor of barium phytate.

The barium phytate was rechecked in the slower drying "Antoxide" 373-844. As an additional control, a paint was made without any barium salt (4). The two paints containing the barium lanolate and barium phytate were about equal; but surprisingly, the barium free version appeared most resistant to two weeks salt spray exposure. This observation was rechecked in a vinyl toluene oil modified formulation (5) and again the barium free formulation proved superior in salt spray resistance. (Judgment is based on visual estimation of creepage from cross cut, blistering, if any, and film continuity.) Other properties, e.g. dry, penetration and sedimentation seemed independent of barium content.

On the basis of these observations, barium lanolate was eliminated and the following pigmentation was adopted for future studies:

			Wt. %
W-275	Lamp Black	-	5
W-78	Zinc Oxide	-	20
W-132	Talc	-	75

2. Colored and Inhibiting Pigments

Several paints were made (6) in which the black pigment was (a) merely eliminated, (b) replaced by iron oxide, W-878, and (c) replaced by red lead, W-349. In a V54 linoleate-Aroplaz 6006 vehicle, all behaved similarly and equivalently to a black control. This suggests that primers may be formulated with a variety of colorants or rust inhibiting pigments, if the latter can be justified.

*373-861, a faster drying version of 373-844 with a medium oil alkyd, RC-311, replacing RC-141.

3. Zinc Oxide

An effort was made to judge the role of zinc oxide in "Antoxide" pigmentation and to evaluate W-19, a low oil absorbing ZnO, and W-78, a fine particle ZnO. In a V54 linoleate-Aroplaz 6006 blend (7) and at 33.3% PVC, the W-19 level was set at 27 weight %, 22.3 weight % and 16.2 weight %. After 2 weeks in salt spray, all three films behaved equivalently. This experiment suggests that a more favorable cost-property balance may be achieved by reduction of ZnO content.

In a linseed oil-Keltrol 1001* formulation (8) a W-78 Zinc Oxide pigmentation seemed more flocculated and dried faster than an equivalent W-19 Zinc Oxide formulation. In a V54 linoleate/vinyl toluene copolymer (9), the W-78 formulation was slightly better in corrosion resistance. In a V54 linoleate-Aroplaz 6006 formulation (10), the W-19 version dried faster but possessed equal corrosion resistance to the W-78 version. The only consistent difference was that the W-19 version needed more thinner. In general, the differences between W-19 and W-78 are marginal and usage should probably ultimately depend on cost and production conveniences.

4. PVC

Because the pigment volume concentration (PVC) of "Antoxide" 373-844 is 33.6%, many of our studies were also in the 33% PVC range. However, a group of V54 linoleate-Aroplaz 6006 paints were formulated at 30, 33, and 36% PVC (10). All withstood 2 weeks salt spray, but the 36% version offered the advantage of better 45°F dry and better penetration. A similar study was made of the Keltrol 1001-V54 linoleate system (8). In this series, the 36% PVC version had the best wetting properties and performed best in the salt spray. This study indicates that there may be an optimum PVC for each paint system.

B. Vehicle Studies

The vehicles studied have been limited to several combinations of a hard viscous material and a low viscosity oil. The hard resins include Keltrol 1001, several vinyl toluene copolymers of drying oils, Aroplaz 6006 and a heat reactive hydrocarbon, Piccodiene 2215 (Penna. Ind. Chem. Corp.). The oils were limited to linseed oil and V54 linoleate. These were used because they were available and low in cost. Obviously, the choice of vehicles could be extended. In this section, those properties which may be influenced primarily by the vehicle will be discussed.

*Keltrol 1001 - Spencer Kellogg's vinyl toluene oil copolymer, 60% in mineral spirits.

1. Keltrol 1001-Linseed Oil

Before adopting Keltrol 1001, several other commercial oils were evaluated (11). Keltrol 1001 was chosen because its mineral spirits solvent led to easier brushing. Other varieties may be chosen for spray application. Several laboratory styrenated oils were also tested (1), but these did not dry fast enough to justify replacement of "Antoxide." Some questions with respect to the use of Keltrol 1001 remain because Keltrol 1001 proved to be package unstable (5).

Most of the work was based on 40 parts Keltrol 1001 and 60 parts linseed oil, H-253. This ratio was arrived at empirically as the most favorable balance of dry and penetration.

2. Keltrol 1001 - V54 linoleate

Comparing two primers, one at 60/40 and the other at 50/50 levels of V54 linoleate/Keltrol 1001 (12), the 60/40 primer wetted better and demonstrated better corrosion resistance. Comparing V54 linoleate with linseed oil at a 60/40 oil/Keltrol 1001 primer, the use of V54 linoleate seemed beneficial. Very little work was done with this system because the alkyd Aroplaz 6006 proved to be better in corrosion resistance.

3. Aroplaz 6006 - V54 linoleate

Primers based on the use of a 60/40 blend of V54 linoleate and Aroplaz 6006 were better than those based on a 50/50 ratio. Other properties such as dry, hardness and penetration were within a commercial range (13) (6) (10) (7) (14).

4. Hydrocarbons - V54 linoleate

A number of hydrocarbon resins - Nevindene 1,[a] RBH 510,[b] Piccodiene 2215, RK-662[c] and RCW-53225[d] - were compatible with V54 linoleate (15). In many instances, the V54 linoleate improved both dry and hardness. Of some interest is Piccodiene 2215 because it sells for only 11¢/lb. Good corrosion resistance was obtained with a 60/40 V54 linoleate/Piccodiene 2215 formulation (16).

5. Copolymers of V54 linoleate and vinyl toluene

Several vinyl toluene copolymers of linseed oil, recently described (17), were used in "Antoxide" formulations (18). These products dried rapidly and performed well in salt spray. In

(a) Nevindene 1 - Neville Chemical Co. - Coumarone Resin
(b) RBH-510 - Interchemical fossil hydrocarbon resin
(c) RK-662 - "Budium"
(d) RCW-53225 - Copolymer α-methyl styrene and butadiene

another experiment (9), the copolymer proved to be less corrosion resistant than the Aroplaz 6006 - V54 linoleate primer. It was found that these copolymers possessed poor adhesion to steel (19).

C. Paint Performance

In this section, a number of miscellaneous factors will be considered and the formulae and properties of several prime candidates will be recorded. These formulae are suggested as starting points for future studies and field tests.

The primers in this study were made by first dispersing the pigments in the linseed oil or V54 linoleate via roller mill grinding and then adding the hard viscous resin. This yielded a product with some degree of penetration (17). Grinding in the viscous resin generally led to better suspension and poorer penetration (20).

1. Flocculants

In our previous report (1) a number of flocculants were listed. But because good flocculation and penetration were finally achieved without a flocculant, no systematic effort was made to find specific reagents for each system studied. On the basis of the early work, it is evident that a wide variety of chemicals can be used whenever needed.

2. Driers, Accelerators and Inhibitors

The V54 linoleate blends were treated with 0.1% cobalt and 1.0% lead as naphthenates. In all cases studied, the paints proved package stable, non-skinning and non-wrinkling. The Keltrol 1001 linseed oil blends were treated with 1.0% lead, 0.025% cobalt and 0.1% manganese, but even at this low cobalt level, skinning and wrinkling often occurred. In addition to service as an auxiliary drier, the lead naphthenate also functions as a wetting agent. No effort was made to seek an optimum drier level nor were any efforts made to utilize auxiliary agents.

3. Drying Behavior

In general a 3 mil drawdown of a V54 linoleate product dried hard in about 8 hours at 77°F. and 50% RH. At 45°F., thin films dried overnight whereas thicker films (4 mil drawdown and higher) remained tacky for several days. When removed to room temperature, the films dried within 8 hours. At 140°F., the films hardened without wrinkling (21). The Keltrol 1001-H-253 is more temperature sensitive; at 45°F., the films remained soft; at 140°F., the films wrinkled. After 48 hours bake at 105°C, the Keltrol modification was more elastic than the "Antoxide" control. The overall good behavior of V54 linoleate merits consideration.

4. Effect of Film Thickness

A group of primers - V54 primer 7067-5001, "Antoxide" 373-844, Red Lead primer 67-800, alkyd enamel 25-415 were evaluated together with a group of experimental paints (22) at 3 levels of film thickness over clean and rusted steel and over Kenneth Tator panels. The film thicknesses (dry) were controlled at 3, 6 and 9 mils by spraying. In all cases, the 3 mil films failed to protect the steel from salt spray corrosion whereas the 9 mil films were generally excellent. The "Antoxide", red lead, alkyd and the Keltrol 1001-H-253 wrinkled at 9 mils. The best primer in this series, on all counts, was the V54 linoleate-Aroplaz 6006 formulation.

5. Intercoat Adhesion

Several topcoats were applied over the experimental and control primers (23). These included Epoxy 823-8021, phenolic 353-922, alkyd 25-415, #50 House Paint, sign enamel 802-003 and vinyl 6201. Except for the vinyl, the air dried topcoats adhered to the primers, improving as the films aged. If baked, the vinyl also adhered to the primers. On this basis, no unusual difficulty in intercoat adhesion is anticipated.

6. Exposures

Experimental primers W40748C (a Keltrol 1001-linseed oil), W40749A (styrenated oil-linseed oil), W40756A (vinyl toluene-V54 linoleate copolymer) and W40757B (Keltrol 1001-V54 linoleate) were exposed by J. Ansul in a paper mill in Florida in October 1961.

Kenneth Tator panels were painted with W40764E (Keltrol 1001-linseed oil), W40766F (V54 linoleate-Aroplaz 6006), W40773F (Piccodiene 2215 - V54 linoleate), 373-844 "Antoxide", 67-800 Red Lead Primer and 7067-5001A V54 Primer). These are aging on the roof of the #231 Building in Philadelphia.

7. Representative Formulations

"Antoxide" 373-844

		Weight
H-287	Mineral spirits	52.0
G-383	Barium lanolate	32.1
H-253	Linseed oil	218.5
H-624	24% Lead naphthenate	19.8
RC-141	Long oil alkyd soln.	105.0
W-132	Talc	475.0
W-78	Zinc oxide	95.5
501-205 (W-225	Lamp black	70.4
501-205 (H-253	Linseed oil	121.6
H-173	6% Manganese naphthenate	5.0
VH-7552	Inhibitor soln.	5.0
H-287	Mineral spirits	29.0
		1188.9

PVC = 33.6%

H-253/RC-141 = 80/20

Initial viscosity = 83-87 KU

Solids = 91.5%

Drying rate - 1 mil film - 77°F and 50% RH - soft 24 hours

- 1 mil film - 45°F - wet

W40766F V54 linoleate - Aroplaz 6006

		Weight
-	V54 Linoleate	324.0
H-624	24% Lead naphthenate	22.5
W-225	Lamp black	38.8
W-19	Zinc oxide	307.0
W-132	Talc	620.0
H-200	Aromatic solvent	50.0
G-1136	Aroplaz 6006	430.0
H-172	6% Cobalt naphthenate	9.0
H-200	Aliphatic hydrocarbon	45.0
		1846.3

PVC = 36%

V54 linoleate/Aroplaz 6006 = 60/40

Initial viscosity = 83 KU

Solids = 83%

Drying Rate - 1 mil film - 77°F and 50% RH -7 hours

3 mil " " " " " -overnight

1 mil " 45°F - overnight

3 mil " 45°F - sl. tacky overnight

W40772D-Piccodiene 2215 - V54 linoleate

		Weight
-	V54 Linoleate	117
W-78	Zinc oxide	127
W-225	Lamp black	30
W-132	Talc	474
W-287	Aliphatic hydrocarbon	173
-	75% Piccodiene 2215 in H-287	190
H-624	24% Lead naphthenate	14.8
H-172	6% Cobalt naphthenate	1.5
H-173	6% Manganese naphthenate	5.9
		1133.2

PVC = 36%

V54 Linoleate/Piccodiene = 45/55

Initial viscosity = 82 KU

Solids = 80%

Drying Rate - 1 mil film - 77°F + 50% RH - 16 hours

W40760 V54 linoleate/vinyl toluene copolymer

			Weight
W40758A -	1:1 copolymer weight basis V54 linoleate - vinyl toluene	-	142
W-78	Zinc oxide	-	127
W-225	Lamp black	-	30
W-132	Talc	-	474
W40758A	Copolymer	-	285
H-624	24% Lead naphthenate	-	17.9
H-172	6% Cobalt naphthenate	-	7.0
H-287	Aliphatic hydrocarbon	-	250
			1332.9

PVC = 32.6%

Initial viscosity = 83 KU

Solids = 81.1%

Drying Rate - 1 mil film - 77°F and 50% RH - 7.5 hours

3 mil " - " " " "-overnight

1 mil " - 45°F -tacky overnight

W40764E - Keltrol 1001 - Linseed Oil

		Weight
H-253	Linseed oil	117
W-19	Zinc oxide	127
W-225	Lamp black	30
W-132	Talc	474
H-287	Aliphatic hydrocarbon	80
H-253	Linseed oil	95
-	Keltrol 1001	237
H-624	24% Lead naphthenate	14.8
H-172	6% Cobalt naphthenate	1.5
H-173	6% Manganese naphthenate	5.9
H-287	Aliphatic hydrocarbon	40
		1222.2

PVC = 36%

Keltrol 1001/H-253 = 40/60

Initial viscosity = 82 KU

Solids = 83%

Drying Rate: 1 mil film - 77°F and 50% R.H.=8 hours

3 mil " - " " " "-overnight

1 mil " - 45°F - tacky overnight

8. Economics

The cost of the preferred experimental primers is dependent on the cost of V54 linoleate. With V54 alcohol purchased at 48¢ lb. and at a manufacturing volume of 1 million lb. per year, it is estimated that the mill cost of V54 linoleate will be 46¢ lb. (24). Using this cost and one of 30¢ lb., the mill costs in 5 gallon cans of a V54 linoleate-Piccodiene 2215 and a V54 linoleate-Aroplaz 6006 primers were calculated (26). These were contrasted to the current mill cost of "Antoxide" 373-844 and V54 Primer 7067-5001.

	Mill Cost in 5 Gallon Containers
"Antoxide" 373-844	$2.43
V54 Primer 7067-5001	$5.07 assuming V54 esters at 44¢/lb.
W40776A V54 Linoleate-Aroplaz 6006	$3.20 for 46¢ V54L
	$2.84 for 30¢ V54L
W40776B V54 Linoleate-Piccodiene 2215	$2.40 for 46¢ V54L
	$2.10 for 30¢ V54L

9. Advantages and Disadvantages

The following table summarizes as far as we know certain advantages and disadvantages of several of the primers discussed in this report.

	V54L-Aroplaz 6006 W40766F	V54L Piccodiene 2215 W40772D	"Antoxide" 373-844	V54 7067-5001
Drying Behavior	6-8 hours	16-24 hours	over 24 hr.	4-8 hours
Film hardness-24 hours	moderately hard	medium	soft	hard
% Solids	83	80	91	95
Cost	2.84 - 3.20	2.10 - 2.40	2.43	5.07
Topcoat adhesion	OK	OK	OK	OK
Wetting & penetration	moderate	good	excellent	excellent
Corrosion resistance	OK 2 weeks salt spray	OK-2 weeks salt spray	Commercially acceptable	2 weeks salt spray

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