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T E C H N I C A L O P E R A T I O N S

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Series No.54.

VISCOSITY CONTROL

(C.12.)

by

J. RICKMAN.

Viscosity Measurements Using the
Brookfield Viscosimeter.

The Brookfield Synchro-Electric Viscosimeter is an electrically driven instrument which measures viscosity in terms of the drag produced on a spindle rotated in the material under test.

The spindle is driven by a synchronous motor, the dial of the instrument also revolves and is driven directly from the motor, whereas the drive to the spindle is transmitted through a spiral spring. The spring absorbs the drag produced on the spindle by the material under test. The distance by which the spindle lags behind the dial is communicated to a pointer which indicates this distance on the dial. Dial readings can be converted to viscosity measurements in absolute units.

The dial may be rotated at four different speeds, speed changes being made whilst the motor is running.

There are seven interchangeable spindles of different sizes, to cover the measurement of viscosities from $\frac{1}{2}$ - 20,000 poises.

The viscosities of eight materials were measured at 25° C. using

(a) Gardner Bubble Tubes

(b) The Brookfield Viscosimeter with No. 3 Spindle.

Materials and results are tabulated below :-

MATERIAL	VISCOSITY IN POISES AT 25° C.		
	Brookfield	Bubble-Tube	Effortless
Lubricating Oil	1.6	1.6	-
Stand Oil (39)	3.7	2.7	-
SA.5591	5.4	5.0	0.2
Glycerine	5.7	5.0	0.7
Stand Oil (Made in Lab.)	20.0	approx. 10	approx. 0.2
Cylinder Oil	25.5	18.7	0.6
V.3082	27.8	approx. 20	approx. 0.4
Stand Oil (45)	28.0	approx. 20	approx. 1.

VISCOSITY CONTROL (C.12)
(Continued)

Over this viscosity range results by the two methods show close similarity. Where there is disparity the Bubble Tube Method is more probably at fault because

- (1) When the rate of rise of the bubble in the material under test does not exactly coincide with that of one of the standards, the reported figure can only be an approximation.
- (2) Variation in diameter of the empty comparison tubes causes error.

Determination of effect of varying the motor speed using the No.1 Spindle.

Materials used :-

Lubricating Oil, Glycerine and Stand Oil (89).

These materials gave readings at two or more motor speeds.

MATERIAL	R.P.M.	Reading	Reading converted to 20 r.p.m.	Reading converted to poises.
Lubricating Oil	2	16	160	1.6
	4	32	160	1.6
	10	80	160	1.6
	20	160	-	1.6
Glycerine	4	112	560	5.6
	10	287	574	5.7
Stand Oil (89)	4	54	270	2.7
	10	132	268	2.7
	20	272	-	2.7

Conclusion:

When in any material two or more motor speeds give satisfactory readings without changing the spindle, it is immaterial which speed is used as the viscosity figures calculated from the readings are virtually identical.

Viscosity of Texture Paint. 4.L.1891.

Spindle No.6.

Temperature 80 F.

Motor Speed	1st reading	2nd reading
4 r.p.m.	238	230
2 r.p.m.	122	120

Mean reading at 4 r.p.m. = 234 = 1170 poises.

cont.

VISCOSITY CONTROL

(C.12)

(Continued)

Viscosity of Lino Adhesive B.4950.

Spindle No.5	1st reading	400
Temp. 85° F.	2nd reading	400
R.P.M. 10		
	=	320 poises

Viscosity of Pigmented Emulsion Paste Cleaner (Formulation now different).

This material did not appear to be homogeneous and there was a tendency for liquid to separate after stirring. Successive readings showed lack of agreement. Sometimes the pointer would remain steady for several revolutions and then rapidly jump to a new position. Normally the pointer reaches the stable position without any jerkiness.

The following readings were obtained using spindle No.6 at 4 r.p.m. at room temperature (60° F.) :-

182, 176, 154, 180, 176, 178 and 180

Mean of 1st, 2nd, 4th, 5th and 6th readings

= 178 = 890 poises

Preparation of Stand Oil.

4 gallons of (2) in a 5-gallon pot was brought to 560° F. and held for several hours. Periodic viscosity determinations were made immersing the Brookfield Viscosimeter in the hot oil. After each determination a small sample of oil was withdrawn and the viscosity determined by the Bubble Tube method.

The readings from the Brookfield were obtained using No.1 Spindle at 20 r.p.m.. This covers the lowest viscosity range viz. 0.5 p. - 5 p. Readings below 0.5 p are stated to be unreliable. All the readings were below this limit, and therefore unreliable.

The final viscosity of this stand oil at 25° C. by Bubble Tube was 12.9 p. A 27 p. stand oil (the stoutest available) was heated to 560° F. and its viscosity measured by the Brookfield. The viscosity was 0.17 p., again well below the accuracy limit of the machine.

contd.

VISCOSITY CONTROL
(Continued)

(C.12)

Time	Temperature	Brookfield Reading	Brookfield Poises	Bubble Tube @ 25° C.
2 p.m.	560° F.	2.0	0.1	2.65
2.40 "	560° F.	2.0	0.1	5.0
3.0 "	560° F.	2.25	0.11	6.3
3.30 "	568° F.	2.25	0.11	8.8
4.0 "	560° F.	2.5	0.12	10.8
4.30 "	560° F.	2.5	0.12	12.9
-	560° F.	3.5	0.17	27.0

When the Brookfield Viscosimeter had been immersed in Stand Oil at 560° F. the adhering film of Stand Oil present on removal of the instrument set practically immediately and was most difficult to remove. The spindle and calibration sleeve were given a rough cleaning after each immersion. At the end of the experiment the baked film on these and the side arms was extremely hard and could not be removed with paint remover. The film was eventually softened by refluxing with methylene chloride for eight hours.

Conclusion.

The instrument is unsuitable for use in hot stand oil varnish because

- 1) of the trouble with baked films described above.
- 2) the low viscosity of bodied materials at the temperature of cook is usually below the range of the instrument.

Preparation of Alkyd (S.A.5142)

RM.731	504	gms.
(368)	250	"
(350)	455	"
(458)	171	"

The 731, (368) and (350) were heated to 500° F. and held for 2½ hours when the experiment was brought to an end by an electricity cut. The specified works cooking time is 3 hours but even in the limited time

contd.

VISCOSITY CONTROL
(Continued)

(C.12)

the alkyd was over bodied. Periodic viscosity measurements were made at the cooking temperature using the Brookfield viscosimeter with Spindle No.1 at 20 r.p.m.. All readings except the final one (when the alkyd was over-bodied) were below the accurate range of the instrument. Nevertheless, when viscosities were plotted against time a smooth curve was obtained.

Time	Brookfield Reading at 500° F.	Reading Converted to poises.
1 hr. 10 mins.	4.25	0.21
1 hr. 28 "	5.0	0.25
1 hr. 45 "	6.0	0.30
2 hrs.20 "	9.75	0.48
2 hrs.45 "	15.0	0.75

After immersion in the alkyd at 500° F. the adherent film of liquid on the instrument set hard immediately and was most difficult to remove. The spindle and calibration shield were roughly cleaned after the first immersion. They were not cleaned again until after the third immersion by which time a thick film had been deposited. Most of this was removed prior to the fourth immersion. The fourth and fifth readings were taken without an intermediate cleaning. After the fifth reading the calibration sleeve had a hard layer of alkyd which was only removed with great difficulty after standing in (458) overnight. Alkyd had built up on the spindle, and on the rim had a thickness of about 1/2". This deposit was most difficult to remove even after standing overnight immersed in solvent.

Conclusions:

The Brookfield Viscosimeter provides a rapid and accurate method of determining the viscosity of varnishes, stand oils, lubricating oils, texture paint and lino adhesive in absolute units. Other materials for which it could be used include Brunswick Blacks, aromatic Petroleum residues and paints over a wide range of viscosities. For normal varnishes the instrument is less convenient than the Bubble Tube as a much greater volume of liquid, namely, 600 c.c. must be brought to standard temperature.

The instrument is not suitable for controlling varnish cooks etc. because :-

- (1) The instrument lacks accuracy at viscosities below 1 p.l.u.

contd

VISCOSITY CONTROL
(Continued)

(C.12.)

- (2) The material under test rapidly bakes hard on the immersed parts of the instrument.

The instrument is sufficiently robust for careful use in the laboratory but is too delicate for use on the works.

CELLULOSE SURFACES AND STOPPERS (C.13.)

by

W. P. KEMP.

As it was desired to release for E.S. Enamels supplies of Cellosolve, at present fully used in our primer surfacers, alternative formulas not requiring cellosolve were tested. The classes compared were :-

- (A) Standard 4G.44, 45, 46, based on Shellac and Blown Castor Oil.
- (B) Sobac Formula 4G.9001, based on Ester Gum and Dibutyl Phthalate.
- (C) "Glasco" Type, similar to 4G.117 Stopper, based on Backacite 100 Pale and Treated Linseed Oil.
- (D) 1W.1321 type based on Ester and Damar and D.B.P. + Castor Oil with some Selenium-treated Tung Oil.

Types (B) and (D) were discarded as being considerably poorer than standard for adhesion, but Type (C) was very promising. When tested alone they appeared slightly brittle, and so an extra 2% of T.L.O. was added. The unaltered material was, however, better under enamel, being equal to standard after 1-2 days drying.

After 3-4 weeks ageing, however, both the unaltered and altered formulas were too brittle, and it was found after extended tests that an additional 8% of T.L.O. was required to give satisfactory elasticity & ageing, but this made the material too soft for satisfactory flattening when freshly applied. To overcome this difficulty further tests are in hand using maleic type resins in place of the Backacite 100 Pale when it is hoped to combine the good hardness and adhesion with satisfactory solvent release.

NITROCELLULOSE AIRCRAFT FINISHES. (C.20.)

by

C.A. WEBB.

Some experimental work has been done to determine the cause of the precipitation and coagulation which has been occurring with D.T.D.754

contd.

NITROCELLULOSE AIRCRAFT FINISHES
(Continued)

(C.20.)

Camouflage Finishes on ageing. This trouble has been experienced throughout the range.

It was thought that it was probably caused by the action of lactic acid, formed by the hydrolysis of the ethyl lactate in the solvent base, on reactive pigments in the finishes. White was the most severely affected. This is made on Zinc Oxide and it is known that this pigment is prone to coagulation if the pH value of the medium is too low, as it would be in this case if lactic acid were being liberated. Another pigment which might react with the lactic acid and which is common to the whole range is Zinc Stearate.

Several series of experimental mixings were made to determine whether our theory as to the cause of the trouble was correct. One major difficulty was that the coagulation takes several months to develop and it was necessary for us to obtain, speedy results. We therefore tried a number of methods to accelerate the reaction. These included the incorporation of various quantities of lactic acid, the addition of large quantities of ethyl lactate and in one case the addition of water to speed the hydrolysis of the ethyl lactate in the solvent base.

Four series were made, each on a different pigment.

<u>I</u>	Zinc Oxide
<u>II</u>	Lead Chromate
<u>III</u>	Zinc Stearate
<u>IV</u>	Titanium Oxide.

The titanium oxide series was introduced as a control because this is an inert substance and would confirm whether the pigments were taking any part in the reaction.

Tabulated on the following pages are the details of the finishes and the observations made.

contd.

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Series No. 11

NITROCELLULOSE AIRCRAFT FINISHES (G.20.)
(Continued)

Series I Pigmented with Zinc Oxide.

	Initial Observation	Over- night	1 week	1 month	2 months
Standard (4% (455)) Solvent Base	O.K.	O.K.	O.K.	O.K.	O.K.
A. Contains 20% (455)	O.K.	O.K.	O.K.	O.K.	Coagulation commencing
B. 30% (455) and 5% Lactic Acid	Immediate Coagulation				
C. 20% (455) - 3 1/2% water	O.K.	O.K.	O.K.	Slight signs coagula- tion	Coagulation commencing
D. 30% (455) - 3 1/2% Lactic Acid	Immediate Coagulation			-	
E. 1% Lactic Acid	Immediate Coagulation			-	
F. 0.1% Lactic Acid	O.K.	O.K.	O.K.	O.K.	O.K.
G. 0.01% Lactic Acid	O.K.	O.K.	O.K.	O.K.	O.K.
H. 0.001% Lactic Acid	O.K.	O.K.	O.K.	O.K.	O.K.

CONT

NITROCELLULOSE AIRCRAFT FINISHES
(Continued)

(C.20.)

Series IIPigmented with Lead Chromate

	Initial Observation	Overnight	1 week	1 month	2 months
Standard (4% (455)) Solvent Base	O.K.	O.K.	O.K.	O.K.	O.K.
A. Contains 20% (455)	O.K.	O.K.	O.K.	O.K.	O.K.
B. 20% (455) and 5% Lactic Acid	O.K.	Shows Coagu- lation does not dry.			
C. 20% (455) + 3 1/3% water	O.K.	O.K.	O.K.	V. Slight signs coagula- tion	V. Slight signs coagulation
D. 1% Lactic Acid	O.K.	Shows coagu- lation. Slight tack.			
F. 0.1% Lactic Acid	O.K.	O.K.	O.K.	O.K.	O.K.
G. 0.01% Lactic Acid	O.K.	O.K.	O.K.	O.K.	O.K.
H. 0.001% Lactic Acid	O.K.	O.K.	O.K.	O.K.	O.K.

cont.

NITROCELLULOSE AIRCRAFT FINISHES (C.20.)
(Continued)

Series III Pigmented with Zinc Stearate.

	Initial Observation	Overnight	1 week	1 month	2 months
Standard (4% (455)) Solvent Base	O.K.	O.K.	O.K.	O.K.	O.K.
A. Contains 20% (455)	O.K.	O.K.	O.K.	O.K.	O.K.
B. 20% (455) and 5% Lactic acid	Coagulation, apparent dissolution of pigment. Does not dry.				
C. 20% (455) + 5% Lactic acid	O.K.	O.K.	O.K.	V. Slight signs of coagulation	no change
D. 20% (455) + 2% Lactic Acid	O.K.	Coagulation Non-drying			
E. 1% Lactic Acid	O.K.	O.K.	O.K.	O.K.	O.K.
F. 0.1% Lactic-acid	O.K.	O.K.	O.K.	O.K.	O.K.
G. 0.01% Lactic Acid	O.K.	O.K.	O.K.	O.K.	O.K.
H. 0.001% Lactic Acid	O.K.	O.K.	O.K.	O.K.	O.K.

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Series No. 54.

NITROCELLULOSE AIRCRAFT FINISHES (G. 20.)

(Continued)

Series IV. Pigmented with Titanium Oxide.

	Initial Observation	Overnight	1 week	1 month	2 months
Standard (4% (455)) Solvent Base	O.K.	O.K.	O.K.	O.K.	O.K.
A. Contains 20% (455)	O.K.	O.K.	O.K.	O.K.	O.K.
B. 20% (455) and 5% Lactic Acid	O.K.	O.K.	O.K.	O.K.	O.K.
C. 20% (455) + 3 1/2% water	O.K.	O.K.	O.K.	O.K.	O.K.
D. 20% (455) + 3 1/2% Lactic Acid	O.K.	O.K.	O.K.	O.K.	O.K.
E. 1% Lactic Acid	O.K.	O.K.	O.K.	O.K.	O.K.
F. 0.5% Lactic Acid	O.K.	O.K.	O.K.	O.K.	O.K.
G. 0.01% Lactic acid	O.K.	O.K.	O.K.	O.K.	O.K.
H. 0.001% Lactic Acid	O.K.	O.K.	O.K.	O.K.	O.K.

contd

NITROCELLULOSE AIRCRAFT FINISHES
(Continued)

In the control series on Titanium no coagulation has occurred, whereas it has developed in some cases in all the other series. Lactic Acid in any appreciable quantity will show almost immediate coagulation, the concentration necessary to effect this reaction varying somewhat with the pigment in the finish.

In the case of the Zinc Oxide Series, coagulation has commenced in the finish containing 20% of Ethyl Lactate, and it has been shown that water in the finish will accelerate the process. There has as yet been no change in any of the finishes made on the standard solvent base which contains 4% of Ethyl Lactate.

The result of these tests therefore confirm our assumption as to the cause of the trouble, that is, it is due to the reaction of the pigments with lactic acid formed by the hydrolysis of ethyl lactate. Normally, in the finishes of the standard solvent base the concentration of lactic acid takes some months to become high enough to have any effect.

Consequently, we are altering the formulae of all our Camouflage Colours to DTD.754 leaving out the ethyl lactate.

PERMANENT RED 2.B.

(D.5.)

by

R. HEATON.

C.2995 is the barium toner and C.3028 the calcium toner of Permanent Red 2.B. In each case manufacture falls into two sections (1) the production of Permanent Red 2.B. sodium salt by coupling diazotised 3-amino - 4 - chlor - toluene - 5 sulphonic acid, and (2) forming the alkaline earth metal salt. However the dye is produced under somewhat different conditions for C.2995 and C.3028 and laking of these dyes is carried out under different conditions. Analysis of the quality of the factory C.3028 is much more uniform than that of C.2995. The present work was undertaken to find out whether the sodium salt of Permanent Red 2.B. will give a barium salt equal to C.2995 standard.

As a preliminary experiment C.3028 was prepared in the laboratory with the calcium chloride in the formula replaced by an equimolecular quantity of barium chloride. This product was much deeper and weaker than C.2995.

In the second experiment the sodium salt was prepared as in C.3028 but the laking was carried out as in C.2995. Here the self-colour was much brighter and somewhat paler than standard C.2995; the undertone checked well with standard but the toner was 10-20% weaker than C.2995.

PERMANENT RED 2.B.
(Continued)

(D.5.)

Variations of pH of dye suspension before laking have shown that the barium salt will not form if the pH value is 5.0 or less - (c.f. calcium lake which will not form if the pH value is pH 5.5 or more).

Work is continuing.

CHROMAT

(D.8.)

by

H. WEBB.

Present production of Chromat is on casein/soda ash binder, although it is known that a casein/lime bound water-distemper produces a better, harder film which can be recoated. However, casein/lime distempers have poorer dispersing properties and tend to froth more than those based on soda ash.

A recent publication by Technical Products, Ltd. recommends the use of Teepol X as a dispersing agent for aqueous suspensions, particularly useful in presence of hard water. Casein/lime distempers were therefore made up as follows :-

- (2) unmodified to act as control.
- (3) containing 1% Teepol on total weight of distemper.
- (5) containing 2% Teepol on total weight of distemper.

They were then dispersed in water with the results set out in the following table.

	Control	1% Teepol X	2% Teepol X
Ease of mixing with water	Standard	Not noticeably different from control	As 1% Teepol
Frothing	Fair	Equal to Control	Equal to Control
Skimming on standing	Pronounced	About equal to control	Slightly less than control
Settling on standing.	Bad	Amount about the same as Standard but rather more readily dispersed	

CROMAT
(Contd.)

(D.8.)

Series No.54.

Conclusion:-

Whilst Teepol X has a slightly beneficial effect on casein/lime distempers, especially in relation to settling it will not bring them up to the standard of our normal product.

Sodium pyrophosphate has also been recommended as a dispersing agent in the case of inorganic solid/aqueous dispersions, in quantities of about 0.01 Molar percent. (this is equivalent to about 1 oz. on a 5-cwt. batch of Cromat). A laboratory batch of a green Cromat was made up in which about 0.01 Molar % of sodium pyrophosphate was incorporated. This was dispersed in water and compared with a sample of corresponding run-of-plant material. This particular colour was used because of the difficulty of wetting the green which tends to float on the water. The treated batch wet at about the same rate as the untreated batch and settled to about the same extent overnight.

The subsequent dispersion of the settled Cromat did not appear to be easier in the case of the treated batch and no advantage of brushing or in the dried film was apparent.

Conclusion: Sodium pyrophosphate does not appear to improve the quality of Cromat in any way.

MAROON LAKE

(D.15.)

by

C. M. WEEB.

Various maroon pigments have been on exposure in Proxcelia formulae for the past 1 yr. 9 months, and the following is a summary of the results to date. (Exposure Reference PX.1638 A & B.). On the whole these results confirm previous experience, although the poorer results obtained with C.1465 cause some apprehension regarding the durability of deep shades in which this predominates. This is somewhat allayed by the improvement shown in the blends, and by the consideration that the above materials were based entirely on Ester Gum instead of the Ester-Damar blend now in use.

Routine tests on current manufacture should provide further information in 6-12 months, and further tests are in hand using a proportion of alkyd resin.

contd.

Exposure Tests on Maroon Enamels. (D.15)286/271/1.

There are no signs of breakdown in the finish. Rapid deterioration of the gloss and polish occurred but otherwise this exposure is fairly good. The colour became dirty after about six months and there were slight signs of chalking.

286/254/2.

The colour of this finish is still quite good. Gloss and polish are fair and slight chalking and pitting have occurred. On the whole this is quite satisfactory.

Q.1833

The colour of this is still quite good. Gloss and polishing are fair. The finish is chalking and pitting and checking is just commencing.

Q.2048

This blued up fairly rapidly. The gloss is fair and polishing poor. The finish has chalked very slightly.

Q.1465

The colour is now slightly blue. Gloss and polishing have gradually deteriorated. The finish has chalked badly and checking which was very slight is now beginning to spread over the panel.

Q.2033 1 pt.)

Q.1465 5 pts.)

The colour of this is still quite good. Gloss and polish have deteriorated. The finish has chalked slightly. There are no signs of pitting and checking.

Q.2048 1 pt.)

Q.1465 3 pts.)

Colour fair. Polishing and gloss are poor. Slight checking has occurred and there is no pitting or chalking.

EFFECT OF STEARAMIDE ON CHROME GREEN.....D.17by M. WEBB

The surface active agent Stearamide has been recommended by its producers as an ideal material for reducing flotation in Chrome Greens. They maintain that satisfactory results are obtained if the paint medium, chrome green and stearamide are milled together. About $\frac{1}{2}\%$ on the weight of green is recommended.

The material was tested as follows:-

(1) $\frac{1}{2}\%$ stearamide was ground into Y5089 and the treated pigment ground up on a palette with varnish V5929.

(2) 1% stearamide used precisely as above.

(3) $\frac{1}{2}\%$ stearamide was added to C1686 slop, thoroughly stirred, filtered and dried. The treated C1686 was then made up to Y5089 and a paint made up as in (1).

(4) Y5089, $\frac{1}{2}\%$ stearamide and V5929 were all ground together to make up a paint.

(5) A control was made up as in (1) omitting the stearamide.

The paints were brushed out side by side and examined after $\frac{1}{2}$ hour and after several hours. No difference in flotation was observable.

Parallel tests in stand oil (45), and linseed oil (3) gave exactly similar results.

Conclusion. Stearamide doesn't appear to have any beneficial effect on the floating of chrome greens.

PARA REDS D.18.BYJ.K. BARRACLOUGH.

Following the Laboratory Report in the issue Series 52 recommendations were made to the production department to replace sulphonated loral in C2461 by Calsolene Oil H.S. This has now become established works procedure and during the past two months 19 batches have been made on the revised formula, 17 were satisfactory to standard, the other two were slightly deep. (N.B. the formula was revised because run-of-plant material was tending to be too pale).

Production will be watched and if present standard of output is maintained the formula will be taken as satisfactory.

HANSA YELLOWS.D. 21.BY N. EDIS.

U.S. Patent No. 2,261,626 claims that by the introduction of quaternary ammonium compounds during the formation of azo pigments, brilliance and tinctorial power are improved.

The patent gives formulae for Hansa Yellows prepared in presence of about 12 quaternary ammonium compounds - none of which are commercially available in England. However the Lissolamines (I.C.I.) are compounds of this type and two of these, viz., Lissolamine A50 and Lissolamine V have been tried.

Using the formula quoted in the patent, dirty brown products were obtained, but using our Standard Hansa Yellow G (C2510) formula along with suitable quantities of the Lissolamines pigments of slightly greater strength and slightly impaired self-colour have been obtained. Although of little interest from a paint viewpoint they are of importance from that of printing-inks since in stand oil they give films of greatly enhanced finish when compared with C2510 or C2776.

Light-fastness tests are in hand.

AZO ORANGE, C.2611.....D.25BYJ.K. BARRACLOUGH AND R. HEATON.

Background. C2611 prepared by coupling diazotised o-nitro-aniline with B-naphthol is known in England as Azo Orange and in U.S.A. as o-nitro-aniline Orange. L.B. & S. are the only makers in England. The formula as used prior to this work has been used for many years giving results which fluctuated round a mean with such regularity the batch and batch blending gave results equal to standard. However, some time ago, a Works run of material came out consistently too red in self-colour and too blue in undertone.

Records of earlier work, unfortunately, do not exist.

Some of the work described was carried out in the laboratory before further works batches were made, the rest of the work described in the report was made as works trials were in progress.

Laboratory Results. Two basic formulae were used during the laboratory work, these are designated (A) and (B). (A) was used during the earlier work and (B) during the later work. They are:-

<u>A.</u>	<u>B.</u>	
8.0 gm.	8.0 gm.	Re 370 (o-nitro-aniline) mixed
50 ml.	100 ml.	thoroughly with water; to this
5.3 mls.	5.0 mls.	(60) (sulphuric acid) was added and
		stirred for 15 minutes. After cooling
		to 0°C. the Re 370 was diazotised with
3.8 gm.	3.8 gm.	Re 329 (sodium nitrite) as 10% solution.
		When diazotisation was complete the
		diazo solution was settled with
10 gm.	10 gm.	Re 20 and the clear liquor used for
		coupling.
7.9 gm.	7.9 gm.	Re 400 (B. naphthol) and
3.8 gm.	3.8 gm.	Re 343 (caustic soda) were dissolved in
200 ml.	150 ml.	hot water and diluted to
600 ml.	600 ml.	In case B
	20 ml.	10% (66) (Turkey Red Oil) was added and
		then coupling was carried out.
19 mls.		In case A
		10% (66) was added immediately after
		coupling.

After coupling the dye was stirred for 2 hrs. and washed twice with 2000 ml. water by decantation. It was then filtered and dried for 18 hrs. at 70°C.

The variants examined are listed below:-

(A) Surface Active Agents.

Experimental No. & Formula based on	Surface active Agent used	colour, against standard
379/70/1 A	(66)	S/c deeper than standard
379/70/2 A	Sulphonated lorol added before striking No. (66) used	S/c = standard U/t - slightly yellow to standard Strength = standard
379/70/3 A	Sulphonated lorol added after striking No. (66) used	S/c slightly dirty otherwise equal to standard

(B) Speed of Coupling.

Experimental Number	Formula used	Time of coupling	Colour against standard
379/76/2	379/70/2	30 secs.	S/c deeper than standard
379/77/1	379/70/2	20 mins.	S/c deeper than 76/2
393/71/A	B	20 secs.	Paler & yellower than standard.
393/71/D	B	20 mins.	Redder than standard

(C) Presence of Excess Nitrite in Diazo.

Experimental Number	Formula used	Variant	Colour against standard
379/79/1	379/70/2	No excess nitrite	S/c slightly paler and cleaner than standard.
379/79/2	379/70/2	Excess nitrite	S/c very deep and dirty.

(D) Amount of Acid present during diazotisation.

Experimental Number	Basic formula used	% of acid (Basic formula = 100%)	Colour against standard
379/70/2	A	100%	S/c = std.
379/82/1	A	98%	S/c slightly redder than std.
379/85/1	A	100%	S/c deeper than 82/1.
379 83/2	A	100%	S/c paler than 83/1.
379/90/2	A	98%	S/c = 379/82/1
379/90/3	A	105%	S/c deeper than 90/1
379/91/1	A	94%	S/c slightly yellower than std.
393/62/B	B	100%	S/c cleaner, brighter and a little yellower than standard.
393/63/A	B	102%	S/c a little yellower than std.
393/64/A	B	104%	S/c yellower than 63/A.

The end of coupling pH of the series 393/62/B, 63/A & 64/A varied only from 10 to 10.3

(E) Variations in R₁ 379.

Experimental Number	Basic formula used	Cask No. of R ₁ 379	Colour against standard
379/94/1	A	1	S/c slightly paler than standard U/t sl. yellow strength = standard
379/94/2	A	2	S/c sl. deeper than std. U/t sl. blue strength = standard
379/94/3	A	3	S/c much deeper than std. U/t sl. yellower strength = standard.

(F) Temperature of Coupling.

Experimental Number	Basic formula used	Temperature of coupling.	Colour against standard
393/57/A	B	45° C.	S/c little paler than standard.
393/62/E	B	25° C.	S/c little cleaner & yellower than std.
393/61/B	B	30° C.	S/c equal to std.

(G) Excess of Naphthol. An accidental spillage of a small quantity of diazo gave rise to a finished dye which was "out of place" in a series. For this reason the following series was carried out.

Experimental Number	Basic formula used	% of theoretical diazo	Colour against standard.
393/68/A	B	90%	S/c redder than standard.
393/68/B	B	95%	S/c redder than standard.
393/68/C	B	98%	S/c redder than standard. Less red than A or B.
393/68/D	B	100%	S/c slightly redder than standard. Less red than A, B or C.

(H) Filtration of diazo solution. On settling with R20 a clear solution is not obtained. 393/66/B was settled in the normal way, 393/66/D was filtered instead. The two finished products were identical.

(I) Reverse coupling. Normally the diazo is run into the naphthol solution. In the series 393/67/A, /B & /C the reverse procedure was employed. All the dyes were very dirty in self-colour.

SUMMARY OF RESULTS:- N.B. This summary also includes conclusions drawn from factory experience.

1. The two chief factors influencing the colour of o-nitrobenzene azo -2-naphthol are - speed of coupling, and the presence or absence of B-naphthol as impurity.
2. Sulphonated Lorol is a more satisfactory auxiliary than Turkey Red Oil.
3. The speed of striking, in the Works, can not be less than 15 minutes, (otherwise dirty products result).
4. Excess Nitrite in the diazo is to be avoided.
5. The amount of acid used in diazotisation is not critical - for this reason the new formula now in use in the factory contains only minimal quantities.
6. Temperature of coupling is not critical.
7. No advantage would result from filtering rather than settling the diazo before coupling.
8. Coupling Naphthol to diazo is unsatisfactory.

There seems to be no doubt that differences in the self-colour of C 2611 will occur from batch to batch during manufacturing; but it is believed that we know which factors are most important, and can adjust the formula, in such a way to produce redder or yellower products.

It is not proposed to carry out any further Laboratory work on this colour unless factory experience proves conclusions to be false.

Incidentally whilst engaged on this programme it has been brought to our notice that C 2611 cannot be used in stoving enamels in which Cobalt driers are present. No corresponding colour change is imparted by Lead or manganese drier.

Stonhard Resurfacer. by J. D. Stefanuti

Hardness Test. A simple test is being devised to give some indication of the hardness of Resurfacer.

The hardness of the material was determined by measuring the impression produced by a steel ball when dropped from a fixed height.

The procedure adopted was as follows. A sheet of carbon paper was placed face uppermost on the specimen to be tested and covered with a sheet of paper. The steel ball was then dropped on the specimen and the diameter of the impression obtained on the white paper measured.

The size of steel ball used in these experiments was 1 1/2".

The height from which the steel ball was dropped, 3 ft.

The mean diameter of a number of impressions was taken.

Comparative data of different materials was obtained by testing in the same way.

<u>Material.</u>	<u>Average diameter of impression.</u>
A. Concrete. 14 days old.	0 mm. diam.
B. Magnesite (composition flooring) Old.	6 mm. diam.
C. Wood floor.	9.0 mm. diam.
D. Resurfacer. 14 days old.	7.5 mm. diam.
Resurfacer (using sawdust as filler) 14 days old.	12.0 mm. diam.

Moisture Absorption. A number of similar resurfacer specimens were prepared and dried in the incubator at 40°C until constant weight was reached. The specimens were then immersed in water and the equilibrium moisture determined by frequent weighing. Maximum absorption was obtained where there was no further increase in weight.

The average absorption of a block weighing 120-150 grams was found to be 1.4 to 1.6% as compared with a concrete block having water absorption of 2.94%.

The water absorption of a Resurfacer flooring is therefore less than that of a concrete floor of the same sand/cement ratio, in this case 2 to 1.

Notes on priming of surfacers.

(using 60/80 Bitumen Primer and 180/220 Bitumen Primer)

1. Absorbent surfacers, such as concrete.

Dry. Two coats of primer were applied to a dry concrete surface using primer diluted in the ratio of 1:1 and 2:1 and allowed to dry out. It was found that the primer could be scraped off the concrete surface, showing poor penetration.

Damp. The experiments were repeated on a damp surface and although the time taken for the primer to dry out was longer (about 2 hours), the penetration of the primer into the concrete surface was greater, the primer film not being easily scraped off.

STONHARD P. 3 (CONTD)

2. Less or non-absorbent surfaces, such as hard wood, metal or stone.
When allowed to dry out, the adhesion of the primer at 1:1 and 2:1 dilution to this type of surface was found to be good.

Conclusions to be drawn from the results obtained are:-

- (a) For concrete a damp surface gives greater penetration of primer and therefore better bonding.
- (b) Non-porous surfaces can be primed satisfactorily.
- (c) That the most effective way of priming a surface would be to use the dilute solution (ratio 1:1 primer to water) as the first coat and the more concentrated solution (2 pts. Primer and 1 pt. water) as the second application.

Introduction of coloured pigment in Stonhard Resurfacer.

Experiments are being carried out with the view of determining the suitability of various pigments as colouring agents for Resurfacer. The results will be reported later.

Preparation of a tackier Primer using 180/220 Bitumen.

The raw materials used with the exception of the substitution of 60/80 Bitumen by the 180/220 variety in varying quantities, were similar to those in the preparation of the standard primer.

The final emulsion proved to be a satisfactory product but less viscous than the 60/80 Primer.

For brush application less water was required for dilution, i.e. 3 pts 180/220 Primer to 2 pts water as compared with 1 pt 60/80 Primer - 1 pt water.

The 180/220 Primer appears to give the tackier surface but on practical tests showed no advantages.

Resurfacer (180/220 Bitumen)

Similarly a quantity of Resurfacer was prepared using the same basic formula as in the case of 60/80 Bitumen Resurfacer.

The new product when mixed with sand and cement did not give a mortar which could be trowelled easily. There was a tendency for the material to adhere to the trowel. Also the material took a longer time to set and never attained the hardness of 60/80 Resurfacer. The material is also susceptible to heat (40°C) and becomes soft.

The use of 180/220 Bitumen in Resurfacer is therefore not recommended.

STONHARD P. 3 (CONTD)Stonhard "Stonpach".

The conclusion that may be drawn from studying the details given in the Stonpach literature is that the material probably consists of a mixture of sand, cement and a cement hardener possibly calcium chloride.

The following preliminary experiments were carried out with the object of formulating a similar product.

A number of mixtures containing 2 parts sand and one part Portland Cement and to which varying amounts of calcium chloride (ground) was added, were prepared and the initial and final setting times recorded at 15°C. Observations were also made on the appearance of the resultant materials when set, (using Vicat Needle), e.g.

- (1) 200 gms. cement & 400 gms. Leighton Buzzard Sand,
+ 65 gms. water.
Initial Vicat setting time = 15 mins.
Final " " " = 3½ hrs.
No sign of efflorescence after setting over-night.
- (2) 200 gms. cement & 400 gms. Leighton Buzzard Sand,
+ 2 gms. ca.cl. 2, i.e., 1% of cement.
Initial setting time = 12 mins.
Final " " " = 2 hrs.
- (3) 200 gms. cement + 400 gms. Leighton Buzzard Sand,
+ 1½% calcium chloride.
Initial setting time = 12 mins.
Final " " " = less than 2 hrs. (slight)
- (4) 200 gms. cement + 400 gms. Leighton Buzzard Sand,
+ 2% calcium chloride
Initial setting time = 7 mins.
Final " " " = 35 " efflorescence)
Definite efflorescence.

(1), (2), (3) & (4) were allowed to stand several days. No marked difference in appearance was noted. Comparative strength tests are to be carried out at a future date.

A dry mixture of sand and cement containing calcium chloride (1½% of wt. to cement) which was exposed to the atmosphere for over a week did not show any signs of deterioration.

Conclusions (tentative).--

- (1) Addition of calcium chloride to a sand and cement mix accelerated setting as is well known.
- (2) The amount of calcium chloride in the mix should not exceed 2% of the cement content.
- (3) The mix does not deteriorate rapidly on exposure.

Further work is in progress to confirm package stability under normal conditions of storage and to determine the maximum water content of sand used in conjunction with cement sold as a dry mix.

Works Test with Resurfacer (overall area test).

Area = 45 ft. long by 2 ft. ins. wide.
Laid to a thickness of ½" on the ramp leading to Paint Store (Basement No. 10). Owing to adverse climatic conditions 48 hrs. elapsed before the resurfacer had set hard enough to take traffic. Examination of the floor after several days

STONHARD P. 3. (CONTD).

did not reveal any sign of cracking or lifting.

There was no modification in procedure of laying the Resurfacer except (1) The primed surface was still damp when the Resurfacer was applied (2) Ratio of Primer to water was 2:1 instead of 1:1.

The floor was given a good trowel finish.

To date the test is very satisfactory. It has confirmed that it is not necessary to leave the first coat of primer for 8 hours, and our literature will be altered accordingly.

(29)

Fire Retardent Paints..... P. 40.By C. Stevens.

Fire Resistance Tests have been conducted on various paints which were regarded as having good fire retardent properties. They have been subjected to the Paint Research Station's "Flame Spread Apparatus", a copy of which has been made.

Deal panels free from knots and of low moisture content were used, size of panels being 9" x 6" x $\frac{3}{4}$ ".

Preparation of Panels.

2 coats applied by brush.
1st. coat Airdried 3 days.
2nd. coat Airdried 7 days.

the whole face, sides and 1" inwards on back of panels were coated.

Materials Tested.

B 2684.	Ignot No. 1.
B 3477.	Ignot No. 3. to BS/ARP 59.
Rx2210.	Matone. Standard formulation.
396/8/1.	Matone. Based on all Antimony Oxide Rm 618.
B 6578.	Burnot.
B 6654.	INC/FRP1 White.
396/9.	Rx1 + 10 - 15% Micronised Mica.
Rx4576.	Matroil Cream.
396/27.	Matone TYPE. 50% Antimony Oxide, 10% micro Mica, 40% Barytes.
396/26.	Modified B 6654 (10% micro Mica in place of China Clay)
345/328.	Rx1 + Wood Ash = pts by volume.
345/328/1.	Rx1 + Coal Ash = pts by volume.
345/328/2.	Rx1 + 50% Wood Ash 50% Coal Ash by volume.

Flame Spread Tests.

(29)

MATERIAL.	REF. NO.	TIME BEFORE IGNITION IN SECS.	FLAME SPREAD IN INS.	BLISTERS IN SECS.	CONDITION OF PANEL AFTER TEST. 5½ MINUTES HEATING.
Protexit. 345/ Wood Ash. 528		70	2½	30	General small blisters. Discolouration 6" along panel. Heavy charring 2½"
Protexit 345/ Coal Ash. 328/1		25	2¾	25-30	General slightly larger blisters. Discolouration 5½" along panel. Heavy charring 2¾".
Protexit 345/ 50% Wood Ash 50% Coal Ash	328/2	27	3½	25-30	Blistering similar to Coal Ash. Discolouration 5½" along panel. Heavy charring 2¾".
Ignot No. 1.	B2634	--	Nil	15	Huge blisters which crack on cooling. Wood untouched by heat. Panel buckled.
Ignot No. 3	B3477	--	Nil	30	Slight blistering. Discolouration of Film 5" along panel. Some slight charring. No breakage of film. Panel buckled.
DNC/ FRPI White	B6654	35	8.75	30	Slight small blistering. Film charred 8½" along panel. Heavy burning of wood 3" along panel. Panel buckled.
Pxl-10% Micro Mica	396/9	35	3	10	Heavy general blistering. Discolouration 7" along panel. Wood burnt 2½", charring up to 5" along panel.

CONTD.

Flame Spread Tests.

MATERIAL	REF. NO.	TIME BEFORE IGNITION IN SECS.	FLAME SPREAD IN INS.	BLISTERS IN SECS.	CONDITION OF PANEL AFTER TEST. 5½ minutes heating.
Matone All RM618.8/1	396/	25	3.75	30	Very slight isolated small blisters. Slight discolouration up to 6½ ins. Charring 3½". Heavy burning of wood 3".
Matone Std Figs.	RX 2310	12	6.4	30/35	Only very slight local blistering. Discolouration and charring 6½". Heavy burning 1½".
Burnot.	B6578	22	4	12	General small blisters. Discolouration up to 6½" along panel. Heavy charring 3½".
Matroil	4576	22	2.4	Nil	No blisters. Discolouration 2½" along panel. Heavy wood charring 2½".
Matone 50% RM618 40% RM21 10% Micro Mica	396/27	12	5	15	Heavy charring. Small blisters in patches. Discolouration 6½" along panel.
DNC/FRP1 Modified	396/26	7	2½-2¼	15	Local blistering. Heavy charring 1½" along panel. Discolouration 6½".

Class 1 requirements are:- A flame spread of not less than 1½".
 Class 2 requirements are:- A flame spread of between 1½" & 3½".
 Class 3 requirements are:- A flame spread of between 3½" & 8".
 Class 4 requirements are:- A flame spread of more than 8".

Contd.

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Water Resistance on Fire Retardent Paints.

Glass slides prepared with each paint, which were then airdried 7 days, then immersed in distilled water for 48 hours. The amount of water absorbed being calculated

MATERIAL.	REF. NO.	CONDITION OF FILM AFTER IMMERSION.	% WATER ABSORPTION.
Ignot No. 1	F2684	Film removed from glass.	-
Ignot No. 3	B3477	Film removed. Partly dissolved.	-
DNC/FAP1 White	B6654	Untouched.	6.65
Ixl + Micro mica	396/9	Some blistering. Softened.	45.0.
Matone All R.G18	396/3/1	Film lifted. Softened.	12.77
Matone Std.Figs.	2210	Film lifted bottom edge only.	10.0
Burnot White	B6573	Completely removed from glass.	-
Matroil	4576	Softened.	2.05
Protexit Wood Ash	345/328	Small blisters. Softened.	28.82
Protexit Coal Ash	345/328/1	Moderate blisters. Softened.	25.46
Protexit 50% Wood 50% Coal	345/328/2	General fine blisters. Softened.	28.32

Conclusions to date are that, R.G18 is advantageous. Oil promotes blistering, it is therefore preferable to use varnish. Micronised mica is an advantage for flame resistance. "Protexit" gives dark grey colour and both Coal and Wood Ash are water soluble.

FINING CUL/80 P.54
By C. Stevens.
YASCO SUPER SYNTHETIC ENAMELS

Exposure of 12 months duration

Chrome Used.	Exposure No.	Checking.	Gloss.	Colour	Remarks.
Y3508	835 ^A	8/7	Good	Bad	Very dark and brown Moderate Dirt Retention
BY9671	835 ^B	9	Good	Bad	"
C2973	835 ^C	Incipient	Poor	Bad	Very slight blistering Greenish in tone. Moderate Dirt Retention
C1686	837 ^A	"	Good	Moderate	Much deeper and darker Fair dirt retention
C2664	837 ^B	"	Good	Fairly Good	Best colour in comparison with others. Fair dirt retention.
C3103	837 ^C	"	Good	Moderate	Green and dirty Fair dirt retention

C2664 is the best of series for stability of colour C1686 and C3103 are next best.

C2973 worst for gloss.

Pompeian Super Synthetic Enamel Saxon Green Panels Nos. 833 & 9 & 40 when made on these same individual yellow pigments in conjunction with Monastrol Blue and Chinese Blue give very similar results to the straight chromes etc. It is noted that where Monastrol Blue is used the general tone is dark and blue whereas with Chinese Blue the general tone is dark and rank.

C2664 with BY8352 (Chinese Blue) gives the best colour retention followed by C3103 & BY8352. The checking reported has occurred since the last examination, see this report Series 52.

POLYKEM.P. 81.BY J. NICKMAN.Bergermaster.

Investigation of properties of Styrene copolymer vehicle SA 5320 and paints Nos. Rx 6980-6985.

1. Film weight at which wrinkling occurs at 100°F.

Tested: each colour and the vehicle.

<u>Paints.</u>	Below 2 ozs/sq.yd.	no wrinkling.
	at 2½ " " "	Incipient wrinkling and wrinkling of fat edges.
	at 3½ " " "	Pronounced surface wrinkling.
<u>Vehicle.</u>	At 1.8 " " "	First sign of wrinkling.
	2.3 " " "	Slight surface wrinkling.
	3.0 " " "	Pronounced surface wrinkling.

2. Heat Stability.

Half pint samples of each colour and the vehicle were tested through the Ford Cup and then placed in the incubator at 104°F. for 14 days.

Settlement:-

6980 Broken White	: Fairly hard settlement
6981 Cream	: " " "
6982 Biscuit	: Hard rubbery settlement
6983 Terra Cotta	: Little settlement
6984 Turquoise	: Virtually no settlement
6985 Berk. Green	: Fairly hard settlement

Where settlement had occurred the paint could be knocked up to give the original homogeneity. All the paints and the varnish were brushed out and gave satisfactory panels.

Viscosity: In most cases there was a small increase in viscosity.

Colour	No.	Viscosity; Ford Cup No. 4. Seconds @ 25°C	
		Initial	Final.
Broken)	6980	147	158
White)	6981	129	130
Cream)	6982	107	123
Biscuit)	6983	198	242
Terra)	6984	175	183
Cotta)	6985	169	176
Turquoise)	SA 5320	171	205
B. Green)			
SA 5320)			

3. Rain Spotting after 1-3-7 days' application.

Tested: All colours and the vehicle.

In all cases rain spotting was pronounced.

It was difficult to detect differences between panels splashed after 1 day and those splashed after 3 days.

The panels splashed after 7 days showed slightly less pronounced spotting.

4. % Water Absorption. (Specification 2 x 17 method).

6980	Broken White	5.5
6981	Cream	4.2
6982	Biscuit	4.4
6983	Terra Cotta	2.6
6984	Turquoise	2.3
6985	Berk. Green	2.8
SA 5320	--	2.3
5433	Pompeian - Berk. Green	4.3
6833	Super Synthetic Pompeian) Berk. Green)	6.5

5. Bloom of SA 5320 applied over matt black, compared with SA 2165.

SA 5320 copolymer and SA 2165 alkyd, were applied side by side on a panel coated with ES 6120.

The varnishes dried with equal gloss and gave no bloom initially or after ageing in laboratory for several days.

6. Adhesion between enamel coats when applied at 6-16-24 hour intervals.

Each colour was tested

In every case adhesion at each time interval was satisfactory.

No difference could be detected between panels brushed at the different time intervals.

Contd.

7. Petrol Resistance; MTD 260 Method.

Tested all colours and the vehicle.

Paints. Slight loss of gloss
Film weakened
Bend test satisfactory after immersion

Would pass MTD 260 only as border-line case
owing to loss of film strength.

Note:- Turquoise - In case of Turquoise a
colour change occurred, the treated film being
bluer due to solution of Hansa Yellow.

SA 5320. No loss of gloss
Bend test satisfactory
Film softened considerably
Owing to last factor SA 5320 would pass
MTD 260 only as a border-line case.

8. Oil Resistance MTD 260.

Tested all colours and SA 5320
All films unaffected by oil

9. Gloss Retention of Bergermaster compared with
Pompeian Super Synthetic.

Tested Berkeley Greens only.

4 panels were coated with U/c. 6765
2 were given one coat Bergermaster 6985
2 " " " " P.S.S. 6833

After drying overnight the panels were placed over
water in a sealed tank and left for 7 days at room
temperature.

Initial gloss was very similar

After treatment P.S.S. had very slight bloom

Bergermaster had no bloom

Super Synthetic Pompeian.....P.82.

By J. F. Wells.

Comparative Water Absorption of House Paints and Enamels.

Aluminium panels were given 1 coat (brush application) of material, then air dried for 7 days, films weighed, then immersed in distilled water for 24 hrs. and reweighed. Films were also examined after a further 4 days air drying for any permanent injury (i.e. bubbling - loss of gloss). Tests were made in duplicate and following results are averages, duplicates gave comparable results in all cases.

Colour.	Ref. No.	Film wt.	Berger master. % age absorption	Film appearance 4 days after immersion.
B. Green	6985	0.347	6.55	O.K.
Terra Cotta	6983	0.272	6.25	O.K.
Stone	6982	0.267	5.63	O.K.
Cream	6981	0.363	6.29	O.K.
White	6980	0.362	3.63	O.K.
Turquoise	6984	0.283	6.04	O.K.

Pompeian Enamel.

B. Green	3453	0.303	13.78	O.K.
Terra Cotta	3420	0.298	8.0	O.K.
Stone	3407	0.586	11.04	O.K.
Cream	3487	0.612	12.48	O.K.
White	3488	0.594	16.05	O.K.
Turquoise	3458	0.510	16.48	O.K.

Super Synthetic Enamel.

B. Green	6833	0.294	16.41	O.K.
Terra Cotta	6820	0.230	12.53	O.K.
Stone	6807	0.247	15.10	O.K.
Cream	6887	0.326	14.53	O.K.
White	6888	0.316	10.29	O.K.
Turquoise	6858	0.313	22.35	O.K.

"B. P." Paint.

B. Green	687	0.575	23.5	Film soft.
Terra Cotta	687	0.393	22.05	Lost gloss (Flat)
Stone	688	0.825	25.35	Film sl. soft.
Cream	688	0.905	35.25	Film soft.
White	680	0.748	26.55	Film sl. soft
	685	0.522	7.76	Film soft.

(xx) Rr 686 is B.P. middle Green as no Turquoise shade is made in this range.

It is observed that the styrene copolymer enamel, Bergermaster has higher water resistance than Pompeian (Zinc oxide-oleo-resinous vehicle) or Super Synthetic (Titanium-Antimony in Alkyd). "B.P." Lead Paint has, surprisingly, a very much higher water sensitivity than the other lines.

SUPER SYNTHETIC MAYFAIR CREAM ENAMELS. P.82.BY J. F. WELLS.

Enamels were ground using various pigments and pigment combinations, tinted with Hansa Yellow. The finished enamels were then exposed, mainly to check colour and gloss retention.

Type formulation. 253/284/1. Equal parts Titanium Rm 713
 " " Antimony Rm 618
 224 Gms. 713
 224 " 618
 Tint C 2664
 674 Ccs. SA 5291
 102 " (25)
 32 " (86)
 6 $\frac{3}{4}$ " V 3906
 6 $\frac{3}{4}$ " V 4031

This is the present Super Synthetic and Kemitone formulation RX 6887. The above pigment was substituted as follows, the total quantity varying to maintain opacity.

284/2.	565	Gms.	pigment comprising	2 parts	Antimony Rm 618
				1 part	Titanium Rm 713
284/3.	896	"	"	"	all Antimony Rm 618
284/4.	448	"	"	"	all Titanium A10 Rm 610
284/5.	565	"	"	"	2 parts Antimony Rm 618
				1 part	Titanium A10 Rm 610
284/6.	585	"	"	"	8 parts Antimony Rm 618
				4 parts	Titanium A10 Rm 610
				1 part	Zinc Oxide Rm 6
284/7.	486	"	"	"	4 parts Antimony Rm 618
				4 parts	Titanium Rm 713
				1 part	Zinc Oxide Rm 6

These enamels were exposed on steel panels over Primer and Undercoating against Pompeian Mayfair Cream Rx 3487 (all Zinc Oxide pigmentation) with the following results.

After 1 month	284/1	} Showed definite fading
	284/2	
	284/4	
	284/5	

After 3 months Rx 3487 and 284/3 (all Antimony) show very good colour retention.

284/1 and 284/4 - very bad fading
 284/2 and 284/5 - bad fading, but better than 284/1 and /4.
 284/6 and 284/7 - redder and darker in colour, also bad dirt retention.

(33)

Exposures are continuing, but it will be already observed that Antimony retains colour in pale tints, whereas Titanium pigments fade badly. The addition of Zinc Oxide as a third component stops fading, but shows darkening to occur. N.B. The Zinc formulations were applied to panels the same day as ground, since such enamels are not stable with the vehicle used.

Water Absorption Tests.

Absorption tests were also made with the above pigments, together with additional enamels ground on the same type formula and then all standardized on a constant pigment to Medium basis of 115 Pigment - 88% media.

Panels used were aluminium, 1 coat of enamel dipped and then air-dried for 7 days before being immersed in distilled water for 24 hours. Tests were done in triplicate and the following results are the averages obtained, in no case was there any appreciable variation in the results of the three determinations.

Ref.No.	Pigment	Film Weight.	% Absorption.
284/11	Rm 6 Zinc Oxide	0.725	16.3
284/9	Rm 8 Lithopone	0.679	10.72
284/8	Rm 713 Titanium	0.948	5.7
284/4	Rm 610 A.10 Titanium	0.760	5.35
284/3	Rm 618 Antimony	0.759	4.7
284/10	Rm 770 White Lead	0.821	4.16
284/1	(1 part Rm 618 Antimony (1 part Rm 713 Titanium	0.862	7.19
284/2	(2 parts Rm 618 Antimony (1 part Rm 713 Titanium	0.860	5.42
284/5	(2 parts Rm 618 Antimony (1 part Rm 610 A.10 Titanium	0.850	3.33
284/7	(4 parts Rm 618 Antimony (4 parts Rm 713 Titanium (1 part Rm 6 Zinc Oxide	1.120	7.22
284/8	(8 parts Rm 618 Antimony (4 parts Rm 610 A.10 Titanium (1 part Rm 6 Zinc Oxide	1.084	7.03

Conclusion.

Antimony, White Lead and Antimony-Titanium A.10 combination give the best results, while lithopone, as expected, worse. The result on Zinc Oxide may be false as this pigment feeds badly with the medium used, and gives as a result a very thick film on which to gauge results.

Contd.

SUPER SYNTHETIC POMPEIAN P. 82.

by C. Stevens.

Pompeian Super Synthetic Enamels Rx 6888 Type made on various alkyd resins and exposed for 12 months. E.P. 808 - 816.

WHITE ENAMEL.

Resin	Chalking Rating 0-10	Colour	Gloss
Beckosol 1427	4	Good	Poor
" 1450	4	Good	Poor
" 1441	4	Good	Poor
Synolac 157 A	4	Good	Poor
Paralac 10	5	Good	Poor
" 11	6	Good	Flat
" 15	6	Good	Fairly Good
W. Resin 6	6	Good	Poor
" 8	6	Good	Fair
SA 5089)	7	Slight)	Fair
SA 5090)		Brown)	
SA 5291		Good	Fair/Poor
Alprokyd 607 P	6	Good	Fair
SA 5239	7	Yellowed	Fair

CREAM ENAMEL.

Beckosol 1427	4	Good	Poor
" 1450	6	Good	Poor
" 1441	6	Good	Flat
Synolac 157 A	7	Faded	Fairly Good
Paralac 10	7	Good	Poor
" 11	7	Good	Flat
" 15	6	Good	Fair
W. Resin 6	6 ?	Good	Poor
" 8	6 ?	Good	Fair
SA 5089)	4	Slightly	Poor
SA 5090)		dirty	
SA 5291		Good	Poor
Alprokyd 607 P	5	Good	Poor
SA 5239	3	Fraction dirty	Poor

POMPEIAN SUPER SYNTHETIC GREEN ENAMEL.

Beckosol 1427	10	Good	Fair
" 1450	10	Slightly Pale	Fair
" 1441	10	"	Fair
Synolac 157 A	10	"	Fair - Light dirt retention
Paralac 10	10	"	Good
" 11	10	Good	Fairly Good
" 15	10	"	Fairly Good

Contd.

POMPEIAN SUPER SYNTHETIC GREEN ENAMEL (CONTD).

Resin	Chalking Rating 0-10	Colour	Gloss
W. Resin 6	10	Good	Fairly good
" 8	10	Good	Fair - Light dirt retention
SA 5089 }	10	Fraction)	Very fair
SA 5090 }		Blue)	
SA 5291	10	"	Fair
Alprokyd 607 F	10	Good	Fair
SA 5239	10	Good	Fair

All panels are free from failure by crazing, checking or flaking. Berger resins appear to be equal to competitive products.

For light fastness of Super Synthetic Enamels see Item P.54.

FINISHES FOR PREFABRICATED HOUSES.

P.86

BY - F.C. Everett - D.E. StarTHE ALUMINIUM HOUSE.

A few changes in formulation have been necessary due to raw material difficulties & for other reasons.

DEEB 101 - B6842 - off-white primer is unchanged, but some trouble has been experienced with the dip-tank at Messrs Blackburn, due to excessive loss of naphtha caused by the method of agitation used (air-blowing). Thinners for this material will in future consist of (357) - naphtha only.

DEEB 104 - B7205. The omission of V6598 has been necessary, due to the shortage of aluminium Stearate. Although retained reference tins of numerous batches showed no pigment sedimentation complaints were received from the consumer. As a result the stearate was replaced. The solvent in the medium has been changed from (522) to white spirit (i.e. Medium change from V7394 to V5024) without loss of drying speed. The use of RM27 china clay as a possible alternative to whitening has been investigated & found satisfactory.

Some failures as regards specification drying & bend tests have been experienced with production batches & these appear to be due to variations in the non-volatile content of the medium or to the pigment non-volatile medium ratio.

DEEB 306 SA3560 Tests of old reference tins of this material have indicated that serious darkening occurs on storage, when the urea resin from Messrs Pinchin Johnson (467)A - which contains 3% of water & soluble salts - has been used. This colour change is noticeable on stoving for 1 hr at 300° F. in the normal way. This trouble has been traced to contamination by iron from the black plate tins following the softening of the protecting lacquer. Such contamination does not occur in tin plate containers or in the tinned plate 5 - gallon drums normally used as bulk packages. The Customer should therefore not meet any trouble.

A similar defect has occurred when batches of SA3560 are made in a steel ball mill & this practice has been discontinued.

Conservation of butanol, & xylene have been necessary. The latter has been replaced by (469) a light solvent naphtha & the former has been considerably reduced. Ethyl lactate is now omitted from SA3560, the present formula for which is now below:-

<u>SA3560</u>		<u>SA5404</u>	
150 lbs.	- 713	Alkyd Medium	
40 "	- 27	72 lbs	- (197)
Tint 64 "	- 22	33½ "	- RM731
60 "	Gls. SA5404	125 "	- (368)
20 "	(467) or (467)A	205 "	- RM731
14 "	(469)	125 "	- (365)
Tn yield	100 galls.	60 "	- (453)
		60 galls	- (469)
		11½ "	- (432)
		2½ "	- (38)
		Tn yield =	110 galls.
		Manufacture as SA5554.	

contd.....

The present-formula for SA3560 leaves much to be desired as regards flexibility when stored for at 300° F. It has been found possible to obtain satisfactory performance to specification & much improved flexibility by making a substantial reduction in the quantity of U.F. resin & tests are proceeding on these lines.

DDFB 109 -ES6301. This enamel based on SA5338, - a 50% oil length linseed alkyd - showed a tendency to excessive rain-spotting when subjected to the specification weathering test. A series of 4 accelerated weathering tests of the standard 4 -coat system were tested, using 4 different media. The following results were obtained:-

Complete system on SA5338 -shade S52 - Gloss fair, rain spotting moderate. Colour retention rather poor, film OK.

Complete system on SA5072 -shade S52 - Gloss fairly good, rain spotting causes blue spots. Colour retention rather poor. Film shows local blistering.

Complete system on Paralac 11V - shade S54 - Gloss good, rain spotting moderate, colour retention good, film OK.

Complete system on Paralac 16X - shade S54 - Gloss fairly good, rain spotting slight, colour retention good, film OK.

This series of tests again demonstrated the superior weathering properties of the Paralac resins. A more recent test on ES6301 has however given a very much better result to that obtained above & no immediate change in medium is contemplated.

Bittiness has been met with in this particular material - a defect not apparent in the medium. The enamel does however exhibit a strong tendency to skin & this is probably the cause of the trouble.

WHITE LEAD PAINTS.

P98.

C.Stevens.

- (1) Further Examination of exposures after 10 months EP867 - 874.
 Comparison of Cooksons' - Lonabarc, Metal Colors grades of RM 770 with
 Octagon High Stain and Octagon Low Stain and electrolytic process
 White Leads. (2) The evaluation of blends of White Lead with zinc oxide
 and Antimony Oxide.
 All paints were Tinted Pale Blue with BY8352

TYPE OF LEAD	MEDIUM	CHALKING	COLOUR	GLOSS	REMARKS.
Cooksons	V7431	5	Faded Badly	V. Poor	Heavy dirt retention
Cooksons	345/109	6	"	"	"
Cooksons	345/110	6	"	"	"
Cooksons	V4201	6	"	"	Slight erosion
Lonabarc	V7431	5	"	"	Heavy dirt retention Very slight blistering
Lonabarc	345/109	5	"	"	Heavy dirt retention
Lonabarc	345/110	5	"	"	"
Lonabarc	V4201	5	"	"	Slight blistering. Moderate to heavy erosion
Metal Colors	V7431	5	"	"	Heavy dirt retention. Slight erosion
Metal Colors	345/109	5	"	"	Heavy dirt retention
Metal Colors	345/110	6	"	"	"
Metal Colors	V4201	5	"	"	Fairly heavy erosion

(44)

WHITE LEAD PAINTS

. G. Stevens.

TYPE OF LEAD	MEDIUM	CHALKING	COLOUR	GLOSS	REMARKS
Octagon High Stain	V7431	5	Badly Faded	Poor	Heavy dirt retention
Octagon High Stain	345/109	5	"	"	"
Octagon High Stain	345/110	5	"	"	"
Octagon High Stain	V4201	5	"	"	"
Octagon Low Stain	V7431	5	"	V. Poor	Moderate dirt retention Slight erosion
Octagon Low Stain	345/109	5	"	"	Moderate dirt retention
Octagon Low Stain	345/110	6	"	"	"
Octagon Low Stain	V4201	5	"	"	"
Electrolytic ref. 241/220	V7431	4/5	"	Poor	"
Electrolytic ref. 241/220	345/110	4/5	"	"	"
Octagon H.S.3 Rm6 1	345/110	6	"	"	"
Octagon L.S.3 Rm6 1	345/110	6/5	"	V. Poor	Moderate dirt retention Slight erosion
Ionabarc Rm6 1	345/110	6/5	"	Poor	Moderate dirt retention
Cooksons Rm6 1	345/110	6/5	"	V. Poor	"

WHITE LEAD PAINTS

TYPE OF LEAD		MEDIUM	CHALKING	COLOUR	GLOSS	REMARKS
Metal Colors	3	345/110	7/6	Badly Faded	V. Poor	Moderate dirt retention
Rm 6	1					
Metal Colors	4					
Rm 6	1	345/110	7/6	"	"	"
Rm 618	1					
Octagon H.S.	4					
Rm 6	1	345/110	6	"	Poor	Isolated blistering
Rm 618	1					
Octagon L.S.	4					
Rm 6	1	345/110	6/5	"	"	Moderate dirt retention
Rm 618	1					
Cooksons	4					
Rm 6	1	345/110	7/6	"	"	"
Rm 618	1					
Lonabarc	4					
Rm 6	1	345/110	7/6	"	"	"
Rm 618	1					

CONCLUSIONS:- The influence of the addition of 33% linseed stand oil or of 33% linseed wood oil Stand oil is only slight.

Thin Stand oil is worst of the four media tried, on account of erosion.

Only small differences are apparent & it is not possible to select any one type of lead as being best.

The addition of antimony & Zinc Oxide to any of the White Leads so far shows a slight improvement, re chalking. Zinc Oxide as a sole additive has no apparent effect.

RAILWAY COACH & ENGINE FINISHES

P103

C.Stevens.

GREAT WESTERN RAILWAYVARNISH TESTS

Various varnishes have been exposed to the G.W.R. specification for 18 months, with the following results.
E.P. 718 - 723.

(116) Ex. Pale Oak Vsh.	Heavy checking, Poor colour, No gloss. Heavy Dirt Retention. Checking first visible after 5 months on 1 coat of Vsh.
(116)+7 $\frac{1}{2}$ (45) 314/186	Moderate to Heavy checking, Poor Colour, No Gloss, Heavy Dirt Retention. Checking first visible after 4 $\frac{1}{2}$ months on 1 coat of Vsh.
(116)+15 (45) 314/186/1	Moderate to Heavy checking, Poor Colour, Slight Gloss, Checking first visible after 4 $\frac{1}{2}$ months. 1 ct. of Vsh. Heavy Dirt Retention.
(118) "Perfecto"	Heavy checking, Moderate erosion, Poor Colour, No Gloss Checking first visible after 4 $\frac{1}{2}$ months. Slightly more heavy dirt retention than on others. 1 Coat.
(116)+7 $\frac{1}{2}$ V7371 314/187	Moderate checking, Best colour, Fair gloss. Checking first visible after 4 $\frac{1}{2}$ months 1 coat Moderate dirt retention.
(118)+15 V7371 314/187/1	Moderate checking. Dirty Colour No Gloss. Heavy Dirt Retention. Checking first visible after 4 $\frac{1}{2}$ months 1 coat.

From these tests it appears that (116)+7 $\frac{1}{2}$ V7371 Conlinox Stand Oil is slightly the best. Panels to be withdrawn.

Conlinox is Conjugated Linseed Oil manufactured by Younghusband Barnes.

Process of Panels.

Sandblasted steel panels painted with 1 coat Bxl291 White Lead paint thinned with V4079 and White spirit. Finished as follows:-

- 3 coats Varnish on 1st inch of panel
- 2 coats Varnish on 2nd inch of panel.
- 1 coat Varnish on centre of panel.

In every case the two coat system is better than one coat in respect of gloss & checking & the three coat system only fractionally better than two coats.

LABEL VARNISH & WATERPROOFER
by D.R. Star.

P.108

A sample of a label varnish from a competitor was analysed
& a match prepared using the following formula:-

332/240/1

102	grms.	-	Gelva 7
84	-	-	(24)
414	-	-	(38)

Cold cut.

This was tested on many different types of paper & labels in the Printing & Advertising Department & found to give satisfactory impregnation combined with a semi-gloss finish. Waterproofing properties were also good in all cases. The flexibility was far superior to the standard manilla type Varnish & showed no damage when folded.

REFRIGERATOR AND EQUIPMENT FINISHES.KEMPLASTITE.P. 117.

Further work has been put in hand to determine the relative merits of SA 5334 and SA 5404. Five pots of each have been made under joint Technical Service and Technical Operation supervision, tanked and converted into Kemplastite in the works.

Manufacture of Media.

For the purpose of this test the following standard formulae and procedures were adopted.

SA 5307

1. (58 lbs. (197)
(27 " RM 731
2. (120 " (368)
(164 " RM 731
(104 " (365)
- 48 " (453)
57 gls. (240)

Yield = 94 galls.

SA 5334

1. (72 lbs. (197)
(33½ " RM 731
2. (125 " (368)
(205 " RM 731
(126 " (365)
- 60 " (456)
50 gls. (240)
11½ " (432)
2½ " (38)

Yield = 110 galls.

Manufacture.

(1) run to 300°F. in 1 hr. and hold till clear, (2) added and whole raised to 575°F. in 3 hrs. and held 5-10 mins. for very tough bead just showing cold fracture.

Manufacture.

(1) run to 300°F. in 1 hr. and held till clear. Then (2) added and whole raised to 500°F. and held for an average time of 2 hrs. 20 mins. for a very tough bead showing cold fracture.

The progress of the reaction at 500°F. was carefully followed in the case of SA 5334, samples of base being removed at intervals during the cook and made into a solution in xylol of 50% solids.

The following constants refer to such a solution.

Time at 500°F.	Viscosity poises	Acid value of solution	Colour (Gardner-Holdt)
½ hrs.	3.4	20.12	6
1 " "	4.0	17.52	6½
1½ " "	4.0	17.24	6½
2 " "	4.4	15.39	8
2½ " (Pot off fire)	5.0	15.04	8
3 " (450°F.)	5.6	15.31	8
3½ " (423°F.)	6.2	15.10	9
4½ " (thinned)	12.9	15.35	9

KEMPLASTITE P.117 (CONTD)

Constants of 5 blended pots each of SA 5307 & SA5334.
These were as follows:-

	SA 5334	SA 5307
Acid value	(17.19) 16.75	10.5
% Solids	52.65	47.68
Visc. poises @ 25°C.	(1.65) 2.40	3.7
Colour (Berger)	(4) 4.5	5
S.G. @ 15°C.	0.991	0.981

The figures in brackets represent constants determined on samples from each of the five pots blended in the laboratory.

Remarks.

The constants of the SA 5334 bulk are normal. The viscosity of the SA 5307 is however considerably lower than standard (6-8 poises).

From an examination of the changes occurring during the cooking of SA 5334 it is apparent that the greatest viscosity increase is obtained during the cooling period and the duration of this period (normally 2-2½ hrs. in the case of SA 5334) is obviously important.

An attempt was made to reduce the acid value of one pot of SA 5334 by increasing the CO₂ blow towards the end of the cook. It was not found possible to effect any substantial reduction in acid value by this method, nor is it possible to check accurately the rate at which CO₂ is being passed with the existing works equipment.

The discrepancy between the constants determined upon the laboratory blend of the 5 batches and the bulk when tanked would appear to be due to loss of volatiles when the tank is mixed by blowing. A similar difference was noted in the case of the SA 5307. Incidentally moisture is introduced on air blowing.

Other aspects of this investigation relating to SA5307 have been covered by Technical Service.

Preparation of Kemplastite enamels on special batches.

on SA 5307
150 galls. McFranti Cream SA 3805/5215G-ground-40 hrs.
50 galls. Armstrong Maroon SA 3705/3219G - ground - 60 hrs.
75 galls. Apple Green SA 3609/29623/A - ground - 48 hrs.
150 galls. Serigloss Brown SA 3754/3327G - ground - 40 hrs.
on SA 5334
150 galls. Leaf Green SA 3536/3449G - ground - 70 hrs.
75 galls. Apple Green SA3609/29623/B ground - 48 hrs.

These gave the following results after ageing for 6 weeks when stoved for ½ hr. @ 220°F. on tin plate.

Reference No.	Gloss	Condition just after stoving.	Condition 4 days after stoving.	Factory report just after making
SA5307				
SA3805/5215	Excellent	Sl. short	Hard & tough	O.K.
SA3705/3219	Fairly good.	very "	F-hard & "	sl. soft
SA3609/29623A	Good	" "	very short	O.K.
SA3754/3327	Satisfactory	Rather"	Hard & tough	-

KEMPLASTITE P.117 (Contd)

Reference No.	Gloss	Condition just after stoving.	Condition 4 days after stoving.	Factory report just after making.
5334. SA3536/3449	Fairly good	Rather short	Hard & tough	O.K.
SA3609/2862B	Good	f. hard & tough	Hard & Tough	O.K.

Remarks.

The general improvement of adhesion and toughness of Kemplastite films on ageing is very apparent in this series but nevertheless does not always develop with works batches.

Conclusion.

From this series of tests it is not possible to differentiate between SA 5334 and SA 5307. The slight advantage in hardness and gloss shown by SA 5307 in a previous test is not apparent. This may be due to under-cooking of SA5307 in this instance as indicated by its abnormally low viscosity.

There is no evidence which warrants a reversion from SA 5334 to SA 5307.

"TWELVE STAR" ALKYD ENAMEL

P120

C.Stevens.

Comparison of alkyd resins SA5291 and SA5239 after having been
exposed 11 months on Steel. Primed with RX4102.
E.P. 821 = 826

Colour	Medium	Chalking	Colour	Gloss	Remarks.
White	SA5291	10	Fairly Good	Fair	Fine extensive blistering
White	SA5239	10	Fraction Yellow	Fairly Good	Fine extensive blistering
Mayfair Cream	SA5291	10	Faded	"	Colour nothing like original Fine general blistering.
Mayfair Cream	SA5239	9	Fraction Faded	Good	Fine general blistering
Pale Biscuit	SA5291	10	Very Dirty	Fairly Good	Slight checking, dirt retention. Hansa C.2510 disappeared.
Pale Biscuit	SA5239	10	Slightly Dirty	Good	Checking 8. less dirt retention than with SA5291. Chrome C1522
Crimson Oxide	SA5291	10	Good	Fair	Checking 7
Crimson Oxide	SA5239	10	Good	Poor	Checking 8
Turquoise	SA5291	10	Fraction Pale & blue	Good	Slight dirt retention, Very slight blistering
Turquoise	SA5239	10	"	Good	Checking 8/7. Slight dirt retention
Early Green	SA5291	10	"	Good	Slight dirt retention Checking 8
Early Green	SA5239	10	"	Good	Slightly more yellow than with SA5291. Slight dirt retention.
Saxon Green	SA5291	10	Little brown	Good	Slight dirt retention
Saxon Green	SA5239	10	"	Good	Checking 8/7
Berkely Green	SA5291	10	Fraction dark	Good	Checking 9.
Berkely Green	SA5239	10	"	Fairly Good	Slightly darker than SA5291 Checking 9

(52)

Series 54.

Colour	Medium	Chalking	Colour	Gloss	Remarks.
Dark Lincoln Green	SA5291	10	Fraction deep & blue	Good	Incipient checking
Dark Lincoln Green	SA5239	10	"	Good	Checking 8
Terra Cotta	SA5291	10	Fraction deep	Good	Incipient checking
Terra Cotta	SA5239	10	"	Good	Incipient checking. Bloom over Film
Black	SA5291	10	Good	Good	Checking 7
Black	SA5239	10	Good	V.Good	Checking 5
Light Tan	SA5291	10	Red & Deep	Poor	Slight bloom on film Checking 9/8
Light Tan	SA5239	10/9	"	V.Good	Slight bloom on film checking 7
Chocolate	SA5291	10/9	Good	Fairly Good	Slight bloom on film Checking 7
Chocolate	SA5239	10/9	Good	Good	Slight bloom on film Checking 7

Conclusions All shades in present range have satisfactory light fastness with the exception of Hayfair Cream. Other exposures have shown that this shade should contain antimony as sole white pigment. This involves considerations of supply & price.

SA5239 is rather better for gloss retention than SA5291 but the differences do not justify the marketing of two lines of house or coach paint on the two mediums.

The blistering observed is unusual in this type of exposure & must be due to the abnormally wet year (plus insufficient protection of the metal surface by the primer) ?

BY W.H. STITSON & G.GALE.

Report on investigation to produce a reasonable oil type substitute for "Kemplastite" stoving enamel.

The first stage was to try out our standard oil type stoving medium V 5144 with Kemplastite pigmentation at various times and temperatures. It was found that at 200° - 220°F and both ½ and 1 hour stoving times gave reasonable gloss and colour retention on pale tints but a very soft film which tended to become generally rather brittle after 7 - 10 days. Above 220°F the pale shades tended to discolour although the initial hardness of film was improved.

V 7027 and V 4407 were then tried, both separate and blended, at ½ hr. at 250°F but the films produced were very short and discolouration took place, particularly with V 7027.

We decided to work on V 4407 and blends of this and V 5929, V 5438 and (118) were tried with far more promising results, particularly at 1 hour at 200° - 220°F. Higher temperatures gave discolouration and a reduction of the stoving time tended to produce an initial softness.

V 5438 was also tried alone and blended with (118) but with less promising results. Alone, when stoved below 250°F, it was very soft although colour retention was good. Some of the blends with (118) were harder but were still soft through after 7 - 10 days.

On examination of a summary of our tests it was decided to give a trial to the combination of 2 Parts V 4407 and 1 Part V 5929. A small works batch was put through on flatstone mill grinding and larger tests carried out. Samples were handed to Technical Service for their opinion. The main snag with our material was that it sprayed wetter than Kemplastite and in order to obtain a solid job one tended to apply too heavy a coat which produced bad runs and tears during stoving. It therefore became necessary to replace all or part of the White Spirit in the varnish and a trial was made replacing half the thinner in the V 4407 with (522). This gave very much better results so a larger works batch was made of a Pastel Green shade. This produced a far more favourable report from Technical Service although results were of course no comparison with Kemplastite in toughness. It was also found that the enamel tended to become somewhat brittle on ageing 7 - 10 days.

We also considered using a white undercoat for the paler and less opaque shades and although Rx188 White Undercoat gave fair results, ES6119 Air Drying Kemitone White Undercoat gave even better results on adhesion, so that the final set up has become as follows.

Pigmentation generally on Kemplastite basis but in a few cases, namely the very pale and less opaque colours, it has been increased with the addition of lithopone.

To be sent out in thin brushing condition 50-60 secs. F/C 4 and to take 10% of thinner V7119 for spray.

Stoving time 1 hour at 200°F which can be varied to a higher temperature at a shorter time although attention must be paid to colour change on the paler and brighter shades.

Air drying in 4 hours (dust free) and hard in 8 hours. ES6119 recommended as undercoat for the pale shades when stoved at 1 hour at 200°F.

V4407 has been adjusted in formulation to contain equal parts by volume (522) and (18) and this has received the new reference V7519.

We are however not entirely satisfied with this formula, and tests are still being carried out to effect an improvement in elasticity on ageing. Some promising results have been obtained on a Penta resin DCO type medium 387/100/1 both alone and blended with our V7519. Work is still proceeding so a full report cannot be given yet.

Tests are also being carried out on our present formulation to determine the stability in a dipping tank.

The existing formula for Dymard medium being high in ester gum content viz., 2 parts V7519 and 1 part V5929 stoving tests were done on the Pentalyn varnishes made.

V 7519.

300 lbs.	R ₁ 258
6 1/4 "	R ₂ 14
25 galls.	(81)
23 "	(18)
23 "	(522)
1/2 "	V 4031

Y = 94 galls.

V 5929.

168 lbs.	R ₁ 259
45 galls.	V 3098
50 "	(18)
1/2 "	V 4031
1 1/2 "	V 3906

Y = 107 galls.

Stoving 1 hour 200°F irrespective of oil length.

Penta Grade	Film	Package stability.
387/71/1	Tough, non mar.	Skins.
387/71/2	" slight mar.	"
387/73/1	" mar	Signs of skinning.
387/74/2	" mar	No skin.
362/181/2	Sl. brittle, mar	" "
362/182/1	V. sl. brittle, mar	" "
362/183/1	" " " "	" "
362/184/1	Brittle	" "
V4046 Std.	Tough, sl. mar	Edge skin.
V4046/4	" non mar	" "
V4046/5	Soft through	Sl. edge skin.
362/189/1	Tough, mar	No skin.
362/190/1	" sl. mar	" "
355/194/4	" non mar	" "
	Bad surface rivel.	" "
355/194/5	Tough, non mar	" "
	Bad surface rivel.	" "

DYMARD STOVING ENAMEL P. 121 (Contd)

S. 54

355/194/6	SA 5291	Tough, non mar Sl. edge rivel.	No skin.
355/196/3	G	V. tough, non mar Sl. edge rivel.	" "
355/196/4	X	V. tough, non mar Sl. edge rivel.	Sl. skin.

362/190/1, 387/71/1 and V4046/4 were selected for trial in Dymard. White viz.,

175 gms.	R: 713
14 "	R: 163
7 "	C 1686

810 ccs.	Varnish
----------	---------

Results:- 389/65/1 on 362/190/1. V. tough and flexible. Good gloss. Tougher than standard and more flexible.

389/65/2 on 387/71/1. V. tough and flexible. Tendency to rivel.

389/65/3 on 4046/4. Tough and flexible. Sl. clam.

362/190/1 is best, but supplies not being sufficient to employ this as the sole vehicle makes it necessary to conduct further blends.

362/190/1

300 gms. Pentalyn X	Resin and (45) taken to
240 " (45)	565°F in 1/2 hr. Hold for
240 " (194)	5 mins. Add (194). Take
700 " (18)	to 565°F in 20 mins. Hold
25 ccs. V4033	20 mins. Cool to 450°F
8.5 " V4031	and thin.

Arising from this V7524 has been fixed using lighter solvent.

V7524 Medium for Dymard.	To be used equal parts with V7519.
164 lbs. Pentalyn X	Resin and (45) to 565°F in
15 galls. (45)	1/2 hr. Add (194). Take to
15 galls. (194)	565°F in 20 mins. Hold 20 mins.
27 galls. (18)	Cool and thin.
27 galls. (522)	

Y = 100 galls.
Wt. per gl. 9 lbs.
Solids 50%
Viscosity 2-2.5 p. @ 25°C.

V7519 Dymard Blending Medium	Heat (81) to 500°F. for body.
300 lbs. R: 258	Add half the 258, followed by
6 1/2 " RM 14	14. Hold at 500°F. for string
25 gls. (81)	Check with rest of 258 and heat
23 " (18)	to 500°F. Hold for hard pill
23 " (522)	
1/8 " V4031	

Y = 94 galls.
Wt. per gl. 9.3 lbs.
Viscosity 1.5 p. @ 25°C.

Following work on Pentolyn varnishes the following blends were made up and tested by pouring on degreased tin-plate and stoving 1 hour @ 200°F.

Equal parts	387/100/1 with V7519.	Good tough film. Slight tendency to rivel.
" "	387/100/1 with V5929.	Soft film. Slight tendency to rivel.
" "	387/100/1 with V5438.	Soft film. Rivals badly.
" "	387/100/1 with V7514.	Tough film, but rivals.
" "	387/100/1 with V6960. (lab. manufacture)	Soft film. Rivals very badly.

The 387/100/1 used in the above tests had an addition of 2% V3906 (Lead and Manganese drier) as a test of 387/100/1 undried was still very tacky after 1 hour stoving at 200°F.

The following blends were tested in the same manner as outlined above.

Equal parts	387/100/1 plus 1% V3906 with V7519.	Good tough film. No signs of rivelling.
" "	387/100/1 undried with V7519.	Good tough film. No signs of rivelling.
One part	387/100/1 undried with 2 parts V7519.	Good hard film, but rather brittle. No sign of rivelling.

A test of 387/100/1 plus 1% V3906 was also made. This gave a tough film but it rivelled badly. The blend of equal parts undried 387/100/1 (now V7524) with V7519 was chosen as being the most promising combination and a lab. batch of R6406 made up on the following formula.

175 lb.	713	
14 "	163	
7 "	C1686	
45 galls.	V7519	) formulae given above.
45 "	V7524	
5 "	(522)	

Brush applied against our present standard on bare steel and stoved 1 hour @ 200, showed the new formula to give better gloss and is far more elastic on ageing than our old one. As soon as we are able to carry out spraying operations further tests will be made and its stability in a dip tank will be determined.

A dip tank test has been carried out with our present formula giving the following results.

Running time 8 hours. The second solids taken after material had been thinned to original viscosity.

Ref. No.	Viscosity at commencement of test.	Viscosity at end of run.	% Solids at commencement of run.	% Solids at end of run.
Lx6408	26 secs.	45 secs.	64.35%	58.5%
Rx6408 + 1% V3452	25 secs.	35 secs.	63.8%	63.5%

COLOURED STENCILLING PAINTS IN SOLID FORM FOR WAREHOUSE USE.

BY F. EVERETT. (ITEM P.122)

1. Yellow. The Warehouse stencilers have for some years been using yellow stencil paint in the form of solid cakes, which have been made in the laboratory on the following formula:-

3 parts R_x 258
1 part R_x 7002 (C.Dept. Yellow Tinting Paste)

Melt Ester Gum at about 380°F. Remove from fire and pour in yellow tinting paste. Pour mixture into flat tins while still hot.

As these cakes have proved more convenient and economical in use than the liquid stencil paints used for other colours, the laboratory was requested to develop similar cakes in Red, Yellow and Black.

2. Red. It was first attempted to make red cakes by substituting a Helio Red Tinter for the R_x 7002 in the above formula, but it was found that the Helio Red could not withstand the temperature of the molten Ester Gum. It was therefore necessary to make a special tinter on a heat-resisting pigment.

Red Tinter. 70 g. C3119 (Ba salt of Perm.Red 2B)
250 cc. (2)
150 cc. (23)

Red Stencil
Cakes. 2700 g. R_x 258
420 g. Red Tinter (above)
150 cc. (2)

Made as for yellow cakes. The extra (2) was found necessary to soften the cake.

3. Black. A mixing was made as follows:-

2700 g. R_x 258
650 g. R_x 7022 (C Dept. Black Tinter)
200 cc. (2)

This was tried out by the Warehouse but had insufficient covering power and it was necessary to add 700 g. Oil Black dye 35395 ex Williams (Hounslow) to obtain sufficient opacity.

4. White. C. Dept's White Tinting Base B3553 was found to be quite useless for this purpose, being made on lithopone, and having far too poor an opacity. Laboratory grindings have been made using R_x 713 ground in (2) but the cakes so far produced are still insufficiently opaque and mixings are now being made to incorporate larger percentages of R_x 713.

WAX POLISH. V. 3.

by W.H.Stitson and C.Stevens.

The Guardeese Wax Polish is in production on the following formula:-

Wt: 28

52 lbs. Special Polish Wax No. 736
ex Poth Hille.
18½ galls. (23) White Spirit

Max. possible theoretical yield 200 lbs.

This polish gives a harder and more water resistant film than Super Wax ex 590.

Anti Skinning Agent. V.30 by J.H. Dale.

A sample of 2:4 Dimethyl - 6 tertiary butyl phenol (24M6B) received from I.C.I. Billingham was tested as an anti skinning liquid.

V 4046 (a spar varnish) which is known to skin under certain conditions in a narrow tube was used as control.

A solution (a) 10% 24M6B in (23), by volume, and a solution (b) of V3452 10% Guaiacol were used.

1% and 2% of solutions (a) and (b) were added in turn to V4046 and the four solutions, thus obtained, left in contact with air against a control.

The additions of 1% and 2% of solutions (a) and (b) resulted in concentration of 0.1% and 0.2% of the anti oxidants in the varnish.

V4046 alone skinned over between 24 and 48 hours.

0.1% addition of 24M6B resulted in the retardation of skinning for 5 weeks when a skin appeared at the edge. The skin spread rapidly and within 4 days had spread to the centre.

0.2% addition of 24M6B and 0.1% and 0.2% additions of Guaiacol showed no skin after 25 days.

A skin appeared on the V4046 containing 0.2% 24M6B after 50 days.

No skin was present on either of the Guaiacol additions.

Films were poured of the four solutions against the control.

After 6 hrs. drying
films were as follows:

After 18 hrs. films
were as follows:

Control	Hard set, slight tack.	Slight clam.
V4046 +		
0.1% 24M6B	Surface set, strong tack.	Slight clam.
V4046 +	Surface set, very	Slightly more
0.2% 24M6B	strong tack	pronounced clam.
V4046 +		
0.1% Guaiacol	Wet	Very tacky.
V4046 +		
0.2% Guaiacol	Wet	Very tacky.

Conclusion.

24M6B appears to be less effective as an anti skinning agent than Guaiacol weight for weight. Its effect on the drying is correspondingly less.

It is a poisonous liquid. Guaiacol is toxic to a certain degree.

The results obtained justify works trials in place of the Izal phenoloids used as a Guaiacol substitute.

RUBBING COMPOUNDS V. 31.Guardeese Cleaner.

by W.H. Stitson and C. Stevens.

Work has been carried out to improve the stability of the wax emulsion used in the preparation of the cleaner. The formula adopted is:-

Wx 101 Emulsion for Guardeese Cleaner.

Rx 387	105 lbs.
(18)	21½ galls.
(59)	55 lbs.
Water	62 galls.
Triethanolamine	14 lbs.
Yield	100 galls.
Wt/gln.	9.6 lbs.

1. Heat wax, thin with White Spirit, add cleic acid and use at 230°F.
2. Heat water and triethanolamine to 180°F in agitator.

Add 1 to 2 and emulsify in agitator.

Wx 59. Guardeese Cleaner.

Rx 381	560 lbs.
Rx 65	52 lbs.
Rx 93	1½ lbs.
Rx 742	4½ lbs.
Rx 317	11½ lbs.
Rx 722	96 lbs.
Wx 101	157 galls.

Yield 1 ton

Make in edge-runner.

Keeping tests have been carried out on the cleaner to determine whether 0.5% borax is sufficient to prevent mould growth.

After 4½ months at 40°C.

Blank showed fine growth around the edges.

0.5% borax	- no mould
0.1% Mercuric chloride	- slight black mould
0.5% Sodium pentachlorophenate	- no mould
0.5% Sodium ortho phenyl phenate	- no mould

Supplementing the work done with penta-erythritol in alkyds, reported under item P 82, experiments have been carried out with the Hercules range of Pentalyn Resins and Gray Valley M.R.107.

The resins examined were:-

- Hercules Pentalyn G. This is a maleic modified penta-erythritol resinate claimed to have a slight dispersing effect on drying oil polymers.
- Hercules Pentalyn L. This is a phenol modified version which is very reactive and is useful for imparting high viscosity to varnishes made on low viscosity oils. Hardness is a special feature and it is claimed that Pentalyn L is useful in spar varnishes being better than modified phenolics but somewhat inferior to pure phenolics.
- Hercules Pentalyn X. Is a highly maleic modified penta-erythritol resinate which like L has little effect on the dispersion of oil polymers but is useful in coats with soft oils.
- Gray Valley
Grayvallas MR 107. This is an unmodified penta-erythritol resinate similar to Pentalyn A. Pentalyn A is not at present available.

The above resins were incorporated in a variety of formulae. Remarks concerning manufacture are given separately on the following pages.

Media were prepared, using the V 5438 type formula and replacing the ester gum by Pentalyns M, G, and X, ex Hercules and Crayvallac M.R. 107 from Cray Valley Products.

The following formula was used:-

448 grms. Penta ester
500 ccs. (81)
300 ccs. (45)
1000 ccs. (23)
20 ccs. V4031.

Manufacture. Heat penta ester, all (81) and 100 cc. (45) to 560° F. in $\frac{1}{2}$ hour and allow to rise to 580° F. Check with balance of (45), cool and thin.

Examination of media. The following results were obtained:-

Resin used	Pentalyn G	Pentalyn M	Pentalyn X	Cray- vallac MR 107	Ester Gum
Colour	6 $\frac{1}{2}$	7	6 $\frac{1}{2}$	7 $\frac{1}{2}$	
Viscosity 25°C.	1.4 p.	6.1 p.	2.5 p.	0.9 p.	
Acid Value	6.68	6.61	6.79	7.96	
Solids %		60.16	62.40		
<u>Drying:-</u>					
5 hours.	Surface set, fairly tacky	Surface set tacky	Frosts badly	Surface set, fairly tacky	Surface set, strong tack
Overnight.	Tack free Harder	Tack free Harder		Tack free Harder	Tack free
24 hr. water immersion of films A.D.	very slight	very slight clouding,	very slight clouding,	very slight clouding	
3 days.	clouding	slight blistering.		very slight blistering.	

Conclusions.

Pentalyn G in this instance gave a similar viscosity to ester gum, Crayvallac M.R. 107 was lower whereas Pentalyn M caused the greatest body. It was not found possible with Pentalyn X to produce a V5438 type of varnish free from frosting tendencies and grade X also had this tendency to a lesser degree.

Pentalyn G appears to be a suitable substitute for use in V5438.

All the media were repoured approximately two months after making. Those on Pentalyns G, M & X all frosted badly, whereas the varnish on Crayvallac M.R. 107 did not show this defect. The particularly cold weather prevailing at the time may have contributed to this failure.

PREPARATION OF PENTA-ERYTHRITOL ALKYDS. By D.E.Star.

Two formulae of this type i.e. SA 5401 and SA 5541 were checked in the laboratory to determine their suitability for large scale manufacture and what losses might be expected. Both are alkyds of approximately 60% oil length and comparable with SA 5291.

SA 5401.

The following formula and procedure were used:-

200	grms.	731	
120	"	732	penta-erythritol ex I.C.I.
216	"	(965)	
181	"	V4201	3 poise stand oil
630	ccs.	(18)	
370	"	(16)	

Manufacture. Take all except thinners to 460° F. in 47 mins. and hold 1 hour with vigorous mechanical stirring and moderate CO₂ blow. Then increase CO₂ blow and hold for rubbery bead with 10 inch string - about 50 mins.

Total actual yield = 1400 ccs. (includes 7% (16) extra to standard formula) equivalent to 1310 ccs. on the original formula.

The RM 732 used had Ash 2.07% and Penta-erthritol content 83.5%.

SA 5541.

A	200	gms.	731	
B	120	"	732	penta-erythritol ex Chemical Supplies
C	210	"	(965)	
D	180	"	(2)	
	600	ccs.	(18)	
	100	ccs.	(16)	

Manufacture. Take, A, B & C to 400° F. in ½ hr. and hold ¾ hour. Add D, take to 475° F. in ¾ hr. and hold ¾ hour for 4 inch string, CO₂ blow and mechanical stirring throughout, increased during last stage.

Total actual yield = 1276 ccs. (includes 8% (16) extra to standard formula), equivalent to 1180 ccs. on the original formula.

The RM 732 used had Ash 0.84 % and penta-erythritol content 74.2%.

Remarks.

SA 5401. This medium was clouded due to impurities in the penta-erythritol used. Most of the foreign matter remained as a hard cake in the bottom of the flask used for the preparation, but the suspended matter did not settle on standing nor was it readily removed by centrifuging.

The yield represents a total of 87 galls. on the standard batch, compared with the theoretical yield of 96 galls. or the actual amount obtained in the Works of 72 galls.

SA 5541. This was quite bright and free from sediment. The time taken to reach the end point from 475° F. (i.e. ¾ hr.) was considerably less than that specified in the standard instructions (1 - 1½ hrs.)

PREPARATION OF PENTA-ERYTHRITOL ALKYDS (CONTD) by D.E. STAR.

The yield corresponding to 78½ galls. on the standard batch compared with the calculated yield of 80 galls.

Constants of media.

These were as follows. SA 5291 figures are given for comparison:-

	SA 5341	SA 5401	SA 5291
Visc.-poises } @ 25° C.	4½ (3½)	5 (3½)	3 - 4
Colour	8 (clear)	6½ (cloudy)	6 - 7
Solids	52.3% (60%)	44.7% (50%)	58 - 60%
A.V.on solids	45 (15)	30 (30)	30 - 37

(Figures in brackets are those supplied by Research Dept.)

Drying plus 2% V 3906 and 2% V4031. Both penta alkyds were approximately equal to SA 5291 at all stages. In the case of SA 5401, the film was somewhat seedy.

Water Resistance. Both penta alkyds gave a somewhat less cloudy film than SA 5291 when immersed in water, but they were rather weaker than the latter.

Conclusions. The I.C.I. penta-erythritol gives alkyds which are cloudy and difficult to clarify, whereas the purified material from Chemical Supplies Ltd. is free from this defect. The general properties of penta alkyds of 60% oil length differ from a corresponding glycerol alkyd insofar as viscosity is higher for a given solids and acid value.

(cc)

SHORT OIL VARNISHES.

By J.H. Dale. S.54.

Three resins from the Hercules Powder Co. Pentalyn G. Pentalyn M and Pentalyn X and one from Gray Valley Products (MR 107) which are of the penta-erythritol - ester gum type, were used in the experiments. Varnishes of the V6592 type were made according to the cooking schedules suggested for the respective resins.

1. Pentalyn G - 600 gms. 362/181/2
(45) 600 ccs.
(18) 850 ccs.
V4031 20 ccs.

The oil and half the resin were heated to 585° F. in one hour, the rest of the resin added and top heat regained in 15 mins. The mixture was cooled, thinned to approximately 2 poise and driers added.

2. Pentalyn M - 600 gms. 362/182/1
(45) 600 ccs.
(18) 1300 ccs.
V4031 20 ccs.

The oil and resin were heated to 565° F. in one hour, cooled, thinned to approximately 2 poise and driers added.

3. Pentalyn X - 600 gms. 362/183/1
(45) 600 ccs.
(18) 1200 ccs.
V4031 20 ccs.

The oil and resin were heated to 565° F. in one hour, cooled, thinned to approximately 2 poise and driers added.

4. M.R. 107 - 600 gms. 362/184/1
(45) 600 ccs.
(18) 700 ccs.
V4031 20 ccs.

The oil and resin were heated to 565° F. in one hour, cooled, thinned to approximately 2 poise and driers added.

V 6592 used for comparison:-

- | | | |
|--------|---|-----------|
| RM 258 | - | 600 gms. |
| (45) | | 600 ccs. |
| (18) | | 1040 ccs. |
| V 4031 | | 20 ccs. |

RM 258 and (45) heated to 500° F. Hold ¼ hour, cool thin and add driers.

SHORT OIL VARNISHED (CONTD.) BY J.H. DALE. S.54.

The varnishes thus produced were tested and the following results obtained:-

Varnish on.	1 Pentalyne G.	2 Pent.M. X	3 Pent. X	4 MR107	V6592
Viscosity	2 poise	2 poise	1.65 p.	2 poise	0.5 p.
Colour	7	6½	6½	7½	5½-6½
Acid Value	7.7	10.5	8.2	9.8	7-10

Films of the varnishes (thinned to 0.5 poise) were poured against V6592.

Varnish on.	1 Pentalyne G.	2 Pent.M. X	3 Pent. X	4 MR107	V6592
After 3 hrs. Surface	Surface set Fair Tack	Surface set Fair Tack	Surface set slight Tack	Surface set Fair Tack	Surface set Strong Tack
After 20 hrs. Very	Very hard	Very hard	Vary hard	Very hard	Hard

Judging from the amount of thinners taken, Pentalyne M and MR 107 are the hardest and most reactive, Pentalyne G is soft and yields varnishes of high solids. The X grade is intermediate. Both M & X carry more thinners than ester gum.

Pentalyn M.

As was expected, direct replacement of RM 258 in the standard V4046 formula was unsatisfactory, gelling occurring within one hour at 480° F. Even using equal parts (81) and (2) instead of 2 parts (81) to 1 part (2) still led to gelling. The use of 1 part (81) to 2 parts (2) gave a satisfactory result initially but the resulting varnish was unstable in package.

Pentalyn X.

Gelation occurred also when RM 258 was replaced by Pentalyn X in the standard V4046 formula, but a satisfactory result was obtained using equal parts (81) and (2).

Pentalyn G.

This resin likewise led to a gelation but with equal parts (2) and (81) a satisfactory varnish was obtained after 3½ hrs. total cooking.

The drying of both these varnishes, despite the reduced wood oil content, was satisfactory and equal to V4046 standard. The varnish on Pentalyn X was slightly harder over-night than that on Pentalyn G and standard.

Exposure panel 1066 was prepared.

(69)
VARNISHES MADE ON HERCULES POWDER CO. FORMULAE
BY J. H. DALE. S.54.

Pentalyn resins G, M, X from Hercules Powder Co. and Gray Valley Products' MR 107 were used in the formulae quoted by the Hercules Powder Co. in their pamphlet. MR 107 was used in place of Pentalyn A.

The quantities were the same in all formulae, the resin and making instructions being the only variables.
Type formula is as follows:-

Resin	300 gms.
(45)	240 "
(194)	240 "
(18)	700 "

Making instructions were as follows:-

For MR 107 362/192/1 and 362/192/2.

$\frac{1}{2}$ of the resin, the (45) and (194) were taken to 585° F. in 48 mins. held for 1½ hrs. when the make gelled. The instructions called for holding for 2 hrs. 50 mins. A repeat of the experiment had exactly the same results.

For Pentalyn G. 362/191/1 and 362/191/2.

$\frac{1}{2}$ of the resin, the (45) and (194) were taken to 585° F. in 1 hr. held for 1 hr., when the make gelled. The instructions called for a holding period of 1½ hrs. A repeat of the experiment gave the same results.

For Pentalyn K. 362/189/1.

The resin and (45) were taken to 565° F. in 50 mins., held for 5 mins., the (194) added and the whole taken to 565° F. in 26 mins., cooled to 450° F. and thinned. Driers V4033 - 25 ccs. and V4031 - 8.5 ccs. were added.
Visc. @ 25° C. 3 p.
Colour 7

For Pentalyn X. 362/190/1.

The resin and (45) were taken to 565° F. in 47 mins., held for 5 mins., and the (194) added. The whole was taken back to 565° F. in 20 mins., held for 20 mins., and cooled to 450° F. and thinned. Driers V4033 - 25 ccs. V4031 - 8.5 ccs. were added.
Visc. @ 25° C. 2.75 p.
Colour 6½

The two successful makes have been exposed.
Exposure panel 1065.

The Pentalyn M Varnish was surface set and dust free in 7 hrs. and was hard-dry overnight but with a pronounced clam.

The Pentalyn X Varnish was surface set and dust free in 6 hrs. hard overnight free from clam, and tougher. This varnish shows an advantage over V6960 which is of similar composition but an ester gum and is approximately equal to V 4046 for which V6960 was a war-time substitute.

Further work is indicated, water absorption tests etc. using Pentalyn X in this class of varnish.

MODIFICATION OF ALKYDS WITH PENTALYNS G AND X

by F. EVERETT.

Three methods suggested by the Hercules Powder Company were tried out.

1. Addition of Pentalyn at end of varnish cook.
355/194/4 and 355/194/5.

A sample of SA 5291 base was removed from a works pot at the end of the cook. To portions of this were added (a) 20% Pentalyn G and (b) 20% Pentalyn X. Each was then thinned with the appropriate amount of thinner and driers. A sample was taken from the same batch of SA 5291 when thinned in the works, for use as control. 355/194/6. Exposed 27/1/47. Panel No. 1063.

2. Addition of Pentalyn as solution in White Spirit.

50% (v/v) solutions of Pentalyns G and X in (18) were made by churning. These dissolved eventually but not very easily.

Additions of these solutions were made to SA 5291, equivalent to 20% Pentalyn on the non-volatile base of the varnish. 355/196/3 and /4. Exposed 27/1/47. Panel No. 1064.

3. Addition of Pentalyn during grinding of paint.
355/195/1 and / 2.

Samples of Pompeian Super-Synthetic White were placed in ball-mills and additions of Pentalyns G and X were made equivalent to 20% of the non-volatile portion of the medium. The ball-mills were run overnight to break up the resins and and dissolve them in the paint. Exposed 27/1/47. Panel No. 1067.

There was no noticeable effect on the drying of the varnishes on adding the Pentalyn by either method. After a week 355/194/4 was not quite as tough as the others. All varnishes gave equal gloss to standard and showed good build and face before exposure.

The two white enamels containing Pentalyns showed slightly improved initial gloss over the standard R 6888.

Comparisons of Beck Koller R 1028/2/5 (now Beckosol 1428) with Paralac 10 and SA 5401. (60% oil length)

Beck Koller sample and Paralac 10 were thinned with (2S) to a viscosity of $2\frac{1}{2}$ p. @ 25°C. SA 5401 had a viscosity of $1\frac{1}{2}$ p. To each varnish was added 2% by volume of V4031 and V5906.

Property	Sample	Paralac 10	SA 5401 experimental (insufficient sample)
Sp. G.	0.933	Insufficient sample	Insufficient sample
Colour, original	5 $\frac{1}{2}$	5	3 +
Colour, after thinning	5 +	1 $\frac{1}{2}$	
Viscosity, original	12 p.	Very viscous.	14 p.
Viscosity, after thinning	$2\frac{1}{2}$ p.	$2\frac{1}{2}$ p.	
Solids, original	60.5%	100%	47.6%
Solids, after thinning	48.6%	55.6%	
Acid Value, original	1.1	10.7	25.6
Drying time of thinned materials			
S.S.	2 hrs.	2 hrs.	2 hrs.
H.S.	3 "	3 "	3 "
T.F.	4 "	4 "	Slightly tacky after 24 hrs.
Films.	Clear, good gloss, cheesy after 8 hrs. Hard after 16 hrs. slightly harder than Paralac 10	Clear good gloss, cheesy after 8 hrs. Hard after 16 hrs.	Clear, gloss similar to others, pronounced tack after 8 hrs; tacky & soft after 16 hrs.
A set of poured films after exposure for 8 hrs. were immersed in water overnight.	Milky film weakened. Slightly better than Paralac 10 Regained original condition on standing	Slightly milkier, slightly weaker than Beck Koller. Regained original condition on standing	Very milky & film very weak Film remained permanently opalescent & bubbled.

Comparison of Beck Koller R 1028/2/5 with Paralac 10, SA 5406 (65% oil length) & 327/68 (Mr. Gray).

The Beck Koller & Paralac 10 Samples were the ones used for the work reported above. They had stoutened and were thinned with (25) to 2½ poises. The sample of SA 5406 was treated with 2% by vol. V4031 & 2% V3096, and then thinned to 2½ poise. The 327/68 had a viscosity of 2½ poise. This was not thinned but the usual drier addition was made.

	Beck Koller	Paralac 10	SA 5406	327/68
Drying Time:-				
S.S.	1	1	1	1½
H.S.	1½	1½	1½	1½
T.F.	3½	3½	3½	4½
Film	Cheesy, but	Cheesy,	Cheesy	Cheesy,
After 8 hrs.	hardest	similar to		softest
		SA 5406		
" 24 "	Hard	Hard and	Hard &	Hard, but
	toughest	tough	tough	softer than
				others.
" 48 "	Hard, very	Hard and	Hard &	Hard but not
	tough	tough	tough	as tough as
				others

Conclusion.

SA 5406. Better than original sample 327/68, very similar to Paralac 10 but not quite as good as Beck Koller's sample.

These results confirm the belief expressed by the Research Dept. that Paralac 10 and Beck Koller 1028/2/5 are nearer 65% in oil length.

Formulae and Standard Figures:-

<u>327/68.</u>	R.M. 731	150 gms.	Acid Value 9.0
	R.M. 732	109.2 "	Solids 60%
	(385)	340.8 "	Viscosity 3-4
	(18)	416 ccs.	Colour 7-8
	(86)	60 "	
<u>SA 5401.</u>	R.M. 731	133 lbs.	Acid Value 30
	R.M. 732 I.C.	1.80 "	S.G. 0.92
	(385)	141 "	Colour 6½-7
	V4201	121 "	Viscosity 3-3½ p.
	(18)	42 gls.	Solids 50%
	(16)	18 "	

Formulae and Standard Figures

SA 5406.1st works batch of 327/68 above.

R.M. 731	150 lbs.	Acid Value	9.0
R.M. 732			
Rex Campbell	109 "	Colour	7-8
(365)	341 "	Viscosity	3-4 p.
(453)	10 "	Solids	60%
(18)	41½ gls.		
(86)	6 "		

Water Resistance of SA5406 compared with Beck Koller
H1028/2/5 and Paralac 10.

Dried films of these materials were immersed in water at room temperature for 44 hours. Beck Koller's sample and Paralac 10 were unaffected. SA 5406 was milky and the film wrinkled at the edges. After 24 hours this film had not regained its original appearance, but remained wrinkled at the edges and slightly orange peeled on the surface. film strength had been regained and the milkiness had disappeared.

Conclusions. It is apparent that Penta-erythritol Rosin Esters cannot be substituted directly for rosin ester in an effort to make last minute economies in glycerine usage. The above work, incomplete as it is, illustrates the variations in cooking procedure for each of a number of varying types of varnish.

The use of Penta Esters of Rosin, straight or modified, do not greatly reduce the drying time of an ester gum varnish. Hardness is increased especially by the use of Pentalyn. X.

The use of these resins in Stoving Varnishes is reported separately as Item P.121.