JOURNAL of the OIL AND COLOUR CHEMISTS' ASSOCIATION



Vol. 44, No. 12

December, 1961

TRANSACTIONS AND COMMUNICATIONS

THE EXTERIOR DURABILITY OF PAINTS BASED (LITHOPONE & ZINC SULPHIDE	ON 	835
AN INVESTIGATION INTO DRYING PHENOMENA EXPERIENCED WITH ALKYD RESINS BASED ON SOYA BEAN C. G. T. Williams and P. D. Cox		851
THE LIMITATIONS OF LEACHING RATE DETERMINATIO OF ANTI-FOULING COMPOSITIONS A. Partington and P. F. Dunn	NS 	869
REVIEWS		882
CORRESPONDENCE		885
NFORMATION RECEIVED		888
SECTION PROCEEDINGS	1.09	889
NOTES AND NEWS	2	898
NDEX		908

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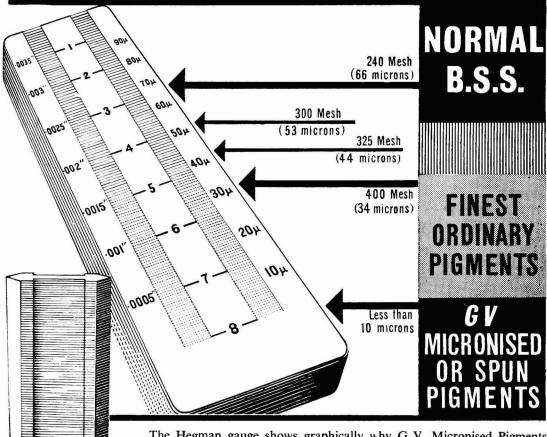
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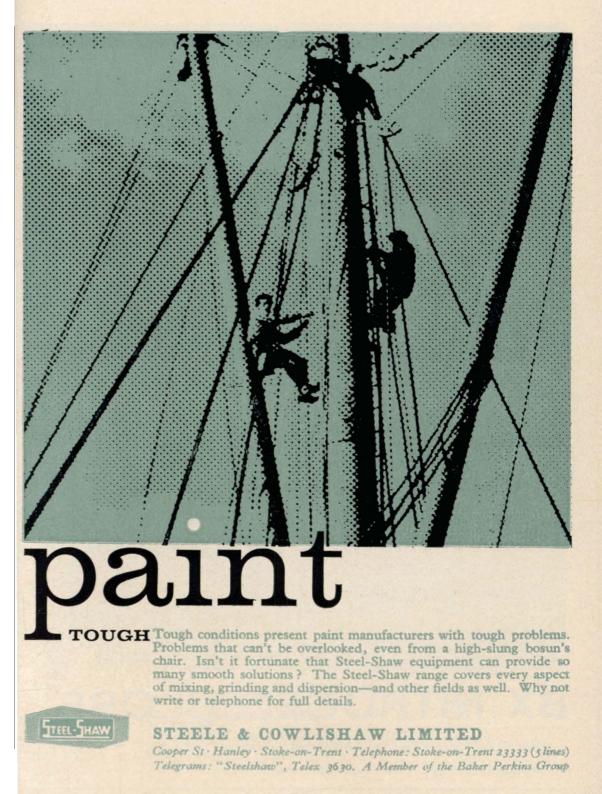


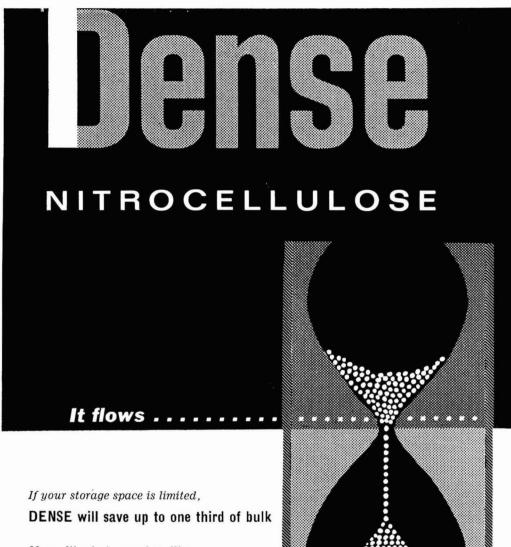
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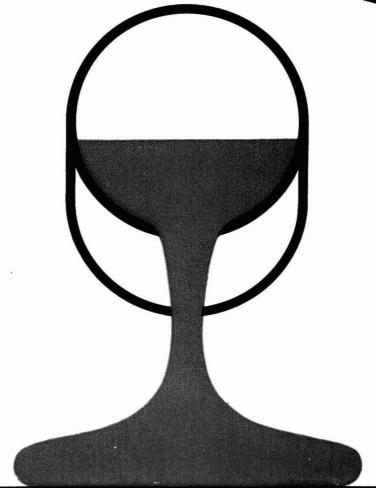
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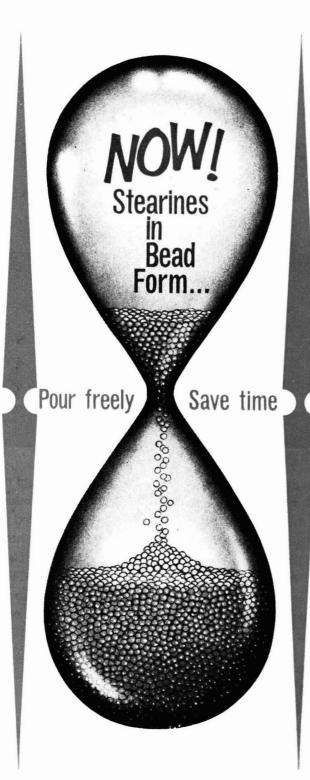
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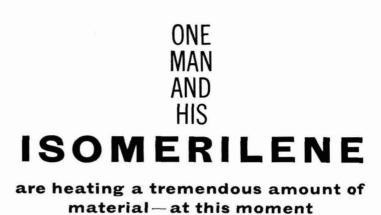
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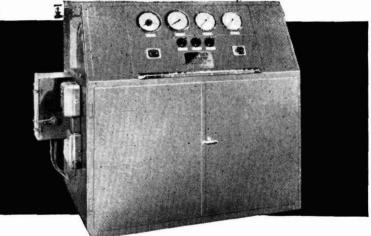


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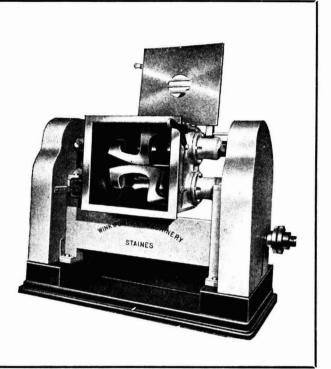
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SPECIFICATION (tentative) & PROPERTIES

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Volatile	Xylol
Viscosity	
Colour	
Acid No.	
Specific Gravity	0.949
White Spirit Tolerance	
Suggested thinner proportions :	
Xylol	100%

Recommended Driers: 0.01% Co, 0.04% Zr on base resin

Average Drying Time: Surface dry in 15-20 minutes. Stoving Schedule: 30 minutes at 140°C.

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OUTSTANDING PROPERTIES: Rapid touch-dry, economic curing schedules, chemical resistance, hard-ness combined with flexibility, good adhesion

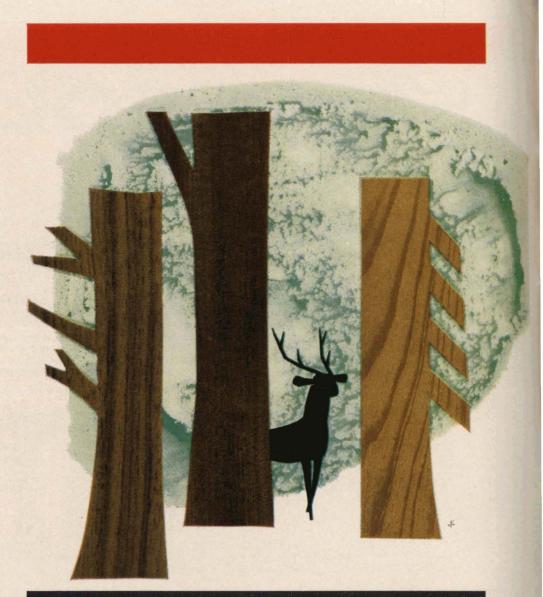
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Compa	tible with :	
Amino	Resins	Good

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Hexylene Glycol

By NEIL R. FISK

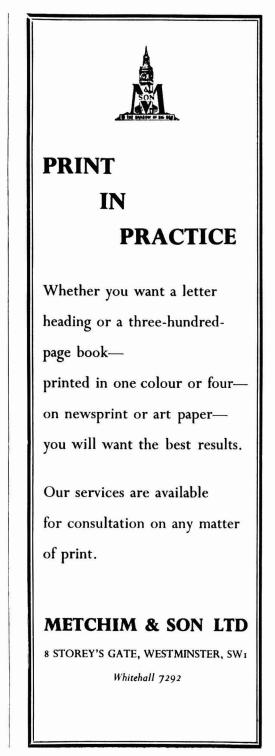
What is it? Although not to be found anywhere in a reference book recently brought out, the solvent itself is not unimportant: it aids coalescence, and hence levelling, of emulsion paints, and penetration of inks, and it was mentioned after Dr. Talen's stimulating paper on film formation at the London Section meeting on 10 October, and in one of Dr. Barakan's tables the following week.

Hexylene glycol properly so called is the 1 : 2-compound, that is to say, hexane-1 : 2-diol. But the hexylene glycol of industry is not that substance . . . nothing like it, in fact. One's next guess would be the 1 : 6, but, wrong again, for this is hexamethylene glycol. (Nobody makes either in bulk, by the way.)

So when I had to place the substance in Fisk's Paint Year Book and A-Z Buyers' Guide there was nothing for it but to ask one of the manufacturers, and then I was told at once that in reality it is 2-methylpentane-2 : 4-diol. In other words it is not really a hexylene glycol at all, but a branched-chain pentylene glycol, and, even at that, not the one that would be expected by a "pure" chemist.

The next thing was to ask the other manufacturers to identify their product, and each gave me the same answer as the first. So, naturally, we have called it 2methylpentane-2:4-diol in *FPYBBG*, but of course we have cross-referred it from "hexylene glycol" as well.

This is "just one of those things" to which I alluded in my little piece on homonymy and synonymy in the November J.O.C.C.A. There were scores of problems of that sort in the two-and-a-quarter thousand headings in the book, and that is why I am not yet quite ready to invite orders for it, although a fair number have come in, some from overseas.



December

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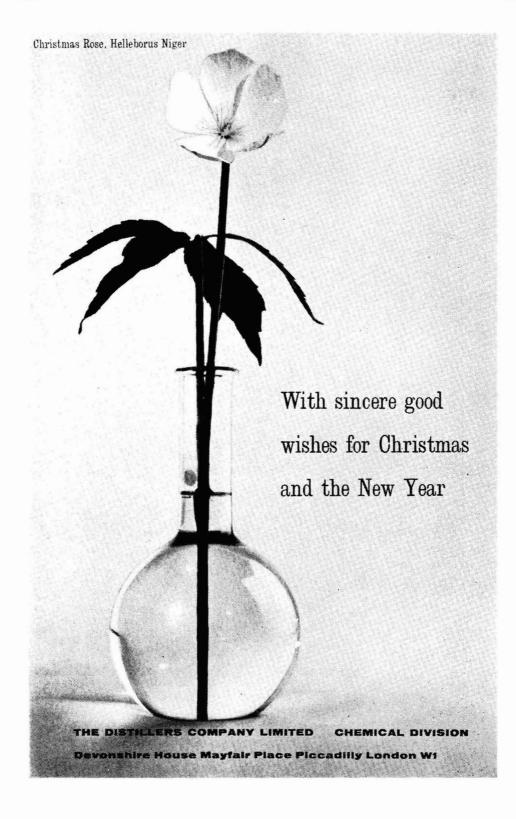
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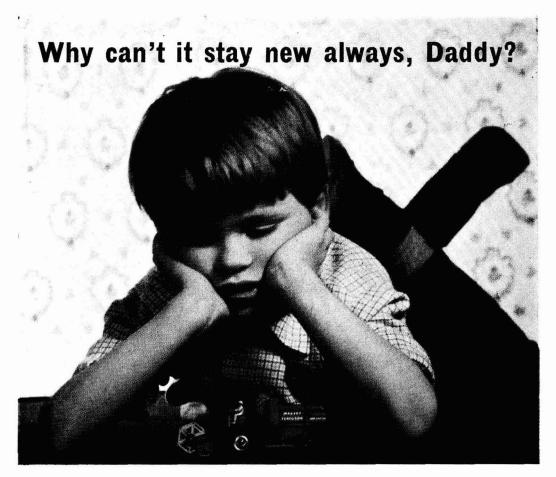


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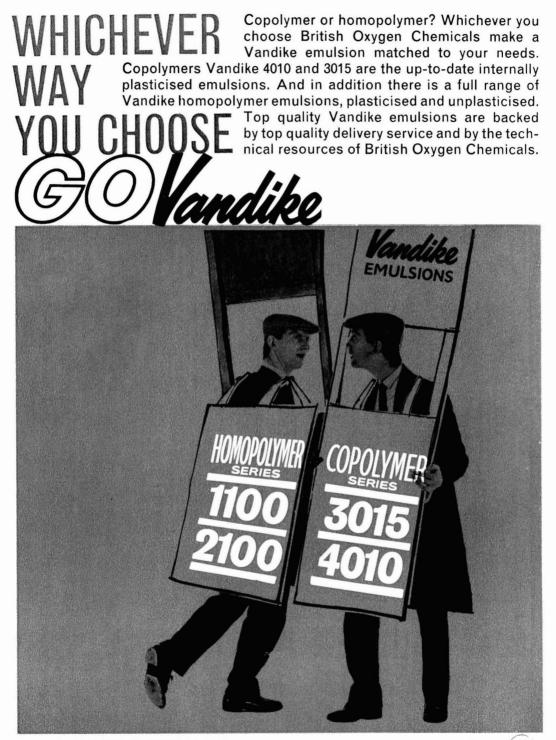
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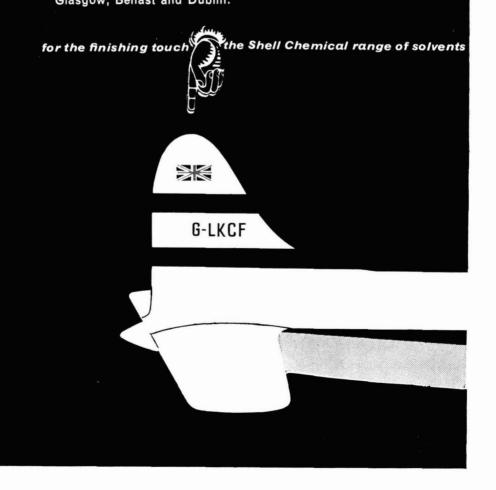
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xxviii



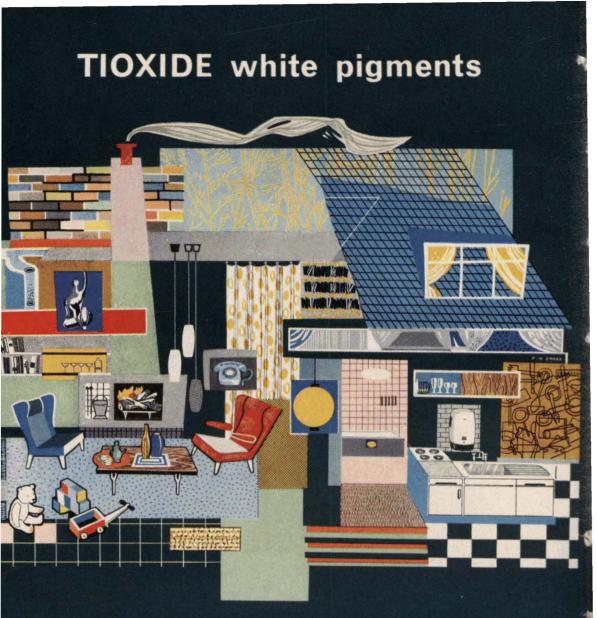
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INDEX	OF	АĽ) V E	RTI	ISERS
		в			
Badische, Anilin	n-& Soda-H	B Fabrik	A.G.		. xii
Beadel, James, Beck, Koller &	& Co. Ltd.		5		. Cover
B.I.P. Chemical	s Ltd.	ina) Li	a		xi xix
B.I.P. Chemical British Oxygen British Resin Pr British Titan Pr B Plastics Li	Co. Ltd				. xxiv
British Titan Pr	oducts Ltd	Ltd.	•		ii xxx
B.X. Plastics Lt	.d		•		iv
		С			
Ciba (A.R.L.) I C.S.R. Chemica	.td.				. xxviii
Consolidated Z	INC COLDOL	апоп і.	td.		. xv . xl
Corv. Horace.	& Co. Ltd.				. xxxii
Croxton & Gar Cyanamid of G				• •	. xxxix
Distillers Co. L	td. The	D			. xvii
English Clause I	avaring D	E ashin (P. C.	TAJ	
English Clays, I Esso Petroleum	Co. Ltd.	ocnin a	x Co.	Lta	. xxv
Fisk, Neil R.		F .			. xiv
Geigy Co. Ltd., Golden Valley Greeff, R. W.,	The .				. xili
Golden Valley	Colours Lte	d		• •	. !
Green, R. W.,	& Co. Ltd.	•	•		. xxxvi
Hercules Powde Heydon, Harolo	a r r	н			
Hercules Powde	r Co. Ltd.	rt		9 B	. xxxv . Cover
	,				
Imperial Chem	ical Indus	I trios I	1d (1	Jugatus	r
Division)	incar mous	····		Jyesiui	
		к			
Kestner Evapor	ator & En		Co.	Ltd	. viii
				Dia.	
Laporte Titaniu	m Ltd	L			. Cover
Lonabarc Ltd.					X
		М			
Metal Propeller Metchim & Sor	s Ltd				ix
Metchim & Sor	n Ltd	•	•	•	xiv
		N			
Novadel Ltd.	••	•	•	•	xxii
	-	0			
Oakley, H. G.,	& Co. Ltd	• •	•	•	xxi
		Р			
Paint, Oil & Co				а . 1	. xviii
Pascall Enginee	ring Co. Li	id			. xxxvi
D		R			
Resinous Chem	icals Ltd.		•		•• XX
Chander Ort	S. C. I	S			
Shandon Scient					. xvi . xxvii
Shell Chemical					
Shell Chemical Steele & Cowlis	shaw Ltd.	•			
Shell Chemical Steele & Cowlis Styrene Co-Poly	shaw Ltd.				. xxiii
Steele & Cowlis Styrene Co-Poly	shaw Ltd. ymers Ltd.				xxiii
Steele & Cowlis Styrene Co-Poly Union Carbide	shaw Ltd. mers Ltd. Ltd.	. U	•		xxiii
Steele & Cowlis Styrene Co-Poly	shaw Ltd. mers Ltd. Ltd.	U : :	•		xxiii
Steele & Cowlis Styrene Co-Poly Union Carbide	shaw Ltd. ymers Ltd. Ltd o. Ltd	U : : W		•	xxiii

JOURNAL OF THE OIL & COLOUR CHEMISTS' ASSOCIATION

Vol. 44 No. 12 December, 1961

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No. 12

TRANSACTIONS AND COMMUNICATIONS

The Exterior Durability of Paints Based on Lithopone & Zinc Sulphide*

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Summary

Co-operative exposure tests are made by members of the Zinc Pigment Development Association Technical Committee using 28/30 lithopone or zinc sulphide, mixed with leaded zinc oxide and micronised mica as the pigment. The media are based on linseed oil, phenolic and alkyd varnishes and exposures are made for a 26-month period, at 45° South exposure, in industrial, marine and rural environments.

Results indicate that, with white paints, serious faults other than loss of gloss and erosion are prevented by use of the mixed formulations. The addition of stable tinting pigments gives considerable improvement in the general performance of these paints. All 28/30 lithopone- and zinc sulphide-based paints are in excellent condition for repainting at the end of the exposure period and require only a minimum of preparation prior to recoating.

INTRODUCTION

Zinc sulphide pigments have for a long time been used with success by paint manufacturers in undercoats for both interior and exterior paint systems. They have not, however, found a similar acceptance as the main pigmentary constituents of exterior finishing paints, since their presence introduces a tendency towards rapid failure on weathering by chalking, checking and heavy erosion.

Assertions have appeared occasionally in the technical literature that this breakdown is due to oxidation of the zinc sulphide with the eventual formation of water-soluble zinc sulphate. The late Noel Heaton, in particular, perpetuated this assumption in his classical text-book¹ "Outlines of Paint Technology". Nevertheless, satisfactory proof that such a breakdown occurs in exterior finishing paints on exposure to natural sunlight and weathering has never been established.

One of the authors has, indeed, spent a considerable effort in endeavouring to oxidise zinc sulphide and has found that this material is extremely resistant towards breakdown of this type. Even uncalcined zinc sulphide of very fine

^{*}Co-operative exposure tests by members of the Zinc Pigment Development Association Technical Committee.

December

particle size, when suspended in water suspensions with oxygen passed through for a period of several weeks, shows only very slight zinc sulphate formation. The presence of short-wave ultra-violet light slightly increases the oxidation, but radiation of this type is, for practical purposes, absent from visible light under normal atmospheric conditions. Even if it were not, it would be difficult to correlate the slight amount of oxidation with the heavy breakdown on weathering of zinc sulphide in exterior paints.

There are indications in the literature^{2, 3} that the durability of films based on zinc sulphide can be greatly improved by modification of the formulation, notably by the addition of zinc oxide (to increase absorption of longer wave ultra-violet light and to impart basicity) and mineral extenders of a fibrous or plate-like structure (to give reinforcement and flexibility). In this manner the breakdown can be reduced to eventual failure by mild chalking and erosion, leaving surfaces which can be repainted with a minimum of surface preparation. This improvement might be expected to be more pronounced with coloured paints, provided that the coloured pigments are themselves stable to the action of light and weathering.

With the object of testing paints of this type, particularly coloured paints, the Technical Committee of the Zinc Pigment Development Association produced a series of paints and exposed them at various sites in November, 1954.

PREPARATION OF THE PAINTS

All the undercoats and finishing paints were prepared in the laboratories of the Imperial Smelting Corporation Ltd. at Avonmouth, by pre-mixing, followed by three passes through a *Holmes* laboratory single-roll mill. After milling they were allowed to mature for seven days after which, where necessary, the consistency was adjusted with white spirit to give a paint which could be brushed easily. The viscosities were determined, after the thinning operation, with a *Krebs-Stormer* viscometer. The values recorded in Tables I-VII are the number of grams required to rotate the paddle at 200 r.p.m. Viscosity measurements were also made after a period of 18 months' storage. Full details of the compositions of the paints are given in Tables I-VII (see Appendix 1).

Primers

For metal panels, a zinc-rich primer based on chlorinated rubber was used:

Constituents		Per cent by weight
Zinc dust (95-97 per cent metallic)		76.0
Alloprene B (chlorinated rubber)		2.4
Cereclor I (chlorinated wax plasticiser)		1.6
Xylol	••	20.0
		100.0

1961 PAINTS BASED ON LITHOPONE AND ZINC SULPHIDE 837 For wooden panels, the primer was a conventional pink lead paint:

Consti	Per cent by weight				
Dry white lead	,		••	֥	71.83
Non-setting red lead			••	••	3.29
Acid refined linseed oil	23.20				
16 per cent lead naphth	enat	e solut	ion	••	0.188
6 per cent cobalt naphtl	nena	te solu	tion		0.047
White spirit					balance

Undercoat

This was a common undercoat for all systems based on 28/30 lithopone, acicular zinc oxide and micronised mica, in a ratio of 4:1:1 by weight, respectively. The medium was a mixture of acid refined linseed oil and 30-poise linseed stand oil, in a ratio of 4:1 by weight, respectively, with lead and cobalt naphthenate driers. The undercoat was tinted grey with vegetable black to enable erosion of the weathered paint systems to be more easily detected. The composition of the undercoat is given below:

Cor	Per cent by weight				
28/30 Lithopone			• • •	÷.	47.29
Acicular zinc oxide	**	••	•••		11.82
Micronised mica	••	••	••		11.82
Acid refined linseed of	oil	••	••	••	18.92
30-poise linseed stand	l oil	••			4.73
16 per cent lead naph	thenat	te solut	ion		1.47
6 per cent cobalt nap	hthena	te solu	tion	÷.*	0.05
Vegetable black	••	••			0.12
White spirit	••	••	••		3.78
					100.00

Finishing Coats

White paints were pigmented with either 28/30 lithopone or zinc sulphide, with additions of 25 per cent leaded zinc oxide (*Ledzinox* 25) and water-ground mica.

The white bases were then tinted to close matches with Monolite Red GRS, Ferrite Yellow MY 50, Monastral Green GS, chromium oxide green and Monastral Blue LBS.

The following three media were used:

(i) Linseed/linseed stand oil with a low-acid value.

Constitue	Per cent by weight		
Acid refined linseed oil	 		80.0
30-poise linseed stand oil	 		20.0

(ii) Phenolic resin varnish.

Constituents	Per cent by weight	
Epok R.710		10.80
30-poise linseed stand oil		36.45
Tung oil stand oil		12.15
16 per cent lead naphthenate solution		0.68
6 per cent cobalt naphthenate solution		0.17
White spirit		39.75
		100.00

(iii) Alkyd varnish.

Epok A1060/75 with 75 per cent total solids in white spirit. White and red paints based on white lead in linseed/linseed stand oil medium were included for comparison.

The compositions of the finishing paints are given in Appendix 1.

PAINTING OF THE PANELS

The painting of the panels was carried out in the laboratories of Amalgamated Oxides (1939) Ltd. at Dartford. Wooden and mild steel panels were used, each measuring 30 in. \times 9 in. The wooden panels were planed and sand papered, and after the backs had been primed, a previously-primed wooden batten $(8\frac{3}{4} \text{ in.} \times 1\frac{1}{2} \text{ in.} \times \frac{1}{2} \text{ in.})$ was screwed to the backs, near to each end. The metal panels, which were bright and mill-scale free, were degreased with white spirit prior to painting; the faces of the panels were rubbed with emery paper to roughen the surface and provide a suitable key.

Each panel carried six separate finishing coats of a similar colour with common primer and undercoating systems. The white lead finishing paints were, however, painted on separate panels of smaller size.

1961 PAINTS BASED ON LITHOPONE AND ZINC SULPHIDE 839

EXPOSURE OF PANELS

One complete set of wooden and steel panels was exposed at each of three sites in November, 1954. The sites were at Avonmouth (Industrial), Frodsham (Cheshire—Rural) and Burry Port (South Wales—Marine). All the panels were exposed at an angle of 45°, facing due South.

EXAMINATION OF PANELS

Panels were examined at three-monthly intervals by members of the staff of McKechnie Brothers Ltd. and Imperial Smelting Corporation Ltd. for the following properties.

Gloss

This was assessed against a series of standards with ratings from 1 to 6: 1—high gloss, 2—fairly high gloss, 3—medium gloss, 4—low gloss, 5—gloss at grazing angle only, and 6—flat.

Tint Retention

A series of colour standards was prepared for each colour in the laboratories of McKechnie Brothers Ltd. at Widnes by:

- (a) reducing the shade of one of the tinted paints by the addition of varying amounts of white paint, made from the same white pigment and extender composition, so that the amount of coloured pigment in the paint fell from 100 per cent of that present in the original tinted paint, in 10 per cent steps, to 0 per cent (the shade of the white paint).
- (b) increasing the intensity of shade by the addition of further paint prepared from the same tinting pigment to give shades above the 100 per cent colour content, in increasing steps of 10 per cent of additional colour content.
- (c) applying the above paints in strips to painted cards and using these for assessing the tint retention of the weathered paints.

Per cent tint retention = $\frac{\text{Per cent colour in matching strip}}{\text{Per cent colour in original matching strip}} \times 100$

Checking, Cracking, Chalking, Flaking and Erosion

The standards given in the Exposure Standards Manual of the Federation of Paint and Varnish Production Clubs (1941) were used for assessing these failures. The numbering was, however, modified in the following way:

0=no failure, 1=slight failure, 2=moderate failure,

3=appreciable failure and 4=heavy failure.

RESULTS OF TESTS

The abridged results of the examination over a 26-month period are given in Appendix 2. In these tables the figures given are averages of the ratings recorded on wooden and steel panels. Generally there was little difference between the behaviour of respective paints on each of these two substrates, apart from a slightly greater tendency of a few paints to show more erosion on the metal than on wood, particularly at the Burry Port site.

General Results

The results indicate that both 28/30 lithopone and zinc sulphide give a useful degree of durability in all three media and that the performance is improved considerably when coloured pigments are present.

The loss of gloss is fairly rapid with mixed pigment formulations of the type employed. The more serious defects such as cracking and flaking are, however, either absent entirely or present in a few cases only to a very slight degree. Chalking occurred to varying and erratic degrees, depending on the climatic conditions, but it was never heavy for a prolonged period. As might be expected, chalking took place only to a slight degree with the alkyd medium and was worst with the linseed oil medium. Checking was not a serious defect with any of the paints. Erosion developed with the white and red tinted paints towards the end of the 26-month period. However, all the 28/30 and lithopone panels were in good condition for repainting at the end of the exposure period. Erosion was particularly low on the coloured paints, apart from where *Monolite Red* had been used as the tinting pigment. The dirt retention tended to be worst with the alkyd medium, probably because this showed lower chalking and self-cleansing properties.

The red tints showed very poor colour retention and within a few months faded nearly to the colour of the white panels. It was unfortunate that the red pigment had been used for tinting the comparative white lead paints, since they also were subject to this severe fading. In general there was little difference in performance between the white paints and the red tinted paints. The other colours gave appreciable enhancement of durability.

Tint retention, apart from the red tinted paints, was relatively good with all colours. Some fading took place initially, particularly with the linseed oil medium, but this was counteracted by a tendency for the mixed zinc sulphide pigment/zinc oxide/extender to intensify the depth of colour, so that a number of panels actually showed a greater depth of colour after exposure than before. This tendency seemed to be more pronounced at the Frodsham site.

No general difference in performance between the zinc sulphide—and 28/30 lithopone—pigmented paints was detected. In some cases one proved to be more satisfactory and in other cases the effect was reversed. The variations were random and not associated with site or medium. The zinc sulphide pigments behaved at least as well as the comparative white lead paints in cases where direct comparisons were possible.

CONCLUSIONS

It is possible to formulate white paints from zinc sulphide pigments mixed with zinc oxide and mica which will weather for over two years without serious failure other than loss of gloss developing. Since the tests were made at an inclination of 45° facing South, it seems likely that the durability would be more than doubled under more normal aspects.

1961 PAINTS BASED ON LITHOPONE AND ZINC SULPHIDE 841

The durability of these paints is definitely enhanced by the addition of stable coloured pigments, including ferrite yellow, chromium oxide green and Monastral Blue. In particular chalking and erosion are improved considerably.

All the paints based on 28/30 lithopone and zinc sulphide were in a good condition for repainting at the end of the 26-month period and would have required little pretreatment other than a light rubbing down to remove surface chalking.

ACKNOWLEDGEMENTS

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APPENDIX 1

Constituent		Con	nposition	of paints	(%)	
Constituent	1.A.1	1.A.2	1.A.3	1.A.4	1.A.5	1.A.6
28/30 lithopone	38.05	35.46	36.28	36.80	30.80	37.76
25 per cent leaded zinc oxide	9.51	8.87	9.07	9.20	7.70	9.44
Micronised mica	9.51	8.87	9.07	9.20	7.70	9.44
Linseed/linseed stand oil medium	38.10	39.28	37.17	37.42	33.62	37.96
16 per cent lead naphthenate						
solution	2.34	2.41	2.54	2.34	2.11	2.36
6 per cent cobalt naphthenate						
solution	0.09	0.09	0.09	0.09	0.08	0.09
Parathane (Dipentene)	0.60	0.75	0.58	0.53	0.49	0.59
White spirit	1.80	2.72	2.54	2.61	3.61	1.94
Monolite Red GRS		1.55	-	-	-	
Ferrite Yellow MY 50			2.66		-	-
Monastral Green GS				1.81		
Chromium oxide green	-	-	- 1		13.89	
Monastral Blue LBS		-	-	-	-	0.42
	100.00	100.00	100.00	100.00	100.00	100.00
*Viscosity as made (units)	490	490	490	490	490	490
*Viscosity after 18 months	595	590	550	575	600	700
Settlement after 18 months	000	570	550	515	000	100

TABLE I LITHOPONE-LINSEED/LINSEED STAND OIL FINISHING PAINT

*All viscosities were determined with a Krebs-Stormer viscometer, the values given being the number of grams required to rotate the paddle at 200 r.p.m.

TABLE II

	LITHOPONE-PHENOLIC	MEDIUM	FINISHING	PAINT
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Constituent		Composition of paints (%)							
Constituent		1.B.1	1.B.2	1.B.3	1.B.4	1.B.5	1.B.6		
28/30 lithopone 25 per cent leaded zinc oxide Micronised mica Varnish V.33002 Parathane White spirit Monolite Red GRS Ferrite Yellow MY 50 Chromium oxide green Monastral Blue LBS	··· ·· ·· ··	30.40 7.60 7.60 50.60 0.95 2.85	28.45 7.11 7.11 52.11 1.00 2.92 1.30 —	28.90 7.22 7.22 50.50 1.00 2.96 	29.30 7.32 7.32 49.40 1.30 3.86 	25.40 6.35 6.35 45.78 0.79 4.22 	30.32 7.58 7.58 50.46 0.92 2.79 		
Viscosity as made (units) Viscosity after 18 months Settlement after 18 months	 	100.00 275 300 Hard	100.00 275 325	100.00 275 325	100.00 275 300 Soft	100.00 275 300	100.00 275 300		

TABLE III

LITHOPONE-ALKYD MEDIUM FINISHING PAINT

Constituent		Composition of paints (%)							
Constituent	1.C.1	1.C.2	1.C.3	1.C.4	1.C.5	1.C.6			
28/30 lithopone 25 per cent leaded zinc oxide Micronised mica 6 per cent cobalt naphthenate	33.45 8.37 8.37	32.50 8.12 8.12	32.35 8.09 8.09	32.70 8.17 8.17	29.90 7.48 7.48	33.30 8.33 8.33			
solution Parathane Epok A1060/75 46-poise dehydrated castor oil White spirit Monolite Red GRS Ferrite Yellow MY 50 Monastral Green GS Chromium oxide green Monastral Blue LBS	0.11 1.67 40.50 2.51 5.02 	0.15 4.86 41.10 2.44 1.11 1.60 	0.10 4.85 39.80 2.42 2.30 	0.10 4.90 39.98 2.48 2.00 	0.10 4.47 38.23 2.24 	0.10 4.97 40.30 2.49 1.80 0.38			
	100.00	100.00	100.00	100.00	100.00	100.00			
Viscosity as made (units) Viscosity after 18 months Settlement after 18 months	250 375 —	325 700 —	300 200 —	300 600 Soft	325 875	300 675 —			

TABLE IV

Constituent		Composition of paints (%)							
Constituent	2.A.1	2.A.2	2.A.3	2.A.4	2.A.5	2.A.6			
Zinc sulphide	38.05 9.51 9.51 38.10 2.34 0.09 0.60 1.80 	33.72 8.43 8.43 40.82 2.40 0.09 0.51 2.78 2.82 	35.60 8.90 8.90 36.81 2.30 0.09 0.50 1.80 5.10	35.60 8.90 8.90 36.73 2.33 0.09 0.50 3.20 	28.12 7.03 7.03 32.06 1.85 0.09 0.42 4.42 	37.42 9.36 9.36 37.65 2.34 0.09 0.47 2.14 — — 1.17			
	100.00	100.00	100.00	100.00	100.00	100.00			
Viscosity as made (units) Viscosity after 18 months Settlement after 18 months	550 575 —	550 500	550 500 —	550 575 —	550 500 —	550 450 —			

ZINC SULPHIDE-LINSEED/LINSEED STAND OIL FINISHING PAINT

TABLE V

ZINC SULPHIDE-PHENOLIC MEDIUM

Constituent		Composition of paints (%)							
Constituent		2.B.1	2.B.2	2.B.3	2.B.4	2.B.5	2.B.6		
Zinc sulphide 25 per cent leaded zinc oxide Micronised mica Varnish V.33002 Parathane White spirit Monolite Red GRS Ferrite Yellow MY 50 Monastral Green GS Chromium oxide green Monastral Blue LBS	··· ··· ··· ··· ···	30.40 7.60 7.60 50.60 0.95 2.85 	28.00 7.00 7.00 52.50 0.80 2.50 2.20 	25.70 6.42 6.42 45.20 3.11 9.35 	28.50 7.12 7.12 48.40 1.44 4.32 	20.24 5.06 5.06 41.00 3.16 9.48 	29.85 7.46 7.46 49.99 0.93 3.37 — — — — — — — — — 0.94		
Viscosity as made (units) Viscosity after 18 months Settlement after 18 months	 	100.00 225 270 Hard	100.00 225 275 —	100.00 225 275 —	100.00 225 260	100.00 225 260	100.00 225 260		

TABLE VI

ZINC SULPHIDE-ALKYD MEDIUM

Constituent		Composition of paints (%)								
Constituent	2.C.1	2.C.2	2.C.3	2.C.4	2.C.5	2.C.6				
Zinc sulphide 25 per cent leaded zinc oxide Micronised mica 6 per cent cobalt naphthenate solution Parathane Epok A1060/75	33.45 8.37 8.37 0.11 1.67 40.50	31.10 7.77 7.77 0.10 1.70 41.71	31.40 7.85 7.85 0.10 1.75 39.00	33.20 8.30 8.30 0.11 1.70 40.25	25.36 6.34 6.34 0.08 3.78 35.15	33.00 8.25 8.25 0.10 4.95 40.03				
46-poise dehydrated castor oil White spirit	2.51 5.02	2.30 5.05	2.30 5.25	2.49 2.15	1.88 3.37	2.48 1.90				
Monolite Red GRS Ferrite Yellow MY 50 Monastral Green GS	-	2.50	4.50	3.50	-	_				
Chromium oxide green	=	=	=	_	17.70	1.04				
	100.00	100.00	100.00	100.00	100.00	100.00				
Viscosity as made (units) Viscosity after 18 months Settlement after 18 months	300 450 —	300 700 —	300 280 —	300 550 —	300 550	300 800 —				

TABLE VII WHITE LEAD-LINSEED/LINSEED STAND OIL

Constituen				on of Paints	
Constituen	ii.	ĺ	3.A.1	3.A.2	
Dry white lead Acid refined linseed oil 16 per cent lead naphthenat 6 per cent cobalt naphthena White spirit Monolite Red GRS		 	72.75 23.63 0.19 0.05 3.38	72.46 23.40 0.19 0.05 3.36 0.54	
Viscosity as made (units) Viscosity after 18 months Settlement after 18 months	 	 	100.00 700 	100.00 700 —	

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APPENDIX 2

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	Finishing paint (No. and type)	 14 14 Lithopone/linseed 2A1 ZnS/linseed 1B1 Lithopone/phenolic 2B1 ZnS/phenolic 1C1 Lithopone/Alkyd 2C1 ZnS/alkyd 	3A1 White lead/linseed	 IA1 Lithopone/linseed 2A1 ZnS/linseed 1B1 Lithopone/phenolic 1B1 Lithopone/phenolic 1C1 Lithopone/alkyd 2C1 ZnS/alkyd 	3A1 White lead/linseed	1 1.1 Lithopone/linseed 2.A1 ZinS/linseed 2.A1 ZinS/linseed 1.B1 Lithopone/phenolic 2.B1 ZinS/phenolic 2.C1 ZinS/alkyd	3A1 White lead/linseed
	Panel	Avonmouth FIAI FIA2 FIA3 FIA4 FIA5 FIA5 FIA6	F1A7	Frodsham FIB1 FIB2 FIB3 FIB4 FIB5 FIB5	F1B7	Burry Port FIC1 FIC3 FIC3 FIC3 FIC3 FIC3 FIC3 FIC3 FIC3	FIC7

TABLE VIII White Finishing Paint

(Mean readings for wood and metal panels)

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TABLE IX RED FINISHING PAINT (Mean readings for wood and metal panels)

846

D. S. NEWTON AND J. G. RIGG

December

1961 847 PAINTS BASED ON LITHOPONE AND ZINC SULPHIDE

(Mean readings for wood and metal panels)

FERRITE YELLOW FINISHING PAINT

TABLE X

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				Rating of film d	Rating of film defects after indicated period (months)	ed period (mor	iths)				
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TABLE XI

ORGANIC GREEN FINISHING PAINT (Mean readings for wood and metal panels)

848

D. S. NEWTON AND J. G. RIGG

December

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TABLE XI	OXIDE GREEN
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CHROMIUM OXIDE GREEN FINISHING PAINT (Mean readings for wood and metal panels)

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D. S. NEWTON AND J. G. RIGG

December

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TABLE XIII BLUE FINISHING PAINT (Mean readings for wood and metal panels)

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An Investigation into Drying Phenomena Experienced with Alkyd Resins Based on Soya Bean Oil*

By G. T. WILLIAMS and P. D. COX

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Summary

The problem of variable drying, particularly with regard to the variation in the length of induction period experienced with long oil pentaerythritol soya alkyds, is examined with a view to establishing its cause and how it can be resolved. The observed differences between the drying of the pigmented and unpigmented alkyds are shown and a method is given for effecting an improvement in the basic drying ability of the resin. The role of some drier metals is also reviewed, with particular reference to calcium and barium as additives to the standard 10/1, lead/cobalt drier mixture.

INTRODUCTION

The subject of this paper concerns a particular problem encountered in the course of the authors' work and which in itself led to a variety of phenomena, each a problem in its own right. The bulk of the work outlined was carried out using a typical long oil (65 per cent) penta soya bean oil alkyd prepared by the monoglyceride process, but the phenomenon was not confined to this particular alkyd; it was also observed in other soya alkyds, both pentaerythritol and glycerol types. The work originated from the result of an observation that the drying time, or more correctly the induction period, of a driered soya alkyd varied from batch to batch, the effect being greatly increased when the alkyd was pigmented.

When pigmented finishes are mentioned, unless otherwise stated, rutile titanium dioxide was used at a pigment/binder ratio of 0.8/1.0 and the paints were made up by triple roll milling. It must be noted that unless otherwise stated the drier level used was fixed at 0.5 per cent lead and 0.05 per cent cobalt as metal on alkyd base, this level being employed in both clear and pigmented finishes, and in every case the finishes were reduced to a medium solids of 53.6 per cent in order to keep the composition constant.

The variable drying properties of the different batches of resin were, as mentioned previously, confined chiefly to the induction period of the drying film. Once normal drying commenced the dry was then rapid and satisfactory. This final process was similar whether the resin possessed a long or a short induction period. The instrument used for comparing the variations in drying was the *B.K. Drying Recorder*, which gave a permanent record of the results obtained. In every case a standard film thickness of 0.0015 in. was employed, all tests being carried out at 20°C and 65 per cent relative humidity. The type of record obtained is shown in Fig. 1, and the differences between "good" and "bad" resins can readily be seen. The difference between the drying properties of clear and pigmented finishes is particularly marked; the clear

851

^{*}Read before the Midlands Section on 20 January, 1961, and the West Riding Section on 14 February, 1961.

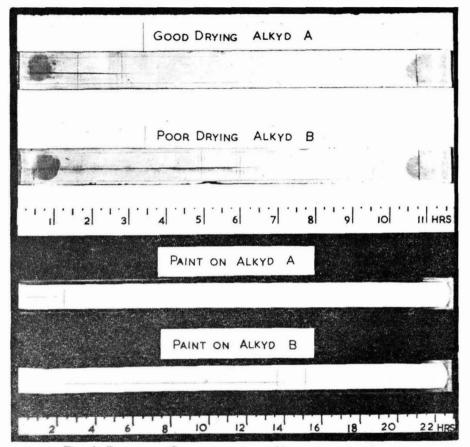


FIG. 1. DRYING OF SATISFACTORY AND UNSATISFACTORY ALKYDS

finish result is obtained on a twelve-hour run, while the pigmented finish requires a twenty-four-hour run in order to record the greatly extended induction period of the poor drying paint.

An examination of the general characteristics of undriered resins with variable dry showed no marked differences, the only minor observation being that the poor drying resins were paler in colour than their good drying counterparts. When it came to an examination of the driered resins, however, a significant observation was made, namely that, although on drier addition all the resins became pink in colour from the cobalt drier present, a gradual colour change to green took place. The rate of change varied from batch to batch of resin: where a rapid change took place the resin had a short induction period; where the change was slow the resin had a long induction period. In order to confirm this observation, three samples of resin with known varying drying differences were tested on the *B.K. Recorder*, in correlation with *Lovibond Tintometer* measurements; the results are shown in Table I. The faster drying the resin, the more quickly the solution developed the yellow (Y) and blue (B) ratios, with a corresponding drop in the red (R).

Maturing	Alk	yd 1—0	Good dry	ing	Al	kyd 2—	Fair dryi	ng	AL	kyd 3—	Poor dryi	ng
period (hr.)	D.T.	R	Ŷ	В	D.T.	R	Y	В	D.T.	R	Y	В
I	14-15	2.3	6.4	0.3	21-31	3.1	5.4		4 -4	3.1	5.4	-
2	11-2	2.0	6.6	0,6	21-3	3.0	5.4		31-4	3.1	5.4	
3	1 -14	1.3	6,6	0.5	$1\frac{3}{4} \cdot 2\frac{1}{2}$	2.7	5.7		23-31	3.0	5.4	
4	14-14	1.2	6.6	0.5	2 -21	2.4	5.7		3 -34	2.6	6.8	
5	1 - 11	1,1	6.6	0.6	$1\frac{1}{2}-2\frac{1}{4}$	2.3	6.5		212-3	2.6	6.8	-
6	3-13	1.1	8.1	1.1	1 -14	2.2	7.1		11-21	2.5	6.9	-
7	1 -13	1.0	8.8	1.3	11-14	1.8	7.0	0.3	2 -21	2.5	7.0	-
8	1 -13	0,9	8.8	1.5	1 - 12	1.6	7.3	0.4	11-21	2.2	7.5	0.
24	14-13		19.0	4.1	1 -13	0.8	13.0	2.6	11-2	0.8	13.0	2.

 TABLE I

 COFFUR CHANGE OF DRIFTING SOLUTION V. DRYING TIME

Since the induction period of a resin solution on drying as a film varied with both the state of oxidation of the drier metal and the time of maturing, the method employed for testing the drying properties of the clear and pigmented resins was standardised. The drying time was determined after four-hour and twenty-four-hour maturing periods, the four-hour test giving an indication of the rapidity of the colour change in relation to the dry. The twenty-four-hour test was used as a standard control test, since it was considered that a good resin should be capable of optimum drying properties on maturing for twenty-four hours.

Since the colour change is one observed on drier addition, and is obviously connected with the "coloured metal" cobalt, it appears that in order to obtain a short induction period the cobaltous cobalt originally added must achieve a certain degree of oxidation in the resin solution. In unpigmented finishes, although the colour change may be very slow, oxidation will eventually occur, particularly if the resin is cast in a thin film in the presence of air. Therefore, although clear finishes do show variations in drying properties, particularly when very short maturing periods are used, after a reasonable time these variations become less noticeable. The initially poor drying resins thus eventually attain the drying properties (and green colour) of the satisfactory materials.

However, early observations had shown (see Fig. 1) that once a resin was pigmented the variation in drying power was greatly increased, the problem becoming much more serious because the pigmented finish, unlike the clear finish, failed to attain satisfactory drying properties on maturing. This phenomenon at first was difficult to explain, but the explanation below appears to be the most probable.

In the pigmented finish the following processes occur:

- (i) The resin reacts with the drier, oxidising the cobalt from the pink cobaltous to the green cobaltic state.
- (ii) Simultaneously the pigment begins to adsorb the drier metals.

December

If reaction (i) is slow the drier is preferentially adsorbed on the pigment and becomes inactivated, with a consequent loss of drying power. If reaction (i) is fast the formation of cobaltic cobalt is largely completed before drier adsorption reaches a degree that will affect the dry; when this change has been effected good drying properties are obtained. Further detailed study is required to determine whether the reason for this is that cobaltic cobalt resists adsorption or that the reacted resin remains in an activated state after the valency change and regardless of adsorption.

A number of experiments bearing on the above explanation was therefore carried out with the following results.

(a) Paints based on anatase titanium dioxide were greatly inferior in drying properties to similar paints based on rutile titanium dioxide when made on poor drying resin. Anatase was found to adsorb driers much more readily and more quickly than rutile, which was determined by adding a white spirit solution of drier to a white spirit pigment slurry, then filtering and observing the filtrate colour compared with a blank containing driers alone. The filtrate from the anatase slurry was colourless while that obtained from rutile showed from its colour that a fair quantity of cobalt remained in solution.

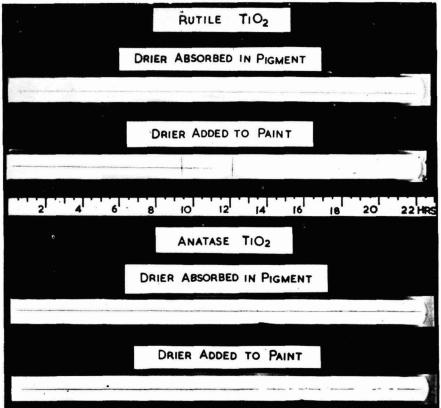


FIG. 2. DRYING POWER OF PRE-ADSORBED DRIER METALS

1961 DRYING PHENOMENA: ALKYD RESINS BASED ON SOYA BEAN OIL 855

- (b) A pigment was allowed to adsorb drier equivalent to the normal quantity required in a paint, then dried (in a low temperature oven) and made into a paint; the results are shown in Fig. 2. Where the drier was absorbed into the pigment, the paint had no drying properties at all, whether anatase or rutile titanium dioxide were used. Where the resin was pigmented and driered in the normal manner, the paint based on rutile showed reasonable drying properties while that based on anatase showed very poor drying properties. In other words, adsorbed drier metal was effectively removed and played no part in catalysing the drier reaction.
- (c) If a resin with known poor drying properties was driered, matured to a green colour and then pigmented, the pigmented finish was superior in drying properties to a similar finish made in the conventional manner.

In the light of work done so far the factors influencing the pink-green colour change were examined. It was found that an alkyd could be rendered capable of effecting this change by a number of treatments including prolonged air blowing, peroxidation with organic peroxides, or filtration. This indicated the possible presence in the alkyd of a material which inhibited the drying mechanism, either by the prevention of the formation of hydroperoxides in the alkyd, or in some other way retarding the cobalt/alkyd reaction. This material appeared to be neutralised in the presence of excess oxygen, hence the eventual satisfactory dry of a clear finish. The fact that filtration improved the dry led to the possibility that the inhibitor (if such existed) was in such a form that it was capable of removal by filtration.

Now that the general scope of the problem was thoroughly understood, the next step was to investigate the basic alkyd, its processing and the raw materials used in its production with a view to establishing the identity of the troublemaker, determining its source, and eliminating it if possible.

EXPERIMENTAL

This poor drying problem could only be brought to light for examination by using a special laboratory technique. Prior to this investigation good drying of soya alkyds was always achieved in the laboratory, using the usual factory procedure of pressing the alkyd into solvent and then filtering the solution thus obtained. In order to accentuate drying differences care was taken to impose the most adverse conditions by omitting filtration and excluding air completely. The special technique adopted, therefore, was to pour every alkyd as base into a container, to which the lid was immediately fitted. The base resin thus produced was cold-cut into mineral spirits and examined for drying as a pigmented finish in the manner previously outlined. All the drying times quoted in the following tables will therefore refer to pigmented finishes and are the times when the finishes are just surface-dry.

The alkyd type chosen for the investigation was of the type in which the problem was exceptionally noticeable, *i.e.* a fusion-cooked 65 per cent soya oil, pentaerythritol, phthalic alkyd, all the raw materials being of high quality and the soya oil having an iodine value between 130 and 135. (This is mentioned in order to show that drying differences should not be due to the variability of the soya oil unsaturation.)

December

As litharge was the alcoholysis catalyst being used where this drying difficulty was experienced, the investigation began by considering the filtration aspect. Litharge naturally forms insoluble lead salts during the alkyd preparation. which are removed partially or completely during a filtration process, depending on the efficiency of the filtering. As has been stated in the introduction, it was noticed that with more efficient filtering the drying of the alkyd was improved. This could be due to removal of the lead salts, which could therefore be the cause of the poor drying experienced with an unfiltered alkyd.

Resin	Catalyst	Viscosity †	Drying	time (hr.)
No.	(%)	(poise)	(Maturin 4 hr.	g period) 24 hr.
1	PbO-0.02	26.3	>12	9 ³ 4
2	LiOH-0.02	31.6	>12	. 12
3	КОН-0.02 РЬО-0.0025	33.1	>12	. 12
4	PbO $-0.02 + H_3PO_4$ (to ppt. as PbHPO ₄)	30.0	>12	>12
7	Ca nap = $PbO - 0.02$	33.1	>12	81
10	$PbO-0.02+H_{3}PO_{4}$ (to ppt. as PbHPO ₄ *)	33.1	>12	>12

	Т	ABLE II		
VARIATION	OF	ALCOHOLYSIS	CATALYSTS	

*This was filtered off at end of alcoholysis resulting in a clear final alkyd. †70 per cent solution in white spirit, at 25°C.

In Table II is shown a series of alkyds in which various alcoholysis catalysts are used, and it can be seen that poor drying results in every case. Even in alkyd No. 10, where the lead was removed as the phosphate immediately after the alcoholysis stage resulting in a perfectly crystal clear base resin, the drying was extremely poor. This could only mean that the alcoholysis catalyst used was not the cause of the problem and that some other component of the alkyd must be responsible.

The oil was the obvious first suspect and hence its replacement by fatty acid and glycerine would show whether it was responsible. The results obtained when this replacement is carried out are given in Table III.

Alkyd No. 6 had a form of alcoholysis carried out by first cooking the fatty acid and polyols together to bring it as near as possible to the previous alkyd cookings in Table II. Alkyd No. 9 had all the materials charged together, and Alkyd No. 11 had the alcoholysis quantity of litharge added in order to examine the alcoholysis catalyst question once again. It can immediately be seen that good drying occurs in all cases and that litharge had no effect. Since the pentaerythritol and phthalic anhydride were from the same source in all cases, no doubt remained as to the oil being the source of the drying problem.

DRYING PHENOMENA: ALKYD RESINS BASED ON SOYA BEAN OIL

TABLE III

Resin	1	Viscosito A	Drying	time (hr.)
No.	Remarks	Viscosity † (poise)	(Maturii 4 hr.	ng period) 24 hr.
6	Soya fatty acids and glycerine cooked together as a monoglyceride before addition of dibasic acid. No catalyst.	34.6	3 <u>1</u>	4
9	All ingredients charged together. No catalyst.	33.1	$1\frac{3}{4}$	13
11	Exactly similar to No. 6 except 0.02 per cent PbO added to "alcoholysis" stage.	33.1	2 <u>1</u>	2

REPLACEMENT OF OIL BY FATTY ACID AND GLYCERINE

†70 per cent solution in white spirit, at 25°C.

This conclusion poses the question as to what in the oil can be responsible for the poor drying, as the iodine value of the oil is exactly the same as the iodine value of the fatty acids used and the speed of drying is quite different. The normal drying mechanism is obviously upset or retarded and a good indication as to where it is retarded is given by the previous observation that a good drying soya resin turns the cobalt driers quickly to the green cobaltic state, while a poor drying resin produces this change only very slowly.

Taking an accepted drying mechanism, the cobaltous cobalt is converted to the cobaltic state by any preformed oil hydroperoxide decomposing:

$$\begin{array}{ccc} \text{Co''} + \text{ROOH} & \longrightarrow & \text{Co'''} + \dot{\text{RO}} + \text{OH} \\ & & \text{or} \\ & & \bar{\text{RO}} + \dot{\text{OH}} \end{array}$$

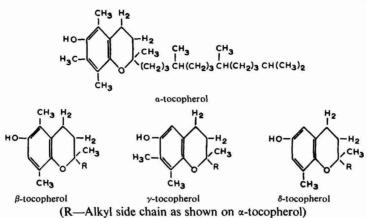
The fact that reaction does not occur, or only does so very slowly, means that either this reaction is prevented, or that the preformed hydroperoxides are decomposed by something before the cobalt can react with them, or that preformation of the hydroperoxides is suppressed by the presence of an oxidation inhibitor in the oil. The third explanation seems to be the most probable; such oxidation inhibitors are known to be present naturally in vegetable oils and particularly in soya oil. These natural inhibitors are the tocopherols and possibly some other materials which have not been fully identified.

The tocopherol structures are given below and they are well known as the "E" vitamins.

In actual fact the tocopherols have been known and used for some time as anti-oxidants in the cooking or edible fats industry to prevent high temperature oxidative degradation during the various processes carried out on the oils. The effect these natural anti-oxidants have on air-drying of the oils has also been known for some time as indicated in a U.S. patent by Vogel¹.

1961

857



In this patent air is blown through the oil in the presence of one of the drying metal catalysts. This preferentially oxidises the anti-oxidant, hardly affecting the jodine value of the oil. Bover² et al. stated that tocopherols have no antidrying power in linseed oil, but act as synergists with any acid phosphatides present in the oil, these phosphatides being powerful anti-oxidants.

The type of oil utilised in this investigation was a good grade alkali-refined soya oil from which virtually all the phosphatides had been removed, to the extent that spectographic examination failed to detect a measurable quantity of phosphorus. The only natural inhibitors known to remain in this type of soya oil, therefore, were the tocopherols and it is to be presumed that they were the reason for the drving difficulties experienced.

Previously it had been shown that by using a fatty acid an alkyd with good or uninhibited drying resulted. In order to examine the possibility of tocopherols being responsible for the poor drying of oil-based alkyds, a quantity of α -tocopherol equal to the amount normally found in soya oil was added to the fatty acid alkyd. The result of α -tocopherol addition on these alkyds is shown in Table IV. It is very evident that compared with the good drying of alkyd No. 9, the drying of alkyd No. 48 has been completely inhibited by adding 0.1 per cent α -tocopherol.

Resin No. Type of cooking		Treatment	Viscosity † (poise)	Drying time (hr.) (Maturing period) 4 hr. 24 hr.		
9	Fatty acid full fusion	No additives	33.1	13	13	
48	Fatty acid full fusion	α -tocopherol added =0.1 per cent oil§	28.5	>12	>12*	

TABLE IV THE EFFECT ON DRYING TIME OF *α*-TOCOPHEROL ADDITION

^{†70} per cent solution in white spirit, at 25°C. *Wet after 24 hours.

§ i.e. similar to proportion found in soya oil.

This is proof that the tocopherol can exist unaffected by the alkyd preparation and act as a strong anti-oxidant. The α -tocopherol addition is of the order of published total tocopherol contents of oils. This is given in Table V, together with that of some other oils, for the purpose of full illustration and comparison.

Oil	Alpha (%)	Gamma (%)	Delta (%)	Total (%)
Linseed oil	-		_	0.11
Safflower, crude	-			0.080
Soybean, refined	0.020-21	0.074–78		0.094-99
Soybean	0.020	0.098	0.050	• 0.168
Sunflower seed	-	-		0.07
	1		1	1

 TABLE V

 TOCOPHEROL CONTENT OF FATS AND OILS³

When it had thus been shown that α -tocopherol had the identical effect on a good drying alkyd as was experienced when using an oil, a series was prepared as shown in Table VI, where the tocopherol content of the oil or fatty acid was varied by subjecting the same oil or fatty acid to various treatments.

TABLE VI

EFFECT OF TOCO	PHEROL CONTENT	ON OIL C	DR FATTY	ACID	FROM THE	SAME SOURCE

Resin No.	Oil or F.A. used	Tocopherol content (mg./100g.)	Catalyst	Viscosity † (poises)	(h Matu	g time r.) uring tiod 24 hr.
59	Sample No. 1 neutralised unbleached Soya oil	64	PbO	31.6	>12	31/2
60	Sample No. 2 neutralised bleached soya oil	56	РЬО	33.1	8 <u>1</u>	2 <u>3</u>
61	Sample No. 3 autoclave split undistilled soya fatty acids	25	_	31.6	8 <u>1</u>	23
62	Sample No. 4 autoclave split distilled soya fatty acids	0		34.6	2 <u>1</u>	13
63	Sample No. 5 chromatographically refined soya oil	0	PbO	31.6	13	134
64	Sample No. 6 specially refined soya oil	64	РЬО	31.6	10 1	4 <u>3</u>

†70 per cent in white spirit, at 25°C.

The oils and fatty acids were all derived from the same unrefined batch of soya oil and the separate samples obtained as stated in Table VI, the sample No. 6 having been refined by an alternative unspecified process. In Table VI it will be seen that where tocopherols are absent from the oil or fatty acid there is no inhibition of the drying. In fact the chromatographically refined oil gave an alkyd with exceptionally good drying although the iodine value for this oil was only 128. Where there is a quantity of tocopherol present in the fatty acid-based resin No. 61, inhibition of the drying occurs, and although the fatty acids for both resin No. 61 and No. 62 were derived from the same source, the tocopherol-free fatty acid gave alkyd No. 62 which dried extremely well. Similarly, where there were quantities of tocopherols present in the oil-based alkyds, inhibition of the drying occurred, larger tocopherol contents giving more inhibited drying.

The determination of the tocopherol contents for this series was carried out by a modified Emmerie-Engel test. The tocopherols were extracted after saponification of the oil and dissolved in a known quantity of alcohol. By taking a portion of this alcoholic solution, adding excess alcoholic ferric chloride solution and then excess 2-2' dipyridyl in alcohol, a red coloration was produced, the intensity of which was measured on an adsorptiometer. Estimation was based on the tocopherols quantitatively reducing the ferric ions to ferrous ions. The ferrous ions form a coloured complex with the dipyridyl which will be in proportion to the concentration of the tocopherol content.

As a matter of interest, due to the possibility of any other semi-drying oil having this inhibited drying problem, two alternative oils were examined, as shown in Table VII. There the segregated sunflower oil, which contained a

Resin No.	Type of oil	Tocopherol content of oil (mg./100g.)	Alcoholysis catalyst	Viscosity† (poise)		ng time nr.)
		(ing./100g.)			(Maturir 4 hr.	ng period) 24 hr.
56	Segregated sunflower oil	40	РЬО	30.1	>12	3
57	Tobacco seed	10	РЬО	31.6	31	2

TABLE VII

ALKYDS BASED ON SEMI-DRYING OILS

†70 per cent solution in white spirit, at 25°C.

moderate quantity of tocopherols, dried as expected, with some definite inhibition, whilst the tobacco seed oil, which contained only just detectable quantities of tocopherols, dried without any real sign of inhibition.

This then fairly well established the cause of the problem, but it remained for a method to be found to overcome the drying difficulty.

860

INVESTIGATION INTO METHODS OF REMOVING THE Drying Problem

One obvious conclusion from the first part of the investigation was that the use of a good distilled soya fatty acid would produce alkyds which did not exhibit the drying difficulty at all. The only dangers arising with the method were associated with the use of a cheaper, inadequately distilled soya fatty acid which would contain some inhibitors.

Trying to find a method for using oil was a lot more difficult. The use of chromatographically refined soya oil had been shown to give the desired result, but this method was definitely uneconomical and about the only known way of actually removing the tocopherols. The only remaining solution was to destroy the tocopherols in the oil before cooking the alkyd.

Resin No.	Oil treatment	Catalyst	Viscosity† (poise)		ime (hr.) 1g period)
				4 hr.	24 hr.
30	Oil exposed U.V. light for 6 hr. under air bubbling	РЬО	31.6	>12	>12
33	Oil treated with 0.05 per cent M.E.K.P. and 0.05 per cent T.B.H.P.	РЬО	34.6	>12	>12
36	Oil air blown for 90 min. at 130°C in presence of 0.001 per cent Co	РЬО	31.6	>12	>12
45	Oil treated with 0.2 per cent T.B.H.P. in the presence of 0.001 per cent Co for 1 hr. at 120°C	РЬО	41.25	>12	>12
51	Oil washed three times at 55°C with methanol	РЬО	37.8	>12	>12

TABLE VIII OIL TREATMENTS TO OVERCOME INHIBITION OF DRYING

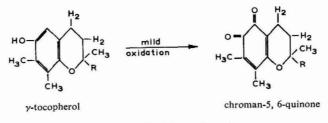
†70 per cent solution in white spirit, at 25°C.

Some of the various processes which have been tried to destroy the inhibitors in the oil before preparation of the alkyd are indicated in Table VIII; none of them has been found to show any improvement.

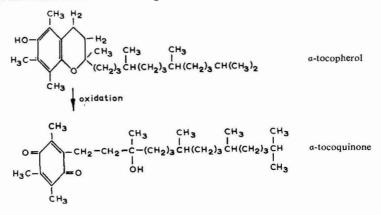
For alkyd No. 30 the oil treatment was based on the fact that the tocopherols are known to oxidise readily under ultra-violet light, particularly when exposed to air. The oil treatment for alkyd No. 33 was based on supplying a large quantity of oxidising material in the form of peroxide, which would decompose on heating the oil and accordingly oxidise the inhibitors. The basis for the treatment carried out on the oil for alkyd No. 36 was taken from the previously mentioned patented process by Vogel¹, which definitely claimed to oxidise the inhibitors and render them inactive. Similarly, the treatment used on the oil in alkyd No. 45 gave visual proof that oxidation of the tocopherols was taking place in that the oil on reaching 120°C, with the peroxide and cobalt, developed a pronounced red coloration which subsequently disappeared on

December

prolonged holding at 120°C. This coloration was attributed to the formation by mild oxidation, of chroman-5, 6-quinone from the γ -tocopherol present in the soya oil. This oxidation process is shown below. Since this quinone is highly coloured, as little as 50 mg./100 g. of oil is enough to produce quite a pronounced coloration of the oil.



The subsequent disappearance of this coloration was presumably due to further oxidation splitting the ring system and producing a yellow coloured p-quinone. Other tocopherols must similarly be oxidised to their respective quinone, *e.g.* as illustrated for α -tocopherol.



A treatment not relying on oxidation was the basis for the oil treatment used for alkyd No. 51 in Table VIII, in that it was found that hot washing of the oil with methanol removed a portion of the tocopherols each time. However, three washings apparently failed to remove sufficient of the tocopherols to give any improvement in drying. Nor was there any improvement in drying obtained by the oxidation treatments as can be seen from Table VIII. This can only mean that during the alkyd cooking process any oxidised inhibitor can be reactivated to a form which will again act as an inhibitor.

In order to ascertain the validity of this statement a further series of resins was prepared, to which the oxidation product of α -tocopherol, *i.e.* α -tocopherylquinone, was added. The results obtained in this series are shown in Table IX, where all the resins were full fusion soya fatty acid alkyds prepared in the same way, except for the variation indicated in the table.

Resin No. 9 showed no inhibition (see also Tables III and IV). The addition of α -tocopherol to this resin as shown in resin No. 9 (a) produced definite

1961 DRYING PHENOMENA: ALKYD RESINS BASED ON SOYA BEAN OIL 863

Drying time (hr.) Remarks Resin Viscosity[†] No. (poise) (Maturing period) 24 hr. 4 hr. 9 No addition 34.7 11 11 34.7 9 (a) 0.1 per cent α -tocopherol, based on the oil 101 41 content, added to the completed resin 9 (b) 0.1 per cent α -tocoquinone, based on the oil 34.7 13 13 content, added to the completed resin 70 0.1 per cent α -tocoquinone, based on the oil 30.0 7 21 content, added to the initial charge of the resin

 TABLE IX

 Effect of a-Tocopherol and a-Tocopheryl Quinone Addition

†70 per cent solution in white spirit, at 25°C.

inhibition, while the addition of α -tocopheryl-quinone in resin No. 9 (b) produced no inhibition. This proved that tocopheryl-quinone was not an anti-oxidant, but by incorporating it in the initial charge of the resin preparation anti-oxidant properties were produced, as shown by the inhibited drying of resin No. 70.

Thus actual removal from the oil of the inhibitors or their oxidation products is necessary before a suitably good drying alkyd can be produced without fail. Removal is completely impracticable, but there still remains the possibility of treatment of the finished alkyd resins as the means of overcoming the problem.

As mentioned in the introduction, the addition of organic peroxides to the alkyd was found capable of improving the drying properties, the most suitable peroxides being the type more readily decomposed at low temperatures, *e.g.* M.E.K.P., M.I.B.K.P., *etc.* Unfortunately, their effect was only short lived, presumably owing to their decomposition leaving the system inhibited as before. Their sole purpose would therefore appear to be one of providing a "bank" of available oxygen to initiate the drying mechanism and not to affect the nature of the inhibitor in any way.

During the study of work that had been carried out on edible oils, where anti-oxidants were a necessity for stability and where the tocopherols were particularly desired both as a source of anti-oxidant and of vitamin E, it had been observed that metal ions, particularly copper, must be absent or deactivated by chelation⁴, otherwise the tocopherols were rapidly destroyed by catalytic oxidation⁵. As it was necessary in this investigation to destroy these inhibitors, the introduction of metal ions in the presence of oxidising agents was examined as a possible means of improving poor drying alkyds.

From this satisfactory results were obtained using metals, such as copper, cobalt, ferric ion and many others, for catalytically oxidising the tocopherols with such oxidising agents as air, oxygen, peroxides or hydroperoxides, the

December

reaction being carried out at temperatures above 50°C. The process at present forms the basis for a patent application⁶ and Table X shows the type of improvement obtained by a typical treatment using copper naphthenate as the source of metal (0.5 p.p.m.) and M.E.K.P. as the oxidising agent (0.12 per cent active peroxide on base alkyd), the treatment being carried out on the mineral spirit thinned solution.

TA	DI	E	V
IA	DL		Λ

	CATALYTIC	OXIDATION	TREATMENT	ON A	POOR	DRYING	SOYA	ALKYD	SOLUTION
--	-----------	-----------	-----------	------	------	--------	------	-------	----------

Resin	Viscosity [†] (poise)	Drying time (hr.)		
	(poise)	(Induction period 4 hr. 24 l		
Soya alkyd solution before treatment	26.3	12	8	
Soya alkyd solution after 2 hr. treatment	26.3	1 7	13	

†70 per cent solution in white spirit, at 25°C.

It will be seen from Table X that this method affords a satisfactory means of ensuring that regardless of variation in the anti-oxidant content of a soya oil alkyd, the anti-oxidant can be reduced to a level which renders the alkyd satisfactory in drying properties without affecting any other properties.

THE EFFECT OF SOME DRIER METALS ON SOLVING THE PROBLEM

Although a satisfactory process for overcoming the phenomenon had been obtained, this was not the only line of work that had been investigated. Before the nature of, and the cure for, the main problem above had been determined, further work had been in progress on the resin in order to determine whether a particular addition or combination of drier metals could effect an improvement in the drying properties of the alkyds, particularly when pigmented, and the following general results were obtained.

The obvious course of action was to increase the drier level already being used, and, as was to be expected, an increase in the drier level improved the dry. However, this was not regarded as a satisfactory approach to the problem, since the quantity of drier had to be varied to meet the specific requirements of a particular resin sample. If the resin had particularly poor drying properties, excessive quantities of drier would be required. However, an interesting observation noted was that a resin with initial good drying properties retained better dry after two years' storage when driered at a level of 0.07 per cent cobalt, 0.7 per cent lead, rather than at the level of 0.05 per cent cobalt and 0.5 per cent lead. The paint containing the higher drier level had an induction period of $2\frac{1}{2}$ hours after two years' storage compared with four hours with the standard drier. Any further increase failed to improve the retention of dry beyond this point.

A number of very interesting facts were observed when 0.1 per cent calcium naphthenate was examined with 0.5 per cent lead, 0.05 per cent cobalt as a

864

1961 DRYING PHENOMENA: ALKYD RESINS BASED ON SOYA BEAN OIL 865

dry improver in paints. First, with a poor drying resin pigmented with rutile titanium dioxide the method of incorporation of the drier greatly affected the resulting paint. If the three drier metals were pre-mixed and stood for some time, and the drier blend then added to the paint, very poor drying properties resulted. However, if in the same drier combination the calcium were added first, followed after a suitable period by the lead/cobalt mixture, or if the lead cobalt were added first, followed after a time by the calcium, greatly improved drying properties resulted.

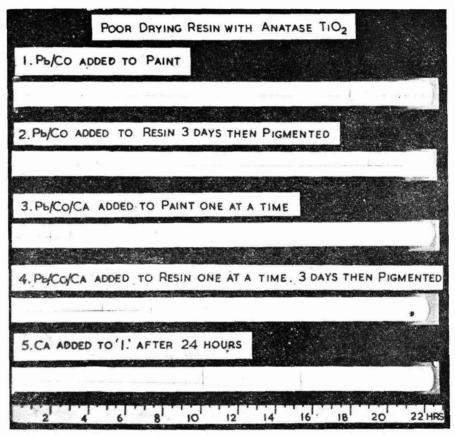


FIG. 3. THE EFFECT ON INDUCTION PERIOD OF METHOD OF DRIER ADDITION

An example of the effect drier metals play on this type of sensitive drying material is demonstrated in Fig. 3. In this experiment a series of paints was made up on a poor drying resin sample, the effect being enhanced by pigmenting with anatase titanium dioxide. Initially only paint No. 1 had a poor dry, the remaining paints being very satisfactory. The figure shows the results obtained after two years' maturing of the paints, and immediately the differences become apparent. Both the paints containing lead and cobalt have lost their drying properties, while the paint in which the lead, cobalt and calcium were added one after the other has retained a very good dry. The remaining paints,

December

which contain the same drier concentration as No. 3, vary appreciably in dry retention, depending on the method of incorporating the driers in the paint.

The reasons for the peculiar differences with calcium which are dependent on the method of incorporation are still to be fully investigated, but it is significant that calcium is a very "active" metal; addition of calcium to an alkyd or oil can cause an immediate viscosity increase or, in the case of lightly bodied oil, the formation of tiny gel-like particles. In other words, calcium appears to react with the medium, possibly forming complexes with low viscosity, slow drying material. Similarly, calcium also appears to form a complex with cobalt, for when the two drier metals are blended together an intense "lilac" colour develops. This "lilac" material, unlike cobalt alone, is resistant to ready oxidation (difficult to obtain pink-green change) and is also resistant to adsorption to pigments, even anatase titanium dioxide. This resistance to adsorption is similar to the effect obtained when cobalt is complexed with a known complexing agent such as 1 : 10 phenanthroline; in both cases after slurrying with anatase there remained a coloured filtrate.

A further drier metal examined with promising results was barium, but the work carried out on this metal is only in its early stages in pigmented finishes. However, this preliminary work appears to show that barium, like calcium, when mixed with cobalt produces a blend which is resistant to adsorption, but unlike calcium, barium activates cobalt, making the cobalt more readily oxidisable.

A level of about 0.25 per cent barium was required to give equivalent dry retention to that obtained with 0.1 per cent calcium, but these figures resulted from only three months' observation. A series of experiments were, however, carried out using a sample of unpigmented resin with a control based on lead/cobalt, and additions of lead/cobalt/calcium and lead/cobalt/barium (both calcium and barium being employed at 0.1 per cent concentration). In one case the calcium and barium were pre-mixed with the lead/cobalt before addition to the resin and in the other case the calcium and barium were added one hour before the lead/cobalt. All of the resins had similar drying properties after twenty-four hours' maturing $(1\frac{1}{2}$ -hour induction period), but after one year these had changed appreciably with the results shown in Fig. 8. The control based on lead/cobalt alone had deteriorated to an induction period of nine hours, and similarly the pre-mixed calcium and the separately added barium trials both showed long induction periods ($6\frac{1}{2}$ and seven hours respectively). The induction periods of the resins with the separately added calcium and the pre-mixed barium were, however, quite short, both being $1\frac{1}{4}$ hours.

It is thus evident that both calcium and barium are satisfactory drying improvers when added correctly, the barium preferably being mixed with the lead/cobalt before addition to the paint and the calcium being added separately.

Zinc with lead/cobalt, unlike calcium or barium, renders the drying properties of the resin less satisfactory than lead/cobalt alone. Similarly, the replacement of 10 per cent of the paint thinner (white spirit) with dipentine caused poor initial dry and a rapid loss of dry in a pigmented finish on storage.

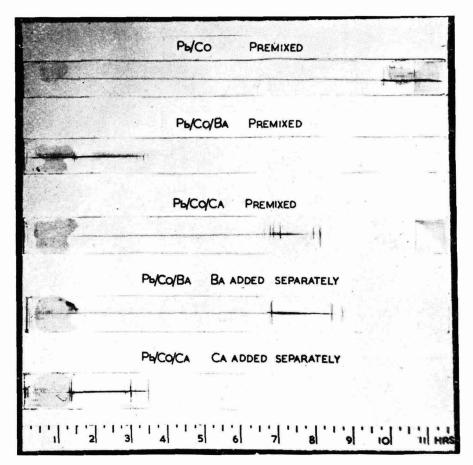


FIG. 4. THE EFFECT ON INDUCTION PERIOD OBTAINED USING BARIUM OR CALCIUM

CONCLUSIONS

A chance observation that was routine in itself initiated a series of interesting experiments and the accumulation of much useful information. The investigation has revealed that the anti-oxidant materials known to be present in soya bean oil remain active even after the incorporation of the oil in an alkyd, and a method has been developed for destroying or rendering innocuous these antioxidants present in the resin. A number of useful drier recommendations and facts concerning the resin/drier/pigment relationship were also obtained, although the work is far from complete. In fact some of the aspects of the problem have not yet been examined at all fully and will need to be the subject of future work.

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[Received 18 May, 1961

DISCUSSION

(West Riding Section)

MR. P. HAIGH asked what was the effect of an anti-skinning agent, such as methyl ethyl ketoxime, on the sensitive soya alkyds.

MR. WILLIAMS replied that these products evaporated off normally and did not retard the drying properties. However, in very bad conditions of high humidity and low temperature their effect was observed.

MR. N. F. LYTHGOE inquired whether cobalt naphthenate affected the colour retention of the dried film.

MR. WILLIAMS felt that cobalt naphthenate used as a drier metal in soya alkyds should not affect the colour retention of the dried film any more than when used with any other type of alkyd at a similar concentration.

MR. D. GRAY wondered whether the authors had approached oil manufacturers to produce tocopherol-free soya oil.

MR. Cox confirmed that this had been done and that pure material had been obtained chromatographically for several experiments. Unfortunately the cost of the process was far too high for commercial production.

MR. GRAY further inquired whether the authors had any evidence that the source of the soya beans had any effect on the variation of the different batches of alkyds made from that oil.

MR. Cox stated that they had not been able to follow this up, since the source of the seed was unknown to the suppliers of the oil.

December

The Limitations of Leaching Rate Determinations of Anti-Fouling Compositions

By A. PARTINGTON and P. F. DUNN

Research Organisation of Ships' Compositions Manufacturers Ltd., Poole, Dorset

Summary

The leaching rate technique is described and the experimental method for its determination is outlined. Several limitations of this technique are mentioned, together with some unusual and anomalous results that have been obtained by some workers in this field. Experiments are carried out to establish a suitable technique for leaching rate determination, with the ultimate object of devising an accelerated test which will predict the performances of anti-fouling compositions in service, and further to examine the relationship between leaching rates and different methods of storage—raft, speed apparatus, agitator, laboratory tank and power station outfall.

INTRODUCTION

The leaching rate technique was published in England by Harris¹ and in America by Ketchum² as a means of assessing the efficiency of anti-fouling compositions. It is based on the concept that an anti-fouling composition acts by producing a surface layer of a poisonous solution which repels, or is lethal to, the spores or larvae of marine organisms which attempt to settle on the composition. The maintenance of a lethal concentration of poisons in the surface layer necessitates a gradual wastage of the poisons. If an anti-fouling composition is to be efficient, the rate of loss of poisons from it must be neither too high nor too low; otherwise a composition would either have a very short life or would be ineffective. The leaching rate technique was designed to measure the rate of loss of poison under standardised conditions of test and is expressed in micrograms of poison-as metallic radical-liberated from one square centimetre of film per day (μ g./cm.²/day). For any single poison the leaching rate which just prevents fouling is known as the critical leaching rate. In the case of cuprous oxide it has been demonstrated by several investigators^{1, 2, 3, 4} that the critical leaching rate is 10 μ g./cm.²/day. When used in the presence of toxic mercury compounds the critical value is lower than 10 μ g./cm.²/day, but there is no reliable information regarding the critical value when the two poisons are used together; certainly, the effect is not simply additive.

Experimental Technique

A brief description of the experimental method^{1, 2, 5, 6} used in determining leaching rates now follows. Ground glass slides are painted with anti-fouling compositions and, after standard drying times, are immersed in sea water either in the sea itself or in a tank of flowing sea water in the laboratory. The slides are removed periodically from storage and leaching rate determinations are carried out by immersing them for a certain period of time in an appropriate volume of continuously aerated sea water. The slides are then removed and the quantity of copper or mercury in the leachates is determined colorimetrically using sodium diethyl dithiocarbamate and diphenylthiocarbazone, respectively. A modification of the technique for use at inland stations has been described by Hanson and Jervis⁷, and another modification consists of rotating

December

painted glass tubes permanently in a certain volume of sea water which is replaced periodically and analysed. Evidence of the composition industry's interest in leaching rate techniques is also shown by the joint correspondence in connection with the report by Harris¹, the contributions by Keenan and Brooks to the discussion of the paper by Lawrence and Gale¹⁴ at the O.C.C.A. Conference in 1947, and again in a contribution by Keenan¹⁵ at the Premier Congrés Technique in Paris, also in 1947.

There are several complications in the technique. If zinc or iron is present it can react with the sodium diethyl dithiocarbamate and either produce turbidity or interfere with the determination. Alternative colorimetric reagents for copper have been studied by Gran⁸ and Tuck and Osborn⁹.

Bacterial slime may form on the anti-fouling compositions during storage and slough off during the leaching rate test to produce turbidity. The slime may also contain appreciable accumulations of copper which will result in apparently higher leaching rates.

If the concentration of copper in the sea water exceeds 0.5 μ g./ml., the leaching of the copper from the anti-fouling composition is suppressed. Copper also interferes with mercury estimations, but a method has been developed by Barnes¹⁰ which eliminates the interference.

Limitations

The advantages of leaching rate determinations over raft trials of anti-fouling compositions are that the former can be used at any time of the year when fouling organisms are absent and, by using tank storage, such determinations are independent of location. As used to date, however, they cannot be regarded as a substitute for raft trials, although these also have limited uses, the ultimate test of an anti-fouling composition being, of course, on a ship in service.

Leaching rates do not provide any estimate of the amount of residual poisons in a composition or, alternatively, the amount of poison lost. Consequently, they are of no use in predicting the effectiveness of a composition at some future date: the rate of loss of copper during leaching rate determinations is not necessarily the same as the rate of loss during raft or tank storage. Likewise, the leaching rate technique cannot be considered as an accelerated method, since the test must be continued up to the point where the anti-fouling composition would fail in raft trials; it is only accelerated in the sense that it can be used in winter to indicate the probable performance of an anti-fouling composition. In view of the above limitations, the "conventional" leaching rate technique can be used satisfactorily only for the following purposes:

- (i) The elimination in the laboratory of completely unsatisfactory experimental anti-fouling compositions.
- (ii) The observation of trends in performances of anti-fouling compositions as a result of gradual variations of the ingredients.
- (iii) The routine checking of batch productions.
- (iv) An indication at any particular time that an anti-fouling composition is in such a condition that it would prevent or allow settlement of fouling organisms.

1961 LIMITATIONS OF LEACHING RATE DETERMINATIONS 871

Unusual and Anomalous Results

A survey of the literature shows that when using the leaching rate technique several unusual and anomalous results can be obtained, some being mentioned below. Harris^{1, 11} showed that when an anti-fouling composition contained mercuric oxide in addition to cuprous oxide, the leaching rate of copper necessary to prevent fouling was less than that required when cuprous oxide was used alone in the same medium. The leaching rate of mercury from copper/mercury compositions was variable and unconnected with anti-fouling efficiency. A copper leaching rate of 5-7 μ g./cm.²/day appeared to indicate satisfactory anti-fouling performance, provided that the mercury was also lost at a rate of at least 0.5 μ g./cm.²/day. Also, mercury leaching rates did not give a true indication of the rate of loss of mercury from a composition.

Barnes¹² demonstrated that considerable changes in the mercuric oxide content of the same matrix had little or no effect on the mercury leaching rate, in contrast to cuprous oxide where variations in content resulted in marked changes in the copper leaching rate. The analysis of paint films containing different percentages of mercuric oxide showed, however, that equal additions of mercuric oxide produced similar rises in the actual rate of loss of mercury and that the losses as estimated by analysis were greater than those indicated by leaching rate determinations.

Ketchum¹³ et al. used the same matrix in the preparation of a series of compositions containing different proportions of cuprous oxide and magnesium silicate at a total pigment volume fraction of 0.12. From the average steady-state leaching rates over a period of two to five months they calculated the intrinsic rate of solution of the matrix. The calculated values decreased from 46 at a poison weight fraction of 0.04 to 31 at a poison weight fraction of 0.30. Since the same matrix was used for all compositions, the calculated rate of solution would be expected to remain constant, irrespective of the cuprous oxide contents. This result was possibly caused by the matrix combining to some extent with the cuprous oxide.

The rate of loss of copper from an anti-fouling composition, as determined from the leaching rate, has been found to be 1.2 times the actual rate of loss on a raft, and the rate of loss in service twice the mean rate of loss on a raft³.

EXPERIMENTAL

When it was decided to undertake leaching rate determinations at the R.O.S.C.M. Laboratory, an experiment was carried out in order to select a technique to be eventually employed, the desideratum being ultimately to devise, if possible, an accelerated method which would enable an accurate forecast to be made of the performances of anti-fouling compositions under service conditions. From a consideration of the work which had been done previously it was decided that the experiment should be designed not only to test the accuracy of the technique chosen at Poole but also to determine the relationship between leaching rates and different methods of storage, since this was thought to be a very important factor.

Five methods of storage were therefore employed: raft, speed apparatus, agitator, laboratory tank and power station outfall, in conjunction with the leaching apparatus illustrated in Fig. 1.

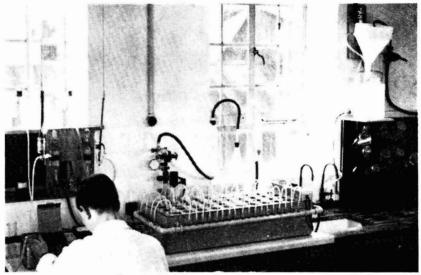


FIG. 1. LEACHING RATE APPARATUS

The apparatus is based on that described by Harris¹. It consists of a water bath (36 in.×13 in.×8 in.) thermostatically controlled at a temperature of 23°C and designed to accommodate forty hydrometer jars of internal measurements approximately $6\frac{1}{2}$ in.×1 $\frac{1}{2}$ in. Air is supplied from a compressor storage tank to each jar by means of 0.7 mm. bore glass capillary tubes attached to a $\frac{3}{4}$ in. galvanised pipe fitted around the sides of the bath. The air is supplied at 50 lb./in.² and reduced to 8 lb./in.², enabling 5 litres per hour to be delivered to each jar. In order to prevent any possible contamination from copper supply lines, the air is dried before use with calcium chloride and then re-saturated by bubbling through sea water. Sea water for the apparatus is drawn from a 10-litre aspirator jar and is aerated continuously before use in order to stabilise the pH.

The panels consist of pieces of p.v.c. extrusion (6 cm. $\times 3.33$ cm.=20 cm.²) with a projection on the back which slides into another type of p.v.c. extrusion. The panels are sandpapered, weighed and then placed in a jig so that replicate panels may be painted simultaneously. The wet and dry weights of the anti-corrosive and anti-fouling compositions are recorded, and the rates of application adjusted as near as possible to pre-selected values. The final coats of compositions are allowed to dry overnight before weighing and immersion in sea water (Fig. 2).

When carrying out leaching rate determinations, 100 ml. of aerated sea water are introduced into each hydrometer jar by means of an automatic pipette; the jars are placed in the water bath (cold) and air bubbled through by inserting the capillary tubes. The thermostat is then switched on to increase the temperature to 23°C and, in the meantime, the panels are removed from storage, washed with a jet of sea water and placed in dishes. The slime is removed from all unpainted parts of the panels with a foamed plastic sponge and the panels again rinsed in sea water before being placed in the jars. After two

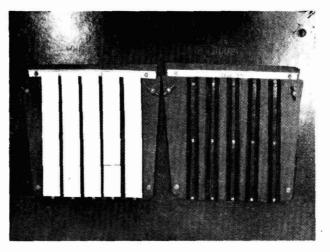


FIG. 2. METHOD OF MOUNTING PANELS FOR RAFT STORAGE

to four hours, according to the leaching rates, the panels are returned to storage and the leaching jars removed from the bath. The copper in the leachate is determined by adding, with shaking, 5 ml. of 20 per cent citric acid, 5 ml. of 10 per cent ammonium hydroxide and 2 ml. of a 0.1 per cent solution of sodium diethyl dithiocarbamate in distilled water. The solutions are added in that order as soon as possible, and the leachate is then left undisturbed for forty minutes before measuring the intensity of the colour by means of a 10 cm. cell in an electrical absorptiometer fitted with a 601 violet filter. By using a calibration curve for the instrument the concentration of copper is determined and from this the leaching rate, *i.e.* the rate of loss of copper in μ g./cm.²/day, is obtained.

In order to study the effect of speed of storage, the apparatus shown in Fig. 3 was used. It consists essentially of a plastic cylinder which can be rotated

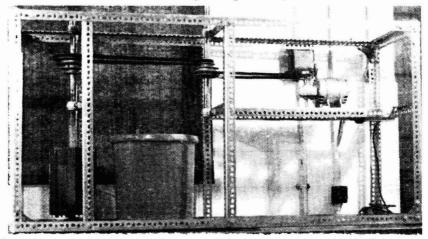


FIG. 3. Apparatus Used for the Study of Speed of Storage Between Leaching Rate Determinations

December

in sea water at peripheral speeds varying from $\frac{1}{8}$ to three knots. The panels are mounted in slots on the sides of the cylinder and are removed periodically for leaching rate determinations. Agitated conditions of storage were obtained by mounting panels in a domestic washing machine.

The experiment was confined to copper determinations and included compositions based on five types of modified rosin matrices:

- (i) Boiled oil/pine oil/hydrogenated methyl abietate.
- (ii) Vinylite/tritolyl phosphate.
- (iv) Chlorinated rubber/hydrogenated methyl abietate.

- (iii) Bedesol oil.
- (v) A neutral resin.

The first four matrices contained varying quantities of cuprous oxide. Compositions based on the fifth matrix contained equal amounts of cuprous oxide and different proportions of a neutral resin.

RESULTS

The results showed in the middle stages of test that:

(a) Compositions containing equal quantities of cuprous oxide and based on matrices modified with different proportions of the same neutral resin gave almost identical minimum leaching rates when stored under agitated conditions between leaching measurements. The results are illustrated in Fig. 4. Also

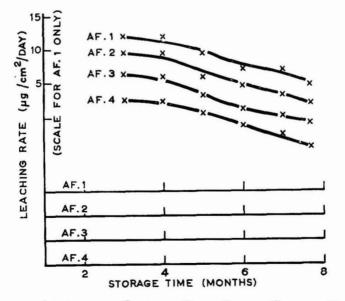


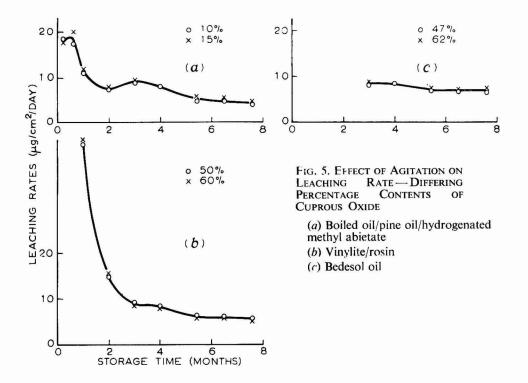
Fig. 4. Effect of Agitation on Leaching Rate-Similar Cuprous Oxide Contents

874

1961 LIMITATIONS OF LEACHING RATE DETERMINATIONS 875

when stored under static conditions the leaching rates approached a constant maximum. Widely separated curves were given by the same four compositions when using raft storage.

(b) Compositions based on the same matrix and containing different percentages of cuprous oxide gave almost the same minimum leaching rates when stored under agitated conditions between leaching measurements. These results are illustrated in Fig. 5.



(c) The maximum leaching rates for compositions based on soft matrices or containing low percentages of cuprous oxide were obtained by using static storage between leaching rate determinations, *i.e.* sea water which was changed periodically but did not flow over the panel surfaces (Fig. 6 (a)). Intermediate rates between the maxima and the minima (agitator storage) were obtained by using raft storage or by storing in flowing sea water. Maximum leaching rates for compositions based on hard matrices (*e.g.* Vinylite or Bedesol oil) containing high percentages of cuprous oxide were obtained either by storing the panels in flowing sea water or by using raft storage between leaching measurements (Fig. 6 (b)). The lower rates for static storage are considered to be attributable to the accumulation of resins and comparatively insoluble basic copper salts on the surface.

December

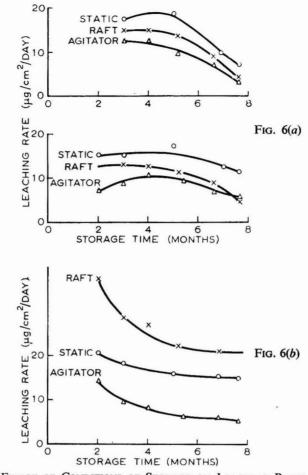


FIG. 6. EFFECT OF CONDITIONS OF STORAGE ON LEACHING RATES (a) Soft matrices with low cuprous oxide contents (b) Hard matrices with high cuprous oxide contents

DISCUSSION

These results can best be explained in terms of the following concepts of the mode of release of poisons from an anti-fouling composition. A soluble matrix anti-fouling composition, based on rosin/neutral resin and cuprous oxide, in the middle stages of its life is considered to consist of a matrix receding at a constant rate and containing a sparingly soluble salt, cuprous oxide, which may or may not form a "pebble dash" on the surface of the composition. If a "pebble dash" is formed then some mechanism would be expected to obtain which would allow cuprous oxide to persist on a surface such as rosin, which has a solubility rate of about one-third that of cuprous oxide. The solution of the matrix is essentially a slow chemical reaction which should be almost independent of the rate of movement of water over the surface. On the other hand, the rate of solution of the cuprous oxide will depend on the concentration of

1961 LIMITATIONS OF LEACHING RATE DETERMINATIONS 877

copper (mainly as $CuCl_2$) in the surface layer, or the rate at which the surface layer is removed. Under sufficiently agitated conditions of storage any exposed particles of cuprous oxide would be dissolved comparatively rapidly to leave "craters". Further, under almost static conditions of storage the matrix would recede to leave a "pebble dash" of cuprous oxide which may be in excess of its proportion in the formulation.

From this it follows that the leaching rate determination, as normally carried out after raft or static tank storage, is merely a measure of the relative intensities of the "pebble dash" on the surface of the matrix. Under such conditions the leaching rate is approximately proportional to the cuprous oxide content when using the same total pigment volume concentrations and matrix. The leaching rate cannot, however, be considered as a measure of the loss of copper unless, fortuitously, the rate of agitation in the leaching jars is the same as the average rate of agitation during storage. Paradoxically, high leaching rates may indicate a low average rate of loss if cuprous oxide particles have accumulated on the surface during storage. There still, however, remain several points which require elucidation. Immediately after storage under highly agitated conditions, the anti-fouling compositions had appreciable leaching rates. Further, the leaching rates were almost independent of the cuprous oxide contents when using the same matrix, and were almost identical when using the same amounts of cuprous oxide in matrices modified with different proportions of the same neutral resin. It is suggested that under such conditions copper is liberated entirely by diffusion through the matrix and this can best be illustrated by Fig. 7.

The simplified theory outlined above refers only to anti-fouling compositions that are formulated to give steady-state leaching conditions during the middle stages of their lives. Certain soluble matrix anti-fouling compositions give continuously falling leaching rates during the whole of the middle and later stages of their lives, and in such cases copper is considered to be lost by diffusion through the matrix; these types of compositions usually contain high percentages of rosin or rosinates or liquid plasticisers such as hydrogenated methyl abietate. Further, the simplified theory does not apply to matrices containing high percentages of cuprous oxide or insoluble resins. If insoluble resins or basic copper salts accumulate on the surface, then even under static conditions of storage the leaching rates would be expected to approach a constant lower value irrespective of the cuprous oxide contents, owing to the rate of release of copper being limited by the comparatively insoluble copper salts and by diffusion through the film of insoluble resins. In such cases the maximum leaching rates would be obtained by providing just sufficient agitation between leaching determinations so as to prevent the formation of insoluble basic copper salts on the surfaces of compositions and by ensuring that the concentration of copper in the storage water was below 0.5 µg./cc. This is illustrated in Fig. 8, which shows the types of curves obtained from three anti-fouling compositions containing 20, 30 and 40 per cent cuprous oxide.

After reaching steady-state leaching conditions the compositions were stored in static unchanged sea water for fourteen days and then subjected to increasing speeds of rotation between leaching rate determinations. Immediately after VARIOUS CONCENTRATIONS OF CUPROUS OXIDE PARTICLES AT SURFACE DEPENDING ON MATRIX USED, DRYING TIMES, SOLVENTS, ETC

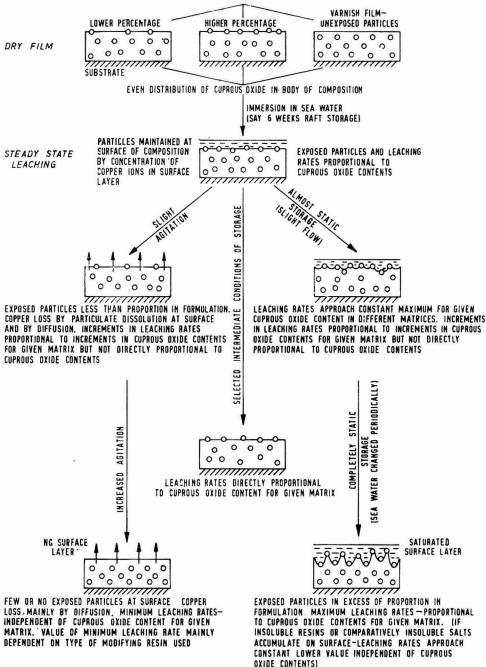


FIG. 7. ILLUSTRATION OF THE EFFECT OF STORAGE CONDITIONS ON LEACHING RATES OF SOLUBLE MATRIX ANTI-FOULING COMPOSITIONS CONTAINING MODERATE PROPORTIONS OF CUPROUS OXIDE

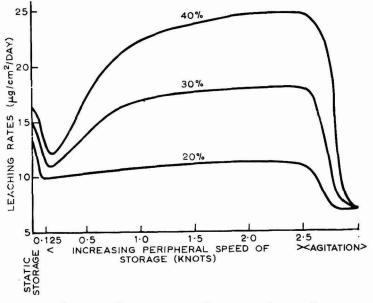


FIG. 8. EFFECT OF STORAGE CONDITIONS ON LEACHING RATES-COMPOSITIONS CONTAIN DIFFERENT PERCENTAGES OF CUPROUS OXIDE

storage in static unchanged sea water there were only slight differences in the leaching rates of the three compositions and only that containing 20 per cent cuprous oxide had attained its maximum leaching rate. The compositions containing 30 and 40 per cent cuprous oxide gave maximum leaching rates after storage at peripheral speeds in excess of one knot, although the actual rate of water flow over the surfaces was almost certainly considerably lower than one knot because of the laminar layer effect. These results are considered to indicate that, when using tank storage between determinations, the peripheral speed of rotation must be adjusted according to the leaching rate in order to obtain conditions of steady-state leaching. In the case of a satisfactory composition with a leaching rate in the region of 10 μ g./cm.²/day, for example, the peripheral speed in excess of $\frac{1}{8}$ knot would be sufficient. In all cases it would, of course, be essential to maintain the concentration of copper in the storage water below 0.5 μ g./cc., *i.e.* to prevent the storage water becoming saturated with basic copper salts.

In these experiments the compositions were subjected to agitated conditions of storage after a peripheral speed of $2\frac{1}{2}$ knots had been reached. The agitation was sufficient to remove the laminar layer and results confirmed those obtained in previous experiments, *i.e.* for a given matrix the leaching rate after agitated conditions of storage was independent of the cuprous oxide content. It will also be noted that after agitated conditions of storage the leaching rates of all three compositions were below the critical value of 10 μ g./cm.²/day.

The practical application of the foregoing concepts is that, under the comparatively stationary conditions in port, copper is considered to be lost from an anti-fouling composition by particulate dissolution at the surface; when underway the loss is mainly due to diffusion through the matrix. The

December

most "dangerous" time in the life of an anti-fouling composition is when it is subjected to potential fouling conditions in port immediately after a voyage³, for its efficiency will then depend on the time required for it to present a lethal surface layer. This will necessitate eventual exposure of particulate cuprous oxide, but in the interim period there will be some anti-fouling action as a result of diffusion. In most cases, however, the leaching rate of copper in the interim period will be below the critical value of $10 \ \mu g./cm.^2/day$ and at such times the use of compounds such as mercury rosinate would ensure that the composition presented a lethal layer at its surface.

The above concepts can be applied to anti-fouling compositions containing particulate mercuric oxide, but it is not necessary to consider agitation; the high solubility of mercuric oxide ensures that any particles exposed during storage dissolve rapidly to leave "craters" and the rate of solution is not limited by the concentration in the surface layer. When subjected to the leaching test mercuric oxide is lost entirely by diffusion through the matrix and, apart from odd particles which become exposed, the rate is independent of the mercuric oxide content.

Further, if these concepts for the mode of release of copper and mercury are correct, then it is not surprising that difficulties are encountered when attempting to incorporate organic poisons in anti-fouling compositions. Unless the solubilities are very low the leaching rate after a voyage or turbulent conditions on a raft would be almost zero—there would be no diffusion until sufficient matrix had dissolved to expose more poison.

CONCLUSIONS

The leaching rate test can be satisfactorily employed only to eliminate completely useless anti-fouling compositions, to check batches of a formulation, to observe trends in the performances of a series and to indicate at any particular time whether an anti-fouling composition will prevent fouling. It is an arbitrary test and the values obtained depend largely on storage conditions between determinations. Even with raft storage the readings will vary not only according to design and siting of the raft, but also according to prevailing tidal conditions, both during storage and at the time of the determinations. It gives no indication of the future performance of an anti-fouling composition or any indication of the ability of a composition to recover its lethal surface layer either after turbulence on a raft or after a long voyage in service, which is the *sine qua non* of anti-fouling properties.

Alternative methods of carrying out leaching rate determinations, such as rotating painted glass tubes in sea water and analysing periodically the leachate, have the disadvantage that, whilst they give an indication of the true rate of loss of poison, they do not give an indication of the ability of a composition to retain or produce a lethal concentration of poisons at the surface where it is required. From a consideration of the defects and the results of work which has been done, it is thought that the best approach to the laboratory testing problem would be to rotate an anti-fouling composition in sea water at a progressively increasing speed and determine periodically the leaching rate by rotation at a constant speed. Work is now being carried out on these lines at the R.O.S.C.M. Laboratory.

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Reviews

CHEMISTRY OF NATURAL PRODUCTS

International Union of Pure and Applied Chemistry. London: Butterworths Publications Ltd., 1961. Reprinted from *Pure and Applied Chemistry*, Volume 2, Nos. 3-4, 347-635. Price 70s.

At the outset it is necessary to say that this is not a textbook on natural product chemistry and that the contents do not have any direct bearing on chemical problems of concern to the paint industry. The book consists in fact of the special lectures presented at an International Symposium in Australia in 1960. Included therein are, however, some of the most exciting stories of modern organic chemistry. At this point of history it is fascinating to compare the classical organic and the modern organo-physical approaches to current problems.

In many cases the two are used in combination, but the extremes are admirably illustrated by the story of the total synthesis of chlorophyll by Woodward, using a bewildering variety of organic reactions, and the description of the techniques for determining three-dimensional molecular structure from empirical formulae and X-ray diffraction data only by Mathieson. Biological chemistry is well represented, and in the paper on bacterial lipids by Lederer it can be noted how little some of the fatty acids responsible for pathological phenomena differ chemically from the acids of the common glyceride oils. For example, tuberculostearic acid, responsible for tubercules, differs from stearic acid only by one methyl side chain. There are many other interesting papers and the book is divided into sections on heterocyclic chemistry, physical methods, aliphatic and homocyclic chemistry and biological chemistry.

L. A. O'NEILL.

ELECTRONIC PACKAGING WITH RESINS

By C. A. HARPER. London: McGraw Hill Publishing Co. Ltd., 1961. Pp. 339. Price 85s. 6d.

The sub-title "A Practical Guide for Materials and Manufacturing Techniques" establishes the aims of the book. There is little chemical theory but a great deal of information of value to engineers and others who wish to apply the techniques of encapsulation and embedding to electrical and electronic equipment.

A range of encapsulating and embedding resins is dealt with but, quite rightly in view of their widespread use, epoxides occupy 63 pages against 30 for all other types. Fillers are well covered as are low density resins, and a very interesting section dealing with processing equipment demonstrates the wide range of such equipment available in the United States. In the United Kingdom, much of the processing equipment has been individually built by the user company because of the lack of suitable commercial equipment.

The most serious criticism of this book is that it deals only with materials of American origin, many appearing under trade names unknown in the United Kingdom. An example is on p. 35 where the only European manufacturing REVIEWS

company mentioned is British Resin Products Ltd., perhaps the least known suppliers of epoxide resins in the United Kingdom. There is no mention of C.I.B.A., British Bakelite, Shell or other British suppliers.

Inevitably when so much information is provided, a large amount of it originates in manufacturers' literature; this results in a multiplicity of units. Examples are on pages 27 and 34; in the former power factor is stated as 2.27, 2.48, *etc.*, presumably (but not stated) expressed as a percentage; in the latter it appears as 0.002, 0.003, *etc.* Similarly °F and °C appear in a more or less random order.

An interesting and novel section is that devoted to the effects of environment, including radiation hazards. Fortunately, the curing agents are stated by their chemical names, but the resin ERL 2774 does not appear to be the same as any marketed in the United Kingdom; this detracts from the usefulness of this information.

Most of the criticisms are minor ones. This book should prove of great value to the many engineers, technologists and others concerned with the applications of resins to electrical and electronic equipment. The printing and binding are well up to the usual high standard expected from the McGraw-Hill Publishing Co. Ltd.

H. G. MANFIELD.

ISOPROPYL ALCOHOL

By L. F. HATCH. London: McGraw-Hill Publishing Co Ltd., 1961. Pp. x+184. Price 54s. 6d.

Isopropyl alcohol was first produced in quantity in 1920 and is the first petroleum chemical. Its annual production from propylene, of which a good account is given in this volume, now approaches half a million tons. Much of this is diverted for conversion into acetone, aliphatic esters and aromatic derivatives, but isopropyl alcohol is also consumed directly for many purposes. Indeed the reader will be surprised to see listed at the outset no less than 250 applications. Descriptions of its uses as solvent cover twenty pages and include varnishes, nitrocellulose lacquers, adhesives, cosmetics, perfumes, detergents, pharmaceutical preparations, reaction and crystallisation media and drying agents. Brief mentions follow of isopropyl alcohol as an extraction medium for vitamins, anti-oxidants, vegetable oils and petroleum products. Some miscellaneous uses cited are its possible exploitation as a major constituent of motor fuels, for de-icing aeroplane wings, in anti-freeze mixtures, as a corrosion and foam inhibitor and for the recovery of fuel oil. Another chapter surveys its pharmacological applications.

Somehow or other the whole field of chemical and physico-chemical industry seems to be embraced, but the author omits theoretical discussion of the principles which may be involved in these very varied uses and makes no claim to evaluate the efficacy ascribed to isopropyl alcohol for any particular purpose. Although industry will find a use for this monograph, it cannot have been intended for the use of students.

December

REVIEWS

There are forty-five pages (twenty-two tables) of physical data relating to isopropyl alcohol and its derivatives as well as 1,203 literature references. The book is attractively produced, as is usual with books from this publishing house.

A few minor points: the English reader is nowadays not unprepared for "color", "odor" and "sulfur", but he will look twice at "molds" (p. 72), "carburetor" (p. 65), "mediums" (p. 49), "Soybean" (p. 58), "preignition" (p. 66), "fulfills" (p. 75) and "makeup" (p. 4). What is the usefulness of printing recipes which include such ill-defined materials as *Aquaresin GMC* (p. 42), *LPG* (p. 71), and *TEL* (p. 65)? "Caustic" (p. 59 *et seq.*) would be better replaced by "Sodium hydroxide" and "glycerine" (p. 61 *et seq.*) by "glycerol".

S. MARKS.

POLYPROPYLENE

By O. J. KRESSER. New York: Reinhold Publishing Corporation, 1960. Pp. 268+ix. Price 52s.

The author of this book has gathered together all the information available on polypropylene at the time of writing. Items covered are properties, chemistry, processing, and applications in packaging, durable goods and soft goods. There is also a chapter on possible future developments. The processes and applications involved in the use of the plastic are so varied that the treatment has had to be, in some cases, brief.

Nevertheless, anyone capable of processing, say, polyethylene should be able to obtain enough information to handle polypropylene. Paper and printing are good, and the illustrations are clear. Altogether this is a useful book for anyone interested in the manufacture of products ranging from transparent films to filter funnels, from water pipes to textiles.

G. L. FULTON.

Correspondence

TECHNICAL EDUCATION IN THE PAINT INDUSTRY

The Report of the Study Group of the Paint Manufacturers' Co-operation Committee is an interesting and intelligently constructed document which is no doubt even now playing a useful part in helping people to grasp the confused and confusing position obtaining in technical education and technical qualifications. It will, however, be of substantial value in the framing of policy only if two fundamental questions are first considered. These are: What field is to be open to the technically trained man in the paint industry? What is the relationship to be between the paint industry and the rest of the chemical industry?

Openings for the Technical Man

It does not seem to me to matter all that much whether we consider the technical man as having O.N.C., H.N.C., City and Guilds, Dip. Tech., B.Sc., or Ph.D. The only point to remember is that the higher the qualification the broader the range of subjects the man can tackle. The main thing is a training which will result in an inquiring mind which takes nothing for granted. Having got that inquiring mind, how far can the technical man get? Or—what *ought* to be the same thing—what salary can the technical man hope to get? This matter has two aspects: Can the able technical man hope to become Managing Director? Can the competent but limited technical man expect to get as good a salary as he would get for the same kind of job in another industry? Unless the answer is *yes* to both questions the paint industry cannot expect to get good recruits; why should it?

I am rather afraid that the answer at the present time is *no* to both questions. Many firms still cling to the outworn idea that the technical man should be *on tap but not on top*, that the place of the technical man is the laboratory, and that questions of cost and management should be left to persons whose only qualification to deal with them is a complete lack of training in clear thinking. I have even heard the argument advanced that, because of the shortage of technologists, firms cannot afford to promote technical staff. I am not, of course, saying that all senior jobs should be filled by technical people—although that is not such a bad idea—but simply that the possession of technical training ought not to be a bar to promotion. The very structure of the paint industry makes for difficulties in this direction, since the large number of family firms implies a large amount of nepotism—using the word purely descriptively and in no pejorative sense. All that can be suggested here is that the families concerned will have to exercise some degree of self-restraint in making senior appointments.

The other aspect is more serious; it matters less, in a sense, that the Managing Director is able than that the formulating staff is competent. And it is of no use expecting to have a pool of promising juniors if you pay twelve pounds a week to an O.N.C. man of twenty-eight or £850 a year to a B.Sc. of forty; these are both cases within my knowledge. Ignorance is no excuse; the R.I.C. publishes from time to time a salary survey of its members which is not a recommended

scale but a statement (more or less) of fact, while the A.Sc.W. publishes a recommended scale for unqualified staff. This latter scale, while somewhat divorced from the facts of life at the higher levels, ties up nicely with the bottom end of the R.I.C. survey. It may thus fairly be adopted for men of National Certificate or City and Guilds level: those who do not graduate at the expected age can have their scales assimilated to the lower quartile or lower octile of the R.I.C. scale. Salary increases are always a hard matter; the guidance obtainable from published scales should be accepted gladly.

Relationship with the Chemical Industry

Because there are so many small firms in the paint industry the opportunities for promotion by change of job are fewer than occur in other industries. Yet there must be such opportunities if able men are to be attracted, quite apart from the general point that people in this country do not change their jobs often enough. I see no way out of this difficulty except by a conscious acceptance by the paint industry of the fact that it is a part of the chemical industry. The paint industry must be ready to accept staff from other parts of the chemical industry. taking advantage of their probably superior scientific status and accepting the necessity for teaching newcomers its own mysteries. More important, the paint industry must allow its technical staff to fit themselves to depart to other parts of the chemical industry by encouraging the broadest contacts with technical matters in general. Industries (and firms) can attract good staff only if they are prepared to lose them. In this connection the proposed establishment of a special technological qualification for the paint industry would appear to be a retrograde step; I cannot welcome the idea that competent paint technologists are going to become *adscripti glebae*, economic serfs, as it were, bound to a single industry. It would be far better to explore the possibility of some kind of liaison with the Plastics Institute, the Institute of Physics, and the Society of Dyers and Colourists, so that their well-recognised qualifications could, with suitable modification, be made available to the paint technologist. It is a very happy chance that recognised qualifications exist in the main three technological directions in which a paint technologist would wish to develop.

But the real point is that the problem presents difficulties which are quite likely unique, and that this uniqueness has not been fully appreciated. We have an industry mainly composed of small family firms, an industry which has been slow to accept the concept of applied science, an industry whose technical disciplines involve in the main three different widely separated scientific disciplines, highly specialised and offering unusual difficulties to any attempt at simplification. We need to construct an educational system based essentially on the physical chemistry of high polymers, the theory of colour, and rheology. To that we must add the requirement that this formidable trinity is to be presented at an intellectual level essentially below that of a pass degree. We then have to warn the candidates that the intellectual climate of the industry is not such as to welcome or even to accept such a scientific approach, that the salaries are lower than those obtaining elsewhere, that substantial promotion is unlikely, and that they run the risk of disqualifying themselves from moving outside the paint industry. Put this way it is surprising that anyone ever comes in at all.

1961

CORRESPONDENCE

I am old-fashioned enough to believe that supply is related to demand. If the paint industry really wants good technical people it must offer the appropriate rewards of salary and status. (To get the ball rolling it may have to offer salaries higher than appropriate.) When that is done the mechanics of training will sort themselves out.

Ivy Cottage, Moore, via Warrington. M. H. M. ARNOLD 15 October, 1961

Information Received

(In case of difficulty regarding addresses, members and subscribers to the Journal should apply for details to the General Secretary of the Association at the address shown on the front cover.)

Information has been received from THOMAS INDUSTRIAL AUTOMATION LTD. concerning a new version of their *Leveltron* Industrial Level Controller, the *CS 101*, together with a new continuous level indicator and controller—the *Leveltrator*—which will operate visual and audible alarms. Further, they have/supplied information on their *Add-on* alarm unit which gives signal light or annunciator display of plant conditions. It was designed to meet the demand for a complete "pre-packaged" alarm unit and is intended for flush mounting on instrument panels.

It has been announced by HOWARDS OF ILFORD LTD., a member of the LAPORTE INDUSTRIES GROUP, that they are undertaking two further projects. One is a unit for the production of the plasticiser dicyclohexyl phthalate which should be completed in 1962. The second plant will produce cyclohexanol and cyclohexanone by the direct oxidation of cyclohexane in air; this unit should be completed in 1963.

A magnetic level indicator for radioactive, corrosive, toxic, inflammable, volatile, pressurised or valuable liquids has been developed by ALAN COBHAM ENGINEERING LTD. It can be fitted from above or below on any tank and is reported to be simple to operate.

JOHNSON, MATTHEY & CO. LTD. have announced that they have acquired the majority of the shares of the leading Belgian precious metal company Pauwels Freres S.A. The new company will operate under the title JOHNSON, MATTHEY & PAUWELS S.A.

The following publications have been received: The Cellobond Polyester Story from BRITISH RESIN PRODUCTS LTD.; Plasticised PVC Film, Polypropylene Film, Polythene Film, Nylon Film and Polyvinyl Alcohol Film from BRITISH CELLOPHANE LTD.; C.V.P. Versamid Polyamide Resins: Specifications and Compatibility Data, Protective and Decorative Coatings from CRAY VALLEY PRODUCTS LTD.; Micronised Pigments in the Cowles Dissolver, and the Torrance High Speed Cavitation Dispenser, together with an information sheet on Spun Phthali—Chrome Green ZE. 64505 from GOLDEN VALLEY COLOURS LTD.; Kestner Fluid Heat Transmission Systems from KESTNER EVAPORATOR & ENGINEERING CO. LTD.; Dispercel Cellulose Lacquer Chips: Colour Range Card and Information Booklet 102 from RUNNYMEDE DISPERSIONS LTD.; Flooring Based on Epikote Resins from SHELL CHEMICAL CO. LTD.; a complete set of Information Sheets, complete with folder, giving details, and specifications and uses of SPELTHORNE METALS LTD.'s metallic lead.

Several hundred aerosols collected from all over the world will be on display at a small exhibition, "More and More in Aerosols", staged by THE METAL BOX CO. LTD. in their showrooms in Baker Street until 15 December.

COMMENT

The stated object of a new film entitled "Why Thixotropy?", presented by CRAY VALLEY PRODUCTS LTD., is to demonstrate the benefits of thixotropic paints, particularly in relation to a reduction in labour costs. This it does very persuasively and in a straightforward manner which will be readily comprehensible to non-technical users, at whom it is aimed. The film is in beautiful colour and extremely well produced by FILMS OF INDUSTRY.

Bristol Section

PATENTS AND THE PAINT CHEMIST

The first meeting of the Section's 1961-2 session took place at the Royal Hotel, Bristol, on 29 September, when Mr. F. Armitage took as his subject "Patents and the Paint Chemist", twenty-seven members and friends being present.

The lecturer gave details of the historical origins of the Patent Rolls and letters patent, and described the development of patent law and the organisation and responsibilities of the Patent Office. The steady increase in the number of patents granted was commented on.

The method of applying for a patent and the cost involved were then discussed, and the lecturer illustrated these by reference to the patent history of styrenated media, a product in which his own company had a considerable interest, acrylic resins and unsaturated polyesters. Dealing with patents in general, the lecturer produced an analysis of the filing of British Patents, and showed that, whereas in 1956 43 per cent of all British Patents dealing with surface coatings were filed by the United Kingdom companies, in 1961 52 per cent of all British Patents were filed by American companies, 25 per cent by European firms and only 23 per cent by the United Kingdom concerns. His conclusions from these figures were that, first, British companies as a whole were not putting enough effort into research, and that they preferred to allow continental and United States firms to carry out research and to obtain the patents, and then to negotiate a licence to use them. The lecturer considered this to be a dangerous trend which could well lead to trouble in the future, both from the economic and the scientific manpower aspect.

The discussion was opened by Mr. A. B. Lock and questions dealing with patent infringement, blocking patents, the terms of reference of the Patent Office, and the patent position in the U.S.S.R. were posed by Mr. Phillips, Mr. Woodbridge, Mr. Thompson and others. A vote of thanks was proposed by the Publications Secretary and unanimously endorsed.

D. S. N.

London Section

THE OTHER MAN'S TECHNOLOGY

The first meeting of the twenty-fourth session took place on 27 September at Manson House when, in accordance with established custom, the new Chairman, Mr. A. T. S. Rudram, addressed the Section, taking as his subject "The Other Man's Technology".

Mr. Rudram discussed the functions of Research Associations and the relations existing between them and industry. The development of the idea behind the original foundation of the Research Associations was traced, and the objections put forward by industry at various times were noted, in order to show that the Research Association development had not always been wholly acceptable to industry: in some cases this was true even today. Mr. Rudram thought that this development represented a factor of major importance in British industrial development and had provided a pattern for similar developments abroad. The changes in the functions of the Research Association as technological research in industry had increased in quantity and quality were reviewed and possibilities of the Research Association undertaking studies in operational procedures with a view to increased efficiency and productivity were put forward. He thought that the original main work of the Research Associations could be classified under the following headings:

(i) Basic research on the raw materials and processes of the industries they served, and

- December
- (ii) Applied research on technical problems of general concern to their industries;

but under present conditions the following five should be added:

- (iii) Development of research results to levels required by their industry, or such sections of it as may be unable to do so for themselves;
- (iv) Operational surveys with a view to increased efficiency and productivity;
- (v) Library and information;
- (vi) Liaison and advisory services to their members;
- (vii) Technical and technological education.

The speaker continued by discussing operational research, quoting various authorities in defining the subject, and he suggested that success in this field might be achieved by an objective approach which might suggest that Research Associations could apply the techniques very effectively. At the same time it had to be remembered that the human being was a vital factor in industrial efficiency and all operational approaches should take human welfare into consideration. He quoted the D.S.I.R./Medical Research Council Committee on Human Relations final report which said: "It is a peculiar condition of social science that the best results come from those persons who are able to operate at both the practical and theoretical levels." He suggested that this should be the aim of all members of the staff of Research Associations.

Mr. Rudram then turned his attention to education for industry and mentioned the training courses held at the Paint Research Station and the way in which they had been supported by industry so that they had always been oversubscribed. The interests of the professional societies and groups connected with the paint and printing ink industries had been shown by such activities as the Apprenticeship Councils and publications such as the Printing Ink Manual and the O.C.C.A. Training Manuals. He stressed the support given by the industry to this educational activity and surveyed the recommendations given in the report *Technical Education in Industry* produced by the Study Group on Technical Education in the Paint Industry. He suggested that the drive for better professional status for the paint and ink industries and the broadening of training policies to embrace techniques and knowledge in other industries should be intensified, quoting Dr. L. Valentine's use of this theme and his development of it in his address at the last annual meeting of the Paint Research Association. He concluded by examining the way in which the technologies in the plastics industry were developing and to some extent usurping those of the surface coating industries. He suggested finally that these latter industries should apply the same principle and learn from the other man's technology and, having learnt, improve it, and further, that the stimulus for this should be provided by the co-operative Research Associations. O.C.C.A. and the National Trade bodies.

Following the address the meeting was continued by a discussion to which several of Mr. Rudram's predecessors in the Chair contributed. Mr. H. A. Newnham inferred that the paint industry seemed to be far from efficient. He was worried about the effects of diversification, either by the dispersal of manufacturing units or by increase of departments. He thought that this might lead to a diminution in progress in the original purpose of the industry—the manufacture of paint. Mr. Rudram remarked that his idea was that diverse industries had hints and tips to give the paint industry which were worth consideration.

Mr. N. R. Fisk quoted a report, summarised in *The Times* of 31 July, 1961, of a committee on which stockbrokers had been largely represented and in which the paint and pigment industry had been rated as sixth in efficiency out of thirty seven industries surveyed. The ability of such a committee to pass judgment on such matters was doubted by Mr. Rudram.

Mr. J. A. L. Hawkey queried the wisdom of equating efficiency with profits and the possibility of complacency arising from the showing of good sales and profits by the industry. He thought that this tendency to look backwards and at figures sometimes led to a reluctance to plan for the future by undertaking long term research. Mr. Hawkey also suggested that it should be acknowledged frankly that the industry was less efficient than it might be and that, even in a mainly efficient factory, it was always possible to find pockets of inefficiency which should be eliminated. He thought that in a Research Association approach to operational research, the first object should be to educate the management. Mr. Rudram replied that the D.S.I.R. had an operation unit which ran courses for management.

Mr. F. Armitage raised again the question of diversification and quoted American and German paint companies who had entered into the adhesive and laminating fields. He suggested that in fifty years' time the paint industry might no longer be making paint.

Dr. H. G. Rains cited figures given in the Paint Research Station Monthly Memorandum comparing personnel employed and output in the United Kingdom and the United States; he suggested that they gave considerable food for thought on the question of efficiency.

Mr. G. W. Mack suggested that the time of a Research Association should be divided into 50 per cent fundamental, 25 per cent general and 25 per cent propaganda, publication and education. He thought that in particular Research Associations could well combine the experience of the industry by a survey technique. The education of the user and consumer was a problem requiring serious thought for, in some cases, if a manufacturer were honest, an order could be lost, and he wondered whether that function might devolve on a new Development Association or even D.S.I.R.

Mr. D. E. Roe commented on the professional animosity which existed between the chemist and the accountant; he felt that this might be due to the lack of skill of the chemist in this industry in presenting his story in a form understandable to the accountant. He quoted the obvious difference in the pharmaceutical industry where accountants authorised large expenditure on research projects that must be beyond their understanding.

Mr. Rudram concluded the meeting by commenting on the liveliness of the discussion, typifying a statement made by Mr. Roe that the subject of the address was one on which everyone was an expert.

Some Considerations on the Formation of Films

The second technical meeting of the session was held in the lecture room of the Chemical Society at Burlington House on 10 October. Dr. H. W. Talen, Director of the Paint Research Institute, Delft, Holland, gave a lecture entitled "Some Considerations on the Formation of Films".

Dr. Talen divided film formation methods into nine types involving three classes: purely physical, physical/chemical, and purely chemical; and three mechanisms within each class. He defined each type by example and proceeded to discuss three types in detail. In type 2, the physical class involving the evaporation of a non-solvent, he discussed the various theories put forward to explain the formation of coherent films by the coalescence of polymer particles from aqueous emulsions. He showed how the theory most relevant to practical observations and experimental facts was that put forward by Brown. He considered the forces applying in film formation and the way in which the spherical polymer particles were squashed together into polyhedra, illustrating this by some excellent electron micrographs. In type 4, the physical/chemical class involving the evaporation of solvent followed by reaction with outside agents, he considered linseed stand oil and alkyd paints and discussed the effects on film properties of segregation of stand oils by molecular distillation and acetone extraction. The effect of method of cooking on the properties of alkyds was postulated as being more important than time of cooking, and Dr. Talen demonstrated that mechanical properties correlated with hydroxyl value rather than with viscosity or phthalic anhydride content. He concluded that the length of the glyceryl phthalate chain was the factor most influencing the film properties.

The speaker continued by considering type 6, the physical/chemical class involving solvent evaporation followed by the chemical reaction of the components, and itemised in particular two-can coatings of the epoxy/amine and epoxy/polyamide type. He showed how film properties could change by the neglect of changes in conditions during drying, and illustrated the methods of following the chemical reaction by infra-red spectroscopy or methyl ethyl ketone extraction. The effects of solvent retention on apparent drying were well illustrated by this technique where, in two formulations with the same reactants ratio but different solvent mixtures, film properties varied considerably due to retention in the film of high boiling solvents although the chemical reaction was completed in the same time.

Dr. Talen concluded by a consideration of class C, the purely chemical class. After itemising the advantages of solventless systems, he pointed out that one of the outstanding examples of this class of materials was linseed oil. The artistic use of this film-former was delightfully illustrated by a number of excellent slides of the work of such masters as Rembrandt and Van Gogh.

During the discussion which followed the lecture, and to which contributions were made by Dr. L. Valentine, Dr. R. F. Bowles, Dr. M. L. Ellinger, Mr. J. J. Scowron, Mr. J. A. L. Hawkey, Mr. H. A. Idle, Mr. W. H. Moss, Mr. P. N. Whitby, Dr. L. A. Jordan and Dr. S. H. Bell, the whole field covered by Dr. Talen in his lecture was surveyed. The influence of water-soluble constituents and coalescing agents in emulsion paints, the effect of high boiling solvents on film gloss, the effect of selective evaporation of solvent/non-solvent mixtures, and the artistic use of film-formers as opposed to the more utilitarian uses, were some of the points raised.

The meeting was concluded by the proposal of a vote of thanks by Mr. H. A. Idle, who said that in the printing ink field the types and classes enumerated by Dr. Talen could not be so readily accepted in view of the necessity to consider in addition the effects of the medium upon which the print was applied and, in particular, penetration, filtration, pH, *etc*.

SOLVENTS IN THE SURFACE COATING INDUSTRY

The third meeting of the session was held at Manson House on 10 October, when Dr. L. M. Barakan, of the Distillers Co. Ltd., presented a paper entitled "Solvents in the Surface Coating Industry".

Dr. Barakan discussed solvents on the basis of the different types of surface coating and stated that the choice of solvent for any particular use was dependent, first, on the type of resin to be used, secondly, on the method of application and, thirdly, on the drying process. The properties of solvents which could be of importance were compatibility, solvent power, evaporation rate, dilution ratio, blush resistance, electrical characteristics, flammability, odour, toxicity and price. It was usually necessary when making a final choice to adopt a compromise. In the case of alkyd resin-based formulations, the usual solvents were hydrocarbons, and generally there were few difficulties in obtaining suitable blends. Odourless solvents had become more popular in the United States than in the United Kingdom; these had low solvent power, but this could be offset by the addition of solubilising agents. Dr. Barakan then proceeded to a detailed consideration of the properties required in solvents for nitrocellulose lacquer formulations. He commented on the use of alcohols as latent solvents and demonstrated the effect on dilution ratios of the addition of alcohols, such as methylated spirit and butanol, to ketones and esters. The problem of blushing and solvent balance was also discussed. The question of toxicity and odour and their interrelation was considered, the lack of an objective method of measurement of odour being regretted. The use of threshold limit values and the comfort level of odour were mentioned as guides to the imminence of a dangerous concentration of solvent in the atmosphere.

The use of vinyl resins in lacquer formulations involved an appreciation that the solids/viscosity relationship with this type of resin was even more important than with nitrocellulose; and ketones were the most important of solvents in this field. Acrylic lacquers were in use in the automobile industry in the United States, where a larger use was made of air-drying formulations. Ketones, esters, aromatic hydrocarbons and glycol ethers were the solvents necessary in this field. So far as the speciality formulations based on epoxy and polyurethane resins were concerned, it was important to remember that only anhydrous solvents could be used with the isocyanates, and esters, particularly the glycol ether esters, had been recommended for this use. Dr. Barakan continued by discussing the way in which application method affected the choice of solvent and, in relation to electrostatic spraying, he listed the dielectric constant and resistivity of a number of well-known solvents. He ended by suggesting that, although the impact of water as a solvent was increasing and had obvious advantages over organic solvents, the two would ultimately lead a life of peaceful co-existence.

During the course of the discussion after the lecture, Mr. W. F. Daggett enquired about the possibilities of the use of nitroparaffins in the United Kingdom. Dr. Barakan said that these solvents were not produced in this country and that might have hampered their development. He referred to a paper by Bennett in Official Digest, September, 1960, when an extensive survey of the properties of 2.nitropropane was given, and developed on the basis of solubility parameters. Mr. R. A. May asked for advice on a suitable solvent system for a urea-formaldehyde resin surface coating unmodified by alkyd resins. Dr. Barakan replied that, although he did not know of any such coatings, the normal xylene/butanol mixtures should be the most suitable. Mr. R. N. Wheeler requested further enlightenment on the matter of the importance of the electrical properties of the solvent in electrostatic spray application. Dr. Barakan said that there seemed to be little fundamental knowledge on the subject. From his own experience diacetone alcohol, glycol ethers and mesityl oxide as solvents improved the performance of paints in an electrostatic field, but he could give no explanation of the causes. He thought that it was a subject which required very much more study than had been given to it so far.

Mr. J. R. Berry asked whether there was an aromatic hydrocarbon solvent which, when mixed with butanol, would give a flash point above 73°F. Dr. Barakan suggested solvent naphtha. Mr. N. R. Fisk raised the question of odour measurement, pointing out that some original work not confined to threshold values had been carried out on the subject which had been noted in correspondence appearing in the technical press about five years ago. He continued by making a very strong appeal that the correct chemical nomenclature for solvents should be used in trade literature and the industry generally. He thought that the misuse of synonyms and homonyms was probably at its worst in the solvents section of the raw materials industries and quoted hexylene glycol as a conspicuous example of a misleading misnomer. Dr. Barakan agreed that the use of misnomers was very reprehensible, but suggested that it was so prevalent because of convenience. On the matter of odour measurement he stated that he had mentioned odour in conjunction with threshold values only when the level of a powerful odour might give early warning of danger. Mr. P. E. Randall continued the discussion on

odour and threshold limit values in relation to toxicity and said that threshold limit value depended on the comfort level. He thought that it would be wrong to use a table of threshold limit values as a basis for the deduction of the relative toxic effect of solvents. He also commented on the uses of alcohols as latent solvents in combination with methyl isobutyl ketone and the use of isopropanol with this solvent and with methyl ethyl ketone. Dr. Barakan agreed that threshold limit values had to be treated with caution and due allowance made for both the odour and volatility of the solvent.

Mr. S. Kettle asked whether wet on wet spray application complicated the selection of solvent mixtures. Dr. Barakan replied that there was complication in the formulation, particularly when the two coats were not of the same product.

The meeting concluded by the proposal of a vote of thanks to the lecturer by Dr. R. J. Wicker, who commented on his experiences with misnomers in the solvent field. He was relieved that Dr. Barakan had not attempted to introduce any new solvent and he noted the interesting and devastating effects on transport in the United States caused by nitromethane.

C. R. P.

SOUTHERN BRANCH

FLOODING AND FLOATING IN PAINTS

The second meeting of the current session was held on 9 October at the Royal Beach Hotel, Portsmouth, when a paper entitled "Flooding and Floating in Paints" was read by Mr. L. A. Logue.

After defining the terms "flooding and floating" as he meant them, the speaker described some of the research that had been directed towards the solving of this problem. Experiments concerning various alkyds and pigment combinations had shown many effects without pointing to the cause. Mr. Logue showed evidence of the control by additives which, if incorporated correctly, could cure or at least minimise these defects.

More recent work on the study of the nature of the molecules of titanium dioxide and phthalocyanine blues had indicated possible reasons for flooding and floating. However, since the defects were also found in varying degrees, depending on the vehicle and solvent used, the findings could not be conclusive.

Mr. Logue concluded by saying that an awareness of correct and balanced formulating, choice of additive if needed and true balance of solvent could do much to reduce these faults.

Following the lecture an interesting discussion took place in which Mr. P. Targett, Mr. J. Smith, Mr. R. D. Orwin and Mr. F. W. Davies took part. Mr. J. Smith proposed the vote of thanks which was endorsed by the meeting. In conclusion, Mr. Logue introduced a new film entitled "Titanium Oxide", which presented the aspects of manufacture and use of this pigment in a most pleasing manner.

A. F. R.

Manchester Section

THE MODERN CHEMISTRY OF ORGANIC PIGMENTS

To a very good attendance in Manchester, Dr. Gartner presented the first lecture of the season on 13 October, dealing with "The Modern Chemistry of Organic Pigments". The lecturer gave a brief survey of those developments in pigment chemistry which had led to the creation of particularly high grade organic pigments. He described how the advent of the phthalocyanines had provided pigments suitable for universal use, since they possessed maximum all-round fastness. However, their shade range was still restricted to blues and greens, despite tremendous efforts to extend it. The search for similar fastness in other shades had led to the investigation of other chemical classes, and had produced the thioindigo pigments, anthraquinone pigments, and the perinone and perylene pigments, all of which were either identical with or chemically related to vat dyestuffs. Further, this work had led to the development of the dioxazines and the quinacridones which were polycyclic compounds no longer related to vat dyes. Quite a number of these new pigments approached the fastness of the phthalocyanines, and a few actually reached it.

In addition, work in the field of azo pigments, still by far the most important class, had led to remarkable improvements in fastness properties. One line of work had been to use complicated diazo components and to prepare various metal salts and metal complexes in the production of pigments by the usual coupling method. Another line of work had been the production of azo pigments of higher molecular weight by reversing the reaction sequence in synthesis. These "azo condensation" pigments differed from conventional azo pigments in that their fastness properties were well balanced and of a higher general standard. Up to the present time they had been mainly used in the coloration of plastics. The leading brands of the range approached the fastness standard of the phthalocyanines and could thus be employed in high quality organic coatings.

A survey of the chemistry of high grade pigments showed that most of them were neutral compounds without salt-type or complex groups, and that many of them stemmed from chemical classes not previously used in pigment chemistry.

A further survey showed what shades the various chemical classes had yielded to date as a result of the search for high-grade pigments. One specific group of shades dominated in each class with the exception of the anthraquinones, which covered a remarkably wide range. However, the phthalocyanine standard in respect of both fastness and brilliance had by no means been reached in all shades. Furthermore, in no case had it been possible to get down to the phthalocyanine price level with other high-grade pigments.

An interesting discussion followed the lecture, covering the relation of molecular weight to light fastness, dispersion properties, polymorphic forms of quinacridone pigments and the flocculation of phthalocyanine blue. Dr. Gartner was congratulated in a vote of thanks for the excellent presentation of his lecture which had provided a most enjoyable evening for the large audience present.

I. S. M.

Midlands Section

THE PERFORMANCE OF METALLIC AND ORGANIC FINISHES

On 22 September at Regent House, Birmingham, Mr. R. J. Brown who is Materials Engineer for B.M.C. Ltd., presented a paper on "The Performance of Metallic and Organic Finishes"; Mr. N. H. Seymour was in the chair.

Mr. Brown began by describing the conditions which cars and spare parts had to resist, particularly during shipment to and operation in tropical and Arctic conditions. The main objective in applying organic finishes was corrosion resistance, combined with the covering of surface blemishes and the production of an attractive appearance.

The performance requirements were specified for each type of paint. For finishing coats the film had to withstand wide temperature and humidity variations, abrasion, weathering and possibly chemical attack, whereas for engine parts, oil and chemical resistance were of paramount importance.

Certain standard routine tests, such as viscosity thinning ratio, total solids content and pigment/vehicle ratio, were carried out on the paint itself. Tests on the finished coating included gloss, hardness, adhesion, flexibility, chip resistance, corrosion resistance and freedom from blemishes. The speaker discussed the tests which had been developed for assessing these requirements and how the results from these related to results under actual performance conditions. It was evident that considerable work had been carried out, particularly on resistance to staining and blistering and the factors affecting durability.

Of the three main metallic finishes, chromium plating, stainless steel and anodised aluminium, the first of these was dealt with in some detail and mention made of the duplex processes. Performance tests on metal finishes were severe, being mainly accelerated corrosion tests. A number of interesting examples of finishes were shown and their properties described during the lecture.

The discussion which followed was lively, and questions by the Chairman Mr. Edwards, Mr. Clarke, Mr. Morris, Mr. French and Mr. Taylor were dealt with very comprehensively by Mr. Brown. Mr. Northeast proposed the vote of thanks to Mr. Brown for his competent and interesting talk.

R. D. C.

Newcastle Section

JOINT MEETING

On 18 October the Section were guests of the local branch of the National Federation of Master Painters and Decorators at a pot-pie supper in the Crown Hotel, Newcastle. The branch President, Mr. Robson, was in the chair.

After the meal, Mr. D. C. Wall gave a short talk on corrosion and this was followed by a two-hour discussion on the lines of an informal brains trust. Considerable interest was expressed by the decorators in rapid drying enamels, and although none had adverse comments to make on thixotropic paints there was no enthusiasm either. On the assumption that only reputable paints were used, price seemed to be the prime factor in making any selection, yet it was obvious at the same time that water-thinned systems were being approached with great caution.

The use of emulsion paints as exterior undercoats was condemned, but it was agreed that certain advantages were apparent for interior work. Some time was spent in discussing the durability of clear varnishes and the effect on this property of pigmentation. The demand for clear varnishing work was decreasing as graining became a lost art.

The decorators appreciated the improvement in colour retention shown by first-class enamels over the past few years, but they considered that the development of a good, one-pack, fast drying knifing stopper with no shrinkage and excellent durability would be a worthwhile research project.

The O.C.C.A. members who answered most of the questions were Mr. J. Clark, Mr. D. C. Wall and Mr. E. A. Watson, whilst Mr. Gow and Mr. R. A. Bibby led for the decorators. The Chairman of the Section, Mr. A. W. Blenkinsop, thanked their hosts for a pleasant and useful evening.

J. A. W.

Some Considerations on the Formation of Films

The first meeting in the 1961-62 programme was held in the Royal Turks Head Hotel, Newcastle, on 5 October. Mr. A. Blenkinsop in the chair, welcomed the members to this session and thanked all those who had contributed in various ways by assisting Mr. E. Farrow at the very successful local O.C.C.A. Exhibition which had been held in Newcastle in connection with the Commonwealth Training Week.

Dr. H. W. Talen of the Paint Research Institute T.N.O., Delft, presented a paper entitled "Some Considerations on the Formation of Films". He introduced this by pointing out that film-formation was the last and very important stage in the sequence of chemical and physical processes leading to the end product of the paint industry, *i.e.*, the protective or decorative coating.

He differentiated between nine types of film-formation, these being divided equally into three distinct classes, namely formation by (a) purely physical processes, (b) purely chemical processes and (c) a combination of (a) and (b).

A fuller description was furnished of the processes involved during the drying, of three typical examples, *viz.*, a p.v.a. emulsion, linseed oil and alkyd resins and an amine cured epoxy system.

Mr. A. Duell proposed the vote of thanks to Dr. Talen and said that he felt that the great interest in the paper lay in the fundamental nature of the work, being of the type which the average paint technologist found little opportunity to tackle.

E. A. G.

West Riding Section

METAL PREPARATION AND PAINTING

At the first meeting of the session held on 12 September at the Hotel Metropole, Leeds, Mr. D. H. du Rieu, a member of the Section, delivered a paper on "Metal Preparation and Painting". Introducing the subject, Mr. du Rieu said that it would be restricted to the surface preparation of structural steel work and would not include factory production systems of surface preparation. He did not intend to deal with painting schemes in detail, but would mention painting where it was particularly related to the method of surface preparation.

Mention was made of the fact that, although everyone was made very conscious of the cost of corrosion now, owing to the considerable publicity the matter received, in practice the advice was often disregarded. The case for good surface preparation was primarily a matter of economics. In an average painting job the cost of preparation and application was approximately 80 per cent of the total cost and only the remaining 20 per cent was for materials. Consequently, it was sound economics to use good quality materials and good surface preparation. Since labour costs were increasing, it was likely that the differential between the cost of preparation and application and the cost of materials would be increased further.

In the first section the lecture dealt with physical adhering contaminants, such as oil, dirt and old paint. General methods were given for removing these contaminants, but it was pointed out that because of the wide variety of possible contaminants, particularly chemical ones, each individual case had to be considered separately.

The next section dealt with the removal of chemically bonded contaminants, which included rust and mill scale. The four main methods for effecting removal were then described, being manual cleaning, including the use of power driven tools, flame de-scaling and abrasive blasting. In each case the equipment required, the method used, and the various safety precautions necessary were outlined. The lecturer discussed the advantages and disadvantages of each method, drawing considerably on his own practical experience. Finally, a comparison was made between the various methods available, and the speaker stated that, although other methods had their place, the only reliable one in the majority of cases was abrasive blasting, and that this method should always be used wherever possible.

The lecture was concluded by a vote of thanks from Mr. Cass, who mentioned that he was very pleased to hear a lecture from one of the members of the Section and hoped that more local members would give papers in the future. After a short break there was a question time and it was obvious from the number and character of the questions that those at the meeting were extremely interested in this particular subject.

897

1961

FOURTEENTH TECHNICAL EXHIBITION, 1962

MINISTER FOR SCIENCE TO OPEN EXHIBITION

The Exhibition Committee is pleased to announce that The Rt. Hon. Viscount Hailsham, q.c. (Lord President of the Council and Minister for Science), has accepted its invitation to be the Guest of Honour at the Fourteenth Technical Exhibition Luncheon and to reply to the Address of Welcome by the President, Dr. H. A. Hampton.



THE RT. HON. VISCOUNT HAILSHAM, Q.C.

The Exhibition Luncheon will take place at the Criterion Restaurant, Piccadilly, London, W.1, at 12.15 p.m. for 12.45 p.m. on Monday, 26 February, 1962. At 3 p.m. on the same day, Lord Hailsham will the marked attendance of overseas visitors, declare open the Exhibition (which takes and interpreters will be present to help place at the Royal Horticultural Society's both visitors and exhibitors from overseas. Hall. The Exhibition will be open until languages.

7 p.m. on 26 February and on the following three days it will be open from 10 a.m. to 7 p.m.

The charge for Exhibition Luncheon tickets will be £1 12s. 6d. and forms of application for these will be included in all copies of the Exhibition Official Guide, which will be sent individually to all members of the Association in the New Year. They will also be sent to manufacturing companies in the United Kingdom as well as to chemists and technologists individually in Western Europe and Scandinavia. Non-members wishing to obtain copies of the Official Guide may obtain them, without charge, by making application in writing to the General Secretary of the Association, at the address given on the front cover of this Journal

The Exhibition provides a focus for the technical display of advances in raw materials, plant and machinery for use in the paint, printing ink and allied industries. The Fourteenth Technical Exhibition will cover a larger floor area than any previous Exhibition, and there is participation from companies in countries in both the European Free Trade Area (Denmark, Sweden and Switzerland), the European Common Market (France, Germany and Holland) as well as in the United States of America. There are, of course, many overseas companies who will be showing through associated companies in the United Kingdom.

A feature of recent Exhibitions has been Old and New Halls, London, S.W.1) by On this occasion the invitation cards to the cutting a tape at the entrance to the Old Exhibition are being printed in four

Alphabetical List of Exhibitors

Albro-Fillers & Engineering Co. Ltd. Allied Colloids Ltd. *Amoco Chemicals Corporation A.P.V. Company Ltd., The Armour Hess Chemicals Ltd. Associated Lead Manufacturers Ltd. *Astles Chemical Co. Ltd. Bakelite Ltd. **Baldwin Industrial Controls** *Barter Trading Corporation Ltd. (Kemisk Vaerk Koge, A/S) Beck, Koller & Co. (England) Ltd. Berk, F. W., & Co. Ltd. B.I.P. Chemicals Ltd. Boehm, Frederick, Ltd. Boulton, William, Ltd. British Celanese Ltd. British Oil & Cake Mills Ltd. British Oxygen Chemicals Ltd. British Resin Products Ltd. British Titan Products Co. Ltd. *Buhler Brothers (England) Ltd. Burts and Harvey Ltd., incorporating Alchemy Ltd. *Bush, Beach & Segner Bayley Ltd. Campbell, Rex, & Co. Ltd. and the *Runnymede Dispersions Ltd. Chemical Supply Co. Ltd. Carless, Capel & Leonard Ltd. *Chemische Werke Huel A.G. Ciba (A.R.L.) Ltd. Ciba Clayton Ltd. Cornelius Chemical Co. Ltd. *Coulter Electronics Ltd. Cray Valley Products Ltd. Crosfield, Joseph, & Sons Ltd. Croxton & Garry Ltd. Distillers Co. Ltd. and Honeywell & Stein Ltd. *Dow Chemical Co. (U.K.) Ltd. **Dunlop Chemical Products Division** Durham Raw Materials Ltd. Elliott, H. J., Ltd. Esso Petroleum Co. Ltd. Evans Electroselenium Ltd. Farbenfabriken Bayer Aktiengesellschaft Ferranti Ltd. Geigy Co. Ltd. Golden Valley Colours Ltd. Goodyear Tyre & Rubber Co. (G.B.) Ltd. Hardman & Holden Ltd. Hercules Powder Co. Ltd. Imperial Chemical Industries Ltd. Johnson, Matthey & Co. Ltd. *Joyce, Loebl & Co. Ltd.

Kek Ltd. *Kingsley & Keith (Chemicals) Ltd. Kunstharsfabriek Synthese, N/V Laporte Titanium Ltd. *Lennig, Chas., & Co. (G.B.) Ltd. Marchant Brothers Ltd. Metal Propellers Ltd. Micafine Ltd. Mitchell, L. A., & Co. Ltd. Monsanto Chemicals Ltd. *Morris & Ingram (London) Ltd. National Adhesives Ltd. *Noss Mayo Dispersion Products Ltd. Novadel Ltd. Paint Manufacture Paint, Oil & Colour Journal Paint Research Station, The Paint Technology Pascall Engineering Co. Ltd. *Pechiney-Saint-Gobain Plastanol Ltd. Premier Colloid Mills Ltd. Price's (Bromborough) Ltd. Research Equipment (London) Ltd. Resinous Chemicals Ltd. *Scado-Archer Daniels, N.V. Schenectady-Midland Ltd. Scott Bader & Co. Ltd. Shawinigan Ltd. Sheen Instruments (Sales) Ltd. Shell Chemical Co. Ltd. Silverson Machines (Sales) Ltd. Spelthorne Metals Ltd. Surface Coating Synthetics Ltd. Svenska Oljeslageri Aktiebolaget Swada Ltd. & H. Haeffner & Co. Ltd. Tin Research Institute Trade & Industrial Press Ltd. (The Paint Journal) *Translation & Technical Information Service Union Carbide Ltd. Universal Oil Co. Ltd., Premier Oil & Cake Mills Ltd. & J. L. Seaton & Co. Ltd. Vinyl Products Ltd. Winkworth Machinery Ltd. Wolf, Victor, Ltd. Younghusband Stephens & Co. Ltd. **Technical Education Stand** O.C.C.A. Information Bureaux

^{*}Denotes exhibitors who have not shown at previous Exhibitions

NOTES AND NEWS London Section



[Photograph by

Halifax Photos]

Members of the London Section Junior Group pictured here with their hosts at the works of Burrell & Co. Ltd. during a recent visit. Present on this occasion were Mr. T. Whitby, Mr. H. Elliott, Mr. C. Condon, Mr. R. Furner, Mr. M. Heath, Mr. R. Golds, Mr. J. Bird, Mr. K. Shaw, Mrs. J. Horvath, Mr. G. Gully, Mr. P. Pitfield, Mr. A. Ferris and Mr. J. O'Neill

JUNIOR GROUP'S WORKS VISIT

Fourteen members of the Junior Group took part in the works visit on 21 September to Burrell & Co. Ltd. The members of the party were met by Mr. Maloney and Mr. Dennett from the company and a short discussion developed during the serving of light refreshments.

commenced, covering the wide field of extenders, etc., toners and lakes. Finally,

the party reunited to visit the processing and blending departments. This proved to be a most interesting and comprehensive visit.

Refreshments were then supplied and their hosts were most helpful in answering questions. Mr. C. Condon, the Junior Group's Chairman, proposed a vote of The party then split up and the tour thanks which brought the visit to a close.

M. H.

Midlands Section

PRESENTATION TO MR. S. MARKS

On 5 October the Midland Sections of O.C.C.A. and the Royal Institute of Club in a presentation to Mr. Sidney Chemistry, together with the Midland Marks on his retirement from teaching at Varnish, Paint and Colour Manufacturers' the Birmingham College of Technology.

Midland Association and the Paint Students' Association, joined the Birmingham Paint, Varnish and Lacquer



Photograph taken at the presentation of a silver tray and an illuminated address to Mr. S. Marks on his retirement. From left to right are: Mr. G. King (Chairman, Midlands Section of the Royal Institute of Chemistry), Mr. N. H. Seymour (Chairman, Midlands Section, O.C.C.A.), Mr. C. J. A. Taylor, who made the presentation, Mr. S. Marks, Mr. H. J. Clarke (President, Birmingham Paint, Varnish and Lacquer Club) and Mr. L. V. Jennings (President, Midlands Paint Students' Association).

was also a member of each of the five Sidney Marks and Mrs. Marks. associations represented. During his time in the Department of Chemistry, Mr. Marks had not only instructed and guided so many students in paint and other industries but had also won their respect and affection. Mr. Taylor also described in particular Mr. Marks's close association with Dr. R. S. Morrell and his long and valuable services to the City and Guilds Institute.

Mr. C. J. A. Taylor, who made the showing the emblems of the five assopresentation, had been closely associated ciations, together with a silver tray, was a with Mr. Marks during this period and token of the esteem and affection held for

> Mr. Marks recalled his early days at the College of Technology and how the pattern of lectures had changed since 1922. A whole new technology in resins, pigments and paint manufacture had been built since the days when Mr. Sturge and Dr. Newton-Friend carried out the preliminary work. In thanking Mr. Taylor and the associations concerned, Mr. Marks expressed his great pleasure in seeing so N.H.S.

The presentation, an illuminated address many of his friends present.

Register of Members

The following elections to membership have been approved by the Council. The Sections to which the new members have been attached are given in italics.

Ordinary Members

ADAM. RICHARD, "Dunedin", 40 Ashtown Grove, Navan Road, Dublin 7, Eire.

(Bristol) ASTLES. PHILIP GOOLDEN, B.SC., CHEM. ENG., 75 Mashiters Walk, Romford, Essex.

(London)

December

BLACKMAN, FRANCIS ALLEN, Hercules Powder Co. Ltd., Church Manorway, Erith, Kent. (London)

- BOWKER, KENNETH, L.I.R.I., Industrial Dyestuffs Ltd., Bonding House, 26 Blackfriars Street, Manchester, 3. (Manchester)
- BOYLE, JAMES JOSEPH, B.SC., M.I.C.I., Shell Chemical Co. Ltd., 33/34 Westmoreland Street, Dublin, Eire. (Bristol)
- CATHERALL, KENNETH DAVID, B.SC., 15 Swan Street, Alvechurch, Worcs. (Midlands)
- CHRISTENSEN, GUNNAR, M.SC., 14 Kastrupvej 3 sal., Copenhagen S, Denmark.

(Overseas)

- HILL, WILLIAM VICTOR, A.S.T.C., 19 Macdonald Avenue, Auburn, New South Wales, Australia. (New South Wales)
- JACOB, DEREK ERNEST, Armour-Hess Chemicals Ltd., 6 Arlington Street, St. James's, London, S.W.1. (London)
- KOTWAL, HOSHIDAR PESHOTAN, B.SC., 20 Eduwi Dinshaw Building, Preedy Street, Karachi (3), Pakistan. (Overseas)

MOLE, LEONARD WILLIAM, 35 Strode Road, Forest Gate, London, E.7. (London)

- O'BRIEN, BARRIE JAMES, 354 Avoca Street, Randwick, New South Wales, Australia. (New South Wales)
- OTT, EUGENE H., B.SC., Grand Rapids Varnish Corporation, 1350 Steele Avenue S.W., Grand Rapids 2, Michigan, U.S.A. (Overseas)

ROZEMA, PIETER, 101 Ryde Road, Pymble, New South Wales, Australia.

(New South Wales)

Associate Members

ELLIS, RONALD, 11 Ash Street, Wardley, Swinton, Manchester. (Manchester) FERGUSON, JOHN RICHARD, 261 Worona Road, Engadine, New South Wales, Australia. (New South Wales)

HARLEY, ROY ERNEST, 21 Adelaide Street, West Ryde, New South Wales, Australia. (New South Wales)

JONES, JAMES BRUCE, I.C.I. Ltd., 4 Blytheswood Square, Glasgow, C.2, Scotland. (Scottish)

- JONES, THOMAS JAMES, Bitumen & Oil Refineries (Australia) Ltd., Bunnerons & Military Roads, Matraville, New South Wales, Australia. (New South Wales)
- YOUNG, GRAHAM BISSET, I.C.I. Ltd., Templar House, 81/87 High Holborn, London, W.C.1. (London)

Junior Member

KNUCKEY, CHARLES HOWARD, 22 Queens Road, Skewen, Neath, Glam. (Bristol)

Obituaries

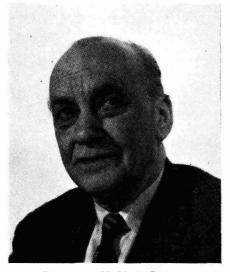
MR. HUGH REYNOLDS

Mr. Hugh Reynolds, Technical Director of Cowan Bothers (Stratford) Ltd. of Stratford, London, E.15., died on Friday, 13 October, aged 65. His health had been poor for the past two years and became worse during the past few months.

Mr. Reynolds was educated at Tottenham Grammar School, the Borough Polytechnic, and Sir John Cass Institute. He joined Cowan Brothers in 1912 in the Laboratory and Works and became a Director in March 1921.

He had an extensive knowledge of pigments and allied materials and was a keen member for years of the Borough Old Students Society and the Oil and Colour Chemists' Association. He also served for several years on the Pigment Panel of the Paint Research Station.

A very modest and courteous man, he was highly respected by his many friends in the pigment, paint, printing ink and plastic Industries for his sincerity and integrity. He leaves a widow and two sons.



PROFESSOR H. V. A. BRISCOE

PROFESSOR HENRY VINCENT AIRD BRISCOE, D.SC., A.R.C.S., D.I.C., 1888-1961

A Vice-President: 1950-51

An Appreciation

- On discovery: "Whether the discovery be great or small, the making of it affords the keenest satisfaction we scientists can enjoy".
- On serendipity in research: "It is important for any investigator or any research institution to do a sufficient proportion of 'd... silly' experiments''.
- On assisting in the revision of a well-known chemical dictionary: "It taught me to read and write and to cope with tyrants".
- On motoring: "I am a hardened motorist, who bought a motor cycle in 1911 and have owned some kind of petroliferous vehicle ever since"
- On having his cigarette lighted: "With a match for a cigarette you're the quickest man on the draw I know'

These are some of the things said by "H.V.A.B." (many people referred to him with affectionate brevity by his initials) from which those who did not know him could deduce much about him, including his somewhat dry humour. But only those who knew him can have the pleasurable remembrance of the thoughtful smile and the occasional exuberant laugh that accompanied such remarks.

He was a man of parts, of skills, of versatility, and of wide service to others his retirement (1954). collectively and individually. He was habitually pleasant and gentle in manner, and of the many subjects which engage

and could be gay. He could tell a good story and could cloak wisdom in humour; he could be firm, but seldom stern.

For many years he suffered physical discomfort and pain, but it seemed to disturb none of his characteristic readiness to give a friendly ear to individuals' problems and, having listened (a virtue this), to give advice which in the event was so frequently found to be sound. Such an appreciation of him could be written only by one who knew him well and experienced such things, but many others could testify similarly.

So strong are these personal impressions that one could almost wish to write only of the man (and that extensively) and omit his works; but the record of his considerable and diverse activities tells so much of his character and helpfulness. His range of interests was enormous, which revealed his ability and energy rather than diffuseness, for he was much concerned lest excellence should be sacrificed to conformity.

A Londoner and proud of it, he went in 1906, at the age of 18, to the Royal College of Science, which was soon to become a constituent college of the Imperial College of Science and Technology. After graduation, he assisted Sir Edward Thorpe in the revision of the famous Dictionary of Applied Chemistry, and became a demonstrator to, among others, a young student named Louis Arnold Jordan.

Resigning his full-time academic appointment in 1916, he became concerned with chemicals and munitions, but maintained contact with the college by lecturing in engineering chemistry. He also assisted the Sir John Cass College. Then followed a series of university appointments: Armstrong College (now King's College) Newcastle, Professor of Inorganic and Physical Chemistry (1921), Head of the Chemistry Department (1925); University of Durham, Dean of the Faculty of Science; Imperial College, Professor of Inorganic Chemistry (1932), Director of the Laboratories of Inorganic and Physical Chemistry (1938), and, finally, Emeritus Professor on

Such, briefly, was his academic career,

his attention he will perhaps be best Imperial College and the University of remembered for his work on the "newer" elements and his even earlier work on the on Recruitment and Training of Science precise determinations of atomic weights.

He was ever mindful of practical applications of scientific knowledge and made contributions to studies of industrial dust hazards, anti-pest measures for stored foodstuffs, the chemistry of dairy products and the purity of water supplies.

His interest in paints and hence in our own Association was in parallel with his advisory work for the Walpamur Company from 1926 onwards, and with his close of his diverse interests, already apparent. interest in the Research Association of One activity however is perhaps especially British Paint, Colour and Varnish Manu- significant in rounding off the story of this facturers (from 1927) of which, after remarkable man, whose first occupation on considerable Council service, he became graduation, additional to his laboratory the Chairman of the Technical Advisory work, was to assist with Thorpe's Diction-Committee in 1945 and President in 1951, ary, Just before his death he was actively Many will remember his address at the engaged editorially in the preparation of 21st birthday celebrations at the Paint supplementary volumes to Mellor's monu-Research Station entitled "Following the mental "Comprehensive Treatise on In-Unexpected"; it was then that he made the organic and Theoretical Chemistry". memorable utterance about doing "d . . . silly experiments".

He was always interested in the welfare of young people and of those who taught them; the two came together in his investigations of the accuracy of the marking of examination papers. He gave much attention to the training of technicians in university service. He was Chairman of technician training committees at the

London, and of a National Joint Committee Laboratory Technicians. He was for many years Treasurer of the Association of University Teachers, and his election in 1958 to the Fellowship of Imperial College (a rare honour) was in special recognition of his services to his college and to university education generally.

Many other chairmanships and committee memberships could be noted, but would serve only to emphasise the major trends

Finally, it is proper we should return from his works to the man; to one who, in the words of The Times obituary notice, "loved contact with men of all varieties of experience and outlook, for he always argued that education is acquired mainly 'through the skin' ".

We shall remember him.

S. H. B.

NEWS OF MEMBERS

Mr. H. Warson, an Ordinary Member attached to the Midlands Section, has been appointed Development Manager, Polymers, of Dunlop Chemical Products Division.

APPLICATIONS FOR ORDINARY MEMBERSHIP

At the Council meeting held on 15 February, 1955, the Council passed a resolution in accordance with Association Rule 5 (ii), reaffirming the standard of competence for the election of candidates to Ordinary Membership of the Association, and it is felt that members would like to be aware of this information when sponsoring candidates for election.

The Council's resolution reads 38 follows:

THAT the qualifications for the granting of Ordinary Membership shall be-

1. A degree in a scientific subject or any generally accepted equivalent qualification; or any technological qualification in a subject covered by the Association.

2. Or where there is adequate evidence of the technical competence of the candidate other than the obtaining of the qualifications mentioned above, the qualifying period of practice in the industries covered by the Association shall normally be not less than seven years.

BINDING OF THE ASSOCIATION'S JOURNAL the Royal Photographic Society are invited,

Members will be pleased to know that W. Heffer & Sons Ltd., Hills Road, Cambridge, will undertake the binding of back volumes of the Association's *Journal*, sent in by individual members, at a cost of 18s. 6d. per volume.

Members wishing to avail themselves of this facility should send the parts direct to Heffer & Sons Ltd., enclosing a remittance of 18s. 6d. and ensuring that notes bearing their names and addresses are enclosed with the parcels.

CHANGE OF ADDRESS

Since membership of the Association is on a personal basis, members are asked to note that, although the General Secretary's office may be notified of the change of address of companies, this does not mean that such changes are automatically noted on the record cards of members employed by those companies.

Where a member uses his company's address for communication purposes, it is necessary for him to inform the General Secretary's office of any change in the address and, at the same time, to quote his personal number, if possible.

THE F. W. CLARK MEMORIAL PRIZE

It has been announced that the 1960 F. W. Clark Memorial Medal and Prize has been awarded to Mr. D. P. Maple, a plastics chemist and a student of East Ham Technical College, London. Mr. G. Copping, now an Honorary Member of the Association, raised a sum of approximately £250 from friends in the Paint Industries Club to commemorate his colleague, Mr. F. W. Clark, who was killed during the war while on a technical mission to the United States. The fund was passed to the Association, who in turn passed it to the City & Guilds of London Institute to provide an Annual award.

A.P.T. 1962 MEETINGS

The first meeting of the Association of Printing Technologists, to which members of the Scientific and Technical Group of

the Royal Photographic Society are invited, will take place on 23 January and will consider two papers by Mr. F. Pollak (Kodak Ltd.). His subjects are "The Contrast of Moiré Patterns in Half-Tone Gravure" and "Split Filter Exposures in Colour Reproduction".

Operational research, what it is and what it can do for the industry, will be discussed by a panel of experts on 21 February. The last meeting will be addressed by R. C. McIntosh and H. V. Purdy (Pan World Press Ltd.) on "A New System of High-Speed Photocomposition".

The Sixth Conference will be held on 16 April and the programme details will be announced early in the New Year.

CENTENARY OF PLASTICS, 1862-1962

A Joint Committee of the Plastics Institute and the British Plastics Federation has been set up to organise a number of events in connection with a Centenary of Plastics, 1862-1962.

It has been decided to celebrate the centenary in 1962 because it was at the International Exhibition in London in 1862 that Alexander Parkes, patentee and exhibitor, of Birmingham, showed for the first time to the public *Parkesine*.

The programme of events to be organised by the Joint Committee includes the following:

- (a) A banquet in Guildhall in June.
- (b) The preparation of historical records on tape and possibly on film.
- (c) The publication of a book giving the story of plastics and containing a detailed history of celluloid.
- (d) Supplements in the national press on the history of plastics.
- (e) Specially designed stickers for letters and for office franking machines.
- (f) Contributions in the national and technical press and on radio and television.
- (g) Open days in firms in the plastics industry.
- (h) Special meetings and lectures throughout the centenary year culminating in Interplas 1963.

SIXTH I.S.F. CONGRESS

As already announced in the July issue of the Journal, the Sixth I.S.F. Congress will take place from 9-13 April, 1962. The Congress will be formally opened by the Lord Fleck, President of the Society of Chemical Industry, at the plenary meeting on 10 April, when he and the President of the Congress, Dr. E. G. Woodroofe, will welcome delegates.

papers has been arranged, copies of which liminary programme of the Congress will a half days and will be held at the Imperial 14 Belgrave Square, London, S.W.1.

College of Science and Technology, London, finishing on the morning of 12 April.

The Congress banquet will take place at the Hyde Park Hotel on 11 April. Visits have been arranged to research and industrial establishments in the London area for 12 April, while there will also be visits to both Oxford and Cambridge the following day. A separate social programme has been arranged for ladies A programme of some sixty scientific accompanying the delegates. The premay be obtained in advance by intending be available soon and copies may be participants. Lectures will occupy two and obtained from the Congress Secretariat,

Forthcoming Events

(Note: Details are given of meetings arranged in the U.K. up to the 15th of the month following publication, and in South Africa and the Commonwealth up to the 15th of the second month after publication.)

Friday, 1 December

Midlands Section. Ladies' Evening at the George Hotel, Solihull. Reception 7 p.m. for 7.30 p.m.

Newcastle Section Junior Group. "Decorative Paints", by Dr. A. J. Seavell, in the Rutherford College of Technology, Northumberland Road. Newcastle upon Tyne, at 3 p.m.

Monday, 4 December

Bristol Section. Annual Dance at the Arnos Court County Club, Bristol.

Hull Section. "The Alkali Fusion of Fatty Acids", by Professor Weedon. Joint meeting with Oil and Fats Group of the S.C.I. and Hull Section of the R.I.C., at the Royal Station Hotel, Hull, at 7.15 p.m.

Wednesday, 6 December

Victorian Section. Annual Dinner, Melbourne University Union, Main Buffet. Guest speaker to be announced.

Thursday, 7 December

Association Council Meeting at 11.30 a.m. at Wax Chandlers' Hall, Gresham Street, London, E.C.2.

New South Wales Section. Annual Dinner at the "Amory".

London Section. "Aircraft Paints", by A. Macmaster, M.SC., A.R.I.C. (Ministry of Aviation), in the Lecture Theatre, Royal Society of Tropical Medicine and Hygiene, Manson House, 26 Portland Place, London, W.1, at 7 p.m.

Newcastle Section. "Driers: Past, Present and Future", by C. T. Morley-Smith, at the Royal Turks Head Hotel, Newcastle upon Tyne, at 6.30 p.m.

Bristol Section. "Paint Progress—a Myth?"—Debate. Joint meeting with Birmingham Paint, Varnish and Lacquer Club, in Birmingham, at the Imperial Hotel, at 6.30 p.m.

Friday, 8 December

Manchester "Architectural Section. Approach to Colour in Building", by A. E. Hurst, F.I.B.D., to be given at the Manchester Literary and Philosophical Society, 36 George Street, Manchester, at 6.30 p.m.

Monday, 11 December

London Section-Southern Branch. "The Interaction of the Pigment with the Medium", by Dr. V. T. Crowl (Paint Research Station), at the Royal Beach Hotel, Southsea, Hants., at 7.30 p.m.

Thursday, 14 December

Scottish Section. "Wines Tasting and Testing", by H. F. Barnes, in the Grand Hotel, Charing Cross, Glasgow, at 7.30 p.m.

Friday, 15 December

Bristol Section. A paper by I. C. R. Bews, B.SC., A.R.I.C. (Honorary Editor, J.O.C.C.A.), at the Royal Hotel, College Green, Bristol, 1, at 7.15 p.m.

Saturday, 16 December

Scottish Section Student Group. Film Show at More's Hotel, 18 India Street, Glasgow, C.2, at 10 a.m.

Wednesday, 27 December

Western Australian Branch. Dinner and Golf Day.

Thursday, 4 January

Newcastle Section. "Have Cationic Surfactants Any Use in the Manufacture of Paints?", by M. K. Schwitzer, at the Royal Turks Head Hotel, Newcastle upon Tyne, at 6.30 p.m.

Saturday, 6 January

London Section—Junior Group. Lecture by A. R. H. Tawn (preceded by a short Annual General Meeting) in the Memorial Hall, Farringdon Street, London, E.C.4, at 7 p.m.

Monday, 8 January

London Section—Southern Branch. A paper from I.C.I. Ltd. (Paints Division) on colour appreciation, at the Polygon Hotel, Southampton, at 7.30 p.m.

Hull Section. "Milling of Titanium Oxide Pigments in Various Paint Machinery", by H. D. Jefferies (British Titan Products Co. Ltd.), at the Royal Station Hotel, Hull, at 7.15 p.m.

Tuesday, 9 January

West Riding Section. "The Preservation of Timber", by Dr. Crook, at the Hotel Metropole, Leeds, at 7.30 p.m.

Friday, 12 January

Newcastle Section—Junior Group. Film Show in the Rutherford College of Technology, Northumberland Road, Newcastle upon Tyne, at 3 p.m.

Manchester Section. "Thermo-Setting Acrylics", by K. E. Piggot, B.Sc., A.INST.P., at the Manchester Literary and Philosophical Society, 36 George Street, Manchester, at 6.30 p.m.

Scottish Section. Annual Dinner-Dance in Kintyre Suite, Central Hotel, Glasgow. 6.30 p.m. for 7 p.m.

Monday, 5 February

Victorian Section. Works Visit to Altona Petrochemical Co. Pty. Ltd., Steam Cracking Plant.

INDEX TO VOLUME 44-1961

Key:

- (C) = Correspondence
- (D) = Discussion
- (E) = Editorial and Comment
- (N) = Notes and News
- $(\mathbf{R}) = \mathbf{R}$ eviews
- (S) = Section Proceedings
- (T) = Transactions and Communications

AUTHORS

A

Abbott, P. H. J. The Physical Behaviour of Paint Films (D) 594 Adams, P. I. A Method of Hiding Power Determination and its Application as an Adamson, A. W. Physical Chemistry of Surfaces (R) Arbuckle, K. H. The Design of Unsaturated Polyester Resins for Surface Coatings 295 65 60 (D) . . Armitage, F. Lacquer Coating of Aluminium/Magnesium Alloys (D) 109 . . Armstrong, J. W. Lacquer Coating of Aluminium/Magnesium Alloys (D). Arnold, J. E. The Physics of Brushmarks (D). 110 • •

PAGE

632

885

. .

. .

B

Ballentyne, D. W. G., and Walker, L. E. Q.							
		in Ch				and	
	Mathe	matics (R)				311
Bank, W. H. (ed.). Printing Inks and Colour							313
Barton, J. F. Lacquer Coating of Aluminium	n/Magn	nesium /	Alloys	(D)			108
Beggs, M. J. Lacquer Coating of Aluminiun	/Magn	esium A	Alloys (D)			108
Beikmanis, J., Kovacs, L., and Russell, R.					for Sto	ving	
Enamels (T)							150
Benfold, A. Ships' Paints (D)							256
Bernhardt, E. C. (ed.). Processing of Thermo	plastic	Materi	als (R)				544
Bharucha, N. R. Pigments in Corrosion Pro							515
Black, W. see Moilliet, J. L. (R)							817
Blais, J. F. Amino Resins (R)							63
Boer, J. H. de (ed.). The Mechanism of Hete							384
Bowles, R. F. (ed.). Printing Ink Manual (R)			-				382
Brace, A. W. Lacquer Coating of Aluminiur							110
Brace, H. W. History of Seed Crushing in G				(2)			66
Bridle, P. F. The Correlation of Service Beha				hser			00
Characteristics in Air Drying Pa							687
Brunt, N. A. The Coating and the Substrate							809
Bullett, T. R. Solar Reflectivity of Paints (D							711
The Physical Behaviour of Pai			••	••	••	••	593
		IS (D)		••	•••	•••	
The Physics of Brushmarks (D			C. hatm	··· (T	· · ·	••	631
Bullett, T. R., and Rudram, A. T. S. The Co							787
Buser, A. L., and Polianskii, N. G. The U		Organic	Reage	ents II	n Inorg	anic	210
Analysis (R)	•••	••	••	••	••	••	210
Bussell, K. R. see Rischbieth, J. R. (T)	••	••	••	••	••	•••	351
see Rischbieth, J. R. (T)							367

C	PAGE
Cannegieter, D. The Coating and the Substrate (D)	812
Carter, C. F., and Williams, C. R. Science in Industry—Policy for Progress (R)	279
Champion, F. A. Lacquer Coating of Aluminium/Magnesium Alloys (D)	108
Chilton, C. H. (ed.). Cost Engineering in the Process Industries (R)	156
Clay, H. F. Pigments in Corrosion Protection (D)	538
— The Coating and the Substrate (D)	808
Cohen, J. D. The Behaviour of Printing Ink on Rollers (D)	616
——— The Wetting, Adhesion and Penetration of Surface Coatings on Wood	
$(D) \qquad \dots \qquad \dots \qquad \dots \qquad \dots \qquad \dots \qquad \dots \qquad \dots \qquad \dots \qquad \dots \qquad $	785
Colborne, R. S. Water-Dilutable Stoving Finishes (D)	149
Collie, B. see Moilliet, J. L. (R)	817
Colyer, L. E. Lacquer Coating of Aluminium/Magnesium Alloys (D)	109
Cox, P. D., and Williams, G. T. An Investigation into Drying Phenomena experi-	
enced with Alkyd Resins based on Soya Bean Oil	
	851
Cutforth, R. E. The Behaviour of Titanium Dioxide Pigments in High Speed	
Impeller Dispersion Mills (C)	547

D

Davidson, R. R. The Behaviour of Printing Ink on Rollers (D)	• •			617
The Physical Behaviour of Paint Films (D)	• •			591
Davis, D. S. (ed.). Chemical Processing Nomographs (R)				61
Dowling, D. G. The Behaviour of Titanium Dioxide Pigments	in	High	Speed	
Impeller Dispersion Mills (T)				188
The Behaviour of Titanium Dioxide Pigments	in	High	Speed	
Impeller Dispersion Mills (C)				547
Dunkley, F. G., and Earp, D. P. The Correlation of Service Behav	iour	of Pai	nt with	
Observed Physical Characteristic	cs in	n Air	Drying	
Paint for Structures (T)				649
Dunn, P. F. see Partington, A	• •			869

Earp, D. P. see Dunkley, F. G. (T)		• •	••	• •	649
Eckersley, B. Ships' Paints (D)			• •		256
Edwards, J. A. Laboratory Management and Techniques (R)				542
Ellinger, M. L. Water-Dilutable Stoving Finishes (D)		••			147
Entwistle, T. Ships' Paints (D)					256

F

Fink-Jensen, P. H. The Behaviour of Printing Ink on Rollers (D)	616
Solar Reflectivity of Paints (D)	712
Fisk, N. R. Some Aspects of Physical Assessment of Emulsion Polymer Films (
——— The Design of Unsaturated Polyester Resins for Surface Coatings (
——— The Wetting, Adhesion and Penetration of Surface Coatings on Wo	ood
(D)	785
Fordham, S. (ed.). Silicones (R)	161
Freidlina, R. Ku., and Karapetyan, Sh. A. Telomerisation and New Synthe	etic
Materials (R)	816
Fry, E. S. J. Lacquer Coating of Aluminium/Magnesium Alloys (D)	108
Water-Dilutable Stoving Finishes (D)	148

Gardam, G. E. Lacquer Coating of Aluminium/Magnesium Alloys (D)			108
Gellman, A. Water-Dilutable Stoving Finishes (D)	• •		148
Glossop, J. Preparation of Some Metals for Painting (T)	• •	• •	269
Goodwin, F. The Exploration of the Solar System (R)	• •	••	816

Gray, D. R. Absorption of Paint Me			rates (]	D)		
——— The Physical Behaviour					••	· · ·
Gray, V. R. Absorption of Paint Med	(5)					••
The Coating and the Sub The Correlation of Service		of Paint	with C) hserve	d Phys	 ical
Characteristics in Air Dr					u i nys	icai
——— The Wetting, Adhesion a					on Wo	bod
(T)					••	••
Green, K. W. The Design of Unsatur	ated Polyester	Resins	for Su			(D)
Guminski, R. D. Lacquer Coating of					• •	••
see Meredith, F. M.	. P. (T)	• •	•••	••	•••	•••
	н					
Hall, R. W. The Design of Unsaturat	ted Polvester	Resins f	or Surf	face Co	atings	(D)
Hammond, E. Water-Dilutable Stovin						
				• •		
Hampton, H. A. Ships' Paints (D) Handscomb, J. A. Water-Dilutable St			••	••	••	••
Harper, C. A. Electronic Packaging v	vith Resins (R	.)	•••	• •		
Harrison, V. G. W. Absorption of Pa	int Media int	o Porou	is Subs	trates ()	D)	• •
Hawkey, J. A. L. Lacquer Coating of	Aluminium/I	Magnesi	um All	oys (D)	• •
Hedgecock, K. L. Lacquer Coating of	of Aluminium	/Magne	sium A	lloys (I		• •
Helvey, T. C. Moon Base—Technical					••	••
Herdan, G. Small Particle Statistics (Hindley, H.R. The Coating and the S	K)	• •	••	• •		• •
					· ·	•
Hoffmann, E. Efflorescence Phenome Sealers for Insulating F	Daint Films wi	(1) ith a Ui	ah Cor	 stent of		$\dot{\mathbf{T}}$
Hofling, Dr. Lacquer Coating of Alu						(1)
Holt, M. R. The Design of Unsaturat	ted Polvester I	Resine f	or Surf	ace Co	 atings (, ini
Holt, M. R. The Design of Chisatura	icu i oryester i	itesins i	or burn		utings (D)
	J					
Jeffreys, A. E. Michael Faraday: A Li	ist of his Lectu	ures and	Publis	hed W	ritings	(R)
Jenkins, V. F., Mott, A., and Wicker,	R. J. The D	Design o	f Unsa	turated	Polves	ster
Resins for Surfa	ce Coatings (Γ)				
Johnson, T. E. Ships' Paints (D)		·				
Value in 7 The Casting and the Ca	K					
Kalewicz, Z. The Coating and the Sul		••			•••	•
Karapetyan, Sh. A. see Freidlina, R.	KU. (K) d Daumravia (Р\	• •	* *	••	••
Karsten, R. Bauchemie für Schule un Kershaw, R. The Wetting, Adhesion a	a Baupraxis (R)	face C	oatings	on We	···
(D)			lace C	oatings		
Kiessling, G. C. see Teach, W. C. (R)	··· ··	••	••	••	••	
Kingcome I C Shins' Paints (T)						• • •
Kingcome, J. C. Ships' Paints (T) Kovacs, L. see Beikmanis, J. (T)			••	••	••	×
Knapman, C. E. H. (ed.). Gas Chrom	atography Al	stracts	1960 (R)		•••
Kraus, A. Der Cold-Check Test (R)	atoBraphy /it	, sti uets,	1700 (••
Kresser, O. J. Polypropylene (R)				**		
	L					
Laar, J. A. W. van The Coating and	the Substrate	(D)	••	••		
The Physical Beha	viour of Pain	t Films	(D)	••		
——— The Physics of Br	ushmarks (D)		••		••	
Solar Reflectivity	of Paints (D)	• •				
Some Aspects of	Physical Ass	essment	of Er	nulsion	Polyn	ner
Films (D)						
Landmann, A. W. The Wetting, Adhe	sion and Pene	etration	of Sur	face Co	atings	on
Wood (D)						
Lewis, J. D. The Design of Unsaturat			or Surfa	ace Coa	atings (D)
Liebermann, O. Water-Dilutable Stov			••	• •	••	••
Pigments in Corrosio	n Protection (D)		• •	• •	• •

	Μ			
Mack, G. W.	Absorption of Paint Media into Porous Substra	ates (T)		
	The Coating and the Substrate (D)			
	Some Aspects of Physical Assessment of Emulsi			
	The Wetting, Adhesion and Penetration of Surfa	ace Coatings	on Wo	bod
	(D)		••	••
Meason, M. J.	. F. see Pass, A. (T)	• ••	••	•••
	C. Water-Dilutable Stoving Finishes (D)		f i aaa	•••
Merediin, F. I	M. P. Methods of Testing Certain Mechanical J Films (T)		•	
	and Cuminali B D (T)	• ••	••	••
MillCCT	The Behaviour of Printing Ink on Rollers (T) .	• ••	••	•
Moilliet J. L.	, and Collie, B. Surface Activity (R)			••
Morris, C. H.	The Design of Unsaturated Polyester Resins for	Surface Co	atings ((D)
Morrison, G.	A. Systematic Qualitative Analysis: An Introdu	iction (R)		
	Runnerless Moulding (R)			
Moss, W. H.	The Correlation of Service Behaviour of Paint w	ith Observe	d Physi	cal
	Characteristics in Air Drying Paint for Structur	es (D)		••
Mott, A. see	Jenkins, V. F., et al. (T)		• •	
M.K. D.C				1
MCKee, R. S.,	, and Staddon, A. W. E. The Alcoholysis and	Acidolysis		
MeLoon W/	ceride Oils (T) .		• •	•
McLean, w. C	G. Ships' Paints (D)		•••	•••
	Ν			
Narcus, H. N	Metallizing of Plastics (R)			••
Newell, G. A.	Absorption of Plastics (R) Absorption of Paint Media into Porous Substr	ates (D)		
	A. Pigments in Corrosion Protection (D) .			• •
	. Ships' Paints (D)			••
Newton, D. S.	., and Rigg, J. G. The Exterior Durability of P		on Lit	10-
	pone and Zinc Sulphide (T).	• • •	•••	•••
North, A. G.	The Physics of Brushmarks (D)			• •
	Water-Dilutable Stoving Finishes (T)	 Sumfana Ca		•••
	The Wetting, Adhesion and Penetration of Wood (D)	Surface Co	atings	
	Wood (D)	• ••	••	••
	0			
Oates, J. A.	The Wetting, Adhesion and Penetration of Surface	ce Coatings	on Wo	bod
	(D)			••
O'Neill, L. A.	The Design of Unsaturated Polyester Resins for	Surface Co	atings ((D)
Orchard, S. E.	. see Smith, N. D. P., et al. (T)		••	• •
	Р			
Pantony D A	A. A Chemist's Introduction to Statistics, Theory	v of Error a	nd Des	ion
Lunceny, D. /	of Experiment (R)	, or Error a	IC Des	
Partington, A.	, and Dunn, P. F. The Limitations of Leaching	Rate Deter	minatio	ons
5,	of Anti-Fouling Composition	ns (T)		
Partington, J.	R., and Wheeler, T. S. The Life and Work	of William	h Higg	ins
Pass. A., and	Meason, M. J. F. Zinc Dust as a Protective F	Pigment: Pa	rt III	(T)
	Organic Coating Technology (R)			• •
Phillips, G.	Absorption of Paint Media into Porous Substrate	es (D)		• •
]	The Behaviour of Printing Ink on Rollers (D) .		· · ·	•••
	The Correlation of Service Behaviour of Paint wi		1 Physi	cal
(Characteristics in Air Drying Paint for Structures	s (D)	••	••
	The Physical Behaviour of Paint Films (T) .		••	•••
	The Physics of Brushmarks (D)	a a a		• •
Pinder, A. R.			••	• •
Pollanskii, N.	G. see Buser, A. L. (R)		• •	• •
Poucher, W. A	A. Perfumes, Cosmetics and Soaps (R)		· ·	
Pue C P T	. D. Lacquer Coating of Aluminium/Magnesiur	n Alloys (D		• •
rye, C. K. L	acquer Coating of Aluminium/Magnesium Alloy	s (D)	••	

	R						PAGI
Rains, H. G. Water-Dilutable Stovin Reiser, K. Pigments in Corrosion Pro-						••	149 539
Reynolds, G. E. J. The Design of Uns	aturated Polyes	ter Res	ins for		ce Coat	ings	
(D) Rhind-Tutt, A. J. see Smith, N. D. F	at al (T)	• •	• •	1 P	• •	••	60
Ridge, M. J. Gypsum Plaster as a Su	bstrate for Pair	t Film	с. с.(Т)	•••	•••	•	618
Rigg, J. G. see Newton, D. S. (T)		it i iiiii	5(1)	• •	• •	•••	83
Rischbieth, J. R., and Bussell, K. R.	Climatic Effec	ts on			of Pai	nted	35
	Steel (T) The Evaluation	of Pr	imina	Dointe	by Co		33
	sion Tests (T)				b by Ct	110-	36
Robinson, A. G. Water-Dilutable Sto			••		••		149
Rochow, E. (ed.). Inorganic Synthese							384
Routley, A. F. An Improved Method	for the Assess	nent of	Primi	ng Pai	nts on S	Steel	50
Under Marine Condi	tions (T)						25
Rudram, A. T. S. see Bullett, T. R. (T)			•••		•••	78
Pigments in Corro	sion Protection	(D)				• •	53
Russell, R. H. see Beikmanis, J., et a	<i>l.</i> (T)	•••	• •				150
	S						
Scott, R. P. W. (ed.). Gas Chromato	granhy 1960 (2)					20
Sell, G. (ed.). A Glossary of Petroleu			••	•••	••	• •	48
Smith, H. Ships' Paints (D)		•••			••	••	25
Smith, N.D. P., Orchard, S. E., and Rhi	nd-Tutt A. J.	The Pl	nysics c	f Brus	shmark	s(T)	61
Smith, W. J. Water-Dilutable Stovin	g Finishes (D)						14
Staddon, A. W. E. see McKee, R. S.	(T)						49
Steiner, H. (ed.). Introduction to Pet						•••	38
Stevens, W. H. The Physical Behavio	our of Paint File	ns (D)		14			59:
Stoyle, F. W. The Behaviour of Prin	ting Ink on Rol	lers (D)				610
Strain, C. G. The Design of Unsatura	ated Polyester F	tesins f	or Surf	ace C	oatings	(D)	5
Strain, R. N. C. The Solar Reflectivit	y of Paints (T)	• •	••				68
Sunderland, E. The Correlation of	Service Behav	iour of					
Physical Characterist							68
Some Aspects of Phys	ical Assessment	of Emu	Ision F	olyme	er Films	;(D)	72
— The Wetting, Adhes	ion and Penetr	ation of	of Surf	face C	Coating	s on	
Wood (D)			• • •	* *	•••		78
	Т						
Talen, H. W. Some Aspects of Physic	cal Assessment of	of Emu	lsion P	olvme	r Films	(D)	72
——— The Physics of Brushm							63
Targett, P. Pigments in Corrosion Pr		•••					53
Teach, W. C., and Kiessling, G. C.							
Tengstrand, G. The Coating and the S	Polystyrene (R)		• •				
			••	• •		••	6
Tobolsky, A. V. Properties and Struc	ubstrate (D)	 	••	••		••	6 81
Tobolsky, A. V. Properties and Struc Turner, J. H. W. Some Aspects of	bubstrate (D) cture of Polyme	rs (R)		•••	•••	**	6 81
Turner, J. H. W. Some Aspects of	bubstrate (D) cture of Polyme Physical Asses	rs (R)	 of Er	 nulsio	 on Poly	 /mer	6 81 15
Turner, J. H. W. Some Aspects of Films (D)	Substrate (D) cture of Polyme Physical Asses	rs (R) ssment	 of Er	 nulsio	 n Poly	 /mer	6 810 15 72
Turner, J. H. W. Some Aspects of	Substrate (D) cture of Polyme Physical Asses	rs (R) ssment	 of Er	 nulsio	 on Poly	 /mer	6 810 15 72
Turner, J. H. W. Some Aspects of Films (D)	Substrate (D) cture of Polyme Physical Asses	rs (R) ssment	 of Er	 nulsio	 n Poly	 /mer	6 810 15 72
Turner, J. H. W. Some Aspects of Films (D) Twyford, H. Ships' Paints (D)	bubstrate (D) cture of Polyme Physical Asses	rs (R) ssment	 of Er	 nulsio	 n Poly	 /mer	6: 810 15 72 25
 Turner, J. H. W. Some Aspects of Films (D) Twyford, H. Ships' Paints (D) Valentine, L. The Physical Behaviour 	bubstrate (D) cture of Polyme Physical Asses	rs (R) ssment	 of Er 	 nulsio 	 on Poly 	 /mer	6 810 15 72 25
 Turner, J. H. W. Some Aspects of Films (D) Twyford, H. Ships' Paints (D) Valentine, L. The Physical Behaviour Vickery, R. C. Analytical Chemistry 	bubstrate (D) ture of Polyme Physical Asses V r of Paint Films of the Rare Ea	rs (R) ssment	 of Er 	 nulsio	 on Poly 	 /mer	6. 810 15 72 25 59 28
 Turner, J. H. W. Some Aspects of Films (D) Twyford, H. Ships' Paints (D) Valentine, L. The Physical Behaviour 	bubstrate (D) ture of Polyme Physical Asses V r of Paint Films of the Rare Ea	rs (R) ssment	 of Er 	 nulsio 	 on Poly 	 /mer 	6 811 15 72 25 59 28
 Turner, J. H. W. Some Aspects of Films (D) Twyford, H. Ships' Paints (D) Valentine, L. The Physical Behaviour Vickery, R. C. Analytical Chemistry 	bubstrate (D) cture of Polyme Physical Asses 	rs (R) ssment	 of Er 	 nulsio 	 on Poly 	 /mer 	6 811 15 72 25 59 28
 Turner, J. H. W. Some Aspects of Films (D) Twyford, H. Ships' Paints (D) Valentine, L. The Physical Behaviour Vickery, R. C. Analytical Chemistry Vollmann, H. F. Detection of Nitroc 	bubstrate (D) cture of Polyme Physical Asses V r of Paint Films of the Rare Ea cellulose (T) W	rs (R) ssment is (D) rths (R	 of Er 	 nulsio 	 on Poly 	 /mer 	6 811 15 72 25 59 28
 Turner, J. H. W. Some Aspects of Films (D) Twyford, H. Ships' Paints (D) Valentine, L. The Physical Behaviour Vickery, R. C. Analytical Chemistry Vollmann, H. F. Detection of Nitroc Waddington, F. B. The Irradiation F 	bubstrate (D) cture of Polyme Physical Asses 	rs (R) ssment (D) rths (R 	 of Er 	 nulsio 	 on Poly 	 /mer 	6 81 15 72 25 59 28 30
 Turner, J. H. W. Some Aspects of Films (D) Twyford, H. Ships' Paints (D) Valentine, L. The Physical Behaviour Vickery, R. C. Analytical Chemistry Vollmann, H. F. Detection of Nitroc Waddington, F. B. The Irradiation F Waele, A. de Determination of Thixe 	bubstrate (D) ture of Polyme Physical Asses 	rs (R) ssment (D) rths (R 	 of Er 	 nulsio 	 	 /mer 	6: 810 15 72 25 59 280 303
 Turner, J. H. W. Some Aspects of Films (D) Twyford, H. Ships' Paints (D) Valentine, L. The Physical Behaviour Vickery, R. C. Analytical Chemistry Vollmann, H. F. Detection of Nitroc Waddington, F. B. The Irradiation F 	bubstrate (D) ture of Polyme Physical Asses 	rs (R) ssment (D) rths (R 	 of Er 	 nulsio 	 Poly 	 /mer 	6: 810 15 ⁻ 72 ⁻ 25 ⁻ 59 280 303 17 ⁴ 37 ⁻ 31

Wheeler, R. N. Water-Dilutable Stoving Finishes (D)				• •
The Wetting, Adhesion and Penetration	of Su	rface (Coating	s on
Wood (D)				
Wheeler, T. S. see Partington, J. R. (R)				
Whiteley, P. The Correlation of Service Behaviour of Pair	nt with	Observ	ed Phy	sical
Characteristics in Air Drying Paint for Struc				
——— Pigments in Corrosion Protection (D)				
White, T. Pigments in Corrosion Protection (D)				
Wicker, R. J. see Jenkins, V. F., et al. (T)	•••	•••	• •	
Wilkinson, E. M. The Design of Unsaturated Polyester Re	eine fo	r Surfa	Co Coa	tings
	• •	•••		•
Williams, B. R. see Carter, C. F. (R)	• •	• •	• •	• •
Williams, G. H. Homolytic Aromatic Substitution (R)				
Williams, G. T. see Cox, P. D	- · · .			
Williams, H. Some Aspects of the Physical Assessment of				
Woinarksi, C. H. Z. Thoughts on the Education and Adu	. 12		· · · ·	. • •
Woinarksi, C. H. Z. Thoughts on the Education and Adu	ninistra	ation c	of Tech	nical
Staff (T)				• •
Wormwell, F. Pigments in Corrosion Protection (D)				• •

SUBJECTS

	Α							PAGE
Acrylic Emulsion Paints (S)								167
								166
Adhesism (NI)								734
Adhesion and Penetration, The Wetting, o	of Sur	face	Coatir	igs on V	Wood	(T).		756
Administration of Technical Staff, Thoug	hts on	the	e Educa	ation an	d (T)	(-)		264
Absorption of Paint Media into Porous S								737
Alcoholysis and Acidolysis of Triglyceride								497
Acidolysis, The Alcoholysis and, of Trigly	vceride	e Oi	ls (T)					497
Alkyd Resins based on Soya Bean Oil,						Pheno		
experienced with (T)								851
Aluminium/Magnesium Alloys, Lacquer (93
Amino Resins (R)								63
Analysis of Paint Materials (S)								392
Analytical Chemistry of the Rare Earths								280
Annual General Meeting, Proceedings of								479
Annual Reports—1960								442
Anti-Fouling Compositions, The Limitation	ons of							869
Anti-Fouling Paints (S)								822
Applications for Ordinary Membership (N	N)							88, 494
A DT 10/0 M I (ND								905
								826
Association Conference 10(1 (NI)	•							287, 344
Association of Printing Technologists, Th							.,,	233
Atomic Energy Installations, Surface Coa								316
Automobile Painting Processes (S)								285
	В							
Bauchemie für Schule und Baupraxis (R)				• •				312
Behaviour of Printing Ink on Rollers, The	e (T)					· ·		596
Behaviour of Titanium Dioxide Pigments	in Hi	igh	Speed	Impelle	r Disp	ersion	Mills,	
								188
Behaviour of Titanium Dioxide Pigments	in Hi	igh	Speed	Impelle	r Disp	ersion	Mills,	
The (C)			• • • •	•				547
Binding of the Association's Journal (N)		• •	••					88, 494

									F	AGE
Book Reviews:										()
Amino Resins Analytical Chemistry of the Rare Earths	••			••	••	••	•••		**	63 280
Bauchemie für Schule und Baupraxis		•	•••	••			•••	••		312 61
Chemical Processing Nomographs Chemistry of Natural Products		•••				••	••	• •		882
Chemistry of the Terpenes, The	•••					••••	• •	• •	• •	311
Chemists Introduction to Statistics, Theo Cost Engineering in the Process Industrie		error an			Experin	nent, A			•••	818 156
Der Cold-Check Test										210
Dictionary of Named Effects and Laws in Electronic Packaging with Resins	Chen						 3.2	•••	• •	311 882
Exploration of the Solar System, The	••	• •	•••	•••		•••	•••	••	••	816
Gas Chromatography, 1960	• •	• •	••		••	••		•	× ×	209
Gas Chromatography Abstracts, 1960 Glossary of Petroleum Terms, A	•••			•••		•••		• •		818 484
History of Seed Crushing in Great Britain	1		**	••	• •	• •			1.1	66
Homolytic Aromatic Substitution Inorganic Syntheses Volume VI	•	•••	•••	••				••	••	159 384
Introduction to Petroleum Chemicals						••	•••		3 8 • •	386
Isopropyl Alcohol	•••	• •	••		• •			••	••	883
Laboratory Management and Techniques Life and Work of William Higgins, The			•••	••	• •	• •				542 485
Mechanism of Heterogeneous Catalysis								**		384
Metallizing of Plastics Michael Faraday: A List of His Lectures	and P	ubliche	d Wr	tinge	201201	1.01.01	•	••	••	61 545
Moon Base-Technical and Psychological			u wn	ings 	••				••	816
Organic Coating Technology		• •						• •	• •	385
Perfumes, Cosmetics and Soaps Physical Chemistry of Surfaces	••	• •	**	••	••	••	•••	5 B	**	160 65
Polypropylene						••	••			884
Polystyrene	••	• •	••		••	••	•.•	••		65 313
Printing Ink and Colour Printing Ink Manual						••				382
Processing of Thermoplastic Materials		•	••	••					• •	543
Properties and Structure of Polymers Runnerless Moulding		• •		•••	••		5404	• •	••	157 157
Science in Industry—Policy for Progress	••	• •	••	••		••	••	• •		279
Silicones	• •	••	••	**		••	••	• •		161
Small Particle Statistics	•••	••					••	••		158 159
Surface Activity										817
Systematic Qualitative Analysis: An Intro	ductio	n	••	••	•••			€0.e	••	543 816
Telomerisation and New Synthetic Mater Use of Organic Reagents in Inorganic An	alysis.	The	•••	**	••	••	••	• •	8 E 	210
Brains Trust (S)				• •						398
Bristol Section: Annual Dinner (N)										172
Bristol Section: Annual General Meet	ing (S) .					••			490
Bristol Section: Formation of the Iris	h Bra	nch (]	N)						9	827
Bristol Section: Irish Branch (N)				• •					560,	639
Bristol Section: Programme Changes				• •						639
Bristol Section: Works Visit, 1961 (S)			•	••	• •	•••	••	•	0	490
British Aerosols Manufacturers' Asso				••			• •	1.5	0	233
British Colour Makers' Association, 7	ne (1	N) .	•	••		••	••	170		89
British Standards (N)	• •		•	• •	• •	••	• •		, 734,	
Brushmarks, The Physics of (T)		٠	•	8 (M)		• •	• •	• •		618
		С								
Can the Climatologist Help You? (S)										391
Centenary of Plastics, The (N)					• •					905
Change of Address				• •					88,	494
Chemical Processing Nomographs (R))			• •	•••					61
Chemistry of Natural Products (R)						• •				882
Chemistry of the Terpenes, The (R)			•	•				. 21		311
Chemists's Introduction to Statistics, T	heory	ofEr	ror a	nd De	sign of	Exper	iment.	A(R))	818
C.I.T.A. (N)			•	• •	•••	• •	••			734
City Livery Companies (N)			•	• •	••	• •				399
Clark, F. W., Memorial Prize, The (N			· (T)	• •	•••	••	• •	•		905
Climatic Effects on the Rusting of Pai	inted	Steel	(1)	••	•••	••	••			351
Coating and the Substrate, The (T)			•		••	••	• •	• •		787
Coconut Oil/Isophthalic Alkyds for S				201	••		• •••		901	150
Considerations on the Formation of I	mins,	, some	e (3)	• •	3 8 8	••	••		891,	020

Comment:											
Anderson, James, & Co. (Colours) Ltd.	••	• •			• •	• •			••	68
Butterworths Publications Ltd.	••			• •	••	••		•••		••	68
Fatoils Ltd Imperial Chemical Industries Ltd.	* *	141.47	• •	• •	••		••	••	••	••	69
International Council of Scientific			••	••		••	••	••	••	••	162 69
Laporte Titanium Ltd						•••					819
Stockdale Engineering Ltd	**										68
Vinyl Plastisol		••	••	• •	••	• •	••	••	••	••	282
Committees of Council	••									••	2,409
Considerations on the Formatio											891
					• •					• •	
Correlation of Service Behavior	ir of P	aint w	ith (The	ved P	hysical	Char	acteri	stice	s in	
Correlation of Service Behaviou									stics	s in	640
Air Drying Paint for Struc	tures, 7	The (T).		ved P		• •	acteri	stics	s in 	649
Air Drying Paint for Struc Corrosion of Iron and Steel, Th	tures, 7 ne (S)).								487
Air Drying Paint for Struc Corrosion of Iron and Steel, Th Corrosion Prevention, Pigments	tures, 7 ne (S) s in (S)	The (T) .				• •	•••		••	487 316
Air Drying Paint for Struc Corrosion of Iron and Steel, Th Corrosion Prevention, Pigments Corrosion Protection, Pigments	tures, 7 ie (S) in (S) in (T)	The (T) .	•	 	 				•• ••	487 316 515
Air Drying Paint for Struc Corrosion of Iron and Steel, Th Corrosion Prevention, Pigments Corrosion Protection, Pigments Corrosion Tests, The Evaluatio	tures, 7 the (S) s in (S) in (T) n of Pr	The (T	") .	•	 	· · · · ·	 	 		••• ••• ••	487 316 515 367
Air Drying Paint for Struc Corrosion of Iron and Steel, Th Corrosion Prevention, Pigments Corrosion Protection, Pigments Corrosion Tests, The Evaluatio Cost Engineering in the Process	tures, 7 the (S) s in (S) in (T) n of Pr	The (T	") .	•	 	 	 			••• ••• ••	487 316 515
Air Drying Paint for Struc Corrosion of Iron and Steel, Th Corrosion Prevention, Pigments Corrosion Protection, Pigments Corrosion Tests, The Evaluatio	tures, 7 the (S) s in (S) in (T) n of Pr	The (T	")		 (T)	 	··· ··· ··	··· ··· ··		 	487 316 515 367

PAGE

D

Der Cold-Check Test (R)	• •	210
Design of Unsaturated Polyester Resins for Surface Coatings, The (T)		42
Detection of Nitrocellulose (T)		308
Determination of Thixotropic Coefficient (T)		377
Development of Organic Pigments for Printing Inks, The (S)		389
Dictionary of Named Effects and Laws in Chemistry, Physics and Mathemat	tics, A	(R) 311
Dispersion of <i>Tioxide</i> Pigments in Modern Day Machinery, The (S)		551
		234
Drying Phenomena, An Investigation into, experienced with Alkyd Resins	based	on
Soya Bean Oil (T)		851

			E								
Editorials:											
A.I.P.T					••	••		••		••	819
11,000 Miles Apart	This	• •	• •	••	••		••	••	••	• •	162
Flowers that Bloom in the Spring, From Corrosion to Conflict	The		•••		••		••	•••	• •	••	213 162
	 					••	**	••	••	••	
Education and Administration o			Staff,	The	oughts	on (T)	• •	••		••	264
Efflorescence Phenomena on Pla		F)	• •		• •		• •	• •			32
Electronic Brushability Meter, A	n (S)				••	••	• •				637
Electronic Packaging with Resins	s (R)				·• •					1 4 •	882
Electron Spin Resonance (N)											644
Emulsions, Acrylic (S)					••	••					166
Emulsion Paints, Acrylic (S)											167
Emulsion Polymer Films, Some	Asnec	te of t	he P		1 Acc	· ·	t of (T) ···		•••	713
						essmen		1)		••	367
Evaluation of Priming Paints by			rests,	Inc	s(1)	•••	•••	•••		••	
Exploration of the Solar System,	Ine	(K)									816

Evaluation of Finning Faints by Corrosion Tests, The (1)					507
Exploration of the Solar System, The (R)					816
Exterior Durability of Paints based on Lithopone and Zinc	: Sulphi	de, Th	e (T)	••	835

F

FATIPEC Sixth Conference (N) .			•••				90, 293,	645
Films, Some Considerations on the Forma	ation	of (S)	••			••	891,	896
Finishing in the Motor Industry (S) .			•••	• •	••		••	395
Floating, Flooding and, in Paints (S) .	•		•••		•	•••		894
Flooding and Floating in Paints (S) .	•	••		• •	•	• •	server from the server server	894
Fourteenth Technical Exhibition, 1962 (N				• •	• •	• •	489, 560,	731
Future Trend of Paint Technique, The (S)		••	••	••	• •			390

			G							PAGE
Gas Chromatography, 1960 (R)										209
Gas Chromatography Abstracts,	1960 (R)								818
Glossary of Petroleum Terms, A	(R)									484
Gypsum Plaster as a Substrate fo	r Pain	t Fil	ms (T	()						11
-51										
			Н							
Hiding Power Determination as a	an Aid	to I	Paint	For	mulatio	on, A	Metho	d of (S)	216
Hiding Power Determination and	its Ap	oplic	ation	as a	an Aid	to Pa	int For	mulatio	on, A	
Method of (T)										295
History of Seed Crushing in Grea	at Brita	ain (R) .							66
History of the Paint Industry (S)										319
Homolytic Aromatic Substitution		••		•	• •	• •	• •		• •	159
Honorary Members of the Assoc	ation	••				• •	••	• •	• •	10, 416
Hull Section Meetings (S)	••	•••			••		••		•••	316, 389
Hull Section: Men's Night (N)	••		•	•	••	• •	• •	• •	• •	561
Hull Section: Social Events (N)	••	•••			••	• •	• •	• •		345
			I							
Impeller Dispersion Mills, The	Rehavi	our	of Ti	itan	ium Di	iovide	Pigme	ente in	High	
Canad (T)		oui	01 1	itan		IOXIGE	Figine	ins in	righ	188
Improved Method for the Asses	 sment	of	Primi	ing	Paints	on S	teel U	nder M	arine	100
Conditions, An (T)	Sincin	01				on o		ider ivi		258
Industrial Finishes, Pigments for	High-	Grad	de (S)	•		•••	•••	•••		74
Industrial Finishes, Water-Solubl	e Syste	ems	for (S	5)						216
Information Received:										
ABCO Chemical Co.										70
Alan Cobham Engineering Ltd.					•••		•••			000
Albright & Wilson Group										. 730
Amalgamated Oxides (1939) Ltd. Armour Hess Chemicals Ltd			•••				• •		•• •	202 720
Arnold, M. H. M							• •			. 635
Associated Chemical Companies (Sa			• •				• •			282
Association of British Chemical Ma Berk, F. W., & Co. Ltd					•••					634, 820
Brandram-Henderson			••							. 163
British Celanese Ltd British Oxygen Chemicals Ltd.										214
Burts & Harvey Ltd										. 730
Coates Brothers Ltd		••	•••	• •	•••	• •	••		·· ·	175
Cowan Bros. (Stratford) Ltd.										71
Crosfield, Joseph, & Sons Ltd.		•••		• •	• •					634
Cyclo Chemicals Ltd		•••								720
Dupont	••			••						. 163
Du Pont Co. (U.K.) Ltd Elektrochemische Werk Munchen						•••	•••			1/7
Golden Valley Colours Ltd.										. 214
Gunn, D. A., (Eng.) Ltd I.C.I. Ltd		• •	••	• •	• •	••	• •	••	71 634	634
Johns-Manville Co. Ltd										202
Johnson, Matthey & Co. Ltd. Keystone Paint & Varnish Co. Ltd.		••		••		••				720
Kunstharsfabriek Synthese N.V.									·· ·	
Laporte Chemicals Ltd										. 821, 888
Marchon Products Ltd. Metal & Pipeline Endurance Ltd.	•••		•••			••		••		. 70
Midland Silicones Ltd										. 163, 634
Monsanto Chemicals Ltd Noss Mayo Dispersion Products Lt	 d	••	• •	•••	•••	••	• •		•• •	030
Novadel Ltd	u. 									720
Parkinson Cowan Measurement										. 820
Price's (Bromborough) Ltd Pye, W. G., & Co. Ltd										
Randall, J. H., & Son Ltd										. 214
Resinous Chemicals Ltd Rose, William, Ltd		••	••	••	••	••	••	••		. 315, 634
Salford Electrical Industries Ltd.		::				•••			·· · ·· ·	214
Shandon Scientific Co. Ltd	••	••					••			. 729
Shell Chemical Co. Ltd Shell International Chemical Co. Ltd.	td.	::		::		::	::			. 214. 315

									PAGE
Short & Mason Itd		1							70
Short & Mason Ltd Smith, H. A., Ltd		••				• •			635
Stainless Steel Fabricators' Association of					• •				215
Thomas Industrial Automation Ltd. T. P. Chemical Engineering Co. Ltd.		••	**	•••	• •	••			163, 888 729
Translation & Technical Services									164
Turner, E. R. & F., Ltd Ultrasonics Ltd.	••		••	•••	••	• •		• ••	729 729
Ultrasonics Ltd Union Carbide International Company	•••	 		•••		••			315
Union Carbide Corporation		• •		• •	••	• •	8.8 · · ·		634
Vinyl Products Ltd								• •	71, 214 821
Information Retrieval Systems (S)									551
Inorganic Syntheses Volume VI (R)	•••				•••				384
Instrumentation in the Assessment of	Printi	ng Inl	c Ch			(S)	•••		388
Interplas '61 (N)						(~)			293
Introduction to Petroleum Chemicals	(R)				••				386
Investigation into Drying Phenomena				h Alky	d Res	sins ba	sed on		
Bean Oil, An (T)									851
Iron and Steel, The Corrosion of (S)	••	•							487
Irradiation Resistance of Varnishes, T	he (T)	• •			• •	• •		• •	179
Isopropyl Alcohol (R)	••			•••	•••	••	• •	•••	883
		J							
Joint I.R.I./P.I. Examinations (N)									830
Joint I.K.I./F.I. Examinations (IV)	• •	••		• •	1.1	• •	••	• 35	0.50
		K							
Dr. H. W. Keenan in Norway (N)									86
Kevnote Address (N)									734
		L							
Laboratory Management and Techniq				• •				• •	542
Lacquer Coating of Alumium/Magnes	sium A	lloys	(T)			• •			93
Lacquer Films, Methods of Testing C	ertain	Mech	anica	al Pro	pertie	s of (T)	· · ·	111
Leaching Rate Determinations of Anti							ations of	of (T)	869
Lead, Sealers for Insulating Paint Filr	ns with	n a H	igh (Conter	nt of,	(1)	•••	• •	509
Lectures on Solvents (N)	165	1025	Th	(D)		• ×	• •		89
Life and Work of William Higgins (17	/65 10	1825)	, Ine	$e(\mathbf{R})$		• •	19 X	19. E	485
Limitations of Leaching Rate Determine Lithopone and Zinc Sulphide, The External	ination	is, In		f Doi.	ata har		(T)	• •	869
London Section: Analysis of Paint Ma				л ган 	its bas	sed on t	1)	• •	835 289
London Section: Annual General Mee				• •		••	••	••	399
London Section: Formation of the So				N	•••	•••	••	• •	81
London Section: Junior Group Works					••		••	••	290
London Section: Junior Group Works									491
							••		81
London Section: Meeting of Junior M	lember	's (N)			• •	• •			173
	-	Μ							
Manchester Section: Annual Cricket M					• •	• •	• •		561
Manchester Section: Annual General				• 1•1	• •	•••	• •	• •	400
Manchester Section: Cricket Match v.				N)	••	••	••	• •	639
Manchester Section: Dinne Dance (N	•)			• •			• •	• •	83
Manchester Section: Joint Meeting	ті			· · ·	• •	• ••	• •	• •	393
Manchester Section: Visit to Granada					· · ·	• •	• •	• •	828
Manufacture of Titanium Dioxide Pig Marine Conditions, An Improved Me	thed	u Pali	11S, I	ne (S) nt of	Deimin	a Dain		488
					ant of	rimin	g rain		258
Steel under (T)	ne M	athod	of	 Festin	 a	ain (T	· · ·	• •	258
Mechanism of Heterogeneous Catalysi	is The	(P)	5 01	i esun	g certa	am, (1		• •	384
Metallic and Organic Finishes, The Pe	orform	ance	of (S	· ·	••	••	• •	• •	895
Metallizing of Plastics (R)			л (5)	,	••	••	••	•••	61
Metal Preparation and Painting (S)	•••	••		••	••	• •	••	••	897
inclar reparation and randing (5)	••	• •		8 B1				• •	0,1

							PAGE
Metals for Painting, Preparation of (S)	• •	••			••		165
Metals for Painting, Preparation of Some (Τ)	•••	• •				269
Method of Hiding Power Determination a	and its	Applicati	ion as	an /	Aid to	Paint	
Formulation, A (T)	• •			1 3.	· ·		295
Method of Hiding Power Determination as						5)	216
Methods of Testing Certain Mechanical Pro)	• •	111
Michael Faraday: A List of His Lectures an	na Pub	lished wr	tings ((K)	•••	• ••	545
Microbiology in Paints (S) Midlands Section: Annual General Meeting	· (ND)	• •	••	• •	••	•••	318 491
Midlands Section: A.G.M. and Dinner (N)				••	• •	19 B	290
Midlands Section: Celebration Dinner (N)		•••		• •	•••	••	345, 561
Midland Section: Ladies' Evening (N)				••	•••	••	230
Midlands Section: Presentation to S. Marks							900
Midlands Section: Works Visit to Barry (N			• •				563
Modern Chemistry of Organic Pigments, Tl	he (S)			• •			894
Moon Base—Technical and Psychological A	Aspects	(R)		• •		• •	816
Motor Industry, Finishing in the (S)	• •	• •		• •	• •		395
	N						
Newcastle Section: Annual Dinner Dance (• 14:	• •	••	••	402
Newcastle Section: Joint Meeting (S)		•••••		• •	••	••	896
New Coatings and New Coatings Raw Mat				• •	••		234, 348
Newer Methods of Testing Paint Films (S) Newer Methods of Testing Paint Films (S)	• •	• •		10.00	••	••	165 286
News of Members (N)	••	• •	••		88 176	232	293, 404
	••	••	••	4	94 570	643	733, 830
New South Wales Section: Annual Dinner	(N)						564
New South Wales Section: Committee (N)							491
Nitrocellulose, Detection of (T)					• •		308
1996 Direkt Szketerintrikaketeket LAK Uric A Decidi Le R - e 1995 - Maretik A R - A 19							
	0						
Obituaries (N):	0						
Obituaries (N): Bond E. J.	0						. 233
			• •			• •	233 903
Bond, E. J	 	•••••	•••			•	. 903 . 902
Bond, E. J			• •	••		• •	903 902 830 87
Bond, E. J. Briscoe, H. V. A. Reynolds, H. Tweedie, E. L. Waters, R. B. Woinarski, C. H.			•••			• •	903 902 830 87
Bond, E. J. Briscoe, H. V. A. Reynolds, H. Tweedie, E. L. Waters, R. B. Woinarski, C. H. O.C.C.A. Technical Exhibition (C)			• • • • • •			• •	903 902 830 87 176 67, 212
Bond, E. J		······	•••	· · · · · · · · · · · · · · · · · · ·		•	903 902 830 87 176 67, 212 385
Bond, E. J	 mance		•••	:: :: ::		· · · · · · · · · · · · · · · · · · ·	903 902 830 87 176 67, 212 385 895
Bond, E. J. Briscoe, H. V. A. Reynolds, H. Tweedie, E. L. Waters, R. B. Woinarski, C. H. O.C.C.A. Technical Exhibition (C) Organic Coating Technology (R) Organic Finishes, Metallic and, The Perform Organic Pigments for Printing Inks, The Do	mance	of (S)	···	··· ··· ···	··· · · · · · · · · · · · · · · · · ·	· · · · · · · · · · · · · · · · · · ·	903 902 830 87 176 67, 212 385 895 389
Bond, E. J Briscoe, H. V. A	mance	of (S)	···	··· ··· ···	··· · · · · · · · · · · · · · · · · ·	· · · · · · · · · · · · · · · · · · ·	903 902 830 87 67, 212 385 895 389 219
Bond, E. J Briscoe, H. V. A	mance evelopr blems	of (S) encounter	 	 he U	se of (S	· · · · · · · · · · · · · · · · · · ·	903 902 830 87 176 67, 212 385 895 389
Bond, E. J Briscoe, H. V. A	mance evelopr blems	of (S) Encounter	 ed in t	 he U	··· · · · · · · · · · · · · · · · · ·	· · · · · · · · · · · · · · · · · · ·	903 902 830 67, 212 385 895 389 219 552
Bond, E. J Briscoe, H. V. A	mance evelopr blems	of (S) encounter	 	 he U	se of (S	· · · · · · · · · · · · · · · · · · ·	903 902 830 830 87 67, 212 385 895 389 219 552 894
Bond, E. J. Briscoe, H. V. A. Reynolds, H. Tweedie, E. L. Waters, R. B. O.C.C.A. Technical Exhibition (C) Organic Coating Technology (R) Organic Finishes, Metallic and, The Perform Organic Pigments for Printing Inks, The De Organic Pigments in Stoving Lacquers, Pro Organic Pigments, The Modern Trends (S) Organic Pigments, The Modern Chemistry Other Man's Technology, The	mance evelopr blems of (S)	of (S) nents of (S Encounter		 he U	 se of (S	· · · · · · · · · · · · · · · · · · ·	903 902 830 830 87 67, 212 385 895 389 219 552 894
Bond, E. J. Briscoe, H. V. A. Reynolds, H. Tweedie, E. L. Waters, R. B. O.C.C.A. Technical Exhibition (C) Organic Coating Technology (R) Organic Finishes, Metallic and, The Perform Organic Finishes, Metallic and, The Perform Organic Pigments for Printing Inks, The De Organic Pigments in Stoving Lacquers, Pro Organic Pigments, The Modern Trends (S) Organic Pigments, The Modern Chemistry Other Man's Technology, The	mance evelopr blems of (S) P of Sur	of (S) nents of (S Encounter		 he U	 se of (S	· · · · · · · · · · · · · · · · · · ·	903 902 830 830 87 67, 212 385 895 389 219 552 894
Bond, E. J. Briscoe, H. V. A. Reynolds, H. Tweedie, E. L. Waters, R. B. O.C.C.A. Technical Exhibition (C) Organic Coating Technology (R) Organic Finishes, Metallic and, The Perform Organic Pigments for Printing Inks, The De Organic Pigments in Stoving Lacquers, Pro Organic Pigments, The Modern Trends (S) Organic Pigments, The Modern Chemistry Other Man's Technology, The Paint and Printing Ink Industries, The Use Paint Films, Gypsum Plaster as a Substrate	mance evelopr blems of (S) P of Sur	of (S) nents of (S Encounter		 he U	 se of (S	· · · · · · · · · · · · · · · · · · ·	903 902 87 67, 212 885 895 389 219 552 894 889 216 11
Bond, E. J Briscoe, H. V. A	mance evelopr blems of (S) P of Sur for (T	of (S) nents of (S Encounter		 he U	 se of (S	· · · · · · · · · · · · · · · · · · ·	903 902 87 67, 212 885 895 389 219 552 894 889 216 11 72
 Bond, E. J. Briscoe, H. V. A. Reynolds, H. Tweedie, E. L. Waters, R. B. Woinarski, C. H. O.C.C.A. Technical Exhibition (C) Organic Coating Technology (R) Organic Finishes, Metallic and, The Perfort Organic Pigments for Printing Inks, The Defort Organic Pigments in Stoving Lacquers, Pro Organic Pigments, The Modern Trends (S) Organic Pigments, The Modern Chemistry Other Man's Technology, The Paint and Printing Ink Industries, The Use Paint Films, Gypsum Plaster as a Substrate Paint Films, The Physics of (S) Paint Films, Newer Methods of Testing (S) 	mance evelopr blems of (S) P of Sur f for (T	of (S) nents of (S Encounter		 he U	 se of (S		903 902 87 67, 212 385 895 389 219 552 894 889 216 11 72 165
 Bond, E. J. Briscoe, H. V. A. Reynolds, H. Tweedie, E. L. Waters, R. B. Woinarski, C. H. O.C.C.A. Technical Exhibition (C) Organic Coating Technology (R) Organic Finishes, Metallic and, The Perfort Organic Pigments for Printing Inks, The Defort Organic Pigments in Stoving Lacquers, Pro Organic Pigments, The Modern Trends (S) Organic Pigments, The Modern Chemistry Other Man's Technology, The Paint and Printing Ink Industries, The Use Paint Films, Gypsum Plaster as a Substrate Paint Films, Newer Methods of Testing (S) Paint Films, The Physical Behaviour of (T) 	mance evelopr blems of (S) •• • • • • • • • • • • • • • • • • •	of (S) nents of (S) Encounter	s in th	 	se of (S		903 902 87 67, 212 885 895 389 219 552 894 889 216 11 72
 Bond, E. J. Briscoe, H. V. A. Reynolds, H. Tweedie, E. L. Waters, R. B. Woinarski, C. H. O.C.C.A. Technical Exhibition (C) Organic Coating Technology (R) Organic Finishes, Metallic and, The Perform Organic Pigments for Printing Inks, The Deform Organic Pigments in Stoving Lacquers, Pro Organic Pigments, The Modern Trends (S) Organic Pigments, The Modern Chemistry Other Man's Technology, The Paint and Printing Ink Industries, The Use Paint Films, Gypsum Plaster as a Substrate Paint Films, The Physics of (S) Paint Films, The Physical Behaviour of (T) Paint Formulation, A Method of Hiding Point 	mance evelopr blems of (S) •• • • • • • • • • • • • • • • • • •	of (S) nents of (S) Encounter	s in th	 	se of (S	· · · · · · · · · · · · · · · · · · ·	903 902 810 67, 212 385 389 219 552 894 889 216 11 72 165 575
 Bond, E. J. Briscoe, H. V. A. Reynolds, H. Tweedie, E. L. Waters, R. B. Woinarski, C. H. O.C.C.A. Technical Exhibition (C) Organic Coating Technology (R) Organic Finishes, Metallic and, The Perform Organic Pigments for Printing Inks, The Decorganic Pigments in Stoving Lacquers, Pro Organic Pigments. The Modern Trends (S) Organic Pigments, The Modern Chemistry Other Man's Technology, The Paint and Printing Ink Industries, The Use Paint Films, Gypsum Plaster as a Substrate Paint Films, The Physics of (S) Paint Films, The Physical Behaviour of (T) Paint Films, The Physical Behaviour of (T) Paint Formulation, A Method of Hiding Poan Aid to (T) 	mance evelopr blems of (S) P of Sur for (T wer De	of (S) nents of (S) Encounter		 	se of (S	· · · · · · · · · · · · · · · · · · ·	903 902 810 67, 212 385 389 219 552 894 889 216 11 72 165 575 295
 Bond, E. J. Briscoe, H. V. A. Reynolds, H. Tweedie, E. L. Waters, R. B. Woinarski, C. H. O.C.C.A. Technical Exhibition (C) Organic Coating Technology (R) Organic Finishes, Metallic and, The Perform Organic Pigments for Printing Inks, The Deform Organic Pigments in Stoving Lacquers, Pro Organic Pigments. The Modern Chemistry of Organic Pigments, The Modern Chemistry of Other Man's Technology, The Paint and Printing Ink Industries, The Use Paint Films, Gypsum Plaster as a Substrate Paint Films, The Physics of (S) Paint Films, The Physical Behaviour of (T) Paint Formulation, A Method of Hiding Poral Actional Ac	mance evelopr blems of (S) P of Sur for (T wer De	of (S) nents of (S) Encounter		 	se of (S	· · · · · · · · · · · · · · · · · · ·	903 902 87 67, 212 385 895 389 219 552 894 889 219 552 894 889 216 11 72 165 575 295 216
 Bond, E. J. Briscoe, H. V. A. Reynolds, H. Tweedie, E. L. Waters, R. B. Woinarski, C. H. O.C.C.A. Technical Exhibition (C) Organic Coating Technology (R) Organic Finishes, Metallic and, The Perform Organic Pigments for Printing Inks, The Deform Organic Pigments in Stoving Lacquers, Pro Organic Pigments. The Modern Trends (S) Organic Pigments, The Modern Chemistry of ther Man's Technology, The Paint and Printing Ink Industries, The Use Paint Films, Gypsum Plaster as a Substrate Paint Films, The Physics of (S) Paint Films, The Physics of (S) Paint Films, The Physical Behaviour of (T) Paint Firms, The Physical Behaviour of (T) Paint Formulation, A Method of Hiding Poant Industry, History of the (S) 	mance evelopr blems of (S) P of Sur for (T wer De	of (S) nents of (S) Encounter		 	se of (S	· · · · · · · · · · · · · · · · · · ·	903 902 87 67, 212 889 67, 212 889 219 552 894 889 219 552 894 889 216 11 72 165 575 295 216 319
 Bond, E. J. Briscoe, H. V. A. Reynolds, H. Tweedie, E. L. Waters, R. B. Woinarski, C. H. O.C.C.A. Technical Exhibition (C) Organic Coating Technology (R) Organic Finishes, Metallic and, The Perfort Organic Pigments for Printing Inks, The De Organic Pigments in Stoving Lacquers, Pro Organic Pigments, The Modern Chemistry Other Man's Technology, The Paint and Printing Ink Industries, The Use Paint Films, Gypsum Plaster as a Substrate Paint Films, The Physics of (S) Paint Films, The Physics of Testing (S) Paint Films, The Physical Behaviour of (T) Paint Formulation, A Method of Hiding Poan Aid to (T) Paint Industry, History of the (S) Paint Industry in Other Countries (S) 	mance evelopr blems of (S) P of Sur for (T wer De ower De	of (S) nents of (S Encounter face Agent face Agent face Agent terminatic		 	se of (S	· · · · · · · · · · · · · · · · · · ·	903 902 902 87 67, 212 385 895 389 219 552 894 889 216 11 72 165 575 295 216 319 74
 Bond, E. J. Briscoe, H. V. A. Reynolds, H. Tweedie, E. L. Waters, R. B. Woinarski, C. H. O.C.C.A. Technical Exhibition (C) Organic Coating Technology (R) Organic Finishes, Metallic and, The Perform Organic Pigments for Printing Inks, The De Organic Pigments in Stoving Lacquers, Pro Organic Pigments, The Modern Chemistry Other Man's Technology, The Paint and Printing Ink Industries, The Use Paint Films, Gypsum Plaster as a Substrate Paint Films, The Physics of (S) Paint Films, The Physical Behaviour of (T) Paint Formulation, A Method of Hiding Poant Aid to (T) Paint Industry, History of the (S) Paint Industry in Other Countries (S) Paint Media into Porous Substrates, Absorption 	mance evelopr blems of (S) P of Sur for (T wer De ower De ower De	of (S) nents of (S Encounter face Agent face Agent terminatic eterminatic f (T)		 	se of (S	· · · · · · · · · · · · · · · · · · ·	903 902 87 67, 212 385 895 389 219 552 894 889 216 11 72 165 575 295 216 319 74 737
 Bond, E. J. Briscoe, H. V. A. Reynolds, H. Tweedie, E. L. Waters, R. B. Woinarski, C. H. O.C.C.A. Technical Exhibition (C) Organic Coating Technology (R) Organic Finishes, Metallic and, The Perform Organic Pigments for Printing Inks, The Deform Organic Pigments in Stoving Lacquers, Pro Organic Pigments, The Modern Trends (S) Organic Pigments, The Modern Chemistry of the Man's Technology, The Paint and Printing Ink Industries, The Use Paint Films, Gypsum Plaster as a Substrate Paint Films, The Physics of (S) Paint Films, The Physics of (S) Paint Films, The Physical Behaviour of (T) Paint Formulation, A Method of Hiding Poant Industry, History of the (S) Paint Industry, History of the (S) Paint Media into Porous Substrates, Absorption 	mance evelopr blems of (S) P of Sur for (T wer De ower De	of (S) nents of (S Encounter face Agent face Agent terminatic eterminatic f (T)		 	se of (S	· · · · · · · · · · · · · · · · · · ·	903 902 902 87 67, 212 385 895 389 219 552 894 889 216 11 72 165 575 295 216 319 74

PAGE

									PAGE
Paint Manufacturers' Joint Exec	utive Co	uncil (N)					• •	348
Paint Materials, Analysis of (S)									392
Paints, Ships' (S)									168
Paints, Solar Reflectivity of (T)									689
Past Presidents									1,407
Patents and the Paint Chemist (S	a								889
Penetration, The Wetting, Adhes	ion and	of Su		Coatin	as on V		т)		756
					-		1)	••	895
Performance of Metallic and Org					•••	••	•••	••	160
Perfumes, Cosmetics and Soaps		••	••	••	••	••	•••	••	
Perkin Centenary Trust, The (N)		••	••	••	••	••	••	• •	89
Perkin Centenary Trust Awards	(N)	••	••	••	••	••	••	••	831
Petrochemical Industry, The (S)	••		••	••	••	••	••	••	637
Physical Behaviour of Paint Film		(T)	••	••		••	••	••	575
Physical Chemistry of Surfaces (R)								65
Physics of Brushmarks, The (T)						••		••	618
Physics of Paint Films, The (S)									72
Physics of Polymers (N)									90
Physics of Semi-conductors, The									644
Pigments for High-Grade Indust									74
Pigments in Corrosion Preventio		•••	,						316
Pigments in Corrosion Protection									515
Plaster, Efflorescence Phenomena									32
Plastic-Coated Steel Strip (S)			••	••	••	••	••	••	76
	••	••	• •	••	••	••	••	••	644
D1 (1 1 (C))	••	••	••	••	••	••	••	••	283
Plastisols (S)	···	i. D	. •	c		(T)	••	••	
Polyester Resins for Surface Coa					turated	(1)	••	••	42
Polyester Surface Coatings, Rece				(S)	••	••	••	••	73
Polymers that are Different (S)	••	••	••	••	••		••	••	217
Polypropylene (R)									884
Polysulphide Coatings and Seala	nts (S)								73
Polystyrene (R)									65
Polyurethanes (S)									170
Polyurethane Varnishes and Pain	nts, Rece	ent Ex	perien	ce with	1 (S)				822
Porous Substrates, Absorption of									737
Preparation of Metals for Painti									165
Preparation of Some Metals for									269
Priming Paints by Corrosion Tes				of (T)					367
Priming Paints on Steel Under					nnrove				201
					nprove	u Ivicu			258
Printing Ink Characteristics, Inst		tion is	 tha	 A ccoccr	nant of	(2)	••	••	388
Printing Ink Manual (R)	umenta	ation n		M220221			••	••	382
			••	••	••	••	••	••	404
Printing Ink Manual (N)		••	••	••	••	••	••	••	
Printing Inks and Colour (R)	·.•	· · ·		••	••	••	••	••	313
Printing Ink on Rollers, the Beh	aviour c	$\mathbf{n}(1)$	•••	• •	••	••	••	••	596
Printing Inks, Development of C)rganic l	Pigmer	its (S)	• • •	••	••	••	• •	389
Printing Processes (S)		••	••		• •		• •	••	636
Problems Encountered in Air-D									285
Problems Encountered in the Us			Pigme	nts in S	Stoving	Lacqu	ers (S)		219
Proceedings of the Annual Gene	ral Mee	ting							479
Processing of Thermoplastic Ma	terials (]	R)							544
Programme Liaison Committee									571
Properties and Structure of Poly	mers (R)							157
Protective Pigment, Zinc Dust as			3						417
			1	•••					
		(2						
Queensland Section: Annual Dir	nner Da								401
Queensiana Section. Annual Di	iner Da		,	••	••	••		••	401
		F	8						
Recent Developments in Polyest	or Surfa			(S)					73
	u sund					••	••		13
Recent Experience with Polyure	hana V.	rnicha	and	Dainta	(6)	• •			822

PAGE

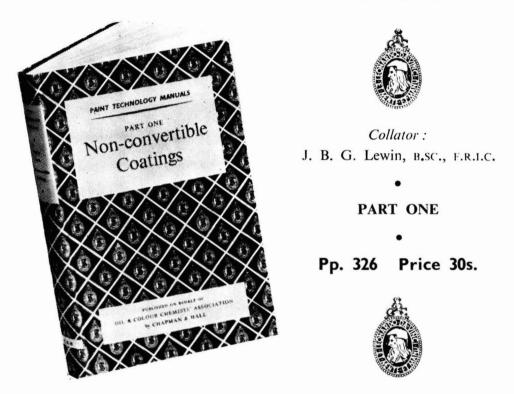
									PAGE
Register of Members (N)		•••							345, 403 829, 901
Research Fund (N) Resin Deve opments for Surface (Restoration of Pointings (S)						+ <i>72</i> , <i>507</i>			
Resin Deve opments for Surface (Coating		••	••			••		167
Restoration of Paintings (S)	couring	50 (0)	•••			•••	•••		220
Restoration of Paintings (S) Reunion Dinner of Past President	s. Past	Hone	orary C)fficers.	Four	nder Me	mbers	and	220
Council (N)									825
Council (N) Runnerless Moulding (R)				••	•••				157
Rusting of Painted Steel, Climatic	Effect	s on th	ne (T)			•••	• •		351
		-	-						
		2	ò						
Science in Industry—Policy for Pr	ogress	(R)		• •		14. 8	• •	• •	279
Scientific Library Methods (S) Scottish Section: Annual General	•••	••	• •	••		••	• •	••	823
Scottish Section: Annual General	Meetin	ig (N)	• •			••	••	• •	402
Scottish Section: Annual Smoking Scottish Section Student Group: H	g Conce	ert (N)	,		•••	•••	1.0.0	• •	403
Scottish Section Student Group: I	Essay C	ompet	tition (N)	•••				291
Scottish Section: Dinner Dance (N Scottish Section: A. H. Whitaker	N)	• •	• •					• •	229
Scottish Section: A. H. Whitaker	(N)	• •				•••	(*)(*)	• •	402
Sealants, Polysulphide Coatings and	nd (S)	112-1	ċ.				• •	÷ 14	73
Sealers for Insulating Paint Films	with a	High	Conter	nt of L	ead (I)	•	•••	509
Section Officers and Committees, Section Programmes for 1961-62 S	1901-0.		••	• •	• •	••	• •		410
Section Programmes for 1961-62 S	session	(IN)		tion I.		Channe (NI		565 828
Section Programme for 1961-62 Se	ession-	-Lond	on sec	tion Ju	mor	Group (• •	1/0
Ships' Paints (S)	• •	• •		••		• •	••	••	237, 906
Ships' Paints (T)		• •	••	•••	•••	••	•••		
Sixth ISE Congress 1962 (N)	• •	••	••	••	• •	••	••	••	494
SLE Third Congress (N)	• •	••		•••	· ·	••	•••	• • •	405, 734
Small Particle Statistics (R)	• •	••	••			201200	• •		
Solar Reflectivity of Paints (T)		• •		• •	• •	••	••		689
Silicones (R) Sixth I.S.F. Congress 1962 (N) SLF Third Congress (N) Small Particle Statistics (R) Solar Reflectivity of Paints (T) Solvents in the Surface Coatings I	 ndustry	(S)	••	···					892
Some Aspects of the Physical Ass	essmen	t of E	mulsio	n Polvr	ner F	ilms (T)	1. (1. 1 .)	• •	713
South Australian Section: The Fu	ture of	the A	rts (N)						291
									159
Soviet Technology Digest (R) Soya Bean Alkyds, Problems Enco	ountere	d in A	ir-Dry	ing (S)		•••	• • •		285
Soya Bean Oil, An Investigation	into D	Drving	Pheno	mena	experi	enced w	ith A		
Resins based on (T) Steel before Painting, The Surface Steel, Painted, Climatic Effects on	••					• •			851
Steel before Painting, The Surface	Prepa	ration	of (S)			1.1			396
Steel, Painted, Climatic Effects on	the Ri	usting	of (T)				• •		351
Steel Strip, Plastic Coated (S)									76
Steel Under Marine Conditions, A	n Impr	oved N	Aethoc	l for th	e Asse	essment	of Prir	ning	
Paints on (T)	••	• •	•••	•••		•••	• •	• •	258
Stoving Enamels, Coconut Oil/Iso	phthali	ic Alky	ds for	· (T)	• •			••	150
Stoving Finishes, Water-Dilutable	(T)			• •		••		• •	119
Stoving I misnes, viater-I minaon						•••		• •	72, 395
Stoving Lacquers, Problems Enco	unterec	i in the	e Use o	or orga	unc i	ignicints	$\ln(S)$;	219
Structures, The Correlation of Se	ervice E	senavi	our of	Paint	with	Observe	d Phy	sical	(40
Characteristics in Air-Drying	Paint	$(\mathbf{r}(\mathbf{I})$			• •	••	• •	• •	649
Students' Competition (N)		••	••	•••	•••	•		•••	830
Substrate, The Coating and the (T) Denti		T)	••		• •	••	÷ 8.	787
Substrate for Paint Films, Gypsun	n riasu	er as (1)	••	• •	(*)	• •	• •	11
Surface Activity (R) Surface Agents in the Paint and P	 rinting	Ink In		e The	Lleo	of (S)	•••	• •	817 216
Surface Coatings in Atomic Energy				es, The	Use	01 (3)	••	• •	316
Surface Coatings in Atomic Energy Surface Coatings, The Design of U				er Reci	ns for	(T)	•••	•••	42
Surface Coatings, The Design of C Surface Coatings, Resin Developm			orycsu	er ivesi			••		167
Surface Coatings, Water-Soluble F	lesine f	for (S)		••		••	••	•••	220
Surface Coatings, Water-Soluble I Surface Coatings on Wood, The V	Vetting	Adhe	esion a	nd Pen	etrati	on of (T)	•••	756
Surface Preparation of Steel befor	e Paint	ing T	he (S)	na ren	enan	on or (1	/	• •	396
Systematic Qualitative Analysis: A						• • •	••		543
-,					1.11			•••	-

DACE

Т		PAGE
Technical Education in the Paint Industry (C)		885
Telomerisation and New Synthetic Materials (R)		816
Testing Paint Films, Newer Methods of (S)		165, 286
Third Australian Convention (S)		547, 640
	171,	223, 321
Thixotropic Coefficient, Determination of (T)		377
Thoughts on the Education and Administration of Technical Staff (T)		264
Titanium Dioxide Pigments, The Behaviour of, in High Speed Impeller Disper Mills (T)		100
Titanium Diavida Diamontal Dainta The Manufacture of (S)	•••	188 488
Torquay Conference Diary (N)	••	553
Torquay Conference Diary (N)	•••	497
rigiveride Ons, The Alcoholysis and Acidolysis of (1)	••	477
U		
		210
Use of Organic Reagents in Inorganic Analysis, The (R)	• •	210 216
Use of Surface Agents in the Paint and Printing Ink Industries, The (S)	••	210
V		
Varnishes, The Irradiation Resistance of (T)	••	179
Visit to Stanlow (N)	••	570
W		
Wallpaper Today (S)		394
Water-Dilutable Stoving Finishes (T)		119
Water-So uble Resins for Surface Coatings (S)		220
Water-Soluble Systems for Industrial Finishes (S)		216
Water-Thinnable Stoving Finishes (S)		72, 395
Waverley Gold Medal (N)		405
West Riding Section: Dinner and Dance (N)		173
Wetting, Adhesion and Penetration of Surface Coatings on Wood, The (T)		756
Wood, The Wetting, Adhesion and Penetration of Surface Coatings on (T)		756
Z		
Zinc Dust as a Protective Pigment: Part III (T)		417
Zinc Dust in Protective Coatings (S)		283
Zinc Sulphide, The Exterior Durability of Paints based on Lithopone and (T)		835

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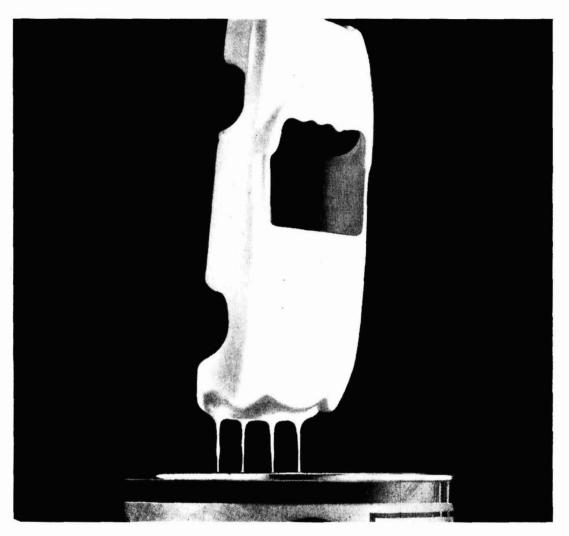
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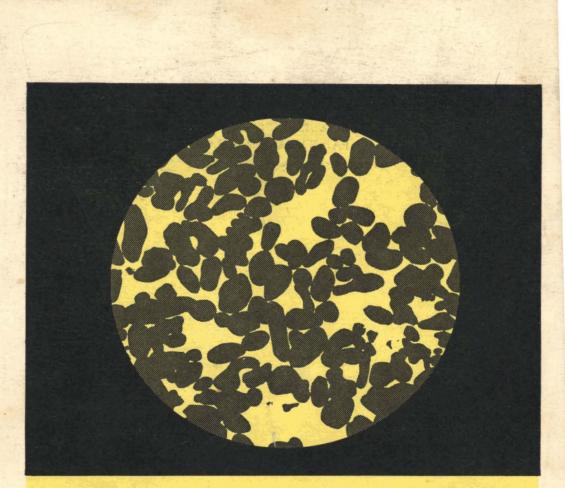
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