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Weathering of paint films. I. Chalking caused by zinc oxide in latex paints

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The painting of galvanised sheet—the performance of priming paints pigmented with 80/20 w/w zinc dust/zinc oxide mixtures

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Testing and investigation of shipbottom paints

A. M. van Londen

Further work on the polarisation of painted specimens

H. F. Clay

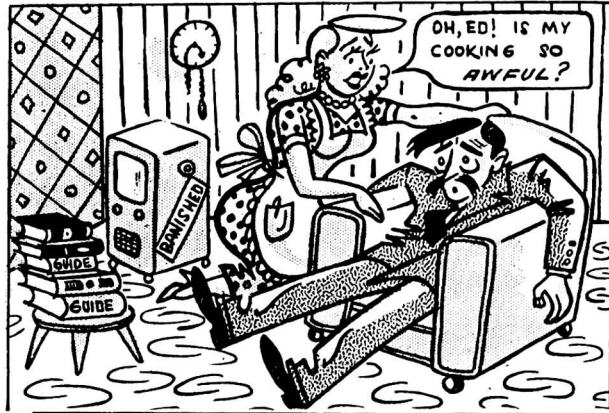
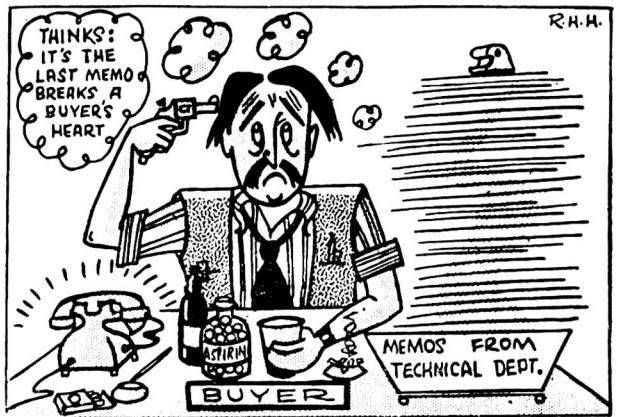
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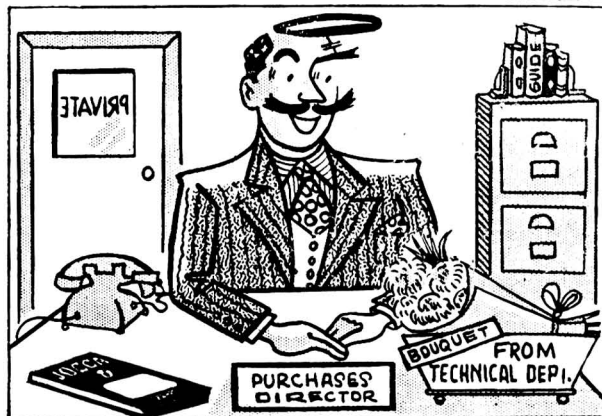
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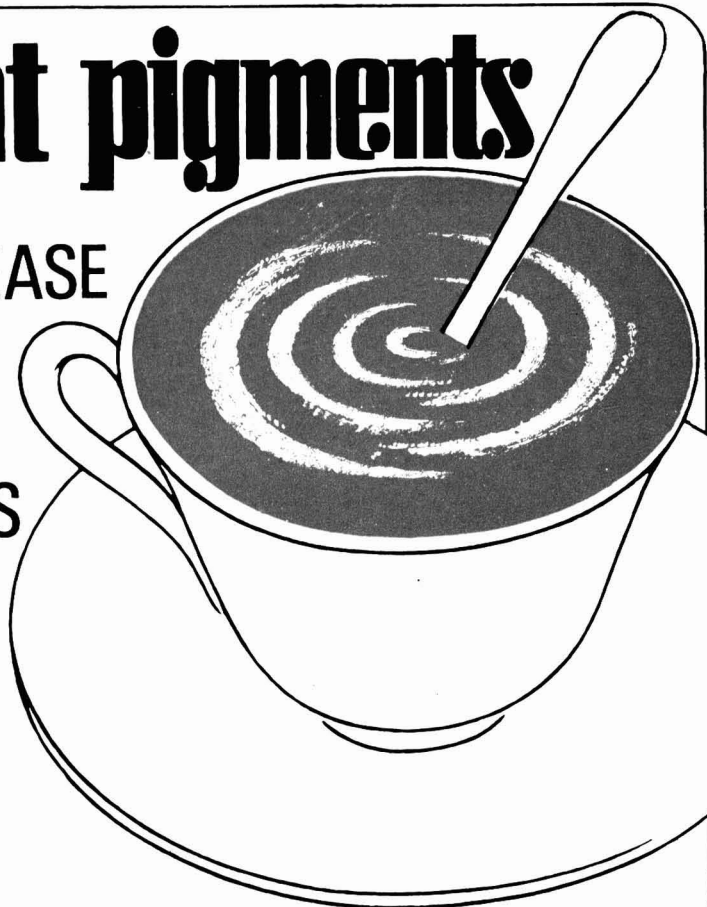
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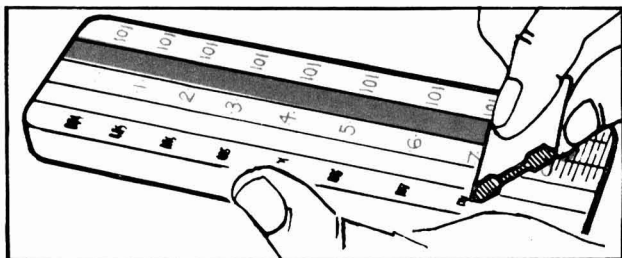
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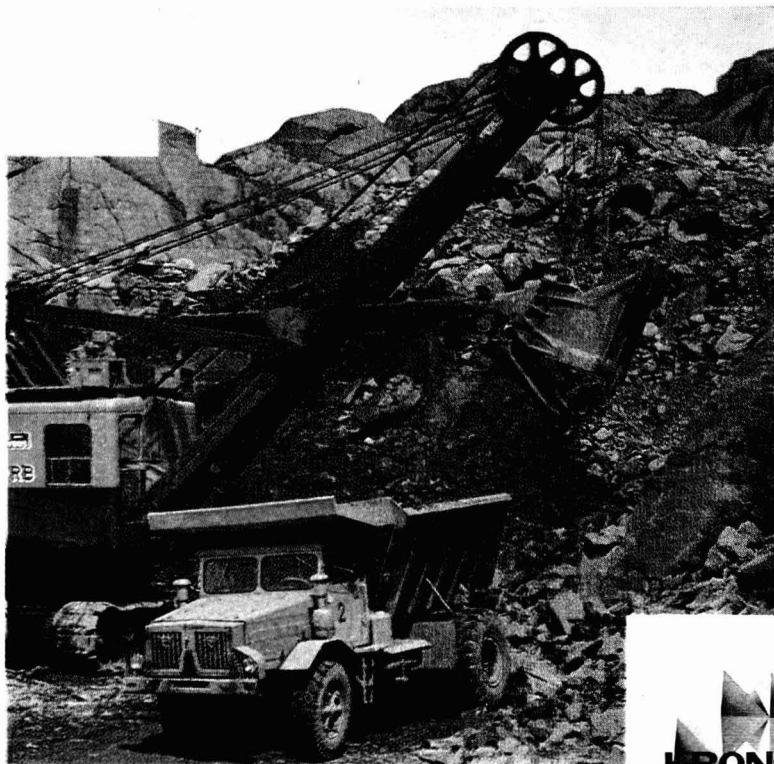
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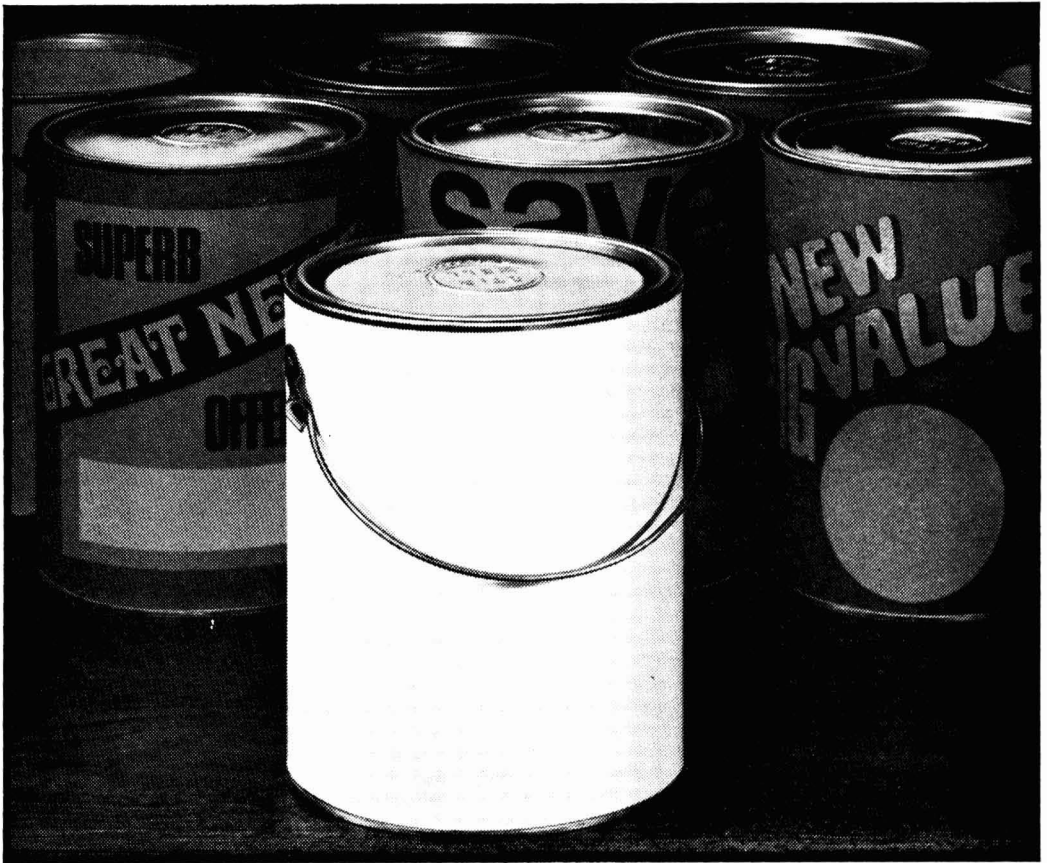
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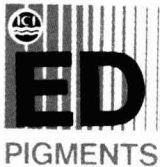
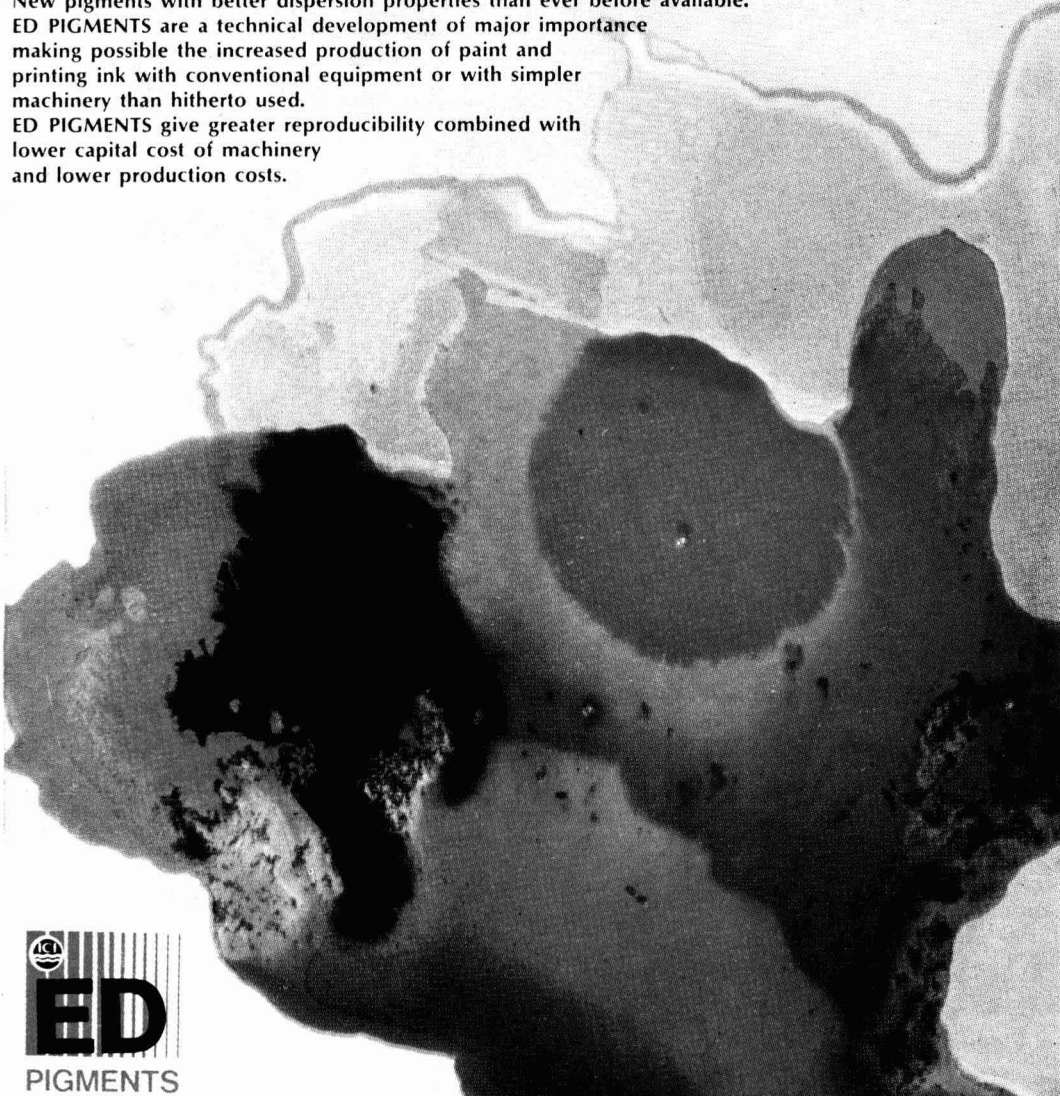
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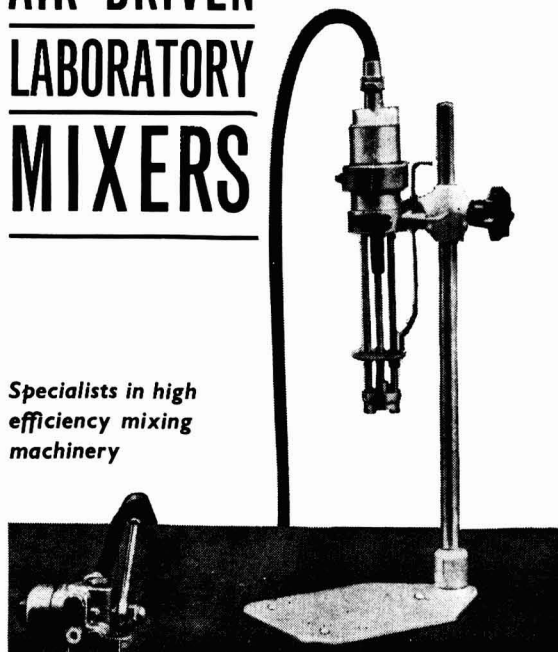
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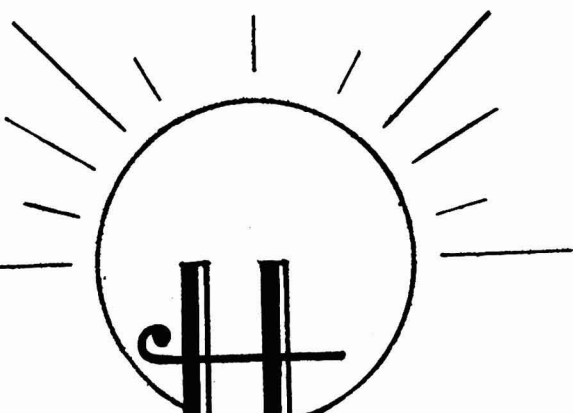


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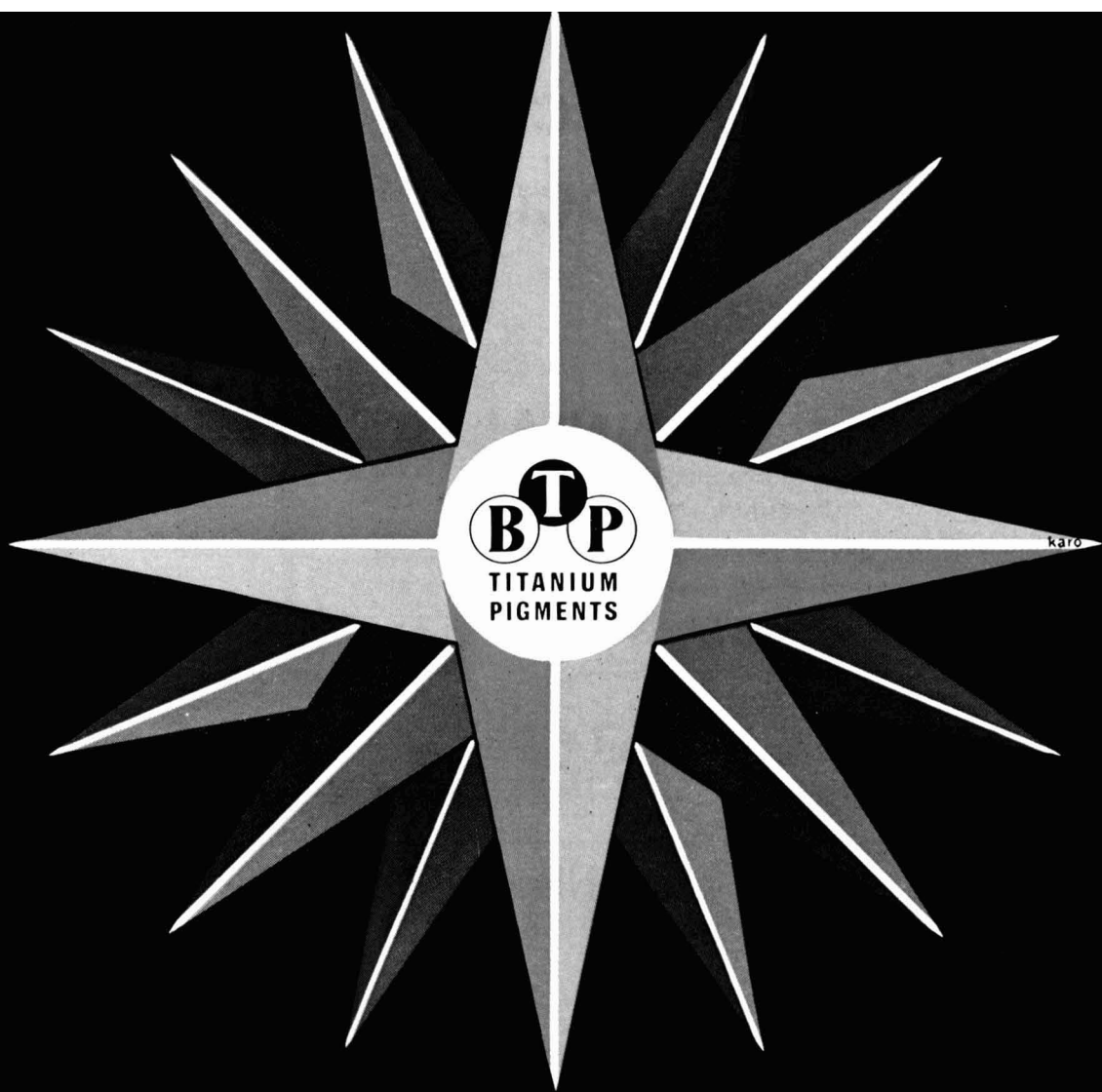
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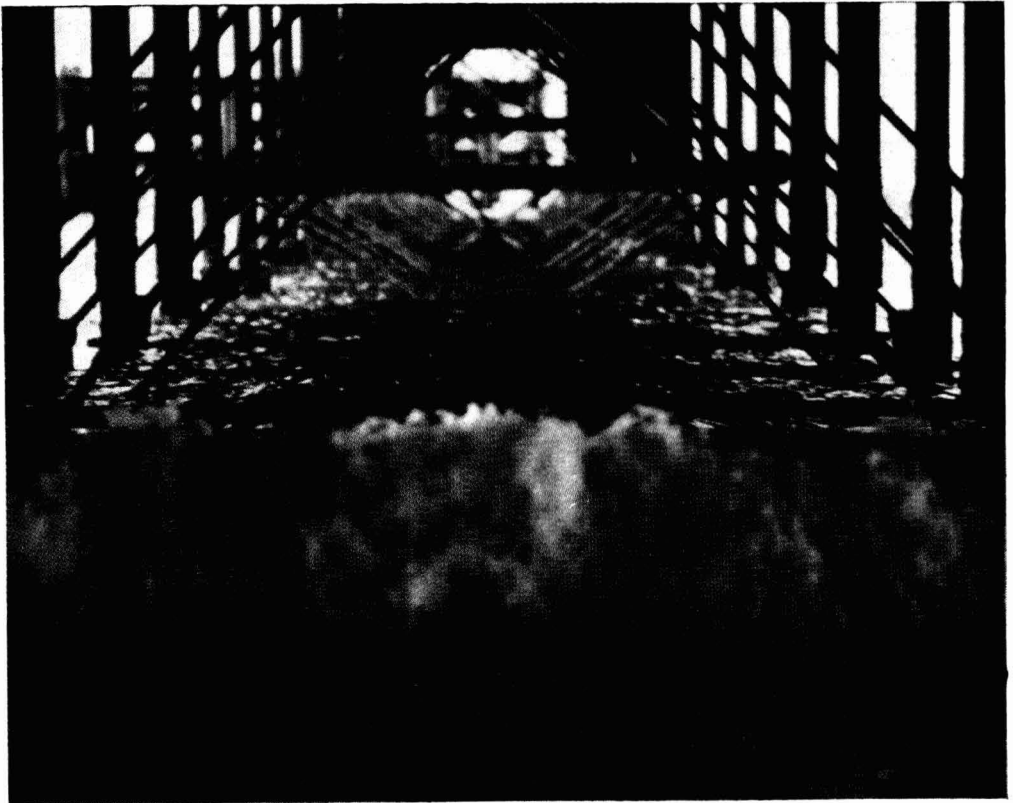
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Transactions and Communications

Weathering of paint films : I. Chalking caused by zinc oxide in latex paints

By E. Hoffmann and A. Saracz

Division of Building Research, Commonwealth Scientific and Industrial Research Organization, Melbourne, Australia

Summary

The chalking of latex paint films caused by various brands of zinc oxide and by mixtures of these with rutile titanium dioxide and stainless steel pigments was investigated in a weatherometer, and at exposure sites at Highett, Victoria, and Lae, New Guinea.

The degree to which the rate of chalking changes with changes in humidity is a characteristic of each brand of zinc oxide. As zinc oxide is progressively replaced by rutile titanium dioxide or stainless steel pigment the rate of chalking decreases approximately exponentially.

The observations made in this study have a bearing on accelerated testing and this is also discussed.

Key words

Types of coating

latex coating

Binders (resins etc.)

acrylic resin
copolymer resin
polyvinyl acetate
polyvinyl chloride
styrene resin

Prime pigments

stainless steel pigment
titanium dioxide
zinc oxide

Properties characteristics and conditions associated with dried or cured films chalking

Analysis, measuring and testing

Processes

accelerated weathering
weathering

Equipment

weatherometer

Le vieillissement de feuil de peinture

I. Farinage provoqué en peintures-émulsions par l'oxyde de zinc

Résumé

Le farinage des feuil de peintures-émulsions provoqué par de diverses marques de l'oxyde de zinc et par leurs mélanges avec de l'oxyde de titane de type rutile ou avec des pigments d'acier inoxydable était étudié dans un weatheromètre, et aux stations d'exposition à Highett, Victoria, et à Lae, Nouvelle Guinée.

La variation du taux de farinage selon la variation de l'humidité se diminue à peu près exponentiellement lors du remplacement progressif de l'oxyde de zinc par l'oxyde de titane de type rutile ou par du pigment d'acier inoxydable.

D'ailleurs, on discute l'importance, au point de vue des essais de vieillissement accélérés, des observations que l'on a fait pendant cette étude.

Bewitterung von Anstrichfilmen

I. Durch Zinkoxidgehalt in Latexfarben Verursachtes Kreiden

Zusammenfassung

In einem Weather-O-Meter, sowie in den Bewitterungsstationen Highett, Victoria und Lae, Neu Guinea wurde durch verschiedene Zinkoxidmarken und deren Mischungen mit rutilen Titanoxiden und nichtrostenden Stahlpigmenten verursachtes Kreiden untersucht. Der Grad des Kreidens jeder einzelnen Zinkoxidmarke wird durch die Veränderungen charakterisiert, welche durch Änderungen in der Feuchtigkeit hervorgerufen werden. In dem Masse, in dem Zinkoxid mehr und mehr durch rutilen Titandioxid oder nichtrostendes Stahlpigment ersetzt wird, vermindert sich der Grad des Kreidens etwa exponentiell.

Die in dieser Untersuchung gemachten Beobachtungen beeinflussen auch die künstlichen Bewitterungsversuche, welche ebenfalls besprochen werden.

Выветривание красочных пленок

I. Известкование причиняемое окисью цинка в латексоподобных красках

Резюме

Исследуется известкование в латексоподобных красочных пленках причиняемое различными сортами окиси цинка и их смесями с рутиловой двуокисью титана и пигментами нержавеющей стали, при помощи прибора для измерения выветривания и при выветривании на открытом воздухе в Хигетте в Виктории и в Лае в Новой Гинее. Степень до которой скорость известкования изменяется с изменением влажности является характеристикой каждого сорта окиси цинка. По мере того как окись цинка прогрессивно замещается рутиловой двуокисью титана или пигментом нержавеющей стали, скорость известкования понижается приблизительно экспоненциально.

Замечания высказанные в настоящей статье имеют отношение к ускоренным испытаниям и этот вопрос тоже обсуждается.

Introduction

Paint films on outside exposure fail mainly in two ways:

(i) by chalking in which the binder is totally decomposed and consequently the pigments and extenders contained in the paint film are set free and are lost.

(ii) by embrittlement due to further polymerization of the binder. The paint film in this case tends to fail by cracking, crazing, or peeling.

It is the aim of the paint industry to produce coatings which keep their protective and decorative value for many years, so an investigation into the causes of chalking and embrittlement is of importance. This paper deals with the former phenomenon.

A great deal has been published on weathering of paint films generally and on chalking in particular, but practically all these publications deal with the subject in a qualitative way.

Zinc oxide pigments have been used in this investigation for two reasons:

(i) the authors have shown that, although paints for indoor use formulated with zinc oxide show an increased mould resistance^{2, 3}, they should not be used outside because they tend to chalk heavily. An investigation into the chalking of the zinc oxide is therefore of immediate practical interest.

(ii) since the brands of zinc oxide used in the investigations cause heavy chalking, any patterns of behaviour which may exist are likely to become evident more quickly with these pigments than with those chalking more slowly.

A great deal of work has already been done in Australia^{4, 5} on the chalking caused by different brands of zinc oxide, but that work dealt with oil-type paints and treated the subject matter in a qualitative way only. A number of papers have also appeared dealing with the fluorescence and photo-chemical activity of zinc oxides^{6, 7}.

Experimental

Materials

Four brands of zinc oxide were used, here called A, B, C, and D. Their properties are given in Table 1. A widely-used brand of rutile titanium dioxide, causing very little chalking, and a stainless steel pigment, were also used. The latter

Table 1
Properties of zinc oxide used

Property	Brand of zinc oxide			
	A (Made by indirect process)	B (Made by direct process)	C	D
Surface area, m ² g ⁻¹ , by nitrogen absorption	4.6	2.1	4.6	14
Lead content % (approx.)	0.05	1	0.005	0.005
Appearance under the microscope	Largest particle about 1 micron, but most rather smaller	Most of the particles 1 to 3 micron, with many rock-like crystals 1 × 4 micron	As for A	As for A

has come on to the market lately and, since very little is known about it, it was included in this study. A pva-acrylic resin (85 per cent pva) was used as the binder throughout except for the series of formulations used to study the effect of the binder (Table 8).

Determination of chalking

Various methods have been proposed including an optical method¹, a method in which a photographic paper is pressed against the film surface with a fixed pressure and the amount of chalk sticking to the paper is judged visually⁸, one in which the amount of chalk that can be taken off the paint film is weighed⁹, and another in which the loss of weight of the paint film after taking off the chalk is determined¹⁰.

Of these methods only the last would appear to be of any value, because none of the other procedures takes into account the amount of chalk lost through the action of rain and wind.

The procedure used in this investigation was as follows: weighed stainless steel panels $2\frac{1}{2}$ in \times 3in were painted, dried to constant weight at 38°C (which took approximately 24hr) weighed again and exposed. After exposure the panels were kept at 38°C for 24hr, and then, after the chalk had been rubbed off with a soft cloth, they were reweighed.

Exposures were carried out at Lae, New Guinea, at Highett, Victoria, and in a xenon-arc weatherometer. At Lae the panels were exposed at 45° facing 31° east of north. The weatherometer was run under three conditions;

Condition P: Cyclic conditions (a) Xenon-arc on 3.8hr, black body temp. 72°C, RH 58 to 65 per cent, air temp. 55°C

(b) Xenon-arc off for 1hr, RH 95 per cent air temp. 55°C

Condition R: Steady conditions Xenon-arc on all the time, black body temp. 73°C, RH 14 per cent, air temp. 55°C

Condition S: Steady conditions as P(a)

The weight losses of the paint films were eventually expressed as loss of resin because only this gives a correct picture of the decomposition of the resin caused by the pigment, e.g. a paint film pigmented to a 40 per cent pigment volume concentration (PVC) will show a larger weight loss, for a given amount of resin decomposed, than will a paint film of 20 per cent PVC. The loss of film thickness, too, will vary with the PVC of the paint film for a given amount of resin decomposed (see Table 2).

Table 2
Calculated* decrease in film thickness
for a resin loss of 10mg cm^{-2}

PVC (%)	0 (clear film)	1	2	4	10	20	30	40	50	70
Decrease in film thickness ($\text{cm} \times 10^{-3}$)	8.7	8.7	8.9	9.0	9.6	10.9	12.4	14.5	17.4	29.0

Note: 2.5×10^{-3} cm = 0.001in.

*The decrease in film thickness has been calculated as follows: 10mg resin (density 1.15) spread over 1cm^2 has a thickness of 8.7×10^{-3} cm. From this the loss of depth, t , for a given percentage PVC, may be obtained from $8.7 \times 10^{-3} = (1 - \text{PVC}/100)t$.

The results of the experiments are summarised in Tables 3 to 13. The panels were withdrawn from the weatherometer at intervals of 133hr of irradiation,

which at condition P is equivalent to one week of running. Tables 3 and 4 show the weight loss for each period. For the zinc oxide pigments the maximum variation during the time from the second to the fifth week was 8 per cent. For the rutile titanium dioxide pigment the variations were much bigger, but one has to take into account the fact that the maximum total weight loss for any one period between the second and the twenty-first period was only 7mg. It is for this reason that in the tables giving the results the loss of resin in the weatherometer has been averaged over the different periods. In calculating the average the first period was omitted because in this time the loss was as a rule exceptionally high. The reason for this is probably that in the initial period polymers of low molecular weight were decomposed.

Table 3
Loss of resin* for weatherometer condition P
(Zinc oxide at 40 per cent PVC)

Period†	Pigment		
	Zinc oxide A	Zinc oxide B	Zinc oxide C
1	1250	550	1180
2	1090	550	1030
3	1100	500	1020
4	1160	550	1060
5	1070	550	1060

*Loss of resin in $\mu\text{g cm}^{-2}$ period⁻¹
†1 period = 133hr of irradiation

Table 4
Loss of resin* for weatherometer condition P
(Rutile titanium dioxide at 40 per cent PVC)

Period†	1	2	3	4	5	6	7
Loss	150	24	19	6.4	42	19	32
Period	8	9	10	11	12	13	14
Loss	19	1.1	1.1	0	37	32	0
Period	15	16	17	18	19	20	21
Loss	19	19	19	37	0	19	0

Note: A loss of resin of $100 \mu\text{g cm}^{-2} = 4700 \mu\text{g}$ total loss

*Loss of resin in $\mu\text{g cm}^{-2}$ period⁻¹

†1 period = 133hr of irradiation

Table 5
Loss of resin and decrease in film thickness in the weatherometer

PVC (%)	Weatherometer Condition	Pigment used											
		Zinc oxide A		Zinc oxide B		Zinc oxide C		Zinc oxide D		Rutile TiO ₂		Stainless steel	
		L*	T†	L	T	L	T	L	T	L	T	L	T
20	P	1400	1.5	540	0.60	1300	1.4	-	-	32	0.035	13	0.014
20	R	160	0.17	9.6	0.011	130	0.15	-	-	4.8	0.0051	8	0.0089
20	S	1300	1.4	62	0.070	1300	1.4	-	-	14	0.016	4	0.0043
40	P	1100	1.6	540	0.77	1000	1.4	930	1.3	16	0.025	2.1	0.003
40	R	43	0.062	6	0.0085	48	0.068	-	-	2.8	0.0040	nil	nil
40	S	830	1.19	180	0.25	800	1.1	-	-	7.7	0.0011	nil	nil

*L = loss of resin in $\mu\text{g cm}^{-2}$ period⁻¹; 1 period = 133hr of irradiation

†T = the corresponding decrease in film thickness in $\text{cm} \times 10^{-3}$; $2.5 \times 10^{-3} \text{ cm} = 0.001\text{in.}$

Discussion

The effects of humidity and radiation

The influence of humidity on chalking is by now well recognised but no quantitative data on the change in the rate of chalking with humidity have been published. Table 5 shows the behaviour in the weatherometer of a latex paint film pigmented with zinc oxides A, B, C, D, rutile titanium dioxide, and stainless steel at two different PVCs.

Comparing conditions P and R it can be seen that in all cases the rate of chalking was very much reduced at R, the low humidity condition, and that the degree of reduction depended on the PVC of the paint. At 20 per cent PVC, for instance, the rate of chalking with zinc oxide under condition R was reduced to 11.6 per cent of the rate under condition P, whereas at 40 per cent PVC the corresponding figure was 3.9 per cent.

The reduction in the rate of chalking was also found to depend on the type of pigment. Paints pigmented with zinc oxides A and C showed a very similar degree of reduction with humidity, but when the pigment was zinc oxide B there was a much bigger reduction, e.g. at 20 per cent PVC the rate of chalking with type B was reduced at the low humidity condition to 1.8 per cent of the rate at condition P, while the corresponding figures for zinc oxides A and C were 11.6 and 10.0 per cent respectively. A similar difference, though not as large, was observed at 40 per cent PVC.

The different degree to which pigments can be affected by the conditions in the weatherometer can be seen strikingly at condition S. The elimination of the dark period, during which the paint films absorbed a great deal of water, had practically no influence at 20 per cent PVC on zinc oxides A or C, but the chalking rate of B was reduced to 11.5 per cent of its value at condition P. At 40 per cent PVC the same trend was evident although the difference was smaller.

Of the four different zinc oxides investigated type B caused the least chalking; in fact at condition R paints formulated with it were nearly as chalk resistant as those formulated with rutile titanium dioxide.

The mechanism by which water accelerates chalking is unknown. It is obvious that one should try to clarify this process because knowledge of the reaction mechanism could lead to paints of a higher chalk resistance. For the present, one conclusion is drawn concerning the formulation of paints, and one possible reason for the effect of water is discussed in some detail.

The relative humidity affects the water uptake of the paint film. The work which has been carried out on absorption of water by paint films seems to deal mainly with absorption from the liquid phase, but what appears equally important for the phenomenon of chalking is the quantity of water taken up from the atmosphere at different relative humidities. Preliminary work¹¹ has shown that this depends very much on the formulation of the paint and on the relative humidity, and Fig. 1 shows some of the results. It was not possible with

the method used (gain in weight) to investigate what happens below 80 per cent relative humidity, but this could be easily done with a radioactive technique.

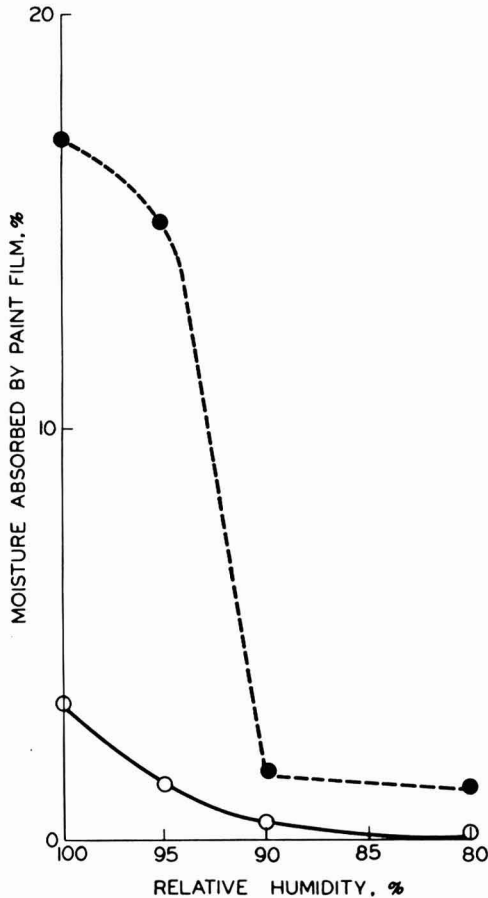


Fig. 1. Absorption of water vapour (per cent by weight) by films of flat alkyd enamels of the same PVC but with different extenders

The two flat alkyd paints referred to in Fig. 1 absorb water vapour to very different degrees. The only difference in formulation is in the type of extenders added. One would suspect that paints which show a low absorption of water vapour would be less liable to chalk than those with higher absorption.

Concerning the reaction mechanism, attention is drawn to one possibility which so far does not seem to have been pointed out. The zinc oxide particles, which will have a layer of water on the surface, will absorb energy from the infra-red part of the radiation and their temperatures will consequently rise. It is very unlikely, however, that each of the zinc oxide particles will behave in the same way. Some particles will, for various reasons, absorb more radiation

than others and their temperature will therefore be higher than those of neighbouring particles, and it is around these that decomposition of the paint medium may be expected to start. The assumption of "hot spots" is not at all unlikely if one considers that the number of particles in 1g of zinc oxide A is of the order 10^{12} . Appendix A gives an estimate of the temperature particles could reach in one second under the assumption that no heat is lost. This of course is not true, nevertheless one can see that there is a possibility that some particles could reach considerably higher temperatures than others.

Zinc oxide B, which has a much larger particle size than zinc oxides A, C, or D, causes considerably less chalking, and the rate of chalking is also much more dependent on the relative humidity than is the case with types A and C. On the foregoing assumption that the decomposition of the medium starts at particles with a somewhat higher temperature than the surrounding ones, this behaviour is understandable, because the possible increase in temperature is inversely proportional to the particle size.

That the finer fraction of a zinc oxide pigment indeed causes more chalking than the coarse one has already been shown¹², but since the author followed the chalking process by Kempf's method⁸ it was not possible to obtain a reliable quantitative estimate of the effect.

Another factor to consider is that zinc oxide B differs from zinc oxide A also by its higher lead content. By treating zinc oxide B with an amount of nitric acid which is not sufficient to dissolve the whole sample, and analysing for lead in the residue, it was possible to show that lead was probably distributed throughout the particle and not just on the surface. (The lead content in the residue in some cases was higher than that of the original sample). Such impurities could lead to less active zinc oxide crystals.

The weight losses observed in the weatherometer were compared with the losses on outside exposure at Highett and Lae. It is not to be expected that the actual values obtained in the weatherometer will be repeated on the exposure sites, yet it is generally taken for granted that the order of the rate of chalking of different pigments will be maintained, that is, if one pigment chalks in the weatherometer at double the rate of another this will also be the case at different exposure sites.

This expectation cannot be justified in view of the finding that the rate of chalking caused by a pigment depends on the humidity in a characteristic way. It is very likely that a variation in total light energy, quality of light, and prevailing ambient temperature will also have a typical influence on each pigment.

The following preliminary experiment shows (Table 6) the effect of decreasing the amount of light energy falling on a painted panel. As there was no provision in the weatherometer to change the total energy the experiment was carried out at Lae. Specimens were placed on both the upper and lower sides of the specimen holder at 45° to the horizontal so that some specimens faced upwards towards the north-east and others faced downwards towards the south-west. In the former case the specimens were exposed to direct solar radiation, sky solar radiation

Table 6

Loss of resin from the upper and lower surfaces of the exposure† panels*

PVC (%)	Pigment used							
	Zinc oxide A		Zinc oxide B		Zinc oxide C		Anatase TiO ₂	
	Upper	Lower	Upper	Lower	Upper	Lower	Upper	Lower
20	2300	450	990	10	2100	310	2600	510
40	2500	480	880	30	—	—	2500	560

*Loss of resin in $\mu\text{g cm}^{-2}$

†Exposure was at Lae, New Guinea, from July to December 1967, the panels sloping at 45° to the horizontal, and facing 31° east of north.

and some ground-reflected solar radiation. In the latter case ground-reflected solar radiation was the main component of the solar radiation received and the average on a clear day was some 29 per cent of that received on the upperside specimen. Rates of chalking with zinc oxide C and anatase titanium dioxide, relative to the appropriate rate of chalking with zinc oxide A, were very nearly the same in the southerly as in the northerly position, but zinc oxide B behaved quite differently. In the southerly position at 20 per cent PVC, paints formulated with zinc oxide B chalked at the rate of only 2.2 per cent of the rate for paints formulated with zinc oxide A. In the northerly position the corresponding figure was 43 per cent. A similar relation existed at 40 per cent PVC. Table 7 bears out what has been said about the relative rate of chalking at the various exposure sites. Zinc oxides A and C behaved in a very similar way in the weatherometer (Table 3) and it can be seen that they behaved in about the same way at the two exposure sites. On the other hand zinc oxide B, which always caused less chalking under artificial weathering, caused approximately the same amount of chalking at 40 per cent PVC in seasons 2 and 3 at Hightett. At Lae B always caused less chalking.

Probably all that can be concluded from the experiments in the weatherometer is that pigments which cause little chalking compared with the zinc oxides (rutile titanium dioxide, stainless steel) will also cause little chalking on other exposure sites. There is, however, no exact quantitative relationship; e.g. under conditions P, R and S in the weatherometer, paints formulated at 20 per cent PVC with rutile titanium dioxide chalk at only 2.3, 3 and 1.1 per cent of the rate of paints which contain zinc oxide A (Table 5), but at Hightett the corresponding figures in the second and third seasons are 13 and 6 per cent and at Lae in the first and second seasons the figures are 6 per cent (Table 7). Such a correlation is probably good enough for most cases, but if one wanted to make

Table 7
Loss of resin and decrease in film thickness for outdoor exposure

PVC (%)	Season	Exposure	Pigment used									
			Zinc oxide A		Zinc oxide B		Zinc oxide C		Rutile TiO ₂		Stainless steel	
			L*	T†	L	T	L	T	L	T	L	T
20	1	Hihgett July '66—Jan. '67 6 months ..	1000	1.1	340	0.36	980	1.0	140	0.15	130	0.14
	2	Jan. '67—July '67 total 12 months	460	0.49	120	0.13	530	0.57	61	0.065	24	0.026
	3	July '67—Jan. '68 total 18 months	850	0.90	120	0.13	900	0.96	51	0.055	40	0.043
40	1	July '66—Jan. '67 6 months ..	620	0.84	340	0.49	590	0.63	110	0.18	130	0.19
	2	Jan. '67—July '67 total 12 months	130	0.19	130	0.19	130	0.19	42	0.06	nil	nil
	3	July '67—Jan. '68 total 18 months	270	0.39	270	0.39	270	0.39	81	0.12	nil	nil
20	1	Lae Sept. '66—Mar. '67 Sample 1 after 6 months ..	2300	2.5	1000	1.1	2100	2.2	140	0.15	130	0.14
	1+2	Sept. '66—Mar. '67 Sample 2 after 12 months ..	3000	3.2	1800	1.9	920	3.4	180	0.19	150	0.16
40	1	Sept. '66—Mar. '67 Sample 1 after 6 months ..	2500	3.6	880	1.3	2100	3.0	120	0.17	150	1.22
	1+2	Sept. '66—Sept. '67 Sample 2 after 12 months ..	3200	4.6	1200	1.7	3700	5.2	180	0.26	140	0.20

Note (a) At Hihgett the same panel was observed. It was removed and after determination of the loss of weight it was exposed again.

(b) At Lae the panels were left for 6 months and 12 months before they were removed and sent back to Hihgett for weighing.

*L = loss of resin in $\mu\text{g cm}^{-2}$ †T = the corresponding decrease in film thickness in $\text{cm} \times 10^{-3}$

a comparison between rutile titanium dioxide and stainless steel pigment one could be misled. In the weatherometer, stainless steel with one exception showed up as better, but there was scarcely any difference between the two pigments at Lae. This could be due to the high humidity at this site, very often over 65 per cent. A run at 90 per cent relative humidity, for instance, in the weatherometer might show that under these conditions there is not much difference between the two pigments.

Table 8

Influence of resins on the chalking caused by zinc oxide A at 40 per cent PVC

Resins used: (I) pva-acrylic copolymer, (II) pva with dibutyl phthalate as plasticiser, (III) pva with tritolyi phosphate as plasticiser, (IV) vinyl-fumarate copolymer, (V) acrylate copolymer, (VI) vinyl chloride-acrylate copolymer, (VII) tetrapolymer of styrene, 2-ethyl hexyl acrylate, methyl-methacrylate and acrylonitrile

Exposure	Resin used													
	I		II		III		IV		V		VI		VII	
	L*	T†	L	T	L	T	L	T	L	T	L	T	L	T
Weatherometer (condition P) ..	1000	1.4	1200	1.7	48	0.69	830	1.2	770	0.11	160	0.23	270	0.39
Highbett Feb. '67—Aug. '67 6 months ..	400	0.57	660	0.94	320	0.46	450	0.65	380	0.54	—	—	—	—
Aug. '67—Feb. '68 total 12 months	900	1.3	240	0.34	120	0.17	800	1.1	1200	1.7	—	—	—	—
May '67—Nov. '67 6 months ..	670	0.96	—	—	—	—	—	—	—	—	420	0.60	400	0.57
Lae Mar. '67—Sept. '67 Sample 1 after 6 months ..	1400	2.0	1400	2.0	1300	1.9	770	1.1	940	1.4	—	—	—	—
Sample 2 after 6 months ..	1200	1.7	—	—	1200	1.7	740	1.1	1050	1.5	—	—	—	—

*L = loss of resin in $\mu\text{g cm}^{-2}$ period⁻¹; 1 period = 133hr of irradiation or 6 months of outdoor exposure

†T = the corresponding decrease in film thickness in $\text{cm} \times 10^{-3}$

Influence of the binder

The influence of the binder on the rate of chalking is shown in Table 8. The one outstanding reduction in chalking was obtained with a pva binder containing tritolyl phosphate as a plasticiser. The rate of chalking of this formulation in the weatherometer was only 4 per cent of that of a paint with the same binder but containing dibutyl phthalate. At Highett, however, the reduction amounted to only about 50 per cent in the first and second seasons, and at Lae there was scarcely any difference. The reason for the bad correlation is probably as follows. Tritolyl phosphate, as the authors have been able to show, decomposes on exposure in the weatherometer or outside, and the decomposition products formed are very likely to be adsorbed on the surface of the zinc oxide, rendering it less prone to chalking. The products formed on natural weathering are probably somewhat different from those obtained under artificial weathering and hence the protective effect of tritolyl phosphate is different in the various situations.

The paints formulated with the tetrapolymer (VII) and vinylchloride acrylate copolymer (VI) also showed an increased chalk resistance under accelerated testing compared with the pva-acrylic copolymer (I), but the difference under natural exposure conditions at Highett was very much smaller.

Effect of PVC

Table 9 shows the chalking in the weatherometer caused by zinc oxide A at different pigment volume concentrations. The rate of chalking depends on

Table 9
Chalking caused by zinc oxide A at different PVCs

PVC (%)	Zinc oxide content by weight (%)	Exposure								
		Weatherometer condition P		Highett				Lae		
		L*	T†	Feb. '67—Aug. '67		Aug. '67—Feb. '68		Mar. '67—Dec. '67		
				L	T	L	T	L	T	
0 (Clear resin)	0	180	0.16							
1	4.6	490	0.43							
2	9	640	0.57							
4	16.7	960	0.86							
10	34.6	1200	1.2	880	0.85	610	0.59	4000	3.6	
20	52	1100	1.2	720	0.77	720	0.77	2700	2.9	
30	67	990	1.2	480	0.59	720	0.89	2700	3.3	
40	76	910	1.3	530	0.76	830	1.2	3200	4.6	
50	83	860	1.5	340	0.59	190	0.33	3000	5.2	
70	92	450	1.3	160	0.46	80	0.24	240	0.69	

Note (a) At Highett the same panel was observed. It was removed and after determination of the loss of weight it was exposed again.

(b) At 70 per cent PVC the paint film at Highett cracked but adhered firmly to the substrate, while at Lae the paint film did not crack.

*L = loss of resin in $\mu\text{g cm}^{-2}$ †T = the corresponding decrease in film thickness in $\text{cm} \times 10^{-2}$

various factors of which the most important are probably the depth to which light and oxygen can penetrate into the paint film, and its water content.

Although zinc oxide does absorb ultraviolet light and therefore could be expected to protect the paint films, it clearly does not do so; on the contrary it accelerates the decomposition. This has been observed in Australia and reported^{4, 6} probably for the first time in 1950. Nevertheless it appears that some workers still maintain that zinc oxide decreases the rate of decomposition of paint binders^{1, 13}. The rate of decomposition of the resin shows a maximum at 10 per cent PVC and then begins to decline slowly, the rate being considerably reduced between 50 and 70 per cent PVC.

Table 9 shows very clearly that zinc oxide A increased the rate of decomposition of the binder. At 1 per cent PVC (4.6 per cent by weight) the rate was 270 per cent greater than the rate when the binder was free of zinc oxide. This is completely contrary to the claim put forward by some authors^{1, 3} that zinc oxide protects the binder. There may of course be some type of zinc oxide which does do that but it cannot be maintained generally that this is so.

The loss of resin from the paint film was at a minimum at 70 per cent PVC, a big drop occurring between 50 and 70 per cent. This was also observed on outside exposure, but at Lae the decrease in the loss of resin was much bigger (the rate at 70 per cent PVC was 8 per cent of that at 50 per cent PVC) than at Highett or in the weatherometer where the rate at 70 per cent PVC was about 50 per cent of the rate at 50 per cent PVC. There was also a difference, at 70 per cent PVC, in the type of failure in the paint film between accelerated testing, outdoor exposure at Lae, and outdoor exposure at Highett. In the first two cases the films failed only by chalking; at Highett the paint film cracked but adhered firmly to the substrate.

Mixed pigments

Tables 10 and 11 show the effect of adding rutile titanium dioxide and stainless steel pigments to paints containing zinc oxide at 10 and 40 per cent PVC exposed in the weatherometer. In all cases a decrease in the rate of chalking was observed but the degree of reduction needs some discussion. It appears to be generally assumed that it is only, or mainly, the ultraviolet part of the sunlight in the region of 290-400nm which affects paint films. It could therefore reasonably be argued on the foregoing theory that if, for instance, half of the volume of zinc oxide were replaced by rutile titanium dioxide the latter would filter out half of the harmful radiation and the chalking would be correspondingly reduced since the chalking caused by titanium dioxide is minimal. It is evident from Tables 10 and 11 that this is far from being true; for example, at 10 per cent PVC (Table 10) the rate of chalking was reduced from a loss of resin of $1300 \mu\text{g cm}^{-2}$ to a loss of $98 \mu\text{g cm}^{-2}$ instead of $650 \mu\text{g cm}^{-2}$ as would be expected on the above view.

Actually the decrease fits an exponential curve better than a linear one, as can be seen from the figures given in Tables 10 and 11 (for calculation of K in these Tables see Appendix B). The authors think that a possible explanation of these observations is that light of greater wave length ($>400\text{nm}$) can also have

Table 10
Chalking under weatherometer condition P by mixtures of zinc oxide A and rutile titanium dioxide
10% PVC

Ratio by volume of zinc oxide to rutile titanium dioxide		ZnO A only	75:25	50:50	25:75	TiO ₂ only	K
Decrease in film thickness in $\text{cm} \times 10^{-3}$ period ⁻¹ *	..	1.2	0.24	0.094	0.048	0.028	
Loss of resin in $\mu\text{g cm}^{-2}$ period ⁻¹	..	1300	250	98†	50	29	
Loss of resin calculated from exponential relation	..		350	98	26		52
Loss of resin calculated from linear relation	..		980	650	320		

Ratio by volume of zinc oxide to rutile titanium dioxide		ZnO only	98.8:1.2	97.5:2.5	95:5	90:10	80:20	60:40	20:80	TiO ₂ only	K
Decrease in film thickness in $\text{cm} \times 10^{-3}$ period ⁻¹	1.1	0.85	0.62	0.39	0.17	0.034	0.018				
Loss of resin in $\mu\text{g cm}^{-2}$ period ⁻¹	800	650	430	270†	120	24	13				
Loss of resin calculated from exponential relation	..	740	470	270	90	8					
Loss of resin calculated from linear relation	..	780	720	640	480	160					13.6

40% PVC

*1 period = 133hr of irradiation

†Value of K calculated from this value—see Appendix B

Table 11

Chalking under weather condition P by mixtures of zinc oxide C with rutile titanium dioxide, and zinc oxide A with stainless steel powder, at 40 per cent PVC.

Ratio by volume of zinc oxide C to rutile titanium dioxide	ZnO C only	75:25	50:50	25:75	TiO ₂ only	K
Decrease in film thickness in $\text{cm} \times 10^{-3} \text{ period}^{-1}$ *	1.2	0.57	0.19	0.076	0.016	
Loss of resin in $\mu\text{g cm}^{-2} \text{ period}^{-1}$	850	400	130†	53	11	
Loss of resin calculated from exponential relation		330	130	51		9.3
Loss of resin calculated from linear relation		640	420	210		
Ratio by volume of zinc oxide A to stainless steel powder	ZnO A only	75:25	50:50	25:75	Stainless steel only	K
Decrease in film thickness in $\text{cm} \times 10^{-3} \text{ period}^{-1}$	1.1	0.068	0.068	0.034	0.011	
Loss of resin in $\mu\text{g cm}^{-2} \text{ period}^{-1}$	750	48	48†	24	8	
Loss of resin calculated from exponential relation		210	48	17		13.7
Loss of resin calculated from linear relation		560	370	190		

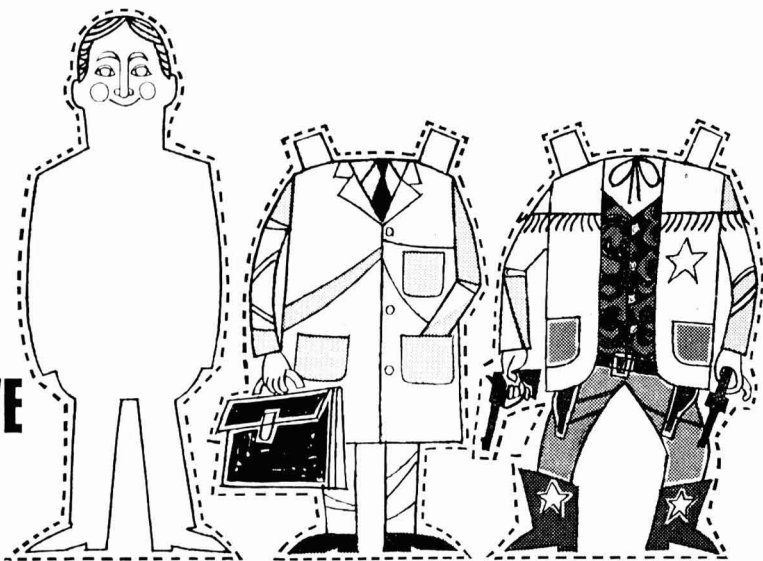
*1 period = 133hr of irradiation

†The value of K calculated from this value—see Appendix B

an effect. Although zinc oxide does not absorb strongly in this region the absorption could be enough to bring the surface of the zinc oxide crystal into a state in which catalytic action occurs. Because of the higher hiding power of rutile titanium dioxide compared with zinc oxide, light of over 400nm will not be able to penetrate as deeply into a paint film containing rutile titanium dioxide as it can into one containing zinc oxide only. The depth of penetration of light into a film will depend exponentially on the concentration of titanium dioxide in it¹⁴.

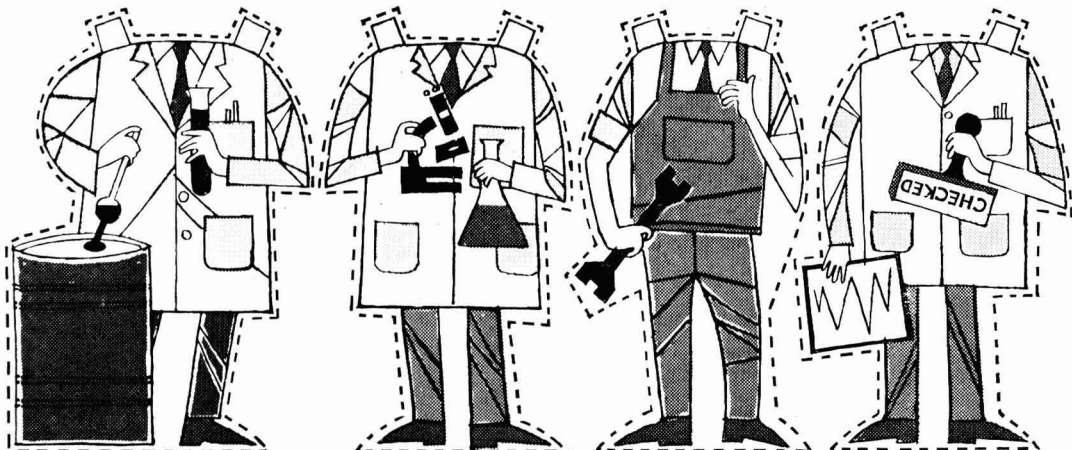
If there is some truth in the outlined hypothesis, two consequences follow: (i) if, instead of using rutile titanium dioxide, a pigment with a higher hiding power is used, the decrease in the rate of chalking should be enhanced (Table 11), and (ii) if mixtures of pigments of the same hiding power are used, the decrease in the rate of chalking should be much less pronounced. Both these

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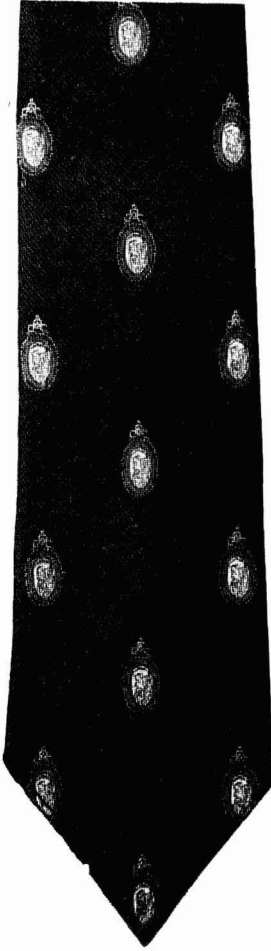
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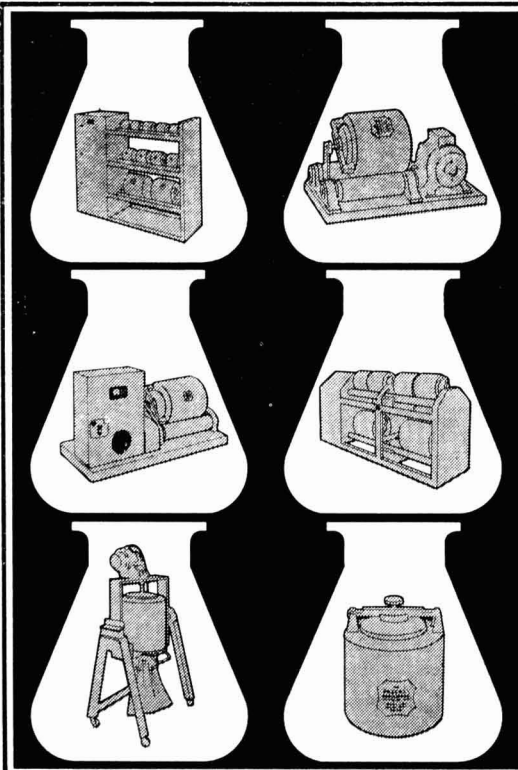
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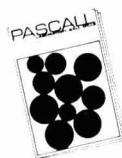
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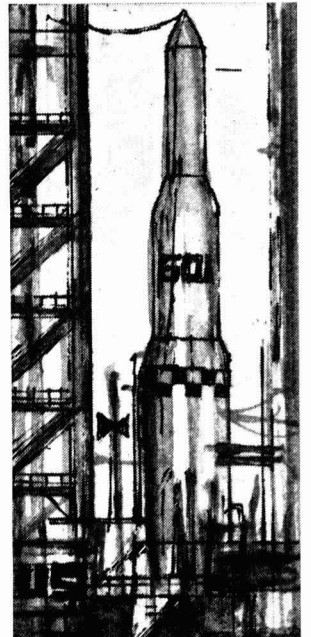
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forecasts are borne out by the observations made with stainless steel pigment (higher hiding power) and a mixture of zinc oxide A and B, which both have a lower hiding power than rutile titanium dioxide (Table 12).

Table 12
Chalking caused by mixtures of zinc oxide A and B at 40 per cent PVC

Ratio by volume of zinc oxide A to zinc oxide B	ZnO A only	75:25	50:50	25:75	ZnO B only
<i>Weatherometer condition P</i>					
Decrease in film thickness in $\text{cm} \times 10^{-3}$ period ⁻¹ *	1.1	1.1	1.0	0.72	0.32
Loss of resin in $\mu\text{g cm}^{-2}$ period ⁻¹ ..	800	800	740	500	220
Highett					
May '67—Nov. '67					
Decrease in film thickness in $\text{cm} \times 10^{-3}$..	0.73	0.73	0.72	0.54	0.46
Loss of resin in $\mu\text{g cm}^{-2}$	510	510	500	380	320
Lae					
July '67—Dec. '67					
Decrease in film thickness in $\text{cm} \times 10^{-3}$..	2.4	2.4	2.1	1.6	1.2
Loss of resin in $\mu\text{g cm}^{-2}$	1700	1700	1500	1100	830

* 1 period = 133hr of irradiation

Experiments at Highett and Lae show (Table 13) that the patterns of chalking behaviour which have been found for exposures in the weatherometer also apply outdoors.

Accelerated weathering

The experiments which have been discussed here have an important bearing on accelerated testing. Every paint chemist knows the unreliability of paint-durability forecasts based on weatherometer data. One author, after trying various weatherometers and cycles, came to the conclusion that, with enough experience and background information, one type of coating might be made to look either better or worse than another by selection of the appropriate conditions for accelerated weathering¹⁵.

It is clear why any of the cycles proposed in the various machines will allow only very unreliable forecasts to be made. The reasons for this have been pointed out in the first part of this discussion. This being so, another more fundamental way of accelerated testing suggests itself: (i) testing should be done in a quantitative way, (ii) before testing the whole formulation, single components and their mixtures should be tested under various conditions of humidity, temperature and light. Once this information is known it is very likely that it will be possible to forecast the behaviour of any coating for any exposure site, provided the climatic conditions for the site are known.

It will be objected, no doubt, that the programme proposed here is impracticably extensive, but the authors do not think that this is the case, because it

Table 13
Chalking caused by mixtures of zinc oxide A and rutile titanium dioxide at 40 per cent PVC

Ratio by volume of zinc oxide A to rutile titanium dioxide	ZnO A only	75:25	50:50	25:75	Rutile TiO ₂ only	K
Hightett						
April '67—Oct. '67						
Decrease in film thickness in $\text{cm} \times 10^{-3}$	1.6	0.6	0.53	0.24	0.20	
Loss of resin in $\mu\text{g cm}^{-2}$	1100	420	370*	170	140	
Loss of resin calculated from exponential relation	5.4
Loss of resin calculated from linear relation	..	630	370	220	..	
Loss of resin calculated from linear relation	..	820	550	270	..	
Lae						
July '67—Dec. '67						
Decrease in film thickness in $\text{cm} \times 10^{-3}$	3.3	1.0	0.06	0.46	0.36	
Loss of resin in $\mu\text{g cm}^{-2}$	2300	700	600*	320	250	
Loss of resin calculated from exponential relation	6.7
Loss of resin calculated from linear relation	..	1200	600	300	..	
Loss of resin calculated from linear relation	..	1700	1150	800	..	

* The value of K calculated from this value—see Appendix B

would be surprising if during such a study rules were not found which simplified things considerably. For instance, the rate of chalking of mixtures of pigments like zinc oxide, rutile titanium dioxide and stainless steel can now be calculated with an accuracy that is probably sufficient for most practical purposes.

To carry out the above programme a weatherometer is needed which allows tests to be made under various humidities, temperatures and wavelengths of light. Unfortunately, reliable equipment of this kind does not seem to be on the market at present.

Conclusions

1. The rate of chalking (expressed as loss of resin) of a paint film decreases with decreasing humidity. The degree to which chalking is affected depends on the pigment volume concentration and appears to be a characteristic quantity for each pigment. The maximum rate of chalking was reached at 10 per cent PVC.

2. The rate of chalking also depends upon the amount and quality of light falling on the paint film surface. The degree to which this happens also appears to be a characteristic quantity for each pigment.

3. The binder most resistant to chalking in the weatherometer was a pva plasticised with tritolyl phosphate, yet at Highett and Lae this binder performed little better than the other film formers which were investigated, a result which shows how unreliable conclusions from accelerated weathering experiments can be.

4. Under accelerated weathering, the rate of chalking of mixtures of zinc oxide with rutile titanium dioxide or stainless steel pigments decreases approximately exponentially with increasing proportions of the latter pigments. The same relationship was found for outdoor exposures at Highett and Lae.

5. All that can be concluded from experiments in the weatherometer is that pigments which cause little chalking (e.g. rutile titanium dioxide) compared with the zinc oxides also cause little chalking on actual outside exposure.

Acknowledgments

Thanks are extended to Mr J. W. Spencer for his estimate of the total energy striking variously-orientated surfaces at Lae, New Guinea, and to Mr B. Molony for his determinations of the surface area of the zinc oxide pigments.

[Received 13 September 1968]

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Appendix A

Possible increase of temperature of a pigment particle owing to absorbed radiation

The maximum solar energy flux at Melbourne incident on a horizontal surface is about $2.68 \times 10^{-2} \text{ cal cm}^{-2} \text{ sec}^{-1}$ ($350 \text{ BTU ft}^{-2} \text{ hr}^{-1}$)¹⁶, of which approximately 50 per cent is infra-red radiation of wavelength greater than 700nm. If it is assumed that as little as 1 per cent of the latter radiation is absorbed in the water layer around the pigment particles the energy absorption would amount to $1.3 \times 10^{-4} \text{ cal cm}^{-2} \text{ sec}^{-1}$. A spherical particle of diameter d , would therefore absorb $0.5\pi d^2 \times 1.3 \times 10^{-4} \text{ cal sec}^{-1}$. The weight of such a particle is $(5.5/6)\pi d^3$ (5.5 is the density of zinc oxide) and the heat needed to increase its temperature by 1°C is $0.11\pi d^3 \text{ cal}$ (specific heat of zinc oxide is 0.11). Therefore the possible increase in temperature of such a particle in one second would be

$$\frac{0.5d^2\pi \times 1.3 \times 10^{-4}}{0.1\pi d^3} = \frac{6.5 \times 10^{-4}}{d} \text{ }^\circ\text{C}$$

For a particle of zinc oxide of diameter 0.2 micron ($2 \times 10^{-5}\text{cm}$) this means a possible increase of 32.5°C per second. This of course will not be the case in practice because an equilibrium will very quickly be reached, but the calculation does show that in an assembly of particles with different particle sizes not all particles can have the same temperature, and that the possible increase in the temperature will be inversely proportional to the particle size. Another reason for some particles being hotter than others is that it is unlikely that all particles will have the same absorption characteristics.

Appendix B

Calculation of K in Tables 10, 11 and 13

The exponential relationship used in attempting to fit the values for chalking is:

$$L = a e^{-KC}$$

where L = loss of weight of resin, $\mu\text{g cm}^{-2}$

a = loss of weight of resin when zinc oxide is the sole pigment, $\mu\text{g cm}^{-2}$

C = pigment volume concentration of rutile titanium dioxide in the paint

K = constant

Example, Table 10. K was calculated from the value at 50:50, $\text{ZnO}:\text{TiO}_2$, 10 per cent PVC, $98 = 1300C^{-0.05 \times K}$, from this $K = 52$.

The painting of galvanised sheet—the performance of priming paints pigmented with 80/20 w/w zinc dust/zinc oxide mixtures

By D. S. Newton

Imperial Smelting Corporation (Alloys) Ltd., Bristol 1

Summary

The performance of newer types of media with 80/20 w/w zinc dust/zinc oxide pigment mixtures as priming paints for galvanised sheet steel is discussed. Long term weathering tests indicate that chlorinated rubber and modifications of the type of drying oil in the oil based primers are equally as good as those specified in US Federal Specification TTP-641-B. The effect of the level of pigmentation is also assessed.

Key words

Binders (resins etc.)

alkyd resin
chlorinated rubber

Oils

linseed oil
wood oil

Prime pigments

zinc dust
zinc oxide

Types of coating

primer

Types of surface

galvanised steel

La peinture de tôle galvanisée—le rendement des couches primaires aux mélanges de 80/20 au poids de poussière de zinc/oxyde de zinc

Résumé

On discute le rendement, en tant que des couches primaires pour tôle galvanisée, des types récents de liant pigmentés aux mélanges de 80/20 au poids de poussière de zinc/oxyde de zinc. Des essais de vieillissement de longue durée démontrent que le caoutchouc chloré et des modifications du type d'huile siccative dans les couches primaires à base d'huile sont aussi effectifs que celles ordonnées dans l'US Federal Specification TTP-641-B. On apprécie, d'ailleurs, l'effet du niveau pigmentaire.

Der Anstrich von Galvanisiertem Blech—das Verhalten von mit 80/20 Zinkstaub/Zinkoxidmischungen pigmentierten Primern

Zusammenfassung

Das Verhalten von neueren Bindemitteln mit gewichtsmässig 80/20 Zinkstaub/Zinkoxid Pigmentmischungen in Primern für galvanisiertes Eisenblech wird besprochen. Langanhaltende Bewitterungsversuche machen es wahrscheinlich, dass sowohl Chlorkautschuk, als auch Modifikationen hinsichtlich der trocknenden Ölart in Primern auf Ölbasis ebenso gut sind, wie die in der US Federal Specification TTP-641-B spezifizierten Sorten. Die Höhe der Pigmentierung wird ebenfalls bewertet.

Окраска гальванизированных плит—эксплуатационные качества грунтовочных красок пигментированных смесями цинковой пыли и окиси цинка в пропорции 80/20 по весу

Резюме

Обсуждаются эксплуатационные качества более новых сред с пигментными смесями цинковой пыли и окиси цинка в пропорции 80/20 по весу, в качестве грунтовочных

красок для тонколистовой гальванизированной стали. Продолжительные сроки испытаний выветривания показывают что хлоркаучук и модифицированные сорта быстровысыхающего масла, в грунтовках на основе масла, одинаково пригодны как и те которые цитируются в Национальной Спецификации С.Ш.А. под номером ТТ-Р-641-В. Оценивается также влияние уровня пигментации.

Introduction

The use of priming paints based on a mixture of zinc oxide and zinc dust has been popular in the United States for many years. The primer, mainly used on galvanised steel, is described in Federal Specification TT-P-641, which details the three types of binders which may be employed, and specifies the total pigment binder ratio as 4.5:1 by weight. Since this specification was first produced, other media have been established in maintenance painting, and more attention has been paid to the level of pigmentation employed.

This paper examines the performance of the specified pigment mixture at various pigment-binder ratios in a number of binders commonly used in the United Kingdom and Europe, both alone and under a conventional top coat.

Formulation of primers

Five pigment binder ratios (2.5:1 to 6.5:1 w/w) were examined in six media, details of which will be found in Appendices I and II.

Paints were prepared as one-pot materials by milling the zinc oxide into the binder, incorporating the zinc dust by high-speed dispersion and finally adjusting to brushing viscosity. Six months' storage tests under laboratory conditions are reported in Appendix III.

Application and exposure of samples

All primers were applied by brushing to hot dipped galvanised steel to BS3083. The systems applied were single coat of primer, two coats of primer, and single and two coats top coated with a single coat of white enamel. Twenty-four hours elapsed between the application of each coat, and the samples were exposed on an industrial site facing south at an angle of 45°, 48 hours after application of the final coat. Film thicknesses were recorded in every case.

Panels were examined for signs of film deterioration at regular intervals over a period of five years.

Records of film thicknesses will be found in Appendix IV.

Results of exposure (by type of binder)

Chlorinated rubber (90003)

Apart from some very slight blistering at pigmentations above 4.5:1 which subsided after 12 months without film fracture in the single coat of primer, no breakdown of any system was noted during the first year. From then onwards both bare primer systems eroded slowly and at the end of five years the single coat showed severe erosion and the two coat system erosion in the brush marks. The top coated systems were in excellent condition and would not have needed any treatment whatsoever.

Alkyd (13008)

Some early blistering of bare primers occurred at all pigment binder ratios, and this remained in evidence for two years. No signs of film fracture at the blister sites was noted however. At the end of the tests erosion was evident on all primer films and those single coats pigmented at 3.5 to 5.5:1 showed signs of rusting. The top coated systems remained in good condition apart from those pigmented at 6.5:1, where blistering at the top coat as opposed to the primer was noted.

Stand oil (40003)

Blistering occurred in the one and two coat primer systems pigmented at 5.5 and 6.5:1 in the early stages of the tests. Erosion was fairly severe, single coats pigmented at 2.5 to 4.5:1 showing rust breakthrough at the end of five years. The top coated systems were in good condition, although some blistering was noted over the 5.5:1 single coat and over both one and two coats of primer at 6.5:1 pigment binder ratio.

Enamel oil (40014)

Erosion occurred on all one and two coat primer systems, with severe rusting of both at 3.5:1 pigment binder ratio. The overpainted primers were in good condition but blistering was noted over those primers pigmented at 5.5 and 6.5:1.

Phenolic/wood oil varnish (33007)

Again erosion occurred on all one and two coat primer systems, but no rusting was noted. The top coated systems all performed excellently but some slight checking of the finish coat was noted over two coats of primer at 2.5:1 pigmentation.

Enamel oil/refined linseed oil (40015)

Erosion was noted in all primer systems, being particularly severe in the case of the single coats pigmented at 3.5 and 4.5:1 where rusting was noted on both panels. All the top coated systems were in good condition.

See Appendix V for full details of results.

Conclusions

The results indicate that within the range of binders examined, and excluding chlorinated rubber, the performance of this type of primer is most dependent on the level of pigmentation.

Failure of the primer occurs mainly by erosion, and thus the surface remains in a satisfactory condition for repainting until rusting occurs. In many cases where rusting does occur there is an appreciable lapse of time between the degree of erosion being classed as "severe" and the onset of rusting.

In general, adhesion to the galvanised sheet is satisfactory, there being only a few cases of blistering, and flaking was absent.

Top coating of this type of primer appears to present no problems. The incidence of blistering is minimal, and no case of flaking was noted.

Of the media examined, chlorinated rubber and the phenolic varnish gave best overall results, both in the primer coats alone and when top coated.

Storage properties varied considerably according to the type of binder, settlement being worse with chlorinated rubber and the phenolic varnish. Skinning also varied. Viscosity increases were not significant, and gassing in this series was not noted. It should be stressed that none of the paints examined contained any structuring or anti-skinning agents and that the judicious use of these would eliminate the adverse properties described in Appendix III.

Acknowledgment

Thanks are due to the Directors of Imperial Smelting Corporation (Alloys) Ltd. for permission to publish this paper.

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Appendix I

Formulations of media examined all in parts by weight

V.90003 chlorinated rubber

Alloprene B (ICI Ltd.)	30%
Cereclor 1 (ICI Ltd.)	20%
Xylene	25%
Dipentene	25%

V.13008 long oil penta alkyd

Epok A1060/75 (BP Chemicals (UK) Ltd.)	19%
30 Poise low acid value stand oil	28.6%
Acid refined linseed oil	14.3%
White spirit	19%
Dipentene	19.1%
Drier addition—0.5% lead as naphthenate 0.5% cobalt as naphthenate						

V.40003 Stand oil

30 Poise low acid value stand oil	85.0%
White spirit	7.25%
Dipentene	7.25%
Drier addition—0.5% lead as naphthenate 0.5% cobalt as naphthenate						

V.40014 25% Wood oil/linseed stand oil

(25% enamel oil) 85% w/w non volatile Wood oil/linseed oil (25/75) stand oil	85%
White spirit	15%
Drier addition—0.05% lead as naphthenate 0.05% cobalt as naphthenate						

V.33007 phenolic wood oil linseed varnish

Epok R.710 (BP Chemicals (UK) Ltd.)	12%
Wood oil/linseed oil (25/75)	48%
White spirit/dipentene (5:3)	40%
Drier addition—0.05% lead as naphthenate 0.05% cobalt as naphthenate						

V.40015 25% enamel oil/refined linseed oil

85% w/w non volatile Wood oil/linseed oil 25/75 stand oil	63.7%
Refined linseed oil	21.2%
White spirit	15.1%
Drier addition—0.05% lead as naphthenate 0.05% cobalt as naphthenate						

Appendix Ia

Formulation coding

PBR	Binder Reference					
	90003	13008	40003	40014	33007	40015
2.5:1	1759	1764	1769	1774	1779	1784
3.5:1	1760	1765	1770	1775	1780	1785
4.5:1	1761	1766	1771	1776	1781	1786
5.5:1	1762	1767	1772	1777	1782	1787
6.5:1	1763	1768	1773	1778	1783	1788

Appendix II

Formulations of priming paints examined all in parts by weight

1. Chlorinated rubber series

Primer Reference

Raw Material	1759	1760	1761	1762	1763
Delaville Superfine zinc dust	200	280	360	440	520
Fricker's White Seal zinc oxide	50	70	90	110	130
V.90003	200	200	200	200	200
Xylene	79	96	104	125	138

2. Alkyd series

Raw Material	1764	1765	1766	1767	1768
Delaville Superfine zinc dust	200	280	360	440	520
Fricker's White Seal zinc oxide	50	70	90	110	130
V.13008	167	167	167	167	167
White spirit	—	—	—	11	23

3. Linseed stand oil series

Raw Material	1769	1770	1771	1772	1773
Delaville Superfine zinc dust	200	280	360	440	520
Fricker's White Seal zinc oxide	50	70	90	110	130
V.40003	118	118	118	118	118
White spirit	24	35	44	56	69

4. Enamel oil series

Raw Material	1774	1775	1776	1777	1778
Delaville Superfine zinc dust	200	280	360	440	520
Fricker's White Seal zinc oxide	50	70	90	110	130
V.40014	118	118	118	118	118
White spirit	35	46	63	76	98

5. Phenolic wood/linseed oil series

Raw Material	1779	1780	1781	1782	1783
Delaville Superfine zinc dust	200	280	360	440	520
Fricker's White Seal zinc oxide	50	70	90	110	130
V.33007	167	167	167	167	167
White spirit	—	11	25	44	51

6. 25% Enamel oil/refined linseed oil series

Raw Material	1784	1785	1786	1787	1788
Delaville Superfine zinc dust	200	280	360	440	520
Fricker's White Seal zinc oxide	50	70	90	110	130
V.40015	118	118	118	118	118
White spirit	3	11	18	25	51

All primers were at equal viscosity (200gms/200rpm on Krebs Stormer Rotational Viscometer at 25°C)

Appendix III

Storage properties of 80/20 zinc dust/zinc oxide primers

Primer Reference	Degree of Settlement	Skinning
1759	Hard & Heavy	Absent
60	do.	do.
61	do.	do.
62	do.	do.
63	do.	do.
64	Soft	do.
65	do.	Very thin
66	do.	do.
67	do.	do.
68	do.	do.
69	do.	Absent
70	do.	do.
71	do.	do.
72	do.	do.
73	do.	do.
74	do.	Very thin
75	do.	do.
76	do.	do.
77	do.	Absent
78	Hard & Heavy	do.
79	do.	do.
80	do.	do.
81	do.	do.
82	do.	do.
83	do.	do.
84	Soft	do.
85	do.	do.
86	do.	do.
87	do.	dc.
88	do.	do.

Appendix IV

	1 Coat	2 Coats	1 Coat + Top Coat	2 Coats + Top Coat
thousandths of an inch				
1759	1.7	2.7	1.9	2.9
60	2.5	3.1	3.1	3.7
61	1.9	2.3	2.5	3.8
62	1.9	2.7	3.1	3.8
63	1.6	2.1	2.4	2.7
64	1.4	1.9	2.2	3.1
65	1.7	2.1	2.5	3.0
66	1.4	2.3	2.2	2.9
67	1.3	1.9	2.1	2.7
68	1.5	2.0	2.3	2.9
69	1.5	2.0	2.1	2.5
70	1.4	2.0	2.4	3.0
71	1.2	2.0	2.0	2.7
72	1.4	2.1	2.6	3.1
73	1.6	2.3	2.6	3.0
74	1.8	2.3	3.0	3.5
75	1.3	1.6	1.9	2.3
76	1.7	2.0	2.5	2.7
77	1.4	2.1	2.1	2.7
78	1.5	2.0	2.4	2.8
79	1.8	2.6	2.8	3.9
80	1.5	2.0	2.3	3.6
81	2.0	2.6	2.9	3.5
82	1.6	2.0	2.2	2.8
83	1.3	1.9	2.5	3.0
84	1.4	1.7	1.9	2.3
85	1.3	1.8	2.2	3.0
86	1.3	1.7	1.9	2.5
87	1.3	2.0	2.0	2.9
88	1.4	2.1	2.2	2.9

Appendix V

Exposure results after 5 years (Industrial site)

E—Moderate Erosion
EE—Severe Erosion

B—Blistering

R—Rusting
RR—Severe Rusting

Single coat of primer

PBR	90003	13008	40003	40014	33007	40015
2.5:1	E	EE/B	R	E	E	E
3.5:1	E	EE/B/R	R	RR	E	R
4.5:1	E/B	EE/B/R	R	E	E	R
5.5:1	E/B	B/R	EE/B	EE	E	EE
6.5:1	E/B	EE/B	EE/B	EE	E	EE

Two coats priming paint

2.5:1	E	E/B	E	E	E	E
3.5:1	E	E/B	E	RR	E	E
4.5:1	E	E/B	E	E	E	E
5.5:1	E	E/B	E/B	E	E	E
6.5:1	E	E/B	E/B	E	E	E

One coat primer and top coat

2.5:1	—	—	—	—	—	—
3.5:1	—	—	—	—	—	—
4.5:1	—	—	—	—	—	—
5.5:1	—	—	B	B	—	—
6.5:1	—	B	B	B	—	—

Two coats primer and top coat

2.5:1	—	—	—	—	—	—
3.5:1	—	—	—	—	—	—
4.5:1	—	—	—	—	—	—
5.5:1	—	—	—	B	—	—
6.5:1	—	B	B	B	—	—

Testing and investigation of ship bottom paints*

By A. M. van Londen

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Summary

The economic importance of good protection of ship's hull and the need for adequate research in this field are discussed.

The testing of anti-corrosive and anti-fouling coatings is dealt with separately. Current testing methods are scrutinised and some improved methods are indicated or described.

The corrosion and fouling problem, being of world-wide interest, calls for a close international co-operation.

Key words

Types of coating

antifouling coating
corrosion resistant coating
ship bottom paint

Methods and processes of analysis measurement and testing

accelerated corrosion tests
electron microscopy
exposure test
laboratory test
x-ray spectroscopy

L'essaiage et l'investigation de peintures de carène

Résumé

On discute l'importance, au point de vue économique, d'assurer une bonne protection aux carènes de navire et également la nécessité dans ce domaine des recherches compétentes.

L'essaiage des revêtements anti-corrosifs et anti-fouling se traite sous des rubriques particulières. Les méthodes d'essai courantes sont examinées et quelques méthodes perfectionnées sont mentionnées ou décrites.

Les problèmes de corrosion et de fouling, étant d'intérêt universel, exigent de la co-opération à la fois internationale et serrée.

Prüfungsmethoden und Untersuchungen von Schiffsbodenfarben

Zusammenfassung

Die Wichtigkeit wirksamen Schutzes der Schiffshülle aus wirtschaftlichen Gründen, und die Notwendigkeit genügender Forschung auf diesem Gebiet werden besprochen. Prüfmethode für Korrosionsschutz- und Antifoulinganstriche werden getrennt betrachtet. Die z.Zt. üblichen Prüfungsmethoden werden unter die Lupe genommen und auf einige verbesserte Methoden wird hingewiesen, oder diese werden beschrieben.

Das Korrosions- sowie das Antifoulingproblem erfordert in Anbetracht des dafür bestehenden weltweiten Interesses enge internationale Zusammenarbeit.

Испытание и исследование красок для подводной части судов

Резюме

Обсуждаются экономическое значение надлежащей защиты корпуса судов и необходимость соответствующих исследований в этой области.

* Presented to the London Section, Southern Branch, on 11 March 1968 and to the Newcastle Section on 12 March 1968.

Испытания противокоррозионных и необрастающих покрытий рассматриваются отдельно. Анализируются современные методы испытания и отмечаются и описываются некоторые усовершенствованные приемы.

Проблема коррозии и загрязнения имеет всемирное значение и требует международного сотрудничества.

Introduction

Ship bottom paint systems are used to protect the ship's hull under the water-line against corrosion and fouling. This protection is not only necessary for material preservation but also for maintaining a smooth surface.

The great economic importance of a smooth surface is still not sufficiently realised or generally accepted. Considerable sums are spent to give a ship a special shape providing a minimum resistance. To obtain this an optimum surface treatment is, however, a second requisite. It is remarkable that relatively little attention is paid to this part of the work, for the surface roughness largely determines the total resistance of a ship to motion. This in turn has a considerable effect on fuel consumption

On the basis of an economic study of the relationship between the treatment of ships' hulls and fuel consumption carried out in the Netherlands¹ it is possible to calculate that for the British merchant navy an annual saving of at least £10,000,000 may be achieved on fuel consumption alone within the present-day technical possibilities. This means adequate pre-treatment of the steel substrate, the use of good paint systems and additional corrosion protection by a cathodic installation.

In order to fulfil its dual function, a ship bottom paint system consists of two separate parts, an anti-corrosive and an anti-fouling part. Adequate anti-corrosive and anti-fouling functions cannot be combined in one paint. This is due to the fact that an anti-corrosive coating has to be as impermeable and unchangeable as possible in order to separate the metal from the sea-water and to avoid the passage of water, gases and ions, which effect the corrosion reactions.

An anti-fouling coat, on the other hand, can only be effective if toxic material is being released from the paint layer. This means that transport of material through the paint film is a necessity. Thus the requirements for an anti-corrosive and an anti-fouling coating are opposed and for this reason a ship bottom paint system consists of an anti-corrosive part with an anti-fouling coat on top.

In the last decade, real progress has already been made in the conservation of ships by paying more attention to the pre-treatment of the steel, the use of pre-construction primers, the application of thick anti-corrosive coatings, using better anti-fouling paints and the installation of cathodic protection devices. Furthermore, the development and use of airless spray equipment and high pressure water cleaning have been of help.

Especially in the anti-fouling field, more progress is desired and will be made possible if research provides a better understanding of fouling deposition and the actual processes which prevent the fouling organisms from depositing. The development of advanced analytical techniques is making this goal increasingly attainable.

This paper deals with the testing and investigation of ship bottom paints with particular reference to anti-fouling compositions. Besides an evaluation of existing test methods, other possibilities are suggested and some results are discussed.

It will be convenient here to distinguish the terms *testing* and *investigation* as used by the author. Generally speaking, testing can only be used to find out whether a certain product or procedure is better than another one, presupposing the testing method to be correct. An investigation is not carried out to differentiate between existing paints or procedures, but to gain knowledge in order to be able to make distinct improvements.

Paint making has long been an art and still lacks adequate scientific basis. Only research and a sound scientific approach can lead to the better understanding needed for real progress.

Testing and investigation of anti-corrosive ship bottom paints

Testing methods

The testing of anti-corrosive ship bottom coatings is being carried out by means of exposure tests in the sea (raft testing), practical tests on ships, and accelerated tests in the laboratory.

Raft testing does not offer the best possibilities for assessing the anti-corrosion properties of paint layers. The exposure test can be carried out with or without an anti-fouling coat on top of the anti-corrosive priming coat. In neither case, however, is the desired direct observation of the anti-corrosive priming coat possible. This coat is hidden by the anti-fouling coat or quickly becomes covered with fouling organisms during the exposure². Moreover, the anti-fouling coat or the fouling organisms may influence the behaviour of the anti-corrosive coating.

Exposure tests in fouling-free sea-water offer better possibilities. The exposure can be carried out in the laboratory in a tank with filtered, natural, running sea-water excluding light to prevent algal growth. An additional advantage of such a test is the possibility of standardising conditions such as temperature and oxygen-content. A difficulty may be the supply of fresh natural sea-water in sufficient quantities.

Practical tests on ships are usually done by means of test patches on the hull. This method has some important drawbacks^{2, 3}. The application of the paints has to be carried out on new ships during the building period on the stocks or on old ships during a dry-docking period. Thus the conditions during application cannot be controlled sufficiently. The inspection of test patches can only be done in dry dock, which is not the most suitable place for making careful and accurate observations. The test only offers an instantaneous picture, information pertaining to the course of the test not being obtained. The test has the same drawback as raft tests where direct evaluation of anti-corrosive paint layers is concerned.

Some of these disadvantages can be overcome by using the so-called bilge-keel test panels. The paints are applied on special test panels, which can be fastened on the bilge-keel (see Fig. 1). These test panels can be prepared in the

laboratory under standard conditions and the examination can be done in the laboratory as well, using all the required instruments.

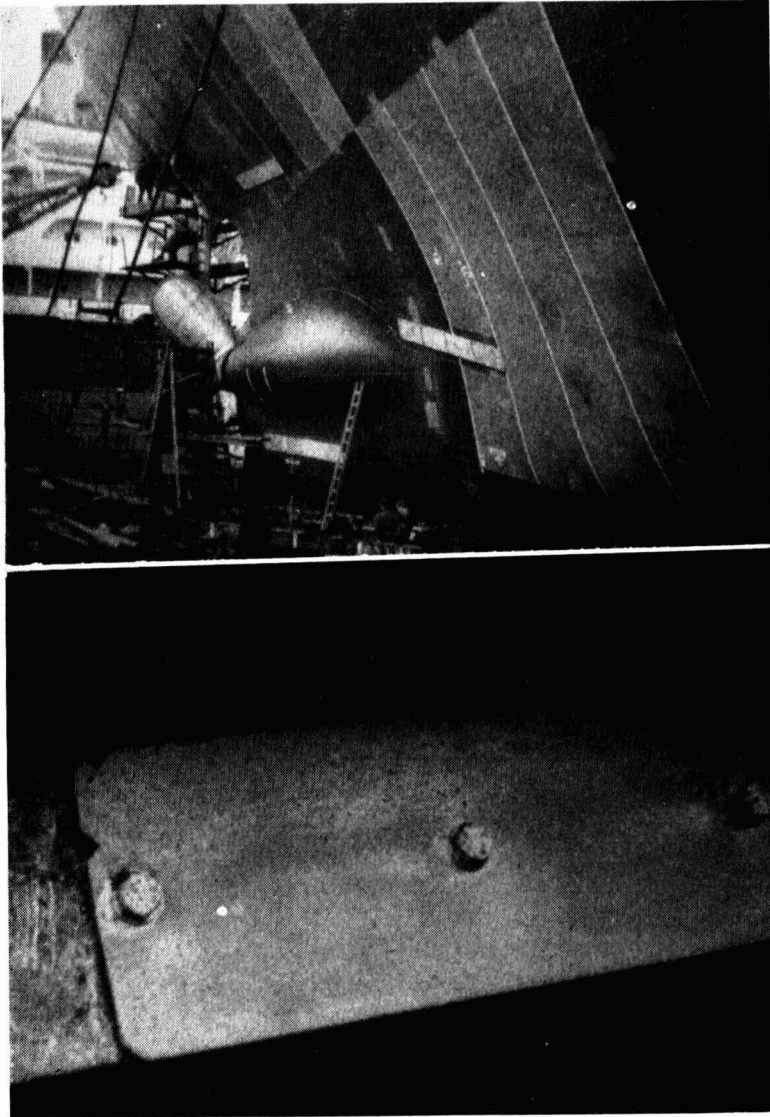


Fig. 1. Practical testing on ships by means of test patches and using bilge-keel test panels

Laboratory tests make it possible to control the conditions and to accelerate the changes by using special techniques. There is a real risk, however, of using a test procedure in which the conditions differ so much from those in practice that the results have no practical meaning. This must especially be kept in mind if accelerated tests are being used, such as the salt spray test, artificial weathering tests, etc.⁴ Even the use of artificial sea-water may give doubtful results because of the absence of trace elements and organic substances and the different buffering action⁵.

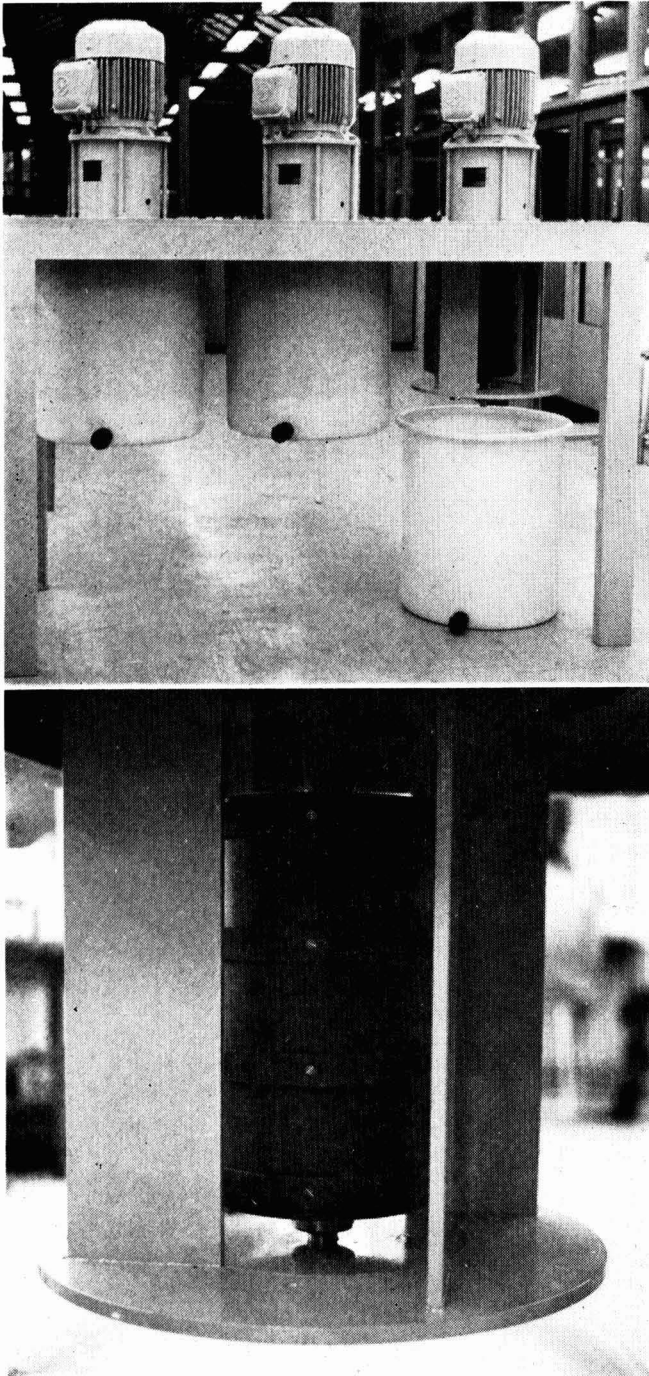


Fig. 2. Rotor apparatus (see also page 146)

More reliable results can be obtained using natural sea-water. An acceleration of the test can be obtained by aeration or by imitation of the water movement along the ship's hull. In the TNO laboratory a so-called rotor test is in use for this purpose. Appropriate test panels are fastened to a cylinder, which can be rotated in sea-water at a known peripheral speed. (Fig. 2.) Information can be quickly obtained on anti-corrosive action and paint coating defects such as cracking, blistering and flaking.



Fig. 2. Rotor apparatus with details of the fastening of the test panels and showing some serious defects in paint systems after ageing

Assessment of defects

The assessment of defects arising during tests and the evaluation of the results are far from simple.

For the assessment of rust formation on steel test panels coated with anti-corrosive ship bottom systems, the European Rust Scale is not a suitable standard. This scale is based on the rusting of steel coated with one type of paint system exposed to a particular atmosphere. The same type of paint exposed to another environment may give rise to quite another type of rust formation. This is certainly the case with test panels exposed to sea-water.

Another drawback of the scale is that the figures obtained cannot be evaluated statistically in a simple way. An assessment of rusting based upon percentage of area^{6, 7} covered with rust is much more suitable. It may be useful to give, besides the percentage of rust, an additional description of type of rust, size and distribution of rust spots, and the colour, structure and adherence of the corrosion products. However, the big advantage is in the possibility of reducing different kinds of rust formation to one denominator. The results can be interpreted without any special standard for comparison and may easily be evaluated

statistically. Another advantage is that the same method of assessment can be used with different defects in the paint coating such as blistering and flaking.

The use of a graduated templet simplifies the estimation of the area on which a defect occurs. In Fig. 3 an example of such a templet and its use is given.

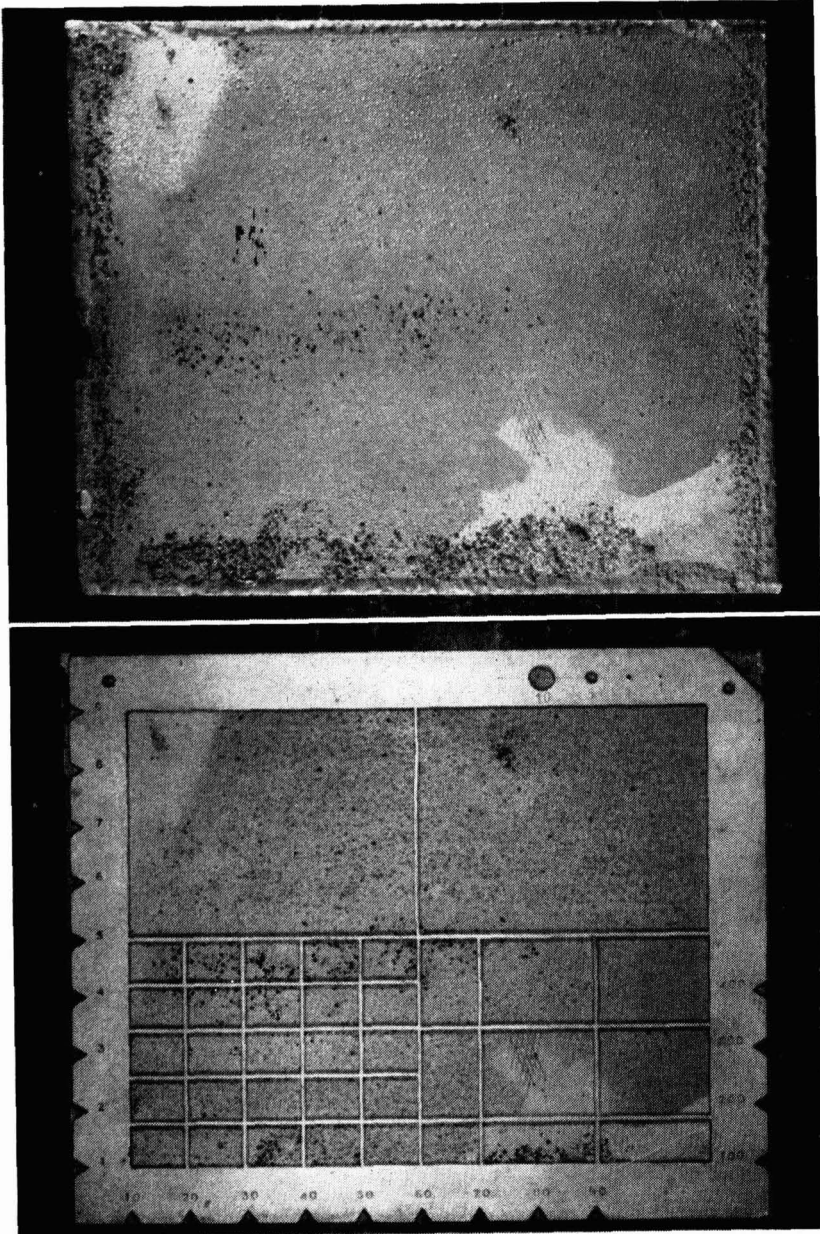


Fig. 3. Assessment of defects on a test panel using a templet to simplify estimation of the percentage of the area with the defects

Edge effects are masked by a strip 2.5cm wide around the periphery of the panel.

Research possibilities

A lot of work has been carried out for many years in testing by comparison anti-corrosive paints, pre-treatment methods and methods of application by means of some kind of exposure test. All over the world thousands of test panels have been carefully prepared and exposed; the exposure conditions and other data have been recorded and the formation of rust and defects in the paint coats have been described. All this work has merely resulted in files of data indicating that one paint formulation or working procedure is better than another. Such work seldom results in more basic knowledge of processes and factors involved. Therefore, research work must be directed to obtaining basic knowledge of the anti-corrosion mechanisms of anti-corrosive coatings. The real problem is to find the material properties that are responsible for the complicated anti-corrosion action.

Research on the permeability of coatings⁸, ion exchange⁹, diffusion of ions^{9, 10}, water uptake^{12, 13}, electrical resistance and capacitance^{14, 15}, seems to be the correct way to tackle the problem.

Besides the use of radioactive material, another technical possibility for studying the processes in paint coatings and at the interface between coating and steel substrate may be in cross-sectioning the anti-corrosive coat and its steel substrate together¹⁶ and analysing the cross-sections using X-ray micro-analysis. This procedure not only provides qualitative and quantitative data but also gives local information about spots as small as some cubic microns.

Testing and investigation of anti-fouling paints

Testing methods

There are three main ways in use for testing anti-fouling paints, analogous to the testing of anti-corrosive ship bottom paints, viz. raft testing, ships trials and laboratory tests.

Raft testing is so well-known that a detailed description of this method seems to be superfluous. Besides such drawbacks as geographical differences¹⁷, local differences¹⁸ and a scale effect (fouling on a relatively small test panel compared with the wet area of a ship's hull)¹⁹, a serious objection can be made to the method as it is currently practised. To make this clear, the mechanism of action of an anti-fouling paint must be recalled to mind.

An anti-fouling paint contains a poison, which is slowly released into the surrounding sea-water by one or another diffusion process. As a matter of course, the rate of poison release determines the effective life of an anti-fouling paint coat. This rate is affected considerably by the conditions of ageing of the anti-fouling coat.

There is a definite and important difference between the ageing under the dynamic conditions on a ship's hull and the ageing under the static conditions on a raft²⁰. The velocity of water flowing along the ship's hull plays a decisive role in this process, as can be seen in Fig. 4. However, there is not only a difference in the rate of release but also in the way release occurs, as is demonstrated in Fig. 5.

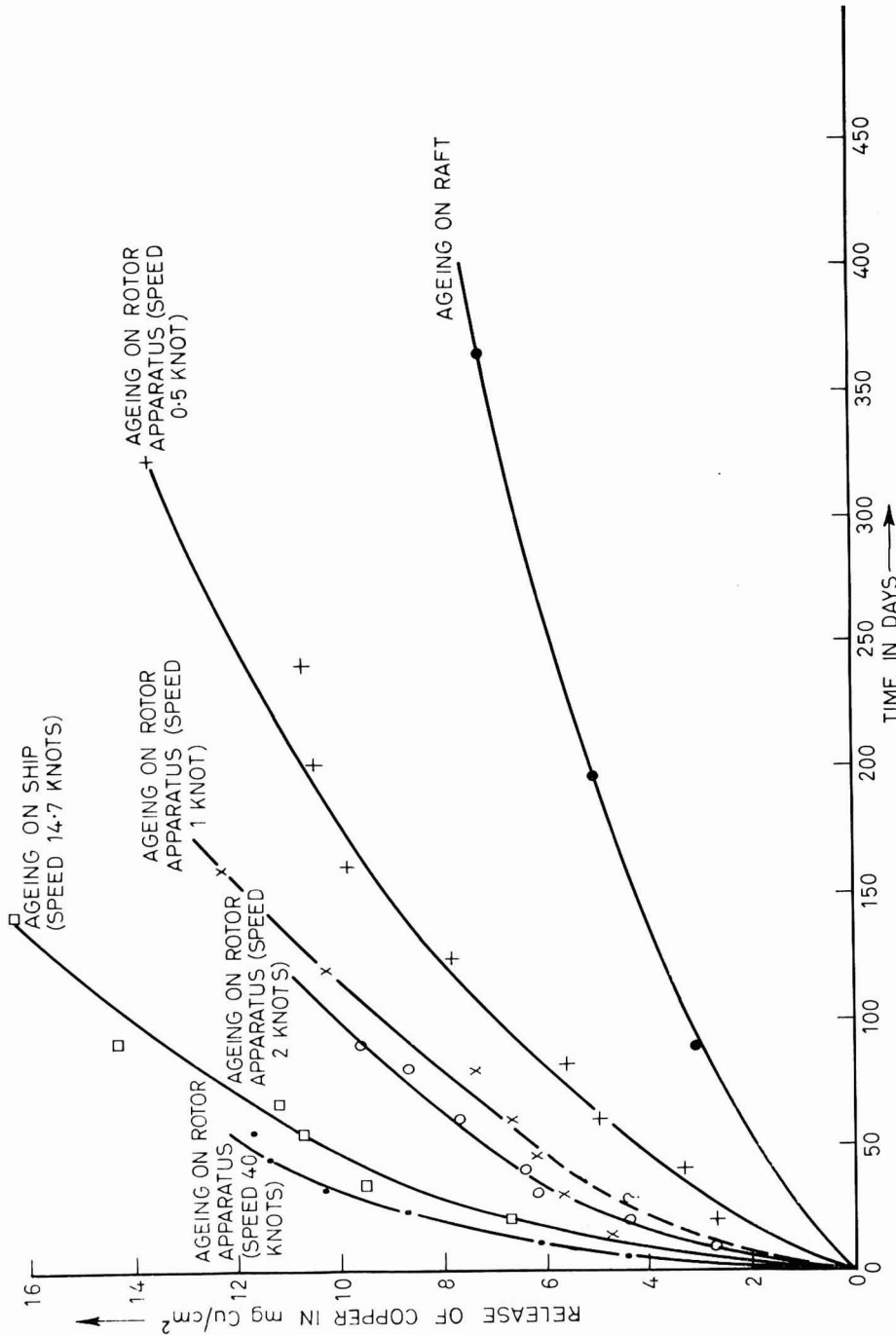


Fig. 4. Effect of water velocity on ageing

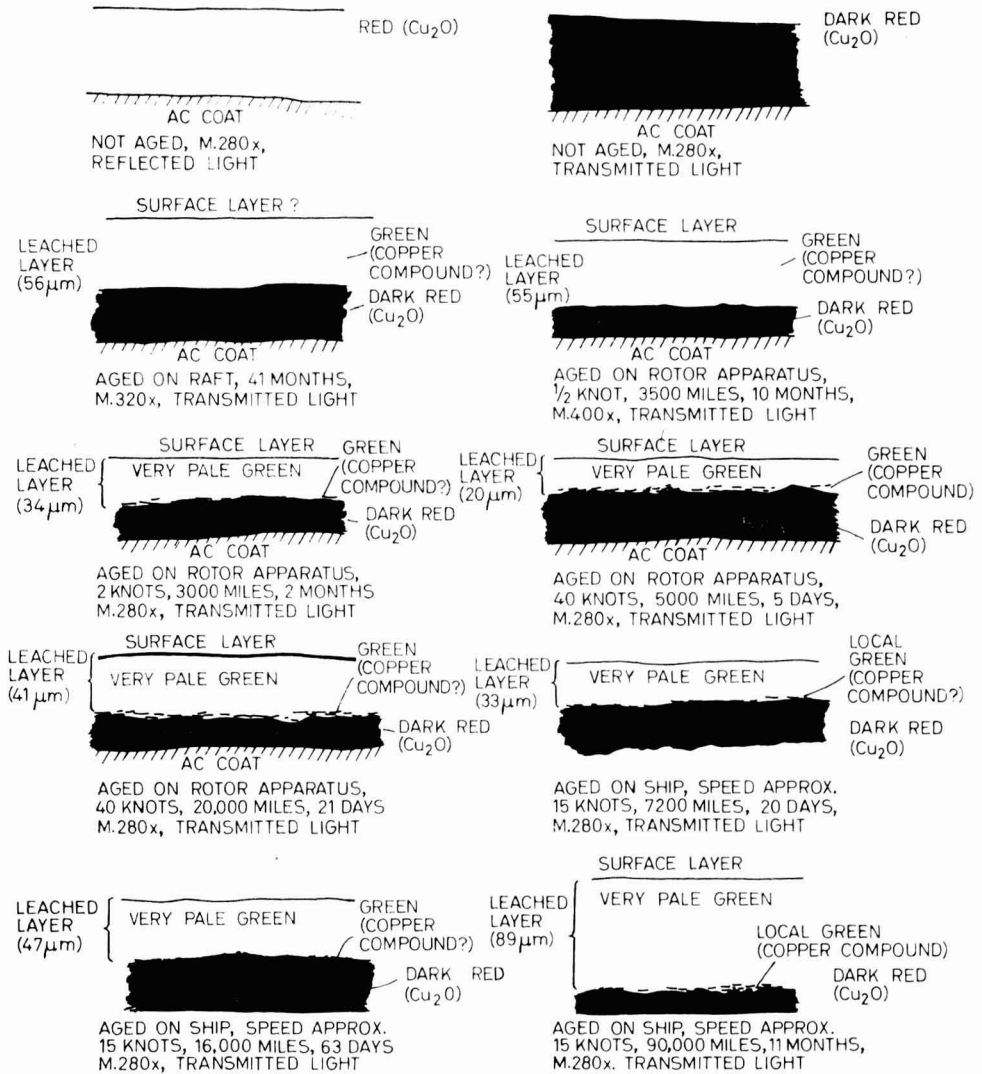


Fig. 5. Sketches of cross sections of paint aged by various methods

The raft test, as it is generally being carried out, is a combination of a method of ageing and a biological test. These, however, cannot be satisfactorily combined on rafts under static conditions. To obtain an ageing comparable with practice the requisite water velocity around the test panels prevents deposition of fouling organisms. Or, on the other hand, when fouling organisms are able to settle the ageing is not as desired.

A solution of this problem lies in using raft testing only as a biological test and applying another method of ageing. It may be possible to use raft tests with a relatively low water velocity around the test panels but this needs further investigation.

Accelerated ageing by means of a rotor apparatus provides a way of ageing analogous to that in practice²⁰. See Fig. 5.

The preferred method of conducting practical tests on ships is by means of bilge-keel testing for the reasons mentioned earlier.

Laboratory tests on anti-fouling paints which have been known for years, are the glycine test, acid-alkali test and the determination of the leaching rate²¹. These tests have been developed for copper based anti-fouling paints and cannot be used without modification for paints containing other poisons. The usefulness of the glycine and alkali-acid tests has never been unequivocally demonstrated. They may be used for routine checking of batches of one and the same paint formulation^{22, 23, 24}. The value of the tests is open to criticism, on the grounds that the release of copper is proceeding otherwise than under natural conditions^{23, 24}.

The determination of the leaching-rate by a standardised method is more generally accepted to establish the anti-fouling properties of a paint. The limitations of the method have been described²⁵. In fact, the method can only serve as a comparative test. It may be used as an indication of whether or not an anti-fouling paint will prevent fouling at a particular moment.

For copper based paints, the minimum critical value to prevent fouling is given to be $10\mu\text{g Cu cm}^{-2}\text{day}^{-1}$. This figure is debatable, as many times paints having a lower leaching-rate do show a sufficient anti-fouling action²⁰. Furthermore, it has to be realised that the leaching-rate is a mean value for the total area of the test panel. This means that, on the test panel, areas may be present with such a low leaching-rate that fouling organisms can settle locally²⁶. If only 0.1 per cent of the total area were represented by such spots on a ship, fouling could occur to an amount of, for example, 50 barnacles, with a diameter of 0.5cm, per square metre.

A leaching-rate determination, therefore, should be considered as a chemical substitute for a biological test. The latter is to be preferred and may be irreplaceable on occasions.

A good method for testing anti-fouling paints, irrespective of the poison used, is the ageing in the laboratory by means of rotor testing already described, followed by exposure in the sea during the fouling season (Fig. 6). A useful additional figure is the amount of residual poison in the anti-fouling paint after ageing. This can be determined analytically and, sometimes, by examining cross-sections²⁰. If this is done after several ageing periods in combination with exposure tests to check the anti-fouling activity, it is not only possible to assess the effective life of the paint but also to see if the release of poison is as desired (Fig. 7).

Assessment of defects and fouling

The assessment of defects in the anti-fouling paint coat may be carried out according to the directions of the British Admiralty⁷.

The assessment of the deposited fouling is a complicated matter. At the moment a practical, accurate and world-wide applicable method is not available. In the Netherlands work is in hand to find a reliable method, in which

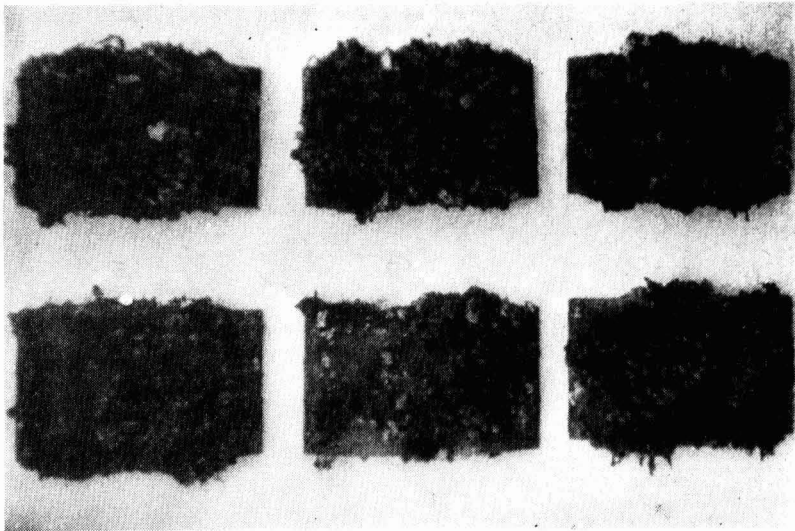


Fig. 6. Six anti-fouling paints completely fouled after an ageing equivalent to 20,000 miles on a rotor apparatus and a 9 weeks' raft exposure. Without the preliminary ageing the paints showed a fouling-free life of two years exposed from the same raft.

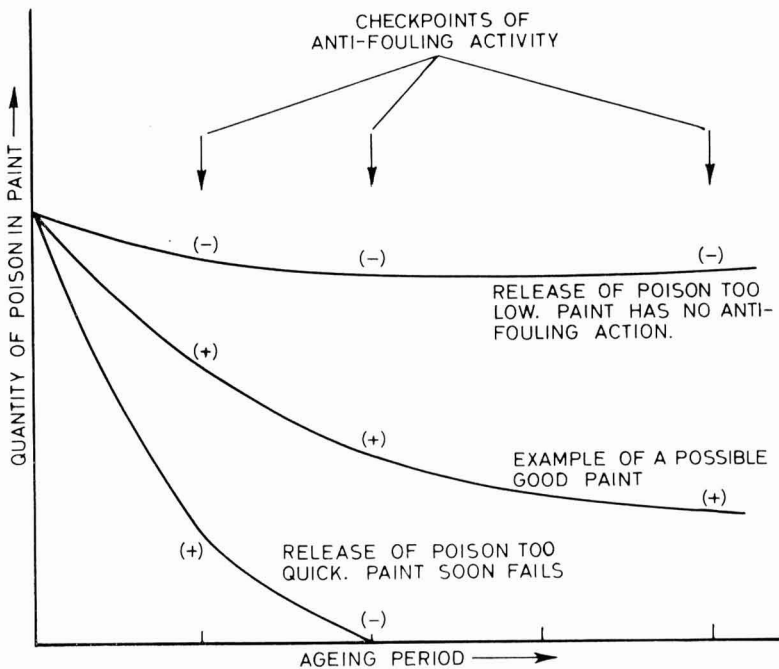


Fig. 7. Assessment of poison release by estimation of residual poison

account can be taken of geographical and local differences, the natural mortality and, in case of raft testing, the scale effect.

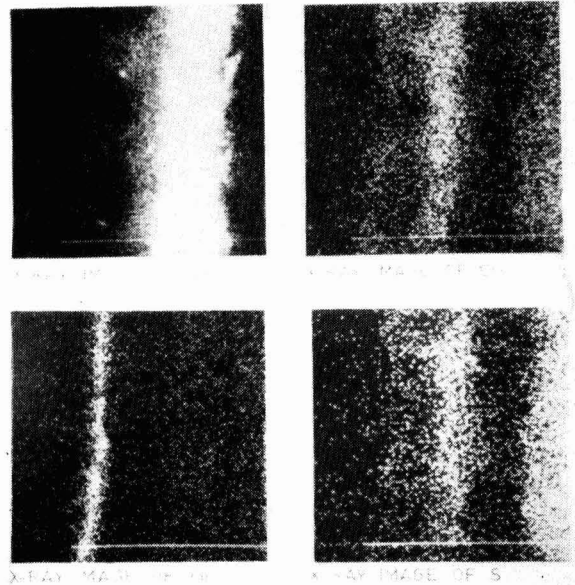
Investigation by means of cross-sections

The processes taking place in the paint coating on ageing are of direct interest

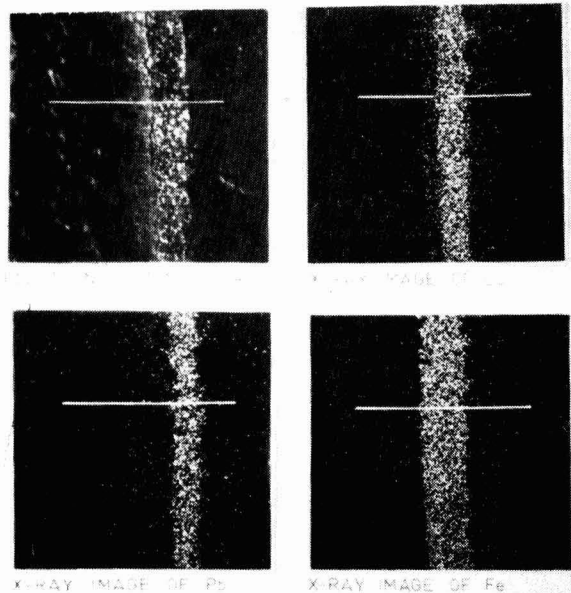
for obtaining a better insight into the mechanism of action. The method which is used for this purpose in the TNO laboratory at Den Helder is cross-sectioning of the paint film²⁷ followed by a microscopic examination and/or electron microprobe analysis, in which the principles of optical and electron microscopy and X-ray spectral analysis are combined. Hence it is possible to study not only the release of material from the coating, but also the penetration of material into the paint film.

In Fig. 5 sketches are given of a number of cross-sections. These demonstrate clearly the possibilities of this technique and also the limitation of a mere microscopical examination. The release of copper from the paint coat can easily be traced. However, in the leached layer and at the surface, unknown products are being formed, the nature of which cannot be established by microscopical examination.

The additional information which can be obtained through the use of the electron microprobe is well illustrated in Fig. 8. Its full interpretation must, however, await the collection of more data.

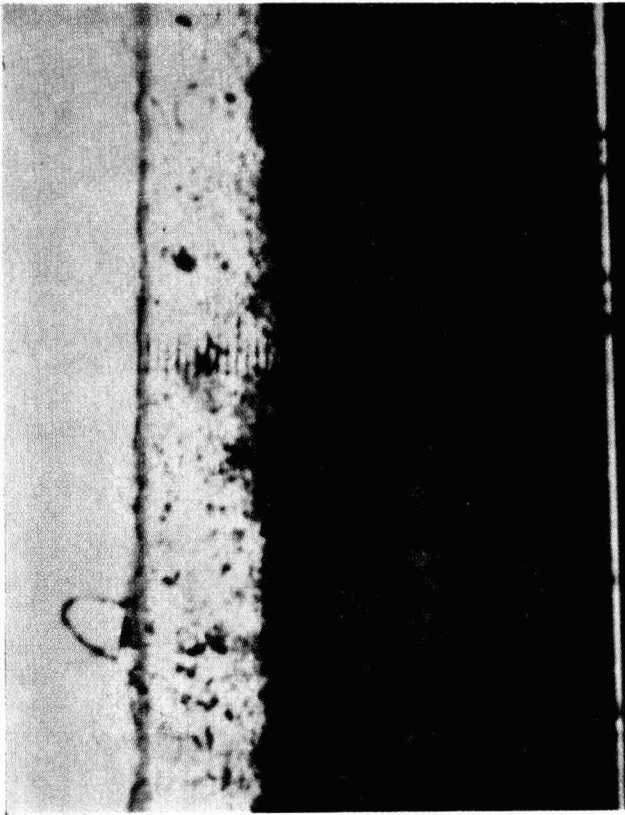


The x-ray images of an anti-fouling paint pigmented with cuprous oxide only, aged on a rotor apparatus 20,000 miles



The x-ray images of an anti-fouling paint pigmented with cuprous oxide and red iron oxide, aged on a rotor apparatus 35,000 miles

Fig. 8. Photo micrographs and x-ray images of aged anti-fouling paints, showing the possibilities of electron microprobe analysis. (See also pp. 154 and 155)



AF paint

AC paint

Photo micrograph of cross-section of an aged a.f. paint pigmented with cuprous oxide only

Future work

Theoretical considerations have led to the concept that the diffusion of poison in the coating may take place in different ways, depending on the nature of the poison, the solubility of the poison in the vehicle, the "sea-water sensitivity" of the paint coat and its PVC. This concept is to be checked by using accelerated ageing and the examination and analysis of cross-sections of the aged paint films.

General discussion

It has been shown that it is economically justifiable to undertake research on anti-corrosive and anti-fouling compositions. It is considered that this research must be aimed at the anti-corrosion and anti-fouling mechanisms.

A critical evaluation of the testing methods in use demonstrates the necessity

for modifications. Possible improvements have been indicated.

New methods of investigation have proved their usefulness in anti-fouling paint research. The same methods may be applicable to the study of anti-corrosive paint coatings.

The solution of the problems concerning the protection of ships' hulls is of world-wide interest. A form of international co-operation on research in this field has been realised in the Permanent International Committee for Research on the Preservation of Materials in the Marine Environment. However, more member countries are needed in this committee to enable successful international co-operative work. This may not only provide useful information concerning geographical conditions and differences, but also permit research beyond the facilities of any one country.

Conclusions

The following conclusions may be drawn.

More attention to the treatment of ships' hulls can lead to considerable savings for the ship-owners.

Good anti-corrosion and anti-fouling properties cannot be combined in one paint.

Raft testing is not suitable for testing anti-corrosive ship bottom coatings. Laboratory tests offer better possibilities.

Assessment of rusting using a percentage-based scale is a practical, generally applicable, method.

Practical tests on ships using test panels may offer better investigation possibilities than test patches on the hull itself.

The ageing of an anti-fouling paint during raft testing differs quantitatively and qualitatively from the ageing under dynamic conditions.

Raft testing can only be used as a biological test for establishing anti-fouling properties.

The assessment of the deposition of fouling organisms is very complicated and a practical, generally acceptable method is not available.

Ageing of anti-fouling paint coats on a rotor apparatus is closely comparable to ageing on a ship's hull.

The examination and analysis of cross-sections of anti-fouling paint coats is a useful method of investigation and the method can also be used for other coatings.

More international co-operation is desirable in the field of marine corrosion and fouling research.

Acknowledgment

The research work on anti-fouling and anti-corrosion action of ship bottom compositions in the Netherlands is being made possible by a close co-operation between the industry and the Organisation TNO.

The following industrial organisations participate in the work:
—ship-owners (Koninklijke Nederlandsche Reeders Vereniging).



AF paint

AC paint

Photo micrograph of cross-section of an aged anti-fouling paint pigmented with cuprous oxide and iron oxide

- ship-yards (Centrale Bond van Scheepsbouwmeesters in Nederland).
- paint manufacturers (Nederlandsche Vereniging voor Research op het gebied van Scheepshuidverven).

In the Organisation TNO this work has resulted in a close collaboration between the following institutes:

- The Netherlands Ship Research Centre TNO.
- The Central Laboratory TNO.
- The Metal Research Institute TNO, and
- The Paint Research Institute TNO.

Thanks are due to the industry for its support and to the collaborators of the various institutes engaged in this work.

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Next month's issue

The Honorary Editor has accepted the following papers for publication and these are expected to appear in the March issue:

"Formulation of fungus-resistant paints. IV: Addition of tetramethyl thiuram disulphide," by *E. Hoffmann and A. Saracz.*

"The migration of lead from paint films in the rat gastro-intestinal tract," by *J. C. Gage and M. H. Litchfield.*

"The use of an electrical resistance method for investigating the performance of polymer coatings," by *G. W. Rothwell.*

"The examination of the structure of anti-fouling coatings by scanning electron microscopy," by *J. H. Bishop and S. R. Silva.*

Further work on the polarisation testing of painted specimens*

By H. F. Clay

Cromford Colour Co. Ltd., Matlock, Derbyshire

Summary

This paper is a continuation of an earlier one which set out a case for applying electrochemical polarisation techniques to the study of the corrosion resistance of painted specimens. A method of potentiostatic testing is described and the results are compared with those obtained by the galvanostatic method previously described.

The two methods of testing give similar pictures, although the potentiostatic method is rather more informative. The techniques are useful in supplementing visual testing and show promise of increasing the knowledge of the mechanism by means of which paints control corrosion.

Key words

Types of coating

alkyd coating primer

Types of surface

steel

Prime pigments

iron oxide pigment

Apparatus associated with analysis, measurement and testing

electrochemical apparatus

salt spray cabinet

Properties, characteristics and conditions associated with dried films

corrosion resistance measurement
galvanic action

De nouvelles études sur l'épreuve des éprouvettes peintes par la technique de polarisation électrochimique

Résumé

Cet article est une continuation d'un article précédent dont l'auteur posait la cause pour s'en servir des techniques électrochimiques en étudiant la résistance à corrosion des éprouvettes peintes. On décrit une méthode d'essai potentiostatique et l'on fait comparer les résultats auprès de ceux obtenus par la méthode galvanostatique décrite au préalable.

Les deux méthodes d'essai donnent des indices pareilles bien que la méthode potentiostatique soit plutôt plus informative. Les techniques sont utiles en augmentant les épreuves visuelles, et elles démontrent la possibilité d'augmenter la connaissance du mécanisme par lequel les peintures peuvent régler le degré de corrosion.

Weitere Arbeiten über die Polarisierungsprüfung von Lackproben

Zusammenfassung

Diese Abhandlung ist die Fortsetzung einer früheren Arbeit, die es sich zur Aufgabe gemacht hatte, auf die Bedeutung der Eignung elektrochemischer Polarisierungstechniken zum Studium des Widerstandes gegen Korrosion von mit Anstrichen geschützten Proben hinzuweisen. Es wird eine potentiostatische Prüfmethode beschrieben, und deren Resultate mit denen verglichen, die bei der früher beschriebenen galvanostatischen Methode erhalten worden waren.

Diese beiden Prüfungsmethoden ergeben ähnliche Bilder, mit Hilfe der potentiostatischen Methode erhält man allerdings etwas bessere Information. Die Techniken ergänzen nützlicher Weise visuelle Prüfungen und versprechen die Kenntnisse von dem Mechanismus, mit Hilfe dessen Anstrichmittel Korrosion unterdrücken, zu erweitern.

*Presented to the Manchester Section, 11 November 1968.

Дополнительная работа в области испытания поляризации красочных образцов

Резюме

Статья является продолжением ранее опубликованной статьи в которой описывалось применение техники электрохимической поляризации в исследовании сопротивления красочных образцов против коррозии. Описывается метод потенциалостатического испытания и результаты сравниваются с результатами полученными при помощи ранее описанного гальваностатического метода.

Оба метода испытания дают однородную картину, хотя потенциалостатический метод является более информационным. Техника полезна как дополнение к визуальным испытаниям и подает надежду более широкого понимания механизма при помощи которого краски контролируют влияние коррозии.

Introduction

Polarisation techniques involve the application of external currents, and they have been widely used in recent years for studying corrosion processes and for forecasting the likely course of corrosion of bare metals. The author has applied these techniques to the study of painted steel. There are a number of ways in which polarisation curves can be obtained, and these are well summarised, and the nomenclature is discussed, in a paper by Pourbaix and Vandervelden¹. The author² initially used a galvanostatic technique (Pourbaix prefers the term "intensiostatic"). More recently, use has been made of a potentiostatic technique (or, more correctly, a potentiokinetic technique). In both methods the painted panel being studied is immersed in a corrosive solution, and the potential difference between the specimen, and a reference half-cell immersed in the solution, is recorded whilst the panel is subjected to a polarising current from a large platinum electrode also immersed in the solution.

In galvanostatic testing, the polarising current is kept constant, by including a high electrical resistance in the circuit, and the potential of the specimen is plotted against time. If there is little change in potential when the specimen is made anodic, it is assumed the current can be readily utilised by the normal anodic reaction, i.e. iron ions can go into solution. Similarly, if there is little change in potential when the specimen is made cathodic, it is assumed the normal cathodic reaction, i.e. the reduction of dissolved oxygen, can readily take place. In the absence of evidence of control of either cathodic or anodic processes, the corrosion rate must be controlled by the internal and external ohmic resistance between the anodic and cathodic areas.

Originally, an oscilloscope was used to display the galvanostatic curves. This worked very well, and will continue to be used because of the advantage that the 35mm paper records can be mounted to give a convenient record of the experiments. A high-impedance pen recorder, purchased for the potentiostatic work to be described, has also been used, in conjunction with a six-cam timer, to control the polarising cycle and to trigger the electronic time base of the recorder. The galvanostatic curves reproduced in the present paper have been obtained with this apparatus.

In potentiostatic testing the polarising current is adjusted, preferably by a potentiostat (of which more will be said later), so that the specimen potential is gradually changed through a predetermined range. The resultant curves are

plots of current against potential. If only a small current is required to maintain a potential of, say, $+0.6$ v.h.s., it is assumed that there is considerable anodic inhibition. Similarly, if only a small current is required to maintain a negative potential of, say, -0.6 v.h.s., it is assumed that there is considerable cathodic inhibition.

The apparatus for potentiostatic testing, and the comparative differences between the results obtained by galvanostatic and potentiostatic methods, will be discussed in detail.

Apparatus for potentiostatic testing of painted specimens

As in the previous paper, the painted panels were examined after they had been subjected to salt spray or immersed in sea water for various lengths of time. During the test itself they were immersed in a glass tank containing 5 l of sea water or ammonium salts solution, according to their previous environment. The tank was provided with a liquid bridge connecting a silver/silver chloride half-cell and, in the case of scratched panels, terminating close to the scratch. A large platinum electrode was used for the supply of the polarising current.

The electrical apparatus as shown in Fig. 1 can be divided into three parts:

- (1) a programme unit which determines the range of potential over which the test is to be made.
- (2) a potentiostat which supplies the necessary current to maintain the specimen at the programmed potential.
- (3) an X-Y recorder which automatically plots a graph of current against voltage.

An added refinement is a voltage scale, similar to that described in the earlier paper, which enables calibration lines on the hydrogen scale to be recorded.

The essential part of the programme unit is potentiometer C driven by a reversible motor. By running the motor forward, or in reverse, the programme potential can be moved in the anodic direction or cathodic direction. Two auxiliary potentiometers, A and B, enable the range of the potential to be selected. The mechanical drive is via a gear box which enables the time of polarisation to be varied. Limit switches are provided so that the motor stops when the slider reaches either end of the potentiometer.

A potentiostat can be regarded as a sensitive proportional relay. When no potential is applied across the input, no potential appears at the output, but when a negative or positive potential is applied to the input an appropriate negative or positive potential appears at the output. The term "potentiostat" was first used by Hickling, who has described a practical instrument, and this and several other instruments are available commercially. Hickling's design has been strongly criticised by Shaw and Edwards³, who describe a transistorised instrument. They also give a circuit for a valve potentiostat based on a design by Roberts. This modified Roberts potentiostat was built and has proved very successful. It gives a maximum current of 30mA and a current of 5mA for an input of 30mV. The limited output is an advantage when the instrument is used with painted specimens, since it decreases the possibility of accidental damage.

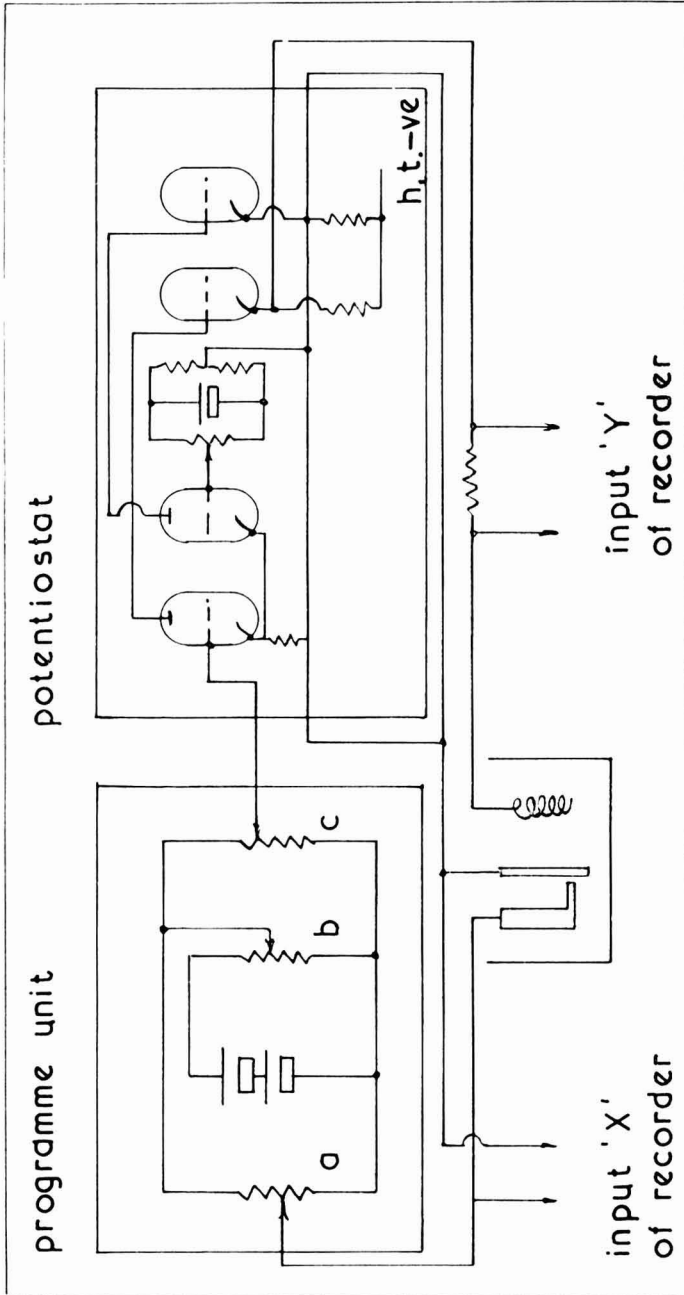


Fig. 1. Outline circuit of apparatus for potentiostatic testing of immersed specimens

The methods of carrying out polarisation tests and the interpretation of the results

Polarisation tests on *bare* metal specimens are carried out over long periods of time, often several hours, with the object of identifying inflexions of the curves with particular reactions. There is the obvious danger that, if this procedure is

followed for painted specimens, the normal life of the paint film might be reduced. As a quantitative indication, if the cathodic current was used entirely in releasing hydrogen, a current of 2mA for one minute releases 12 cubic millimetres of gas which might well be supposed to initiate blistering. It is necessary, therefore, to keep the current, and the period of time for the test, as small as is possible although currents of 2mA are only required when breakdown is moderately advanced and release of hydrogen is less likely to cause damage. High currents could also damage the film by generating alkali.

In the earlier galvanostatic tests, cathodic polarisation was applied for 24 seconds followed by anodic polarisation for 24 seconds. This procedure is still being followed, although it has been found worthwhile also to make tests in which the anodic curve precedes the cathodic one. The total current may be as low as 5 microamps for an 8in \times 4in panel with a complete paint system, which has only been immersed for a short period, to 5 milliamps for a specimen showing advanced breakdown. The current is kept constant, of course, for any given series of tests. One hour is allowed between cathodic/anodic and anodic/cathodic tests.

The selection of the method of carrying out potentiostatic polarisation tests is much more difficult. Whilst in the testing of bare metals, it is usual to start either at the most negative potential and gradually to increase the potential to the most positive value, or *vice versa*, such techniques were considered to be dangerous and the practice has been to start the tests with the programme potential (as indicated by a voltmeter temporarily placed across the programme unit) at 0.7 volts. This is equivalent to a potential on the specimen of about -0.4 volts on the hydrogen scale, which is about the resting potential of a painted specimen in a corrosive solution. The motor is then started and allowed to run until stopped by the limit switches or until the current becomes excessive. The motor is then reversed and again allowed to run until the other limit switch operates.

Usually two cycles, i.e. cathodic/anodic/cathodic/anodic, are made, followed not less than one hour later by anodic/cathodic/anodic/cathodic.

The programme potentiometer is usually driven at 1 rpm, which means that about 1min 26sec is required for one cycle. Alternatively, a shaft speed of 1 revolution in 13min is available, when 19min is required for one cycle.

A comparison of results obtained by galvanostatic and potentiostatic testing of painted specimens

With the object of illustrating this paper, four iron oxide in long oil alkyd medium primers were prepared. The first, No. 852, contained no inhibitive pigment. The other three, Nos. 853, 854 and 855, contained 15 per cent by weight of the pigment in the form of anti-corrosive pigments. Two or three panels of each system were evaluated in a number of ways, and galvanostatic and potentiostatic tests near to the end of the exposure will be shown.

Intact primer only; panels subjected to ammonium sulphate spray

Duplicate panels painted only with the primer were placed in a continuous salt spray cabinet, the salt spray consisting of 3.5 per cent ammonium sulphate

and 0.25 per cent ammonium chloride as recommended by J. B. Harrison⁴. To make the test, the specimens were immersed in a solution similar in composition to the spraying solution for $\frac{1}{2}$ to 2 hours before applying polarisation.

The galvanostatic curves are shown in Fig. 2, where it will be seen that there is good agreement between the duplicated panels.

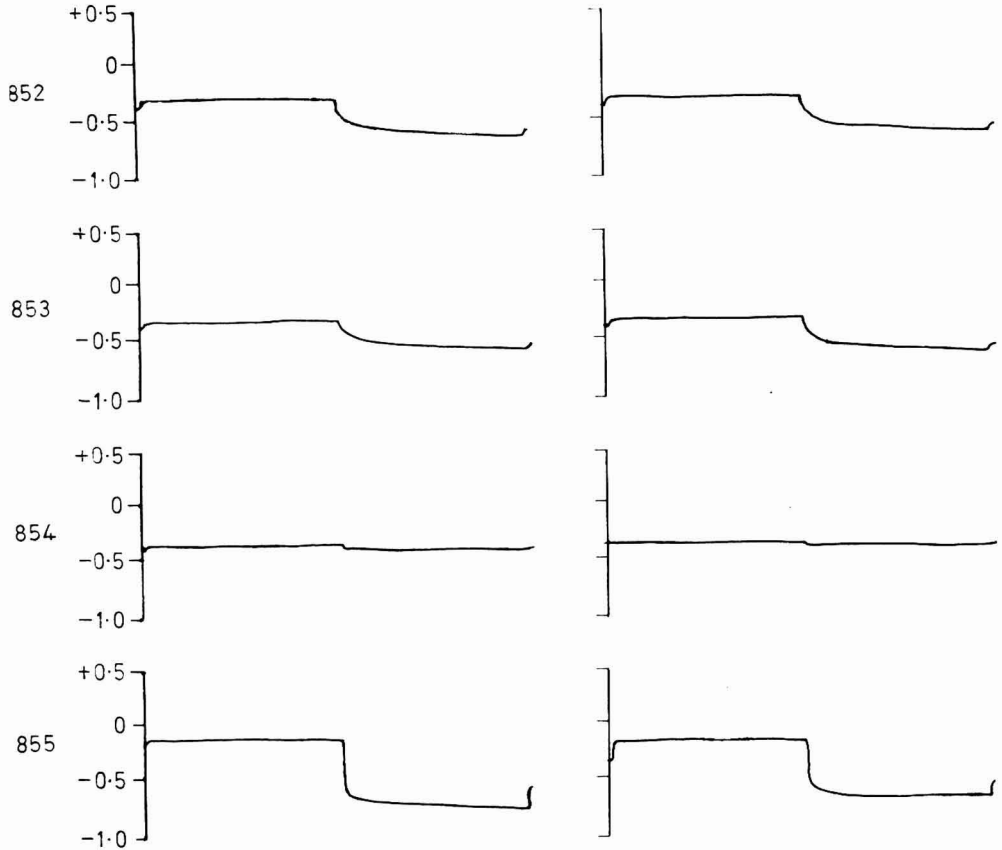


Fig. 2. Galvanostatic tests on intact primer-only panels which have been subjected to 81 days ammonium salts spray. Anodic tests before cathodic tests. Current 3mA

The bottom pair appear to be the best, the third pair the worst, and the first and second pairs intermediate in behaviour. It will be seen that the control appears to be largely cathodic. Tests made with cathodic current applied before anodic current were similar except that the anodic curves were asymptotic, suggesting that some reduction of the surface had taken place in the preceding cathodic polarisation.

These curves correctly represented the breakdown of the panels, as can be seen from Fig. 3, which shows the condition of the panels when withdrawn from the test a few days later.

One of each pair of panels has been stripped. There was no pitting of the fourth panel showing the smallest area of attack, indeed, the deepest attack

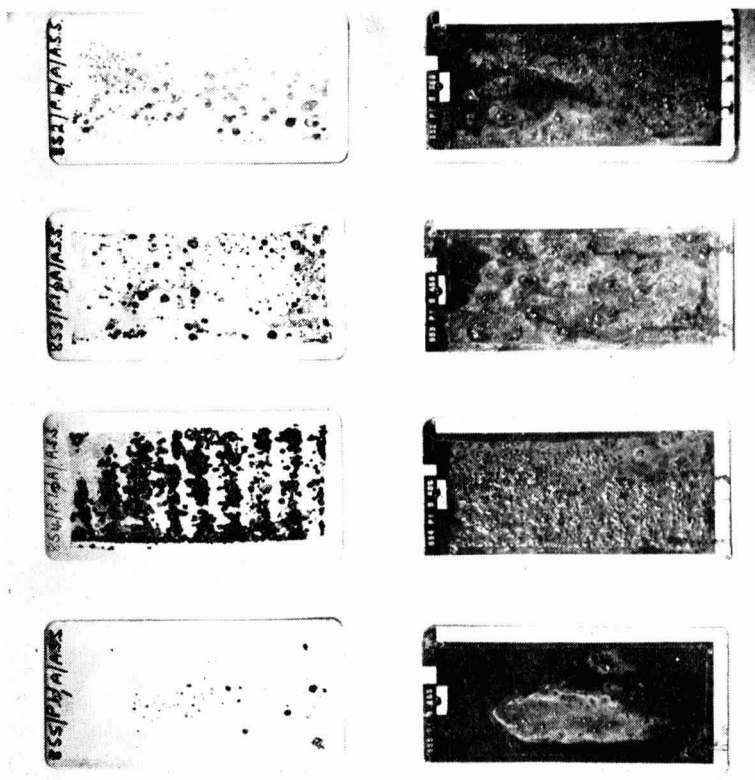


Fig. 3. Panels withdrawn at the end of the ammonium sulphate spray test. Those on the left have had the paint film removed

was on the third panel. Pitting is attributed by Evans to incomplete anodic inhibition in the absence of cathodic control, and the freedom from pits on the fourth panel can be attributed to effective cathodic control of the corrosion current.

Potentiostatic tests on these panels are shown in Fig. 4. Again, the curves give a correct indication of the breakdown. In interpreting the potentiostatic curves, it appears that the significant part of the curve is that near the resting potential. The third primer shows that a considerable current is required to raise or lower the potential from the resting potential, i.e. there is little anodic or cathodic inhibition of corrosion. On the other hand, the fourth set of panels required relatively little current to raise or lower the potential and it is assumed that neither the cathodic nor anodic processes could readily take place.

Two cycles of polarisation have been applied and, in the case of the fourth primer, these are identified by the numbers 1 and 2. It will be seen that the cathodic curves are almost coincident and, except in the case of the third set of panels, there is little hysteresis. All the anodic curves show hysteresis and, particularly in the case of the fourth set, there is a big difference between the first and second anodic curves. It is believed that, in the case of the last set of

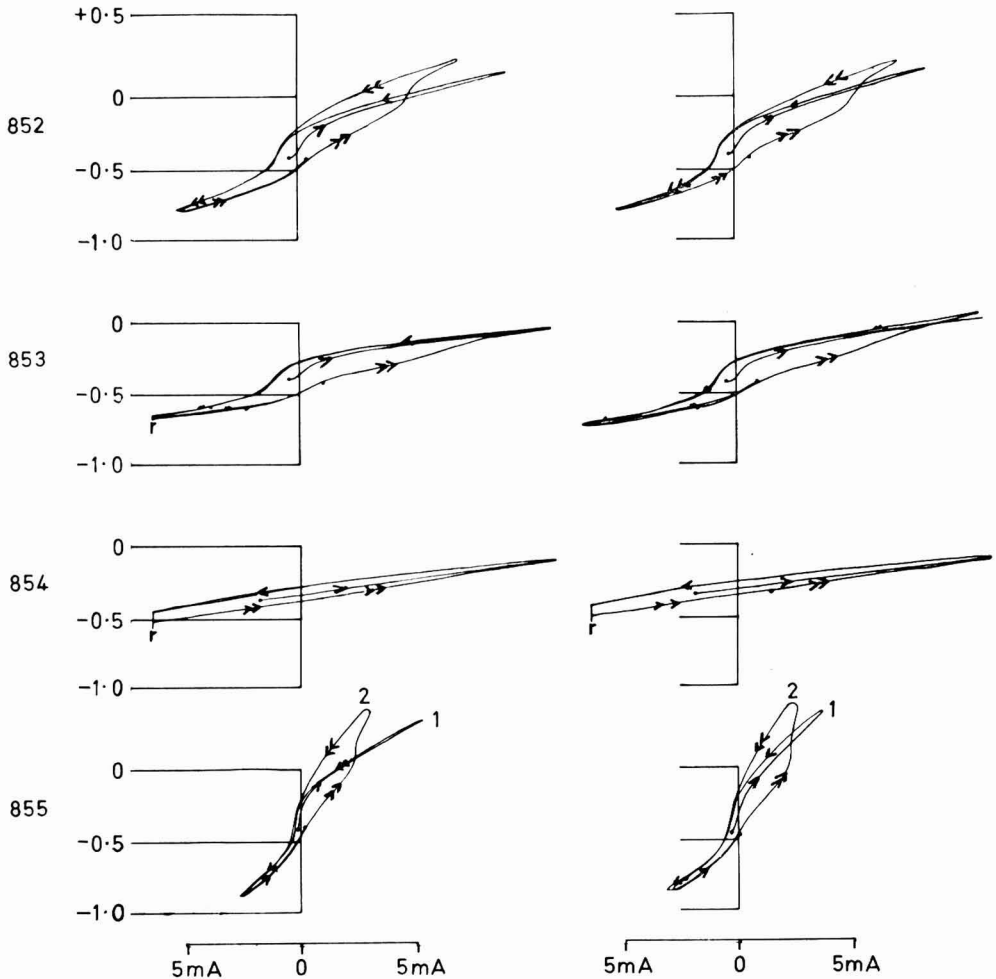


Fig. 4. Potentiostatic tests on intact primer-only panels which had been subjected to 81 days in ammonium salts spray. Sequence: anodic/cathodic/anodic/cathodic

panels, the metal surface is in a highly oxidised state, and that this is subsequently reduced by the cathodic current. Consequently, the greater current observed in the second anodic curve is due to re-oxidation of the surface.

Fig. 5 shows potentiostatic tests in which cathodic polarisation was made before anodic polarisation. There is relatively little difference between the first and second curves in these tests, and they agree quite well with the second curves in the tests made with anodic polarisation preceding cathodic polarisation. There is a suggestion from these curves that, whilst the cathodic currents reduce the oxidised surface, the anodic current does not give rise to a more highly oxidised surface than that present at the start of the test.

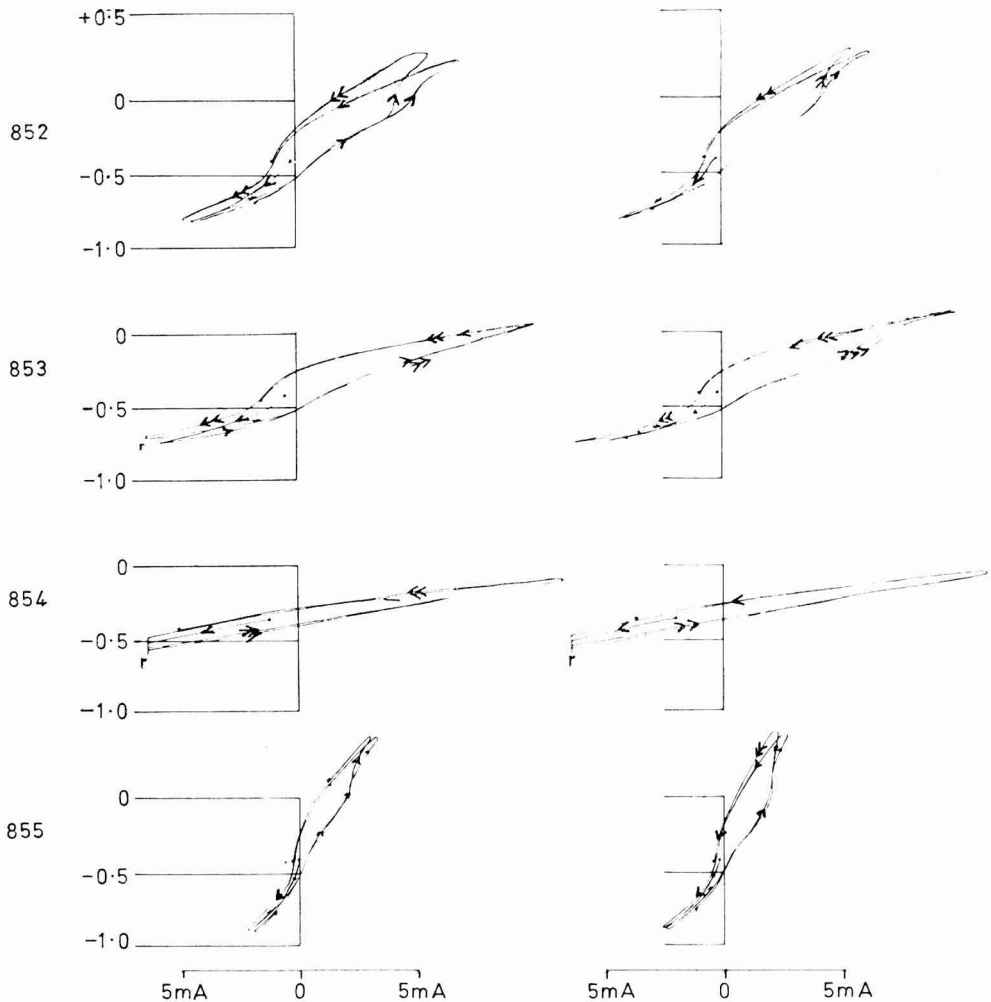


Fig. 5. Potentiostatic tests on intact primer-only panels which had been subjected to 81 days in ammonium salts spray. Sequence: cathodic/anodic/cathodic/anodic

Scratched primer + finish panels immersed in sea water

Triplicate panels coated with primer and finish were scratched, the scratch being 40mm long and 1mm wide. The panels were immersed in sea water and galvanostatic tests are shown in Fig. 6.

During the life of these panels, two of each set (the first and third as shown) were subjected to a large number of polarisation tests, many more tests than would normally be applied, since this was during a period when work was being done on the development of the potentiostatic technique. The third panel (i.e. the centre panel as shown) was only tested at the end of the period of immersion. There is no evidence from this series of tests that the frequent polarisation testing has damaged the panels.

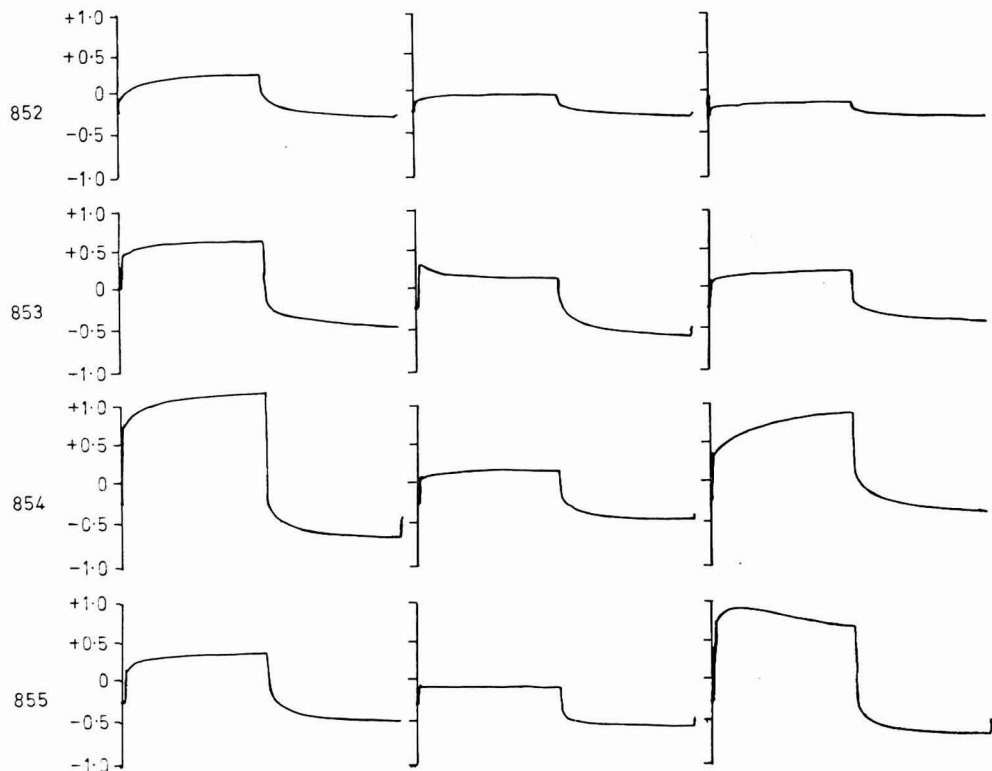


Fig. 6. Galvanostatic tests on scratched primer + finish panels immersed in sea water for 86 days. Anodic tests before cathodic tests. Current 2.5mA

There is no doubt that the first primer has given the worst performance, and the third primer, showing very good anodic polarisation with the first and third panels and good cathodic polarisation in all three, appears to be the best primer.

Galvanostatic tests in which the cathodic current was passed before the anodic current are shown in Fig. 7. These give a rather different picture. Whilst the first panel is still the worst, on balance, the fourth panel is the best, showing rather more cathodic polarisation on all three panels than the third primer.

Visual examination of the stripped panels confirms that the first primer is the worst and shows the fourth primer to be very distinctly the best. This suggests that, in this case, the cathodic curves are more important than the anodic ones. The first panel of the fourth primer was less good than the other two.

Whilst the third primer showed about the same attack on the scratch as the fourth primer, there was extensive but light filiform corrosion. This filiform corrosion was particularly noticeable on the first panel in spite of the excellent polarisation curves.

The first primer showed much more attack on the scratch than the other three.

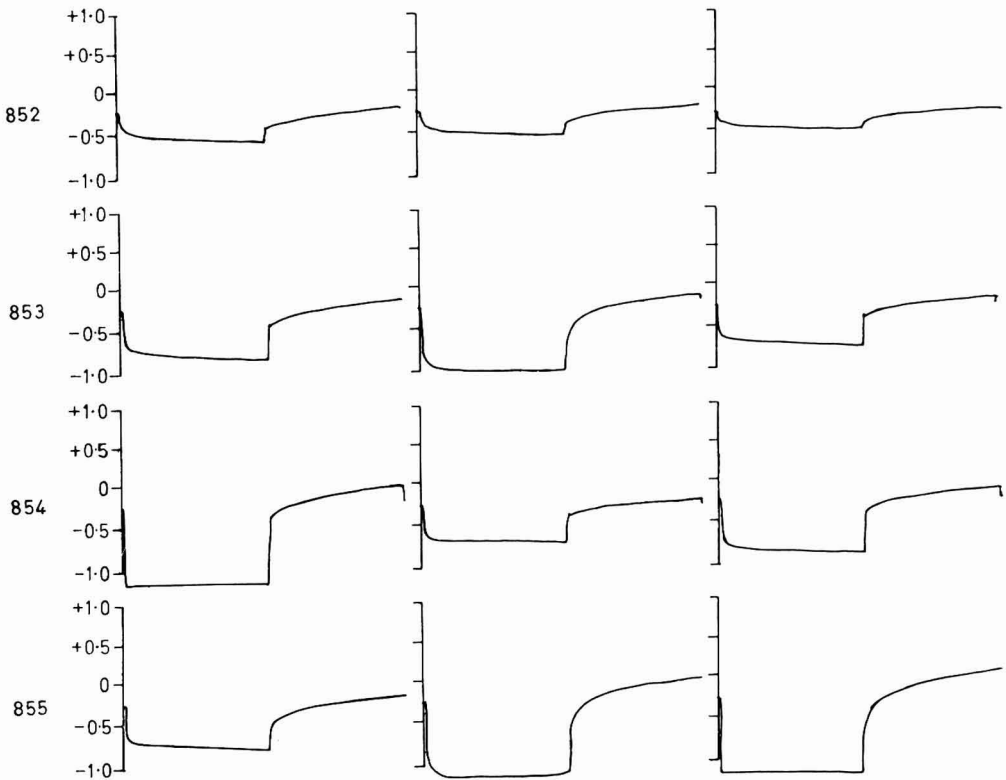


Fig. 7. Galvanostatic tests on scratched primer + finish panels immersed in sea water for 86 days. Cathodic tests before anodic tests. Current 2.5mA

Potentiostatic curves of these scratched primer + finish panels are shown in Fig. 8. Again, the first primer is much the worst and, again, an exaggerated value is placed on the first specimen of the third primer. It appears that both galvanostatic and potentiostatic tests are indicative of the conditions at the scratch rather than of the conditions "under the bulk of the paint film."

If one accepts that the important part of the polarisation curve is that near the resting potential, one correctly assumes that the second and third panels of the fourth primer are extremely good.

Intact primer + finish panels immersed in sea water

Triplicate panels were also prepared of *intact* primer + finish systems and immersed in sea water. Again, one panel in each set—the centre one in Fig. 9—was only subjected to polarisation testing at the end of the period of immersion.

This time, the panels which had not been previously subjected to polarisation testing are showing much more polarisation than the others. This difference, however, is not reflected in the visual appearance of the panels before or after stripping; indeed the middle panels of the third and fourth primers show more attack than do the panels which have been previously polarised.

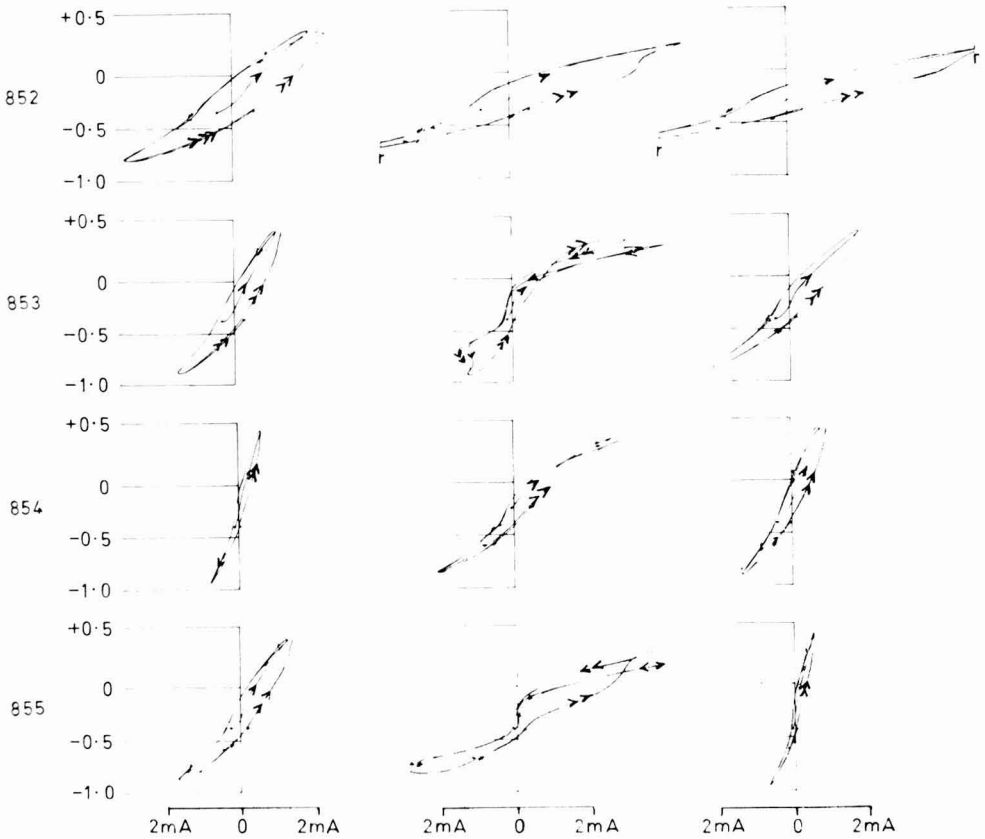


Fig. 8. Potentiostatic tests on scratched primer + finish panels immersed in sea water for 86 days. Sequence: anodic/cathodic/anodic/cathodic

Potentiostatic tests (Fig. 10) also showed more polarisation with the panels not previously subjected to polarisation. If one disregards the second panels, the potentiostatic curves are a good indication of the performance, 853c and 855a showing no breakdown when stripped, and primers 853 and 855 being superior to 854 and 852 which both showed light but extensive attack.

Of course, one cannot ignore the big differences in the curves of the middle panels, and, so far as intact panels are concerned, it appears that whilst the polarisation tests do not affect the life of the paint film, they can produce some change which influences later polarisation curves. A possible explanation for the effect is that, in the early stages of breakdown, the path of the corrosion current is largely restricted to the paint film itself and to the interface between the primer and the metal. The polarising current must, however, pass through the film. Supposing that, under the influence of the anodic polarising current, Fe^{2+} ions move through the paint film towards the solution, and, under the cathodic current, Na^+ ions move towards the metal, the result would be increased conductivity of the film, and the potential difference between the panel and the solution would more closely approximate to the potential at the metal surface. This increased conduction would not necessarily have much

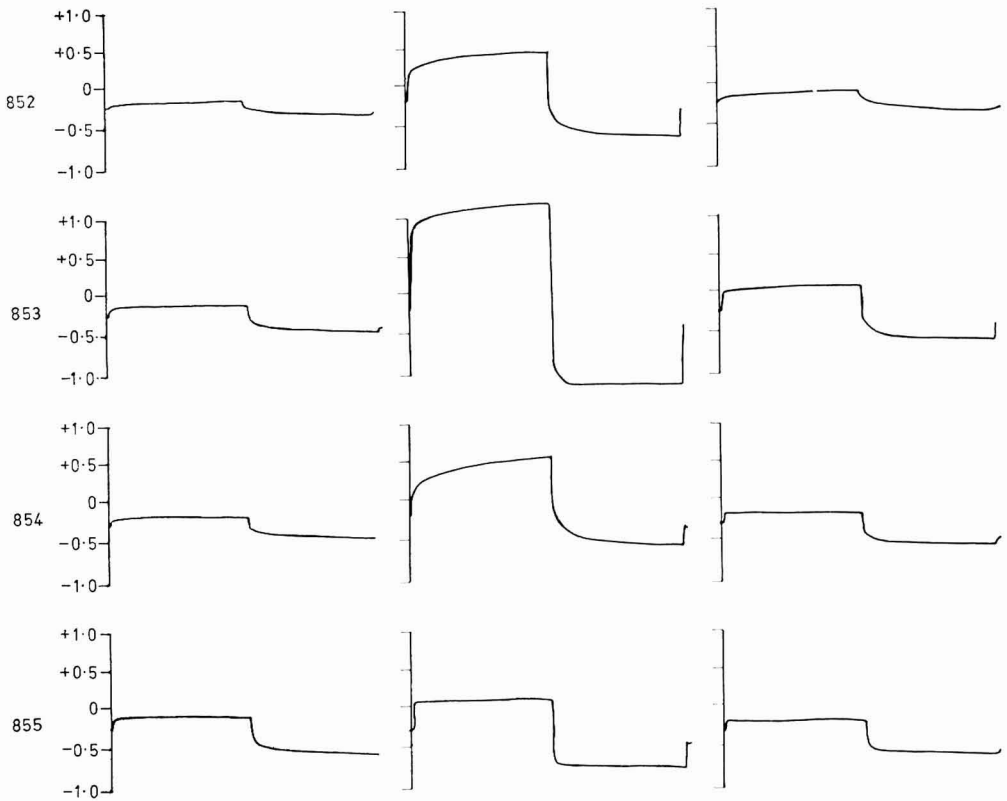


Fig. 9. Galvanostatic tests on intact primer + finish panels immersed in sea water for 93 days. Anodic tests before cathodic tests. Current 0.5mA

effect on the resistance between anodic and cathodic areas, and would not necessarily affect the rate of breakdown.

Conclusions

In general, galvanostatic and potentiostatic polarisation tests on painted specimens give similar pictures, although the potentiostatic method is rather more informative, particularly in providing information on the state of oxidation of the panel surface.

Polarisation methods give a correct indication of the weight of iron being corroded when scratched specimens are tested, but, if the paint film is still in good condition, the test will indicate the condition at the scratch rather than under the paint film.

Polarisation methods give a good indication of the rate of corrosion of unscratched primer-only panels, providing breakdown of the film has commenced.

At the present stage of development, tests on intact primer + finish panels are not completely reliable. In any given set of experiments, all the panels must be subjected to similar periods of polarisation, not because the test accelerates breakdown, but because prior tests can affect subsequent tests.

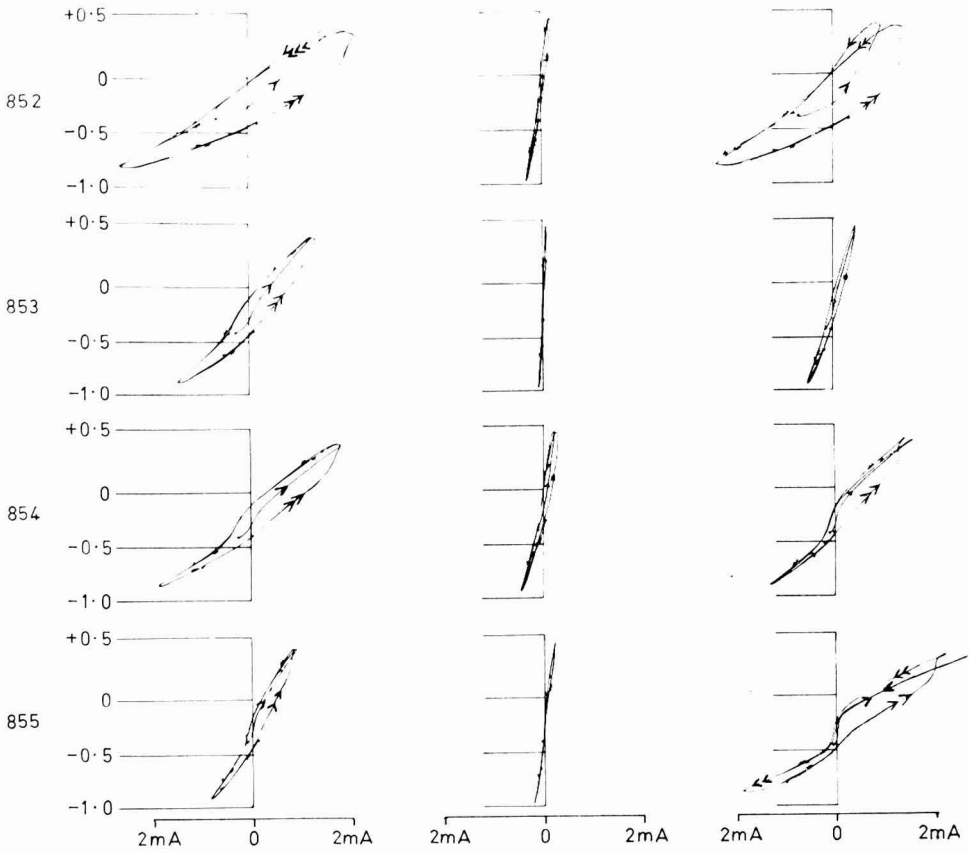


Fig. 10. Potentiostatic tests on intact primer + finish panels immersed in sea water for 93 days. Sequence: anodic/cathodic/anodic/cathodic

Acknowledgment

The author thanks the Directors of Cromford Colour Co. Ltd., Matlock, for permission to publish this work.

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Reviews

ANNUAL REPORT 1966-67, Regional Research Laboratory, Hyderabad

The Regional Research Laboratory, Hyderabad, is one of the constituent national laboratories of the Council of Scientific and Industrial Research, New Delhi. Like that of some well known institutions in other countries, its work is centred on raw materials and industries which are primarily regional, and this reviewer was surprised at the wide range of topics which this embraced.

Extensive studies are reported on oilseed constituents, oil analysis, evaluation of keeping quality, the synthesis of fatty acids, fatty acid derivatives and phospholipids. The emphasis placed on castor is not surprising in view of its importance to the Indian national economy.

In the general field of coatings, studies have been undertaken on the production of polymer raw materials from castor oil, the use of kamala seed oil, fish oil and cashew nutshell liquid, the synthesis of phenolics from indigenous raw materials, and the manufacture of barium potassium chromate. Basic physico-chemical work has been done on unsupported films, the corrosion of metals and adhesion. An aluminium/zinc oxide primer has been developed to replace imported red lead primer, and other coatings, developed in the Laboratory, are expected to find national outlets following the leasing of CNSL products to industry.

Some of the other activities reported are concerned with handmade papers (the Report itself is printed on a handmade paper produced in the Laboratory), entomology (especially insect control), biochemistry with special reference to the metabolism of moulds, low temperature carbonisation and gasification of coal, active carbons, microwave ceramics, crystallography, instrumentation and chemical engineering.

A. R. H. TAWN

CONCEPTS IN CATALYSIS

By ERIC K. RIDEAL. London: Academic Press 1968. Pp. 194, price 50s.

With the explosive growth of scientific study in recent years, one finds that specialisation is a must. Achieving a thorough understanding of a subject requires an increasing amount of time; as a result there is a natural narrowing of the subjects one is able to cope with. At one time heterogeneous catalysis fell within the province of the surface chemist, and previously surface chemistry within that of the ubiquitous physical chemist. Now the specialist in heterogeneous catalysis has little time to keep in touch with other aspects of surface phenomena.

This general trend is reflected in the production of textbooks which fall into two categories. One is a general text, dealing with the broad front of one discipline or sub-discipline, laying the foundation of understanding from which the student may develop a speciality. The other type is a text written by specialists, for specialists, giving an authoritative and up to date account of the current position in a particular field, with extensive bibliographies.

This short book falls into neither of these categories, and it is difficult to see whom it is aimed at. In his introduction, the author says that personal reasons

led him to portray some aspects of the subject (heterogeneous catalysis) which have impressed him. The result is therefore naturally a highly subjective text, in which the author briefly considers many topics, grouped under eight broad chapter headings. These range from General Properties of Catalysts, and Condensation and Evaporation, through Thermal Changes and Reaction Kinetics, Catalytic Oxidation and Hydrogenation, to Catalysis in hydrocarbons and Organic Syntheses and Decompositions. Each chapter is subdivided into short sections, typically of two or three pages, each dealing with specific reactions or topics. For example, the chapter on hydrogenation covers, The Dissociation of Hydrogen; Adsorption of Hydrogen; The Ortho-Para and Deuterium Exchange Reactions; The Synthesis of Ammonia; Catalysis at Electrodes; and Oxy-Redox Catalysis.

The treatment of each topic is often very short and superficial, and yet the author presupposes a familiarity with the basic concepts of heterogeneous catalysis which goes beyond undergraduate level. However, one can not easily follow up any point of interest because of the complete absence of references.

A note on the dust cover declares the object of the book to be an indication of "the direction in which the subject is now moving." However, in some sections at least, the review is out of date by more than a reasonable amount. This is well illustrated by reference to the section, one-and-a-half pages long, devoted to sulphur dioxide oxidation.

Most of this section is concerned with the historical work in which a platinum catalyst was used, only a few lines are given to vanadium catalysts. It is inferred that stoichiometric gas composition is practical and used, whereas in fact it is highly impractical and never used in industrial contact processes.

The promoted vanadium catalyst gets a mention, in the section on the "Influence of Crystal Field." However, the reaction is still treated as a heterogeneous one involving the solid state. This mechanism became untenable when the active phase was shown to be liquid, over 20 years ago. It has been accepted for many years now that the reaction occurs in solution and is in fact homogeneous, under conditions prevailing in working catalyst.

The style of writing does not lend itself to easy reading, a fact which is aggravated by the lack of punctuation. Typographic errors are present, but not in unacceptable numbers, unless the punctuation faults are such.

I doubt if this book will be of value to those other than students of science history, for whom it may provide a latter day glimpse of a great man's view of his ever growing subject.

E. E. EMMETT

PRACTICAL EMULSIONS

Volume II—Applications. By H. BENNETT, J. L. BISHOP, and M. L. WILFINGHOFF. New York: Chemical Publishing 1968, Pp. vii + 204, price: \$13.

The presentation under one cover of formulations of emulsions used in a wide variety of practical applications could constitute a useful handbook. Its value, however, would depend on the formulations being truly representative

of products in the marketplace and on their being supported by critical comments on the significance of at least major variations in composition. This book provides no such comment, although it may be argued that this can be obtained through the literature references. This, of course, leads to the thought that there would be no problem in turning to the technical and trade literature directly without consulting this book as the first step.

Omissions in a book of this type are, perhaps, to be expected, but nevertheless it is surprising to find, for example, no references to nitro-cellulose emulsions for leather, to latex finishes for paper or to multi-colour emulsions for decorative purposes. The "water-paint" formulations have a quaint and sometimes historical aspect, and could hardly claim to be a starting point for development purposes. For emulsion paint formulation it would be more rewarding to turn to the publications of raw material suppliers. The section on emulsion polymerisation is equally no launching pad for development.

This publication may be of some value to a few people, but never to a level equating with its price of \$13.

M. E. D. JARRET

Information Received

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

It has recently been announced that **Unilever** and **Otto Aldag** of Hamburg have concluded an agreement under which the **Unilever NV** group will acquire Aldag's oleochemical factory at Hamburg as from 1 January 1969.

It is intended that a new company will be formed by Unilever to handle the sale of the products, which include distilled fatty acids, stearine, oleine and glycerine. Otto Aldag's trade in oils, fats and sundry chemicals will be continued by Otto Aldag independently.

In co-operation with the **Chemical Industries Association, ICI Ltd., Heavy Organics Division**, has recently announced the introduction of a pilot scheme to supply drivers of its road tankers containing chemicals with hazard warning cards.

On the outside of the card, which is contained in a wallet and has the driver's despatch documents stapled to it, is printed the name of the product, details of the hazards, protective devices required, action to be taken in case of accident, first aid measures and an emergency telephone number.

At present only road wagon tank loads are covered, but it is intended to extend the scheme to all bulk deliveries of hazardous materials.

Baker Perkins Holdings Limited has recently published "The history of Baker Perkins," by Augustus Muir, tracing the history of the company since Jacob Perkins' early experiments with weapons, and his company's contract to print the penny black.

The availability of two new non-toxic preservatives has recently been announced by **Bayer Chemicals Ltd.** The two new products are *Preventol A3*, an anhydrous preservative for use in oil varnishes, alkyd coatings and stoving enamels, and *Preventol D1*, a non-phenolic preservative for use in aqueous systems such as metal cutting fluids, emulsion paints, wax emulsions, glues and adhesives.

Also from Bayer is *Alkydal F 48*, a new binder for trichloroethylene coatings. *Alkydal F 48* is supplied as an approximately 55 per cent solution in trichloroethylene, for cold dipping.

The **Chemical Industries Association** has recently issued a third amendment sheet to the third edition of their booklet "Marketing containers." Copies are available from the CIA.

It has been announced that as from 1 January 1969 the name of **Hoechst Chemicals Limited**, together with certain other companies in the UK group, will become **Hoechst UK Limited**.

Compagnie Francaise Goodyear SA have recently published a booklet giving information on the use of *Pliolite* resins in masonry paints. The booklet, which is intended as a guide to architects contractors and painters, is available in this country from **Hubron Sales Ltd.**

It has recently been announced that a licence agreement has been reached between **Kunstharsfabriek Synthese NV**, a member of the Dutch KZO Group, and **BCL Coatings Ltd.**

Under this agreement Synthese obtained the right to manufacture certain polyurethane resins developed by **Cargill Inc.** of the USA, and to sell all Cargill polyurethane resins in the EEC countries (excluding Belgium) and in Finland. BCL acquired the right to manufacture certain alkyd and melamine resins developed by Synthese.

It was also agreed that all Synthese resins for the paint industry will be marketed in the UK by **Victor Blagden and Co., Ltd. Berk Limited** will continue to sell the full range of Synthese resins in the Republic of Ireland, and Synthese printing ink resins only in the UK.

The Building Research Station has recently issued *Information 68*, a select list of its publications and films. Copies are available free of charge from the BRS.

SODIP SA, a Belgium company, has recently issued details of their services in information retrieval.

The company has gathered, over a period of some years, a steady supply of information about the periodical press throughout the world. This information is available to the public, free of charge.

Up-to-date information can quickly be obtained on the subscription rate of any publication in sterling, and a list of publications covering any particular field is readily available.

Recently published by the **British Iron and Steel Research Association** are three brochures outlining some of its established services to industry. These are the Corrosion Advice Bureau, the Steel User Service and the Information Services. Copies of the brochures are obtainable on demand from BISRA.

The APV Company Limited has recently published for the first time a booklet giving details of the complete range of plant and services which the APV Group offers to the chemical processing and allied industries.

Williams (Hounslow) Ltd. has recently announced that its parent American company, **Morton International Inc.**, has acquired the outstanding shares of **Nederlandse Kleurstofindustrie**, manufacturers of food colours and aniline dyestuffs etc., and this company becomes a wholly owned subsidiary of Morton. It will operate within the Williams Hounslow group of companies.

A 32 page booklet has recently been issued by **Bush Boake Allen Ltd.** giving details of its range of essential oils, of which the company is claimed to be the world's foremost producer.

Copies are available from Bush Boake Allen on demand.

"New buildings in the UK as a market for paint" is the title of a report published recently by **Business Intelligence Services Limited**. Further details of the report, which is based on a detailed survey carried out over six months amongst manufacturers, specifiers, users and distributors of architectural paints, are available from BIS.

Section Proceedings

Hull

Dispersible organic pigments

The second meeting of the session was held on 4 November at the College of Technology, Hull. The Chairman, Mr E. A. Brown, introduced Dr F. M. Smith who gave a very well illustrated lecture on "Dispersible organic pigments."

Dr Smith demonstrated what he defined as dispersibility—the ability to form a stable dispersion with the minimum expenditure of energy in grinding, and showed that individual pigments behaved quite differently according to the fineness of the particle size in the dispersion. Some pigments gave better colour development as the particle size was reduced whilst others might become more transparent when the particle size became too small. Stability of dispersion was important, since lack of it could result in a change in colour and/or covering power; changes might occur in opposite directions, according to whether the dispersion increased due to further "wetting out" or the pigment flocculates, decreasing the dispersion.

It could not be claimed that dispersible pigments solved all the problems but they did have advantages in reducing the number of grinds that had to be made in paint, or ink, manufacture and they could be supplied also in a granular easily handled and easily dispersed form.

Dr Smith defined aggregates as particles in which the primary crystals adhered together by their faces and agglomerates where the crystals adhered together by their corners or edges. The latter were much easier to separate than the former. With aggregates no dispersion occurred until considerable mechanical work had been done and the particles would reaggregate unless something was adsorbed on the surface to prevent it. A number of electron photo-micrographs were shown to illustrate the forms of aggregates and agglomerates and the chain-like structures that could be formed on reaggregation.

When a dispersion was prepared it was necessary (1) to separate the particles and (2) to adsorb some component on the surface to avoid reflocculation, also (3) it should be realised that adsorption from a medium might denude it of the adsorbed fraction. In the case of dispersible pigments, steps (1) and (2) had already been performed and (3) did not arise because adsorption on the surface had already taken place.

Dispersible pigments were available in three forms.

1. Pourable liquids in which the pigment was really dispersed and here the continuous phase of the dispersion must be compatible with the medium to which it was added. Most dispersions of this type were aqueous for use with emulsion paints, but some non-aqueous dispersions were available.
2. Solid granular form containing sufficient dispersing and stabilising agents to make them readily dispersible with a high speed or similar mixer; such pigments could be dispersed in viscous ink media.
3. Chip form, in which the pigment was well dispersed, generally with the expenditure of a great deal of energy in a hot medium, which subsequently solidified and could be broken up.

Messrs Brown, Wynn, Gilroy, Mell, Wenham, Read, Gamon, Anderson and Dr Taylor took part in the discussion that followed the lecture. A vote of thanks was proposed by Mr L. W. Wynn. Twenty-four members and six visitors were present.

S.R.F.

Surface preparation

The third ordinary meeting of the section was held on 2 December at the College of Technology, Hull. The Section Chairman, Mr E. A. Brown, introduced Mr T. R. Bullett of the Paint Research Station who gave a lecture on "Surface preparation." Unfortunately Mr D. Dasgupta, the co-author, was unable to be present owing to illness.

The lecture closely followed the lines of one given previously to the London Section in January 1968, and which has been adequately described in *JOCCA* 1968, **51**, 371.

A discussion followed the lecture in which Messrs Brown, Read, C. C. Mell, Gay, Proudley, Lythgoe and Hasnip took part.

A vote of thanks to the lecturer was proposed by Mr P. J. Gay, and was cordially supported in the usual manner. Twenty members and two visitors were present.

S.R.F.

Manchester

Modern liquid inks for the flexible packaging industry

The first Liverpool meeting of the session was held on 13 December at the Liverpool Building and Design Centre. The Chairman, Mr W. F. McDonnell, introduced Dr H. G. Smith of Fishburn Printing Ink Co. Ltd., who delivered a lecture "Modern liquid inks for the flexible packaging industry."

Flexographic and gravure inks were defined as containing colourants, resins and solvents together with additives to improve transfer, the final product being at low viscosity. Flexographic printing from rubber stereotypes required inks to be free from solvents which could swell or distort the printing surface. Water or alcohols achieved this effect. Five types of flexographic inks were described:—dye (aniline), cellulose, polyamide, acrylic and water-based. Dye inks were used on wrapping papers. Polyamide inks were used on polyolefine films, for example, on sacks and food packages, since the resins were low melting, heat sealing must be in non-print areas and the prints could not be laminated. Acrylic inks gave good adhesion, particularly on pvc, and could be heat sealed; they were used on sausage wraps where extensibility was important and on "boiled-in-the-bag" food packages. Water-based inks were simple, non-flammable and were used on cartons and multiwall sacks.

Because gravure printing imposed no solvent restrictions, a wider range of resins could be used and a wider range of substrates printed. High quality prints could be obtained in long runs from permanent metal cylinders. The requirements for gravure inks were basically the same as for flexographic: good printability, adhesion, low toxicity, high solvent release, odourless prints, abrasion resistance; with resistance to deep freezing conditions, detergents and chemicals being additional for particular purposes.

Dr Smith commented that paper, film and foil could all be printed by gravure or flexography. About 65 per cent of liquid inks were for paper, 20 per cent for foil and 15 per cent for films, and the latter proportion was increasing at the expense of usage on paper. Printing on film could be on the surface, or on the reverse, the latter method being used for laminating. Laminated packaging using polyethylene as the inner layer was increasing in importance because the shelf life of the packaged product was increased.

The future trends in liquid ink applications were predicted by Dr Smith as; increasing use of films from hydrocarbon polymers, films which required no separate coating to be heat sealable, higher press speeds and the use of microwave heating, and the use of "electrostatic gravure" to improve ink transfer.

A lively and valuable discussion followed the paper, the vote of thanks was then proposed by Dr F. Dalton and carried with acclamation.

D.A.P.

Midlands

Instrumental colour matching

At a meeting of Midlands Section, held at Birmingham Chamber of Commerce on 15 November with Mr G. W. Whitfield in the chair, Mr R. P. Best gave a lecture on "Instrumental colour matching."

Mr Best said that colourant calculation techniques were based upon both objective and subjective aspects of colour. There were two types of problem: (a) to obtain an initial match as close as possible to a customer's sample and (b) to make subsequent batches within a given tolerance. The traditional craftsman's method of matching depended on inspired guesswork. Instrumental methods gave numerical values to parameters and so permitted computation. Computation could fail if pushed beyond its limits, if there was a wrong choice of instrumentation or if sampling was poor.

Instrumental techniques were based on the spectral reflectance curve. The plot of the ratio of reflectance against wavelength gave an unambiguous characterisation of colour, but this curve was of little direct use for matching because it could not be reproduced in practise. The CIE system, where the distribution of reflected light was observed in terms of red, green and blue, enabled colour to be examined in terms of retinal cones. An observer's brain integrated the stimuli from the retina.

In order to use computers to integrate tristimulus values, use was made of the Kubelka and Munk equation, which stated that the ratio of adsorption and scattering coefficients, K/S , was equal to $(1-R)^2/2R$ where R was the spectral reflectance. This equation was valid for textiles but was not so good for paints and was of no use for plastics. A modified equation where the term K/S was raised to the power α was found to give better results with plastics. By measuring mismatches on the CIE system and feeding the results into a suitably programmed computer, like COMIC, corrections were obtained in a matter of seconds.

A number of slides was then shown to illustrate various aspects of instrumental colour matching.

During the discussion period Mr Best ably answered a number of questions. Mr H. J. Griffiths proposed a vote of thanks.

L.R.S.

Newcastle

Fundamental aspects of durability in paint films pigmented with rutile titania

The second meeting of the present session was held on 7 November 1968 at the Royal Turks Head Hotel, Newcastle upon Tyne, when a paper entitled "Fundamental aspects of durability in paint films pigmented with rutile titania" was presented by Mr G. Willison and Dr W. Hughes.

Mr Willison began by stating that the use of newer paint media such as the thermosetting acrylics has demanded new types of rutile titania pigment to give the best durability. Such new pigments, which might be of the sulphate or chloride types, did not necessarily give good durability in the older types of medium. Chalk resistance was the traditional mark of good durability, but gloss retention was now often more important. He stated that accelerated methods must be used for assessing high-durability paint films, and discussed the merits of the various methods.

Mr Willison then introduced Dr Hughes, who went on to say that he proposed to deal only with the breakdown of systems pigmented with rutile titania, i.e., chalking systems, together with a study of UV radiation as the destructive agent.

Measurements were presented of the UV absorption spectra of several media and of rutile pigments. These were related to the rate of chalking through two coefficients,

- (1) the intrinsic rate of photo-degradation of unpigmented medium under standard conditions, and
- (2) the photo-catalytic activity of the rutile pigments.

A mathematical expression was deduced for the rate of chalking in terms of the UV absorption coefficients, and the pigment volume concentration.

The physics of sub-micron crystals and their surface was discussed in relation to photo-catalytic activity, and the role of defects given prominence.

A mechanism was presented for photo-catalysed oxidation at the rutile surface. This was based on the work of Stone at Bristol, and involved photo-adsorption of oxygen on active sites.

Dr Hughes concluded by saying that durability was affected by coating the pigment. The chemical and physical nature of the coating was described briefly, and the means of achieving super-durability outlined in principle.

Mr James closed the meeting by thanking the lecturers for an excellent paper, and was pleased to note that this was given by members of the Newcastle Section.

A.L.

Scottish

Fire retardant coatings

The November meeting of the Scottish Section was held on 14 November in the Whitehall Restaurant, Glasgow, when Mr F. C. Adams of the Ministry of Technology Fire Research Station spoke on fire retardant coatings.

Mr Adams opened by describing the successive stages of a fire inside a building from source of ignition through involvement of contents and compartment linings to "flash-over," where all combustibles were involved and life could no longer be sustained. The effect of the type of lining material in delaying "flash-over" and thus increasing the chances of escape was shown by a series of dramatic illustrations.

Comparison of this property of lining material was made by the Surface Spread of Flame Test (BSS476: part 1: 1953) which recorded the rate of flame travel over the surface of a 3ft long panel under specified conditions, and classified materials into groups from Class 1, surface of very low flame spread (e.g. plasterboard) to Class 4, a surface of rapid flame spread (e.g. hardboard). This classification was used in the Building Regulations 1965, and it was to be noted that Classes 3 and 4 had limited or no acceptability because of fire risk, and that this could eliminate many conventional board lining materials were it not for flame retardant coatings. Such coatings could reduce the rate at which flame would travel over the surface so that an improved class of performance was achieved.

Flame retardance could be obtained by impregnation with suitable chemicals, or by surface coating which could be either intumescent, forming a blanket of protecting foam, or of the "conventional" type giving a static protecting skin, which of course also had decorative properties. Little was yet known of the effects of multi-layering or overcoating. Mr Adams laid emphasis on the fact that classification was on a coated surface, and could not be applied to a coating material *per se*, since a particular

coating might protect a Class 3 plywood so that Class 1 was obtained, but only improve Class 4 fibre insulating board to Class 2. Also, coating thickness was critical, and should be carefully specified.

Mr Adams then went on to discuss a new test which measured the contribution of a lining material to fire when totally consumed, the Fire Propagation Test (BSS476: part 6: 1968). In this, the test sample formed one side of a rectangular box fitted with a heat source and a flue for exit gases, whose temperature could be measured. A time-temperature curve of these effluent gases was drawn while the test material was burnt. Careful calibration of the apparatus was most important. This test was particularly useful for low melting point plastics which could not be subjected to the Surface Spread of Flame Test, as they would not remain in position long enough under test conditions. Also, the apparatus was simple to construct and easy to operate, so that coating and lining manufacturers could easily carry out their own comparative tests, which was not possible with Surface Spread of Flame Test with its costly equipment and operating difficulties.

The nature of the products of combustion of lining or coating materials was also of great importance in helping or hindering escape and fire-fighting. Such products might have a toxic or lachrymatory effect, or the density of smoke arising might greatly reduce vision and cause panic. Neither of the tests so far described measured these properties, and new tests were being developed at the Fire Research Station, but the proposals were at an early stage. Mr Adams looked forward to a time when a smoke emission and toxicity test, the Fire Propagation Test and the Surface Spread of Flame Test would together give a fuller and fairer assessment of the potential hazards of building materials.

A vigorous discussion, in which many members took part, followed the lecture, indicating the interest aroused by the speaker. A number of specific coating and lining materials were discussed, and a possible "ignitability test" mentioned. Some problems in the interpretation of the Building Regulations and their marine equivalents were highlighted, when it appeared that many of these arose from the lack of a body of knowledge of the effects of overpainting with different materials and film thicknesses. Finally, a vote of thanks for a most interesting and stimulating lecture was proposed by Mr R. Gardiner and carried with acclamation.

J.D.E.

Chemistry in the Caribbean

For the last lecture of 1968, held in the Whitehall Restaurant, Glasgow, on 12 December the Scottish Section left workaday matters and the local climate behind, and accompanied Professor R. A. Raphael of the University of Glasgow on a visit to the West Indies. Last year Professor Raphael spent three months as Royal Society Visiting Professor on the three campuses of the University of the West Indies in Jamaica, Trinidad and Barbados, and his lecture "Chemistry in the Caribbean" described and illustrated his experiences.

After outlining the geography and history of the islands, Professor Raphael went on to describe the Jamaica campus of the University which was made up of strikingly modern buildings in a setting of unusual natural beauty. Opened in 1948 with 33 students, it now had a roll of about 2,000, and the main problem with these students was that of natural products isolated from the 14,000 odd plant species found in Jamaica, many of which had pharmacological activity. Barbados presented an interesting contrast to Jamaica both in the character of its people and the appearance of its urban building, including the university campus overlooking the tropical sea.

The lecture was enlivened throughout by Professor Raphael's perceptive and witty comments on the life and culture of the West Indies, and was illustrated with coloured slides of breathtaking scenery, a splendid climate, and the attractive towns and inhabitants of the islands, including some outstanding pictures of carnival in Trinidad.

Discussion after the lecture disclosed a strong feeling among the members that chemistry in Glasgow compared but poorly with that in the Caribbean. Thereafter Mr Rowley expressed the thanks of all for a most enjoyable evening and was supported with acclamation.

J.D.E.

President's cruise—first pictures

The full report will appear in the March issue

South Africa

During the course of their world cruise on which they embarked on 28 October 1968, Mr and Mrs Sowerbutts left the "Northern Star" at Capetown and flew to Durban, where they were met at the airport by Mr K. M. Englebert, a Vice-President of the Association, past Chairman and Section Treasurer.

chat on Association and Section Affairs. (See picture on page 184).

On the following evening, before the ship sailed, some Members went aboard to see the bound volume of illuminated addresses and congratulatory messages presented to the Association at the 50th Anniversary Celebrations in May 1968, which Mr Sowerbutts took on the cruise



Seen greeting Mr and Mrs Sowerbutts at the airport are Mr J. Boroky (left), Chairman, South Australian Section OCCAA, and his daughter Jane

They were entertained at a smorgasbord by the Chairman, Mr P. A. Draper and a number of Past Chairmen with members of the Committee and their ladies at the palatial Edwards Hotel, where the opportunity was taken for an informal

for the benefit of the Overseas Sections.

Australia

When the "Northern Star" docked at Fremantle at breakfast time on 26 November, the President and Mrs



South African Members and their ladies shown with the President are: (left to right) standing, R. Siebert, Publications Secretary, N. A. Brown, Committee Member, P. A. Draper, Section Chairman, H. A. J. Van der Merwe, Committee Member (Transvaal), K. R. McDonald, past Chairman, P. Gregson, Section Secretary, D. J. Pienaar, past Chairman, Mrs D. J. Pienaar, K. M. Engelbert, Vice-President, past Chairman and Section Treasurer. Seated: Mrs N. A. Brown, Mrs H. A. J. Van der Merwe, Mrs P. Gregson, Mrs F. Sowerbutts, the President, Mrs F. Schollick, Mr F. Schollick, past Chairman, Mrs P. A. Draper, Mrs G. J. Kidd

Sowerbutts were met by Mr A. James, Chairman of the West Australian Section of OCCA Australia and Mrs James, complete with presentation spray of roses, who had that morning driven 120 miles from their home at Bunbury to welcome them. Following a day's sight-seeing in and around Perth, including some of the neighbouring bush country, Mr and Mrs James, with two other Committee members and their ladies, were hosts at a very jolly dinner party, unfortunately depleted in numbers owing to the arrival of the "Northern Star" two days behind schedule.

The next day the President and Mrs

with Members of the Section and their wives, at the Hilton North Motel, North Adelaide, gave an opportunity for discussion of topics affecting the two Associations when much interest was shown in the 50th Anniversary scrolls. In a brief address Mr Sowerbutts conveyed the good wishes of Council for the success of the Australian Association.

The following day the hosts had arranged a tour of the Barossa Valley, South Australia's wine growing area, and visits to Gramp's winery at Tanunda, as well as places of interest in and around Adelaide.



The President (front left) showing the Commemorative scrolls to members of the South Australian Section of OCCAA. (Back row left to right) Mr J. Boroky, (Chairman), Mr D. Turnbull, Mr D. Bruce (Past Chairman), (front row, right) Mr V. Costello

Sowerbutts flew to Adelaide, where they were welcomed at the airport by Mr J. Boroky, Chairman of the South Australian Section of OCCA, whose daughter Jane presented Mrs Sowerbutts with a bouquet of magnolia. With them also was Mr R. Paddick.

In the evening an informal gathering

After a crowded schedule Mr and Mrs Sowerbutts, who were considered excellent ambassadors by the hospitable South Australians, left en route for Melbourne.

The photographs on pages 183 and 185 were taken by Mr R. Paddick, of Bell Publications Pty., Publishers of the Australian Paint Journal.

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JOCCA

TECHNICAL EXHIBITION

The Exhibition Committee reports that copies of the *Official Guide* to the Exhibition have now been despatched to all members of the Association and will shortly be sent, through the courtesy of the trade organisations, to each of their member firms. Copies have also been sent individually to chemists and technologists on the continent of Europe and, since a notable feature in recent years has been the large number of overseas visitors attending the Exhibition, interpreters (French, German, Italian and Spanish) will be present to help both visitors and exhibitors.

There will be 107 stands at the Exhibition, which takes place at Alexandra Palace, London N22, on the following dates:

24 March	15.00-18.30hr
25 March	10.00-18.00hr
26 March	10.00-18.00hr
27 March	10.00-18.00hr
28 March	10.00-16.00hr

There is an increased representation of overseas firms exhibiting on this occasion, either directly or through British associates, and the Exhibition covers a larger floor area than on any previous occasion. As before, the theme of the Exhibition will be the presentation of technical advances in those industries

supplying the paint, varnish, printing ink, linoleum and other allied industries. There will be no charge for admission or for the *Official Guide*, which gives details regarding each stand, plans of the Exhibition and general information concerning travel arrangements, etc.



Lord Kings Norton

Copies of the *Official Guide* can be obtained prior to the Exhibition from the Director and Secretary of the Association at the address shown on the front cover of this *Journal*.

Technical Education Stand

A feature of the Exhibition which has proved popular in the past has been the Technical Education Stand. Members of the technical colleges and the trade organisations covering the industries concerned have volunteered to staff the stand, and parties of senior science students will attend on the mornings of 25, 26 and 27 March, when they will be given a short introductory lecture, followed by a visit to the Exhibition.

Exhibition Luncheon

On the opening day an Exhibition Luncheon will be held at the Savoy Hotel, London WC2, when the Guest of Honour will be Lord Kings Norton, who will reply to the Address of Welcome by the President of the Association. Lord Kings Norton is the Chairman of the Council for National Academic Awards and President of the Institute of Marketing and the National Council for Quality and Reliability.

Officers of the following bodies have so far accepted the Committee's invitation to be present as guests at the Exhibition Luncheon:

The Presidents of:

- The British Plastics Federation.
- The Institute of Metal Finishing.
(Represented by a Past President).

News of Members

Mr A. K. Ames, an Ordinary Member attached to the Manchester Section, was appointed Chairman of the British Colour Makers' Association at its AGM on 6 November 1968.

Mr W. B. Cork, an Ordinary Member attached to the Hull Section, will be Vice-President, and Mr F. B. Mortimer-Ford, an Ordinary Member attached to the London Section, will be Hon.

- The Paintmakers Association.
- The Plastics Institute.
- The Royal Institute of Chemistry.
- The Society of British Printing Ink Manufacturers.
- The Society of Chemical Industry.
- The Society of Dyers and Colourists.

The Chairmen of:

- The British Colour Makers' Association.
- The British Standards Institute.
London Section of OCCA.
PIRA.
- The Surface Coating Synthetic Resin Manufacturers' Association.

The Directors of:

- The Paintmakers Association.
- The Paint Research Station.
PIRA.

The Master of the Worshipful Company of Painter-Stainers.

European Liaison Lecture

The London Section of the Association will be holding its European Liaison Lecture on Thursday 27 March 1969 at 18.00hr in the Alexandra Room, Alexandra Palace, London N22. The title of the lecture will be "Progress in electrodeposition in Europe" and the lecturer will be Dr Karl-Heinz Frangen. A licensed bar will be available after the lecture and an application form will be enclosed in each copy of the *Official Guide*. Any interested person can obtain a form on application to the Association's offices.

Treasurer, for the year 1968-69, and Mr G. E. Hillier, an Ordinary Member attached to the London Section, and Messrs P. Perkin and J. Smethurst, Ordinary Members attached to the Manchester Section, will serve on the BCMA council for the same period.

Mr P. Walsh, an Ordinary Member attached to the Midlands Section, has

been appointed Plant Manager at the new Four Ashes carbon black facility of Columbian International (Great Britain) Limited.

Mr D. G. Trickett, an Associate Member attached to the Midlands Section, who was previously Works Director at Four Ashes, has been appointed Deputy Managing Director of Columbian International, and will move to the company's head office at Hounslow.

Mr C. N. Taylor, an Ordinary Member attached to the London Section, has been appointed a Director of British Titan Products Company Limited, with effect from 1 January 1969.

Mr B. A. Richardson, an Ordinary Member attached to the London Section, has recently obtained larger premises for his consultancy. The new premises, which will allow more extensive activities, are at Penarth House, Otterbourne Hill, Nr Winchester, and the consultancy is now to be known as Penarth Research Centre.

Dr F. A. Askew, an Ordinary Member attached to the London Section, has been appointed a Director of the newly formed company Coates Brothers (Litho Plates) Ltd., a member of the Coates Brothers group.

Mr M. H. Davies, an Ordinary Member attached to the Manchester Section, has relinquished his appointment as Technical Director of the Lead Development Association to form a consultancy, Fowler Davies and Company, to advise industry on the control of toxic materials and other hazards to health.

Dr H. G. Rains, an Ordinary Member attached to the London Section, and a past member of Council, retired from his position as group technical controller of Berger Jenson & Nicholson Group Ltd., at the end of November 1968.

Institute of Corrosion Technology

The Yorkshire branch of the Institute of Corrosion Technology is to hear a paper by Dr J. B. Harrison, of Goodlass Wall & Co. Ltd., entitled "The prediction of anti-corrosion paint performance" at its meeting on 12 March.

The Institute has extended an invitation to any interested Member of the Association to attend the meeting, which will be held at 6.15 p.m. in Lecture Theatre B, Floor E, The University of Sheffield, Department of Metallurgy, St. Georges Square, Sheffield 1. Tea and biscuits will be served outside the Lecture Theatre at 5.45 p.m.

Symposium "Progress in pigments"

The Society of Dyers and Colourists, London Region, has organised a half-day symposium entitled "Progress in pigments" from 14.00—19.45hr on Friday 7 February in the Charing Cross Hotel, London WC2. There will be papers entitled "Developments in the chemistry of pigments" by Dr D. Patterson, of Leeds University, "Pigments, plastics and progress" by Mr D. Thornley, of ICI Plastics Division. "Pigments in printing inks" by Dr H. G. Smith, of Fishburn Printing Ink Co. Ltd. and "The pigment printing of textiles" by Mr J. Mitchell, of Walsden Bleaching and Dying Co.

For registration forms apply to Mr M. J. L. Fadil, Geigy (UK) Ltd., 42 Berkeley Square, London W1. Telephone 01-493 2131.

TNO Conference

The TNO Conference on "Surface coatings" is to be held on 27 and 28 February, in Rotterdam. Programmes and application forms have now been produced, and can be obtained on request from:

The Secretariat of the TNO Conference,
c/o Holland Organizing Centre,
16 Lange Voorhout,
The Hague,
Netherlands.

Register of Members

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

Ordinary Members

- BANBURY, DAVID EDWARD, 80 Albert Road, Grappenhall, Warrington, Lancs. (*Manchester*)
- BULOW, ULF, AB Wilh Becker, Fack, 10270 Stockholm, Sweden. (*Overseas*)
- CANTIN, MICHAEL CYRIL, Mauritius Paints Ltd., Port-Louis, Mauritius. (*Bristol*)
- CHANDLER, LESLIE, 2 North Close, Chigwell, Essex. (*London*)
- CHAPMAN, PAUL IAN JACKSON, 21 Ilminster Close, Barry, Glam. CF6 7BU (*Bristol*)
- CHILDS, DEREK LESLIE GORDON, 26 Gilders, Sawbridgeworth, Herts. (*London*)
- CONSTABLE, PETER JAMES, 69 Clarkhill, Harlow, Essex. (*London*)
- FORMANEK, LEOPOLD, Research Institute of Synthetic Resins and Lacquers, S. K. Nemanna, 1316 Pardubice, Czechoslovakia. (*Overseas*)
- GHANEM, NADIM A., Department of Polymers and Paints, The National Research Centre, Dokki, Cairo, U.A.R. (*Overseas*)
- HARBOTTLE, TERENCE, 10 Broadwood View, Chester-le-Street, Co. Durham. (*Newcastle*)
- JOHNSTON, RUTH MARIE, D & H, Color Centre, Davidson & Hemmendinger, Inc., 2857 Nazareth Road, Easton, Pennsylvania, 18042, USA (*Overseas*)
- KERRIDGE, JOHN CHARLES, ICI Limited, Heavy Organic Chemicals Division, Organic House, Billingham, Teesside. (*Newcastle*)
- LILAONITKUL, RAVI, 12/2 Sukumvit 68 Rd., Bangkok, Thailand. (*Overseas*)
- MONTGOMERY, COLIN WILLIAM, BASF United Kingdom Limited, PO Box 4, Earl Road, Cheadle Hulme, Cheadle, Cheshire. (*Manchester*)
- OLLIER, DAVID, 19 Arkwright Road, Marple, Cheshire. (*Manchester*)
- ORCEL, JEAN, 53 rue Jean Mermoz, 13 Marseilles 80. (*Overseas*)
- PERRY, JOHN LYONS PRESS, 54 Finchmoor, Harlow, Essex. (*London*)
- RODGERS, MARTIN, 76 Vicarage Crescent, Redditch. (*Midlands*)
- SEOW, KEE YING, c/o Torginol Paints Ltd., 10 Jalan Rajawali, Singapore 21. (*Overseas*)
- SHEIKH, SAEED M., 11 Amil Colony, No. 1, Karachi—5, W. Pakistan. (*Overseas*)
- SOBOL, HECTOR Z., Fuente de Piramides, 282 Mexico 10, D. F. Mexico. (*Overseas*)
- SPARROW, BERNARD WALTER PARKINSON, Corrofell, Yealm Road, Newton Ferrers, Plymouth, S. Devon. (*Bristol*)
- STOCK, BARRY STUART, Stylux Paints, 86-94 Ligoniel Road, Belfast 14, Ireland (*Irish*)

Student Members

- COBB, ROBERT, 58 Christchurch Residences, Lisson Street, London NW1. (*London*)
- DEAN, DAVID JAMES, The Walpamur Co. Ltd., Hollins Road, Darwen, Lancs. (*Manchester*)
- HANDFORTH, VERNON, 69 Longdale Drive, Hattersley, Hyde, Cheshire. (*Manchester*)
- HOWELL, DAVID, 31 Cleveland Road, Basildon, Essex. (*London*)
- WHATLING, ALLAN, 17 Montagu Road, Offerton, Stockport, Cheshire. (*Manchester*)

Forthcoming Events

Details are given of meetings in the United Kingdom up to the end of the month following publication, and in South Africa and the Commonwealth up to the end of the second month.

Monday 3 February

Hull Section. "Colour in the Home" by Mr Haines of Sissons Bros Ltd., to be held in the Bullock Lecture Theatre, Hull College of Technology at 7 p.m.

Thursday 6 February

Newcastle Section. "Protective Coatings on Gas Pipelines and Storage Vessels" by Dr J. T. Harrison of The Gas Council, to be held at the Royal Turks Head Hotel, Grey Street, Newcastle upon Tyne. at 6.30 p.m.

Friday 7 February

Thames Valley Section. Buffet Dance at "Great Fosters," Egham.

Monday 10 February

London Section. Joint Meeting with Colloid and Surface Chemistry Group, Society of Chemical Industry. "The Mechanism of Action of some Deflocculating Agents for Pigments in Paint Media," by Mr W. Black and Dr A. Topham of ICI Ltd. Paints Division, at 14 Belgrave Square, London SW1, at 6 p.m.

London Section, Southern Branch. "Modern Interior Decor" by a speaker from ICI Decorative Products Dept. to be held at the Keppel's Head Hotel, The Hard, Portsmouth, at 7.30 p.m.

Tuesday 11 February

West Riding Section. "Protective Action of Paint on Metals" by Dr J. E. O. Mayne of Vinyl Products Ltd. to be held at the Griffin Hotel, Boar Lane, Leeds 1, at 7.30 p.m.

Thursday 13 February

Scottish Section. "Phthalocyanine Pigments" by Mr R. H. Harper and Dr R. Pascoe of ICI Ltd. Dyestuffs Division, to be held in the Whitehall Restaurant, Renfield St., Glasgow, at 6.00 p.m.

Friday 14 February

Manchester Section. "Market Research," by Mr D. Clark of English China Clays Sales Ltd., to be held at the Manchester Literary and Philosophical Society at 6.30 p.m.

Saturday 15 February

Scottish Section-Student Group. Annual General Meeting, followed by Competitive Panel Game, to be held at the Lorne Hotel, Sauchiehall Street, Glasgow, at 10.00 a.m.

Tuesday 18 February

Thames Valley Section. "Polyurethanes in Surface Coatings" by Dr G. Mennicken of Fabenfabriken Bayer AG, to be held at the Royal White Hart Hotel, Beaconsfield, Bucks, at 7.00 p.m.

Thursday 20 February

Bristol Section. Annual Dinner Dance at the Mayfair Suite, Bristol Entertainment Centre.

Friday 21 February

Midlands Section. "Inorganic Surface Coatings" by Mr R. J. Cole of the Paint Research Station to be held at the Chamber of Commerce House, 75 Harborne Road, Birmingham 15, at 6.30 p.m.

Newcastle Section. "Ladies' Night" at the Five Bridges Hotel.

Wednesday 26 February

Scottish Section—Eastern Branch. Joint Meeting with the Society of Dyers and Colourists at the Carlton Hotel, North Bridge, Edinburgh, at 6.30 p.m. "Safety in Industry with particular reference to Toxicity" by Mr S. J. Silk, H.M. Chief Factory Inspector.

Thursday 27 February

Midlands Section—Trent Valley Branch. "Sintered Plastic Coatings," by a member of the staff of Aquitaine-Fisons Limited, to be held at Loughborough University, No. 2 Lecture Theatre, Edward Herbert Building, at 6.30 p.m.

Friday 28 February

Bristol Section. "The Design of Room Temperature Curing Acrylic Resins," by Mr A. G. North of Cray Valley Products Ltd., at the Angel Hotel, Cardiff, at 7.15 p.m.

Irish Section. "Corrosion Inhibiting Surface Coatings" by Mr R. B. Simon of the Institute for Industrial Research and Standards, to be held at the Clarence Hotel, Wellington Quay, Dublin, at 8.00 p.m.

Monday 3 March

Hull Section. "Recent Developments in Driers" by a speaker from Hardman & Holden Ltd, to be held in the Bullock Lecture Theatre at the Hull College of Technology, at 7 p.m.

Wednesday 5 March

London Section. Joint meeting with the Organic Finishing Group, Institute of Metal Finishing. "Insulation Coatings in the Electrical Industry," by R. Snadow of AEI to be held at Borough Polytechnic, Borough Road, London SE1, at 6.30 p.m.

Thursday 6 March

Newcastle Section. "Emulsion Paints: Instrumental Aids to Binder Selection," by Dr C. Bondy of Revertex Ltd, to be held at the Royal Turks Head Hotel, Grey Street, Newcastle-upon-Tyne, at 6.30 p.m.

Monday 10 March

London Section—Southern Branch. "Paints, Preservatives and Water Repellents' Treatments for Timber" by Mr P. Whiteley of the Building Research Station, to be held at the Keppel's Head Hotel, The Hard, Portsmouth, at 7.30 p.m.

Tuesday 11 March

West Riding Section. "Ethylene-grafted Emulsions," by Mr K. A. Safe of Vinyl Products Ltd., to be held at the Griffin Hotel, Boar Lane, Leeds 1.

Thursday 13 March

Scottish Section. "Acrylic Resins" by Mr R. H. E. Munn and Mr A. R. H. Tawn of Cray Valley Products, to be held in the Whitehall Restaurant, Renfield St, Glasgow, at 6.00 p.m.

Friday 14 March

Manchester Section. "Broad Trends in the Development of Building Assembly Systems" by Professor D. Harper of the Institute of Science and Technology, University of Manchester, at the Liverpool Building & Design Centre Ltd., at 6.30 p.m.

Saturday 15 March

Scottish Section—Student Group. "Modern Managerial Techniques" by Dr D. Cameron of the University of Strathclyde, to be held at the Lorne Hotel, Sauchiehall Street, Glasgow, at 10.00 a.m.

Wednesday 19 March

Scottish Section—Eastern Branch. "Annual General Meeting" followed by a selection of films from the library of Shell Chemicals Ltd., to be held in The Wee Windaes Restaurant, 142 High Street, Edinburgh, at 7.30 p.m.

Thursday 20 March

London Section. "New Resin Systems for use in Coil Coating Application," by Mr L. A. Tysall of Shell Research Ltd., to be held at the New Engineering Block, University College, London WC1, at 6.30 p.m.

Midlands Section, Trent Valley Branch. Joint Meeting with Derby and Nottingham Branches of the National Federation of Master Painters and Decorators. "Timber and its problems as applied to Painting," by Mr G. S. Hall of the Timber Research & Develop-

ment Association, to be held at British Rail School of Transport, London Road, Derby, at 7.00 p.m.

Thames Valley Section. "Pigments—Particular Reference to Printing Inks" by Dr F. M. Smith of Geigy, to be held at the Royal White Hart Hotel, Beaconsfield, Bucks, at 7.00 p.m.

Friday 21 March

Midlands Section. Newton Friend Lecture. "Design and Fashion Through Chemistry." "Contribution of Science to Design and Fashion" by Mr M. W. Alford of Courtaulds Ltd. "Palettes of Fashion"—Film, to be held at the Chamber of Commerce House, 75 Harborne Road, Birmingham 15, at 6.30 p.m.

Thursday 27 March

London Section. "Progress in electro-deposition in Europe" European Liaison Lecture, by Dr K. H. Frangen to be held in the Alexandra Room, Alexandra Palace, London, at 6.00 p.m. p.m.

Friday 28 March

Bristol Section. "Operational Research" by W. C. Henshaw of the Bristol College of Commerce, to be held at the Royal Hotel, Bristol, at 7.15 p.m.

Irish Section. "An examination of Aqueous Interior Semi-Gloss Paints and the Development of an Aqueous Semi-Gloss Vehicle" by Mr J. H. Sparrow of Charles Lennig & Co. Ltd. preceded by the Annual General Meeting at 7.30 p.m.

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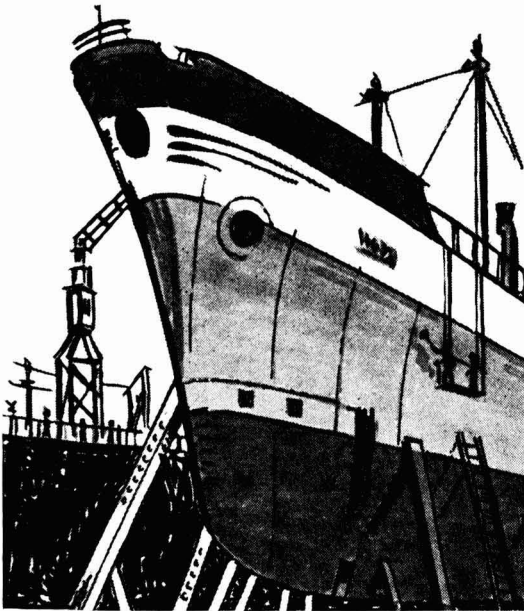
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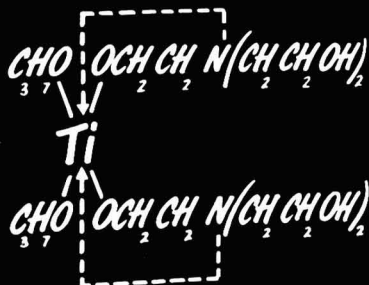
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