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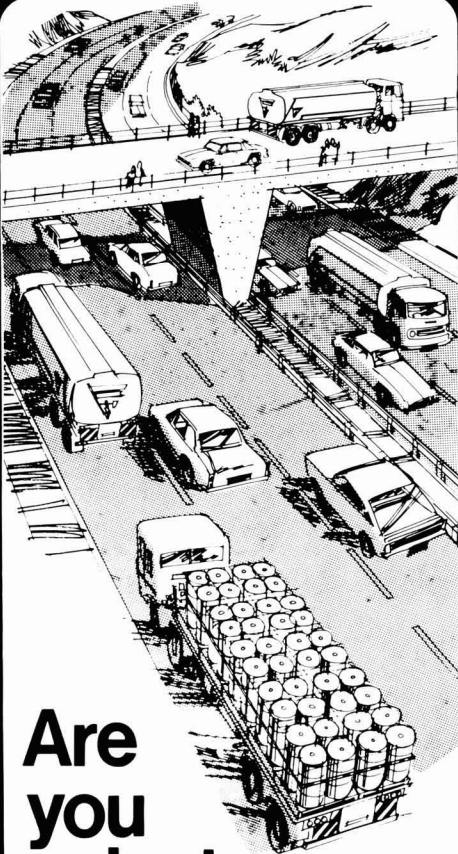
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**JOURNAL OF THE
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CHEMISTS'
ASSOCIATION**

- The painting of timber under adverse weather conditions *J. Boxall*
- The role of new technologies in coatings for the packaging industry *A. J. Newbould*
- Pigmentation of water-soluble alkyd-melamine resin combinations *N. A. Ghanem, F. F. Abd El-Mohsen and S. El-Zayyat*
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- Relative permittivities of some aniline- and toluidine-substituted anthraquinones—Short Communication *G. F. Lewin and S. J. Chapman*



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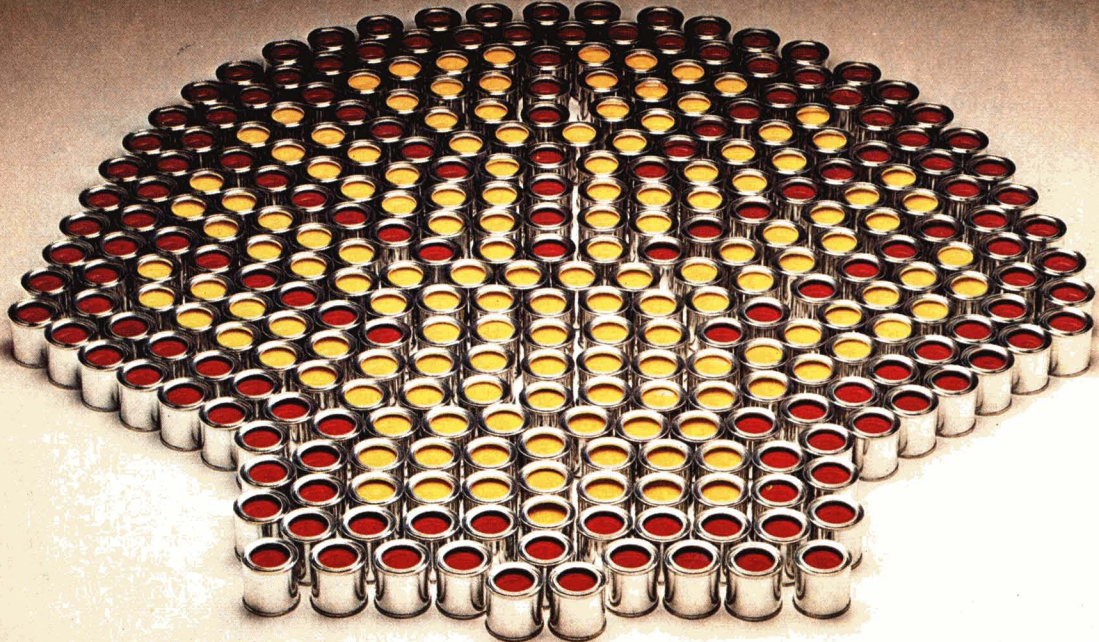
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The Association's twenty-ninth Technical Exhibition will be held at Alexandra Palace, London N22, from 22 to 25 March 1977. Further details regarding the arrangements for OCCA-29 and the many facilities offered at Alexandra Palace appear elsewhere in this issue of the *Journal*.

The Exhibition Committee emphasises on this occasion the quality of the OCCA Exhibitions in providing a *focus* for all those connected (either as suppliers of raw materials and equipment, or as buyers or in some other capacity) with the many and varied coatings markets throughout the world. The Exhibition has long been known as the annual international forum for display and discussion in the surface coatings industries, and the motif for 1977 draws attention to the concept of the annual "focal point" for the industries concerned.

Any organisation which has not previously exhibited and wishes to obtain an Invitation to Exhibit should contact the Association's offices immediately. The address is given on the Contents page of this issue.

"Official Guide"

The Exhibition Committee offers advertising space in the "Official Guide" to the Exhibition, which has proved to be a very popular advertising medium, not only for exhibitors but also for companies who were not showing at a particular Exhibition.

This unique publication will be circulated to all members of the Association, both at home and overseas, and as far as possible to all consuming companies in Great Britain. Copies will also be sent to those applying as a result of the Association's wide-spread publicity. The advantages of an advertisement in the *Official Guide* need hardly be stressed, since it will contain details of each Exhibitor's stand and thus constitute a summary of those recent technical developments which will be shown; consequently, it will be read widely and kept for reference.

For details of advertising in this and other Association publications, write to the Assistant Editor at the address on the Contents page of this issue.

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A reprint of the full regulations for admission to the Professional Grade and application form may be obtained by sending a stamped, self-addressed envelope, marked "Professional Grade leaflet" in the top left-hand corner, to the Association's offices at the address on the Contents page of this issue. It is felt that some overseas Members, in particular, might encounter difficulties contacting the required number of sponsors and any applicant who finds himself in this position is advised to write to the Director & Secretary of the Association in the first instance.

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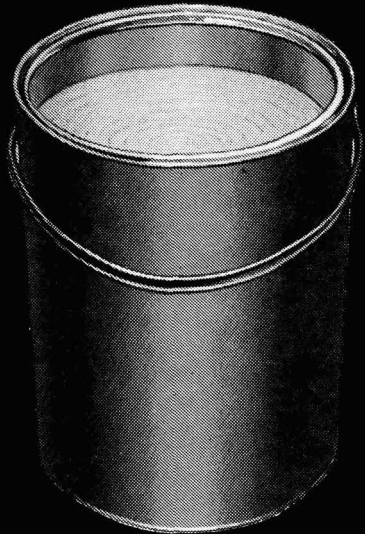
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The painting of timber under adverse weather conditions

By J. Boxall

Department of the Environment, Building Research Establishment, Princes Risborough Laboratory, Princes Risborough, Aylesbury, Buckinghamshire HP17 9PX

Summary

An assessment has been made of the tolerance of a range of paint systems applied to wood under adverse weather conditions. The paints were applied to Baltic redwood panels with a water trap cut into the test face and exposed horizontally. The performance

of the paint systems under test differed during 24 months of exterior exposure, but it has not been possible to relate these differences to the atmospheric conditions at the time of application.

Keywords

Types and classes of coatings and allied products

alkyd coating
primer
undercoat
urethane coating

Types and classes of structures or surfaces to be coated

redwood

Processes and methods primarily associated with the application of coatings and allied products

brush coating

Properties, characteristics and conditions primarily associated with:

coatings during application
drying rate

dried or cured films

adhesion
weather resistance

Le peinturage de bois en temps défavorable

Résumé

On a fait une appréciation de la tolérance d'une gamme de systèmes de peintures qui ont été appliquées au bois en temps défavorable. Les peintures ont été appliquées aux panneaux de bois rouge baltique munis d'une rayure gravée sur la surface sous essai pour attrapper l'eau. Les panneaux étaient disposés horizontalement.

Le rendement des systèmes de peintures qui faisaient l'objet de l'essai se différiaient pendant les 24 mois d'exposition aux intempéries, mais il n'a pas été possible d'établir un rapport entre ces différences et les conditions atmosphériques qui régnaient lors de l'application des peintures.

Das Anstreichen von Holz unter widrigen Wetterverhältnissen

Zusammenfassung:

Eine Bewertung der Toleranz einer Reihe von unter widrigen Wetterverhältnissen auf Holz angewandten Anstrichsystemen. Die Farben wurden auf Tafeln aus baltischem Rotholz, das mit einer in die Prüffläche geschnittenen "Wasserfalle" versehen war, aufgetragen, und horizontal exponiert. In den 24 Monaten der

Aussenbewitterung verhielten sich die der Prüfung unterliegenden Anstrichsysteme verschieden, es war aber nicht möglich, diese Unterschiede zu den beim Auftragen herrschenden Bedingungen in Beziehung zu setzen.

Introduction

Refs. 1-3

It is generally considered that the weather conditions prior to and during painting operations can have a considerable effect on the protective value and the service life of coating systems. In particular, painting during rain, sleet or snow, in very cold weather, or on substrates with a high moisture content or with moisture condensed on the surface, is held to be detrimental to paint performance. This is reflected in British Standard Code of Practice CP 231:1966 *The painting of buildings*¹ and CP 2008:1966 *Protection of iron and steel structures from corrosion*².

CP 231 recommends that painting should not be carried out at temperatures below 4.5°C, at relative humidities above 80 per cent, or under conditions in which water is liable to condense on the surface.

CP 2008 states that the ideal temperature for painting lies within the range 13–32°C at a relative humidity (RH) below 90 per cent. It also recommends that painting operations should be suspended when the atmospheric temperature falls below 4°C or its relative humidity rises above 90 per cent. Painting should also cease when the surface is exposed to rain, snow, fog or mist or in conditions such that condensation has either already occurred on the surface to be painted or is likely to occur in the immediate future.

In practice, however, it is frequently necessary to paint under non-ideal conditions, for example in spells of prolonged cold weather, and also the vagaries of the weather can cause freshly applied coatings to be exposed to adverse conditions. Clearly, guidance would be beneficial on the tolerance of currently used paint systems to application under adverse weather conditions. A programme of work has been undertaken, therefore, by the Princes Risborough Laboratory of the Building Research Establishment to investigate both the

tolerance of paint systems to adverse conditions and also to identify ways of improving paint performance. The experimental work on building substrates other than wood was carried out under an extra-mural research contract by the Paint Research Association (PRA). A brief summary of this work has been published³, and will be reported more fully elsewhere.

The results reported here relate to assessments of paint performance on wood conducted at the Princes Risborough Laboratory.

Experimental

Paints evaluated

The paints used in this project were all drawn from proprietary sources and were typical of those used to coat timber. Four primers, four undercoats and four gloss finishing paints were used in this study in combinations shown in Table 1. The paints were classified as:

<i>Primers:</i>	<i>P1</i>	Pink lead, linseed oil binder
	<i>P2</i>	Oleoresinous joinery primer, "lead-free", quick drying
	<i>P3</i>	Aluminium, oleoresinous binder
	<i>P4</i>	Acrylic emulsion, "lead-free" pigmentation.
<i>Undercoats:</i>	<i>U1</i>	Alkyd undercoat for <i>F1</i>
	<i>U2</i>	Acrylic emulsion undercoat
	<i>U3</i>	Alkyd undercoat
	<i>U4</i>	Polyurethane alkyd undercoat for <i>F2</i> .
<i>Finishes:</i>	<i>F1</i>	Polyurethane alkyd, gloss
	<i>F2</i>	Polyurethane alkyd, gloss
	<i>F3</i>	Long oil alkyd, gloss
	<i>F4</i>	Long oil alkyd, gloss.

The colour of all of the finishing systems was white. The pairs of finishes *F1* and *F2*, *F3* and *F4*, although of the same general composition, were from different manufacturers.

Table 1
Paint systems for exposure testing

Paint	System							
	<i>A</i>	<i>B</i>	<i>C</i>	<i>D</i>	<i>E</i>	<i>F</i>	<i>G</i>	<i>H</i>
Primer	1	2	2	2	2	4	4	3
Undercoat	3	3	2	4	1	3	2	3
Finish	3	3	4	2	1	3	4	3

Panel preparation

Refs. 4, 5

Sapwood of Baltic redwood (*Pinus sylvestris*) was used exclusively and was selected to exclude material having a high porosity (that is, 30 units or less on the PRA porosity meter⁴). It was found impractical to exclude heartwood and knots completely, but these were kept to a minimum. No panel was used with heartwood on the test surface and only 30 per cent of the panels contained a knot.

The design of the exposure panel used for the test was of the type recommended by the PRA for testing joinery paints,

300mm × 100mm × 15mm in size with a groove 250mm × 15mm × 6mm cut on the outer (bark) face.

The test panels were conditioned to a moisture content of 12 per cent prior to preservation with a water-repellent pretreatment comprising:

Per cent by weight

Tri n-butyl tin oxide	1.0
Long oil alkyd resin	10.0 (solids)
Paraffin wax	0.5
Hydrocarbon solvent	88.5

Two preservation treatment methods were used in this study: three minute immersion and double-vacuum impregnation⁵. After preservative treatment, the panels were conditioned at 25°C and 65 per cent RH for a period of two weeks prior to the commencement of painting.

Application of coating systems

All the panels were primed at 25°C and 65 per cent RH, and left to dry for 24 hours under these conditions. This procedure simulated the factory application of primer prior to the delivery of the component to site. The undercoats and topcoats, however, were applied and allowed to dry under the selected test conditions.

Every painting operation was performed in a climate chamber maintained at one of the following sets of conditions:

- (i) 25 ± 1°C; 65 ± 2.5 per cent RH
- (ii) 10 ± 1°C; 90 ± 2.5 per cent RH
- (iii) 5 ± 1°C; 90 ± 2.5 per cent RH
- (iv) 5 ± 1°C; 90 ± 2.5 per cent RH and surface condensation.

The condensation was obtained by storing the test panels in a freezer at -12°C for 30 minutes, after which they were moved to the climate chamber and painted immediately.

All the paint systems were applied, under all test conditions, to both immersion and double-vacuum preserved timber. Duplicate panels were used throughout. After application, the panels were allowed to dry in the test atmosphere. When dry, the panels were stored at ambient laboratory conditions for two weeks prior to exposure.

Exposure testing

The panels were mounted slot uppermost on horizontal racks, one metre above ground level. Exposure was commenced in October 1973 at the Princes Risborough exposure site.

The test panels were assessed every three months and adhesion measurements were performed after 6, 12 and 24 months of exposure.

Adhesion testing

Ref. 6

The adhesion of the coating systems was measured using the torque wrench technique described by Walker⁶. Before fixing the test stud to the surface, the area where the test was to be performed was lightly abraded using 400 grade wet or dry

paper. A two component, epoxy-polyamide adhesive was used for bonding the stud to the surface and the adhesive was left to cure for approximately 20 hours prior to testing.

After removal from the exposure racks, the test panels were conditioned to a moisture content of 16-18 per cent prior to adhesion measurements. The failing load for each test stud was recorded together with the mode of failure. Three types of failures were recognised:

- (i) cohesive failure within the coating system,
- (ii) adhesive failure of the paint system from the wood, and

- (iii) cohesive failure within the wood.

For the adhesion testing performed after 6 and 12 months' exposure, a total of ten tests was performed, five on each duplicate panel, for each combination of the preservative treatment/application conditions. At the end of the 24 months' exposure period, three further tests were performed on each panel. After every measurement, and prior to the next exposure period, the test areas were sealed with a pitch-epoxy compound.

The replication of the results on each individual panel was good and the arithmetic mean was used to determine the results. The adhesion values, expressed in MNm², together with the standard deviations, are shown in Figs. 1-8.

- A - 5°C : condensation
- B - 5°C : 90% RH
- C - 10°C : 90% RH
- D - 25°C : 65% RH

- Lead primer P1
- Alkyd undercoat U3
- Alkyd gloss F3

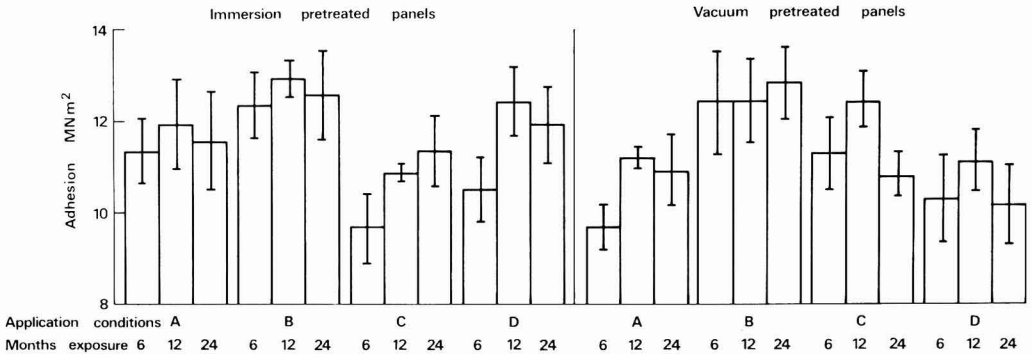


Fig. 1. Adhesion changes during exposure. System A

- A - 5°C : condensation
- B - 5°C : 90% RH
- C - 10°C : 90% RH
- D - 25°C : 65% RH

- Leadless primer P2
- Alkyd undercoat U3
- Alkyd gloss F3

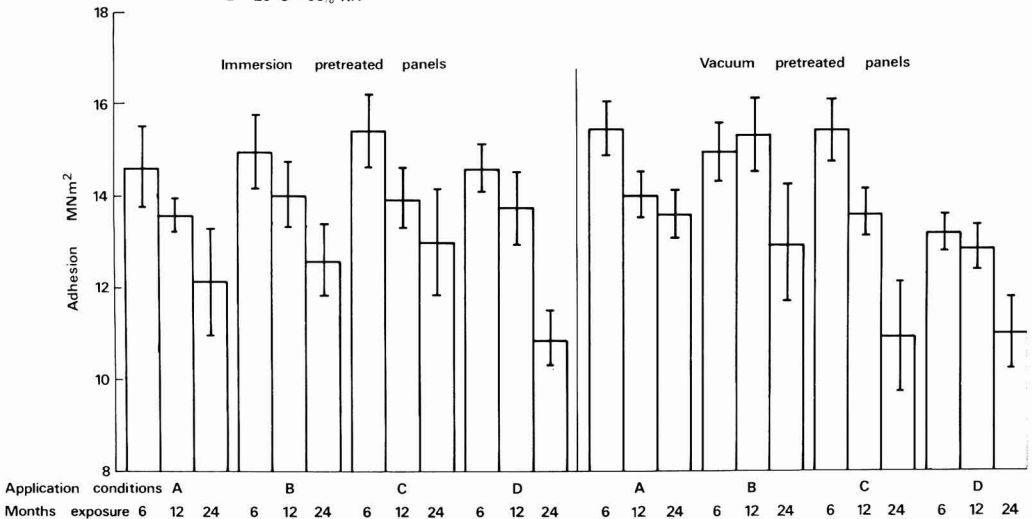


Fig. 2. Adhesion changes during exposure. System B

A - 5⁰C : condensation
 B - 5⁰C : 90% RH
 C - 10⁰C : 90% RH
 D - 25⁰C : 65% RH

Leadless primer P2
 Emulsion undercoat U2
 Alkyd gloss F4

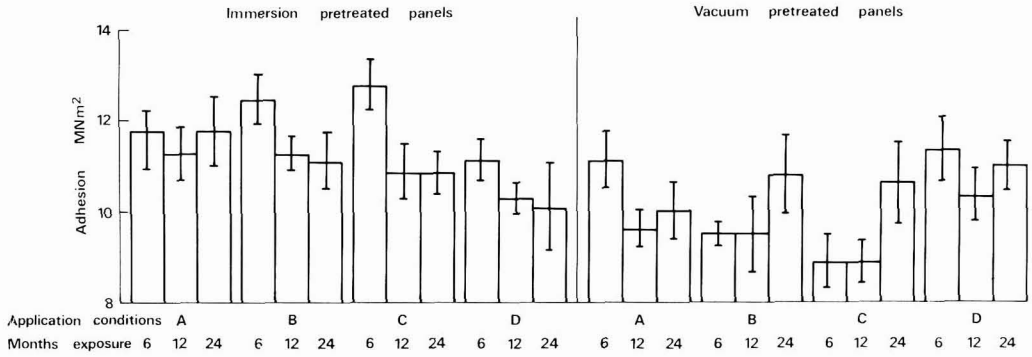


Fig. 3. Adhesion changes during exposure. System C

A - 5⁰C : condensation
 B - 5⁰C : 90% RH
 C - 10⁰C : 90% RH
 D - 25⁰C : 65% RH

Leadless primer P2
 PU - Alkyd undercoat U4
 PU Alkyd gloss F2

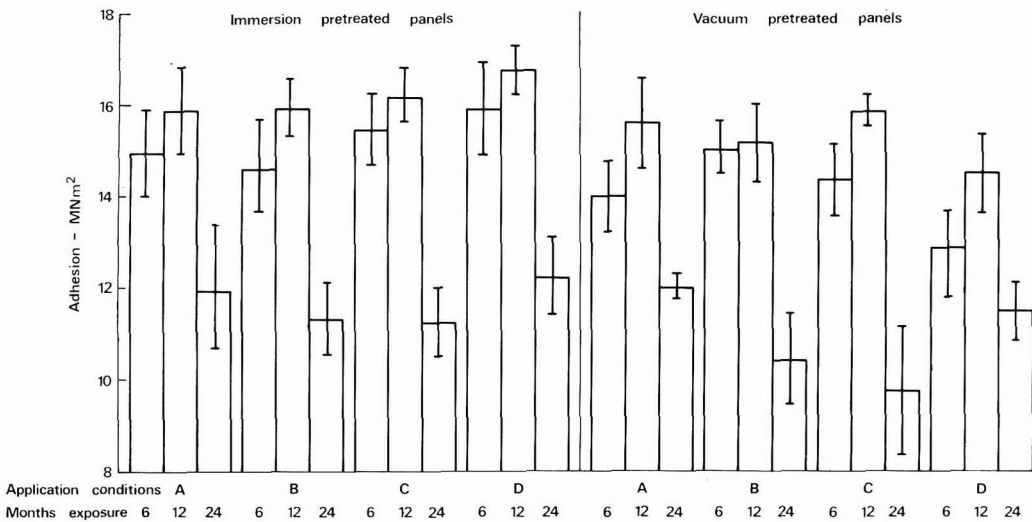


Fig. 4. Adhesion changes during exposure. System D

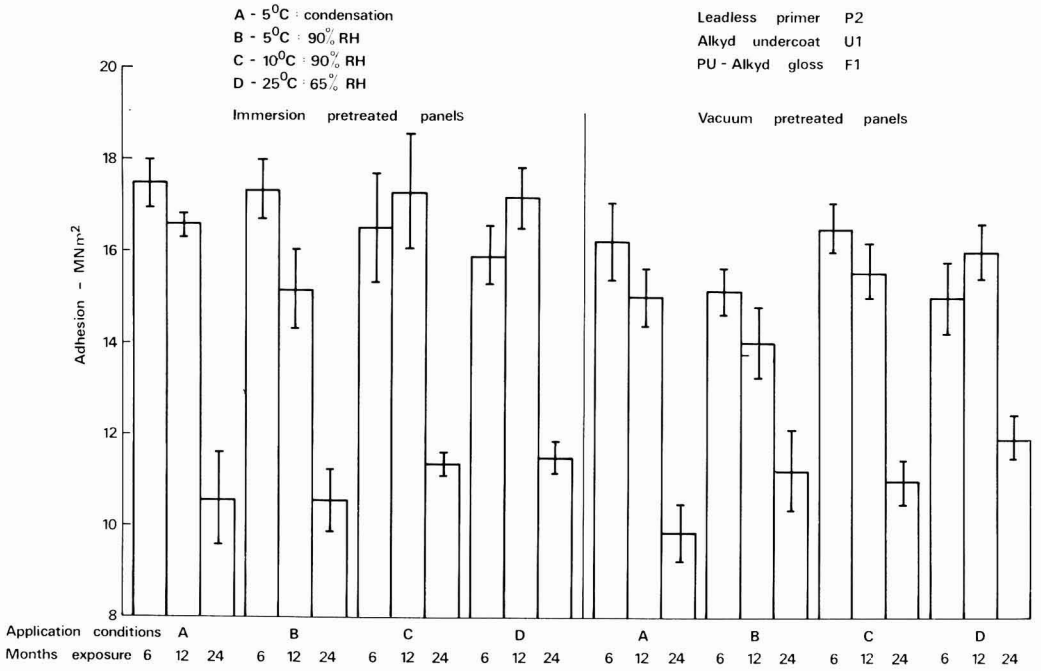


Fig. 5. Adhesion changes during exposure. System E

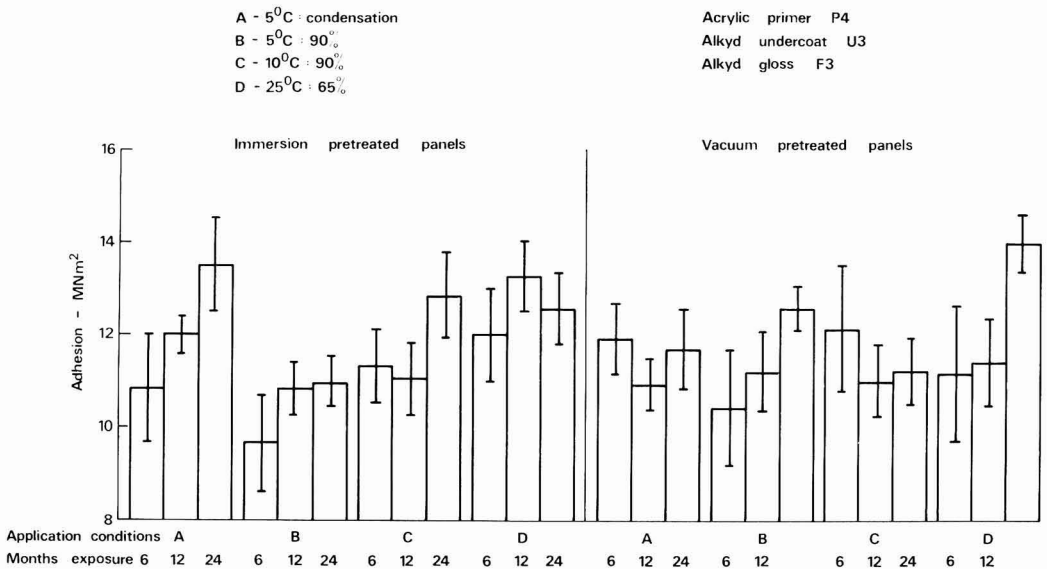


Fig. 6. Adhesion changes during exposure. System F

A - 5⁰C : condensation
 B - 5⁰C : 90% RH
 C - 10⁰C : 90% RH
 D - 25⁰C : 65% RH

Acrylic primer P4
 Emulsion undercoat U2
 Alkyd gloss F4

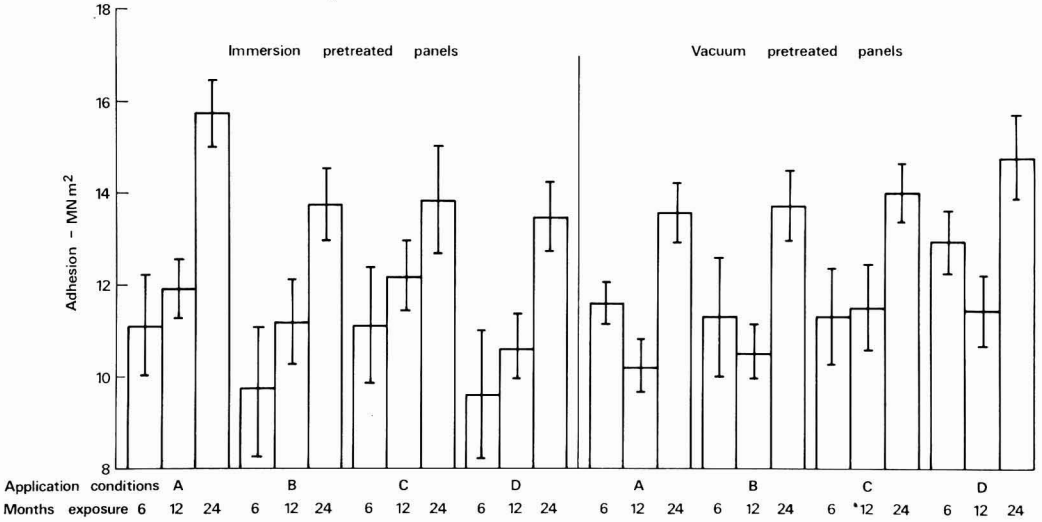


Fig. 7. Adhesion changes during exposure. System G

A - 5⁰C : condensation
 B - 5⁰C : 90% RH
 C - 10⁰C : 90% RH
 D - 25⁰C : 65% RH

Aluminium primer P3
 Alkyd undercoat U3
 Alkyd gloss F3

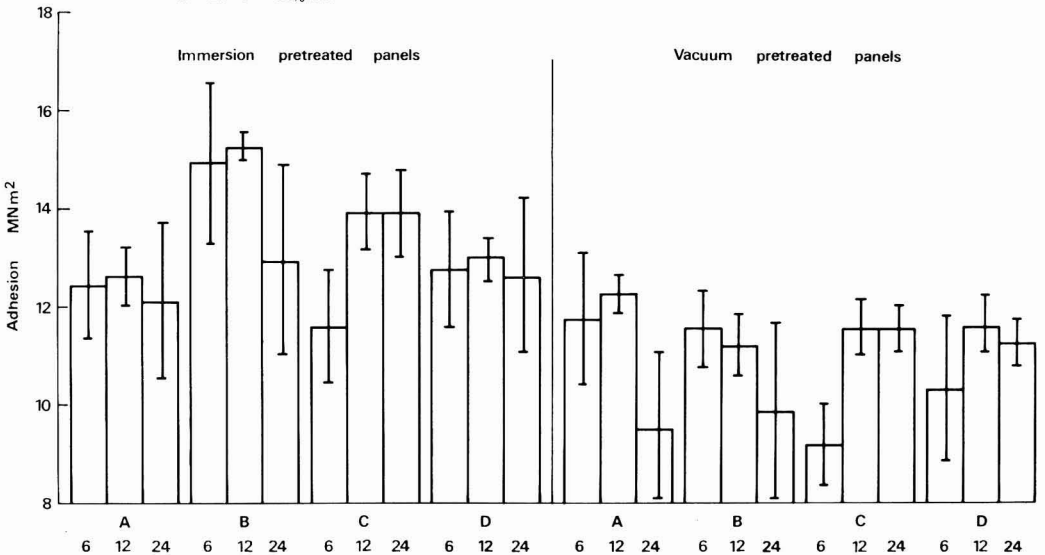


Fig. 8. Adhesion changes during exposure. System H

Drying time studies

The drying times of the paints selected for evaluation were measured using a Beck Koller drying time recorder. For all tests, a wet film thickness of 39 μ m was applied using glass as the substrate. The drying characteristics of the coatings were measured under a variety of simulated atmospheric conditions and the following have been selected for presentation here since they represent a wide range of drying conditions:

- (i) 25 \pm 1°C; 65 \pm 2.5 per cent RH
- (ii) 25 \pm 1°C; 90 \pm 2.5 per cent RH
- (iii) 10 \pm 1°C; 90 \pm 2.5 per cent RH
- (iv) 5 \pm 1°C; 90 \pm 2.5 per cent RH
- (v) 5 \pm 1°C; 90 \pm 2.5 per cent RH and surface condensation
- (vi) 2.5 \pm 1°C; 90 \pm 2.5 per cent RH.

The time (in hours) required by the paint films to achieve a condition where they were not torn by the needle of the test instrument is shown in Table 2. This stage in the drying process can be considered the minimum time before which overcoating can commence.

Table 2
Drying times (hours)

Paint		2.5°C; 90% RH	5°C; condensation	5°C; 90% RH	10°C; 90% RH	25°C; 90% RH	25°C; 65% RH
Primers	<i>P1</i>	32	39	30	18	8.5	8.5
	<i>P2</i>	1	2.75	0.75	1.5	2	1
	<i>P3</i>	30	22	30	28	50	20
	<i>P4</i>	0.6	1	1.25	0.9	1.75	0.2
Undercoats	<i>U1</i>	74	36	34	38	6	9.5
	<i>U2</i>	1	1.25	0.6	0.8	0.5	0.5
	<i>U3</i>	23	23	26	24	6	6.5
	<i>U4</i>	1.5	1.5	1.5	5	7	6.5
Finishes	<i>F1</i>	22	18	18	9.5	5	4
	<i>F2</i>	25	22	22	15	3.5	3.5
	<i>F3</i>	14	10	13	7	5	5
	<i>F4</i>	32	29	24	24	10.5	9.5

Results

Application properties

After application of the films, micro-blistering of their surface was noted in the case of all systems. The effect was not general across the test faces, but occurred in streaks and/or patches. These blisters, which were not hollow, were typically less than 0.5mm in diameter and were much softer than the surrounding paint film.

Exposure trials

After 6 months' exposure, film cracking was evident around the edges of the grooves in all the test panels. The cracking extended along the length of the groove, except for systems *F* and *G*, where only discontinuous cracking was evident. Within each system there were no differences in the degree of cracking which could be related to application conditions.

After 24 months' exposure, there were no major differences in performance within each system which could be related to the application conditions. Detailed differences did exist, however, between and within the systems, as summarised below.

System A

Lead primer, *P1*
Alkyd undercoat, *U3*
Alkyd gloss, *F3*

After 24 months' exposure, slight flaking from the areas of cracking was evident, although the effect was noted only on panels prepared at 5°C with condensation and at 25°C; 65 per cent RH. Other panels in the series were all in good condition. There were no differences between double-vacuum and immersion pretreatments, but all panels showed slight film yellowing, which appeared to be associated with the micro-blisters.

System B

Leadless primer, *P2*
Alkyd undercoat, *U3*
Alkyd gloss, *F3*

After 24 months' exposure, slight flaking was developing around panel edges and this was similar in degree on all panels. Some yellowing of the micro-blisters was also evident on all panels.

System C

Leadless primer, *P2*
Emulsion undercoat, *U2*
Alkyd gloss, *F4*

All panels in this system showed film yellowing after 9 months' exposure; this appeared to be associated with discolouration of micro-blisters on the surface. After 24 months, all panels were subject to slight flaking from cracks near groove edges. There were no differences in performance between immersion and double-vacuum pretreated panels.

System D

Leadless primer, *P2*
Polyurethane-alkyd undercoat, *U4*
Polyurethane-alkyd gloss, *F2*

Yellowing of surface micro-blisters was evident on all panels after 9 months' exposure. After 24 months, panels prepared at 25°C; 65 per cent RH, 10°C; 90 per cent RH and 5°C; 90 per cent RH were all exhibiting flaking of the complete system, both from panel and groove edges and from isolated areas on the face. Panels prepared at 5°C with condensation were in better condition, but even these showed worse flaking than any other system except *H*.

Panels treated by double-vacuum impregnation were in a worse condition than those treated by immersion.

System E

Leadless primer, *P2*
Alkyd undercoat, *U1*
Polyurethane-alkyd gloss, *F1*

After 24 months, slight flaking was occurring around cracks in the film, but only the double-vacuum treated panels were affected. The degree of flaking was similar to that of system *C*. There were no indications that performance was influenced by the conditions under which paints were applied.

Yellowing due to discolouration of the micro-blisters was evident and the effect was most pronounced on double-vacuum pretreated panels.

System F

Acrylic primer, *P4*
Alkyd undercoat, *U3*
Alkyd gloss, *F3*

After 24 months' exposure, only discontinuous cracking was evident around the groove and only one panel, prepared at 25°C; 65 per cent RH, was affected by flaking. All panels exhibited film yellowing attributable to discolouration of micro-blisters. There were no differences between panels treated by immersion or double-vacuum techniques.

System G

Acrylic primer, *P4*
Emulsion undercoat, *U2*
Alkyd gloss, *F4*

After 9 months' exposure, some film yellowing due to discolouration of micro-blisters was evident, panels prepared at 5°C with condensation and at 10°C; 90 per cent RH being most affected. Discontinuous film cracking developed during the first six months' exposure but did not progress.

System H

Aluminium primer, *P3*
Alkyd undercoat, *U3*
Alkyd gloss, *F3*

After 24 months' exposure, flaking of the complete system was observed from panel and groove edges, but no major differences due to application conditions or pretreatment method were evident. The degree of flaking was slightly greater than that exhibited by system *D*. Film yellowing did not occur to any marked degree with this system.

Adhesion testing

In general, the application of the coating systems under adverse conditions did not result in any marked decrease in initial adhesion or variation in the observed site of failure. Variations in performance did exist between systems, however. The adhesion of system *A* remained relatively constant during exposure, whereas that of systems *B*, *C*, *D*, *E* and to some extent *H* tended to decrease, and that of systems *F* and *G* to increase with time. After 24 months' weathering, the adhesion of all of the paint systems were still relatively high.

For any one paint system, no significant adhesion differences were found which could be related to the method of preservative pretreatment.

The site of adhesion failure varied from system to system, but except for systems *D* and *H*, did not vary on exposure (Table 3).

Drying times

In general, decreasing temperatures from 25°C to 5°C result in increasing drying times for the paints, although the polyurethane-alkyd undercoat *U4* exhibited reduced drying times as the temperature decreased.

When the temperature was further reduced to 2.5°C, the drying times for most of the paints were only slightly longer than at 5°C. There were two exceptions: *U1*, and to a lesser extent *F4*, both exhibited a marked increase in drying time at 2.5°C.

The presence of condensation on the surface of the test substrate at the time of application had little effect on the drying of the paint. The lead primer *P1* proved the most sensitive to the presence of moisture and exhibited an increased

Table 3
Adhesion testing, failure sites

System	6 months	12 months	24 months
<i>A</i>	Paint adhesive with wood cohesive	----->	
<i>B</i>	Paint cohesive-adhesive with wood cohesive	----->	
<i>C</i>	Paint cohesive-adhesive with wood cohesive	----->	
<i>D</i>	Paint adhesive with wood cohesive	Paint cohesive-adhesive with wood cohesive	----->
<i>E</i>	Paint cohesive-adhesive with wood cohesive	----->	
<i>F</i>	Paint cohesive-adhesive with wood cohesive	----->	
<i>G</i>	Paint cohesive-adhesive with wood cohesive	----->	
<i>H</i>	Paint adhesive	Paint cohesive-adhesive	----->

drying time compared with its performance at 5°C; 90 per cent RH. It is interesting to note that the three paints known to contain a proportion of polyurethane resin in their composition (*U4*, *F1* and *F2*) were the only materials to exhibit similar drying characteristics, both in the absence and presence of surface condensation.

Discussion

The results presented here indicate that the conditions at the time of application are of secondary importance compared with the nature of the paint system itself.

All of the paints tested here have demonstrated a high tolerance to adverse conditions at the time of application, although they differed in their resistance to outdoor weathering. In particular, the performances of systems *D*, the lead-free primer, polyurethane-alkyd undercoat and gloss, and *H*, the aluminium primer, alkyd undercoat and gloss finish, were markedly inferior to those of the other systems. As other lead-free primer/finishing coat combinations were superior to system *D*, the conclusion to be drawn is that the low durability of this system is attributable primarily to the properties of the polyurethane-alkyd undercoat and/or finish. Similarly with system *H*, as the alkyd undercoat and finishing coat used on this system have performed well on other primers, the implication is that the inferior performance of the system is due to the poor durability of the aluminium primer.

A further consideration is that all the coating systems exhibited some cracking over sharp edges of the test panels within a relatively short period of exposure. Although this did not result in major failure with all except systems *D* and *H*, it is indicative of performance limitations for these classes of coatings; with more severe exposure conditions, these limitations may have resulted in premature film failure.

It is also important to remember that the exposure results presented here are essentially only short-term and it is possible that continued exposure for, for example, the four to five years typically required for local authority maintenance cycles, will reveal differences in performance which can be related to conditions at the time of application.

There is no evidence that the nature of the preservation method markedly influences the adhesion or the overall exterior performance of the paint systems. However, preservation did result in the micro-blistering problem, which must be attributed to entrapped solvents in the timber diffusing out and causing localised areas of film swelling. The presence of solvent within the timber would also result in a more ready migration of wood resins and extractives to the surface and it is probable that the yellow-brown discolouration of the blisters on prolonged exposure is due to oxidation of these resins. The blistering effect was most marked on panels treated by double vacuum and this reflects the greater uptake of preservative achieved by this process. However, due to the longer ageing allowed in practice before application of finishing systems to primed joinery, problems with preservative/paint film interactions are not frequently encountered.

The adhesion results also indicate little influence between the atmospheric conditions at the time of application of the finishing systems and subsequent paint performance. The nature of the paint system appears to be of much greater importance. In particular, systems *B*, *C*, *D* and *E* were all found to exhibit trends towards decreasing adhesion with

exposure time and it is interesting to note that these systems all contain the lead-free primer *P2*. This particular primer is designed to be quick-drying and thus suited for factory finishing; it is probable that this formulation requirement has resulted in a film that becomes brittle relatively quickly on ageing.

An increase in adhesion with time was noted with systems *F* and *G* based on the acrylic emulsion primer *P4*. This is difficult to explain since the adhesion of such paints might normally be expected to remain unchanged in the absence of degradation reactions. It is possible that the increasing adhesion of this system is related to secondary flow of the polymer component of the primer, induced by solar heating of the test panel surface during exposure. Secondary flow within the emulsion undercoat *U2* of system *C* may also account for its lower adhesion losses compared with the other systems (*B*, *D* and *E*) containing lead-free primer.

The adhesion values of systems *A*, *D* and to some extent *H*, were found to reach a peak after 12 months and then to exhibit a reduction in adhesion upon further exposure. This may be related to an initial development of hardness within the coating system, the reducing adhesion values representing the onset of a more brittle behaviour.

One possible important consequence of applying paints under adverse weather conditions is that the drying time may be increased to unacceptable levels, or the process delayed. With the paints examined here, it was found that drying times generally increased progressively as temperature during painting decreased from 25°C to 2.5°C. Furthermore, the presence of condensation had little effect on drying times compared with their drying performance under 90 per cent RH conditions.

It is probable that the performance of paint *U4*, which exhibited reduced drying times as the temperature decreased, is related to the increase in physical hardness of the film at low temperature rather than to any inherent acceleration of the drying process.

With one exception, all of the paints investigated in this study had low temperature drying times of less than 50 hours, many being in the region of 24 hours or less. Therefore, provided that the film was not physically damaged during the drying process, or was not subjected to prolonged, direct water impingement, such as rain, sleet or snow, it is possible that under adverse weather conditions this level of drying performance would be acceptable on site.

With emulsion paints systems, however, the effect of low temperature is more critical and with most conventional matt emulsion paints, problems of film formation, that is coalescence of the film, can be experienced at temperatures below 3°C. In addition, excessively long drying times are experienced below 5°C or at very high humidities with the semi-gloss and gloss emulsions recently introduced into the market. Consequently, when applying such paints under possibly adverse atmospheric conditions, CP 231 painting recommendations should be followed closely.

One further point to consider is the nature of the substrate. Timber was used throughout in this study and the paints investigated were specifically designed for use on this substrate. Because the paints used are found to exhibit a high tolerance to adverse application conditions, it is tempting to extrapolate these results to other substrates and their specific paint systems. However, although it may be possible to draw some broad

conclusions from this work, the results should be applied with extreme caution. For example, the work performed by the Paint Research Association on painting galvanised steel indicated that the adhesion of certain coating systems was severely affected when applied under conditions of 5°C and 90 per cent RH or when condensation was present on the surface.

Conclusions

Refs. 7-9

The tolerance of some currently available paints for timber towards application under adverse weather conditions appears to be of a high order. The subsequent performance of a coating system appears to be due more to its inherent characteristics than to the variations in conditions at the time of application.

The results suggest the need for change in the principles of formulating coatings for timber substrates. In particular, the rapid onset of cracking from sharp edges apparent with all of the systems examined suggests that these coatings become hard very rapidly. Whiteley^{7,8} has indicated that many wood

primers have inadequate flexibility for exterior woodwork, and efforts have been made to introduce primers more suited to the substrate. The recent standard BS 5358:1976 *Low-lead primers for wood*⁹, should enable better lead-free primers to be obtained. The work described here indicates that similar grounds for concern exist with regard to finishing paints.

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The role of new technologies in coatings for the packaging industry*

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Summary

Factors influencing development work within the surface coatings industry are briefly indicated. The requirements of coatings for the packaging industry are discussed, and the various end uses of organic solvent-borne resins in coatings for metal decorating are

tabulated. Alternative technologies, such as ultraviolet curing, water-borne coatings and high solids coatings, are discussed in relation to the needs of the packaging industry; some potential technical and economic benefits are shown.

Keywords

Types and classes of coatings and allied products

can coating
coatings for packages
water base paint

Le rôle de nouvelles technologies dans le domaine des revêtements pour l'industrie d'emballages

Résumé

On indique brièvement les facteurs qui exercent une influence sur les études de développement au sein de l'industrie de revêtements de surface. On discute les propriétés demandées des revêtements destinés à l'industrie d'emballages, et l'on arrange en table les divers emplois des résines solubles dans des solvants organiques au cours de la fabrication de revêtements destinés pour la décoration

de métaux. Au point de vue des besoins de l'industrie d'emballages, on discute des technologies alternatives, telles que durcissement par rayons ultra violets, revêtements à base d'eau ou à teneur élevée en matière sèche. On montre certains avantages éventuels et techniques et économiques.

Die von neuen Technologien der Beschichtungsmittel für die Verpackungsindustrie gespielte Rolle

Zusammenfassung

Die Faktoren, welche Entwicklungsarbeiten innerhalb der Beschichtungsmittelindustrie beeinflussen, werden kurz erwähnt. Die Anforderungen der Verpackungsindustrie gestellten Anforderungen werden besprochen, und die verschiedenen Endverwendungszwecke organischer in Blechlacken verwendeter Harze auf Lösungsmittelbasis werden tabuliert. Zur Wahl stehende

Technologien, wie Ultraviolettärtung, wassergetragene Beschichtungsmittel und solche mit hohem Festgehalt werden mit Bezug auf die Anforderungen der Verpackungsindustrie besprochen; einige potentielle technischen und wirtschaftlichen Vorteile werden aufgezeigt.

Introduction

Through its research and development work, the surface coatings industry is constantly improving the application properties, film performance and economics of coatings.

Over the past few years several factors have affected the direction of the development work. These include the increased cost and, in some countries, shortages of energy; unprecedented increases in materials, labour and insurance costs; pollution of the atmosphere; and the toxicity and inflammability of many solvents.

These pressures have encouraged the development of alternative methods of applying and curing coatings and a number of new technologies are being examined in depth to assess the advantages they can offer.

This paper reviews these technologies and discusses their properties in relation to the needs of the packaging industry.

Requirements for coatings for the packaging industry

Present solvent-borne coatings for the packaging industry have been developed over the last 40 years or so to be applicable by roller coaters or various spray techniques, to have

good substrate wetting and adhesion after curing at relatively low temperatures for short times, to be hard and at the same time flexible, and to be resistant to the wide range of food, beverage and industrial products which come into contact with them. Systems are available for coating the inside and outside of tinplate and aluminium beer/beverage/food cans, of steel drums, of tinplate, tin-free steel and aluminium caps and closures, of tinplate and aluminium aerosol containers and of miscellaneous packages, such as toothpaste tubes. External coatings in general need high gloss, to be hard, mar resistant and flexible enough not to be removed during forming operations, to have sufficiently high slip for high speed filling lines, to be resistant to processing and pasteurisation, and to have good intercoat adhesion.

Internal coatings must not affect the flavour or colour of the product, must be resistant to the product, must be an effective barrier, even when drawn at the lowest possible film weights, to prevent both metal corrosion by the pack and attack on the product by the substrate, must maintain film adhesion and integrity during processing and pasteurisation and in the case of food and drink packs be FDA approved.

The varied requirements of the packaging industry are met by using a wide range of often complex polymer types. Table I

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Table 1
Conventional resins and their end uses

Coating	Resin types	Advantages	Disadvantages	End uses
<i>Sizes</i>	Alkyd Epoxy-amino Vinyl	Good flexibility Good substrate and intercoat adhesion	High volatile solvent content	As size coatings where improved fabrication required
<i>Enamels</i>	Styrenated-alkyd	Cheap Good-fair flexibility Good processing resistance	Poor chemical resistance Poor colour retention	Low flexibility non-process and processing cans
	Polyester	Good flexibility Good colour retention Good process resistance	More expensive than alkyds	Processing and non-processing cans caps, closures Deep drawing caps Aerosols
	Acrylic	Good flexibility Good colour retention Good process resistance	Odours Expensive	Processing and non-processing caps Processing bodies
	Epoxy-ester	Good product resistance Good hardness	Limited flexibility Expensive	Toothpaste tubes
	Vinyl	Very good flexibility Good processing resistance—if modified	Low solids Very expensive Thermoplastic unless modified UV/heat degradable	Deep drawing caps Toothpaste tubes Drawn processing cans
<i>Varnishes</i>	Alkyd	Cheap Good hardness	Fair processing resistance Poor colour retention Flexibility fair	Low flexibility non-process cans
	Polyester	As <i>Enamels</i>	As <i>Enamels</i>	As <i>Enamels</i>
	Acrylic	As <i>Enamels</i>	As <i>Enamels</i>	As <i>Enamels</i>
	Epoxy-ester	Good hardness Good-fair flexibility	Expensive Fair colour retention	Screw caps Crown corks
	Vinyl	As <i>Enamels</i>	As <i>Enamels</i>	As <i>Enamels</i>
	Epoxy-amino	Good-fair flexibility Good colour retention Good processing resistance	Expensive	Caps and ends
<i>Lacquers</i>	Phenolic	Good produce resistance Excellent sulfur resistance Good processing resistance	Very poor flexibility Poor plate wetting	Can bodies
	Epoxy-phenolic (E/P)	Good product, sulfur and processing resistance Good flexibility	Expensive Lowish solids content	Can bodies and ends Internal and external non-compound caps
	Epoxy-ester/ phenolic	Good flexibility Cheaper than E/P	Only fair product sulfur and processing resistance	Cans bodies and ends
	Epoxy-urea formaldehyde	Good product and processing resistance Alcohol resistance good Cheaper than E/P	Only fair drawing properties	Internal spray lacquers for beer/beverage cans
	Vinyl	Excellent flexibility Good alcohol resistance	Low solids content Expensive UV/heat degradable Monomer thought to be carcinogenic	Internal spray lacquers for beer/beverage cans
	Organosol	Excellent flexibility and adhesion Good product resistance Excellent compound adhesion	Only fair flow-out Low process resistance High film weights required Monomer thought to be carcinogenic	High flexibility cap and closure linings Deep drawing lacquers
	Polybutadiene	Very cheap Good product resistance	Odour Only fair flexibility	Beer can bodies
	Oleoresinous	Cheap Good product resistance Good sulfur resistance, with ZnO or ZnCO ₃	High baking schedules required	General food can bodies

summarises the various resins currently used as media for coatings in the packaging industry and their end uses.

New technologies

The alternative technologies which have been developed and evaluated can conveniently be discussed under the following headings:

Type 1 Same application technique/same curing method as conventional coatings.

Type 2 Same application technique/different curing method.

Type 3 Different application technique/same curing method.

Type 4 Different application technique/different curing method.

Type 1

Water-borne coatings

Of the new technologies, water-borne coatings are the most similar to conventional solvent-borne systems; both consist of resins, volatile solvents, and pigment if an enamel, in approximately the same proportions. When a thin film is stoved, the solvents evaporate and the resins cure to form a coating.

Water-borne resins can be prepared as emulsions, colloids or solutions (water reducible).

Emulsions

Emulsions are produced either by reacting a mixture of monomers in the presence of water to give a latex or by dispersing a water-insoluble resin into an aqueous phase with emulsifiers etc., as required to impart stability. The most common emulsions evaluated for packaging applications have been acrylic, vinyl or styrene and mixtures of these.

Because an emulsion is a two-phase system, the viscosity of an emulsion resin will be very much lower than that of conventional solvent-borne resins at the same solids content. As a result, coatings with higher solids contents can be prepared from resins of very much higher molecular weight, giving films with improved flexibility and mar resistance.

The high solids/low viscosity relationship can be used to advantage in the preparation of internal spray lacquers for beer and other beverage cans. Low viscosities are required for good atomisation, coverage and flow. At present organic solvent-borne vinyls, with excellent alcohol resistance, are used. These need thinning with expensive solvents solely to obtain the desired application viscosity, with subsequent reduction in solids content. Emulsions, already at low viscosity, do not need thinning, allowing the application of higher solids coatings and savings in solvent costs.

The flow of emulsion-based varnishes and enamels is usually not as good as that of conventional systems due to the lack of reflow after application and poor thermal flow in the oven. For roller coating application, softer rollers can be used to reduce this flow problem.

Solutions

Solution resins are prepared by introducing water-soluble groups into the resin structure. The commonest way of doing this is to make a resin with a larger number of carboxyl groups than is usually present. On neutralisation with amine, water solubility is conferred on the resins. When stoved, water solubility is lost as the carboxyl groups react with crosslinking resins to form water-insoluble products. Water-soluble equivalents of most of the resins currently used in the packaging industry can be prepared in this way. The crosslinking amino resins are made water soluble by replacing butanol with ethanol or methanol at the etherification stage. These lose their water sensitivity during crosslinking.

Water-reducible resins contain higher levels of organic solvent than do emulsions. Being solutions, their viscosity is very much more dependent on the molecular weight of the resin and to help improve solubility a quantity of solvent is required.

Water-reducible alkyds, polyesters, acrylics and epoxy esters have all been evaluated as the basis of coatings for the packaging industry. Resins have to be formulated much more critically since the requirement for highly flexible coatings is in opposition to the need to produce lower molecular weights (chain lengths) for water solubility.

A wide range of coatings has been prepared with application and film properties equivalent to conventional solvent-borne systems when evaluated on commercial equipment.

Colloids

Colloidal resins lie between solutions and emulsions in their resin properties. They are dispersions of ultra-fine particles of resins, intermediate in molecular weight between emulsions and solutions, and containing acidic or basic polar groups which impart a degree of water solubility. They have not yet been found suitable for packaging end-uses. The viscosity of the resins can be varied from low (as supplied) to high by solubilising the polar groups present with amines etc., or by solubilising the resin with organic solvents. At low viscosities, they behave predominantly like emulsions and can exhibit the same flow problems. Most colloidal resins are very pH sensitive, small changes in pH causing large changes in viscosity, thus making it difficult to control accurately the application parameters. The addition of organic solvents can also lead to rapid increases in viscosity as the high molecular weight components previously emulsified, become solubilised.

High solids coatings

In order to prepare high solids solution coatings (≥ 80 per cent solids content) suitable for application on conventional coating equipment, low molecular weight resin species, which polymerise during stoving, are required. Acrylic, polyester and epoxy resins have been evaluated. The high flexibility and film hardness required of coatings for the packaging industry can limit the use of very high solids coatings based on very low molecular weight resins.

Coatings with higher solids content and higher viscosities (and so with improved flexibility) might be applied on heated rollers, although this could well present additional handling problems.

The application costs for coatings which are used in large volumes can be reduced by appreciably increasing the solids content. Internal organosols, for example, are currently applied at relatively high film weights from relatively low solids coatings.

Because the system is partially a dispersion of polyvinyl chloride and partially a solution of resins, the viscosity is not so dependent on molecular weight.

Non-aqueous dispersions (NADs)

Similar in principle to aqueous emulsions, NADs are dispersions of resin in organic non-solvents. Acrylic and epoxy resins have been prepared and although high solids/low viscosity coatings are obtained, the flow of these two-phase systems on roller coaters can be poor, as with the aqueous emulsions.

Type 2

Ultraviolet curing

The principle of this method as applied to the packaging industry is that UV radiation is used to produce free radicals from photo-initiators. These radicals cause polymerisation of the polymer/monomer mixture used as media and thus cure the film. Low molecular weight/low viscosity monomers are required to give a degree of flow to the coating and make it suitable for application by roller coater. The balance between monomers and polymers needs to be controlled carefully to give good flow from the former and a sufficiently high degree of polymerisation for good flexibility from the latter.

Cure can be carried out in air or in an inert atmosphere. Because of the high surface-to-volume ratio, oxygen inhibition—always a detrimental factor in radical induced polymerisation—is a particular problem. The use of nitrogen or argon blankets attempts to overcome this. There are sealing difficulties when non-continuous substrates are cured, which makes air the more favoured atmosphere in which to operate.

Typical of the resin systems evaluated for UV cure are acrylic, epoxy-acrylic and unsaturated polyesters. Acrylic resins are the most adaptable to the requirements of the packaging industry for hard flexible films with good adhesion after short cure times. A number of UV curable varnishes and white coatings are available, although the latter need formulating more carefully to overcome the reduction in cure rate which can occur due to UV absorption by the pigment.

Because there is little time available for flow in the wet state between application and curing and there is no thermal reflow, the film appearance of UV cured coatings is often not as good as that of conventional coatings.

Electron beam curing

As an alternative curing technique, the electron beam is not new. Work had been carried out in the 1950s to examine the application of this method to the packaging industry. Two types have been considered. In both, electrons are generated from a source and accelerated in vacuum towards a target. The beam of electrons is focussed and can be scanned across

the target—in the case of the packaging industry, a pre-coated sheet of a metallic substrate.

In the simpler air-cure method, the electrons pass through a window into air before hitting the target. The efficiency of the electrons is reduced due to energy absorption during passage through the window and air space. Vacuum cure, where the substrate is irradiated in a vacuum, is most suitable for continuous substrates, such as coil coating. Non-continuous sheet coating requires a more complex system of seals to maintain the vacuum in the irradiation chamber.

Type 3

Electrodeposition

Some work has been carried out to examine the application of water-borne coatings by electrodeposition to the inside of beer and beverage cans. If a current is passed through an aqueous solution containing electrolyte, negatively charged ions will migrate to the anode and positively charged ones to the cathode. Water-reducible resins are generally anionic due to the presence of carboxyl groups used to confer water solubility, and by making a metal can the anode a layer of coating can be deposited on uncoated areas. A subsequent stoving operation converts the coating to a cured film.

Repair coats, to cover damaged areas and film defects, are being applied by this technique over internal lacquers for beer cans.

Powder coatings

A number of different resin types are suitable for the preparation of finely divided solid particles. Acrylic, polyester and epoxy thermosetting powders, and nylon, polyvinyl chloride and polyethylene thermoplastic powders have been evaluated. They cannot be applied by roller coater and other methods, such as electrostatic spraying, are used. Powders can be applied satisfactorily to the inside of drums, but for film continuity at the low film thicknesses required for can coatings, powders with small particle size have to be used. The use of this type of powder requires additional precautions to prevent it from becoming electrostatically charged with subsequent flow problems and application difficulties due to blocking of powder in the spray equipment.

Powder coatings are being used commercially to coat the exterior side seam of beer cans.

Type 4

Hot melt coatings

Based on microcrystalline waxes, hot melt coatings have been evaluated as replacements for low solids vinyls on the inside of beer and beverage cans. Hot liquid wax is applied by spray on to the inside of cans at room temperature. A rough coating results, and this can be caused to flow out by heating at low temperature. In order to achieve the same resistance properties as vinyls, higher film weights have to be used. Their resistance to flavouring does not appear to be as good as that of the vinyls, and this could limit their use.

Conclusion

All the alternative technologies discussed have potential application in the packaging industry. The extent to which each system is used depends on the way in which it can help to overcome the particular economic and technical problems of individual users within the prevailing legislative framework.

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Pigmentation of water-soluble alkyd/melamine resin combinations

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Summary

A stable, rutile grade titanium dioxide pigment has been selected for studies of the effect of pigmentation on the properties of paint films cast from a water-soluble alkyd resin.

It is found that gloss increases with increasing pigment-to-binder ratios up to 1.4-to-one, although at higher ratios the gloss decreases. The gloss is also increased by increasing the thickness of the paint film. It is found to be unacceptably low, however, when the pigment content in the ball mill charge is too great, and at a pigment-to-binder ratio of six-to-one the gloss is almost unnoticeable. The gloss also decreases with increasing stoving temperature.

The degree of whiteness of the film increases with increasing pigment-to-binder ratios and increasing film thickness, but it decreases with increasing stoving temperature.

The hardness of the paint film increases with increasing amino resin content and increasing stoving temperature, but is not greatly affected by variations in the pigment-to-binder ratio.

A flow control additive improves flow characteristics and prevents sagging of the wet film; its presence in a controlled amount does not affect the main properties of the film.

Keywords

Type and classes of coatings and allied products

stoving finish

Raw materials:

binders (resins, etc.)

alkyd resin

melamine resin

water-soluble resin

prime pigments & dyes

titanium dioxide

Processes and methods primarily associated with manufacture or synthesis

ball mill

Properties, characteristics and conditions

primarily associated with dried or cured films

gloss

hardness

La pigmentation des combinaisons de résines alkydés et mélamines solubles à l'eau

Résumé

On a choisi un stable variété de dioxyde de titane du type rutile pour étudier l'effet qu'exerce la pigmentation sur les caractéristiques des films de peintures rendus par une résine alkyde soluble à l'eau. Jusqu'au rapport pigment/liant de 1,4 : 1, on trouve que le brillant s'augmente, bien qu'aux rapports plus élevés il se diminue. Le brillant s'augmente également où l'épaisseur du film de peinture est augmentée. Cependant, il se trouve à un niveau inacceptablement bas lorsque la teneur en pigment de la masse broyante dans le cas d'un broyeur à boulets est trop élevée, et le brillant est largement imperceptible au rapport pigment/liant de 6 : 1. Il se diminue également à mesure que la température d'étuvage s'augmente.

La blancheur du film s'augmente lorsque le rapport pigment/liant et l'épaisseur du film s'augmentent, mais elle se diminue à mesure que la température d'étuvage s'augmente.

La dureté du film de peinture s'augmente lorsque la teneur en résine amine et la température d'étuvage s'augmentent, mais elle n'est pas largement influencée par les variations du rapport pigment/liant.

Un adjuvant pour contrôler l'étalement améliore les caractéristiques d'étalement, et empêche la formation des coulures sur le film frais; sa présence à une quantité précise n'exerce aucune influence défavorable sur les caractéristiques principales du film.

Pigmentierung wasserlöslicher Alkyd-/Melaminharz kombinationen

Zusammenfassung

Eine beständige, rutile Type eines Titandioxidpigments wurde für Zwecke der Untersuchung der Auswirkungen von Pigmentierung auf die Eigenschaften von wasserlösliche Alkydharze enthaltenden Lackfarbenfilmen ausgewählt.

Dabei wurde gefunden, dass sich der Glanz mit ansteigendem Pigment-zu-Bindemittel-Verhältnis bis um 1,4 zu eins erhöht, obwohl der Glanz bei grösseren Verhältnissen zurückgeht. Der Glanz erhöht sich auch mit grösserer Filmdicke. Als unakzeptierbar niedrig wurde der Glanz auch gefunden, wenn bei der Kugelmühlenschickung der Pigmentgehalt zu hoch ist; bei einem Pigment zu Bindemittelverhältnis von sechs zu eins ist Glanz kaum feststellbar. Der Glanz vermindert sich ebenfalls mit ansteigenden Temperaturen bei Ofentrocknung.

Der Weissgrad des Films erhöht sich mit wachsendem Pigment zu Bindemittelverhältnis und grösserer Filmdicke, verringert sich aber mit ansteigender Temperatur bei Ofentrocknung.

Die Filmhärte erhöht sich mit grösserem Gehalt an Aminoharz sowie mit ansteigender Ofenhitze, wird jedoch durch Variationen im Verhältnis Pigment zu Bindemittel nicht wesentlich beeinflusst.

Zusatz eines Verlaufs kontrollmittels verbessert die Verlaufscharakteristika und verhindert Gardinenbildung beim nassen Film; ein solcher Gehalt hat, wenn in begrenzter Menge zugegen, keinen nachteiligen Einfluss auf die Filmeigenschaften.

Introduction

Refs. 1, 2

Pigmentation effects exhibit major differences when solvent-based systems are made water-soluble.

Non-reactive pigments which have low water absorption, low water-soluble content and little tendency to swell in water are preferred for use with the water-soluble alkyd resins prepared with trimellitic anhydride¹. Because of their reactivity, pigments of a highly basic nature, such as white lead, zinc oxide and zinc chromate, should not be used with water-soluble resins. Pigments of mildly basic nature, for example calcium carbonate, barium carbonate and basic lead silicochromate, can be used if careful attention is given to hydrolytic stability. Resin salt solutions tend to react with coated grades of titanium dioxide pigment, particularly those coated with polyamines. This interaction usually results in a steady thickening up of the paint on storage², or the formation at the bottom of the can of a hard pigment/resin layer, which will not redisperse.

It is important, therefore, when evaluating any particular water-soluble resin system, to choose a suitable specified grade of titanium dioxide pigment to give stable paints. Since white paints account for over 50 per cent of the total retail market, a stable titanium dioxide pigmented paint is essential for a water-soluble paint to be commercially viable.

In the present study, the titanium dioxide pigment "Tioxide R-CR", supplied by BTP Tioxide Ltd, was chosen for pigmentation of the water-soluble alkyd prepared, because this is one of the most stable white pigments produced. For all practical purposes it is chemically inert and it is not affected by organic or inorganic acids or alkalis at ordinary temperatures.

Experimental

Refs. 3-7

Titanium dioxide pigment "Tioxide R-CR", described already in the introduction and elsewhere³, was used throughout this study.

A water-soluble alkyd resin⁴ with the following components molar proportions was prepared: dehydrated castor oil (1); glycerol (1); phthalic anhydride (1.5); trimellitic anhydride (3.53); diethylene glycol (6.81). It was solubilised in water by adding triethylamine, and diluted to give a 50 per cent solution. The properties of the finished alkyd were as follows:

Acid value	46
Hydroxyl value	89
Diene number	17.6
Iodine value	41.25

"Cibamin M 100", supplied by CIBA-Geigy AG, is a solvent-free, methylated melamine formaldehyde condensate (polymethoxymethyl melamine¹).

"Bentone 34" and "Bentone 38" anti-settling agents are produced by Titan GmbH. They are gelling agents which have the properties of swelling and gelling in liquid systems.

Preparation of paint films

A spinner coater⁵ was used for paint application: a suitable panel is placed on the horizontal platform which can be

rotated at a uniform, controlled speed. After placing an excess of paint on the panel surface, the rotor is started spinning at a constant predetermined speed for a short period up to about 5 minutes. The plates are then left suitably supported in a nearly vertical position for half an hour to remove slowly the greater part of the volatile amine.

After this, the plates were stoved at the required temperature and for a set time.

Measurement of gloss

The instrument used was the Sheen Instruments Ltd glossmeter. It is used for determining the gloss and mattness of painted surfaces.

Determination of the percentage whiteness

The instrument used to determine whiteness was a Carl Zeiss Leukometer, which is used for the determination of the luminance factor, especially of the degree of whiteness for smooth surfaces and for powdery substances.

Mechanical tests

Impact resistance⁷ was determined using the Gardner impact tester. The weight of the steel rod is 1.81 kg. In this work, the round-nosed steel rod was raised to 160 on the scale. The elasticity⁷ was measured by bending the panel, with coated side up, over a rod 3.2 mm in diameter, attached to the impact resistance tester.

Results and discussion

The effect of pigment-to-binder ratio

The paint formulation was as follows:

<i>Formula 1</i>	Mill charge (g)
Titanium dioxide pigment	80
Alkyd resin (solids)	66.7
Melamine resin ("Cibamin")	13.3
Water mixture (15% t-butanol)	50.0
Antifoaming agent: silicone oil	0.2
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Pigment-to-binder ratio	1:1
Alkyd-to-amino resin ratio	5:1

The paints were prepared by ball milling for 12 hours.

The base paints were diluted with resin solution and water mixture to provide different ratios of pigment-to-binder (ranging from 0.8:1 to 0.1:1). Paint films were prepared using the spinning disk applicator as already described. Three panels were prepared for each ratio. The first panel was stoved at 120°C for 120 minutes, the second at 130°C for 120 minutes and the third at 150°C for 120 minutes. After the films had dried, their whiteness, gloss and hardness were measured. The results are given in Table 1 and shown graphically in Figs. 1 and 2.

Table 1
The effect of pigment-to-binder ratio on the gloss, whiteness and hardness of paint films
 Alkyd : Amino resin = 83.4 : 16.6

Run No.	Pigment-binder ratio	Stoving temperature (°C)	Gloss reflection (%)	Degree of whiteness (%)	Hardness (seconds)	Film thickness (μm)
1	0.4 : 1	120	35	72.6	9.5	30
2	0.5 : 1	120	41	80.8	9.5	30
3	0.6 : 1	120	53	83.5	8.0	40
4	0.7 : 1	120	60	83.8	9.5	50
5	0.8 : 1	120	76	83.3	8.5	50
6	0.4 : 1	130	28	69.2	12.0	40
7	0.5 : 1	130	32	78.8	14.0	40
8	0.6 : 1	130	35	82.5	10.0	40
9	0.7 : 1	130	37	83.8	12.0	50
10	0.8 : 1	130	55	82.2	11.0	50
11	0.4 : 1	150	22	51.0	22.0	40
12	0.5 : 1	150	27	64.2	22.0	50
13	0.6 : 1	150	30	68.2	18.0	50
14	0.7 : 1	150	32	70.7	19.0	60
15	0.8 : 1	150	49	70.1	20.0	60

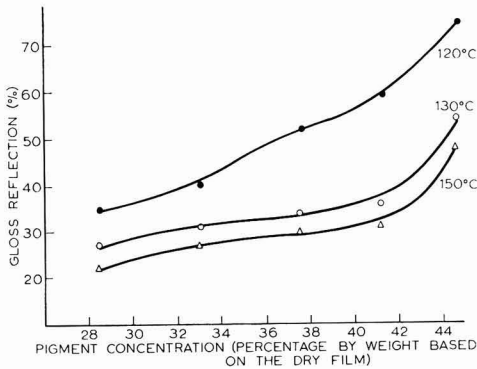


Fig. 1. Effect of pigment-to-binder ratio

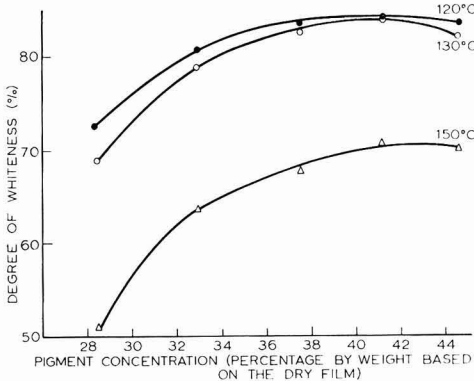


Fig. 2. Effect of pigment-to-binder ratio

The results show that the gloss increases with increasing pigment-to-binder ratio, but decreases by raising the stoving temperature. The hardness increases by raising the stoving temperature, but is not affected by variations in pigment-to-binder ratio. The whiteness increases with increasing pigment concentration up to about 38 per cent by weight pigment

in the dry film and then remains almost constant. The whiteness, however, is decreased by raising the stoving temperature.

The effect of water-soluble melamine resin

Alkyd/melamine resin combinations are widely used for forced-drying and stoving enamels in many industrial applications. The melamine part of the combination provides increased hardness and paler colour than could be achieved using alkyds alone.

Experiments were performed at the stoving conditions 120°C, 130°C and 150°C for 120 minutes using different alkyd/melamine combinations. The results are summarised in Table 2 and shown in Fig. 3, where it can be seen that the hardness increases with increasing percentage of amino resin, and by raising stoving temperature.

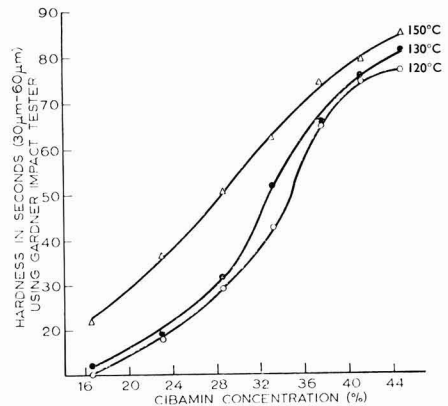


Fig. 3. Effect of increasing the melamine resin content

Study of the effects of different percentages of Bentone on film properties

Although the paint films described above had good gloss and whiteness, they were not without defects. Sagging occurred,

Table 2
The effect of various alkyd/melamine combinations on film hardness, elasticity and impact strength at different temperatures

Pigment : binder = 0.7 : 1

Run No.	Amino resin (%)	Temperature (°C)	Hardness (seconds)	Film thickness (μm)	Elasticity*	Impact strength*
16	16.6	120	10	50	p	p
17	23.0	120	18	60	p	p
18	28.5	120	29	50	p	p
19	33.3	120	43	50	p	p
20	37.5	120	66	55	p	p
21	41.1	120	75	50	p	p
22	44.4	120	77	55	p	p
23	16.6	130	12	50	p	p
24	23.0	130	19	50	p	p
25	28.5	130	32	50	p	p
26	33.3	130	52	50	p	p
27	37.5	130	65	60	p	p
28	41.1	130	76	50	p	p
29	44.4	130	82	55	p	p
30	16.6	150	22	60	p	p
31	23.0	150	37	85	p	p
32	28.5	150	51	90	p	p
33	33.3	150	63	80	p	p
34	37.5	150	75	55	p	p
35	41.1	150	80	50	p	p
36	44.4	150	85	65	p	p

*p = Passed test

particularly in thick films. Tests were performed to examine the effect of Bentone as anti-sagging agent. The results are summarised in Table 3. It can be seen that Bentone does not affect gloss or hardness, and the degree of whiteness is only slightly lowered (by not more than one per cent). Sagging did not occur in all the paint films containing Bentone.

Table 3

Properties of films after adding Bentone

Pigment : binder = 0.7 : 1
Amino resin = 40% (on weight of total resin)
Stoving conditions = 130°C for 120 minutes

Run No.	Bentone (%)	Gloss reflection (%)	Degree of whiteness (%)	Hardness (seconds)	Film thickness (μm)
37	0	71	82.3	75	50
38	0.5	67	82.2	74	50
39	1.0	69	82.1	73	50
40	1.2	70	81.9	75	50
41	1.4	66	80.8	65	50
42	1.7	68	81.0	66	50
43	2.0	71	81.3	71	50

Appearance of the films : Sagging disappeared from all paint films containing Bentone.

The effect of barytes (barium sulfate)

From an economic point of view, it is an advantage to replace part of titanium dioxide by barytes, since it is cheaper.

Tests were made with different percentages of barytes and stoving was carried out at 130°C for 120 minutes. The results are summarised in Table 4 and shown in Fig. 4.

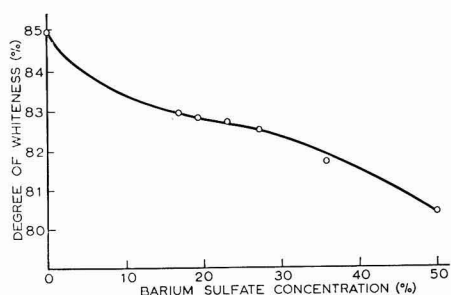


Fig. 4. Effect of partial replacement of titanium dioxide by barytes

Table 4

The effect of barytes on the properties of paint films

Ball mill charge pigment : binder = 1 : 1
Pigment : binder = 0.6 : 1 in final paint
Amino resin = 40% (on weight of total resin)
Stoving conditions = 130°C for 120 minutes

Run No.	Ratio of TiO ₂ to barytes	Gloss reflection (%)	Degree of whiteness (%)	Hardness (seconds)	Film thickness (μm)
44	50 : 50	39	80.6	59	60
45	64.3 : 35.7	39	81.8	62	60
46	72.3 : 27.2	36	82.6	62	60
47	77 : 23	39	82.8	56	60
48	80.8 : 19.2	37	82.9	58	60
49	83.3 : 16.7	39	83.0	60	60
50	100 : 0	39	85.0	56	60

The effect of the ball-mill charge

In this study, the formulation was as follows:

Formula II	Mill charge (g)
Titanium dioxide	240
Alkyd resin (solids)	80
Water mixture (15% butanol)	80
Gel Bentone 10% (0.5% weight of pigment)	12
Disperser N	0.48
Sodium hexametaphosphate	0.48
Silicone oil (French 470)	0.116
Pigment: binder ratio	3:1

The paint was milled for 24 hours. The base paint was adjusted to give the required composition.

Effect of pigment-to-binder ratio

The base paint was diluted with the required amounts of alkyd and amino resin solutions and water to give seven different ratios of pigment to binder in the range 0.4 : 1 to 1.6 : 1. Paint films were prepared using the spinning disk applicator as described earlier. Three panels were prepared for each pigment: binder ratio. One series of panels representing all the ratios was stoved at 120°C for 60 minutes, the second series at 130°C for 60 minutes and the third at 150°C for the same period of time. After drying of the panels, the degree of

From a comparison of Table 1 with Table 5 it can be seen that the gloss decreases with increasing pigment content in the ball-mill charge (formula I mill charge, pigment-to-binder = 1 : 1; formula II, pigment-to-binder = 3 : 1). In formula I sufficient binder is adsorbed on to the surface of the pigment, whereas in formula II the binder content is not sufficient to give optimum gloss.

The effect of grinding time

A sample was taken every 6 hours during the milling of formula II. The amino resin content was 40 per cent (on weight of alkyd). The paint was adjusted to the required viscosity and the panels were stoved at 130°C for 60 minutes. The results are given in Table 6.

Table 6
The effect of time of grinding on the properties of paint films
(pigment : binder = 1:1)

Test Number	Time of dispersing (hours)	Gloss (%) reflection)	Degree of whiteness (%)	Hardness (sec.)	Film thickness (µm)
73	6	59	83.7	84	50
74	12	62	84.2	83	50
75	18	66	83.85	80	50
76	24	62	83.95	75	60

From this Table it can be seen that 12 hours is sufficient for good dispersion.

Table 5
The effect of pigment-to-binder ratio on the properties of paint film

Milling time 24 hours Solids content 60% Amino resin 40% (on weight of total resin) Stoving time 60 minutes Bentone 0.5% (on weight of pigment)						
Run No.	Pigment-binder ratio	Stoving temperature (°C)	Gloss reflection (%)	Degree of whiteness (%)	Hardness (seconds)	Film thickness (µm)
51	0.4 : 1	120	23	78.9	72	60
52	0.6 : 1	120	33	82.5	69	60
53	0.8 : 1	120	36	84.5	80	60
54	1 : 1	120	68	84.2	76	60
55	1.2 : 1	120	72	84.9	77	60
56	1.4 : 1	120	73	85.9	76	60
57	1.6 : 1	120	49	85.0	77	60
58	0.4 : 1	130	15	78.3	73	60
59	0.6 : 1	130	20	82.65	80	60
60	0.8 : 1	130	30	84.32	83	60
61	1 : 1	130	66	82.95	80	60
62	1.2 : 1	130	75	83.40	78	60
63	1.4 : 1	130	73	84.95	85	60
64	1.6 : 1	130	47	84.0	83	65
65	0.4 : 1	150	15	79.6	73	60
67	0.6 : 1	150	33	82.5	87	60
68	0.8 : 1	150	35	84.2	85	60
69	1 : 1	150	67	84.3	90	55
70	1.2 : 1	150	70	83.95	87	60
71	1.4 : 1	150	73	84.9	86	60
72	1.6 : 1	150	32	83.55	94	50

whiteness, the gloss and the hardness were measured. The results are summarised in Table 5.

It was found that the gloss increased with increasing pigment-to-binder ratios up to 1.4 : 1 and then decreased when the ratio of pigment to binder was further increased. The whiteness increased with increasing percentage of pigment and the hardness increases with increasing stoving temperature.

The effect of amino resin content

Tests were performed at a stoving temperature of 130°C for 60 minutes using different ratios of amino to alkyd resin. The results are summarised in Table 7.

It can be seen that the hardness, as well as the gloss and the degree of whiteness increase by increasing the percentage of amino resin in the formulation.

Table 7
Effect of amino resin content (pigment : binder = 1:1)
Bentone = 0.5% (on weight of pigment)

Test number	Amino resin (%)	Degree of whiteness (%)	Gloss (%) reflection)	Hardness (sec.)	Film thickness (μ m)
77	10	77.6	—	Tacky	—
78	20	80.8	40	53	35
79	30	82.40	47	80	35
80	40	82.95	60	70	65
81	50	83.80	55	97	45

All films in these tests were affected by the alkali resistance test, but were not affected by the acid resistance test, except that the adhesion of a few films at low stoving temperatures and short times was reduced. From the results it can be concluded that the addition of small amounts of Bentone does not affect the main properties of the paint films. Some paint films were affected by boiling water, namely, those which were stoved at 150°C for 45 minutes, and those stoved at 130°C for 45 and 60 minutes. This may be due to the presence of small, but significant amounts of base paint in the films.

The effect of pigment-to-binder ratio

The paint formulation in this study was as follows:

Formula III	Mill charge (g)
Titanium dioxide	240
Alkyd resin (solids)	40
Water mixture (15% T-butanol)	100
Bentone 10% (1% on weight of pigment)	24
Disperser N	0.48
Sodium hexametaphosphate	0.48
Silicone oil	0.116
Pigment-to-binder ratio	6 : 1

The paint was dispersed for 12 hours. The base paint was diluted with the alkyd resin, amino resin and water mixture to give different ratios of pigment to binder (from 1.6 to 0.4 : 1). Films were prepared and stoved at 130°C and 150°C for 60 minutes. The results are summarised in Table 9.

Table 8
The effect of varying the concentration of Bentone on the properties of the paint film

Pigment : binder = 1 : 1
Amino resin = 30% (on weight of total resin)

Run No.	Bentone (%)	Stoving temperature (°C)	Stoving time (minutes)	Hardness (seconds)	Film thickness (μ m)	Boiling water resistance*	Elasticity and impact strength*
82	0	150	45	46	30	A	p p
83	0	150	60	50	30	NA	p p
84	0	150	90	60	30	NA	p p
85	0	150	120	65	35	NA	p p
86	0.25	130	45	40	30	A	p p
87	0.25	130	60	44	25	A	p p
89	0.25	130	90	47	35	NA	p p
90	0.25	130	120	49	30	NA	p p
91	0.25	150	45	44	25	A	p p
92	0.25	150	60	51	30	NA	p p
93	0.25	150	90	63	35	NA	p p
94	0.25	150	120	67	25	NA	p p
95	0.5	130	45	38	30	A	p p
96	0.5	130	60	38	35	A	p p
97	0.5	130	90	44	35	NA	p p
98	0.5	130	120	47	35	NA	p p
99	0.5	150	45	38	40	A (slightly)	p p
100	0.5	150	60	38	35	NA	p p
101	0.5	150	90	50	40	NA	p p
102	0.5	150	120	59	40	NA	p p
103	0.75	130	45	45	40	A	p p
104	0.75	130	60	60	42	A	p p
105	0.75	130	90	90	45	NA	p p
106	0.75	130	120	—	—	NA	p p
107	0.75	150	45	48	25	A	p p
108	0.75	150	60	52	25	NA	p p
109	0.75	150	90	—	—	NA	p p
110	0.75	150	120	68	25	NA	p p
111	1.0	130	45	38	25	A	p p
112	1.0	130	60	40	30	A	p p
113	1.0	130	90	45	30	NA	p p
114	1.0	130	120	48	30	NA	p p
115	1.0	150	45	38	40	A	p p
116	1.0	150	60	43	35	NA	p p
117	1.0	150	90	52	30	NA	p p
118	1.0	150	120	56	35	NA	p p

*A = Adhesion affected; NA = Adhesion not affected; p = Passed test

Table 9
Effect of increasing pigment-to-binder ratio
Ball-mill charge Pigment : binder = 6 : 1
Bentone = 1% (on weight of pigment)
Stoving time = 60 minutes

Run No.	Pigment binder ratio	Amino resin (%)	Stoving temperature (°C)	Gloss reflection (%)	Degree of whiteness (%)	Hardness (seconds)	Film thickness (μm)
119	0.6 : 1	40	130	4	82.45	71	35
120	0.8 : 1	40	130	3	82.95	76	30
121	1 : 1	40	130	3	84.1	78	40
122	1.2 : 1	40	130	3	82.5	83	40
123	1.4 : 1	40	130	3	84.3	76	40
124	1.6 : 1	40	130	2	81.3	90	35
125	0.6 : 1	40	150	1	80.60	79	35
126	0.8 : 1	40	150	2	82.9	82	30
127	1 : 1	40	150	2	82.9	82	30
128	1.2 : 1	40	150	2	82.55	90	30
129	1.4 : 1	40	150	2	81.3	95	30
130	1.6 : 1	40	150	2	81.2	96	35
131	0.6 : 1	30	130	—	—	—	—
132	0.8 : 1	30	130	3	81.35	42	30
133	1 : 1	30	130	3	81.35	50	30
134	1.2 : 1	30	130	5	83.5	46	40
135	1.4 : 1	30	130	2	82.8	59	35
136	1.6 : 1	30	130	2	83.8	68	35
137	0.6 : 1	30	150	6	80.1	63	40
138	0.8 : 1	30	150	4	82.25	65	45
139	1 : 1	30	150	3	82.75	69	40
140	1.2 : 1	30	150	3	81.75	69	50
141	1.4 : 1	30	150	3	83.65	70	40
142	1.6 : 1	30	150	2	82.35	69	40

From Tables 1, 3 and 9 it can be seen that the gloss decreases with increasing pigment-to-binder ratio in the mill charge. In case of the ratio 6 : 1 the paint films are matt and this is due to there being insufficient resin in the ball mill charge to wet the pigment particles.

Conclusions

Gloss increases with increasing pigment-to-binder ratio up to 1.4 : 1 and then decreases at higher pigment-to-binder ratios. The gloss also increases with increasing film thickness of the paint. The gloss is greatly decreased when the pigment content in the ball-mill charge is too high; the gloss almost disappears at a pigment-to-binder ratio of 6 : 1. The gloss is also decreased by increasing the stoving temperature.

The degree of whiteness increases with increasing pigment-to-binder ratio, but decreases with increasing stoving temperature. It also increases with increasing film thickness.

The hardness increases with increasing percentages of

amino-resin and increasing stoving temperature, but is not greatly affected by variations in the pigment-to-binder ratio.

Bentone improves the flow characteristics and prevents sagging of the wet film; its presence does not affect the essential properties of the dry film.

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The packaging of hazardous goods*

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Summary

The United Nations recommendations on the packing of dangerous goods are discussed with reference to their possible adoption by the Inter-governmental Maritime Consultative Organisation

(IMCO), European road and rail transport organisations (ADR and RID conventions), and other authorities.

Keywords

Specifications, standards and regulations

labelling laws and regulations

Miscellaneous terms

accident prevention

safety

Le conditionnement des marchandises dangereuses

Résumé

On discute les recommandations des Nations Unies à propos du conditionnement des marchandises dangereuses au point de vue de leur adoption éventuelle par la Inter-governmental Maritime

Consultative Organisation (IMCO), les organisations de transports ferroviaires et routiers en Europe (ADR et RID conventions), et autres organisations officielles.

Das Verpacken riskanter Güter

Zusammenfassung

Eine Besprechung der United Nations Vorschläge für die Verpackung riskanter Güter unter Bezugnahme auf die mögliche Adoption seitens der Intergovernmental Maritime Consultative

Organisation (IMCO), Europäischen Strassen- und Eisenbahnen-Transportorganisationen (ADR und RID Konventionen) sowie anderer Amtsstellen.

Introduction

The exporter of hazardous products has always been in a difficult position because of the differing standards required by the various national and international authorities. For example, in Britain it is necessary to comply with the appropriate road conveyance regulations when transporting a package to the port, then to meet the requirements of the "Blue Book" of the Marine Division of the Department of Trade before the same package can be put aboard ship. In addition, if the exports are to the USA or to Canada, a heavy penalty can be incurred if the requirements of the USA Department of Transportation (or the Canadian Transport Commission) are not met. Similarly, international journeys in Europe are governed by the RID or ADR conventions, which relate to international through-transport by rail and road, respectively. So far as air travel is concerned, the International Air Transport Association (IATA) has Restricted Articles Regulations, which have the force of law in many countries.

Only the more important regulations are mentioned here, but in addition there are many national requirements throughout the world. Not only do the regulations governing packaging differ, but the classification of the goods themselves varies in certain details.

United Nations

Final responsibility for hazardous goods within the United Nations rests with the Economic and Social Council

(ECOSOC) on which all members of the UN have a seat. Recognising the fact that differing requirements created serious obstacles to international trade, ECOSOC authorised work several years ago with the objective of harmonisation with regard to regulation of hazardous goods. This work continues and is the responsibility of "The Committee of Experts on the Transport of Dangerous Goods" on which committee about eight nations, including the UK, serve. To this Committee of Experts respond two groups: these are "The Group of Rapporteurs on the Packing of Dangerous Goods" and "The Group of Experts on Explosives".

The UN Committee of Experts' recommendations on the classification, packing and marking of dangerous goods were published in 1970 and, hopefully, they will eventually be the basis of all international and national legislation.

The UN work classifies products into one of nine classes, for example, Class 3—"Inflammable Liquids", and Class 8—"Corrosives", every one of which has its own symbolic label. These symbolic labels have already been adopted by the Intergovernmental Maritime Consultative Organisation (IMCO), the RID and ADR Conventions, and the USA Department of Transportation.

They are required to be used for all shipments from UK ports or in British ships, and for all road movements within the UK of inflammable liquids, corrosive products and organic peroxides.

*Paper presented to a meeting of the Thames Valley Section on 29 January 1976. Note: Readers will know that it is the policy of this *Journal* to follow, wherever practicable, the recommendations of the International Union of Pure and Applied Chemistry (IUPAC) regarding the style for chemical symbols and formulae, SI units and spelling. Certain concessions have been made in this paper, however, where the author is quoting from an organisation or standard which does not follow the IUPAC recommendations. An appropriate conversion to SI units is given where appropriate in the text for the benefit of readers used to this system, but it should be noted that in these cases the SI equivalent is merely an editorial feature and it does not necessarily form part of the original regulation or standard being quoted.

Package performance tests

Possibly of greatest interest is the work of the United Nations on the performance tests for packages designed to carry hazardous goods. The real merit of the UN recommendations is that they are based on performance only and not on the use of specified thicknesses of given materials. Detailed tests are set out for each of twenty different transport packages and these "type tests" are to be carried out "at the start of production . . . of each design, size, gauge and manner of construction" and repeated after each modification of design and at intervals during production, to ensure that the requisite standards are being maintained. On all twenty package types, drop tests and stacking tests have to be carried out. In addition, where appropriate to the package design and intended contents, air leakage tests and hydraulic pressure tests are also required.

The UN requirements can best be illustrated by referring to metal and plastic drums for liquids, and fibreboard cases containing tins or bottles of similar liquids.

Testing of drums

Drop test

Six drums are filled to 98 per cent of their capacity. The first three metal drums to be dropped so that they strike the target diagonally on the chimb, or if the receptacle has no chimb, on a circumferential seam. The second group of three drums are to strike the target on the weakest part not tested by the first drop, for example a closure or, for some cylindrical drums, the welded longitudinal seam of the drum body.

The normal drop height is 1.2m and the normal content of the test drums is water, the results being valid for all liquids up to a specific gravity of 1.2. Where liquids with a density greater than 1.2 are to be packed, there are two options: testing with the actual contents from 1.2m, or testing with water from a height in metres equal to the specific gravity of the liquid to be carried, this value being rounded up to the first decimal place.

Plastic drums are tested in a similar way to metal drums but they are required to be pre-conditioned so that the drum and contents is reduced to a temperature of -18°C (0°F), at which temperature the drop testing must take place. This necessitates the use of an anti-freeze solution.

Stacking test

Where metal drums are concerned, three specimens are to be tested for a period of 24 hours with a weight placed on a flat surface resting on top of the package and equivalent to the total weight of identical packages which could be stacked on it during transport. In general, the stacking height to be taken into consideration is 3m, but for sea transport, where the packages are not stowed on deck or in a freight container, the height to be taken into consideration is 8m.

The requirements for plastic drums are more onerous in that the test is of 28 days duration and has to be carried out at 40°C . For all practical purposes the maximum stacking height is 3m since 8m is beyond the economic design possibilities of plastic drums.

Air leakage test

Closed-end metal drums and removable-head metal drums (unless they are destined to contain solids which do not react dangerously with moisture) are required to withstand, without leakage, an air pressure of at least $0.2\text{kg}/\text{cm}^2$ (that is, 19.6133 kPa, or approximately 0.2 atmospheres pressure). Not only is this provided for as a type test, but all drums must be so tested. Provision is made for drums to be reused where they have been inspected and found free from corrosion or other damage, and have been retested to an air pressure of at least $0.2\text{kg}/\text{cm}^2$.

Plastic drums have to be tested in a similar manner, but there is an anomaly in that, apparently, there is no exemption for removable head drums destined to contain solids which do not react dangerously with water.

Hydraulic pressure test

Three metal drums are to be tested for a period of five minutes to a hydraulic pressure not less than the total pressure (that is, the vapour pressure plus the partial pressures of any inert material present, if any) which could be developed by the contents at the highest temperature likely to be met during carriage, multiplied by a safety factor of 1.5. The highest temperature is deemed to be reached at 55°C . The drums are not to be mechanically supported during the test.

Similar tests are to be carried out on plastic drums, but to allow for the possible "cold flow" it is required that the pressure be applied and maintained for a period of 30 minutes.

Testing of fibreboard cases

No physical testing is carried out on the inner packages, whether these be tins or bottles, but they must be filled to 98 per cent of their capacity, closed and placed in the outer fibreboard case which, in turn, must be closed ready for transport. The packages to be tested are required to be conditioned for at least 24 hours in an atmosphere maintained at 65 per cent relative humidity and at a temperature of 20°C .

Drop test

Five cases are to be used, one for each of the following drops from a height of 1.2m: flat drop on bottom of case; flat drop on top of case; flat drop on one long side of case; flat drop on one short side of case; drop on to the shortest edge of the case.

There should be neither leakage from, nor serious rupture of any of the tested cases or of the inner receptacles.

Stacking test

Three cases are to be tested for a period of 24 hours with a weight placed on a flat surface resting on the top of the case and equivalent to the total weight of identical packages which could be stacked on it during transport. In general, the stacking height to be taken into consideration is 3m, but for sea transport, where the packages are not stowed on deck or in a freight container, the height to be taken into consideration is 8m.

Implementation of the UN recommendations

IMCO decided at an early date to follow in principle the UN recommendations and the test procedures outlined above are published in Annex I to the International Maritime Dangerous Goods Code. The requirements have already been imple-

mented by the authority responsible in the UK (the Standing Advisory Committee of the Department of Trade) for all new applications for permission to ship new hazardous products, or established products in packages not previously provided for in the "Blue Book".

The RID and ADR authorities have already intimated that their regulations will eventually be brought into line with the UN recommendations and, whilst this may take two or three years to complete, the test requirements for drums for inflammable liquids have already been changed to comply with the UN tests.

The packaging of hazardous goods for movement by road in the UK is likely to be controlled in the future by a series of Codes of Practice. That for organic peroxides has already been published and those for inflammable liquids and corrosive products are at an advanced stage of drafting. What is not clear is when this work will be resumed, following transfer of responsibility to the Health and Safety Executive, and whether UN type testing will be required at least for all new products/packages.

Classification by degree of danger

The UN Committee of Experts has now refined the system of classifying products by type of hazard, and within each class there is a sub-division of products by degree of hazard. In effect, a number of extremely hazardous products has been designated "Group I" and a number of products with a relatively minor hazard as "Group III". The vast majority of products within each hazard class (that is, those not specifically placed in Group I or Group III) are designated "Group II".

For example, within Class 8 "Corrosives" there are:

- Group I — Fuming sulphuric acid
- Group II — Commercial grades of sulphuric acid
- Group III — Filled electric storage batteries

The importance of this classification by degree of hazard is that some of the test requirements are modified. For Group I products, the severity of the drop and air pressure tests is increased by a factor of 1.5 and certain minimum requirements are stipulated for hydraulic pressure. For Group III products, there is a reduction in the height of the drop test by a factor of 1.5. The effect of these modifications is shown in Table 1, firstly for all products with a density not exceeding 1.2, and then for a product with a specific gravity of 1.59 where water is used as the test medium.

Table 1
Modifications to drop test due to degree of hazard

Hazard rating	Drop height— water fill (Specific gravity (SG) ≤ 1.2)	Drop height— water fill (SG 1.59 rounded up to 1.6)
Group II (Standard)	1.2 m	1.6 m
Group I (multiply by 1.5)	1.8 m	2.4 m
Group III (divide by 1.5)	0.8 m	1.06 m

These new recommendation have not yet been adopted by any national or international authority, but the IMCO Subcommittee on the Carriage of Dangerous Goods at its September 1975 meeting decided to do so.

Hazards of transport

Just as different products involve different degrees of hazard, so different types of journey pose different degrees of risk to the packages which are being transported. Every time a package is handled it is at risk and so if in a journey to the customer it is loaded only once on to a vehicle and unloaded at the destination, with no intermediate handling, the package stands a far better chance of arriving undamaged than if it is shipped overseas in a conventional break-bulk cargo vessel. It is, therefore, the considered opinion of the Chemical Industries Association (CIA) that this well-substantiated fact should be taken into account and the drop-test modified for "through loads", for example direct road or rail delivery within the UK and Europe, and full freight container loads (FCLs) shipped overseas. Representations to this effect have been made to the appropriate UK government departments and it is hoped that suggestions in Table 2 will be given realistic and sympathetic consideration.

Table 2
CIA recommendations for modifications to the drop-test for direct delivery journeys

Hazard rating	Drop height (m)		
	Marine shipment —break bulk	Marine shipment —FCLs	Direct road/rail
Group I	1.8	1.2	1.2
Group II	1.2	0.8	0.8
Group III	0.8	0.6	0.6

Similarly, certain types of unit load, for example a box pallet, can afford considerable protection to the packages contained in the load and in such instances a case can be argued for reducing the severity of both the drop test and stacking test on the individual packages. Alternatively, there is scope for the development of tests on the complete unit load.

Outstanding problems

Despite the very real progress which has been made, the complexity of the task of unifying procedures is such that there are inevitably a number of outstanding problems. These may be divided into three broad groups:

Problems of implementation.

Requirements additional to the UN recommendations.

Packages well proven in use, which will not meet the new criteria.

Implementation of the UN recommendations by all international and national authorities is essential if the full benefits are to be derived. In this regard there are two main areas of difficulty—the time-scale, and universal recognition of nationally approved packages. For companies in the UK, who already have to comply with the UN system in order to ship their products overseas, it is counterproductive to comply with other different regulations, such as the American DOT or RID/ADR, which are unlikely to be changed for another two or three years at the earliest. Additionally, there is a disturbing tendency for nations to require the packages to be re-tested (or at least registered) in their country, and early agreement is essential on an internationally acceptable mark which will ensure that officially tested and approved packages are not held up at some point on their journey overseas.

There is also concern over the possibility that some authorities will regard the UN tests as the minimum standards, and that other more onerous requirements will be added to them. The support which the British chemical industry (and others) gave to the UN work over the past decade was based on the belief that the UN recommendations would be the norm, compliance with which would ensure acceptance by all authorities. If this is not to be the case, then the original objective of facilitating the movement of goods will not have been achieved.

Finally, there is concern in several industries that packages well proven in use will not survive the prescribed tests. Whilst it would be unrealistic for industry to claim that all packages used for hazardous goods have been satisfactory at all times, this is a problem which deserves, and is receiving, careful consideration. To change the form of the package, or to increase its strength, where the safety record of the existing package is good needlessly increases costs and puts the UK at a competitive disadvantage in the world marketplace. Particular attention has been drawn to the problem of household chemicals, which are usually packed in tins or bottles overpacked in fibreboard cases. The IMCO Sub-Committee on the Carriage of Dangerous Goods has proposed—subject to ratification by the Maritime Safety Committee—that where hazardous goods are packed in:

- (a) very small packages, they be totally exempt from the provisions of the IMCO Dangerous Goods Code.
- (b) slightly larger packages, the Competent Authority may allow a lower standard of receptacle and outer package than that required for the same substances in the Code, provided it is satisfied that such receptacles and packages are safe and suitable for transport. In such instances, the package should as a general rule comply with UN Group III performance tests, but the Competent Authority may waive this requirement. The maximum permissible size of such a package will vary according to the hazard class and group (Table 3).

Table 3
Examples of maximum net volume per receptacle

Type of exemption	Class 3 Group II Inflammable liquids	Class 8 Group II Corrosive liquids
Total exemption	50 ml*	2 ml*
Reduced packaging standard	3 litres*	1 litre*

*One litre equals 1.000 028 dm³ (for the purposes of the 1963 Weights and Measures Act).

How this will ameliorate the position only time will tell, but it is noteworthy that the long-established American DOT regulations totally exempt from package testing (provided they are packed in "strong outside containers") the following small packages:

Flammable liquids: Metal containers not over 1 US quart (0.95dm³) capacity; other containers not over 1 pint (0.475dm³) capacity or 16 oz (0.453kg) by weight.

Corrosive liquids: Glass bottles, not over 1 lb or 16 oz by volume, each enclosed in a metal can; metal or plastics packages not over 1 lb or 16 oz by volume.

Conclusions

There has been much ill-informed criticism of the UN recommended package performance tests but, provided the position of certain well-proven packages can be adequately safeguarded, their universal adoption by all national and international authorities should be a priority. Until this objective has been substantially achieved it would be helpful if all work on further proposals, such as the penetration test favoured by the Department of the Environment or the chemical testing of plastic drums in Germany, could be suspended; without such a period of adjustment and consolidation it is likely that the position of industry in the short term will be worse than it was when the international work first commenced.

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The influence of metal surface structure on the adhesion of thermosetting resin*

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Summary

Metals are pretreated before painting or adhesive bonding so that the protected surface or the adhesive joint will have a satisfactory service life. The preparation of aluminium for either painting or bonding by chemical etch may involve the same method—that is, chromic-sulfuric acid etching. Moreover, a durable paint and suitable bonding adhesive for this substrate are likely both to be based on the same type of thermosetting resin system.

Keywords

Types and classes of structures or surfaces to be coated

aluminium

Properties, characteristics and conditions primarily associated with dried or cured films

adhesion test

This paper discusses the influence which minor constituents in the etch bath can have upon the strength of the aluminium-to-resin bonds. The etch process is compared with less rigorous treatments.

The work described has been governed by the concepts of adhesive bonding, but it is concerned with several factors at the resin/metal interface which also control surface coating phenomena.

Processes and methods primarily associated with:

analysis, measurement or testing

peel test

surface preparation before coating

chemical pretreatment

chromate pretreatment

L'influence qu'exerce la structure de la surface du métal sur l'adhérence d'une couche de résine thermodurcissable

Résumé

Les métaux sont pré-traités avant d'être peints ou d'être joints par adhésifs, afin que la surface protégée ou le joint aient une vie utile qui donne satisfaction. La préparation d'aluminium par gravure chimique, soit pour peinture ou jointure pourrait entraîner l'emploi de la même méthode, c'est-à-dire, la gravure par l'acide chromo-sulfurique. D'ailleurs dans le cas de ce sujet, une peinture durable et un adhésif convenable sont probablement basés sur le même type de résine thermodurcissable.

Dans cet exposé, l'auteur discute l'influence que sauraient exercer

les constituants peu importants de l'agent de gravure sur la tenacité des liens entre l'aluminium et la résine. On donne une comparaison du procédé de gravure auprès des traitements moins rigoureux.

L'étude ci-décrite a respecté les concepts de jointure par adhésifs, mais elle s'occupe d'ailleurs des divers facteurs régnant à l'interface entre la résine et le métal que exercent une influence sur le comportement des revêtements de surface.

Der Einfluss der metallischen Oberflächenstruktur auf die Haftung eines wärmehärtbaren Kunstharzfilms

Zusammenfassung

Damit die geschützte Oberfläche oder die durch Klebung erzeugte Verbindungsstelle eine ausreichende Lebensdauer besitzt, werden Metalle vor Beschichtung mit Anstrichmaterial oder Klebstoff vorbehandelt. Für die Vorbehandlung von Aluminium, sei es für Anstrich oder für Klebung mit Hilfe eines chemischen Beizmittels, mag dieselbe Methode—und zwar Chromsäure-Schwefelsäureätzung—angewandt werden. Weiterhin werden wahrscheinlich beide, ein dauerhaftes Anstrichmittel und ein geeignetes Klebemittel, für dieses Substrat auf dem gleichen wärmehärtbaren Kunstharzsystem basieren.

In dieser Abhandlung wird der Einfluss, den nebensächlichere Bestandteile im Beizbad auf die Festigkeit der Aluminium-Harzverbindungsstellen haben können, besprochen. Das Beizverfahren wird mit weniger rigorosen Behandlungsmethoden verglichen.

Die geschilderte Arbeit wurde von den Begriffen haftender Bindung geleitet, ist jedoch an verschiedenen Faktoren der Harz/Metall Grenzfläche, welche ebenfalls die mit der Lackierung von Oberflächen zusammenhängenden Beschichtungsphänomene betreffen, interessiert.

Introduction

Refs. 1-7

Metal pretreatment processes can be placed in order of increasing rigour from no treatment at all, through washing techniques to remove contaminants, abrasion methods to remove undefined surface layers, up to chemical treatments which provide a fresh, defined surface on the metal. A British Standards Institution code of practice¹ collects many of these methods together.

This code of practice includes one accepted UK formulation for the etch treatment of aluminium prior to painting and bonding, which has its origin at least a quarter of a century earlier². This etch treatment is a mixture of sulfuric acid and chromium trioxide or sodium dichromate in water. An overall chemical reaction can be written for the process:



This is a formal statement that in the etch bath there are competing reactions of oxidation of the metal and of dissolu-

*Paper presented at a symposium of the Manchester Section held on 6 and 7 April 1976 at the University of Manchester Institute of Science and Technology.

tion of the metal. Dissolution is the more significant, since sheets of aluminium etched in the bath show a loss in weight and thickness.

Variants of this etch process, differing in starting formulae and metal exposure regime, exist in a number of national standards. Three are defined in Table 1.

Table 1
Etchant Standards

Quantity (g l ⁻¹) of ingredient	Country of origin of standard		
	USA ³	UK ⁴	UK ⁵
H ₂ SO ₄	285	350	275
Na ₂ Cr ₂ O ₇ ·2H ₂ O	29	95	—
CrO ₃	—	—	50
Temperature	65–71 °C	60–65 °C	60–65 °C
Etch time	10 min	30 min	30 min

In the UK standards^{4,5} some guidance is given upon the preliminary steps in the prebond process, that is removal of gross contaminants, and also on the control of the etch bath as it ages.

Control of the bath is most important. The bath is expected to have a long and also useful life, in the sense that the treated metal should give a consistent resin metal interface. The bond should always be strong and durable. Despite the long history of the formulation, definitions of conditions to give long life and consistent performance are incomplete.

The structure of the treated aluminium surface is a prime factor in determining the properties of the resin/metal bond. The development of electron transmission microscopy and, more recently, scanning electron microscopy, has given techniques by which the surface morphology of the metal can be defined. A model of the aluminium surface⁶ which is produced by the action of the US etch bath formulation³ is shown in Fig. 1*. It suggests a pitted oxide surface, about 40nm thick, upon the underlying metal.

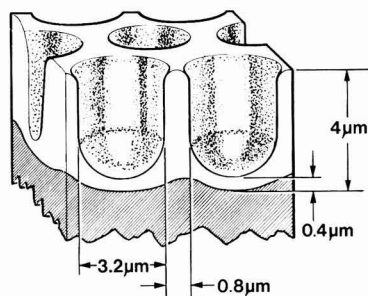


Fig. 1. Model of oxide produced on aluminium by FPL etch (after ref. 6)*

This structure was developed from a study of electron microscopy photographs. The same technique has been used in an extensive study upon etch baths made from chromium trioxide or sodium dichromate and sulfuric acid⁷. In this work, four types of aluminium surface after etching were distinguished:

1. Metal still with residual oxide of the original surface.
2. A smooth surface, free from residual oxide.

3. A surface with filiform oxidation along subgrain boundaries.
4. Surfaces with equally distributed, small etch pits.

The surface from 4 was preferred because with it the best adhesion was obtained.

To obtain the preferred form, 4, there was an optimum range of bath composition and regime: 200–300 g l⁻¹ sulfuric acid, 20–60 g l⁻¹ chromic acid, and an etch time of 10–30 minutes at 50–70 °C.

The surfaces 1–4 were regarded as a progression, the complete sequence being followed as the etch period progressed^{7c}. The formation of pits on the surface was regarded an electrochemical process^{7b}.

Adhesion was measured by testing the peel strength with a vinyl/phenolic structural adhesive "Redux 775" (Ciba-Geigy Plastics and Additives Company, Cambridge). The measurements were of metal-to-metal peel, and a sensitive indicator with the chosen resin system were used. The optimum etch conditions gave the maximum average value (in excess of 7 N mm⁻¹) with the lowest coefficient of variation.

Thus the study established that removal of uncontrolled oxide from the aluminium was not sufficient. The development of an optimum morphology was necessary to obtain good adhesion with a resin system.

Results and Discussion

Scope of work

The relation between adhesion and pretreatment with aluminium and thermosetting resins has been studied. The vinyl/phenolic system mentioned above has been used, but epoxide systems have been examined as well. Surface morphology has been studied by using scanning electron microscopy. Adhesion has been judged by metal-to-metal peel strength and lap-shear strength.

Three aluminium alloys have been used; BS 3L61, BS 3L72 and BS 3L73, the compositions of which are given in Table 2.

Table 2
Alloy compositions (principal elements, per cent:
remainder aluminium)

Alloy type	Metal (%)						
	Cr	Cu	Fe	Mg	Mn	Si	Zn
BS 3L61	0.05	0.1	0.7	0.1	1.2	0.6	0.1
BS 3L72*	0.3	4.3	1.0	0.7	0.8	0.8	0.2
BS 3L73*							

*Clad on both sides with almost pure aluminium

Five types of structure formed by the etch process have been established, even when preferred concentrations of sulfuric and chromic acids have been used:

- A. Surfaces with residual oxides.
- B. Etched impervious surfaces.
- C. Surfaces with filiform oxidation.
- D. Surfaces with fully developed porous oxide, much pitted layers.
- E. Choked oxide layers.

*See also *Errata* note on page 74.

In the author's experience there has not been a progression from *A* to *D*. As the initial oxide is dissolved, a developed structure is revealed. Type *A* is the trivial structure, where residues of the original fabrication oxide layer still remain when the metal has been removed from the bath prematurely. The final form of structure is dependent on minor constituents in the bath and on the type of alloy used.

Type *A* can be avoided by ensuring the bath is at the proper temperature and strength.

When bonds are made up with thermosetting systems it is found that adhesion, as shown by peel strength, is irregular with type *A*, very low with type *B*, improved with type *C* and satisfactory with *D* and *E*.

Surface structure and bond strength

The surface morphology of etched aluminium depends on the influence of noble metals in the bath.

Table 3 shows the results of experiments to etch alloy BS 3L61. The control bath was made from "Analar" chromic acid and sulfuric acid. The efficiency of the etch was judged by the weight change (loss) and by the peel strength with bonding agent "Redux 775".

The pure bath gave very poor results. In workshop conditions steps are often taken to "sweeten" a new bath, for example by allowing scrap metal to dissolve in it. This approach was tested by adding soluble salts to give 500 ppm of metal ion in the acid solution.

These trace additions in most cases were ineffective. However, some did have a beneficial effect.

Table 3
Effect of added metal salts on etch rate and peel

Bath composition: 300 g l⁻¹ sulfuric acid
50 g l⁻¹ chromic acid

Etch regime: 30 min at 62 C

Addition	Group	Electropotential (V)	Etch (g m ⁻²)	T-peel* (N)
Nil (control)	—	—	3.5	35
Mg	IIa	-2.34	3.6	26
Mn	VIIa	-1.05	2.6	21
Fe	VIII	-0.44	3.8	23
Ni	VIII	0.25	3.2	21
Pb	IVb	0.14	6.0	122
Bi	Vb	+0.23	8.2	111
Cu	Ib	+0.35	7.0	166
Hg	IIb	+0.78	10.9	170
Ag	Ib	+0.80	13.2	178

*Mean of at least three specimens, 25 mm wide × 10 cm long

The data in the Table are arranged in order of ascending electropotential for the additives. The potentials are literature values relative to the hydrogen electrode in air. True values for the highly acid etch bath are not known.

From the Table it will be seen that cations with a nominal difference from aluminium larger than 1.81V had a beneficial effect on the etch process. The benefit was evidenced by a much increased etch weight during the immersion period and a least a fivefold increase in peel strength.

The etch weight was not the sole criterion which set the level of peel strength. With sufficiently rigorous conditions, such as increased time and/or temperature, an "ineffective" bath

could give an etch weight equal to that of an "effective" bath. The peel strength remained low.

Scanning electron microscopy showed that the ineffective etch had given an irregular impervious surface structure in all the ineffective baths. Fig. 3 is an example. This is a type *B* surface structure.

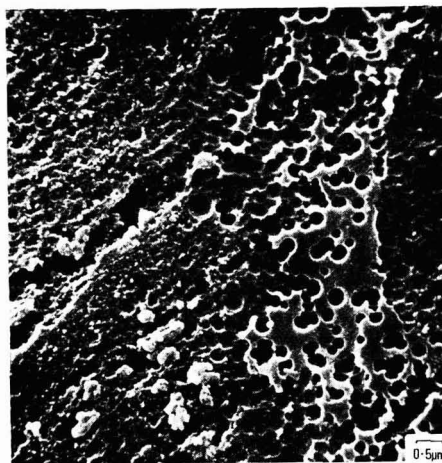


Fig. 2. Aluminium surface partially etched (3L61)

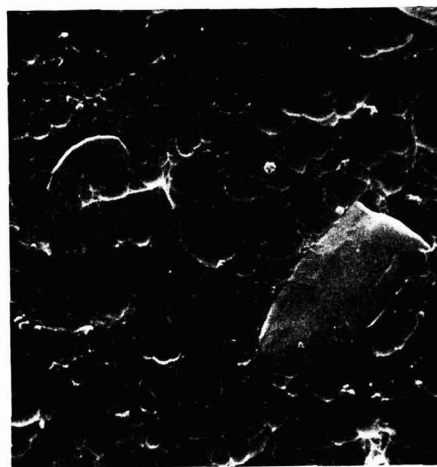


Fig. 3. Aluminium etched in an ineffective bath (3L61)

An active workshop bath typically gives a surface of type *D*. Fig. 5 is an example.

The experimental baths which were effective gave surfaces of type *C*, type *D* or type *E* or states intermediate between these.

Although the range of structures *A* to *E* were defined by these experiments with alloy BS 3L61, it was found that the classification could be applied to other alloys as well. The

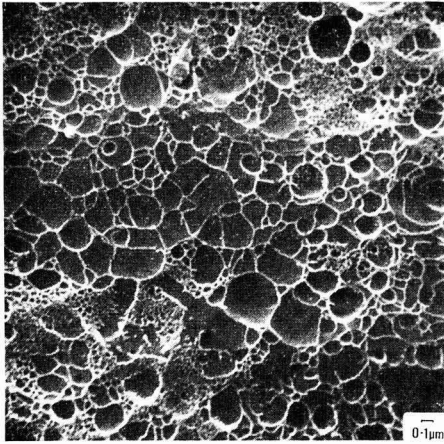


Fig. 4. Aluminium surface showing filiform oxidation (3L61)

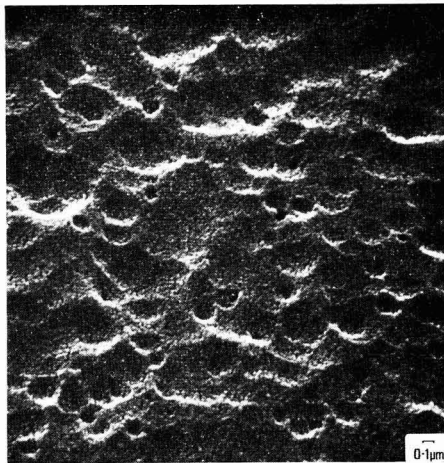


Fig. 5. Etched aluminium with fully developed porous surface (3L61)

alloy BS 3L72 is used in the Ciba-Geigy peel test. This method uses a specimen which is mounted on a drum. The outer bonded strip is drawn off over a roller to give control of the angle of peel. The geometry can be used with other alloys to compare the etch standard—for example, specimens made from BS 3L61 in place of BS 3L72. The comparison is shown in Table 4. Two etch baths were used, the pure control and the modified one containing copper salt.

Table 4
Effect of adherend on peel strength

Etch: 300 g l^{-1} sulfuric acid, 50 g l^{-1} chromium trioxide; 30 min at 62°C

Adherend:	"Alclad 3L72" (standard test)		3L61	
	none	Cu	none	Cu
Etch additive (500 ppm):	none	Cu	none	Cu
Etch weight (g m^{-2})	5.6	5.0	2.9	6.2
Ciba-Geigy peel (N)	268	332	40	199

In both baths, one expected to be ineffective, the other effective, the standard metal gave a surface showing good adhesion—that is, good peel strength. The BS 3L61 aluminium showed the behaviour seen in the T-peel tests: high peel only when the etch contained copper.

Scanning electron microscopy of the surface of the BS 3L72 alloy showed the same structure for specimens from either bath—a highly porous coral-like structure falling into class *D*. (Fig. 7). This behaviour may be explained by the composition of the alloy. Table 1 shows that BS 3L72 is a copper-bearing alloy. Although the alloy is clad with almost pure aluminium, the cut edges of the test sheet expose the copper-bearing metal. This is sufficient to give an effective etch.

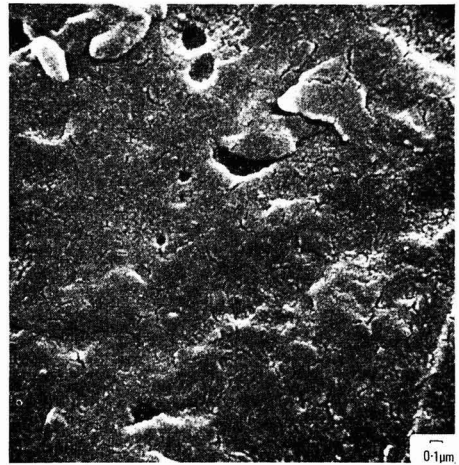


Fig. 6. Etched aluminium with choked surface (3L61)



Fig. 7. Etched aluminium with developed porous surface (3L72)

This hypothesis was supported by using test sheets of BS 3L72 which had the edge sealed with a silicone compound before the etch was carried out. Repeating the Ciba-Geigy peel test gave the results shown in Table 5.

Table 5
Effect of sealed Alclad panel on peel strength (conditions as Table 4)

Etch additive (500 ppm):	Adherend:	
	3L72 (edge sealed)	Cu
Etch weight (g m ⁻²)	4.3	5.9
Ciba-Geigy peel (N)	115	307

Photographs showed that the bath containing copper produced the porous surface of Fig. 7. The pure bath on the other hand, gave an impervious surface falling in the class of type B, shown in Fig. 8.

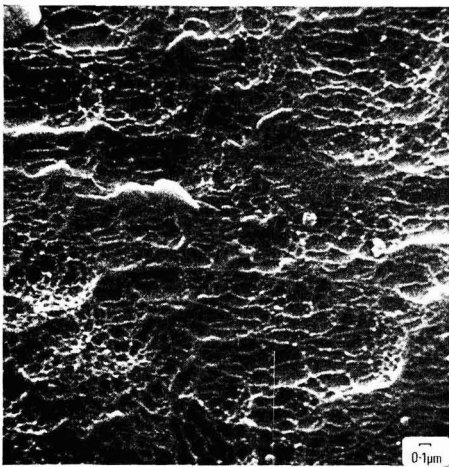


Fig. 8. Etched clad aluminium alloy with sealed edges (3L72)

These experiments with the vinyl/phenolic system showed that adhesion, as exhibited by the peel strength of metal-to-metal joints, was strongly affected by the morphology of the oxide film formed in the etch bath.

Surface structure and durability

Ref. 8

The influence of the metal surface structure on the sustained adhesion values was also examined. In these tests, epoxide systems were used. In the first series the epoxide was "Araldite AV 1566 GB" (Ciba-Geigy Plastics and Additives Company, Cambridge). This one-part adhesive is an aluminium-filled paste. Recommended cure time is 30 minutes at 150°C. It is based on liquid bisphenol A epoxide resin. The curing reaction is akin to that of certain epoxide powder coating formulations. Single lap-shear joints were made up with BS 3L73 "Alclad" alloy and the effect on strength measured by thirty days' immersion in water at 40°C. Three surface conditions were chosen: degreased only; gritblasted; and chromic-sulfuric etched. The degreased metal would still be covered by the oxide layer formed during fabrication. The gritblasted stage would remove about the same amount of substance as an etch bath, but the morphology would be different; this is shown in Fig. 9. The etched metal, prepared in a workshop bath, showed the surface structure of type D.

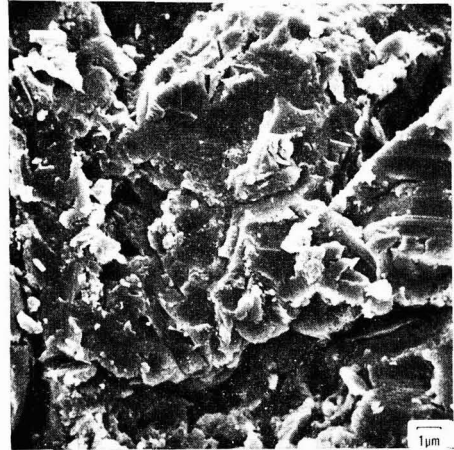


Fig. 9. Aluminium surface after gritblast treatment (3L73)

The results of the experiment are given in Table 6.

Table 6
Aluminium surface structure and resistance of adhesive lap-joints to water

Adhesive: Araldite AV 1566 GB; Adherend: BS 3L73 Alclad
Units: MPa at test temperature shown*

Surface condition	Breaking stress (MPa) at temperatures shown*			
	Initial value		After 30 days in water at 40°C	
	23 C	80 C	23 C	80 C
Degreased	19.0	22.3	2.3	2.3
Gritblasted	19.0	22.3	11.0	5.2
Etched	23.8	35.2	27.5	28.9

*Each entry is the mean of at least four specimens

The etched surface gave the highest initial and final results at both the 23°C and 80°C test temperatures. The margin was very great after the immersion test.

The changes which occurred during the immersion period can be separated into effects at the interface and effects on the cohesive properties of the resin layer. On the etched surface, the plasticisation of the resin layer by water shows a positive effect. The same factor will be operating on the other two surfaces, so that the loss in adhesion at the interface can be estimated as 11 MPa for the gritblast surface and 17 MPa for the degreased surface.

These experiments showed that long-term adhesive properties were also affected by the surface morphology. In addition, they indicated that adhesion was not merely a mechanical "keying", since the gritblast specimens were adversely affected.

In a second series of tests upon sustained adhesion properties, a three-part epoxide coating, "Araldite GY 250" plus "HY 830" and "DY 830" was used. GY 250 is a liquid bisphenol A epoxide resin, with an equivalent weight of approximately 185. HY 830, also a liquid, is a modified aromatic amine which with catalyst DY 830 gives a system curing under ambient conditions. (The mixing ratio was

100:60:1.) T-peel specimens were made up with BS 3L61 using this system and the peel strength determined immediately after cure and after immersion for seven days in water at 40°C.

It has already been shown with this epoxide system that on steel the adhesion is helped by a burst of heat at the commencement of cure⁸, especially for the least drastic pre-bond processes. This concept was tested on the aluminium. The pretreatments were: degreased only; gritblasted; etched in a pure bath (ineffective); or etched in a workshop bath. The results shown in Table 7 were obtained.

such as copper, is present in the chromic-sulfuric acid etch. A pure etch, or a gritblasted or degreased surface give a poorer bond.

Acknowledgments

The author is grateful to Mr D. F. Malin for the scanning electron microscope photographs and to Mrs L. M. Ramsey, Mr B. W. Smith and Mr R. F. Walker, who carried out the adhesive testing.

[Received 19 August 1976]

Table 7
Aluminium surface structure and resistance of T-peel joints to water
Resin system: Araldite GY 250, plus HY 830 and DY 830.
Adherend: BS 3L61.

Surface condition	Adhesion test (N per 25 mm width)					
	24 hr at 20°C		Conditions of cure* 40 min at 100°C		20 min at 125°C	
	Initial	Immersed	Initial	Immersed	Initial	Immersed
Degreased	2	0.3	4.7	3.7	16.5	5.5
Gritblast	6	6	8	7	16	7
Pure etch	2	0.3	7	6	7	6
Workshop etch	13	11	39	31	39	33

*Conditions shown plus 24 hr at 40°C

The data show that the well-etched metal gave joints at least three times the strength, as made or after immersion, of joints with any of the other three pretreatments. The fraction of initial strength lost had the ranking: workshop etch < pure etch < gritblast ≪ degreased. The burst of heat to assist thorough wetting of the metal was successful with all the surfaces. The proportional effect was greatest with the surface which had been degreased only.

This experiment with the epoxide coating system showed the same effect as with the lap-shear tests. The chemical etch of the aluminium surface had to be carried out with the correct conditions to give a morphology which would result in adhesion at the metal-to-resin interface.

Conclusions

The experiments show that the strength and durability of a bond between aluminium and a thermosetting resin depend on the structure of the metal surface. The morphology given by a chemical etch treatment can be affected drastically by the presence, or absence, of noble metals in the system, either dissolved in the etch or present in the alloy. With a vinyl/phenolic system and with two epoxide systems, the strongest, most durable bonds are obtained when a metal,

Errata

The author regrets mistakes in the dimensions shown in his Fig. 1 on page 70 due to an error in conversion to SI units from the original reference 6. The values should read as follows:

- For 4 μ m read 40nm
- For 0.4 μ m read 4nm
- For 0.8 μ m read 8nm
- For 3.2 μ m read 32nm

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- 7(c). Exalto, R., and Bijlmer, P. F. A., *SAMPE 18th Nat. Symp.*, 1973, **16**, 427;
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Next month's issue

The Honorary Editor has accepted the following papers for publication, and they are expected to appear in the March issue of the *Journal*:

Estimation of solubility parameters of low molecular weight compounds by a group contribution technique by H. Ahmad and M Yaseen

The control of settling of some water-soluble paints by N. A. Ghanem, F. F. Abd El-Mohsen and S. El-Zayyat

The influence of plasticiser content on the mechanical properties and durability of chlorinated rubber paint films by D. Caldwell and W. D. Ferguson

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Short Communication

Relative permittivities of some aniline- and toluidine-substituted anthraquinones

By G. F. Lewin and S. J. Chapman

Physics Department, Brunel University, Kingston Lane, Uxbridge and Department of Chemistry, Watford College of Technology, Watford, Herts.

Summary

A simplified apparatus and faster method for determining pigment relative permittivities (ϵ_r) is described. The measured ϵ_r values of

four substituted anthraquinones (used as dispersed dye pigments) are presented.

Keywords

Raw materials: prime pigments and dyes pigments

Equipment primarily associated with analysis, measurement or testing

electrical equipment

Les permittivités relatives de certaines anthraquinones ayant aniline ou toluidine en tant que substituant

Résumé

On décrit à la fois un appareil et une méthode plus rapide pour déterminer les permittivités relatives de pigments (ϵ_r). On présente les valeurs de ϵ_r qui ont été déterminées sur quatre anthra-

quinones substituées qui sont utilisées en tant que colorants insolubles dispersés.

Die relative Dielektrizitätskonstanten einiger anilin- und toluidinsubstituierter Anthraquinone

Zusammenfassung

Beschreibung einer vereinfachten Apparatur und schnelleren Methode zur Bestimmung relativer Dielektrizitätskonstanten von Pigmenten. Die gemessenen ϵ_r Werte von vier substituierten

Anthraquinonen (als dispergierte Farbpigmente benutzt) werden vorgeführt.

Introduction

Previous work on the dielectric constants of pigment powders¹ has been extended by measurements of four dispersed dye pigments (substituted anthraquinones). Because measurements by the method previously described¹ were time-consuming, a modification to the electric circuit was made by raising the frequency to 0.5 MHz whilst keeping the same crystal holder, and the method of measurement was simplified,

making it more suitable for adaptation to industrial requirements.

Experimental

Pellets of pigment, 1-2 mm thick and 16 mm in diameter, were compressed at 27.6 MPa (4000 psi) and silvered with Acheson Silver Dag on both sides. They were inserted in a pellet holder (Fig. 1) and slid into the circular depression

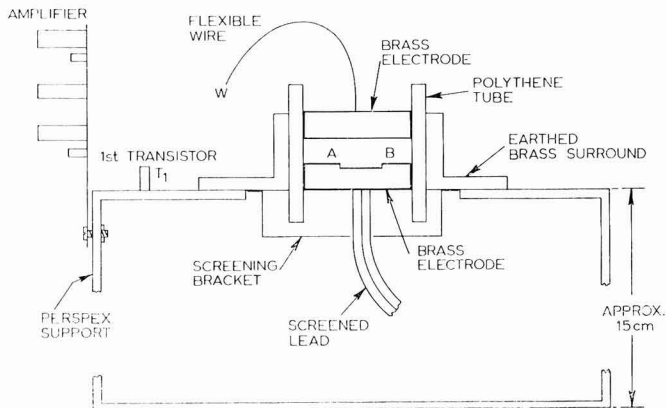


Fig. 1. Pigment pellet holder and wiring

AB in the lower electrode, using tweezers and then slight radial movements with light downward finger pressure. The upper electrode was then replaced and pressed down gently; during a measuring operation, it was held down lightly, if necessary, by the point of a drawn-out glass tube, to ensure that the electrode had not been raised slightly by the flexible lead.

Point *W* was earthed via a 100Ω resistor, and connected via a physically small $0.02\mu\text{F}$ capacitor to the base of the first transistor T_1 of the three-transistor 0.5 MHz amplifier. The circuit used is shown diagrammatically in Fig. 2. Position **1** of the 11-way switch was joined through a screened lead approximately 10 cm long to the pigment holder, and from positions **2-11**, ten screened leads were connected to ten resistors R (nominally 100k , 87k , 82k , 69k , 62k , 47k , 39k , 27k , 22k and $10\text{k}\Omega$) mounted on the perspex support close to the pigment holder and to the first transistor of the 0.5 MHz amplifier. (Only one of the resistors R is drawn in Fig. 2.)

then rotated away from position **1** until nearly the same peak-to-peak height was obtained through one of the resistors R , and then the exact height of 4 cm CRT deflection was obtained again by a slight adjustment of the calibrated 250Ω potentiometer. From the switch and potentiometer settings, and from previous calibrations of the resistors by battery, voltmeter and milliammeter, the capacitance C of the pigment holder (with the pigment pellet in place) was calculated. A cross-check, by measuring pellets previously observed in the earlier apparatus, gave consistent results. From the measured value of C , the air capacitance C_{air} was subtracted, using a previously obtained calibration curve¹ for the pigment holder. The thickness of the silvered pellets was then measured at their centres by micrometer, subtracting $25\mu\text{m}$ (0.001 inch) to allow for two thicknesses of silvering. This correction had been determined by subsidiary measurements on pellets before and after silvering. The advantage of this method is that there is no risk of breaking the pellets before the electrical

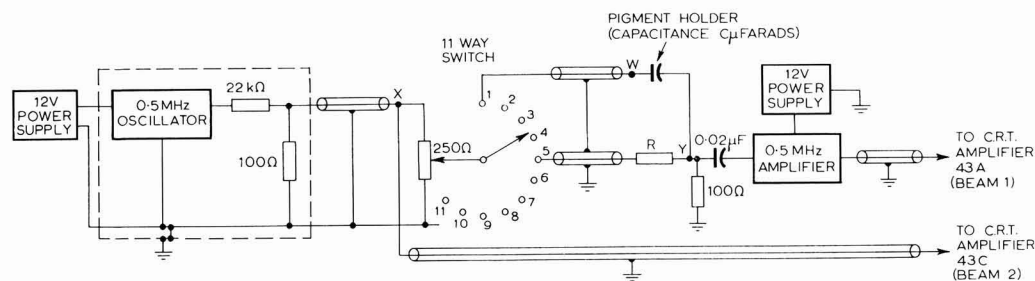


Fig. 2. Circuit diagram of apparatus

The output of the amplifier was connected through a screened lead to the cathode ray tube (CRT) amplifier (type 43A) of a double-beam Telequipment cathode ray oscillograph, using a timebase of $2 \times 10^4\text{ m s}^{-1}$ (2 cm per microsecond). For phase comparison, to allow for the effects of possible resistive loss in a pigment, the upper terminal of the 250Ω potentiometer (point *X* in Fig. 2) was connected via a screened lead to a CRT amplifier (type 43C) feeding the other beam of the oscillograph. By tuning the resonant circuit in the collector lead of the first transistor, whilst observing the CRT with the 11-way switch connected to one of the resistors R , it is possible to adjust the phase to 0° (or, more precisely, to 180° , because of the oppositely directed displacements of the two beams) on R . Then, upon switching to position **1**, the maximum of the sinewave appearing on one beam coincides with the zero crossing of the sinewave on the other beam (90° phase angle) for pigments which are perfect dielectrics without resistive loss.

To determine the permittivity at position **1**, the peak-to-peak height of the output sinewave was adjusted to 4 cm , by altering the setting of the 250Ω variable potentiometer (Fig. 2), taking care that the signal did not become too big to cause any signs of amplifier overloading. The switch was

measurements are completed; moreover, if they break on removal from the pigment holder the thickness can still be measured. This made it possible to reduce the number of pellets used to four per pigment; with four pigments, two to three hours was consumed in making satisfactory pellets, whilst another one hour was needed for measuring these and calculating the results.

Results

Pigment	Average ϵ_r
1:4 dianilino anthraquinone	2.83
1:4 di-p-toluidino anthraquinone	3.19
1 hydroxy 4 anilino anthraquinone	2.44
1 analino 4p-toluidino anthraquinone	2.45

[Received 19 August 1976]

Reference

I. Lewin, G. F., and Chapman, S. J., *JOCCA*, 1974, **57**, 225.



OIL & COLOUR CHEMISTS' ASSOCIATION

CURRENT PUBLICATIONS

Introduction to Paint Technology

The Association's extremely popular book "Introduction to Paint Technology", which forms an excellent introduction to the whole field of surface coatings and related technologies and which has already sold over 16 000 copies, has now been completely revised and updated in a new fourth edition. An important addition in this latest printing is a glossary of most of the chemical and technical terms used in the text: this has been included for the benefit of those readers who require a less superficial knowledge of paint formulation. A brief account of the history and development of chemistry and chemical symbols, formulae and equations is included as an introduction to the glossary itself.

Price: £5.00 (Registered Students of the Association £2.50).

Paint Technology Manuals: Part Seven "Works Practice"

The Association has sponsored the publication of a series of Paint Technology Manuals primarily intended for students and those entering the industry but already acknowledged to be invaluable to the practical man within the industry. "Works Practice" was originally published in the *Journal* as a series of Student Reviews. The volume is concerned with the practical aspects of making paints. In view of the fact that there has been little published material on this topic, a fairly broad coverage is attempted, including factory layout and organisation, paint and media manufacturing processes, legal aspects and safety precautions.

Price: £3.00

Paint Technology Manuals: Part Three "Convertible Coatings"

Part Three of the Association's series of Paint Technology Manuals has been reprinted as a second edition in 1972 and a limited number of copies are still available.

Price: £2.80

Ultraviolet Polymerisation

A volume, bound in limp covers, comprising papers presented at a symposium of the Newcastle Section of the Association in 1975 and subsequently published in the *Journal*.

Price: £5.00

Conference Preprints

The Association organises an international Conference every two years and preprints of the papers presented are prepared for delegates. The complete bound set of papers for both the 1973 and 1975 Conference are still available and offered for sale at £5.00 for each volume.

The 1973 Conference was held at Eastbourne with the theme "Towards 2000" and the bound set of preprints for this Conference includes 16 papers all dealing with various aspects of the surface coatings industry towards the year 2000.

The 1975 Conference, which was held at Scarborough, had as its theme "Performance of surface coatings—does reality match the theory?" and 17 papers were presented.

Many of the papers presented at these two Conferences were later published in various issues of the *Journal of the Oil and Colour Chemists' Association* and back issues are usually available (see below).

The Association's 1977 Conference will be held once again at Eastbourne; further details are given on page 40 of this issue.

History of the Association

As part of its 50th Anniversary celebrations the Association published in 1968 "A Fascinating Story—the History of OCCA 1918-1968". This is an illustrated book comprising 89 pages and bound in hard covers.

Price: £1.50

Journal of the Oil and Colour Chemists' Association

The *Journal of the Oil and Colour Chemists' Association (JOCCA)* is taken each month by thousands of chemists, technologists, and other personnel in the paint, printing ink and allied industries, as well as by numerous science libraries and organisations engaged in research and the supply, production and application of surface coatings, throughout the world. Copies of the *Journal* are now distributed each month to more than 80 countries.

Price: £25.00 (\$50.00) per year

Back issues

Most back issues from recent years and a considerable number of earlier issues, dating back in some cases to the earliest volumes, are available for purchase from the Association's offices at the current *Journal* cover price. The availability of particular issues can be obtained on request from the Association's offices.

Price: £2.50 (\$5.00) each

Consolidated Indexes

Earlier this year the Association published a Consolidated Index, by subject and by author, of all the Transactions and Communications which appeared in the *Journal* during the previous 10 years (January 1966 to December 1975).

This invaluable "quick reference" guide to the literature of the surface coatings industries has been produced in the international A4 size and it can, therefore, be bound with copies of the *Journal*. (Binding facilities for *JOCCA* are available through J. S. Wilson—see page 472 of December 1976 issue.)

Copies are still available of the Consolidated Indexes for the years 1918-1945, 1946-1955 and 1956-1965.

Price: Consolidated Indexes for 1966-1975—£5.00 (The three indexes for 1918-1965—£1.50 each.)

For order form see page xii

Hull

Synthetic clays—their properties and uses

A student lecture was held at the "George Hotel", Land of Green Ginger, Hull on Monday 15 November 1976. Mr B. J. R. Mayes of Laporte Industries Ltd, gave an informal talk entitled "Synthetic clays—their properties and uses".

In recent years, the synthesis of analogues to natural swelling clays have been perfected. "Laponite" inorganic colloids, from Laporte, were designed to modify the rheology and surface properties of a wide range of aqueous systems and had been shown to have an application in a vast range of products and markets. Their most useful property was that of conferring thixotropy and this property could be used in many diverse applications which required thickening, gelling, suspension and emulsion stability. "Laponite" was used in non-drip emulsion paints, aggregate finishes, electro-deposition primers, toothpaste, cosmetics and toiletries, household products and anti-static/anti-soil treatments for carpets.

The lecture covered the background, theory and research and development of these products. The unique properties of "Laponite" were demonstrated in several applications.

The meeting was disappointingly attended, there being only seven in an audience. A vote of thanks was proposed by Mr F. D. Robinson.

D.M.W.

London

Water repellent stains

An evening meeting of the Section was held at Manson House on 7 October 1976 when Mr Hamblin, Technical Manager of Cuprinol Ltd, presented a lecture on "Water repellent preservative stains for use on wooden claddings and external joinery".

Greater use of timber in buildings was made in North America and Scandinavia than in the UK, but there was now an upward trend here. The modified linseed oil/paraffin wax/iron oxide/paraffin solvents type of formulation used in the USA gave inferior performance in the UK.

The four essential ingredients were pigment, resin/oils, preservative and solvent. Iron oxides were the main pigments used to achieve natural wood colour. The choice of binder, such as alkyd resins and drying oils, affected gloss, water repellancy and the preservative effect. Paraffin wax and materials such as silicones were used to give water repellancy. The mode of action was a compromise between physical blocking and surface tension effects to prevent water ingress. The preservative was to prevent the growth of staining fungi.

Performance was governed by film thickness and the stain could be classified as "low build" for up to 25 μ m thickness, "medium build" for 25–50 μ m, and "high build" above 50 μ m. Low-build stains showed poor durability, failing by erosion, even though they gave high penetration into the wood due to low solids and low viscosity. Medium-build stains showed good to very good durability. The high build stains showed very good durability, failing by cracking and peeling, but as there was very little penetration they could not be classed as wood preservative stains.

Durability was tested by exterior exposure on hard and soft woods at two sites. Accelerated exposure results could be misleading. Preservative characteristics depended on penetration and durability of the preservative. Penetration could

be measured by following a soluble dyestuff and comparing against a standard.

Effectiveness against wood rotting organisms was tested according to the British Standard method. Durability of the preservative was tested by first exposing panels to six months' natural weathering during the summer months and then subjecting the panel to staining organisms and examining to what depth staining took place.

The vote of thanks was given by Mr H. A. Hipwood.

B.F.G.

Newcastle

Ion beam etching

The Royal Turks Head Hotel, Newcastle was the venue for the third meeting of the Section on 2 December 1976, when members assembled to hear Mr J. L. Prosser of the Paint Research Association present a paper entitled "Ion beam etching for the study of paint film structure". Using slides and diagrams, Mr Prosser reviewed various methods available for paint film examination and described the procedure used at the PRA of ion bombardment. Slides were shown of various paint films which had been subjected to ion beam etching for various periods of time and the pigment composition of the various "layers" near the film surface was clearly indicated. Mr Prosser concluded that the technique described could be used for the identification of pigments in mixtures, for particle distribution counting and possibly for the forecasting of the onset of chalking. A lively discussion period followed the lecture before the Chairman brought the meeting to a close. It was unfortunate that perhaps due to the inclement weather such a well presented lecture should attract the lowest attendance at the Newcastle Section for many years.

T.H.

Scottish

Car finishing

A lecture on "Car finishing" was given by Dr Bruce, of Goodlass Wall Ltd, in the Beacons Hotel, Glasgow on 18 November 1976 at 6.00 p.m. This was a joint lecture with the Society of Dyers and Colourists.

Dr Bruce's talk was concerned with the protection of car bodies by paint, and although his main interest was the topcoat, he outlined the requirements for phosphating, priming and surfacing prior to this topcoat. Dr Bruce highlighted the importance of correct priming, especially on the lower areas—50 cm from the ground—where the vast majority of corrosion spots could be found. The "pros and cons" of electrolytic priming were detailed. The surfacer was a coat which would allow the formation of a smooth surface for the application of a topcoat and give good topcoat adhesion. It was essential that the surfacer showed no water solubility as this would cause major problems with blistering of the topcoat.

For the topcoat there was now a selection of resins available: alkyd, melamine/formaldehyde, thermosetting acrylic, thermoplastic acrylic and non-aqueous dispersions (acrylics). Dr Bruce outlined the advantages and disadvantages of the various systems both on technical and economic grounds. Pigmentation of the topcoat was also a critical factor—all products must have outstanding fastness properties and not cause degradation of the film. This degradation was typified by the effect of metallics on alkyd films.

In terms of the future, powder coatings were still being examined in depth: satisfactory undercoats had been achieved—especially in combination with electro-coating to cover any areas missed by the powder. Operators in the USA were claiming some encouraging results for powder topcoats. Isocyanate topcoats were also arousing interest and their low temperature curing could give the required economic impetus. Control of spraying was being examined in considerable detail. Machinery for computer controlled matching of spray viscosity and other factors was now being installed.

The vigorous question time which followed indicated the interest shown in the lecture and the meeting thanked Dr Bruce in the usual manner for the clarity and scope of his presentation.

G.H.R.

Eastern Branch

Industrial hydrocarbon solvents

The first meeting of the session was held in the Carlton Hotel, Edinburgh on Wednesday 10 November 1976, when Messrs C. G. Smith and A. Cumbers, of Carless Solvents Ltd, delivered an interesting talk on industrial hydrocarbon solvents.

The basic feedstock for hydrocarbon solvents was naphtha, obtained by distillation of crude oil. After cracking, reforming and distillation, naphtha produced both low and high aromatic content solvents which were supplied to a given boiling range, rather than to any specific chemical formula.

Commercial petroleum ethers were known as SBP solvents, the various boiling ranges being applicable to different industries, such as the adhesive, printing ink and rubber industries. The current trend with these solvents was towards a higher naphthenic (cyclic aliphatic) content in order to increase solvency, thereby reducing the proportion of aromatics. As the naphthenic content rose, so did the Kauri-Butanol value, indicating increased solvent power. A similar trend was apparent with white spirit. Normally white spirit had a KB value of 32 and an aromatic content of 15 per cent. By increasing cyclic aliphatics and reducing aromatics to 5 per cent, the KB value could be increased to 35.

Mr Cumbers discussed the health and safety aspect of these solvents. Although the permissible level of benzene was currently 2 per cent, this was shortly to be lowered. The necessary action might not prove too troublesome since solvents were currently exported to countries where the concentration of certain materials, benzene in particular, was limited to minute quantities (below 10 ppm in some cases).

North Sea oil was not expected to have much effect on feedstock availability, since the correct fractions were not present as starting points for hydrocarbon solvents. As yet, no North Sea oil had been used by manufacturers such as Carless.

A lively discussion period then evolved, during which several interesting points were made. For example, the identification of individual hydrocarbons in a boiling range had proved impossible with GLC and other such methods, so simple empirical tests are more often used. Also, it had been known for customers to enquire as to the benzene content of high boiling fractions, such as white spirit, when in fact the boiling point of benzene was so much lower that it would normally be removed during distillation and be present only in minute or undetectable quantities, if at all.

Mr J. H. Stewart proposed a vote of thanks for a very useful talk, the audience responding in the usual generous manner.

P.S.N.

Put a sparkle into your industrial finishes

The second meeting of the session was held in the Carlton Hotel, Edinburgh, on Wednesday 1 December 1976. Dr D. R. King and Mr R. I. Knowles, of Silberline Ltd, presented

a talk on modern aluminium pastes as manufactured by their company.

After outlining the history and organisation of Silberline Ltd, Dr King stated that the various aluminium powders and pastes available were all manufactured in much the same way, but that quality depended on careful selection of aluminium feedstock for ball milling, and techniques used during screening and bleeding. Choice of additives was also important.

All aluminiums were leaves rather than granules, with length about 200 times the thickness, but there were basically two types—leafing and non-leafing grades. It was the non-leafing grades which were important in automotive finishes, where high resistance properties were required. The latest types on the market gave improved brilliance, or "sparkle", whilst maintaining or even improving the hiding power of the duller types. Opacity was normally achieved by reducing the particle size, which usually led to a duller finish. By control of manufacturing technique, however, brilliance could be incorporated into smaller particle size pastes.

Quality control was very important and properties, such as shade, opacity, flop and particle size distribution, were regulated by fairly simple techniques. "Flop" was that property whereby the finish altered in shade according to the viewing angle, thereby giving better definition to the shape of the car. Such care taken during manufacture of the aluminium paste naturally resulted in a more expensive product, but since only about 2 per cent aluminium was used in automotive finishes, any slight increase in cost was more than offset by more rapid processing and matching times for the customer, thus cutting overheads.

On the question of printing inks, non-leafing aluminium pastes could prove an advantage over the traditional leafing types by providing much improved rub-resistance. Gold effects could be obtained by tinting the paste rather than by the more expensive technique of printing a tinted lacquer over aluminium foil, thereby giving both the brilliance and rub-resistance properties required.

Dr King finished a most informative talk by showing examples of the use of aluminium paste in upholstery finishes.

The vote of thanks was proposed by Mr G. H. Hutchinson and was heartily endorsed by all those present.

P.S.N.

West Riding

Pigments

A meeting was held on Tuesday 9 November 1976 at the Griffin Hotel, Leeds. Mr R. M. W. Wilson, of S.C.C. Colours Limited, gave a talk to illustrate the commercial development of new pigments.

The story was taken up around 1950, when some interesting new pigments showed promise and Pigment Yellows 73 and 74 were introduced. Their shortcomings were soon realised and Mr Wilson discussed these and described the way in which they were circumvented so that the pigments' advantages could be utilised. In particular, Pigment 74 had been developed and had led to the introduction of Pigments 74P and 74T which have interesting possibilities in various applications.

There followed a lively discussion concerned with the lecture itself as well as a variety of other related topics, which were stimulated by the lively, knowledgeable presentation by the speaker. Mr R. Smith echoed the views of all present in proposing a hearty vote of thanks.

R.A.C.C.

Information Received

Chemical investment and export surplus growth

UK chemical industry investment in 1975 was 22 per cent higher in real terms than in 1974 and 50 per cent higher in money terms, according to the recently published Activities Report of the Chemical Industries Association.

A record exports v imports surplus was achieved (£770 million) despite adverse trading conditions in which export volume fell by 16 per cent and import volume by 19 per cent. At the same time export prices were buoyant, showing an increase of nearly 24 per cent. Import prices rose by 11 per cent.

The CIA's work on environment and energy matters and its internal reorganisation on safety and health are among the other topics described in the 16-page report.

Clayton Aniline centenary

We gratefully acknowledge from Clayton Aniline Co. Ltd a copy of its book celebrating the company's 100th year. The 95-page volume has been produced in hard-back version and with many full-colour illustrations, and it describes the progress of the company from its foundation to modern times and the development of the present 57-acre site, which was featured in the August 1976 issue of this *Journal* on page 308.

South Florida test service

Atlas Electric Devices Co. of Chicago, USA, has recently announced the acquisition of South Florida Test Service Inc. of Miami. Long established as one of the leading Florida sites for natural environmental testing, South Florida Test Service maintains inland facilities for direct south exposures at 5°, 45° and vertical, as well as possessing under glass and black box facilities and undertaking speciality work. Additional sites allow salt spray and immersion studies, and laboratory facilities are being expanded to include a variety of Atlas Weather-Ometers.

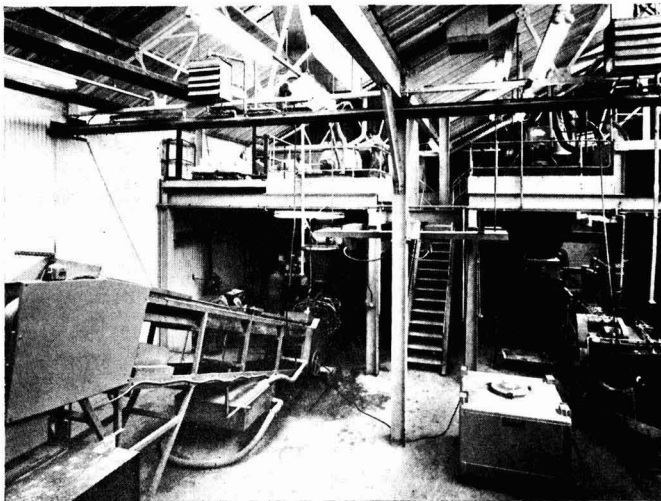
Veba and Huels agreement

An agreement has been concluded between Veba Chemie AG, Gelsenkirchen-Buer, Germany, and Huels (UK) Ltd, Orpington, Kent. With effect from 1 January 1977, Huels (UK) Ltd has taken over the marketing and sale of polyolefins within the United Kingdom.

Byk-Mallinckrodt in new laboratories

After a construction period of only 18 months, and at a total cost of approximately DM3 million, Byk-Mallinckrodt's new research and development centre has been taken into use. This will enable the already largely service-oriented company to set new standards in both cooperation with the international paint and coating industries, and in its contribution to the resolution of problems posed by the paint technology of tomorrow. Apart from this—its traditional market—Byk-Mallinckrodt has recently and successfully introduced new speciality chemicals to solve problems arising in other branches of the chemical industry.

With the new research centre, and the consequent personnel investment, Byk-Mallinckrodt is confident that it has laid a sound base for continued future growth.



A part of Drynamel's powder mixing shop at Hall Green, Birmingham, where thermoset powder coatings are produced for a wide variety of industrial and consumer products

New products

Laboratory device for screening materials sensitive to ultraviolet light and condensation

Wentworth Instruments Ltd, UK agents for Atlas Electric Devices Co. of Chicago, USA, has announced the introduction of the "UVCON", a device for exposing



The "UVCON" unit for screening materials sensitive to ultraviolet light and condensation, which has been introduced by Atlas Electric Devices Co. (UK agent Wentworth Instruments Ltd)

materials to alternate cycles of fluorescent ultraviolet light and condensation. The unit provides a screening test for coatings, plastics, pigments and the like, which may be specifically affected by the UV portion of the sunlight spectrum along with moisture penetration. Screening tests of this

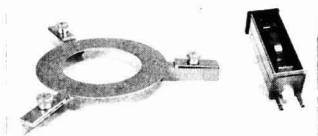
type can give early indications as to how a material may be expected to perform in a natural outdoor environment.

Whilst not intended as a replacement for more complete accelerated weathering systems, the "UVCON" does act as a simple supplement to that equipment where rapid UV screening is desired.

New accessories for lightfastness testers

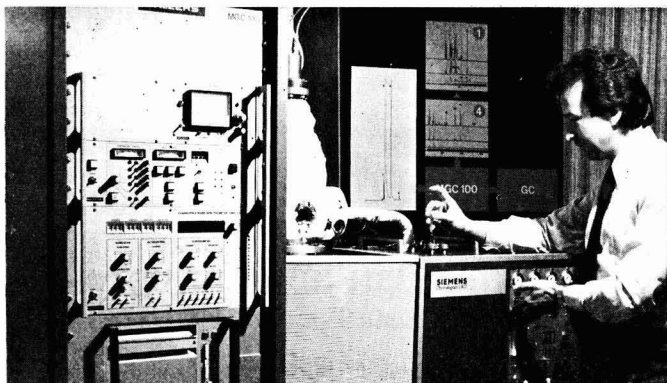
To assist existing users of its Mark I model lightfastness tester, Microscal Ltd has introduced conversion kits giving a wider range of characteristics to suit various needs.

One modification uses a smaller exposure gallery (close frame) to form the Mark I (CF), in conjunction with a different type of lamp (MB/U). The sample capacity is reduced from 134 maximum to 60, and only water-cooled cells are suitable, but the fading rate is increased from 3.4 times daylight for the basic Mark I to 28 times.



Accessories for Microscal's lightfastness tester

Another modification incorporates the same new lamp and a special power unit, without altering the physical layout. This gives a fading rate of 5.4 times daylight, retains the maximum 134 sample capacity, and can use any of the four types of exposure cells available.



Siemens' combined system for gas chromatographic and mass spectrometric analyses. The mass spectrometer can be linked with any desired gas or laboratory chromatograph.

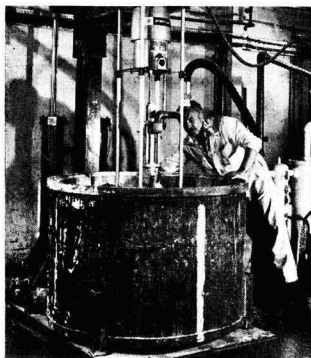
Current production includes all variants of the Mark I and other models, in addition to the conversion kits. Full details are available from Microscal, and the main characteristics of all types are summarised in a new publication "The Lightfastness Tester Family".

New BASF Paliotol pigment

BASF has added a calcium lake of an azo dye, "Paliotol Yellow 0910", to its Paliotol range. This clean, greenish-yellow pigment has been formulated to give excellent overspray fastness and heat resistance. In both mass tone and in combination with nickel-titanium yellow, it should display good weather-fastness. Further information is available in the UK from the D & P Division, BASF United Kingdom Limited, PO Box 4, Earl Road, Cheadle Hulme, Cheshire.

Steeley pumping installation

The Fluid Handling Department of Steeley (Mfg) Ltd has designed and supplied a further pumping installation for use in Berger Chemicals' Newcastle-on-Tyne plant. Designed to handle extremely viscous materials, the new operation employs a high output Graco 10:1 ratio shovel pump



A view of the Steeley pumping installation at Berger Chemical's Newcastle-on-Tyne plant

incorporating a specially designed ram unit fitted with a 133 cm ram plate. This is the largest ram unit yet produced by the Wolverhampton firm and is used to transfer the high viscosity material from a 900 litre mixing vessel, via a filter, to 200 litre

reservoirs which supply the filling line.

The ram unit employs twin air cylinders to apply a controlled pressure in the mixing vessel, thus ensuring positive pump priming for effective high volume transfer. A further advantage is that the ram plate with its dual wiper rings provides sealed protection for the material and also wipes the sides of the vessel, avoiding wastage. To avoid cavitation, the fluid pump is fitted with a priming piston.

The new installation has proved to be highly satisfactory. Running costs are significantly lower than with comparable alternatives and the material is handled quickly and efficiently with no waste or contamination.

Literature

Chemicals directory

A new compact and comprehensive directory of chemicals on the UK market has been published recently by the Chemical Industries Association. Entitled "Chemicals 77", the 250-page book lists over 8000 chemicals in proprietary and trade names and their manufacturers names and addresses. The information is up-to-date to at least the autumn of 1976. Copies are available at £8.00 each (UK) and £10.00 each (overseas), including postage and packing.

Surfactants UK

Tergo-Data of Darlington, a consultancy in detergents, has recently published a new catalogue covering surface active agents available in the UK. Compiled and edited by Dr G. Hollis, this comprehensive book has almost 190 pages of text information and advertisements. Copies are available at £7.50 (UK) and £8.00 (overseas), including postage and packing.

Taschenbuch für Lackierbetriebe 1977

This is the thirty-fourth edition of a handbook for users of industrial finishes, published by Vincent Verlag, Hannover. It contains in a very condensed form the useful subjects dealt with in Industrie-Lochierbetriebe, published by the same company. Modern paints, painting methods and installations are given, and an appendix contains information about German standards and regulations.

Review

Oberflächenbehandlung mittels Strahlmitteln, Ein Handbuch über Strahltechnik und Strahlanlagen

By Dipl. Ing. J. Horowitz

Zurich: Forster-Verlag A.G. 1976
First edition. Pp 238. Price Sw. Fr. 59.00

This extremely well made-up book goes far beyond the field of preparatory work for surface coatings. It discusses and describes in great detail, helped by numerous excellent photographs, drawings and many tables the field of "Surface treatment by means of blasting media" in a rather copious "Manual for blasting technique and blasting plant". This is only volume I. A volume II, which will cover the plant side in particular as well as problems connected with the acceleration of blasting media, fields of application (including use in foundries), protection against undue wear and tear and economical considerations, is in preparation.

Volume I is divided into five chapters. Chapter 1 is an introduction; chapter 2 treats the various types of media (those based on steels, including wire, aluminium and its alloys, zinc, non-metals—mineral and synthetic—such as zirconium, PVC, metal slags, etc). Quartz and sand are practically out, due to the silicosis danger, but glass beads are discussed. Hardness and production of the various grits have, amongst other matters, also been treated. Chapter 3 deals with testing and standardising of metallic blasting media. Excellent photographs illustrate, for example, the wear and tear of miscellaneous media. Chapter 4 is occupied with blasted surfaces, their condition, standardisation and structure. In chapter 5, the choice of blasting media, their type and best granular size for the various purposes are discussed.

The treatment of the subject matter of the book is most thorough, competent and, although rather detailed, the fundamentals are the backbone of the work.

M. HESS



OCCA-29 Exhibition

22-25 March 1977 at Alexandra Palace, London

97 companies representing 16 countries

- ★ Australia ★ Belgium ★ Denmark ★ East Germany ★ Holland ★
★ Hungary ★ Italy ★ Norway ★ Poland ★ Romania ★ Spain ★
★ Sweden ★ Switzerland ★ UK ★ USA ★ West Germany ★

The continuous dialogue between suppliers and manufacturers

New Exhibitors

The Exhibition Committee is pleased to report a number of additions to the list of Exhibitors published in the December and January issues of the *Journal*, and these organisations are mentioned on page 83 opposite.

Any company still wishing to apply for Exhibition space should contact the Director & Secretary immediately, since there is only a limited amount of stand space left in the hall.

Venue

The twenty-ninth annual Exhibition of raw materials, plant and equipment for the paint, printing ink, colour and allied industries organised by the Association will take place at Alexandra Palace, London N22 from 22 to 25 March 1977. Alexandra Palace was the venue for the exhibition held in March 1976 and for the series of exhibitions from 1965 to 1969.

Dates and times

The 1977 Exhibition will take place at Alexandra Palace, London, N.22 on the following dates and times:

Tuesday 22 March .. 09.30 - 17.30 hrs.
Wednesday 23 March .. 09.30 - 17.30 hrs.
Thursday 24 March .. 09.30 - 17.30 hrs.
Friday 25 March .. 09.30 - 16.00 hrs.

Theme of the Exhibition

The Committee emphasises that whilst it naturally encourages the showing of new products it does not stipulate that new products have to be shown by exhibitors each year and it attaches equal importance to the advantage to personnel at all levels of meeting and discussing their common technical problems in an informal atmosphere.

Refreshments on stands

This year in pursuance of this theme the Committee has amended the rule which had previously prohibited the serving of alcoholic refreshments on the stand. Exhibitors at previous Association Exhibitions at Alexandra Palace have commented that the visitors tend to spend longer time at the Exhibition.

Visits by principal officers of other societies

The Exhibition Committee has extended an invitation to the principal officers of many technical societies, research associations and government departments, to a private luncheon at Alexandra Palace on the opening day (Tuesday 22 March). The Committee will be conducting the party around the Exhibition and visiting the stands during the afternoon.

Stand telephones

The Post Office has allocated telephone numbers to Exhibitors who require a telephone on their stands at the Exhibition. Most of these telephone numbers appear with the Exhibitor's editorial entry in the "Official Guide" and the full list will be published in the March "Exhibition Preview" issue of the *Journal*.

Facilities at Alexandra Palace and travel arrangements

Visitors to OCCA-28 last year were impressed by the facilities available, which included two restaurants, two bars, a cafeteria and an exhibitors' bar. Other facilities include this year ample free car parking space, which is of considerable benefit especially to those using the M1 motorway, which links with the North Circular Road.

The Association will once again organise a free bus shuttle service to and from Turnpike Lane Station on the London Underground (Piccadilly Line). The journey from central London on the Piccadilly Line takes approximately 18 minutes and connections to the Piccadilly Line can be made easily from all mainline stations. It is hoped that the new electrification of British Rail services will be completed by March 1977 so that some visitors may find it easier to travel by train from King's Cross to Wood Green Station, from which station the London Transport W3 bus travels to Alexandra Palace. A further link which will be of benefit for those travelling by air will be the extension of the Piccadilly Line to Heathrow Airport, which is scheduled for completion in 1977. This will give a direct link with Turnpike Lane Station and in the meantime a bus service operates from Heathrow Airport to Hounslow West Station. Visitors who prefer to travel from Heathrow Airport to the West London Air Terminal in order to leave their luggage at hotels, can board the Piccadilly Line trains at Gloucester Road Station.

The Association has arranged for the exhibition to be held on its own at Alexandra Palace, which means that the facilities are exclusively available for exhibitors and visitors to this important international meeting place. The Exhibition Committee, therefore, draws particular attention to this aspect of this annual exhibition in providing an annual international focal point for the surface coatings industries, where the display and discussion

of technical developments and knowledge can take place in an informal atmosphere.

"Official Guide"

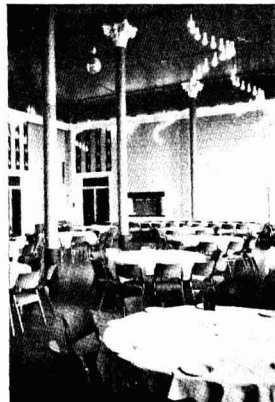
The "Official Guide" to the Exhibition is now being printed and it is expected that copies will be available in February for sending to all members of the Association and those requesting copies as a result of the Association's widespread publicity.

Each Member of the Association, at home and abroad, will be sent a copy of the "Official Guide" and free season admission ticket.

Members are asked to ensure that they bring their tickets to the Exhibition since otherwise the charge for admission will be made and no refund will be applicable in these cases.

As for the 1976 exhibition, copies of the "Official Guide" and season admission tickets will be available several weeks in advance of the exhibition (prepayment only) from the Association's office and they will also be available for purchase at the entrance to the Exhibition Hall.

For the last two exhibitions, it was decided that a small charge of £2.00 (US \$5.00) should be made both for the "Official Guide" and for season admission tickets to the exhibition. This policy undoubtedly deterred casual visitors who might otherwise be attracted to exhibitions



Part of the Edinburgh Room at Alexandra Palace, which will be open between 11.30 and 14.30 hrs on the four days of the Exhibition and where tables may be reserved

for which no admission charge was made and who gathered quantities of technical literature from the stands. The innovation was welcomed by many exhibitors and in no way acted as a deterrent to visitors to this exhibition. A similar charge will be made for the "Official Guide" to OCCA-29.

Information in foreign languages

As in previous years, the Association will be circulating information leaflets in six languages, which will contain application forms for those wishing to purchase copies of the "Official Guide" and season admission tickets before the exhibition.

OCCA-30 Exhibition 1978

The Exhibition Committee is pleased to announce that the Oil & Colour Chemists' Association's thirtieth Annual Exhibition of raw materials, plant and equipment for the paint, printing ink, colour and allied industries, "OCCA-30", will be held from Tuesday 18 to Friday 21 April 1978 at Alexandra Palace, London N22. Further details will be appearing in forthcoming issues of this *Journal*.

News of Exhibitors at OCCA-29 Exhibition

BOC Automation/Hunterlab

BOC Automation, specialising in the design and manufacture of microprocessor and minicomputer based monitoring and control systems, has announced the new "Hunterlab D54P-5" spectrophotometer. This instrument combines the accuracy of a spectrophotometer with the speed and precision of a tristimulus colorimeter and represents a new departure in spectrophotometer design.

A 20 cm (8 inch) diameter sphere is utilised for diffuse illumination. The light source is a quartz halogen tungsten lamp which is filtered to eliminate IR specimen heating and to simulate CIE Illumination D65.

A circular variable interference filter provides high speed spectral dispersion from 400 nm to 710 nm and the light level is sensed by a solid-state silicon diode detector and converted to a digital signal in the sensor.

The processor consists of a pre-programmed micro-computer system, which provides automatic standardisation and a correction factor for the contribution of the sample to the sphere wall. Spectral data for any of the four standard CIE illuminants A, C, D65, F (cool white fluorescent) can be converted into a wide range of colour spaces, colour differences and specific indices of colour, such as yellowness index, whiteness index and special index of metamerism.

The equipment will be shown on Stand 11 at OCCA-29.

Industrial Colours Ltd

As reported in the January issue of the *Journal*, *Industrial Colours Ltd* will be exhibiting on Stand 64 at the 1977 Exhibition and will show a range of aqueous fluorescent pastes and other pigment preparations. The company's address is now: **The Colour Works, Westwood Road, Broadstairs, Kent CT10 2PA.**

Additional Exhibitors at OCCA-29

Eckart-Werke

Eckart-Werke, West Germany, will be exhibiting on Stand 65 at OCCA-29. The company produces special aluminium pastes for automotive finishes. *Eckart-Werke's* agent in the UK is *Johnson & Bloy Ltd*, Metana Works, Priestley Way, Crawley, Sussex.

Polymers Paint & Colour Journal

Polymers Paint & Colour Journal will occupy Stand 1 at the Exhibition and show copies of the journal and other literature for the surface coatings industry.

Strazdins Pty

A. Strazdins Pty Ltd will be exhibiting on Stand 66. The company will demonstrate its full range of designs for the "Blendorama" colourant dispensers.

It will demonstrate the single pump 50 ml capacity dispensing machine for small and

medium stores, together with the double pump 150 ml capacity for larger stores and the large 600 ml capacity double pumps for bulk blending in factories or depots.

Apart from this, the company will display measuring pumps for epoxies or any other product which has to be accurately proportioned prior to use.

A. Strazdins Pty Ltd caters only for a quality market, using stainless steel cylinders and "teflon" slip-rings for all vital parts. In this way, it is possible to offer trouble free, versatile machines with the durability to be expected from high quality precision equipment.

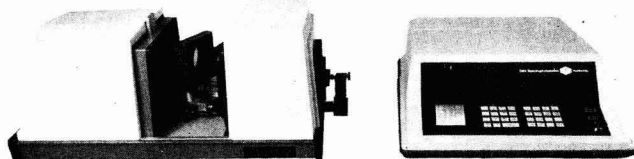
Enquiries for Stand space at OCCA-29 are still being received and it is expected that further space will be allocated after this issue goes to press. A full list of all companies exhibiting will be published in a special Exhibition Preview issue of the *Journal* in March.

Laporte Industries Ltd

Laporte Industries has announced that it will be spending up to £4 million to increase considerably the capacity of the plant at Stallingborough producing titanium dioxide pigments by the chloride process. The work will be completed before the end of 1977. The company reports that demand for the high quality pigments produced at this plant continues in both domestic and overseas markets.

Sherwin Williams

The company will be introducing its new *Powdura Blue*, a new range of dry pigments for the printing ink and carbon paper industries. The stand will also feature the *Alkali Blue* pigments and *Moly-White*, a range of pigments for corrosion-resistant paints, which are marketed by *Sherwin Williams* (UK supplying agent: *K & K Greeff Industrial Chemicals Ltd*).



The new Hunterlab D54 spectrophotometer which will be shown on Stand 11

OCCA Anniversary

Diamond Jubilee

In May 1978 the Association will celebrate the sixtieth anniversary of its foundation and it is felt that Members (and others who wish to take part in the celebrations) would like to have as much notice as possible of the dates. Following the pattern successfully used at the Association's Fiftieth Anniversary in 1968, the main events will be on two consecutive days. On the evening of Thursday 11 May it is planned to hold at a City Livery Hall a Commemorative Lecture, followed by a Dinner to which Past Presidents, Past Honorary Officers of the Association, Honorary Members and the surviving Founder Member will be invited

as guests. On Friday 12 May the Association's Dinner and Dance will be held at the Savoy Hotel, London WC2 and Presidents of other societies, together with their ladies, will be invited to attend.

London Section

Ladies' Night

The Section's Ladies' Night was held on 29 October 1976 at the Piccadilly Hotel, when 200 members and guests attended the annual Dinner and Dance. The President, Mr A. T. S. Rudram, was the principal guest and he proposed the toast to the Section. The response was given by Mr J. Tooke-Kirby, the Section Chairman. Also amongst the guests were chairmen from other Sections. Dancing continued to 1.00 p.m.

ASSOCIATION BIENNIAL CONFERENCE 1977

Conservation of energy, materials and other resources in the surface coating industry

The next OCCA biennial Conference will be held at the Grand Hotel, Eastbourne, Sussex in June 1977; the theme for the Conference will be all aspects of conservation in the surface coatings and allied industries. The Association is pleased to announce that the Federation d'Associations de Techniciens des Industries des Peintures, Vernis, Emaux et Encres d'Imprimerie de l'Europe Continentale (FATIPEC) has nominated "Layer structures of absorbed polymers at pigment/solution interfaces and their influence on the dispersion stability of pigments in paints" by Professor K. Hamann and Dr G. Joppien as the paper to be presented on behalf of the Federation at the Conference. A new paper by Mr S. Haagenrud (*see below*) will be presented during session one and has been nominated the paper to be presented on behalf of the Skandinaviska Lackteknikers Forbund (SLF). It is regretted that the paper by Dr van der Haauw which was described in the November 1976 issue of the *Journal* will not now be given. As already reported in the *Journal*, two papers from America have been designated as being presented on behalf of the Federation of Societies for Coatings Technology (FSCT), thus completing the representation by all four parties of the international alliance.

Copies of the Conference brochure and registration form were enclosed in all copies of the December 1976 issue of the *Journal* sent to Members attached to the General Overseas Section and non-member subscribers to the *Journal*. The brochures have been sent to all Members attached to the UK and Irish Sections with Section circu-

lars. Any non-member who has not yet received a copy of the brochure and wishes to register for this important Conference should apply for the necessary form to the Association's offices. Non-members wishing to avail themselves of the preferential Conference fee for Members may do so, by sending in a **membership application form at the same time as they submit their Conference registration form** and the fee enclosed should cover both the Membership entrance fee, 1977 subscription and the Conference registration fee. Membership application forms can be obtained from the Association's offices.

The registration fees for the Eastbourne Conference will be £40.00 (plus Value Added Tax at the standard rate) for Members, £60.00 (plus VAT) for non-members and £15.00 (plus VAT) for wives.

The Association Conference has been organised on this occasion to include a weekend and it will assemble on the evening of Thursday 16 June 1977 and disperse on the morning of Sunday 19 June. The headquarters of the Conference will be the Grand Hotel, Eastbourne, Sussex, England, where the lecture sessions will be held on Friday 17 and Saturday 18 June. The Association's AGM will take place on 18 June at the Grand Hotel.

Preprints of the papers will be prepared in the coming months and will be circulated to those who have registered for the Conference several weeks before the event, so that lecturers will be able to give a brief introduction to the topic and a longer period will then be available for discussion

purposes. This has been the practice at previous Association Conferences and has proved of immense value to those attending.

New paper

Atmospheric corrosion testing of metallised, metallised and painted, and painted steel

by Dr S. Haagenrud (Norwegian Institute for Air Research)



Dr S. Haagenrud

Summary: Long-term corrosion testing of different types of metallised, metallised and painted, and painted steel on test stations in southern and western Norway will be described. The main goal of the project was to obtain experimental data as basis for the choice of optimum protection systems under different atmospheric conditions. Emphasis has been placed on the measurement and analysis of the climatic parameters determining the extent of local corrosion; the test results should then be generally applicable under other conditions where these parameters can be determined.

Manchester Section Annual Dinner and Dance

The annual Dinner Dance was held at the Piccadilly Hotel, Manchester on 22 October 1976. The principal guests were Mr A. T. S. Rudram, the President, Mr W. B. Giles, Mr J. Tooke-Kirby, Chairman of the London Section, Mr K. V. Hodgson, Chairman of the Newcastle Section and Mr R. P. Bartrum, Chairman of the West Riding Section, and their ladies.

Mr Giles, Managing Director of Crown Decorative Products Ltd, proposed the toast to the Manchester Section. Mr J. E. Mitchell in replying to the toast paid a special tribute to Mr G. Clayton for his untiring leadership of the Manchester Section throughout its busy Golden Jubilee year.

After the formal proceedings, over 370 members and guests settled down to what proved to be a most enjoyable evening. Thanks are due to the Hon. Social Secretary, Mr P. Turner, for his efficient organisation of the event.



A photograph of the top table group at the 1976 Annual Dinner Dance of the Manchester Section, showing from left to right: Mrs Bartrum, Mrs Hodgson, Mr A. T. S. Rudram, Mrs Rudram, Mr J. E. Mitchell, Mrs Mitchell, Mr W. B. Giles, Mrs Giles, Mr J. Tooke-Kirby, Mrs Cook, Mr K. V. Hodgson, Mrs Tooke-Kirby, Mr R. P. Bartrum, Mr H. G. Cook.

News of Members

Dr B. Andersson, an Ordinary Member attached to the General Overseas Section, has been elected President of the Scandinavian Federation of Paint and Varnish Technologists (SLF) with effect from 1 January 1977. Correspondence should be addressed to Dr Börje Andersson, Ab Wilh. Becker, Fack, 102 70 Stockholm 9, Sweden.

Dr G. de W. Anderson, an Ordinary Member attached to the London Section, has been appointed Managing Director of the Paint Research Association in a recent re-organisation of the PRA.

Mr M. H. M. Arnold, an Ordinary Member attached to the London Section and a Fellow of the Professional Grade, and Mr H. R. Touchin, an Ordinary Member attached to the Manchester Section and also a Fellow of the Professional Grade, have introduced a Hazards Survey and Advisory Service through their joint subsidiary A & T Management Services.

The new service is intended to cover all types and aspects of industrial hazards and will be of particular value to chemical manufacturers and users, especially smaller companies which cannot provide from among their own staff the wide range of chemical, toxicological, biological and legal expertise which is required today. A & T Management Services will operate anywhere in the world.

Dr G. Balbi, an Ordinary Member attached to the General Overseas Section and a Fellow in the Professional Grade, has recently been elected Chairman of the Surface Coatings Committee of Unichem (Association for Standardisation in the Chemical Industry), which is the only institution of this kind in Italy, allied to ISO.

Dr W. Carr, an Ordinary Member attached to the Manchester Section and a Fellow in the Professional Grade, who received a Commendation Award in 1976, will be presenting two papers in March 1977. The first, entitled "Theory and practice of pigment dispersion", will be presented at the Manchester Literary and Philosophical Society at 6.30 p.m. on Monday 14 March. The second paper, entitled "Print strength of publication gravure inks", will be presented at the Royal Society of Arts, London WC2 at 6.45 p.m. on Tuesday 29 March.

Mr J. R. Taylor, an Ordinary Member attached to the Bristol Section and Fellow in the Professional Grade, retires this month from BP Chemicals Ltd at Barry, Glamorgan, but will continue to be active in all aspects of work associated with surface coatings and corrosion prevention. It has been arranged that he will act as Consultant on surface coatings to British Petroleum in the New Technology Division, Material Science Branch, Research Laboratories, Sunbury and will continue to be associated with coatings for BP Tankers Ltd and Chemical Plants etc., within the company.



Dr M. L. Ellinger, who is a member of the London Section and a Fellow of the Professional Grade, presenting a paper on "Correlation of weathering results" on behalf of the Association at the Washington Convention of the Federation of Societies for Coatings Technology in October 1976.

Professional Grade

Admission

The following Ordinary Member, having satisfied the regulations, has been admitted as an Associate in the Professional Grade:

VAN LONDEN, ANTON MATTHIJS (*General Overseas Section—Denmark*).

Register of Members

The following elections to membership have been approved by Council. The Section to which each new Member is attached is given in italics.

Ordinary Members

BAK, GREGERS, Dana Paints, PO Box 93/Jng, Jakarta, Indonesia (*General Overseas*)
 BOYD, GORDON JAMES, BSc, 188 Forestwood Drive, Kitchener, Ontario N2N 1B8, Canada. (*Ontario*)
 CARRICK, THOMAS EDMUND, 5 Knightside Gardens, Dunston, Gateshead, Tyne & Wear NE11 9RL. (*Newcastle*)
 COLLINS, GEOFFREY, LRIC, 46 Rothiemay Road, Flixton, Urmston, Manchester M31 3JY. (*Manchester*)
 DEVINE, JAMES, BSc, 28 Ladywell Road, Delves Lane, Consett, Co. Durham. (*Newcastle*)
 ELLIS, KENNETH, LRIC, 87 Saltwell Place, Gateshead, Tyne & Wear NE8 4TH. (*Newcastle*)
 ENGELBRECHT, JOHN FRANCIS, BSc, Revertex (SA) (Pty) Ltd, PO Box 12040, Jacobs, Natal, South Africa. (*Natal*)
 GREIG, MICHAEL STUART McRAE, BSc, 4/264 Balmoral Road, Auckland, 3 New Zealand. (*Auckland*)
 KETTEL, THOMAS JACOBUS, Polyesin Products SA, PO Box 70144, Bryanston 2021, South Africa. (*Transvaal*)
 MACDONALD, DONALD CLARK, 68 Alberton Avenue, Mt. Albert, Auckland 3, New Zealand. (*Auckland*)
 McMAHON, THOMAS MOODIE, Croda Inks Ltd, 170 Glasgow Road, Edinburgh EH12 9BE. (*Scottish-Eastern Branch*)
 MITCHELL, WAYNE MARTIN, BSc, PO Box 1084, Auckland, New Zealand. (*Auckland*)
 NEIL, DONALD, BSc, PhD, Croda Inks Ltd, 170 Glasgow Road, Edinburgh EH12 9BE. (*Scottish-Eastern Branch*)
 PAUL, SWARAJ, BSc, MSc, AB Wilh. Becker, S-10270, Stockholm 9, Sweden. (*General Overseas*)
 PHONE, JERCY, BSc, 40 Nortonville Drive, Agincourt, Ontario M1T 2G8, Canada. (*Ontario*)

SARFAS, DONALD THOMAS, 54 Dumyat Drive, Falkirk, Stirlingshire FK1 5PA. (*Scottish*)
 SPACEK, JAROSLAV, Apartado 61094, Caracas 106, Venezuela. (*General Overseas*)
 THRESHER, DONALD HUGH, Shell Chemicals, Box 494, Johannesburg 2000, South Africa. (*Transvaal*)
 WAGNER, MATTHIAS GERHARD, BSc, 511 Canonberry Court, Apt 402, Oshawa, Ontario L1G 2Z4, Canada. (*Ontario*)

Associate Members

ALLAN, DAVID McDONALD, 97 Avondale Road, Avondale, Auckland 7, New Zealand. (*Auckland*)
 ASCHENBERG, HANS MANFRED, Plascon-Evans Paints (Transvaal) Ltd, Box 3714, Johannesburg, South Africa. (*Transvaal*)
 BJORSTAL, SVENNE, AB Wilh. Becker, Box 2041, S-19502 Marsta, Sweden. (*General Overseas*)
 ENTWISTLE, MARK, Ashland Oil Canada, Industrial Chemicals Division, 150 Bronoco Avenue, Toronto, Ontario, Canada. (*Ontario*)
 GRAY, NICHOLAS CHARLES, Chemtra (Pty) Ltd, PO Box 2399, Durban, South Africa. (*Natal*)
 HARDER, JOACHIM RAINER, Bayer South Africa Ltd, PO Box 1366, Johannesburg 2000, South Africa. (*Transvaal*)
 SIMPSON, GRAHAM MILNES, PO Box 17-236, Greenlane, Auckland 5, New Zealand. (*Auckland*)

Registered Students

BAINS, RANJIT SINGH, 96 North Road, Southall, Middlesex. (*London*)
 KNOTT, DEIRDRE ANN, 14 Fifth Road, Heaton, Bradford 9, West Yorkshire BD9 4RS. (*West Riding*)
 MARCANGELO, RINO ALBERTO, 9 Twyford Abbey Road, Hanger Lane, London NW10 7HH. (*London*)
 READ, HARRY, 67 Kingsdale Crescent, Bradford, West Yorkshire BD2 4DR. (*West Riding*)

Forthcoming Events

Details are given of Association meetings in the United Kingdom and Ireland up to the end of the month following publication and in other parts of the world up to the end of the second month following publication.

February

Thursday 3 February

Newcastle Section: "Some techniques in the management of research" by Mr F. Westwick, Department of Management Studies, Sunderland Polytechnic, to be held at the Royal Turks Head Hotel, Grey Street, Newcastle upon Tyne, commencing at 6.30 pm.

Friday 4 February

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

Monday 7 February

Hull Section: "The alternative technologies for industrial finishes" by Mr J. Rackham, BTP Tiioxide Ltd, to be held at the "George Hotel", Land of Green Ginger, Hull, commencing at 6.30 pm.

Tuesday 8 February

West Riding Section: "Silicone resins in the surface coatings industry" by a speaker from Dow Corning Ltd.

Wednesday 9 February

London Section: "Printing on textiles". Joint day meeting with the Society of Dyers and Colourists and in association with Thames Polytechnic, commencing at 10.00 am.

Scottish Section: "Aspects of packaging" by Mr A. D. Lotte, Metal Box Company Limited. Joint meeting with the Eastern Branch at the Carlton Hotel, Edinburgh, commencing at 7.30 pm.

Friday 11 February

Manchester Section: "Organic versus inorganic coatings" by Mr J. R. Lyon, Goodlass Wall, to be held at The Woodcourt Hotel, Sale, Cheshire, commencing at 6.30 pm.

Thursday 17 February

Midlands Section: "The Health and Safety at Work Act" by Mr M. Kelly of the University of Aston in Birmingham. Joint meeting with Trent Valley Branch to be held at the Birmingham Medical Institute, 36 Harborne Road, Birmingham.

Friday 18 February

Irish Section: Lecture by a speaker from Bayer AG to be arranged.

Newcastle Section: Ladies' Night at the Five Bridges Hotel, Gateshead.

Thursday 24 February

Thames Valley Section: "Masonry paints" by Mr P. Whiteley, Building Research Establishment, to be held at the Beaconsfield Crest Motel (White Hart), Aylesbury End, Beaconsfield, Bucks, at 6.30 for 7.00 pm.

Friday 25 February

Bristol Section: "Paper in relation to printing processes" by Mr A. T. Franklin of Reed Paper and Board (UK) Ltd, to be held at the Royal Hotel, Bristol, at 7.15 pm.

March

Thursday 3 March

London Section: "Flammability testing and its relevance to flame retardant paints" by Mr A. G. Walker, Associated Lead Manufacturers Ltd. Evening meeting at the Royal Society of Tropical Medicine and Hygiene, 26 Portland Place, London W.1, commencing at 7.00 p.m.

Newcastle Section: "The value of mechanical tests in assessing paint performance and their relevance to specifications" by Mr C. E. Hoey, Ministry of Defence.

Monday 7 March

Hull Section: Ladies' Evening: "Antiques" by Mr G. Baitson of the Edwardian Auction Galleries, to be held at the "George Hotel", Land of Green Ginger, Hull, commencing at 6.30 p.m.

Manchester Section: "High solids water reducible antimoplast crosslinkers for modern coatings" by Dr M. Donnez, Monsanto Europe SA, to be held at The Woodcourt Hotel, Sale, Cheshire, commencing at 6.30 p.m.

Tuesday 8 March

West Riding Section: "Titanium dioxide pigment selection for water based glosses" by Mr J. Clark, of BTP Tiioxide Ltd, to be held at the Griffin Hotel, Boar Lane, Leeds, commencing at 7.30 p.m.

Thursday 10 March

Midlands Section—Trent Valley Branch: An informal discussion on paint exporting, with Mr L. Silver, Silver Paint and Varnish Co., to be held at the Crest Hotel, Pastures Hill, Littleover, Derby, commencing at 7.00 p.m.

Scottish Section: "Primers for wood finishes". Progress report on Scottish Section research project to be held at the Beacon's Hotel, 7 Park Terrace, Glasgow, G3 at 6.00 p.m.

Wednesday 16 March

Manchester Section: Student works visit in the afternoon to ICI Ltd, Organics Division, ARTS Block.

Scottish Section—Eastern Branch: Annual General Meeting followed by a film show to be held in the Carlton Hotel, North Bridge, Edinburgh at 7.30 p.m.

Friday 18 March

Midland Section: "Newton Friend" Lecture—Ladies Invitation, to be held at Birmingham Chamber of Commerce and Industry at 7.30 p.m. "A Victorian magic lantern entertainment" by Mr M. J. Simpkins.

Tuesday 22—Friday 25 March

QCCA-29 Technical Exhibition, Alexandra Palace, London—see page 82 for further details.

Wednesday 23 March

Ontario Section: "Organic pigments for trade sales paints" by Mr R. E. Edelman, Du Pont USA. To be held in the Starlight Room, Skyline Hotel, Toronto.

Thursday 24 March

Thames Valley Section: "Return of the vulture" by Dr M. Clarke of the City of London Polytechnic. A talk on corrosion, to be held at the Beaconsfield Crest Motel (White Hart), Aylesbury End, Beaconsfield, Bucks at 6.30 for 7.00 p.m.

Friday 25 March

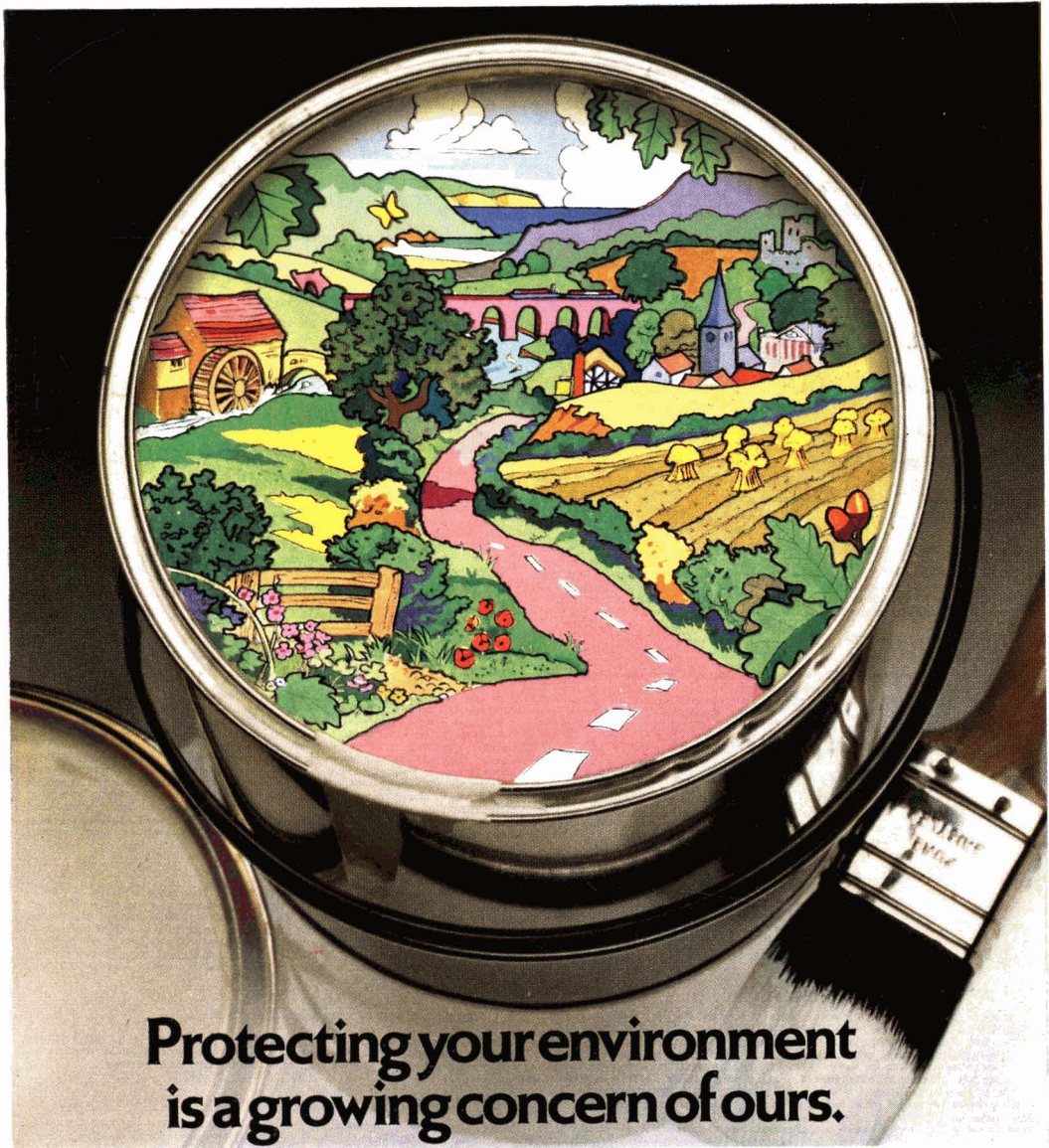
Bristol Section: "Lead chromes, their present uses and future trends, with particular emphasis on recent regulations" by Mr R. M. W. Wilson of SCC Colours Ltd, to be held at the Royal Hotel, Bristol at 7.15 p.m.

Irish Section: "Solvents and safety" by Mr C. W. Andrews, BP Chemicals Ltd, to be held at the Clarence Hotel, Dublin at 8.00 p.m.

Newcastle Section

Second UV Symposium—September 1977

A second symposium entitled "UV polymerisation and the surface coatings industry", sponsored by the Newcastle Section of the Association, is to be held at the University of Durham on 14 and 15 September 1977. Delegates staying at the University will assemble on evening of 13 September and disperse on evening of 15 September. Papers are to be presented by industry covering the practical aspects of the rapidly developing technique of UV curing. Topics will include photoinitiated polymerisation, UV curing equipment, resins, photoinitiators and photosensitive monomers. Further details may be obtained from Mr H. Fuller, Tiioxide International Ltd, Carlton Weathering Station, Yarm Back Lane, Stockton on Tees, Cleveland TS21 1AX.



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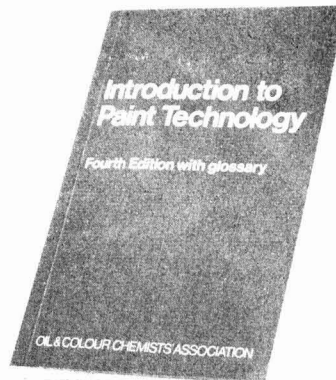
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2. Oils

Drying oils. Semi-drying oils. Non-drying oils. Chemical structure of fatty acids. Drying rate. *Drying oils:* Linseed oil, Stand oils, Tung oil, Soya bean oil. *Semi-drying oils:* Dehydrated castor oil, Tall oil, Tobacco seed oil, Safflower seed oil, Sunflower seed oil, Poppy seed oil. *Non-drying oils:* Castor oil, Coconut oil.

3. Natural and Synthetic Resins

Natural resins: Run gum, Damar resin, Rosin. *Synthetic resins:* Alkyd resins. Manufacture of alkyd vehicles. Phenolic resins. Vinyl resins. Vinyl polymers and copolymers. Solvent soluble vinyl resins. Vinyl chloride dispersions. Styrene containing resins. Polyester resins. Urea resins. Melamine resins. Epoxy resins. Epoxy/pitch media. Polyurethane resins. Acrylic resins. Silicone resins. Coumarone resins. Petroleum resins. Chlorinated rubber. Non-aqueous dispersions.

4. Resins as Media for Paints

Oleo-resinous media: Alkyd resins as media. Cellulose lacquers and media. Solvents for cellulose lacquers. Manufacture of lacquers. Other solvent-soluble, film-forming, high polymers. *Spirit varnishes. Solvent and driers:* solvents, driers. *Some uses of clear finishes:* insulating varnishes, wood finishing by cellulose lacquer. Water-based paints for industrial use.

5. Pigments and Extenders

Dispersion and wetting: wetting, wetting agents. *Inorganic Pigments:* white pigments: titanium dioxide, antimony oxide, zinc oxide, lithopone, white lead, zinc phosphate. Extenders: barytes, blanc fixe, whiting, precipitated chalk, silica, china clay, bentonite, slate powder, mica, asbestos, talc. Black pigments: carbon blacks, vegetable (lamp) black, bone and drop blacks. Metallic pigments: aluminium, bronze powders, zinc. Oxides of lead: red lead. Natural oxides of iron: red oxides, ochres, siennas, umbers, micaceous oxide of iron. Chemically prepared oxides of iron: yellow hydrated oxides, reds and browns, black oxide. Chrome pigments: lead chromes, zinc chromes, zinc tetroxochromate, strontium chromate. Blue pigments: prussian blue, ultramarine blue. Green pigments: chrome (Brunswick) green. Flooding and floating. Cadmium pigments. Nickel titanate. *Organic pigments:* classification of organic pigments: pigment dyestuffs, toners, lakes. Chemical classification: azo pigments, alkali blue or reflex blue, phthalocyanine pigments, phthalocyanine blues, phthalocyanine greens, vat colours, idantrone blue, quinacridone pigments, diioxazine pigments. *Luminescent pigments. Testing of pigments:* staining power or tinting strength. Opacity or hiding power. Oil absorption. Water-soluble matter. Lightfastness. Chemical constitution. Purity. Conditions of exposure, measurement of lightfastness. *Pigment hazards:* toxicity, dust.

6. Decorative and Structural Paints

Types of decorative and structural paints. Sealers. Primers: primers for wood, primers for alkaline and porous surfaces, primers for iron and steel, the mechanism of corrosion, protective mechanism of paint coatings, metal cleaning and pretreatment, blast primers, etch (or wash) primers, zinc-rich primers, zinc silicate primers, zinc phosphate primers, zinc chrome primers, red oxide/zinc chrome primers, red lead/linseed oil primers, calcium plumbate primers, primers for galvanised surfaces.

Undercoats, fillers and stoppers: stoppers, fillers, undercoats. *Finishes:* alkyd-based finishes, 'Non-drip' or thixotropic finishes, semi-gloss and eggshell finishes, oleo-resinous finishes, finishes for structural work, chlorinated rubber finishes, epoxy-resin finishes, epoxy ester finishes, vinyl resin finishes, masonry paints. *Emulsion paints:* nature of emulsions, preparation of polymer emulsions, homopolymers and copolymers, manufacture of emulsion paints, additives, properties and uses of vinyl emulsion paints, acrylic emulsions.

7. Industrial Stoving and Cold-curing Finishes, including Marine Finishes

Industrial finishes. Table of media used for various purposes, alkyds, alkyd/amino combinations, epoxy and epoxy ester resins, epoxy/pitch combinations epoxy/acrylamide acrylic resins, hydroxylated acrylic/amino resins, hydroxylated acrylic, isocyanate resins, thermoplastic acrylic resins, cellulose finishes, polyurethane finishes, vinyl resin finishes, plastisols and organosol finishes, chlorinated rubber finishes, polyester finishes, non-aqueous dispersion finishes, phenolic resin finishes, water-thinnable finishes, silicone resins, zinc silicate finishes, oil-free polyester finishes. *Stoving Equipment:* convection, infrared radiation, induction, dielectric heating, U.V. curing, electron beam curing. *Paint application:* padding, spraying, airless spraying, electrostatic spraying, hot spray technique, dipping, 'roto-dip' process, flow coating, curtain coating, roller coating, silk screen method, vacuum and pressure impregnation, electro-deposition, powder coatings, strip coating, aerosol spraying.

8. Paint Manufacture and Hazards

Paint milling: function of paint mills. General principles of paint mills. *Types of mill:* premixers, mixers for pastes, mixers for slurries, paddle mixers, high speed mixers. Dispersing mills. Dispersing mills requiring premixers. Triple roll mills, single roll mills, colloid mills, the sand mill, sealed sand mill, the 'Dyna' mill. Dispersing mills, direct charge: the ball mill, the attritor mill, high speed dispersers, the kady mill. Thinning mixers. Straining. Filling. *Health and fire hazards:* toxicity, pigments, resins, solvents, fire, other fire risks.

9. Common Defects of Varnishes and Paints

Varnishes: bloom, blushing, pinholing and cissing, silking. *Paints:* blistering, checking, cissing, crawling, dirt collection during drying, flotation, livering, pinholing, pigment sedimentation, rivelling, sagging, curtaining and tears, silking, skimming.

10. Paint Testing

Liquid paint: viscosity, brushability, colour measurement, colour matching, lightfastness, opacity, drying time. *The dried film:* adhesion and elasticity, hardness, gloss, film thickness, wet and dry, fineness of grind, water resistance, humidity resistance, salt spray resistance, durability, weatherometers.

Glossary—Appendices—Index

Order form on page xii

Copies of this book are available at £5.00 each (prepayment only), post free by surface mail, from:

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TEN-YEAR CONSOLIDATED INDEX

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