





Biographies of Conference authors

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A**

**JOURNAL OF THE  
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ASSOCIATION**

Chromate and phosphate pigments in anticorrosive primers  
*H. F. Clay and J. H. Cox*

Fungus-resistant paints for breweries and the humid tropics  
*E. Hoffmann, R. K. Hill and A. Saracz*

The parameters affecting the performance of coal tar/epoxy coatings  
*N. S. Moss*

Some aspects of drying oils technology *G. H. Hutchinson*

The micro analysis of solvent mixtures by reaction gas chromatography  
*J. K. Haken*



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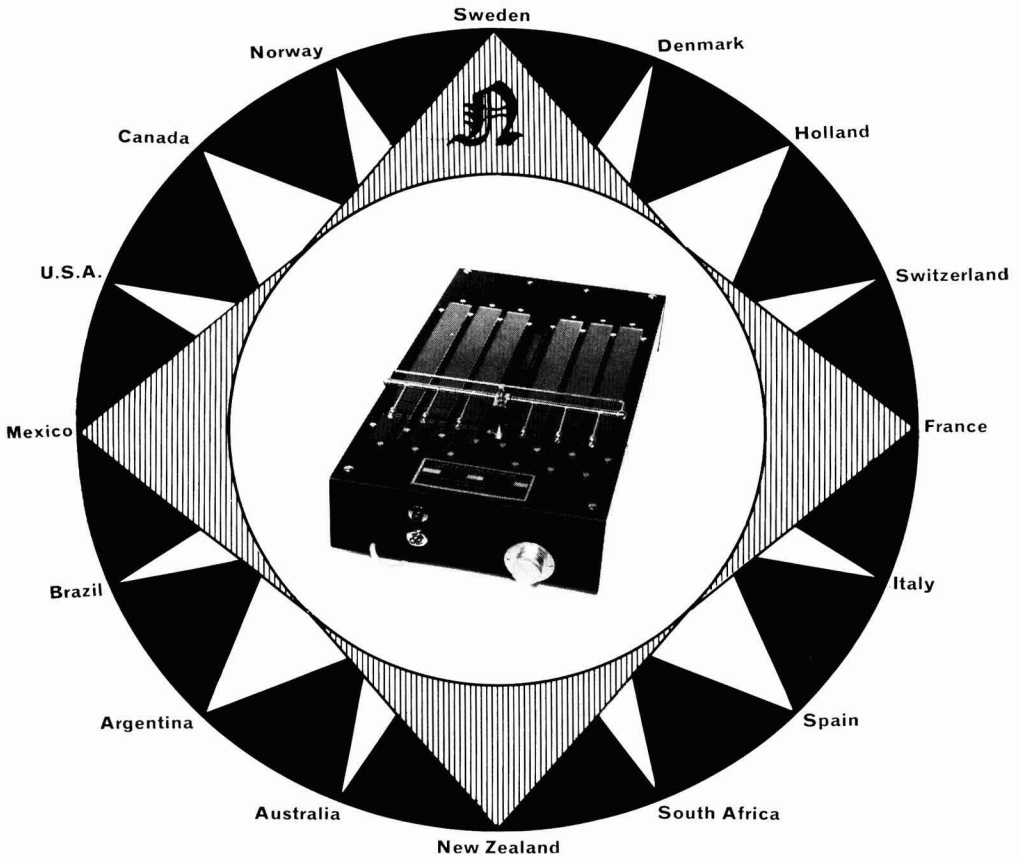
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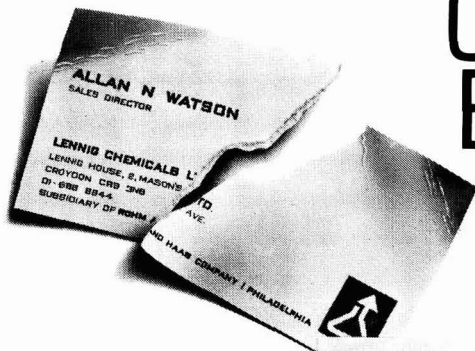
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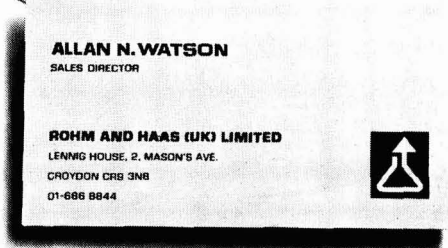


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# Basic products and auxiliaries for surface protection

**Dynamit Nobel**  
CHEMICALS





For Coil Coating:

# DYNAPOL® L

provides coatings eminently suitable for stamping and deep-drawing.

High-molecular, linear, saturated polyesters containing terephthalic acid.

DYNAPOL L types are suitable for the production of coatings having high stamping, deep-drawing and weather-resistant properties and which, in addition to having good adhesive strength, are scratch and impact resistant, and neither yellow nor chalk. DYNAPOL L types are best used for coil-coating. Coating surfaces can have a finish ranging from high gloss to matt as desired. Pigmentations of all shades are possible.

#### Main fields of use:

- Packaging,
- Packaging for foodstuffs (including sterilizable packaging),
- Fascia sections,
- Appliances

DYNAPOL L types are also suitable for single or double coat roller application on aluminium, steel or galvanized iron.

#### Technical data:

Sheen (Gardner)	max. 95 — 100 % approx.
Pencil hardness	max. H
Impact test	180 in. lbs.
T bend	max. depending on type up to T=O
Salt spray test	depending on type 400—1000 hours, unaffected
Kesternich test	Unaffected after 15—20 cycles

These details conform to the test standards of the ECCA (European Coil-Coating Association, Brussels).

# PVF Polyvinylfluoride

for long-life coatings

Wherever particularly long-life and outstanding weather resistance at both high and low temperature are called for, PVF coatings are ideal. For they are corrosion proof, are resistant to chemicals and have excellent shaping properties.

PVF is, among other things, particularly suitable for coil-coating. Surface finishes may range from a silky gloss to matt as desired; they discourage dirt and they neither yellow nor chalk. Many shades are possible.

PVF should be used where the usual coatings afford insufficient protection against corrosion.

#### Main fields of use:

- Building units where industrial pollution is severe
- Facings for industrial plant,
- Prefabricated building sections.

PVF is suitable for single or double coat roller application on aluminium, steel or galvanized iron.

#### Technical data:

Sheen (Gardner)	max. 80 % approx.
Pencil hardness	F — H
Impact test	180 in. lbs.
T bend	OT
Salt spray test	Unaffected after more than 1000 h
Kesternich test	Unaffected after 30 cycles
Friction constant	0.136

These details conform to the test standards of the ECCA



# DYNAPOL® P

Saturated polyesters containing terephthalic acid for the production of powders both for electrostatic powder-coating and for whirl-sintering.

Powders with a DYNAPOL P base are suitable for coating all metals.

By virtue of their special properties (e. g. high resistance to weathering, yellowing and chalking) such coatings are equally suitable for external use.

#### Main fields of use:

- Metal furniture,
- Metal window frames,
- Garden furniture,
- Apparatus,
- Metal facings,
- Tubing,
- Household appliances.

#### Technical data:

Thickness of single coating 50 — 70  $\mu$

Erichsen test 8 — 10 mm approx.

Pendulum test (depending on type) 175 — 185 sec. as per DIN 53 157

Indentation hardness 75 — 120 approx. (depending on type)

Sheen as per Lange. Angle of incidence 45°

80 — 115 (depending on type)

Adhesive strength: very good

Kesternich test up to 30 cycles (depending on type)

## HARD PVC COATING POWDER

A plasticizer-free polyvinyl chloride compound in powder form for whirl-sintering and powder-coating.

#### Advantages:

- Surface toughness
- Weather resistance
- Enduring gloss
- Chemical resistance
- No tendency to brittleness

#### Advantages when processing:

- Pleasant to work with: no offensive odours due to plasticizer vapours,
- High sheen surfaces within seconds,
- No post-treatment necessary.

#### Can be used:

- In the streets: Sign boards, posts for traffic signs, street lighting,
- In industry: Housings, mountings, sectional units, tubing,
- In the building world: Profiles, fittings, fascias,
- In the household: Appliances.

#### Technical data:

Coating thickness depending on method of application 80 — 400  $\mu$

Erichsen test 8 — 11 mm approx.

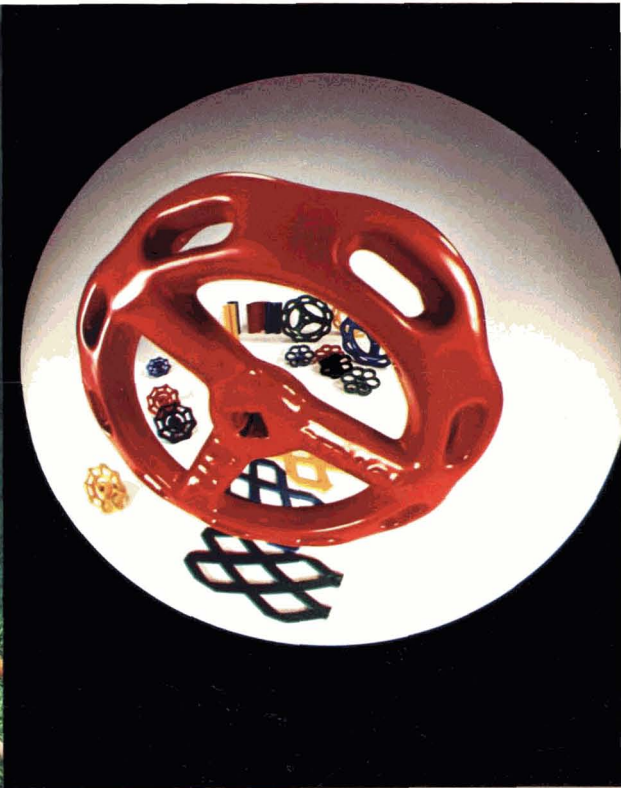
Pendulum test 170 — 185 sec. approx.

Sheen as per Lange. Angle of incidence 45°

120 — 140 %

Kesternich test. Unaffected after 40 cycles

Adhesive strength: with bonding agent, very good





For protection against corrosion even at high temperatures:

# DYNASIL<sup>®</sup> H 500

A binder having a silicic acid ester base for inorganic zinc dust coatings. When it is a question of protecting iron and steel from corrosion, even at temperatures up to 400° C, zinc dust paints having DYNASIL as a binder serve the purpose admirably.

Zinc dust paints with a DYNASIL base are particularly suitable for industrial processing. They can be welded over; they can be applied either by air or airless spraying, brush or roller, and can be used alone as a one-coat process or serve as a foundation for covering with any of the usual colouring methods.

**Main fields of use:**

- Shipbuilding,
- Industrial plant construction,
- Power plant construction,
- Bridge building,
- Large steel structures,
- Mass produced building units.

Test results of DYNASIL based zinc dust coatings:

Salt spray test (DIN 50 021) Unaffected after 1000 hours.

Adhesive strength, grating as per DIN 53 151

Grating value 0.

Temperature loading resistance: Permanent load, maximum 400° C. Short term load, maximum 600° C



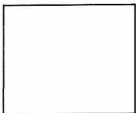
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**DYNAPOL L**



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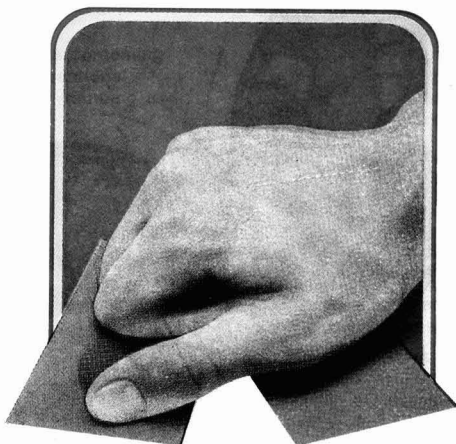
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# Rub-out stability

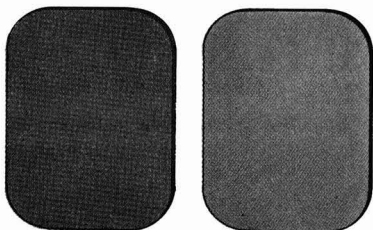


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**1973**

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## Guest Editorial by the President

This, the first issue of its 56th volume, is a landmark in the history of the *Journal* of the Oil and Colour Chemists' Association. For 55 years, since it first appeared in 1918, it has maintained the same basic form, changed only by the colour of the cover and certain typographical adjustments. Now it has moved to this new, larger format, which is to the A4 specification of the international system of paper sizes laid down by the International Standardisation Organisation.

The Council does not believe in change for its own sake. Nor did it take lightly a decision that breaks with such a long tradition, and which affects directly every Member of the Association and every reader of the *Journal*. It believes, as I do, that this change will, in the long term, be greatly beneficial to the *Journal*, and thus also to all Members.

The industries that OCCA serves are, like all science-based industries, being carried forward on a rapidly accelerating tide of technological innovation and advance, faster now, perhaps, than ever before. If it is to continue its valuable contribution to the industries, OCCA must keep pace with this advance. Like its industries, it must both harness the energy and enthusiasm of the young, and absorb the experience of the longer-standing members. To achieve both these goals, it must move with the times, and be seen so to do. The *Journal* is, perhaps, the most important single aspect in forming an impression of the Association. Its contents has always included papers which were abreast, if not ahead, of the state of technology in the industries, blended with news of the Association and its Sections for the benefit of Members and, indeed, no change in editorial policy is envisaged. Now, by adopting a size that is not only part of a modern, scientific system of paper sizes but will also enable papers to appear in a more readable and attractive form, the *Journal* projects a more accurate image of the Association today.

It is not only in the *Journal* that innovations are being made. The Professional Grade has been instituted, and the imposing list of Members admitted to the Grade that appeared in the December issue testified to the success of the venture. The Twenty Fifth (Silver Jubilee) Exhibition will take place in May and certain changes of arrangement have been made which it is felt will be of value to exhibitors and visitors alike. The title of the 1973 Conference is "Towards 2000", and an impressive programme of forward-looking papers has been assembled for the delegates at Eastbourne in June.

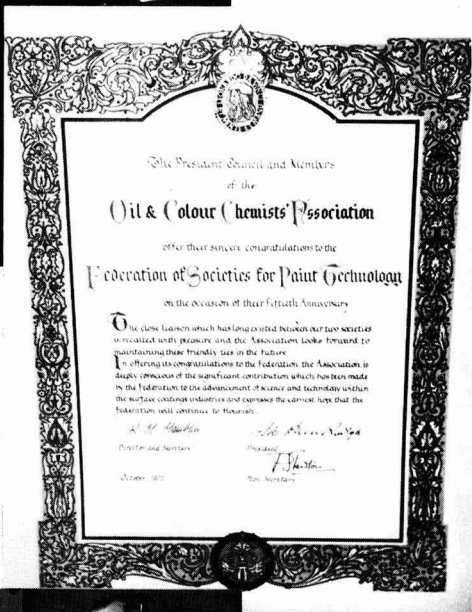
As OCCA looks ahead, let me assure you all that the past is not being abandoned. Rather it is being used as the foundation to build an Association for today and tomorrow. I know that I can count on the support of all Members in structuring OCCA for the future.

A. W. BLENKINSOP

# President's visit to FSPT



Above: Mr Blenkinsop presents FSPT President Mr Teas with the Association's commemorative scroll, shown on the right



Below: At the banquet (left to right) the President (Mr A. W. Blenkinsop), Mr C. Bougery (FATIPEC), Mr Y. Shiota (JSCM), Mr A. Braetsaeter (SLF), Mr J. P. Teas (FSPT), Mr D. Burnham (CPMA) and Mr R. Baudhuin (NPCA)



This year marks the half-century of the Federation of Societies for Paint Technology. The FSPT anniversary celebrations were combined with its Annual Meeting, held at Chalfonte-Haddon Hall, Atlantic City, from 23-28 October. On behalf of the Association, the President, Mr A. W. Blenkinsop, attended the Annual Meeting, and, at a banquet held on Saturday 28 October, presented the President of the FSPT, Mr J. P. Teas, with an illuminated scroll bearing OCCA's good wishes in commemoration of their fiftieth anniversary.

The President was able to meet principal officers of other kindred organisations throughout the world gathered to congratulate the FSPT on its anniversary. Among those

attending were Mr C. Bougery, Secretary-General of the Federation d'Associations de Techniciens des Industries des Peinture, Vernis, Emaux et Encre d'Imprimerie de l'Europe Continentale, Mr Y. Shiota, President of the Japanese Society of Colour Material, Mr A. Braetsaeter, President of the Scandinavian Federation of Paint and Varnish Technologists, Mr D. Burnham, President of the Canadian Paint Manufacturers Association, and Mr R. Baudhuin, President of the American National Paint and Coatings Association.

The Fiftieth Anniversary Annual Meeting was most successful, and the Association wishes the FSPT well for a long and happy future.



## Transactions and Communications

# Chromate and phosphate pigments in anti-corrosive primers

By H. F. Clay and J. H. Cox

S.C.C. Colours Ltd., Matlock, Derbyshire

### Summary

The composition and properties of a number of pigments are described. Particular attention is paid to the percentage of active pigment to be included in a primer, and to the pigment volume concentration to give optimum performance.

Zinc phosphate provides an alternative pigment to chromates: it

has the advantage of being completely non-toxic and can be used with a wide range of media. It is usually superior to zinc chromate when the primers are applied to rusty steel. On bright steel, zinc chromate usually gives an equal or better performance and, because it can be used at a lower concentration, will give a lower cost.

### Keywords

*Chemically active pigments*

zinc chromate  
zinc phosphate

*Types and classes of coating*

corrosion resistant primer

## Les pigments à base de phosphate ou de chromate pour primaires anti-corrosif

### Résumé

On décrit la composition et les caractéristiques de plusieurs pigments. On fait attention exceptionnelle au pourcentage du pigment actif à être incorporé dans un primaire, et également à la concentration pigmentaire par volume afin d'assurer le rendement optimal.

Le phosphate de zinc se révèle en tant qu'un pigment alternatif aux chromates; il possède l'avantage d'être tout à fait non toxique

et il peut être utilisé dans une gamme étendue de milieux. Il est supérieur en général au chromate de zinc dans le cas où les primaires sont appliqués à l'acier rouillé. Sur acier poli, le chromate de zinc donne généralement un rendement égal ou supérieur, et parce que l'on peut l'utiliser à une concentration plus faible, il offre un plus bas prix.

## Chromat- und Phosphat-Pigmente in Korrosionsschutz-Primern

### Zusammenfassung

Beschreibung der Zusammensetzung und Eigenschaften einer Anzahl von Pigmenten:

Der Wichtigkeit des prozentualen Gehaltes an aktiven, in einem Primer enthaltenen Pigmenten, sowie der Pigment Volumen Konzentration wird zwecks Erzielung optimaler Wirkung besondere Beachtung geschenkt.

Zinkphosphat kann wahlweise anstelle von Chromaten eingesetzt werden; es besitzt den Vorteil völlig ungiftig und mit einer grossen Anzahl von Bindemitteln verwendbar zu sein. Wenn Haftgründe auf verrostetes Eisen aufgetragen werden, ist es gewöhnlich dem Zinkchromat überlegen. Auf blankem Eisen zeigt Zinkchromat gewöhnlich gleichgute oder bessere Resultate und ist, weil ein niedrigerer Gehalt genügt, wirtschaftlicher.

## Хроматные и фосфатные пигменты в противокоррозионных грунтовых красках

### Резюме

Описываются состав и свойства ряда пигментов. Особое внимание уделяется проценту активного пигмента входящего в состав грунтовки и объемной пигментной концентрации, для оптимальной производительности. Фосфат цинка обеспечивает альтернативный пигмент по сравнению с хроматами; он имеет преимущество будучи совершенно не-токсичным и может

применяться в широком диапазоне различных сред. Он обычно превосходит хромат цинка, когда грунтовка налагается на ржавую сталь. На полированной стали хромат цинка обычно дает одинаковые или лучшие эксплуатационные качества, и так как он может применяться в пониженной концентрации, он окажется более экономичным.

## The pigments available

Normal zinc chromate does not exist as a pigment and the most commonly used chromate anticorrosive pigment is basic zinc potassium chromate (BS.389, type 2). Zinc tetroxy-chromate (BS.389, type 3) is also used in large quantities.

The neutral chromates of strontium and barium are used to a lesser extent.

Zinc phosphate is now available as an alternative pigment, together with zinc chromate phosphate.

## Properties affecting the choice of chromate pigments

The inhibition produced by chromate pigments in paints probably depends both upon the solubility of the pigment in water and also upon the particular cations present in the solution. A very soluble pigment could be expected to give good initial protection, but there might be some danger of the pigment being quickly leached from the paint, which would then lose its anti-corrosive properties. A slightly soluble pigment, on the other hand, whilst in no danger of being leached out of the film, might not give a concentra-

tion of chromate in water sufficient to produce inhibition. The solubilities of the commonly used chromate pigments are shown in Table 1.

Table 1  
Chromate content and solubilities of chromate pigments

Pigment	Minimum total CrO <sub>3</sub> , %	Solubility in cold water (on shaking 30g with 100ml water), %
Zinc chrome, type 2 (zinc potassium chromate)	43	0.25—0.50
Strontium chromate	46.5	About 0.2
Zinc chromate phosphate	8.15	0.06
Zinc chrome, type 3 (zinc tetroxychromate)	17	} About 0.001
Barium chromate	38.8	

In addition to the requirement of a suitable level of solubility, there are three other considerations:

the effect of the cations; there is considerable evidence that zinc ions are valuable in providing cathodic inhibition of the corrosion of steel;

the reactivity of the pigment with the medium; zinc chromates are basic and react with highly acidic vehicles; and

the freedom from aggressive anions; it is well-known that soluble chlorides, sulphates and nitrates detract from the protection provided by chromates, although in the presence of zinc ions these effects are greatly reduced.

If a pigment with appreciable water solubility is required, the normal choice is zinc chrome, type 2. This pigment has the particular property that, with continued extraction with water, the solubility gradually decreases until the composition of the residue approaches that of zinc tetroxychromate.<sup>1</sup> It is widely used in oleoresinous primers and in one-pack etch primers.

Zinc chrome, type 3 (zinc tetroxychromate) finds its main use in the production of two-pack etch primers, where it probably contributes chromium in different states of valency for film-forming reactions and probably additionally acts as an inhibitive pigment.

Where the basic character of zinc chromes makes their use inadvisable, strontium chromate can often be used as an alternative chromate pigment. It has a slightly lower water solubility than that of zinc chrome, type 2, but is more expensive. So far as conventional primers for application to steel are concerned, it usually performs less satisfactorily than zinc chromate, probably because strontium is not such a useful cation as zinc. It finds its main use in epoxy compositions, particularly those for use in aircraft.

Barium chromate, another neutral pigment, has been used for a long time in jointing compositions, where the rate of leaching of more soluble pigments would be excessive. It is also used in conjunction with strontium chromate in epoxy compositions to provide a reservoir of chromate, and can be used to pigment stable one-pack etch primers.

Phosphoric acid and metallic phosphates have been used for many years for the protection of steel and other metals, but largely as pre-treatments to the metal surface as distinct from paint primers. The first and most widely used paint system incorporating phosphoric acid is the etch primer WP-1.

A number of metallic phosphates have been suggested in recent years, including ferric ammonium phosphate, ferric phosphate, chromic phosphate, calcium and barium phosphates, but the only one which seems to have achieved much success is zinc phosphate. Pioneer work on zinc phosphate in Britain was done by J. B. Harrison and co-workers.<sup>2</sup> Work on the evaluation of zinc phosphate has also been carried out in Germany, where the emphasis has been on mixtures with chromate pigments. G. Meyer<sup>3</sup> discusses the use of a number of extenders together with 3 per cent of zinc phosphate, 15 per cent of zinc tetroxychromate, 5 per cent strontium chromate and 10 per cent zinc oxide.

J. B. Harrison and co-workers (loc. cit.) claim that zinc phosphate on its own, and in combination with other anti-corrosive pigments, including red lead, is superior to other existing anti-corrosive pigments. They also claim that less exacting surface preparation is necessary for zinc phosphate primers, and this view is supported by F. Timmins.<sup>4</sup> Zinc phosphate primers are non-toxic, and steel painted with them can be cut and welded without hazard or discomfort to workers.

Other advantages of zinc phosphate are that, because it is chemically inert, it can be used in a wide range of media and it is unlikely to give intercoat adhesion difficulties. Its virtual insolubility also makes blistering troubles unlikely.

The use of zinc phosphate in non-aqueous primers is the subject of patents<sup>5</sup> but certain pigment manufacturers can issue licences to paint manufacturers.

Zinc chromate phosphate is a relatively new pigment developed in the author's laboratories. It is formed by coprecipitation under controlled conditions of zinc phosphate and basic zinc chromate. X-ray measurements show that it is not simply a mechanical mixture but that it should more correctly be considered as a substituted zinc oxide. In primers applied to bright steel and used at the concentrations employed for zinc chrome, it is usually more effective, but on rusty steel it is often less effective than zinc phosphate. The manufacture and use of this pigment is covered by patents.<sup>6</sup>

### The possible mechanism for inhibition by zinc phosphate

Because zinc phosphate is virtually insoluble and unreactive, it is difficult to explain how it can function as an inhibitor. If steel is placed in an aqueous extract of zinc phosphate, it corrodes at the same rate as it does in distilled water. J. E. O. Mayne<sup>7</sup> has shown that, if steel is immersed in distilled water that has been in contact with a zinc phosphate primer, the rate of corrosion is almost the same as that of a specimen immersed in water that has been in contact with an unpigmented linseed oil film.

The authors thought the picture might have been different if the access of oxygen had been restricted, and with this in mind steel panels were immersed in both slurries and extracts of zinc phosphate. To provide a comparison, similar tests were made with slurries and extracts of blanc fixe. In addition to the tests on the straight extracts, additions of sodium sulphate (= 0.1 per cent of SO<sub>3</sub> of solid material) were made to give more aggressive conditions. Weight losses were lower in the slurries than in the extracts but there was no significant difference between zinc phosphate and blanc fixe.

On the other hand, when anodic and cathodic galvanostatic polarisation tests were made using a method described by one of the authors<sup>8</sup>, marked differences were apparent. Fig. 1

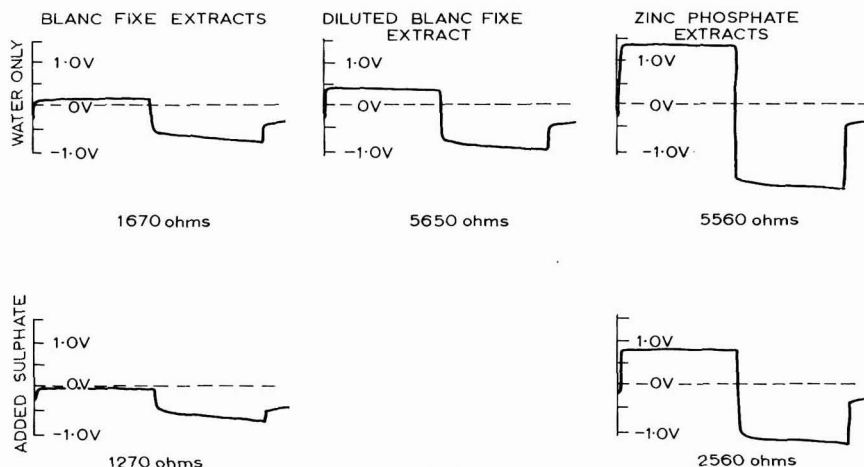


Fig. 1

shows the curves obtained by applying polarising currents, at a density of 100 microamps per sq cm for periods of 25 seconds, to bare steel panels immersed in the extracts of zinc phosphate and blanc fixe with and without added sulphate. The specific resistances of the solutions are shown on the diagram. At these values, the indicated potentials are greater numerically than those at the metal/solution interface and the curves can only be used comparatively.

To minimise the differences resulting from the low conductivity of the zinc phosphate extract, distilled water was added to the blanc fixe extract to give it a similar resistance to that of the zinc phosphate extract.

These tests suggest there is a greater tendency for steel to polarise both anodically and cathodically in extracts from zinc phosphate than in extracts from blanc fixe, and it is assumed that zinc phosphate is sufficiently soluble to cause polarisation effects. It could be that, under a paint film, the active anodic areas are small and the current concentrated, so that the observed anodic polarisation effect becomes of practical importance.

#### The percentage of active pigment to be included in a primer and the most useful pigment volume concentration

Because anti-corrosive pigments are more expensive than inert extenders, there is no point in including more pigment than is necessary; moreover, so far as chromate pigments are concerned, a better primer is not necessarily obtained by including a larger amount of pigment.

There is no doubt that pigment volume concentration plays an important part in determining the behaviour of a primer. Graphs are sometimes seen in the literature which indicate that, as the pigment volume concentration is increased, the corrosion resistance increases until a point near the critical pigment volume is reached, and after this point there is a sharp fall-off in corrosion resistance. There are, however, differences of opinion as to the extent to which particular properties are affected by a change in pigment volume concentration. For example, Brooker, Edwards and

McIntosh<sup>9</sup> indicate that the permeability increases as the pigment volume concentration increases, but this is offset by the tendency to blister's decreasing as the pigment volume concentration is increased, so that optimum conditions are at or below the critical pigment volume concentration of about 55 per cent. On the other hand, other workers, including Hoogerbeets<sup>10</sup> believe the permeability decreases until the critical pigment volume concentration is approached and then increases rapidly.

Brooker, Edwards & McIntosh also describe two statistical techniques used to investigate variables influencing the performance of chromate pigmented epoxy/polyamide primers. As a result of this, they suggest 10-15 per cent zinc tetroxochromate at a pigment volume concentration of 45 per cent, or 25 per cent of barium chromate with a pigment volume concentration of 44 per cent, for optimum performance for the particular resin system. Where there are more than two variables, there are potential advantages in using statistical techniques, but one of the limitations must be the allocation of numerical ratings to a series of subjective assessments.

Very few service specifications directly limit the pigment volume concentration of a primer but most of those applying to zinc chromate insist on a minimum of 15 per cent pigment, and it is found in practice that a pigment volume concentration of 33-35 per cent usually gives the best results. To test whether or not these generalisations applied to a polyurethane oil primer, the experiments summarised in Table 2 were prepared.

Bright steel panels coated with polyurethane oil primers were exposed to

- ammonium salt spray
- sea water spray
- sea water immersion.

In addition, overcoated and scratched panels were subjected to sea water immersion. The sea water immersion tests were assessed by polarisation tests as well as visual inspection, and the numbers show the order of merit, one indicating best behaviour, eight the worst.

Table 2

The effect of variations in pigment concentration and pigment volume concentration in polyurethane oil primers

Environment		Ammonium salt spray	Sea water spray	Sea water immersion		Sea water immersion	
Coating		Primer only scratched	Primer only scratched	Primer only intact		Primer and finish scratched	
Assessment		Visual	Visual	Visual	Polarisation	Visual	Polarisation
%ZC	PVC						
Nil	35	8	8	8	8	8	8
15	30	=2	=5	=3	3	6	=4
15	35	1	=5	=3	2	4	1
15	40	7	7	6	=5	7	=4
25	35	=2	=2	=3	=5	2	=4
35	35	=2	=2	5	=5	=3	3
45	35	=2	=2	2	1	=3	=4
100	35	6	1	1	4	1	2

It will be seen that in every case the worst result was given by the primer which did not contain any zinc chromate. The "datum" mixing, containing 15 per cent zinc chromate having a pigment volume concentration of 35 per cent, behaved well. Decreasing the pigment volume concentration to 30 per cent gave less good results in every case and increasing the pigment volume concentration to 40 per cent gave a distinctly inferior result in every case. In general, there is little evidence to support the use of more than 15 per cent of zinc chromate in a primer based on this medium.

The optimum conditions for the use of zinc phosphate are different from those for zinc chromate. A primer made with only 15 per cent of zinc phosphate will behave less satisfactorily than a similar primer made with 15 per cent of zinc chromate. There is, however, a steady increase in performance as the amount of zinc phosphate is increased and there is no falling off in performance if the zinc phosphate is increased to 100 per cent.

As stated, one of the advantages of zinc phosphate is that it improves the performance of primers applied to rusty surfaces. In making experiments to establish the ideal conditions for the use of zinc phosphate, use has been made of two different media, a styrenated alkyd and a chlorinated rubber. Accelerated tests using bright steel have been supported by outdoor tests on rusty steel.

Two series of primers were prepared:

with 65 per cent zinc phosphate and the pigment volume concentration varied over the range, 27, 30, 33, 36, 39 and 42 per cent;

at a pigment volume concentration of 35 per cent the weight of zinc phosphate in the inorganic solids was varied over the range 55, 60, 65, 70 and 75 per cent.

These variations in levels of pigmentation did not produce very significant differences in behaviour in the accelerated or outdoor tests.

The only tests showing any differences with variation in pigment volume concentration were the outdoor tests in

chlorinated rubber medium (where 27 per cent and 30 per cent pigment volume concentration were slightly better than 33 to 42 per cent pigment volume concentration) and the reverse sides of the accelerated tests using styrenated alkyd medium (where 36 to 42 per cent pigment volume concentration were better than 27 to 33 per cent).

These experiments were followed by further accelerated tests with ammonium salt spray and again using both types of media. The pigment volume concentration was maintained at 33 per cent and 0, 15, 25, 35, 45 and 55 per cent of zinc phosphate were used.

The chlorinated rubber series, with primer only, showed a distinct improvement in performance with increasing amounts of zinc phosphate when examined after 28 days' ammonium salt spray. After 63 days, when the panels were withdrawn from the test, the breakdown had reached a point when there was little difference over the series. The primer and finish panels removed after 63 days' ammonium salt spray showed a progressive improvement between 0 and 55 per cent zinc phosphate. The styrenated alkyd systems, examined as primer only and as primer and finish, also showed a progressive improvement between 0 and 55 per cent zinc phosphate.

From these experiments it appears that 55 per cent of zinc phosphate is required to give optimum performance. The pigment volume concentration does not appear to be very critical and a figure of 33 or 35 per cent should be suitable.

### The relative merits of chromate pigments and zinc phosphate

It has already been pointed out that zinc phosphate has the considerable advantage of being completely innocuous and that primers made from zinc phosphate do not, generally speaking, require elaborate surface preparation. On the other hand, because zinc chromate is much more effective than zinc phosphate at low concentrations, it is often the first choice for primers to be applied to clean steel.

The order of merit of any comparison of pigments will be significantly affected by the type of vehicle and method of assessment. All the pigments mentioned in this paper have specific applications and it is hoped that some guidance on their selection and use has been given.

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# Fungus-resistant paints for breweries and the humid tropics

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## Summary

Fungus-resistant paints for breweries and humid tropical climates are described. Paints containing copper 8-hydroxyquinolate or tetramethyl thiuram disulphide are suitable under conditions in a brewery and alkyd enamels formulated with barium metaborate

and N-trichloromethylthiophthalimide are effective in the humid tropics.

It is shown how fungus-resistant paints can be tested economically on a large scale, and accelerated testing is discussed.

## Keywords

*Types and classes of coating*  
fungus-resistant paints

*Biologically active agents*  
fungicide

*Processes and methods primarily associated with analysis, measurement and testing*  
accelerated testing  
exposure testing

## Peintures résistant à végétation mycélienne pour brasseries et les tropiques humides

### Résumé

On décrit des peintures résistant à végétation mycélienne conçues pour être utilisées dans brasseries et également sous les tropiques humides. Peintures contenant de 8-hydroxyquinolate de cuivre ou de disulfure de tétraméthylthiuram sont convenables pour les conditions sévères qui existent dans les brasseries, et des peintures

alkydes contenant de métaborate de baryum et de N-trichlorométhylthio-phthalimide sont effectives sous les tropiques humides.

On a démontré comment les peintures résistant à végétation mycélienne peuvent être évaluées économiquement à grande échelle, et l'on discute la technique de vieillissement accéléré.

## Pilzanwuchsbeständige Anstrichmittel für Brauereien und die feuchten Tropen

### Zusammenfassung

Eine Beschreibung von pilzanwuchsbeständigen Beschichtungsmitteln für Brauereien und feuchte tropische Klimata. Kupfer 8-hydroxyquinolat oder Tetramethylthiuramdisulfid enthaltende Anstrichfarben eignen sich für in Brauereien vorherrschende Verhältnisse, und Alkydharzemalen, die Bariummetaborat und

N-Trichlormethylthiophthalimid enthalten, sind in den feuchten Tropen wirkungsvoll.

Es wird gezeigt, wie pilzanwuchsbeständige Anstrichmittel im grossen Masstab wirtschaftlich geprüft werden können; Kurzprüfung wird ebenfalls besprochen.

## Краски устойчивые против плесени для пивоваренных заводов и влажных тропиков

### Резюме

Описываются краски устойчивые против плесени для пивоваренной и влажных тропиков. Краски содержащие 8-гидроксинолат меди или тетраметилловый дисульфид тиурама, пригодны для применения в условиях пивоваренных заводов, а алкидные эмали формулированные метабораом бария и N

трихлорметилтиофталимидом, пригодны для применения в влажных тропических климатах.

Показано что краски устойчивые против плесени могут быть испытаны экономично в большом масштабе и обсуждаются ускоренные испытания.

## Introduction

The difficulty encountered in formulating fungus-resistant paints is mainly due to the limited reliability of accelerated test methods that can give a reasonable estimate of the long-term effectiveness of a proposed formulation in a comparatively short space of time. Hoffmann<sup>1</sup> has pointed out that, to develop accelerated test methods successfully, a good deal must be known of the mechanism of the changes that take place, or at least to know which variables influence these changes and the degree to which they do so.

The ground work deemed necessary in order to be able to predict the mould resistance of paints was described briefly in a paper given by Hoffmann to the Second International Biodeterioration Symposium<sup>2</sup>. Of the several types of investigations considered to be important, only one, the

stability of fungicides and fungistatic agents, has been examined by the Division of Building Research, and its importance is too obvious to need any elaboration. This work has provided sufficient knowledge to make it possible to recommend some potentially satisfactory formulations for fungus-resistant paints<sup>3</sup>. The stability tests were carried out by exposing panels under various conditions in the laboratory and outdoors in the tropics. In principle, it is possible to accelerate the weathering of paint films containing the compounds under investigation provided that the correct conditions are selected for the weatherometer in which the tests are carried out, but this has not so far been practicable. However, a recent investigation into the weathering of paint films makes it possible to choose a realistic weatherometer cycle in order to simulate the various climates which are likely

to be encountered. The present state of this project was discussed by Hoffmann in a paper at the 1971 Convention of the Oil and Colour Chemists' Association, Australia<sup>4</sup>.

The present paper deals with fungus-resistant formulations that have been tried out for a considerable length of time in a brewery, where the conditions are very favourable for mould growth, and on a house in the humid tropics at Lae, New Guinea.

### Paints for breweries

The management of a brewery requested the Division to advise on fungus-resistant paints, as the commercially available products had not proved very effective. In most areas of a brewery, the conditions for mould growth are very favourable, firstly because of the high humidity and

secondly because the air is usually contaminated with food particles which are eventually deposited on the surfaces of walls and ceilings.

Because of the uncertainties of extrapolating the behaviour of a fungus-resistant paint from one set of conditions to another, it was decided to carry out experiments before offering any advice. The formulations selected were based on previous experience, especially of what was known regarding the persistence of fungicides under humid conditions. Condensation of water vapour occurs freely on the concrete ceiling over the bottle washing machinery and, consequently, this was chosen as a test area.

Only latex paints were considered, since it would have been impossible to dry the experimental area sufficiently to apply alkyl enamels. The composition of the paints is given in Table 1. The arrangement of the experiment was not ideal

Table 1  
Composition of paints used in brewery

Paint	Pigments	Latex resin	PVC, %	Cu 8-hydroxy- quinolate	N-trichloro- methylthio- phthalimide	Tetramethyl thiuram disulphide
A (blank)	r.TiO <sub>2</sub>			—	—	
B				—	5	
C				5	—	
D	ZnO and r.TiO <sub>2</sub> weight ratio 2 : 1	Vinyl acetate/ acrylate copolymer	25	—	—	Nil
E				—	5	
F				5	—	
G (blank)	r.TiO <sub>2</sub>			—	—	
H				—	5	
I				5	—	
K	ZnO and r.TiO <sub>2</sub> weight ratio 2 : 1	Styrene terpolymer*	25	—	—	Nil
L				—	5	
M				5	—	
N (blank)	r.TiO <sub>2</sub>			—	—	
O				—	5	
P				5	—	
R	ZnO and r.TiO <sub>2</sub> weight ratio 1.2 : 1	Vinyl acetate/ acrylate copolymer	40	—	—	Nil
S				—	5	
T				5	—	
U (blank)	r.TiO <sub>2</sub>			—	—	
V				—	5	
W				5	—	
X	ZnO and r.TiO <sub>2</sub> weight ratio 1.2 : 1	Styrene terpolymer*	40	—	—	Nil
Y				—	5	
Z				5	—	
1	ZnO	Styrene terpolymer*	25			—
2	r.TiO		25			4
3			25			8
4	ZnO		40			—
5	r.TiO		40			4
6			40	Nil	Nil	8
7		Acrylic	40			8
9			40			8
11			40			8
8			40			—
10			40			—
12			40			—

\* Lytron 684

from a statistical point of view but, because of the restricted area available, a compromise had to be made. Nevertheless, the conditions prevailing in this area of the building were very uniform and there can be little doubt as to the correctness of the conclusions drawn from the experiment.

Paints were made up at 25 and 40 per cent PVC levels with each of two different latices, a vinyl acetate/acrylate copolymer (15 per cent acrylate) that is widely used in Australia, and a styrene terpolymer. Experience in the Division has shown that paints made up with the latter resin show a considerably higher adhesion to wet surfaces than do paints made with other latices. The fungicides tested and the reasons for their choice are detailed hereunder.

#### N-trichloromethylthiophthalimide<sup>5</sup>

This fungicide was selected for test because of its comparative stability in a fog room and because it had proved effective when it was used in houses of the Housing Commission, Victoria and in a dye works. The test strip, coated with paints containing this fungicide, is second from the left in Figs. 1 and 2 (areas B, O, H, V), and this shows that,

although there was a slight reduction in mould growth at the end of 11 months (compared with the blank), the effect did not persist to 17 months. For practical purposes the difference was insignificant.

#### Copper 8-hydroxyquinolate<sup>6</sup>

This compound shows a high fungicidal activity when added to paint and is also very persistent in the film. The areas C, P, I, W (third row from left, Figs. 1 and 2) coated with paint showed very little mould after 17 months, and the improvement compared with the blank is worthwhile.

#### Zinc oxide<sup>7</sup>

Latex paints containing zinc oxide and of the same formulation as those used in the present tests are very effective in the dwellings of the Housing Commission, Victoria, and this type is now used exclusively by the Commission.<sup>8</sup> Its effectiveness is illustrated in the first figure in reference.<sup>9</sup> However, under the conditions prevailing in the brewery, this formulation showed no worthwhile difference from the blank at



Fig. 1. Test area on ceiling over bottle washing machine in brewery, 11 months after painting with formulations detailed in Table 1

Left to right:

First strip—Blank. Second strip—Formulations B, O, H, V containing N-trichloromethylthiophthalimide  
 Third strip—Formulations C, P, I, W containing copper 8-hydroxyquinolate. Fourth strip—Formulations D, R, K, X containing zinc oxide. Fifth strip—Formulations E, S, L, Y containing zinc oxide and N-trichloromethylthiophthalimide. Sixth strip—Formulations F, T, M, Z containing zinc oxide and copper 8-hydroxyquinolate.



Fig. 2. Test area of Fig. 1, 17 months after painting

any time, as indicated in areas D, R, K and X in Figs. 1 and 2 (fourth row from left).

#### Zinc oxide and N-trichloromethylthiophthalimide

For about 12 months, paints containing these two compounds show a higher fungus resistance than coatings containing either of them alone, that is, they show a synergistic effect. The effect starts to disappear after about 12 months and by about 17 months there is scarcely any significant difference. This is demonstrated by the areas E, S, L and Y in Figs. 1 and 2 (fifth row from left).

#### Zinc oxide and copper 8-hydroxyquinolate

The combination was chosen to check for any synergistic effects, but there was no appreciable difference between the effects of the combination and of copper 8-hydroxyquinolate alone. The high fungus resistance of the paints

containing the combination can be seen in areas F, T, M and Z in Figs. 1 and 2.

#### Tetramethyl thiuram disulphide (TMTD)<sup>10</sup>

It was concluded that an addition of from 4 to 6 per cent of this fungicide on the solid content of the paint would be likely to increase the mould resistance for more than three years. This prediction was made on the assumption that conditions would be similar to those prevailing in the dwellings of the Housing Commission, Victoria. More condensation takes place in the test area in the brewery than in an ordinary dwelling, and consequently a shorter effective service time could be expected.

The results are illustrated in Figs. 3 and 4 (areas 2, 3, 5 and 6). After 13 months, all the panels coated with paints containing TMTD are still in excellent condition (Fig. 3). After 17 months (Fig. 4), areas 2 and 5 (4 per cent TMTD) show some mould, whereas panels 3 and 6 (8 per cent TMTD)





Fig. 3. Test area on ceiling over bottle washing machine in brewery, 13 months after painting

Panels 1 and 4—Formulation containing zinc oxide.

Panel 3—Formulation containing 8% TMTD, PVC 25%.

Panel 2—Formulation containing 4% TMTD, PVC 25%.

Panel 5—Formulation containing 4% TMTD, PVC 40%.

Panel 6—Formulation containing 8% TMTD, PVC 40%.

are still practically free. TMTD was also tried in the cold room (55°F) of the brewery, and its effect in suppressing the growth of mould for five months is illustrated in Fig. 5. The fungicidal paint is expected to stay effective for a much longer period than on the ceiling over the bottle washing machine, because the condensation on the wall is not as great and also because the growth of mould will be retarded at the lower temperature.

The slight change in intensity of mould growth between paints formulated to 25 and 40 per cent PVC, which is of no practical importance, cannot be ascribed with any certainty to the difference in the PVC. The same comment applies to the influence of the binder on the fungus resistance of the paint.

#### Paints suitable for decoration in breweries

On the basis of the results of the foregoing tests the following paints should be suitable for breweries and other establishments where conditions favour copious growth of mould (high humidity, high condensation of water vapour, and food particles in the air.)

#### Paints containing copper 8-hydroxyquinolate (CuH)

Formulations P or T (Table 1 and Fig. 2) should be suitable and, in areas where the condensation of water vapour is very high, the appearance would be acceptable for between one and two years. The loss of CuH under these conditions appears to be very slow<sup>6</sup>, and it would therefore be expected that the original fungicidal surface would be restored after washing the mould off with hypochlorite solution, a procedure which has been described elsewhere.<sup>11</sup> This could be carried out during the yearly maintenance period and is much cheaper than repainting. The time for which this maintenance would remain effective will depend on the conditions, but it is estimated that even in the worst areas a reasonably pleasing appearance will be maintained for about three years, and for much longer periods on surfaces where conditions for mould growth are less favourable.

No differences have yet been noted between the mould resistance of paints containing CuH and that of paints also containing zinc oxide. The paints formulated only with rutile titanium dioxide as a pigment have a very slight green yellowish colour, whereas the paints containing zinc oxide are a little darker. One disadvantage is that the colour becomes

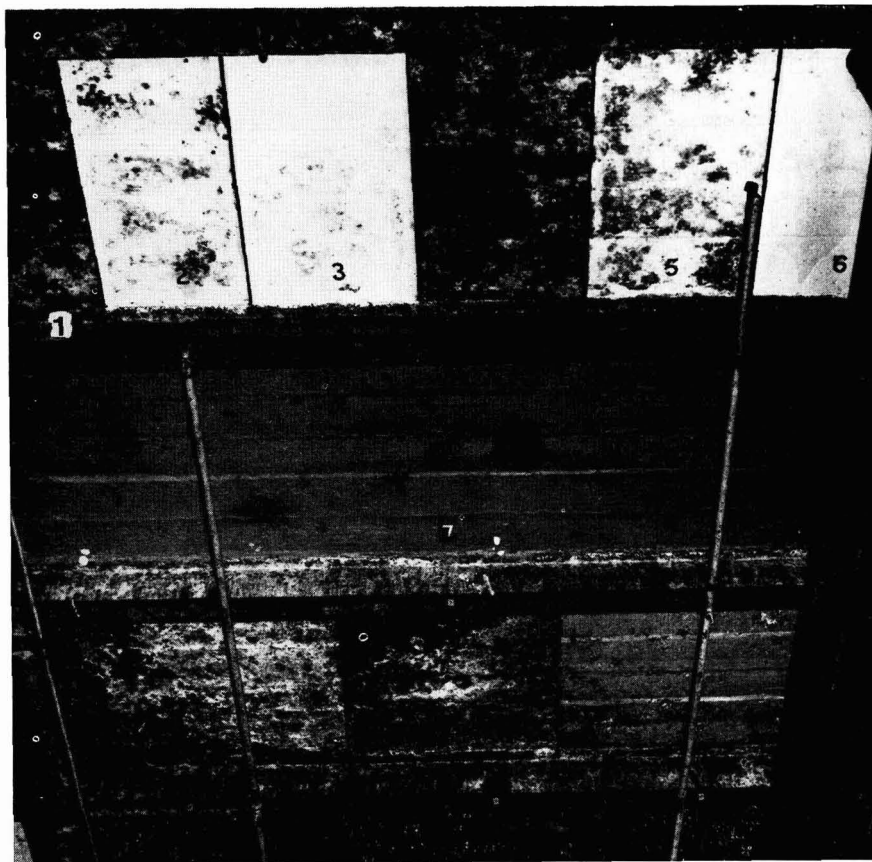


Fig. 4. Test area of Fig. 3, 17 months after painting

somewhat darker with age, probably due to the formation of copper sulphide by the reaction of CuH with hydrogen sulphide, which is a frequent trace constituent in air<sup>12</sup>. The original colour could be restored by treatment with hypochlorites if necessary.

#### *Paints containing tetramethyl thiuram disulphide*

Formulations 2, 3, 5 or 6 (Table 1) should be suitable. Paints 3 and 6 contain 8 per cent of TMTD on the non-volatiles of the paint and would probably remain essentially free of mould for approximately two years, but there is no reason why the concentration of TMTD should not be increased to extend the mould-free life. The fungicide is a cheap commercial product. Washing with hypochlorite probably would not restore the surface to its original fungicidal activity because of loss of the active compound.

The formulation has one severe disadvantage. TMTD is an antioxidant, and if a surface on which it has been used is overpainted with a coating which dries by oxidation, drying will be retarded or completely prevented. Care must be taken not to apply air-drying paints over a formulation containing

TMTD, or, if this cannot be avoided, to apply an insulating coat of a latex paint first.

#### *Paints containing zinc oxide and N-trichloromethylthiophthalimide*

Formulations E, S, L and Y (Table 1 and Figs. 1 and 2) are not sufficiently fungus resistant for the conditions prevailing on the concrete ceiling over the bottle washing machine, but could be of use in areas where the condensation is not so great. The advantage of these formulations is that they can be used for white paints and that they do not contain any antioxidants that could lead to trouble if the surface is overpainted with an air-drying paint.

#### **Paints for the humid tropics**

One of the major inconveniences in the humid tropics is the growth of mould on exterior and interior painted surfaces, giving the decoration an unsightly appearance and necessitating frequent washing or redecoration. It was concluded from the investigations on fungus-resistant paints<sup>3</sup> that certain



Fig. 5. Test area on wall in cold room of brewery, 5 months after painting  
Formulations 7, 9, 11 contain TMTD; others are blanks

formulations should be very effective for exterior conditions, and large-scale tests were carried out on these. In order to obtain reliable results, each wall of a test house was divided into a series of four strips A, B, C and D, and these were varied in a statistically predetermined pattern. Compositions of the four paints used are set out in Table 2. The mould

growth was assessed under the eaves and at the top, middle and bottom of each wall at intervals of approximately three months. The degree of mould growth on the four walls at the end of 23 months is shown diagrammatically in Fig. 6.

Table 2

Main ingredients of paints used on test house at Lae, New Guinea

Paint	Resin* solution	r.TiO <sub>2</sub>	Barium metaborate	N-trichloro- methyl- thiophthalimide
A (blank)	51	32.4	—	—
B	51	32.4	—	2.3
C	51	14	14	—
D	51	14	14	2.3

#### Composition in Parts by Weight

\*Long oil soya bean pentaerythritol alkyd solution (70% NVM)

Stability—Tins of paint 2½ years old and stored unopened for 2 years at Lae showed the following characteristics:

Paint A (blank), easily dispersed

Paint B, partly gelled but could be easily dispersed and brushed out

Paint C, somewhat harder to disperse than paint A

Paint D, easily dispersed, same as Paint A

Paints A, C and D dried normally when brushed out

Paint B, distinctly lower rate of drying



The numbers indicate the assessment according to the scale from 0 (no mould) to 7 (heavily infested). The photograph of a section of a wall (Fig. 7) gives an indication of the

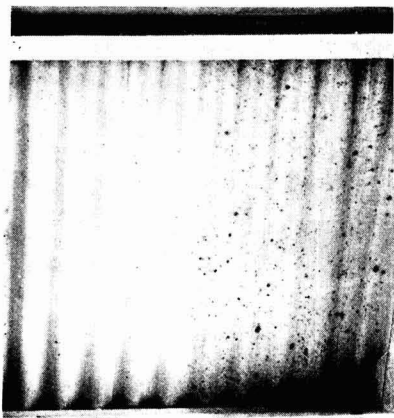


Fig. 7. Mould growth under eaves of test house in Lae. Left, mould rating 0; right, mould rating 5

ratings 5 and 0. To facilitate interpretation of the results of the various assessments, the assessments over each strip have been added up and the sum so obtained divided by the number of strips (Table 3). For example, on the north-west wall there are eight A strips with a total rating of 50, or 6.3 per strip. The number 6 is a good indicator of how the section would look if it were painted with coating A only.

The following conclusions are evident: mould growth appears mostly under the eaves, the exception being on the north-east wall. Paint B is not sufficiently effective under the eaves, but is more effective on the wall sections. Paints C

and D are most effective on all parts of the house. There is an indication that in the long term D may prove to have a slightly higher fungus resistance than C.

On the basis of the experiments described here and elsewhere, the composition of paints recommended for use in the humid tropics is as given in Table 4.

### Accelerated tests

An accelerated test should make it possible to decide in a few weeks or months how long a supposedly fungus-resistant formulation will remain free of mould in a particular environment. For this purpose it is necessary to know what happens to a fungicide or fungistat in a given environment, and how to characterise the conditions for mould growth. The first is comparatively easy to determine, because the variables which can influence the persistence of the fungicide or the fungistat agents in the paint film (e.g. humidity, sunshine, reactions with hydrogen sulphide or carbon dioxide) in the air are known, and the corresponding conditions for any site can be simulated in a suitable device. However, at the moment it is much more difficult to define the conditions for mould growth in any precise way, because these will depend to a large degree on the adaptability of the organism to varying conditions of humidity and food supply. Nothing is known of this so far, and possibly only a study of the metabolism of the mould will clarify the point.

The following examples will illustrate what is meant by the above statement. Paints formulated with zinc oxide are reported not to show any fungus resistance when tested by the Vicklund-Manowitz procedure.<sup>13</sup> This correlates well with the behaviour of these paints in the brewery or on the under side of panels exposed at Lae, New Guinea. However, it does not correlate with the behaviour in other locations, where the same formulation has been shown to be effective,<sup>2,8</sup> and it must be concluded that the Vicklund-Manowitz test reproduces the conditions prevailing in some locations but not in others.

Table 3

Summary of assessments on test house at Lae, New Guinea

Wall	Position on wall	Exposure																							
		7 months				10 months				13 months				16 months				20 months				23 months			
		A	B	C	D	A	B	C	D	A	B	C	D	A	B	C	D	A	B	C	D	A	B	C	D
NW	Under eaves	4	3	0	0	4.5	2.5	0	0	5	4	0	0	6	4.5	0	0	6	5	0	0	6.3	5.5	0.5	0.25
	Top	0	0	0	0	2.5	0	0	0	2.5	0	0.5	0	3.5	0	1.5	0	3.5	1	2	0.5	4.3	1.4	3	0.8
	Middle	0	0	0	0	0	0	0	0	3	0	1	0	4	1	1	0	3.5	1.5	0	0	3.8	1.5	1	0.3
	Bottom	0	0	0	0	3	0	0	0	3	1	0	0	3	2	0	0	3	2	0	1	3.3	2	0.5	0.8
SE	Under eaves	1	0	0	0	2.7	0.6	0	0	4	2.5	0	0	4.5	4	0	0	4.5	4.5	0	0	4.6	4.4	0.3	0.3
	Top	0	0	0	0	1.3	0	0	0	2.5	0	0	0	2.5	0	0	0	2.5	0	1	0	3	0.3	1	0
	Middle	0	0	0	0	0.4	0	0	0	1	0	0	0	0	0	0	0	1	0	0	0	1	0	0.3	0
	Bottom	0	0	0	0	0.6	0	0	0	1	0	0	0	1	0	0	0	1	0	0	0	1.2	0	0.5	0
SW	Under eaves	4	0	0	0	4	0	0	0	4	1.4	0	0	5	2	0	0	6	4	0	0	6	4.4	0	0
	Top	0	0	0	0	1	0	0	0	2	0	0	0	2	0	0	0	3.5	0	1	0	3	0.3	1.5	0
	Middle	0	0	0	0	1.3	0	0	0	2	0	0	0	2.5	0	0	0	2.5	1	1	0	2.5	0.8	0.3	0
	Bottom	0	0	0	0	1	0	0	0	2.5	0	0	0	3	0	0	0	3	0.5	1	0	3.5	0.8	1	0.6
NE	Under eaves	0	0	0	0	1	0	0	0	0	0	0	0	0	0	0	0	1	1	0	0	1	1	0.2	0
	Top	0	0	0	0	1	0	0	0	1	0	0	0	2	0	0	0	2.5	0	1.5	0	3.5	0.5	2.6	0.7
	Middle	0	0	0	0	0	0	0	0	2	1	0	0	3	1	0	0	4	2	0.5	0	4.3	3	0.7	0
	Bottom	0	0	0	0	2.7	0.7	0	0	3.5	2	0	0	2.5	2	0	0	4	1.5	0.5	0	3.8	2	0.6	0



Table 4  
Composition of paints recommended for use in humid tropics

Paint type	Resin (%)	Rutile titanium dioxide (%)	Fungistatic extender or pigment (%)	Fungicide (N-trichloromethylthio-phthalimide) (%)
Gloss enamel for exterior or interior	55*	22	22 BMB	2.8
Flat enamel for interior use	** **	≤35 ≤30	≤20 BMB ≤35 ZnO	2.5 2.5
Flat latex for interior use	** **	≤35 ≤30	≤20 BMB ≤35 ZnO	2.5*** 2.5***
Primer for timber	Either 100% acrylic latex or linseed oil primer plus 1.5% copper 8-hydroxyquinolate			

Percentage by weight on total resins, pigments and extenders.

\* Long soya bean or long safflower oil pentaerythritol alkyl.

\*\* Pigment volume concentration not greater than 50.

\*\*\* Not stable in latex paint and has to be added just prior to use.

BMB = Barium metaborate.

Even more striking are the observations made with N-trichloromethylthio-phthalimide. This fungicide has only a comparatively low rate of disappearance from paint films, and it has been shown to be effective on exterior exposure on panels at Lae.<sup>8</sup> A gloss enamel paint containing the fungicide proved effective on the upper and under sides of the panels, and it could be expected that this formulation would be effective on the test house in Lae. This is only partly correct, and the fungus resistance under the eaves is scarcely better than that of a paint free from the fungicide. This means that the conditions under the eaves differ from those on the under side of the test panel, which would have been expected to be similar. On the wall itself, however, the expected correlation is borne out. At present it is impossible to say how the conditions differ, and it is therefore impossible to devise an accelerated test which simulates these conditions.

Copper 8-hydroxyquinolate is a fungicide which is shown to be effective by the Vicklund-Manowitz test,<sup>9</sup> and it has also been proved to be very persistent in the paint film. On the exposure site at Lae, paints formulated with this fungicide are fungus-resistant on the upper side of the exposed panels but not on the under side. The conditions in the Vicklund-Manowitz test imitate those on the upper side but not those on the under side.

It is therefore evident that any extrapolation from accelerated tests, including panel exposures on a chosen site, is rather uncertain, and it is pertinent to consider how the fungus resistance of a formulation may be readily assessed. It is believed that the most rational method at present is to determine the fungicidal activity by a suitable bio-assay technique—this could be the Vicklund-Manowitz test or one of its variations, the test proposed by Hendey<sup>14</sup> or one of its variations<sup>13</sup>—and then to determine the persistence of the fungicide or fungistatic agent in the paint film on exposure. The latter could be achieved by accelerated testing, because the physical conditions prevailing on the various exposure sites can be established and simulated in suitable equipment. However, it is not sufficient simply to expose a paint film and then see if it still has fungicidal properties, since this has led to very misleading conclusions.<sup>13</sup> Suppose that two fungicidal formulations, after exposure in a weatherometer, are found to retain some fungus resistance properties. The conclusion

from this is not fully reliable, because one fungicide may be very stable whereas the other may have largely decomposed, leaving just enough to show some residual resistance to the growth of fungus. Clearly there is a very important difference between these two cases, and the two compounds tested are by no means equivalent. Paints formulated with the less stable compound will have a very short service life, whereas coatings incorporating the other compound will last much longer, and this difference can be found only by analysing for the remaining active compound. The rate at which a fungicide disappears is the important factor.

Once it has been established that the fungicidal compounds are sufficiently stable, a field test should be carried out in the manner discussed earlier, and similar tests should be used for interior paints. A test such as that described by Hoffmann<sup>9</sup> is comparatively cheap and yields a wealth of information which would be very costly to obtain in any other way.

A warning may be appropriate. Some workers might think that the large-scale test described here could be simplified by dividing the wall into only four parts and applying one of the paints to each of them. This could be very misleading, as an inspection of Fig. 6 will show. On the south-east wall, for example, paint B performs much better in the middle, where the mould growth is assessed as 3, than on the left-hand corner, where the assessment is 6. On the south-west wall, paint B does not show any mould growth in the middle area, whereas there is strong mould growth on the left and right-hand sides of the wall. Another example for the need to subdivide the area to be painted into a reasonable number of strips is given by Hoffmann.<sup>9</sup>

### Cost of the paint

A fungus-resistant paint is more expensive than a normal coating and this is very often considered to be an economic drawback to its use. This is an unreasonable way of looking at the problem, because for every dollar spent on paint in Australia something like \$4-8 is spent on labour to apply it. This means that a 20 per cent increase in the price of the coating makes the whole job only a few per cents dearer, and

anybody who considers this to be an excessive increase cannot have a serious problem as far as mould is concerned.

As most Australian paint manufacturers are prepared to charge the customer only the higher cost of raw material of the paint, the increased price of a fungus-resistant paint should be no serious bar to its use.

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# The micro analyses of solvent mixtures by reaction gas chromatography\*

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## Summary

Reaction gas chromatography applies the classical reactions of organic chemistry to the identification of gas chromatographic peaks by chemical reaction or absorption of specific functional classes of compounds conducted within the gas chromatographic equipment.

Many types of compounds encountered in the surface coatings industry are amenable to chromatographic chemical reaction and the techniques applicable to the identification of alcohols, esters, and ketones and more complex compounds containing these functional groups have been examined and a procedure described for the microanalysis of solvent mixtures.

## Keywords

*Raw materials—solvents*  
solvent

*Processes and methods primarily associated with analysis, measurement and testing*  
reaction gas chromatography

## La microanalyse de mélanges de solvants au moyens de la chromatographie de réaction en phase gazeuse

### Résumé

Dans la technique de chromatographie de réaction en phase gazeuse, on utilise les réactions classiques de la chimie organique pour identifier les pics de la chromatographie en phase gazeuse au moyens d'une réaction chimique ou au moyens de l'absorption d'un genre de composés spécifiquement fonctionnels effectués dedans l'appareil chromatographique.

Beaucoup de types des composés utilisés par l'industrie de revêtements superficiels sont susceptibles à une réaction chromatographique et chimique, et les techniques valables pour l'identification d'alcools, ester, cétones et des composés plus complexes qui renferment ces groupements fonctionnels ont été examinées et l'on décrit un procédé pour effectuer la microanalyse des mélanges de solvants.

## Die Mikroanalysen von Lösungsmittelgemischen durch Reaktionsgaschromatographie

### Zusammenfassung

Reaktionsgaschromatographie verwendet die klassischen Reaktionen der organischen Chemie zur Identifizierung gaschromatographischer Spitzen hervorgerufen durch chemische Reaktionen oder Absorption spezifischer funktioneller Klassen von Verbindungen, welche in der gaschromatographischen Apparatur vor sich gehen.

Viele Arten von in der Lackindustrie benutzten Verbindungen eignen sich für chromatographisch chemische Reaktionen. Die für die Identifizierung von Alkoholen, Estern und Ketonen, sowie komplizierteren Verbindungen, welche diese funktionellen Gruppen enthalten, üblichen Techniken wurden geprüft, und ein Verfahren für die Mikroanalyse von Lösungsmittelmischungen beschrieben.

## Микро-анализ растворяющих смесей при помощи реактивной газовой хроматографии

### Резюме

Реактивная газовая хроматография применяет классические реакции органической химии для опознавания газовых хроматографических пиков, при помощи химических реакций или поглощением специфических функциональных классов соединений, проводимых в газовом хроматографическом оборудовании.

Многие типы соединений применяемые в промышленности поверхностных покрытий, поддаются хроматографическим химическим реакциям, и анализируется техника применяемая для опознавания спиртов, эфиров и кетонов и более сложных соединений содержащих эти функциональные группы, и описывается процедура для микроанализа растворяющих смесей.

## Introduction

Whilst the application of gas chromatography to coating analyses is increasing<sup>1</sup>, the major area of interest continues to be associated with solvent mixtures. With the early realisation that a reliable analysis is generally not possible using a single gas chromatographic examination, a number of supplementary procedures have been reported. Haslam and his co-workers<sup>2</sup> employed two columns of different polarity to resolve complex solvent mixtures, whilst Gatrell<sup>3</sup> used a mixed stationary phase. Temperature programmed operation has been described by Esposito<sup>4</sup>. The limitations of these and other schemes have been described in an earlier

paper<sup>5</sup>, in which an analytical procedure employing the sequential application of solubility tests to remove various chemical classes by pre-chromatographic chemical reaction was described.

Removal of various chemical classes may also be carried out within the chromatograph by chemical reaction or by absorption. Between the injection point and the detector, a reaction zone is employed where, ideally, reaction takes place instantaneously. In practice, this condition is not always achieved but reaction periods of the order of ten seconds are satisfactory, depending on the separation required. An excessively slow reaction may cause peak broadening such

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that identification based on a knowledge of retention data is questionable.

Reaction gas chromatography has now been found of value in the analysis of solvent or thinner mixtures used in the coatings industry, and a technique which has been successfully employed is described in this paper.

With the elimination of chemical reaction external to the gas chromatograph, only minute quantities of sample are necessary and a complete characterisation of the solvent mixture from a fraction of a gramme of coating is possible. The preferred method of removal of solvent from pigments and resins in this laboratory is by low-temperature vacuum fractionation, as described by Haslam and his co-workers<sup>2</sup>. The removal of a particular chemical class is observed after gas chromatographic examination and an interpretation of the identity of the peaks removed is made by reference to retention data.

Several reviews of the literature of reaction gas chromatography have appeared<sup>6, 7</sup>. Many situations that have been reported refer to a specific application, and experimentation is necessary in attempting to determine general applicability.

Molecular sieves<sup>8, 9</sup>, sulphuric acid of varying strengths<sup>10</sup> and mercuric perchlorate<sup>11</sup> have been successfully employed with hydrocarbons for removal of saturates and unsaturates, but are less selective with mixtures that include other chemical types, as partial removal of many other compounds occurs simultaneously. The identification of hydrocarbons that normally occur as mixtures has previously been demonstrated by comparative means after removal of other functional classes by treatment with dilute sulphuric acid<sup>5</sup>. In the present work, comparative or "fingerprint" identification is carried out after use of the abstractor columns.

Instruments containing multiple ovens, columns and detectors, in which analyses of this type may be conducted from a single injection, have been commercially available<sup>12</sup> and have found some application in the petroleum industry. For simplicity, the results reported have been carried out on a simple instrument where small independently heated precolumns are connected via a syringe needle into the injection port of the chromatograph, an injection point on the precolumn allowing admission of the sample.

## Reaction gas chromatography

Reaction gas chromatography allows many of the classical reactions of organic chemistry to be conducted within the chromatographic apparatus. Alcohols, esters, ketones, aromatic and aliphatic hydrocarbons as experienced in solvent mixtures are all amenable to this technique and may be selectively removed by subtractive procedures within the chromatograph.

Boric acid coated supports have been reported to abstract most primary and secondary alcohols by the formation of trialkyl borate esters which are retained in the column. Tertiary alcohols are generally not abstracted, or dehydration may occur with the formation of alkenes<sup>13, 14, 15, 16</sup>. The reaction with tertiary alcohols is increased as the concentration of boric acid in the reaction column increases<sup>15, 16</sup> and has been reported as a means of distinguishing primary and secondary alcohols from tertiary alcohols<sup>16</sup>.

The abstraction of alcohols may also be carried out using a precolumn containing support coated with 3-nitrophthalic anhydride<sup>17</sup>. The column was operated to 200°C, and both

saturated and unsaturated primary alcohols were removed, whilst most secondary alcohols were substantially removed. Tertiary alcohols were partially dehydrated, as is the case with boric acid. Unsaturated alcohols with an allylic double bond, i.e. 2-alken-4-ols, which undergo dehydration with boric acid to form the conjugated diene, are abstracted by the 3-nitrophthalic anhydride.

The saponification of malonic esters using a precolumn containing wet potassium hydroxide has been reported by Janak and his co-workers<sup>18</sup>. The alkaline salts formed are retained in the column, the alcohols formed passing into the gas chromatograph.

Aldehydes may be reacted with aromatic amines to form Schiff bases. Fourteen aldehydes were abstracted by Bieri and co-workers<sup>15</sup> using a 6in column packed with 5 per cent *o*-dianisidine on an Anakrom ABS support and operated between 50 and 175°C. It is reported that cyclohexanone was abstracted, whilst other ketones were not affected until the reagent concentration was substantially increased, to about 20 per cent.

The same workers used a more reactive amine, i.e. benzidine, to abstract aldehydes, epoxides and most ketones, but partial removal of simple esters, ethers and alcohols also occurred. Reagent at a concentration of 20 per cent was used in a 6in column operated between 100 and 175°C. Several recent reports<sup>17, 19</sup> indicate that both of these amines give rise to serious bleed problems and, with their limited selectivity, neither can be considered to be really satisfactory. The stationary phase FFAP<sup>20</sup> abstracts certain aldehydes by an unknown mechanism<sup>21</sup>. A 6in column containing 20 per cent FFAP and operated within the temperature range 75 to 200°C was reported<sup>15</sup> to retain most aldehydes and several epoxides. Recent evaluations of FFAP as an aldehyde abstractor have been reported by Cronin<sup>17</sup> and Withers<sup>22</sup>, and both indicate the serious limitations of the material.

Butylaldehyde was removed from photolysis mixtures by Kerr and Trotman-Dickenson<sup>29</sup> using a mull of sodium bisulphite in ethylene glycol. Acetone was not removed and no mention was made of any other carbonyl compounds.

The established reaction for the characterisation of carbonyl compounds by the formation of their semicarbazones has been applied to reaction gas chromatography by Cronin<sup>17</sup>. A wide variety of aldehyde types were successfully abstracted, as were most methyl ketones and cyclopentanone. Other ketones were partially abstracted. Side reactions, namely self-condensation to form the biurea derivative and the formation of azines, limited the operating temperature to approximately 115°C.

The differential reactivities of metal hydrides towards alcohols, aldehydes, ketones, esters and epoxide in gas phase and solution reactions have been reported<sup>16</sup>. Sodium borohydride, lithium borohydride and lithium aluminium hydride precolumns were prepared by the admission of 200, 10 and 10mg of the respective reagents into an injection port column liner maintained at 150°C. A general lack of selectivity was evident, all oxygen-containing functional groups being removed except ethers, of which only one compound was indicated.

## Experimental

### Gas chromatography

Gas chromatography was carried out using a modified F & M 810/29 Research Chromatograph with simultaneous flame ionisation and thermal conductivity detection and fitted

with an improved flow control system. Two 12ft  $\times$   $\frac{1}{2}$ in o.d. aluminium columns were packed with 10 per cent of the stationary phase on 60/80 mesh acid-washed and silanised Celite 560, and operated under isothermal or programmed conditions as necessary to effect separation of the particular compounds examined.

A recorder with centre zero was found convenient and allowed injection into one column containing the abstractor and comparison injection of the sample into the other column without any manipulation. Retention data from a number of early reports concerning solvent analyses has been tabulated by Hundert<sup>23</sup> while more recent data is also available<sup>24, 25, 26</sup>, and is not reproduced in this work.

#### Abstractor column unit

The abstractor column unit is based on the Beroza-NIL Carbon Skeleton Determinator<sup>27</sup> which provides a convenient temperature-controlled column of dimensions 10in  $\times$   $\frac{1}{2}$ in capable of operation independently of the analytical column. A more versatile design of the abstraction unit is shown in Fig. 1.

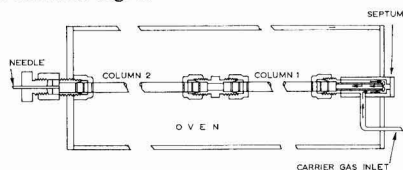


Fig. 1. Diagram showing abstractor column unit

#### Abstractor columns

The formation of bisulphite addition compounds was conducted using 10 per cent sodium metabisulphite deposited on Celite (acid-washed and silanised) 60-72 mesh from aqueous solution. The bisulphite was dissolved in the minimum amount of water necessary to cover the Celite and was evaporated in a rotary evaporator over a boiling water bath in a gentle stream of air to facilitate removal of water. The packing was oven dried at 100°C for 30 minutes prior to preparation of the column. A 6in column was operated at 65°C for optimum formation of the addition compounds.

The boric acid column was prepared by coating Celite, acid-washed and silanised, with 3 per cent boric acid and 10 per cent Carbowax 20M from chloroform solution, using the procedure described above.

The lithium hydroxide column was prepared by coating 3 per cent lithium hydroxide and 10 per cent polyethylene glycol on to Celite, acid-washed and silanised, from methanol using a rotary evaporator as described. A 6in column was used at several temperatures.

#### Results

The compounds that have been examined are presented in Table 1, where the results of the abstraction columns are shown. Some peak broadening was observed for the higher boiling compounds; this was most apparent with the ketones but was not sufficiently serious to interfere with the scheme of identification.

#### Key

- (x) Registered trade name of Shell Chemicals Ltd.  
 (xx) Registered trade name of Union Carbide Corporation  
 (xxx) Registered trade name of ICI Ltd.  
 (xxxx) 3,3,5-trimethyl cyclohexene-2-one-1  
 (1) Completely or virtually completely removed.  
 (2) Partially removed.  
 (3) Considerable band spreading evident.

Table 1—Abstraction of chemical types

Compound	BP (a)	Boric acid column	Bi-sulphite column	Lithium hydroxide and boric acid columns
<i>Alcohols</i>				
Methanol	64.5	1	—	1
Ethanol	78.3	1	—	1
Isopropanol	82.3	1	—	1
<i>tert</i> -Butanol	82.5	2	—	2
<i>n</i> -Propanol	97.2	1	—	1
<i>sec</i> -Butanol	99.5	1	—	1
Isobutanol	107.2	1	—	1
<i>n</i> -Butanol	117.7	1	—	1
2-Methyl butanol	129.8	1	—	1
Pentoxol (x)	165.0	1	—	1
Di-isobutyl carbinol	178.0	1	—	1
2-Ethyl hexanol	184.0	1	—	1
3-Methoxybutanol	161.2	1	—	1
<i>Ketones</i>				
Acetone	56.1	—	1	—
Methyl ethyl ketone	79.6	—	1	—
Diethyl ketone	102.7	—	1	—
Methyl <i>n</i> -propyl ketone	102.3	—	1	—
Methyl <i>isobutyl</i> ketone	116.2	—	1	—
Methyl <i>n</i> -butyl ketone	127.2	—	1	—
Mesityl oxide	128.3	—	1	—
Methyl <i>isoamyl</i> ketone	144.7	—	1	—
Methyl <i>n</i> -amyl ketone	151.4	—	1	—
Cyclohexanone	156.7	—	1	—
Pentoxone (x)	160.0	—	1	—
Di- <i>iso</i> -butyl ketone	169.4	—	1	—
Diacetone alcohol	169.2	1	—	1
Isophorone (xxxx)	215.2	—	1, 3	—
<i>Esters</i>				
Methyl acetate	57.1	—	—	1
Ethyl acetate	77.2	—	—	1
Isopropyl acetate	89.0	—	—	1
<i>n</i> -Propyl acetate	101.6	—	—	1
<i>tert</i> -Butyl acetate	98.0	—	—	1
<i>sec</i> -Butyl acetate	112	—	—	1
Isobutyl acetate	116.5	—	—	1
<i>n</i> -Butyl acetate	126.5	—	—	1
<i>n</i> -Amyl acetate	148.0	—	—	1
Methyl Cellosolve acetate (xx)	144.5	—	—	1
Cellosolve acetate	156.4	—	—	1
Butyl Cellosolve acetate	192.2	—	—	1
Methyl Carbitol acetate (xx)	188.0	—	—	1
Carbitol acetate	217	—	—	1
Butyl Carbitol acetate	246.7	—	—	1, 3
Ethylene glycol diacetate	190.0	—	—	1
3-Methoxy butyl acetate	171.3	—	—	1
<i>Ether</i>				
Methyl Cellosolve	124.6	1	—	1
Cellosolve	135.6	1	—	1
Isopropyl Cellosolve	142.0	1	—	1
Isobutyl Cellosolve	160.0	1	—	1
Butyl Cellosolve	171.1	1	—	1
Methyl Carbitol	194.0	1	—	1
Carbitol	201.0	—	—	1
<i>n</i> -Butyl Carbitol	230.0	1, 3	—	1
Methyl Tri-Icinol (xxx)	—	1	—	1
Tri-Icinol	—	1	—	1
Butyl Tri-Icinol	—	1, 3	—	1
Ucar LM	121.0	1	—	1
Ucar 2LM	187.2	1	—	1
Propasol P	149.8	1	—	1
Propasol B	170.2	1	—	1
Aromatic hydrocarbons	—	—	—	—
Aliphatic hydrocarbons	—	—	—	—



The lithium hydroxide column converts esters to metallic salts which are retained in the abstraction column, whilst the alcohols formed may be passed through the analytical column and the eluted peaks recorded. Alternatively, the alcohols may be passed through a boric acid column coupled in series within the abstraction chamber and then retained in the column as trialkyl borates.

The effect of temperature on the efficiency of abstraction of simple ketones is shown in Table 2, where it is apparent that the percentage absorption decreases as the temperature decreases, and that the decrease with the alkyl ketones tends to be greater as the molecular weight of the ketones is increased.

Table 2  
Influence of temperature on abstraction of ketones

Ketone	65°C	80°C	100°C	130°C
Acetone	100.0	82.0	71.0	65.0
Methyl ethyl	100.0	71.0	65.0	46.0
Methyl isobutyl	100.0	75.0	58.0	54.0
Methyl <i>n</i> -amyl	100.0	70.0	50.0	45.0
Methyl cyclohexanone	100.0	56.0	48.0	38.0
Isophorone	100.0	71.0	42.0	40.0
Diacetone alcohol	100.0	64.0	80.0	79.0

Boric acid columns have been reported to operate relatively independently of temperature within the temperature range 75 to 200°C and in this work operation at both 100°C and 130°C has been found to be satisfactory. Of the alcohols encountered in coatings, tertiary butanol appears to be the only material not abstracted, minor although variable partial reaction being experienced.

The removal of esters by the use of columns containing lithium hydroxide and boric acid in series is shown in Table 3 from which it is apparent that temperature has little effect on the reaction.

Table 3  
Influence of temperature on abstraction of esters

Ester	60°C	100°C	130°C
Ethyl formate	50.0	95.0	94.0
Ethyl acetate	100.0	100.0	—
<i>n</i> -Butyl acetate	100.0	100.0	—
Allyl acetate	100.0	100.0	100.0
<i>iso</i> -butyl acetate	100.0	100.0	—
3-Methoxybutyl acetate	100.0	100.0	—
Cellosolve acetate	100.0	100.0	—

The application of the procedure is demonstrated using the solvent mixture shown in Table 4.

Table 4  
Composition of solvent

Solvent	% v/v
Ethanol	10.0
<i>n</i> -Butanol	7.0
Ethyl acetate	6.0
<i>n</i> -Butyl acetate	20.0
Methyl ethyl ketone	5.0
Shell X55	27.0
Toluene	25.0

The chromatograms for the solvent mixture in Table 4 are shown in Fig. 2, the curves being obtained using a 6ft dinonyl phthalate column at 130°C:

- Original sample.
- After boric acid abstraction with removal of peak 3 (ethanol) and peak 8 (*n*-butanol).
- After bisulphite abstraction with removal of peak 7 (methyl ethyl ketone).
- After saponification and boric acid abstraction with removal of peak 3 (ethanol), peak 8 (*n*-butanol), peak 6 (ethyl acetate), and peak 11 (*n*-butyl acetate).

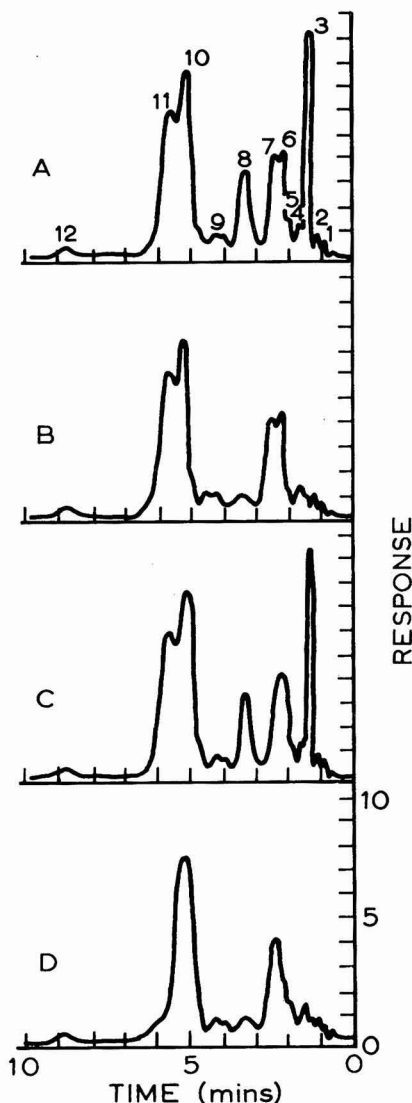


Fig. 2. Gas chromatograms of solvent mixture shown in Table 4, showing effect of abstraction columns

- original solvent
- boric acid abstraction
- bisulphite abstraction
- saponification and boric acid abstraction

A sample of 1 $\mu$ l was used, the attenuation being varied to provide traces with comparable peak heights.

The chromatograms for a mixture with a substantially wider boiling range are shown in Fig. 3. The curves were obtained using a 10 per cent dimethyl silicone OV-1 column with both isothermal and programmed operation:

- Original sample and isothermal operation at 130°C.
- Original sample and programmed operation from 50 to 200°C at 6°C/minute.
- Boric acid abstraction at 130°C and programmed as (b).
- Bisulphite abstraction at 65°C and programmed as (b).
- Saponification and boric acid abstraction at 130°C and programmed as (b).

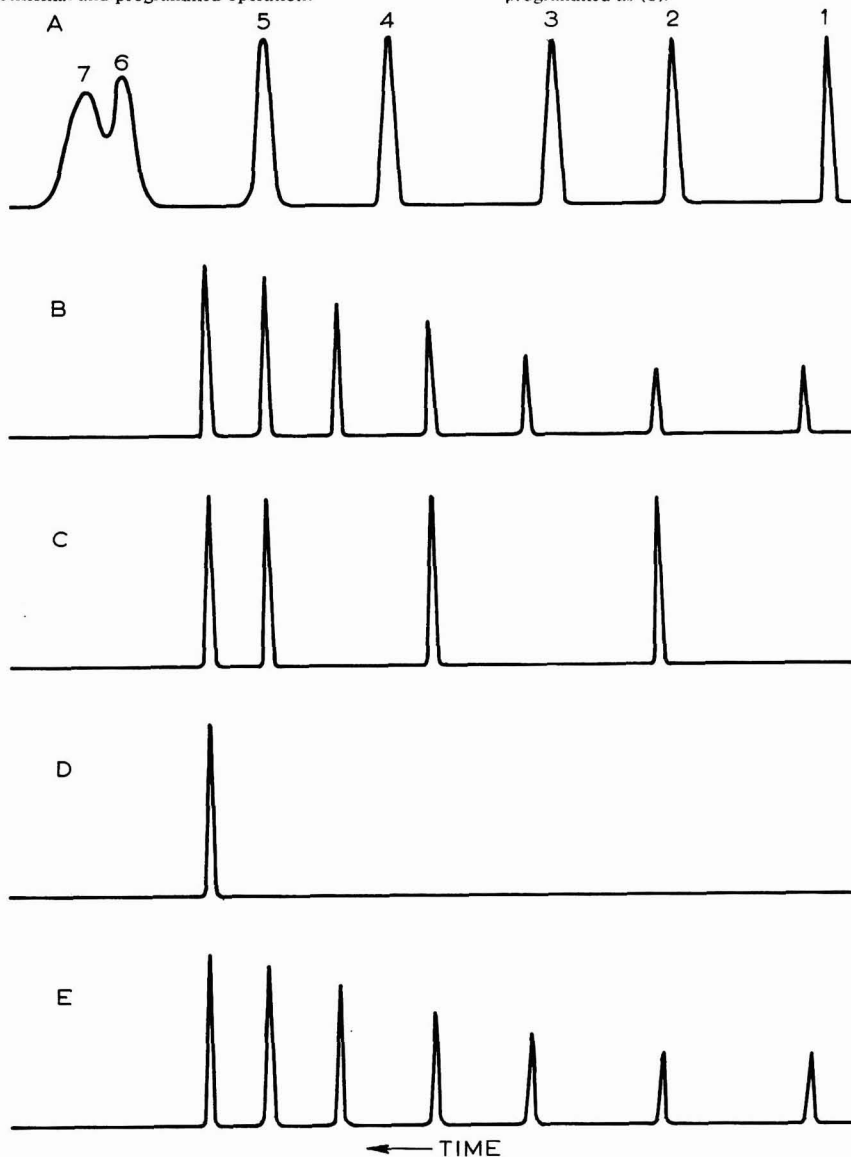


Fig. 3. Gas chromatograms of mixture containing Cellosolve (peak 1), Cellosolve acetate (peak 2), Carbitol (peak 3), Carbitol acetate (peak 4), Tri-Icinol (peak 5), Tri-Icinol (peak 6) and dibutyl Carbitol (peak 7), showing effect of abstraction columns

- isothermal separation of sample
- programmed separation of sample
- boric acid abstraction and programmed separation
- bisulphite abstraction and programmed separation
- saponification and boric acid abstraction and programmed separation

The mixture contained Cellosolve (peak 1), Cellosolve acetate (peak 2), Carbitol (peak 3), Carbitol acetate (peak 4), Tri-Icinol (peak 5), Tri-Icinol acetate (peak 6), and dibutyl Carbitol (peak 7), and was a mixture used previously to produce similar responses under isothermal conditions<sup>28</sup>.

From Fig. 3a and 3b it is apparent that programmed operation enhances the resolution of the high boiling compounds of the solvent mixture, allowing complete resolution of the two components last to emerge from the column, i.e. Tri-Icinol acetate and dibutyl Carbitol. Similarly, if low boiling compounds were present, improved resolution at start of the programme would be achieved due to the 80°C lower initial column temperature. Curve c shows the effect of alcohol abstraction with removal of the ether alcohols, whilst curve e shows the added removal of the esters of these alcohols. The remaining peak is not removed by bisulphite addition (curve d) and is the ether dibutyl Carbitol.

### Acknowledgments

The author is indebted to Union Carbide Australia Ltd., ICI Australia Ltd. and Shell Chemicals (Australia) Pty. Ltd. for supplying samples of their principals' products.

Many of the compounds examined have other trade names<sup>28</sup>. The Union Carbide product names are widely known and, for convenience, these are used where applicable.

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# The parameters affecting the performance of coal tar/epoxy coatings\*

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## Summary

The performance of high build solvent containing coal tar/epoxy coatings has been investigated in relation to several formulation variables, e.g. type of epoxy resin, curing agents, type of liquid extender and quantity of liquid extender.

Intercoat adhesion assessment after seawater immersion of panels prepared with extended time intervals between the first and subsequent coats, and resistance to seawater immersion of panels prepared under ideal recoating conditions, were selected as important factors to be used in the assessment of performance.

## Keywords

*Types and classes of coating*  
coal tar/epoxy coating  
high build coating

## Les paramètres qui exercent une influence sur le comportement des revêtements à base de goudron de houille/résine époxyde

### Résumé

On a étudié le rendement des revêtements "high build" à base de goudron de houille/résine époxyde et de solvant, à l'égard de divers variables de composition, par exemple, le type de la résine époxyde, de l'agent de durcissement, et de l'extender liquide, et aussi la quantité de l'extender.

Choisis comme les facteurs importants dans l'appréciation du rendement, ce sont l'adhérence intercouche après immersion dans l'eau de mer des panneaux où l'on avait interposé une intervalle prolongée entre l'application de la première et des couches suivantes, et également la résistance à immersion dans l'eau de

The results indicate that the best all-round performance is given by aromatic amine adduct cured solid epoxy resins blended with conventional coal tar pitches.

A literature survey has been conducted of previous work related to this topic and that which covers a similar practical assessment of performance variables is discussed in relation to the author's conclusions on the subject.

mer des panneaux confectionnés sous les conditions idéales de rechambrage. Les résultats indiquent que le meilleur rendement global est fourni par un mélange de résines époxydes solides durcies par un adduct d'amine aromatique et de braies de goudron de houille ordinaires.

On a passé en revue la littérature consacrée aux études préalables sur ce sujet, et l'on discute par rapport aux conclusions de l'auteur, celles qui couvrent une semblable appréciation pratique des variables qui exercent une influence sur le rendement.

## Der den Nutzeffekt von Kohlentee/Epoxylacken beeinflussende Parameter

### Zusammenfassung

Das Verhalten von lösungsmittelhaltigen Kohlentee/Epoxylacken wurde unter Vornahme verschiedener Variationen in der Rezeptur untersucht; dazu gehörten u.a. Epoxiharztype, Härter, Art des flüssigen Streckmittels und Anteil des letzteren.

Als wichtige Faktoren in der Beurteilung des Nutzeffektes wurde Zwischenschichthaftung nach Eintauchen der mit immer länger werdenden zeitlichen Abständen zwischen ersten und folgenden Anstrichen hergestellten Prüftafeln, sowie Widerstand gegen Seewasser nach dem Eintauchen von unter für die Anstricher-

neuerung idealen Verhältnissen vorbereiteten Prüftafeln ausgewählt.

Die Ergebnisse wiesen auf, dass feste, mit aromatischem Amin-Addukt gehärtete und mit konventionellen Kohlenteeerpechen verschnittene Epoxyharze allseitig das beste Verhalten zeigen.

Die Literatur über dasselbe Thema wird hinsichtlich sich auf gleiche und ähnliche praktische Beurteilung von Variablen beziehende, vorangegangene Arbeiten unter Bezugnahme auf die vom Autor gezogenen Schlussfolgerungen besprochen.

## Параметры влияющие на эксплуатационные качества каменноугольных, смольных, эпоксидных покрытий

### Резюме

Анализируются рабочие характеристики высокоструктурных растворных каменноугольных смольных эпоксидных покрытий, по отношению к ряду формуляционных переменных, как например: тип эпоксидной смолы, наполнители отверждения, тип жидкого наполнителя и количество жидкого наполнителя.

Оценка межслойного сцепления после погружения в морскую воду панелей приготовленных с продолжительными периодами между первым и последующими покрытиями и устойчивость против погружения в морскую воду панелей приготовленных в идеальных условиях покрытия, принимались как важные

факторы для оценки эксплуатационных качеств.

Результаты показывают что наилучшие рабочие характеристики даются ароматичными, аминовыми, аддитивно высушенными, твердыми, эпоксидными смолами, смешанными с традиционными каменноугольными смолами.

Дается литературный обзор предыдущих работ на эту тему и обсуждаются работы по соответствующей практической оценке эксплуатационных переменных, по отношению к выводам автора по этому вопросу.

\*This paper was submitted as a dissertation in support of the author's successful application for Licentiatehip in the Technology of Surface Coatings. The Professional Grade Committee considers that the format of the paper could well be used as a model on which future applicants could base their dissertations.

## Introduction and literature survey

Coal tar coatings have been used for waterproofing protection for many years and offer cheapness and excellent water and acid resistance. Improvements to the basic coal tar coatings have been achieved by addition of various constituents including epoxy resin/hardener combinations.

Early work on the subject included an assessment of the relative merits of epoxy modified and unmodified coal tar coatings by N. T. Scideler<sup>1</sup> who concluded that the epoxy resin modification offered improved adhesion and corrosion resistance. A consideration of the water permeability characteristics of unmodified and epoxy modified coal tar coatings conducted by J. Eekels and W. A. Spoon<sup>2</sup> in 1957 also reached the same conclusion; that epoxy resin/coal tar combinations showed promise as water resistant coatings.

P. J. Ashby<sup>3</sup> published a paper in 1959 assessing the advantages and possible uses of coal tar/epoxy coatings. The excellent corrosion resistance and ease of application of the high solids coal tar/epoxy coatings has subsequently led to their increasing use as protective systems for ships' hulls. This subject is the primary concern of this paper. Coatings of this type are currently specified by the Ministry of Defence (Navy), e.g. DGS5961 and have now achieved widespread use as protective coatings for ships and other marine structures.

The need has consequently arisen for careful formulation in order to achieve maximum performance with this type of coating. Various papers<sup>1-6, 16</sup> have been published outlining formulations for marine applications and describing the problems encountered with this type of coating system in practice.

The advantage of using the epoxy/coal tar combination is illustrated by S. Guruviah<sup>7</sup> who investigated the relationship between water and oxygen permeability of paint films and the underfilm corrosion on mild steel. Epoxy/coal tar paints are shown to be particularly impermeable to water and oxygen and this is directly related to the rate of underfilm corrosion. In comparison, an epoxy resin/polyamide formulation in the same tests had a high permeability to oxygen and water and consequently a high corrosion rate. Although the same epoxy resin/hardener combination was not stated definitely to have been used in both tests, it is considered that this indicates that the liquid extender (e.g. coal tar) plays an important part in this improvement of corrosion resistance. This is confirmed by Russian technologist S. I. Pimenova<sup>8</sup> who tested tar modified epoxy resin/polyamine combinations in water and fatty acid immersion and found that, whilst the tar modified system gave satisfactory results, the unmodified system failed in four months.

Fillers form an important part of this type of coating and the correct use of fillers can improve the corrosion resistance. Of special interest in this context is aluminium, which has caused a certain amount of controversy. A paper by H. W. Chatfield<sup>9</sup> in 1966 considered the merits of aluminium filler additions to coal tar/epoxy coatings. One year previously, however, a paper by J. C. Kingcombe<sup>10</sup> had discussed the use of aluminium-containing paints and the possibility of a thermite reaction taking place should the coating be heated, for instance by welding. This was stated at that time to be difficult to prove or disprove, but it was a risk that the Ministry of Defence were not prepared to take, and therefore at that time additions of aluminium were not permitted, although it has been found to improve the corrosion resistance properties of the coating.

The current situation regarding the use of aluminium appears to be that it is not permitted by the Ministry of Defence, but finds a certain amount of use in civil applications.

An investigation of the effect of various grades of liquid extender was carried out by A. Adomenas.<sup>11</sup> This investigation was limited to the effect of various grades of tar on the properties of the tar/epoxy system. These tars were produced from coal by different methods and from petroleum sources. Variations of the properties of coatings produced from the different sources of tar were investigated and two vertical retort tars of very high viscosity were noted as especially inferior. The most viscous of these, of minimum distillation point 255°C, gave products which were markedly inferior in salt spray resistance but with satisfactory water resistance.

The same author has also published a paper<sup>12</sup> considering the effect of variation of coal tar content in such systems, but over the range tested found no effect on water and salt spray resistance although hardness, drying time and other chemical resistance tests were affected. An investigation of the effect of coal tar/epoxy ratio on abrasion resistance was carried out by C. H. Klaren.<sup>13</sup> This is the only paper considered here that has taken account of the abrasion resistance of coal tar/epoxy coatings, and it is felt that this test is not of prime importance. Scratch resistance, however, is specified in some Ministry of Defence specifications but the test normally presents no difficulties.

Epoxy/coal tar coatings for marine use are always immersed in seawater after coating and this tends to show up any areas of poor intercoat adhesion and to produce difficulties when it is necessary to recoat the structure. The adhesion of the first coat of a multi-coat system to the substrate is easily controlled by selection of the weather conditions (this type of coating usually being applied in shipyards or dry docks) and by coating immediately after surface pretreatment, but the adhesion of the subsequent coats is dependent on the state of cure and the degree of weathering of the previous coat. In shipyards it is possible that considerable weathering periods can occur between coats with wide temperature variations. A brief study of this factor has been carried out by C. E. Hoey<sup>14</sup> with intervals of 24 hours', seven days' and one month's weathering between coats. This gives only limited information on permissible weathering periods between coats, because this factor will vary with the type of epoxy resin hardener system used and with the weather conditions prevailing.

A survey of the current state of knowledge by P. Brown<sup>15</sup> in 1970 traces the bituminous coating from prehistoric times to the present day and describes work in several countries concerned with the formulation and use of this type of coating.

No conclusions are drawn, but the document offers a useful insight into the uses, formulations and properties of epoxy/pitch coatings and surveys work carried out in Great Britain, Australia and Russia.

A recent paper by F. Blomeyer<sup>17</sup> entitled "Coal tar/polyurethane systems and their field of application" deals with the derivation and compositions of coal tars, bitumens, and asphalts and with the formulation of coal tar compositions based on polyurethane and epoxy/isocyanate combinations.

Whilst epoxy/isocyanate/coal tar coatings are not considered in detail herein, the Blomeyer publication provides a useful insight into the composition of coal tars, and into



the factors affecting weathering stability and general performance of coatings based on coal tars.

The practical work described in this paper studies the influence of composition of solvent containing epoxy/coal tar coatings with particular regard to:

effect of recoating interval and conditions on subsequent intercoat adhesion,  
recoatibility after seawater immersion,  
resistance to corrosion during seawater immersion.

The following variables have been investigated:

the molecular weight of the epoxy resin,  
the hardener type,  
the type of liquid extender used,  
the level of liquid extender used,  
the type of solvent used,  
the type of pretreatment between coats,  
the method of application,  
the addition of laminar pigments.

For convenience, the experimental work detailed below is divided into sections dealing with each of the three aspects shown above.

## Experimental

### Effect of recoating interval and conditions on subsequent intercoat adhesion

*Recoating under controlled temperature/humidity conditions (no weathering between coats)*

#### Test method

A coating of 125 $\mu$  dry film thickness was applied to shot blasted mild steel panels:

the first coat was applied and allowed to cure for 10 days at 10°C and 80-90 per cent relative humidity,

the second coat was applied and allowed to cure for a further 10 days under the same conditions,

the panels were then immersed in synthetic seawater at 20-25°C and the intercoat adhesion assessed at intervals by the cross-hatch method.

#### Formulations

A series of coatings was formulated using a liquid epoxy resin (epoxide equivalent weight 250) (i)\*, in combination with polyamide (ii), polyamide adduct (iii), aliphatic amine adduct (iv), and aromatic amine adduct (v), hardeners. The epoxy resin/hardener mixture was combined with a conventional coal tar pitch at a solids ratio of 1 : 1.

A similar series of coatings was prepared from a solid epoxy resin (epoxide equivalent weight 500) (vi). More detailed typical formulations are quoted in appendix 1.

A third series of coatings was formulated using a liquid epoxy resin cured with a polyamide hardener (a system known to give inferior intercoat adhesion), on its own (system f) and combined in a ratio of 1 : 1 with the following:

\*For details of resins annotated with Roman numerals, and suppliers, see Appendix 2.

- (a) a conventional coal tar pitch (vii);
- (b) a second coal tar pitch (viii);
- (c) a cumarone/indene resin (ix)—25 per cent replacement of the coal tar;
- (d) a fluxing oil (x)—10 per cent replacement of the coal tar;
- (e) tar acids (cresols) (xi)—10 per cent replacement of the coal tar.

## Results

Table 1

*Results of intercoat adhesion of systems based on liquid epoxy resin/coal tar*

Hardener type	Intercoat adhesion after seawater immersion
Polyamide	Failure after 24 hours
Polyamide adduct	Very good after 12 months
Aliphatic amine adduct	Slight failure after 60 days
Aromatic amine adduct	Very good after 12 months

Table 2

*Results of intercoat adhesion on systems based on solid epoxy resin/coal tar*

Hardener type	Intercoat adhesion after seawater immersion
Polyamide	Slight failure after 4 months
Polyamide adduct	Very good after 12 months
Aliphatic amine adduct	Very good after 12 months
Aromatic amine adduct	Very good after 12 months

Table 3

*Result of variation of extender type in a liquid epoxy resin/polyamide system*

System	Intercoat adhesion after seawater immersion
A	Failure after 24 hours
B	Failure after 24 hours
C	Failure after 24 hours
D	Failure after 24 hours
E	Failure after 24 hours
F	Failure after 24 hours

From this it can be concluded that the important factor influencing intercoat adhesion is the hardener type, whilst the epoxy resin and extender are minor influences.

#### Effect of exterior weathering between coats

Two series of tests were conducted to evaluate intercoat adhesion on films which had been weathered between coats, the first at a dry film thickness of 50 $\mu$  per coat, the second at 100-150 $\mu$  per coat.

Whilst the first series of tests at 50 $\mu$  film thickness did illustrate differences between the coatings tested, it was felt that an assessment at 100-150 $\mu$  film thickness on larger test plates with the paint applied by airless spraying would be more meaningful.

**First series—Film thickness 50 $\mu$  per coat: test method 2**

A coating of 50 $\mu$  dry thickness was brush applied to shot blasted mild steel plates.

After curing for 24 hours at 10°C and 80-90 per cent relative humidity the coatings were allowed to weather (45° south facing).

After various time intervals, the panels were washed, allowed to dry for 24 hours and a second coat applied.

This coat was cured for 24 hours at 10°C followed by 6 days at 20°C.

The panels were then immersed in seawater.

Intercoat adhesion was assessed at intervals by the cross-hatch method.

**Formulations**

Epoxy resin + hardener/coal tar (vii) ratio 1 : 1. Both liquid (i) and solid (vi) epoxy resins were evaluated combined with

polyamide (ii), polyamide adduct (iii) and aromatic amine adduct (v) hardeners.

**Second series—Film thickness 100-150 $\mu$  per coat: test method 3**

The following sequence of applications and testing was used.

The film was sprayed on to 250 × 250 × 0.3cm mild steel shot blasted panels by airless spray. (The shot blasting was carried out with G07 angular iron grit.)

The film was cured for 7 days at 10°C and 90 per cent relative humidity, or at 25°C, or outside at 45° upwards south facing, or outside at 45° downwards south facing.

All panels were then exposed to weathering: at 45° south, film facing upwards, or at 45° south, facing downwards.

One panel of each system at each cure schedule, weathered facing upwards and earthwards respectively, was brought inside, washed, allowed to dry for 1 hour and recoated by airless spraying.

The panels were allowed to cure at 10°C and 90 per cent relative humidity for 1 day and followed by 6 days at 20°C.

Finally, they were immersed in seawater and intercoat adhesion assessed at intervals by the cross-hatch method.

**Formulations**

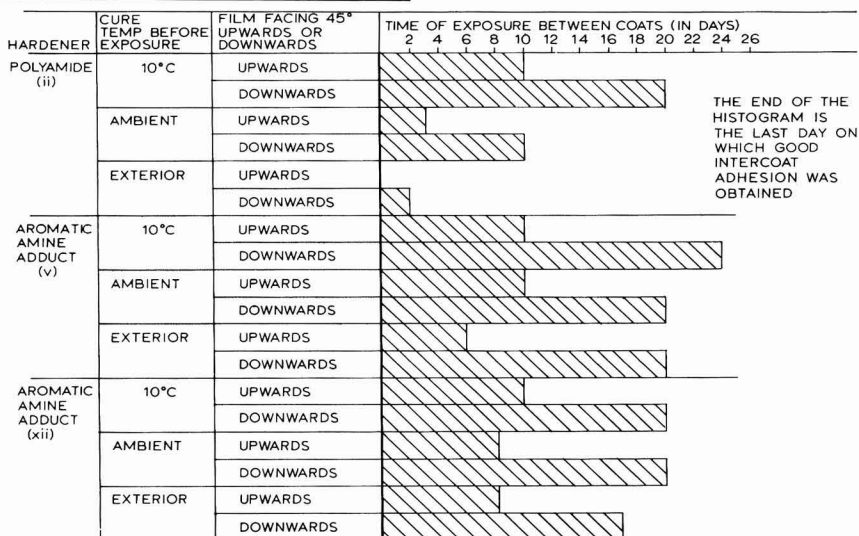
Epoxy resin + hardener/coal tar pitch (vii), ratio 1 : 1. A liquid epoxy resin (i) was combined with a polyamide hardener (ii) or either of two aromatic amine adduct hardeners (v), (xiii).

**Results (See Fig. 1)**

Weather conditions during cure and exposure are given in Tables 5 and 6.

**Table 4**  
**Results**

Epoxy resin type	Hardener type	Period of weathering between coats	Intercoat adhesion after seawater immersion
Liquid (eq. wt. 250)	Polyamide	2 days 5 days	Good after 8 months Failed after 24 hours
Liquid (eq. wt. 250)	Polyamide adduct	2 days 5 days	Good after 8 months Failed after 24 hours
Liquid (eq. wt. 250)	Aromatic amine adduct	2 days 5 days 10 days	Good after 8 months Good after 8 months Good after 8 months
Solid (eq. wt. 500)	Polyamide	2 days 5 days	Good after 8 months Failed after 24 hours
Solid (eq. wt. 500)	Polyamide adduct	2 days 5 days	Good after 8 months Failed after 24 hours
Solid (eq. wt. 500)	Aromatic amine adduct	2 days 5 days 10 days	Good after 8 months Good after 8 months Good after 8 months



**Fig. 1. Intercoat adhesion—effect of weathering between coats**

Table 5

Weather during seven-day curing period for those panels cured outside

Date 1970	Max. temp. °C	Min. temp. °C	R/H at 1200 GMT (%)	Wind direction and speed at 1200 GMT	Rain-fall (mm)
6.2	1.7	-3.3	80	270°; 9 knots	Trace
7.2	7.0	-1.3	86	230°; 18 knots	4.0
8.2	8.1	2.3	70	250°; 22 knots	0.6
9.2	7.0	1.7	68	250°; 18 knots	Trace
10.2	4.9	0.9	77	300°; 16 knots	0.1
11.2	1.5	-4.2	93	270°; 9 knots	Nil
12.2	1.6	-1.7	92	90°; 21 knots	2.2

Table 6

Weather during the possible 27-day exposure period between coats

Date 1970	Max. temp. °C	Min. temp. °C	R/H at 1200 GMT (%)	Wind direction and speed at 1200 GMT	Rain-fall (mm)
13.2	1.8	-5.2	86	330°; 6 knots	0.2
14.2	2.9	-5.1	66	360°; 15 knots	Trace
15.2	2.1	-5.2	81	280°; 8 knots	Nil
16.2	2.0	-3.8	64	310°; 17 knots	0.1
17.2	3.5	-4.9	84	230°; 8 knots	6.8
18.2	2.9	-1.9	94	160°; 6 knots	4.9
19.2	8.5	1.9	83	240°; 5 knots	3.7
20.2	7.7	3.2	61	270°; 30 knots	Trace
21.2	11.3	8.9	88	250°; 17 knots	5.9
22.2	10.3	7.3	78	240°; 12 knots	10.7
23.2	9.2	4.0	58	260°; 18 knots	Trace
24.2	7.5	2.8	71	290°; 19 knots	0.4
25.2	5.3	-0.2	76	330°; 6 knots	0.2
26.2	5.3	0.2	80	20°; 11 knots	1.0
27.2	3.7	-1.8	85	30°; 14 knots	1.6
28.2	4.4	0.5	59	10°; 14 knots	Trace
1.3	7.3	-1.0	74	170°; 15 knots	1.0
2.3	3.9	0.6	60	320°; 16 knots	0.1
3.3	3.6	-3.2	55	310°; 14 knots	Trace
4.3	1.1	-2.9	92	90°; 12 knots	3.0
5.3	1.6	-3.5	93	290°; 18 knots	Trace
6.3	3.9	-4.5	75	290°; 14 knots	Trace
7.3	0.9	-3.4	96	220°; 9 knots	0.9
8.3	4.0	-6.7	70	Calm; 0 knots	Nil
9.3	4.0	-5.8	58	160°; 1 knot	Nil
10.3	5.1	-2.2	70	210°; 7 knots	Nil
11.3	5.5	-1.6	93	200°; 10 knots	9.6

Most precipitation in the period was snowfall and at least one-third of the days were sunny.

From this it may be concluded that the aromatic amine hardeners are less susceptible to weathering between coats than polyamide systems, and that the degree of failure is dependent on the circumstances of cure and weathering as well as the time interval that elapses between coats. The results also indicate that better results are obtained by the use of solid epoxy resins (Tables 1 and 2).

#### Effect of seawater immersion between coats on subsequent intercoat adhesion

##### Test method 4

The dry film thickness per coat was 125 $\mu$ , applied to shot blasted mild steel panels (angular iron shot G07).

The first coat was applied and allowed to cure for 24 hours at 20°C and 60-70 per cent relative humidity.

A second coat was applied and cured for 7 days at 10°C and 85 per cent relative humidity, followed by 7 days at 20°C and 60-70 per cent relative humidity.

The panels were immersed in synthetic seawater at 20°C and after 3 months' and 6 months' immersion were removed. They were then washed with distilled water and allowed to dry for 48 hours. Some panels were lightly sanded.

A third coat was applied and allowed to cure for 7 days at 20°C.

The panels were re-immersed in seawater, and intercoat adhesion tested at intervals by the cross-hatch method.

#### Formulations

In all cases an epoxy resin+hardener/coal tar ratio of 1:1 was used.

Both liquid (i) and solid epoxy resins (vi) were evaluated in combination with aromatic amine adduct (v), polyamide (ii) and polyamide adduct hardeners (iii).

#### Results

Table 7  
Recoating after three months' immersion

Resin type	Hardener type	Surface preparation after intercoat immersion	Intercoat adhesion after 30 months' subsequent immersion
Solid (eq. wt. 500)	Polyamide	None	Good
		Sanded	Good
	Polyamide adduct	None	Good
		Sanded	Good
	Aromatic amine adduct	None	Good
		Sanded	Good
Liquid (eq. wt. 250)	Polyamide	None	Good
		Sanded	Good
	Polyamide adduct	None	Good
		Sanded	Good
	Aromatic amine adduct	None	Fairly good
		Sanded	Good

From this section of the work it can be deduced that:

solid epoxy resins allow more latitude in recoating interval,

there is very little difference between coatings applied by brush or spray.

Table 8

Results of recoating after six months' seawater immersion

(Note that the following tests were carried out only on washed films with no abrasion.)

Resin type	Hardener type	Surface preparation	Intercoat adhesion after 25 months' subsequent immersion
<i>Films brush applied</i>			
Solid (eq. wt. 500)	Polyamide	Washed	Fair
	Polyamide adduct	Washed	Fair
	Aromatic amine adduct	Washed	Fair
Liquid (eq. wt. 250)	Polyamide	Washed	Poor
	Polyamide adduct	Washed	Fair
	Aromatic amine adduct	Washed	Poor
<i>Films spray applied</i>			
Solid	Aromatic amine adduct	Washed	Poor
Liquid	Aromatic amine adduct	Washed	Poor

**Corrosion resistance assessed by immersion in synthetic seawater at 20°C**

A number of variations in composition have been examined with regard to their influence on the corrosion resistance of coal tar/epoxy coatings. The method of test has been kept constant and has been devised to avoid intercoat adhesion failures.

*Test method 5*

Each coat was applied to give 125 $\mu$  dry film thickness, to mild steel shot blasted with angular iron grit G07.

The first coat was applied and cured for 24 hours at 20°C and 60-70 per cent relative humidity.

The second coat was applied and cured for 7 days at 10°C and 80-90 per cent relative humidity followed by 7 days at 20°C.

Panels were immersed in synthetic seawater at 20°C and examined at regular intervals, the blistering being recorded according to the ASTM classification D764. Any underfilm corrosion in these circumstances normally becomes apparent through blistering, but periodically a small section of the film was removed with a knife and the substrate examined.

The work under this heading has been divided into individual sections, each dealing with one specified variable.

*Influence of the ratio of coal tar pitch: epoxy resin/hardener*

For these tests the systems based on solid epoxy resin (vi)

and liquid epoxy resin (i) cured with polyamide (ii) were chosen. To each formulation was added from 0 to 300 parts by weight of coal tar per 100 parts by weight of resin/hardener mixture. The coatings were then tested as specified above.

Table 9

*The effect of varying coal tar content in a solid epoxy resin/polyamide hardener formulation*

Parts of coal tar pitch per 100 resin/hardener (by wt.)	6 months' seawater immersion	18 months' seawater immersion	34 months' seawater immersion
0 (0:1)	Microblisters	Few No. 8 blisters	Few No. 4/6 blisters
50 (0.5:1)	Few No. 6 blisters	Few No. 6 blisters	Few No. 4 blisters
100 (1:1)	Unaffected	Unaffected	Few No. 6 blisters
200 (2:1)	Unaffected	Unaffected	Few No. 6 blisters
300 (3:1)	Unaffected	Unaffected	Trace blistering (2 $\times$ No. 6)

Table 10

*The effect of varying coal tar content in a liquid epoxy resin/polyamide hardener formulation*

Parts of coal tar pitch per 100 resin/hardener (by wt.)	6 months' seawater immersion	18 months' seawater immersion	34 months' seawater immersion
0 (0:1)	Medium No. 4 blisters	Medium No. 4 blisters	Dense No. 4 blisters
50 (0.5:1)	Unaffected	Unaffected	Few No. 2 blisters
100 (1:1)	Trace blistering	Trace blistering	Few No. 2 blisters
200 (2:1)	Unaffected	Unaffected	Few No. 4 blisters
300 (3:1)	Unaffected	Unaffected	Few No. 6 blisters

*Effect of additions of modifying agents in the first coat*

Two basic modifications were tested, the first, an addition of an epoxy silane (xiii), known from previous experience to be helpful in water resistant coatings; the second, the addition of laminar pigments (mica and aluminium). These modifications were made to systems based on solid and liquid epoxy resins in combination with polyamide hardener and coal tar. The epoxy resin/hardener to coal tar ratio was 1 : 1.

Table 11

*Modifications to the system based on solid epoxy resin*

Modification	6 months' immersion in seawater	24 months' immersion in seawater
None	Unaffected	Few No. 6 blisters
3% addition of epoxy functional silane	Unaffected	Unaffected
20% addition of mica	Unaffected	Trace blistering
10% addition of non-leaving aluminium	Unaffected	Trace blistering

Table 12

Modifications to the system based on liquid epoxy resin

Modification	6 months' immersion in seawater	24 months' immersion in seawater
None	Few No. 8 blisters	Few No. 4 blisters
3% addition of epoxy functional silane	Few No. 6 blisters	Few No. 6 blisters
20% addition of mica	Unaffected	Few No. 6 blisters
10% addition of non-leaving aluminium	Unaffected	Few No. 4 blisters

*Effect of solvent composition*

Three solvent blends have been examined in conjunction with a control formulation:

Parts by weight			
Xylene	..	..	4
<i>n</i> -Butanol	..	..	1
Xylene	..	..	1
Ethoxy ethanol	..	..	1
Xylene	..	..	4
MIBK	..	..	1

There was found to be very little difference between coatings based on the solvents after 30 months' immersion in seawater.

*Influence of resin type, hardener type and liquid extender type*

These three parameters were evaluated in one test programme. A number of coal tar pitches and other extenders, such as pale coloured coal tar derivatives and hydrocarbon oils, have been examined in both liquid and solid epoxy systems with a number of different hardeners.

*Materials tested**Hardeners*

- Aromatic amine adduct (v).
- Polyamide (ii).
- Polyamide adduct (iii).
- Solid aliphatic amine adduct (xiv).
- Liquid aliphatic amine adduct (iv).
- Solvent-free aromatic amine adduct (xv).

*Resins*

- Liquid epoxide resin (epoxide eq. wt. 250) (i).
- Solid epoxide resin (epoxide eq. wt. 500) (vi).
- Liquid epoxide resin containing reactive diluent (epoxide eq. wt. 200) (xvii).

*Liquid extenders and pitches*

- (1) Coal tar pitch (vii).
- (2) Cumarone/indene resin (ix).
- (3) Hydrocarbon oil (xvii).
- (4) Hydrocarbon oil (xviii).
- (5) Coal tar pitch (xix).
- (6) Coal tar pitch (viii).
- (7) Bitumen (xx).
- (8) Solvent-free pitch in fluxing oil (xxi).
- (9) Coal tar pitch (xxii).

In each case the coatings were formulated to have an epoxy resin/hardener to liquid extender ratio of 1 : 1.

The various formulations were applied to panels as in test method 5, immersed in seawater and examined periodically.

The results are given in Figs. 2 and 3.

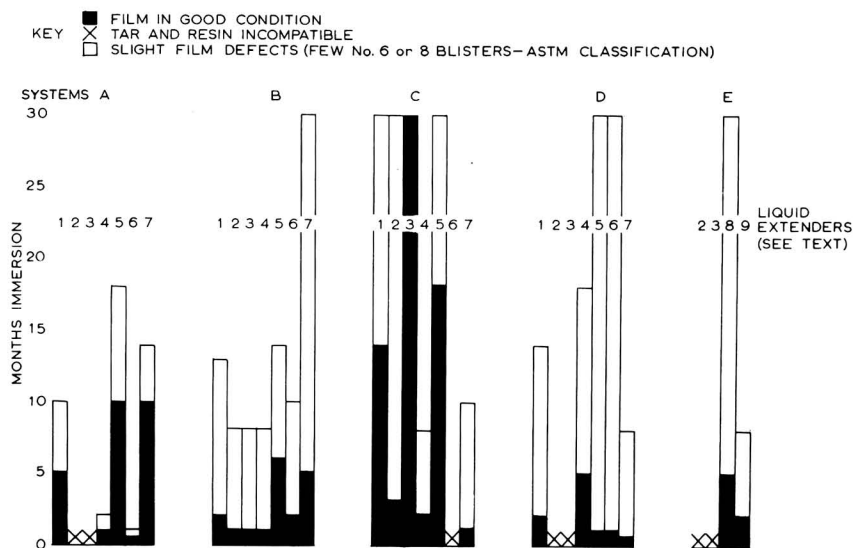


Fig. 2. Performance of epoxy/pitch coatings in seawater immersion



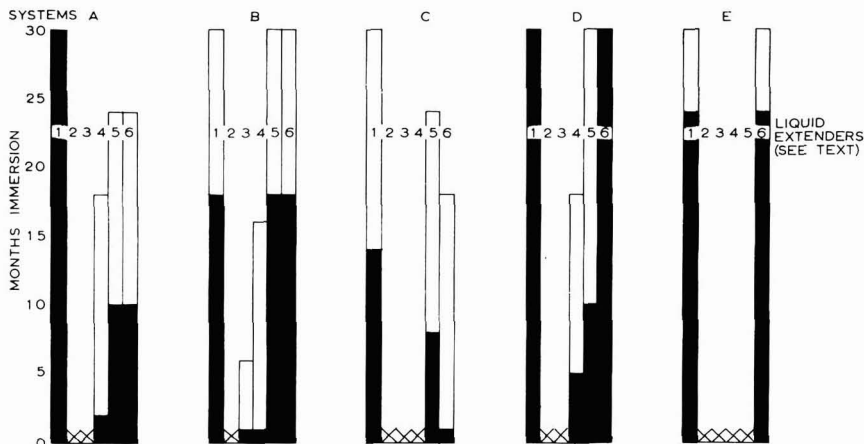


Fig. 3. Performance of epoxy/pitch coatings in seawater immersion

## Conclusions

It is possible to obtain greater latitude in the time interval between coats consistent with obtaining good intercoat adhesion if the correct resin and hardener combinations are selected.

Considering epoxide resins of equivalent weight below 650, the higher equivalent weight resins give superior results. (At epoxide equivalent weights of above 650, cold curing using amine hardeners is impractical.)

Adducts of aliphatic amines and polyamides give greater tolerance to extended recoating intervals than the unmodified products.

Aromatic amine hardeners give greater tolerance to extended recoating intervals than aliphatic amines, polyamides and polyamide adducts.

Coatings allowed to weather between coats, even for very short intervals, show considerably inferior intercoat adhesion compared with similar coatings cured in the laboratory. Recoatability does not appear to be affected by the type of liquid extender. (Omission of the liquid extender from some formulations resulted in no improvement in intercoat adhesion.) The relationship between the hardener type used and the maximum permissible interval between coats indicates that the cause of the early failure is due to the degradation of the surface of the film, perhaps by the uptake of water and carbon dioxide by the amine hardener. In this case, it is logical that the use of hardeners less susceptible to atmospheric conditions during curing, e.g. adducts or aromatic amine hardeners, would give superior results.

Failure taking place after longer weathering periods can be attributed to degradation of the coating surface by normal weathering breakdown (e.g. sunlight and moisture can produce "chalking" of some epoxy resin coatings within two to three weeks).

The results of the work carried out on intercoat adhesion of coatings that had been subjected to seawater immersion between coats show very little difference in performance between the different resin/hardener combinations. The

degree of pretreatment (e.g. washing or abrading) is of some importance, but the method of application, whether by brush or spray, has shown no effect on the subsequent intercoat adhesion results.

Regarding corrosion resistance, the following have been shown.

Conventional coal tars appear in general to give superior seawater immersion resistance in comparison with the other tar derivatives and hydrocarbon oils which have been tested.

The corrosion resistance is considerably reduced if the coal tar/cured epoxy ratio in the binder is reduced to less than 0.5 : 1 (i.e. less coal tar). If the ratio is increased the improvement in corrosion resistance, if any, is not significant and the film will tend to become inferior in other respects (e.g. hardness).

Addition of laminar fillers improves the seawater resistance, especially in the coatings based on liquid epoxy resins. An approximately equivalent effect is obtained by adding either 10 per cent non-leaving aluminium or 20 per cent mica.

Addition of epoxy functional silanes improved the corrosion resistance in the formulations based on solid epoxy resins, but very little effect was noted in the formulations based on liquid epoxy resins.

In combination with most hardeners, a better overall performance is obtained with solid epoxy resins, but a careful choice of liquid extenders is required to achieve the optimum performance. Certain of the liquid extenders tested were incompatible with solid epoxy resin/polyamide adduct combinations, but were compatible with liquid epoxy resin/polyamide adduct combinations. All other examples of incompatibility occurred with both liquid and solid epoxy resins.

Choice of hardeners is important in that some hardeners, e.g. aliphatic amine hardeners, perform best in combination with solid epoxy resin while others, e.g. polyamide adducts, give better performance with liquid epoxy resins.

## Discussion

### Liquid extenders

The choice of liquid extender is one of the most important considerations in the formulation. An extender must be chosen which will give the desired properties with the minimum level of epoxy resin/hardener in order to achieve a competitively priced coating. Certain potential extenders are incompatible with some epoxy resin/hardener systems and, as most are products of coal or petroleum distillation, are not prepared specifically for use in epoxy resin systems.

The report by Adomenas<sup>11</sup> describes the evaluation of a series of liquid extenders prepared from coal and petroleum distillation. The evaluation of 13 of these extenders is given in considerable detail. However, no mention is made of evaluation of extenders other than tars, but an assessment is made of the relative merits of tars prepared by different methods.

The author's conclusion that coal tars give good performance in high build solvent-containing coatings is confirmed by the results quoted in Adomenas's report.

The poor performance of the light coloured liquid extenders, such as hydrocarbon oils and cumarone-indene resins, means that the formulation of light coloured binders that can be pigmented to give coloured coatings is very difficult, and consequently this type of coating is usually brown or black.

The optimum level of liquid extender in the formulation is considered by Adomenas but no effect on salt spray and water resistance is noted. This is probably because the minimum level of extender tested was not below the critical level. This critical minimum level has been found by the author to be between 0.5 : 1 and 1 : 1 liquid extender/cured epoxy resin and, except for flexibility, cure time and hardness of the cured films, there is in theory no limit to the maximum level of extender. The three parameters quoted above place a practical limit of 1.5 to 2:1 on this level. The Russian experience outlined in the paper by Brown<sup>12</sup> indicates that the optimum coal tar/epoxy ratio for fresh water resistance is approximately 1 : 1, which is in agreement with the above-mentioned data.

### Epoxy resins

Adomenas<sup>11</sup> describes three basic types of coal tar/epoxy coatings that can be prepared from amine cured epoxy resins:

Conventional solvent-based coatings prepared from solid epoxy resins of epoxide equivalent weight  $\approx 500$ .

High solids coatings prepared from liquid epoxy resins of epoxide equivalent weight  $\approx 250$ .

Solventless coatings prepared from liquid epoxy resins of epoxide equivalent weight  $\approx 200$ .

Except for a small amount of the work on seawater resistance, the experimental work detailed in this paper is based on the first two types. The conclusion by the author that superior seawater resistance is obtained by the use of solid epoxy resins is only partially confirmed by other work on the topic. Adomenas<sup>12</sup> concludes that the solid epoxy resins give only slightly improved all-round performance.

No work has been carried out to evaluate epoxy resins, other than those derived from diphenylol-propane, in this application.

### Curing agents

The type of hardener used was found to be very significant both in corrosion resistance tests and intercoat adhesion studies. Whilst there is no published work either confirming or disputing the results obtained using aromatic amines, some work has been published<sup>11</sup> which concludes that polyamides give better pot-life, flexibility, impact resistance and intercoat adhesion than amine adducts. It is not disputed that polyamides can give improvements in the first three properties, but the experimental work described in this paper would not confirm that polyamides give superior intercoat adhesion, especially if there is exterior weathering between coats. It is possible that immersion of the films in seawater leads to a more definite breakdown of intercoat adhesion, thus defining the exact differences between the hardeners in this test.

This breakdown of intercoat adhesion after seawater immersion is adequately illustrated by Hoey<sup>14</sup> who quotes results of intercoat adhesion of a controlled system with and without immersion in seawater. This work is based on an aliphatic amine adduct.

The improved recoatability intervals obtained using aromatic amine adducts is considered in more detail in the next section.

### Intercoat adhesion

Usually three coats of coal tar/epoxy paint are applied to a ship's hull (DGS 5961) with an interval of 24-72 hours between coats. If cold weather retards the cure of the initial coats or for any other reason the interval between coats has to be extended, it is necessary to know the effect on intercoat adhesion.

Intercoat adhesion is affected by the length of time between coats, the weather conditions between coatings and the hardener type used. Therefore, the length of exposure between coats consistent with obtaining good intercoat adhesion is largely dependent on the type of hardener used, the weather conditions being usually unpredictable.

Aliphatic amines and polyamides are known to be prone to the formation of deposits on the surface of the film caused by reaction with moisture and atmospheric carbon dioxide.

These deposits will affect intercoat adhesion adversely and can be eliminated only by the use of aromatic amine hardeners which are not prone to the formation of such deposits.

Sanding of the surface has been shown both by the author and by Adomenas<sup>12</sup> to give improved intercoat adhesion. This is probably due to the removal of the degraded surface layer.

From the experimental work outlined it is evident that the direction faced during weathering is very relevant, which would indicate that moisture and sunlight play an important part in this deterioration of the coating surface. Any adhesion failure taking place at longer recoating intervals is attributable to the normal weathering breakdown of the film.

The work by Hoey<sup>14</sup>, although adequately illustrating the need for short intervals, does not compare the effects of different hardeners on permissible recoating intervals or establish the effect of different exterior exposure conditions. However, specific values of adhesion strength are quoted which, when compared with other work, can give a good

indication of the "critical adhesion strength" that is required to give a satisfactory coating. Intercoat adhesion of epoxy/coal tar coatings has been widely criticised, but the high failure rate is largely caused by use under adverse conditions. Other types of coating would also tend to give poor intercoat adhesion under these conditions.

#### Seawater immersion resistance

Resistance to immersion in seawater has been shown to be largely dependent on the type of liquid extender chosen. Subject to correct choice of liquid extender in conjunction with the epoxy resin hardener system, there would appear to be no limit to the length of immersion resistance that can be obtained from a three-coat system. However, incorrect choice of formulation components can lead to very poor results.

#### Acknowledgment

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#### Appendix 1

##### Typical formulations used

	A	B	C	D	E	F	G
Araldite GY280	100	—	100	—	100	—	100
Araldite 6100	—	100	—	100	—	100	—
Special pitch No. 3	180	150	142	121	123	150	148
Araldite DZ093	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Versamid 115	80	50	—	—	—	—	—
Aromatic amine adduct (V)	—	—	55	27.5	—	—	—
HY993	—	—	—	—	23	—	—
EDA870	—	—	—	—	—	50	—
Aromatic amine adduct (XII)	—	—	—	—	—	—	61
Xylene	48	56	36	45	42	78	30
n-Butanol	16	19	12	15	11	—	10
2-Ethoxy ethanol	—	—	—	—	11	50	—

Further solvent as required for brushing or spraying.

Cured resin/pitch	1:1	1:1	1:1	1:1	1:1	1:1	1:1
Percentage solids	85	80	85	80	85	70	85

Fillers (e.g. mica) can be added up to a level of 20-30 per cent of the formulation.

#### Appendix 2

##### Material and suppliers

- Araldite GY 280, CIBA-GEIGY Ltd. (Plastics Division)
- Versamid 115, Cray Valley Products Ltd.
- Versamid 280X, Cray Valley Products Ltd.
- HY 993, CIBA-GEIGY Ltd. (Plastics Division)
- Aromatic amine adduct hardener, CIBA-GEIGY Ltd. (Plastics Division)
- Araldite 6100, CIBA-GEIGY Ltd. (Plastics Division)
- Special pitch No. 3, United Coke & Chemicals Ltd.
- NCB Pitch, National Coal Board
- Dipol L, United Coke & Chemicals Ltd.
- Mobisol 44, Mobil Ltd.
- Tar acids 108, Midland Tar Distillers Ltd.
- Aromatic amine adduct hardener, CIBA-GEIGY Ltd. (Plastics Division)
- Silane A 187, Union Carbide Corp.
- EDA 870, Anchor Chemical Co.
- HY 830/DY 830, CIBA-GEIGY Ltd. (Plastics Division)
- Araldite GY 278, CIBA-GEIGY Ltd. (Plastics Division)
- Epex 100, Hubron Sales Ltd.
- Nevoxy 21, Hubron Sales Ltd.
- LTD 821/XS, Lancs Tar Distillers.
- Bitumen 35, Esso Petroleum Ltd.
- Orgol Tar, United Coke & Chemicals Ltd.
- LTD 821/NS, Lancs Tar Distillers

# Some aspects of drying oils technology\*

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## Summary

Constitutive aspects of drying oils are discussed and, in the assessment of film-forming properties, the importance of considering the effects of impurities in the oils as well as the type and content of polyunsaturated acids is stressed. Simple tests utilising paper chromatography and refractive index measurements can be used to determine the oxidation potential of tall oil fatty acids of different chemical constitution.

A brief review of current theories on the autoxidation of drying oils is followed by a discussion on the chemistry of the volatile products of autoxidation of polyunsaturated oils and the use of

gas liquid chromatography for these studies.

Trends in the modification of unsaturated vegetable oils for surface coating purposes are reviewed and simple molecular models constructed from pipe-cleaners are used to illustrate film-forming properties of various drying oil based media. In efforts to find new outlets for drying and semi-drying oils in the surface coatings industry, some emphasis has been laid on the use of polyunsaturated oils as raw materials for oleochemical intermediates and for the synthesis of novel film-forming polymers.

## Keywords

*Binders, oils etc.*

drying oil  
polyunsaturated oil  
vegetable oil

*Raw materials used in manufacture of ingredients for coatings*

tall oil fatty acids

## Quelques aspects de la technologie des huiles siccatives

### Résumé

On discute les aspects constitutifs des huiles siccatives, et au point de vue de l'appréciation des caractéristiques filmogènes, on souligne l'importance de tenir compte des effets entraînés par les impuretés aux huiles et par le type et la teneur en acides poly-insaturés. On peut se servir des essais faciles, où il s'agit de chromatographie sur papier ou des mesures de l'indice de réfraction, pour déterminer le potentiel d'oxydation des acides gras de tall oil ayant de différentes constitutions chimiques.

Une brève revue des théories actuelles sur l'autoxydation des huiles siccatives est suivie par une discussion de la chimie des produits volatils dégagés lors de l'autoxydation des huiles poly-insaturées, et également de l'emploi de chromatographie gaz-liquide

au cours de ces études.

On passe en revue les tendances dans le domaine de la modification des huiles végétales non saturées pour répondre aux besoins des revêtements superficiels. On utilise de simples modèles moléculaires, construits de nettoies-pipe, pour démontrer les caractéristiques filmogènes de divers milieux à base d'huile siccative.

Au cours des efforts à trouver de nouveaux débouchés pour huiles siccatives ou semi-siccatives dans l'industrie de revêtements superficiels, on a appuyé à quelque degré sur l'emploi des huiles poly-insaturées en tant que matières premières pour des intermédiaires chimiques et pour la synthèse de nouveaux polymères.

## Einige die Technologie Trocknender Öle betreffende Gesichtspunkte

### Zusammenfassung

Mit der Konstitution trocknender Öle zusammenhängende Gesichtspunkte werden besprochen, und im Zusammenhang mit der Beurteilung der filmbildenden Eigenschaften wird die Wichtigkeit die Auswirkung sowohl von Verunreinigungen der Öle als auch der Type und des Gehaltes an polyungesättigten Säuren in Betracht zu ziehen, betont. Zur Bestimmung des Oxidationspotentials von Tallölfettsäuren verschiedener chemischer Konstitution können einfache Versuche unter Benutzung von Papierchromatografie und Refraktivindexmessungen vorgenommen werden.

Einer kurzen Übersicht von v. zt. gültigen Theorien für die Autoxidation trocknender Öle folgt eine Besprechung der Chemie der flüchtigen Autoxidationsprodukte polyungesättigter Öle und

der Anwendung von Gaschromatografie für das Studium derselben.

Die Richtungen, in welchen sich die verschiedenen Modifikationen von ungesättigten pflanzlichen Ölen für Anstrichzwecke bewegen, werden kritisch besprochen und um filmbildende Eigenschaften von auf verschiedenen trocknenden Ölen basierenden Bindemitteln zu beleuchten, werden einfache, molekulare, aus Pfeifenreinigern konstruierte molekulare Modelle benutzt.

In dem Bemühen neue Absatzgebiete für trocknende und halbtrocknende Öle in der Beschichtungsmittelindustrie zu finden, wurde die Verwendung von polyungesättigten Ölen als Rohstoffe für oleochemische Zwischenerzeugnisse und für die Synthese neuer filmbildender Polymerer betont.

## Некоторые аспекты технологии высыхающих масел

### Резюме

Обсуждаются составные соображения высыхающих масел и подчеркивается важность учета, при оценке пленкообразующих свойств, влияния примесей в маслах а также типа и содержания поли-ненасыщенных кислот. Простые испытания, использующие бумажную хроматографию и измерения коэффициента преломления, могут применяться для определения потенциала окисления жирных кислот таллового масла различного химического содержания.

После краткого обозрения текущих теорий авто-окисления высыхающих масел, следует обсуждение химии летучих продуктов авто-окисления поли-ненасыщенных масел и применение газовой жидкостной хроматографии для этих анализов.

Рассматриваются тенденции в модификации ненасыщенных растительных масел для поверхностных покрытий и образцы молекулярных моделей, построенных из проволоки, применяются для иллюстрации пленкообразующих свойств различных сред на основе высыхающих масел.

В попытке найти новые направления для высыхающих и полувсыхающих масел в промышленности поверхностных покрытий, некоторое внимание уделяется вопросу применения полиненасыщенных масел в качестве сырья для химико-масляных промежуточных соединений и для синтеза новых пленкообразующих полимеров.

\* Presented to the Hull Section on 1 November 1971.

## Introduction

Natural drying and semi-drying oils continue to be used in quantity in the surface coating and allied industries, although the demand for these materials in paints, varnishes and printing inks has declined over the past twenty years. In contrast, there has been an increasing trend over this period in the use of synthetic resins and polymers to meet the requirements of faster drying, tougher and more chemically resistant films.

The years following World War II saw the growth of the alkyd resin industry with a trend away from wholly drying-oil based media. Further competition for drying-oil materials came from the expanding field of oil-free water-dispersible paints.

## Constitutive aspects of drying oils

In the past fifteen years there has been much less fundamental research on the constitutive aspects of oils than in the previous decade when, for example, Hilditch and the Liverpool school were actively engaged in studying glyceride and fatty acid composition using low temperature crystallisation and UV spectrophotometric methods. From these investigations, it became apparent that the chemical status of natural drying oils was of importance, rather than their botanical origin. It was shown that, in non-conjugated oils, the total content of polyethenoid acids is an approximate measure of drying power and should not be less than 65-70 per cent.<sup>1</sup> A definite proportion of linolenic acid groups is desirable in a quick drying oil.

New analytical techniques are now widely adopted and the use of gas-liquid chromatography (glc), thin layer chromatography, UV and IR spectroscopy, enables much greater precision in the qualitative and quantitative analysis of drying oils and drying oil-based media.

Table 1 shows the fatty acid composition of some of the drying and semi-drying oils used for paints and printing ink purposes. The analyses were carried out by glc.

Although the "chemical status" of unsaturated glyceride oils has been recognised, it is rarely possible to ensure continuity of supply of oils of constant fatty acid composition. Agricultural factors in seed development, including climate and environment, are responsible for the quite large variations that can occur between different packages of the same botanical species<sup>2, 3</sup>.

Fauve<sup>4</sup> showed that, with linseed oils from which impurities had been removed by careful refining and bleaching, the high iodine value oils (high linolenic content) gave technically different products from those from the low iodine value oils (lower linolenic content). In stand oil and alkyd manufacture, and in the drying and yellowing of white paints based on these oils, there was the expected trend of faster bodying, faster drying and more yellowing of the high iodine value oils.

In work carried out in the author's laboratory several years ago, Canadian linseed oils (high iodine value) were compared with Indian linseed oil (lower iodine value). The importance of considering the effects of impurities in the oils (anti-oxidants, natural colouring agents, phosphatides, etc.) is illustrated by the data in Table 2 and the chromatogram in Fig. 1.

Table 2  
Comparison of Canadian and Indian linseed oils

	Canadian IV = 189			Indian IV = 182.0		
	C	D	AR	C	D	AR
Acid value	3.1	3.8	0.13	1.5	1.5	0.16
Colour Lov. lin cell	29Y 4.8R	59Y 4.6R	8.0Y 0.9R	30Y 2.8R	39Y 3.2R	5.8Y 0.7R
ASTM break, %	0.19	0.06	Nil	0.05	0.01	Nil
Rate of oxidation 116°C (k)	2.9	4.4	4.6	3.3	4.5	4.0

k = polymerisation constant bulk oxidation.

Drying performance (touch dry stage)

Drying conditions		Canadian (hours)			Indian (hours)		
°C Temp.	%Co drier	C	D	AR	C	D	AR
12	0.03	210	216	18	18	18	18
35	0.03	12	12	6	6	6	6
35	0.06	7	6	5	5	5	5
20	0.1	10	10	6	7	6	6

C = Crude. D = Degummed.  
AR = Alkali refined and bleached.

Table 1  
Fatty acid constitution—paint and printing ink oils (typical analyses)

	Soya oil	Sunflower oil	Safflower oil	Linseed Argentine oil	Linseed Canadian oil	Dehydrated castor oil (1½ poise)
Lauric .. ..	0.7	1.1	Trace	0.1	Trace	1.7
Myristic .. ..	Trace	Trace	0.9	Trace	Trace	Trace
Palmitic .. ..	9.3	7.6	7.2	8.6	7.3	1.4
Stearic .. ..	3.7	4.3	3.0	5.3	3.6	1.3
Oleic .. ..	24.5	23.5	12.0	20.3	17.3	4.3
Linoleic .. ..	54.4	63.5	75.8	15.5	14.0	50.5
Linolenic .. ..	7.3	—	1.0	50.2	57.2	—
C 18—cis trans ..	—	—	—	—	—	13.9
cis cis .. ..	—	—	—	—	—	6.2
trans trans ..	—	—	—	—	—	10.7
ricinoleic ..	—	—	—	—	—	10.0

Courtesy of John L. Seaton & Co. Ltd., Hull

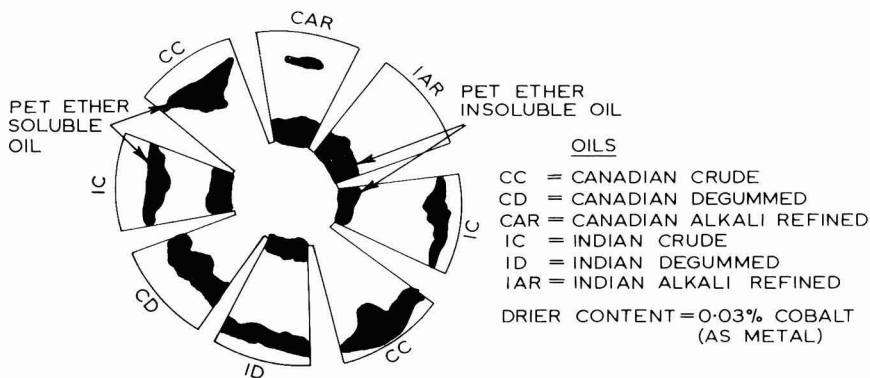


Fig. 1. Influence of refining on oxidation. Canadian and Indian linseed oils

It will be noted that the Indian crude and degummed oils, containing the least impurities according to the ASTM break test, oxidised faster than their Canadian counterparts and exhibited better drying properties; only in the alkali refined and bleached products was the rate of oxidation consistent with the higher iodine value of the Canadian oil.

The type and content of trace non-glyceride impurities has a bearing on technical performance and, in consequence, the degree of refining and bleaching is very important. A very useful contribution by Williams and Cox<sup>5</sup> revealed that natural antioxidants present in soya bean oil were responsible for the inhibited drying of long oil pentaerythritol alkyds. Treatment of a poor drying alkyd by the introduction of metal ions in the presence of an oxidising agent was found to be successful for destroying or inactivating the tocopherols leading to marked improvements in drying properties. Copper naphthenate has been used as the source of metal and an organic peroxide as oxidising agent.

### Tall oil technology

There have been considerable advances in tall oil technology in the past ten years or so and highly refined tall oil fatty acids are now available. These are becoming increasingly attractive as a source of "linoleic rich" fatty acids for alkyd resin manufacture.

Table 3

Fatty acid composition—TOFA American and Scandinavian

Fatty acids	Scandinavian	American
Lower FA .. .. .	0.2	TR
Palmitic .. .. .	0.5	1.0
C <sub>17</sub> branched .. .. .	1.1	0.9
Stearic .. .. .	1.0	3.1
Oleic .. .. .	40.3	55.0
Linoleic .. .. .	48.3	35.7
cis 5, 9, 12— .. .. .	4.4	TR
Higher FA .. .. .	4.2	4.3
<b>Total .. .. .</b>	<b>100.0</b>	<b>100.0</b>
Iodine value .. .. .	155.0	130.0

Courtesy of British Oxygen Chemicals Limited

Table 3 compares the fatty acid constitution of tall oil fatty acids (TOFA) from Scandinavian and American sources. Gas-liquid chromatographic data on the chemical constitution of these fatty acids are shown in Fig. 2.

Tall oil fractionation in the UK has been described<sup>6</sup> and the properties of Scandinavian and American TOFA compared in long oil resins<sup>7</sup>; the drying properties of the Scandinavian TOFA were shown to be superior to the American material. It has been suggested that the good autoxidation properties of *cis*-5,9,12-octadecatrienoic acid present in Scandinavian TOFA contributes greatly to their outstanding drying properties in alkyds<sup>8</sup>.

Recent work in the author's laboratory has been based on measurement of the oxidation potential of unsaturated fatty acid materials. Thin layers of the tall oil fatty acids were oxidised in a temperature-controlled oven under the conditions shown in Table 4.

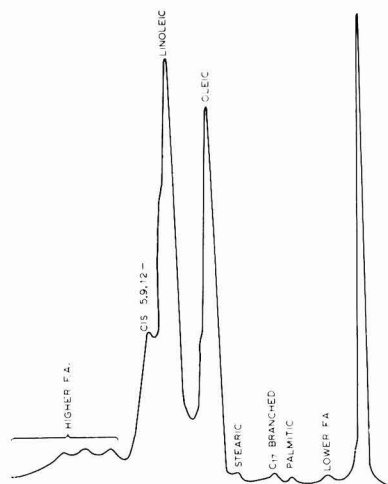


Fig. 2. GLC analysis of methyl esters of TOFA. Scandinavian origin



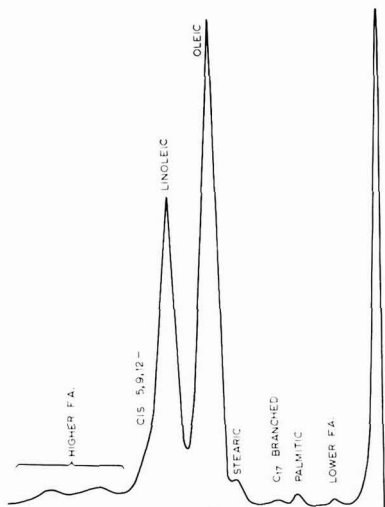


Fig. 2a. American origin

material continues to increase over the five-hour period. The paper chromatograms in Fig. 3 illustrate the comparative rates of development of oxidised fatty acids as determined by petroleum-ether insolubility. Here again the faster oxidation rate of the Scandinavian material is demonstrated. Simple tests of this kind are useful to compare tall oil fatty acids from different sources or of different packages from the same source.

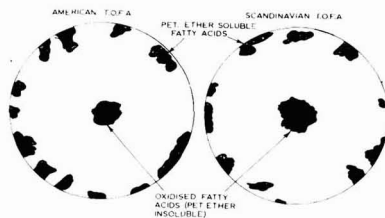


Fig. 3b. Paper chromatograms—oxidation potential of tall oil fatty acids

Table 4  
Oxidation potential of American and Scandinavian TOFA  
Refractive Index  $n_D^{25}$

Hours at 37 C	American	Scandinavian
0	1.4663	1.4703
1	1.4677	1.4712
2½	1.4690	1.4724
3	1.4690	1.4724
4	1.4690	1.4732
5	1.4695	1.4740

5 grams of TOFA exposed in crystallising dish for 5 hours at 37°C.  
Drier content—0.03% cobalt (as metal).

Refractive index increases were used to measure the rate of oxidation. The levelling out of refractive index in the case of American TOFA is noted, whereas that of the Scandinavian

**Autoxidation of drying oils**

Whilst there appears to be general agreement as to the mechanisms operative in the primary stages of oxidation of non-conjugated oils, the reactions of the primary oxidation products leading to polymers and subsequent film formation have not been established with certainty. The oxidation is a free radical chain reaction and the primary product is a hydroperoxide. Polymerisation takes place via the intermediate formation of free radicals from the decomposition of hydroperoxide, the dimers so formed being linked by C-C, C-O-C, or C-O-O-C linkages. Bernstein<sup>9</sup> suggests that the low mechanical strength and poor solvent resistance of freshly formed linseed oil films can be accounted for on the basis that the high molecular weight structure is bonded by secondary rather than by primary valence forces; the association of glyceride molecules could take place by hydrogen bonding between two hydroperoxide groups each on a different molecule, one of these groups being in the free radical form. Poisson and Petit<sup>10</sup> have provided some experimental evidence for formation of polymers in the oxidation of linseed oil films via oxidation of aldehydes formed by scission and arising from the breakdown of oxidised fatty acid chains. The oxidation of the aldehydes in the presence of unsaturated fatty acid chains leads to the

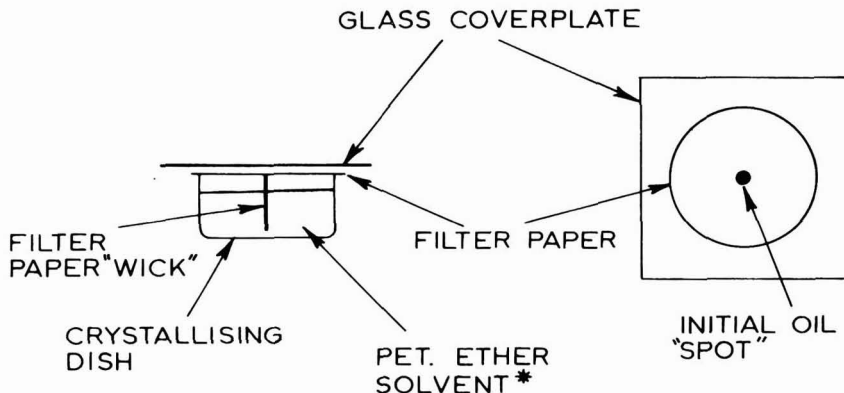


Fig. 3a. Preparation of chromatograms, oxidation of TOFA

formation of polyesters of complex configuration and containing hydroxyl groups. The primary oxidation product of conjugated oils, such as tung oil, is not a hydroperoxide, but some cyclic peroxide would appear to be formed. Most of the polymer formed from the oxidation of conjugated esters appears to be carbon-carbon bonded, the high molecular weight of the polymer suggesting that a chain reaction is operative. Thus evidence from work carried out at the Paint Research Association<sup>11</sup> on the autoxidation of methyl elaeostearate indicates that free radicals are not bridged by peroxide links but that they are mainly C-C bonded, a view in accordance with the general durability of a tung oil film. The polymeric fraction from the autoxidised elaeostearate contains some quite high polymers, of higher molecular weight than those obtained from autoxidation of non-conjugated esters, where the polymer is mainly dimer. The fact that tung oil will dry to an irreversible gel at a much lower oxygen uptake than a linseed oil film (3 per cent as against 14 per cent at the dry point) could be explained by the fact that a true polymerisation in respect of fatty radicals, as distinct from glyceride molecules, is involved. With non-conjugated oils, the main reaction with respect to fatty acid radicals is dimerisation. The data would appear to fit a scheme as shown in Fig. 4.

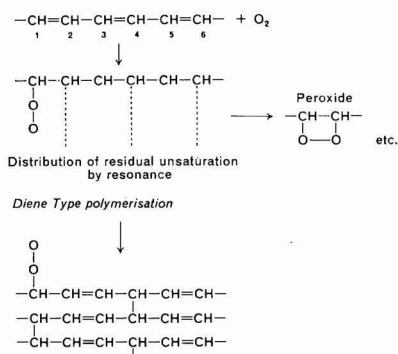


Fig. 4. Mechanism of autoxidation—tung oil

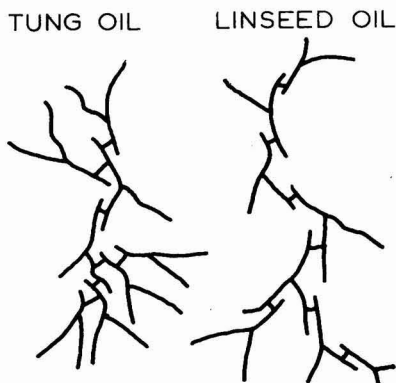


Fig. 4a.

#### Volatiles decomposition products of autoxidation of polyunsaturated oils

Gas-liquid chromatographic studies on the volatile products of degradation arising from autoxidised unsaturated fatty acid esters have yielded useful information on the chemistry of the compounds. From autoxidised linseed oil, propionaldehyde and formic acid have been found as the main volatile products with several minor unidentified peaks<sup>12</sup>. Hexanal was the major volatile compound identified from the low temperature (37°C) autoxidation of sunflower oil whereas deca-2,4-dienal predominated in the larger amounts of volatiles obtained from the thermal decomposition of initially formed hydroperoxides<sup>13</sup>. Mechanisms for the free-radical decomposition of linoleate and oleate hydroperoxides largely explain the formation of identified volatiles (saturated and unsaturated aldehydes, ketones and hydrocarbons)<sup>13</sup>.

Fig. 5 shows gas-liquid chromatograms for the volatile products from the oxidation of a number of drying and semi-drying oils and linseed oil lithographic varnishes.

In these tests cobalt octoate drier at a concentration of 0.1 per cent cobalt (as metal) was added to each oil or varnish and films were cast on to bond paper strips at equal volume and film thickness. Each strip was then placed in a clean 8oz glass bottle sealed with a Suba-seal rubber cap, and the films allowed to dry for a period of 24 hours at room temperature. Using a hypodermic syringe, 5ml samples of air were extracted from each bottle and injected on to the glc column.

Comparison of the linseed lithographic varnishes with alkali-refined linseed oil showed the expected trend of less total volume of oxidation volatiles with increasing viscosity of the varnish (see Fig. 5). Heat polymerisation would consume unsaturation, thereby reducing the number of active centres for subsequent oxidative degradation reactions. Much the same trend was detected in the smelling tests.

Several unidentified peaks were noted on the chromatograms and further work is in hand to attempt identifications of these compounds. For ease of application, the 1,500 poise "strong" varnish was applied as a 50 per cent solution in odourless high boiling aliphatic petroleum hydrocarbon solvent and the sensitivity of the glc instrument adjusted to account for the lower film weight of actual varnish applied to the paper strip.

In odour investigations, an outstanding problem is that it is difficult to correlate glc data with actual smell. For example, a small peak on a chromatogram may contribute much more to the overall odour pattern if the threshold value of the particular compound is low.

#### Utilisation of drying oils in the surface coatings and allied industries

In a discussion on the existing and potential uses for drying oils in the paint, varnish and printing ink industries, it would be appropriate to consider trends in the modification of unsaturated vegetable oils for surface coating purposes.

Attempts to upgrade the film-forming properties of non-conjugated drying oils have involved physical and chemical methods of modification, such as solvent segregation, catalytic isomerisation to conjugated esters, treatment with reactive compounds like styrene, vinyl toluene, cyclopentadiene and maleic anhydride, and replacement of the glycerol component with polyalcohols of higher functionality. For

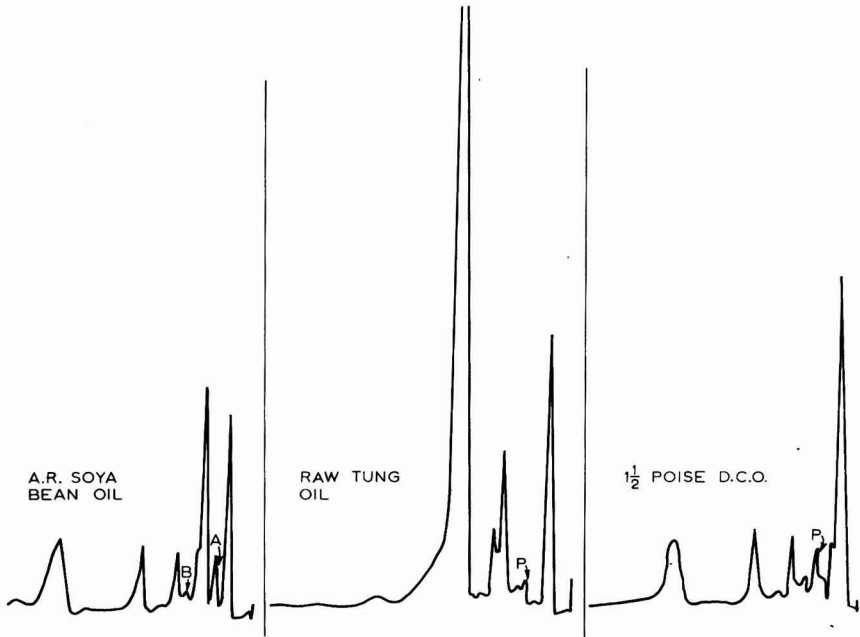


Fig. 5. Odour investigation: autoxidative degradation products  
GLC Column PEG 1000. Temperature 75°C. Detector: flame ionisation

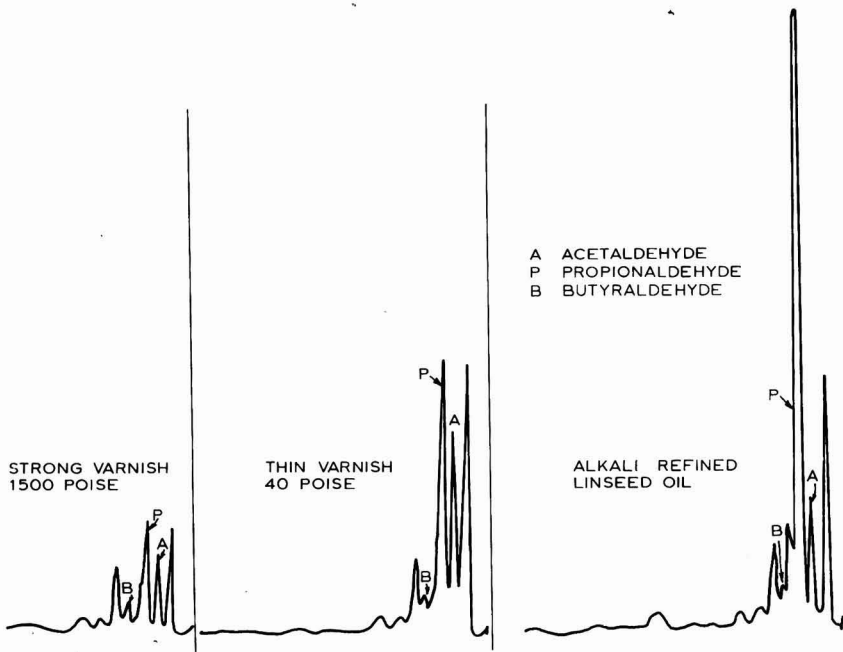


Fig. 5. Antioxidative degradation products

economic reasons, mainly price and availability of oils coupled with the high capital cost of the fractionation plant, solvent-segregation processes have not proved commercially attractive. There has been some interest in isomerised oils, and the tendency appears to be towards the preparation of conjugated materials as a source of oleochemical raw materials which themselves can be utilised for surface coating applications. Thus, treatment of methyl esters of linoleic and linolenic acid with catalytic amounts of alkali metal alkoxide under relatively mild reaction conditions leads to a substantially complete conversion to conjugated isomers<sup>14</sup>. The conjugated acids, polymer, polyamide, and adduct derivatives all find applications in surface coating products<sup>15</sup>. Chemically modified oils themselves, e.g. styrenated oils, have not made a tremendous impact on the paint and printing ink industries, although the underlying chemistry of these materials has been fully exploited in the now well established alkyd resin field. The use of simple molecular models constructed from pipe-cleaners is helpful in gaining an understanding of the film-forming properties of drying oils and drying oil based media. For details on the construction and use of these models,

attention may be drawn to a Student Review<sup>16</sup> in an early issue of the *Journal*. The pipe-cleaner models in Fig. 6 represent three-dimensional molecular structures of air-dried films from different surface coatings materials.

#### Linseed oil film

The "floppy" open structure of the film is to be noted. At the dry point, the film is soft and mechanically weak, and the weak ester linkages are points for attack by acids and alkalis, explaining the lack of chemical resistance in the film. The open structure facilitates diffusion of water and alkalis, etc. into the film leading to eventual deterioration by chemical attack.

#### Linseed oil alkyd resin film

If this model is compared with the linseed oil model it can be seen that the replacement of long linked fatty chains with small compact "phthalic" radicals has made for a tighter packed structure in contrast to the floppy and open structure of the linseed oil macromolecule. The alkyd structure is

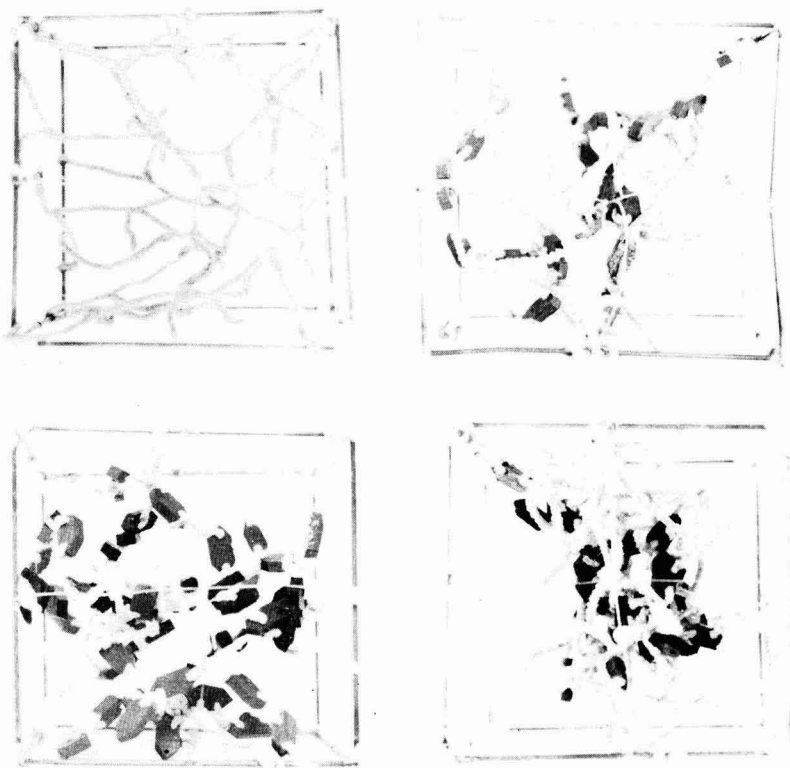


Fig. 6. Three-dimensional molecular structures—pipe-cleaner models

- upper left: freshly dried linseed oil film
- upper right: dried long oil length linseed oil modified glycerol alkyd film
- lower left: dried styrenated alkyd resin film
- lower right: dried urethane oil film



paration of surface coatings polymers or polymer intermediates.

### Unsaturated fatty alcohols

Polyunsaturated fatty alcohols derived from drying and semi-drying oils have been used to prepare esters with film-forming properties superior to those of the oils themselves<sup>26</sup>. Thus, linseed and soya bean oil alcohols have been esterified with certain polyreactive organic anhydrides and acids, e.g. maleic anhydride and trimellitic acid, to yield products with improved air-drying, heat polymerisation and alkali resistance properties. In some work carried out in the author's laboratory, products with useful film-forming properties (hardness and alkali-resistance) were prepared by esterifying the tri-carboxylic acid formed by Diels-Alder addition of fumaric acid to gum rosin, with linseed oil fatty alcohols (see Fig. 7). Unfortunately, the high cost of the alcohols by comparison with fatty acid materials limited commercial exploitation of the products. Polymerised vinyl ethers of linseed or soya bean oil alcohols are potentially useful surface coating products<sup>27</sup>. When the alcohols are reacted with acetylene (atmospheric pressure and basic catalyst) unsaturated vinyl ethers are formed, which may then be polymerised using Lewis acid type catalysts to give unsaturated vinyl ether polymers. Copolymers of the vinyl ethers with lower alkyl vinyl ethers<sup>28</sup> and styrenated derivatives<sup>29</sup> have also been investigated. Cured films from these products are reported to have good hardness, alkali and solvent resistance properties. The performance of the products, however, would not seem to be good enough to compensate for their relatively high cost and they do not appear to have been marketed<sup>8</sup>.

### Polyester-amides from drying oils

The sodium alkoxide catalysed reaction of linseed oil or linseed oil methyl esters with diethanolamine leads to linseed diethanolamides that can be reacted with dibasic acids to yield polyesteramides having interesting surface coating properties<sup>30, 31</sup>. Further research in this field led to the preparation of a new class of urethane derivatives<sup>32</sup>. Excess of the linseed or soya bean diethanolamides is used over the dibasic acid or anhydride to produce polyester-amides terminating in hydroxyl groups, which then react with diisocyanates under triethylene amine catalysis to form urethane polyester-amides. Reaction of the linseed or soya diethanolamides with a di-isocyanate leads to polyurethane amides. Films from the various polymers have a wide range of drying rate, hardness and chemical resistance properties depending on the chemical linkages (ester, amide, urethane) they contain and on the curing condition adopted.

### Products from the reductive ozonolysis of drying oils

Reactive aldehydic products, including aldehyde oils and esters, have been obtained from drying oils by ozonisation followed by reductive decomposition of the ozonolysis products<sup>33</sup>. Reactive aldehyde oils have been found to undergo condensation reactions, e.g. with phenolic compounds, amines and polyalcohols, to give cross-linked polymers. Partial ozonisation of soya bean oil can be controlled to give products containing one or two aldehyde groups per molecule; the dialdehyde oil reacts readily with various diamines to give factice-like cross-linked products<sup>34</sup>.

A continuous pilot plant process for the preparation of the aldehyde ester, methyl azelaaldehyde (MAZ) in the form of its dimethyl acetal (MAZDA) has been described<sup>35</sup>; it employs a sequence of ozonolysis of methyl esters of soya

bean oil, catalytic hydrogenation of ozonolysis products, conversion of resulting aldehydes to acetals and fractionation to isolate MAZDA. Suggested outlets for MAZDA are in plasticisers, polyamides and surface coating polymers. Condensation polymers, namely polyester acetals and polyamide acetals, synthesised from the pentaerythritol acetal of MAZ can be cross-linked at high temperatures to yield adherent, solvent-resistant films having potential uses in protective coatings for glass containers and in glass-reinforced plastics<sup>36</sup>. From a recent communication<sup>37</sup> it would appear that these aldehyde products have not yet reached the commercial production stage, although interest has been developed as shown in a recent patent describing polyaldehyde-polyamine coating compositions<sup>38</sup>.

### Epoxidised oils

Partial epoxidation of linseed oil to an oxirane oxygen content of 1.5 to 3.4 per cent, followed by reaction with phthalic anhydride leads to oil-derived polymers in which the after-yellowing characteristics of the original oil are substantially reduced<sup>39</sup>. Carboxyl-terminated resins of alkyd, acrylic and polyester types have been used to cross-link and cure epoxidised oils from soya bean and linseed oil to form a new class of surface coatings media<sup>40</sup>.

### The future

As long as autoxidative polymerisation is used as a principal mechanism of film-formation, drying and semi-drying oils will continue to find a ready market in the surface coatings and allied industries. No doubt trends in paint and printing ink technology will be influenced by the continuing demand for higher production rates, e.g. faster curing of industrial surface coatings materials, and drying speeds in printing inks to meet the demands of ever increasing speeds of sheet-fed and web-fed presses. Rapid curing by heat or high energy radiation sources is being emphasised and ink systems that cure instantaneously by UV radiation are already available. Such trends might indicate a further decline in the use of drying oils in favour of more reactive monomer and polymer systems derived from petrochemical sources. There seems little doubt, however, that the unique properties of drying and semi-drying oils will continue to be exploited in the preparation of new types of oleochemicals of value to the surface coatings industries and for wider industrial utilisation.

### Acknowledgments

The author wishes to acknowledge the help of his colleague Mr P. L. Watson, who carried out the experimental work on oxidation potential of tall oil fatty acids and the odour investigation. Thanks are due to British Oxygen Chemicals Ltd. and John L. Seaton & Co. Ltd. for supplying analytical data on tall oil fatty acids and drying oils respectively. The paper is published with the permission of the Managing Director of A. B. Fleming & Co. Ltd.

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## Next month's issue

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

**The influence of finish on colour changes in wood** by *S. Dombay*

**A new method for the measurement of ink transfer** by *P. Birkett and P. H. Woodland*

**Rheology of model carbon black ink** by *J. Kollersturm*

**The dynamic mechanical properties of thermosetting acrylic coatings** by *M. Ahoy, S. J. Pargan and E. F. T. White*

## Short Communication

# Chemical study of the alkyd polyamide reaction Part I. Formation of a crystalline reaction product

By J. Jackowska and E. Sunderland

Akt. Alfort & Cronholm, Stockholm, Sweden

### Summary

A crystalline deposit has been found to separate after long reaction of an alkyd resin based on phthalic anhydride with a polyamide. No similar separation appears to occur with alkyds based upon *iso*-phthalic acid. Analysis has shown the separated compound to be diphtalimido-ethane and the yield after long reaction to be sufficient to account for almost all the nitrogen present in the polyamide.

An investigation has been made of thixotropic alkyds in which the reaction time after the addition of polyamide was prolonged beyond the normal.

The base alkyd had an oil length of 70 per cent (tall oil-linseed oil), with a mixture of pentaerythritol and glycerol as polyol and *o*-phthalic anhydride as the only di-basic acid component. The average esterification functionality was 1.920. Two types of polyamide were used separately—Polyamide 930 and Polyamide 943 (Cray Valley Products Ltd.)—and 6.33 per cent on the base alkyd was added in all cases.

The base alkyd was reacted at 260°C to acid values varying from 8 to about 20 before adding the polyamide at 240°C and continuing the reaction at this temperature. The usual effect of a more rapid reaction at higher acid values was noted. Samples were taken at intervals and thinned to 50 per cent solids with white spirit.

In the earlier stages of the reaction with the polyamide, the usual phenomena of clearing (Polyamide 930 only), followed by decreasing thixotropy were noted, and throughout this period the high shear rate viscosity of the products remained constant. After varying periods of time, depending on the type of polyamide and the acid value at addition, the samples lost thixotropy and a slow rise in high shear rate viscosity set in. Some hours after thixotropy was lost, the samples began to deposit a crystalline precipitate, at first after standing some hours, but at later stages of the reaction, immediately upon thinning. The amount of precipitate increased for some hours and then appeared to become constant. In the later stages, crystals appeared in the reaction flask on cooling and did not go into solution again at temperatures up to 260°C. Later work has shown that this lack of solubility (or melting) was due to the upper walls of the reaction flask being well below the temperature in the bulk of the material. The crystalline precipitate was isolated by thinning the alkyd with white spirit, filtering by suction, washing the crystals with heptane and allowing to dry at room temperature. The crystals so obtained were pale buff in colour. Recrystallisation from ethyl acetate or tetrahydrofuran, or better from the two solvents in the order given (two recrystallisations from each) gave colourless shining needles with a very slight greenish yellow cast, MP (uncorrected) = 220°-221°C. The compound proved to be remarkably inert, subliming unchanged and being insoluble in water, alkali, acid and very sparingly soluble or insoluble in most of the common solvents.

The compound contained nitrogen, but neither its infra-red

spectrum nor its general properties corresponded entirely to the simple nitrogen-containing phthalic derivatives phthalimide and phthalimido.

In view of the general interest attaching to the mode of reaction of polyamides with alkyds, and the rarity of well defined crystalline materials in this field, samples of the crystals were submitted both to Cray Valley Products Ltd. and to the Paint Research Association for further study.

The compound was identified independently by both laboratories, as described in Part II, as di-phthalimido-ethane. The identification of the crystalline precipitate made it possible to calculate the amount of polyamide converted. For this purpose, the alkyd was reacted as previously described to the non-thixotropic stage and samples were sealed in thick-walled test tubes, weighed and heated in an oven at 240°C for periods up to six days. Whilst in the oven, all samples remained clear throughout the heating period. Samples removed after three days gave crystals on cooling, even without thinning. Three and six-day samples were thinned with white spirit, the crystals filtered off at room temperature in a sintered glass crucible, washed with heptane and dried to constant weight at 120°C. The yield in both cases was 2.90 per cent, corresponding to a yield, based on polyamide, of 83.5 per cent. It appears, therefore, that the majority of the polyamide is converted to symmetrical diphtalimidoethane and that the reaction is complete in less than 72 hours' reaction time. The earlier evidence suggests a much shorter time for completion, but this has not been confirmed.

Two further experiments were carried out, ancillary to the main investigation. In the first of these a much shorter oil length alkyd (56 per cent oil, esterification functionality 2.05) was used. In this case, gelation intervened at about the time thixotropy disappeared, no crystals having been formed up to this point. In the second experiment, the original base alkyd, but with isophthalic acid replacing phthalic anhydride on an equimolar basis, was investigated. This alkyd finally gelled on cooking, but ample time had elapsed for crystals to have been formed with *o*-phthalic, without any crystals having made their appearance. The possibility of forming a stable imide ring would appear to be a necessary condition for the formation of crystalline derivative.

The following mechanism is proposed.

The first stage is an acidolysis, by residual —COOH groups in the alkyd, of an amide group in a polyamide chain (probably degraded by the long cooking). These residual

carboxyls will be predominantly phthalic carboxyl groups and the next reaction stage is an intramolecular ring formation to give a substituted phthalimide with elimination of an alcohol (complex resin alcohol). The stability of this imide ring will probably ensure its almost complete survival, which then gives a chance for the amide group next in line to undergo phthalic acidolysis in due course. It seems unlikely there would

be any proximity effect across the two methylene groups and the reaction is assumed to be purely random. The freed dimer acid carboxyl group will probably ester-interchange with a phthalic ester carboxyl and the process could clearly go on until all the original diamine residues are converted to the di-imide. Amounts that represent almost complete reaction are, in fact, formed after many hours' cooking.

## Part II. Identification of the crystalline reaction product

By L. A. O'Neill and N. A. Falla

Paint Research Association, Teddington, Middlesex

W. A. Ledger and G. J. Formoy

Cray Valley Products Ltd., St. Mary Cray, Kent

The crystalline reaction product was identified independently in the authors' two laboratories and the combined evidence is summarised below.

### Elemental analysis

The empirical formula was  $C_9H_8NO_2$ . Molecular weights (by vapour pressure osmometry) obtained were from 290 to 310, indicating that the molecular formula was  $C_{18}H_{12}N_2O_4$  (theoretical molecular weight 320).

### Acid hydrolysis

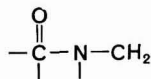
Hydrolysis of the precipitate with excess concentrated hydrochloric acid at  $150^\circ\text{C}$  under pressure for 24 hours yielded *o*-phthalic acid and ethylene diamine.

### Infra-red spectroscopy

The infra-red spectrum resembled that of phthalimide, with a strong carbonyl band at  $\sim 1,710\text{cm}^{-1}$ , but the absence of a NH band in the NH stretch region at  $\sim 3,200\text{cm}^{-1}$  and presence of a strong band at  $880\text{cm}^{-1}$  suggests a N-substituted phthalimide.

### Nuclear magnetic resonance spectroscopy\*

There were peaks at 2.3 and 6.0 tau, with proton ratios 2 : 1. The 2.3 tau peak was assigned to protons on an ortho-disubstituted aromatic ring and the 6.0 tau peak to methylene protons, thus:

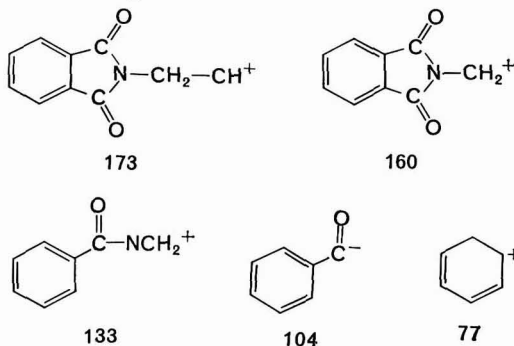


The total number of protons from the molecular formula is 12, and the NMR proton ratios showed that there were eight aromatic to four methylene protons.

\* This was kindly carried out on behalf of Cray Valley Products by Dr D. F. G. Pusey of the Polytechnic of the South Bank.

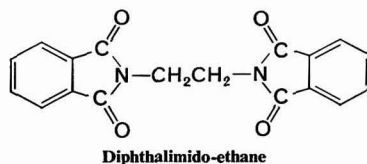
### Mass spectrometry

There were major fragment ions at  $m/e$  173, 160, 133, 104, 77, 76 assigned as:



### Conclusion

The combined evidence thus shows that the structure of the compound is:



The slightly low molecular weight and low melting point (Beilstein, *Handbuch der Org. Chem.*, Vol. II, 1808, gives  $243^\circ\text{C}$ - $244^\circ\text{C}$ ) found may indicate the presence of a small proportion of a compound such as phthalimido-amino-ethane.

[Received 11 October 1972]

## Correspondence

### Change of Journal size

SIR—The announcement that the *Journal* is to change its size to International Standard A4 inevitably brings some regret to those few who, like myself, have known it in much its present format since its birth. I use the phrase "much its present format," because I think I recall that at one time there was a slight enlargement to allow better layout of text, tables, diagrams and advertisements.

When the size of the *Journal* was determined in the very early days of the Association, the requirements of advertisers were quite a minor consideration for, fantastic as it may seem to many Members, the Association was then able to live almost entirely on its subscription income.

A glance at the Income and Expenditure Account for the year ended 1971 shows that, were the Association to be deprived of advertising revenue, it could scarcely afford to publish the *Journal*; it could be a case of A4 size *Journal* or no *Journal*.

Even we old backwoodsmen are content to brush aside nostalgia, resistance to change, these newfangled SI Units in the text and some inconvenience in filing, to keep *JOCCA* in the forefront of scientific publications.

Congratulations to Council and the Publications Committee for their foresight and courage.

Yours faithfully,

N. A. BENNETT

8/3 Gnar il-Lenbi Street,  
Sliema,  
Malta G.C.  
17 October 1972.

SIR—I was disturbed to learn from an editorial in the September issue of *JOCCA* of the proposed change in page size to A4. As I have already indicated in a circular letter to Members of Council, Members of the Publications Committee and certain other Section representatives, I feel this is a bad move for the following reasons:

1. It is good to have *JOCCA* as a completely different size journal so that it is quite distinctive from the piles of A4 size journals that end up on every desk.

2. We have had the *Journal* at this page size since 1918 and the reasons proposed in the editorial are not sufficient to do away with such a long-standing tradition.

3. For individuals who have had their journals bound for some years (either by the official bookbinders or others) it breaks the conformity of a number of years.

There are methods, i.e. film setting, which would have enabled the *Journal* to be printed in its present size but at the same time taking advantage of a wide range of advertising copy.

While I realise that the financial structure of *OCCA* is changing constantly and re-assessments need to be made, I see no reason why Section Committees and Membership at large were not consulted as to their opinion before such changes were finalised.

I realise that Council have acted within the letter of the Articles of Association, i.e. Article 64 which gives them authority to make decisions on behalf of the Membership, but I would suggest that this article does not *prohibit* them from seeking the opinion of Section Committees or Members.

I believe that with a matter as fundamental as this which affects every member of the Association, that they have a duty to widely consult. This matter would seem to call into question the make-up of the Council. I was surprised to find that there are only six directly elected members to this body, the remainder being appointed by one means or another. Numerically, Council appears to be unwieldy to the point that an "inner cabinet," the President's Advisory Committee, has been appointed. It might well be of advantage if some thought was given to making up this Committee by the directly elected members of the Council, so that the President might have the benefit of people who are in close touch with the opinions of the membership at large.

Another thought that might well be considered is to ensure that Members' opinions be directly expressed at the AGM of the Association without the necessity for each member to attend the Conference or AGM. It might be that Sections can arrange to hold their own AGMs prior to the Association's AGM and that they could send a representative to the Association's AGM with a mandate for voting.

There does at the moment appear to be some criticism of the Association being voiced in the industry and I would suggest that consideration of the above points, together with some action along the lines indicated, would be of lasting benefit to the Association at large.

Yours faithfully,

D. C. COLBORN

"Elibank,"  
St. Mary's Hill,  
Ascot, Berkshire.  
26 October 1972.

### The role of research on paint technology in the Australian building industry

SIR—Mr R. R. Hill\* maintains that paint research organisations should be the first ones to be considered for work in paint technology. He is, of course, entitled to his opinion. I have no intention of starting an argument with him. However, I have to comment on some of the statements made by Mr Hill.

(1) If I understand the first paragraph of his letter rightly, he accuses me of the opinion that paint research should be carried out only in Government research organisations. This is definitely not so. All I did was to show up the weaknesses that exist in the various research organisations and the advantages which an establishment like the Division of Building Research has. Of these, the freedom from any pressure from industry or other organisations is the most important one.

Of course, it is clear that the conditions which any institution can provide are only a part of the necessary ingredients

\*R. R. Hill. *JOCCA*, 1972, 55, 1025

for success. The other is the choice of the research worker in charge. I do not want to go into that, except that I should like to mention that someone who is responsible for putting up a research programme in paint technology should have a good knowledge of various scientific fields and, above all, he must have the ability to get acquainted quickly with new ones.

(2) Mr Hill asks why one should build up another technical organisation in which to carry out paint research. I have not asked for this to be done. As a matter of fact, most building research institutions have a small group devoted to paint investigations. However, I have pointed out how much work could be done with very little money in this environment and I have demonstrated this with the work done in the Division of Building Research.

(3) Mr Hill is afraid that work carried out in a building research institute would be too academic. This, of course, could be the case and certainly has happened often enough. However, we have shown that this could very well be otherwise, as all the work carried out by us arose from the complaints of architects, builders and the public.

Yours faithfully,

E. HOFFMANN

*17 Elizabeth Street,  
Mentone,  
Victoria, 3194,  
Australia.  
29 October 1972.*

# Reviews

## Techniques of surface and colloid chemistry and physics

Volume 1. Editors R. J. Good, R. R. Stromberg and R. L. Patrick

New York: Marcel Dekker Inc., 1972.  
pp. ix × 252. Price \$13.75

This is the first volume of a series devoted to the experimental techniques that are available for application in the field of surface and colloid science. These are intended to include not only those which are specifically designed for this area of science but any general technique that might be modified to provide information on surfaces and colloids. Sufficient detail is given to make each technique immediately useful. The fundamentals of the property to be measured are considered together with all the information required for the measurements and relevant computations, including details of available equipment. For example, in the first chapter, on the Langmuir film balance for evaluating intermolecular energies (by N. L. Gershfeld), we are given an account of the derivation of thermodynamic quantities from surface pressure measurements for both single and mixed monolayers. This is followed by a description of the principles and mechanics of the film balance, and provides the mechanical and electronic detail of the sensor attached to the torsion head for pressure measurement, and of the float system, trough etc. The procedure is then discussed, and examples given of computations including full computer programmes. Altogether an admirable account of the film balance technique from which immediate application is possible, and with adequate references for supplementing the information presented.

The remaining four chapters in this volume follow a similar pattern. They refer to the measurement of monolayer permeability (by M. Blank), the techniques of ultrafiltration using semi-permeable membranes (by C. J. van Oss), the formation and properties of bimolecular lipid membranes (by H. Ti Tien and R. E. Howard), and methods used in the detection and measurement of concentration gradients across interfaces (by C. J. van Oss).

For students and academic scientists this series will provide useful information, and the need for such a project has been long felt. However, the majority of *JOCCA* readers would get little help from this particular volume, because of the nature of the topics discussed. But when the editors include wetting, electrophoresis, light scattering etc. the books should make interesting and valuable reading.

G. D. PARFITT

## Introduction to molecular photochemistry

By C. H. J. Wells.

London: Chapman & Hall, 1972.  
pp. xii × 146. Price £1.70

This is a valuable little book in which the author provides an introduction to a subject which most other textbooks treat at a more advanced level. It will therefore be welcomed not only by undergraduates, but also by established chemists who require a working knowledge of the subject. Molecular

photochemistry is assuming increasing importance, not only in organic chemistry in general, but also in its relationship to electronic spectroscopy.

The first three chapters exemplify this relationship, comprising a concise account of the origins of electronic spectra. Chapter 1, introducing the theory of electromagnetic radiation and its absorption, is oversimplified, but chapters 2 and 3 are excellent in their account of simple orbital theory, electronic states and excited states. The treatment of emission is particularly lucid. Although chapter 4 may seem unnecessarily long in its treatment of kinetics, such a study is fundamental to the application of photochemistry to practical problems. The final two chapters give examples of photochemical reactions and are extremely well laid out with examples of a large number of reaction mechanisms, though information on product ratios is scant.

The book is written from a fundamentally theoretical approach at the expense of practical detail. A major criticism is the absence of problems for the student to solve. Such exercises are invaluable to the undergraduate and their omission is unfortunate in a book of this level. There are very few errors in the text, though the dots representing free electrons should have been larger to avoid confusion with stray specks of ink. In more than one place a three-electron hydrogen atom is suggested! As with most textbooks the index is incomplete, but is adequate for a text not intended for reference.

These criticisms apart, the book is very well planned, easy to read, and maintains the high standards of the Chapman & Hall chemistry textbook series.

P. S. NISBET

## Lackrohstoff Tabellen

By Dr E. Karsten  
5th revised edition

Hannover: Curt R. Vincentz Verlag, 1972, pp. 516  
Price 58 Dm.

After an interval of five years, this latest edition of "Surface coatings raw materials tables" has again been extended. It now lists more than 7,800 items, mainly oils, natural and (especially) synthetic resins, but also almost all other raw materials, except pigments and extenders, for the industries with which the reader is concerned. The materials are grouped in 44 main sections, and these again in numerous subsections. Many data are given for each item in column form, e.g. for resins: code of producer, melting point, acid value, colour standard, solubility and supplementary comments, the latter varying in size according to the producer. It must be emphasised that the Tables cover practically all reputable sources of supply in Europe and the USA, and that therefore those of German origin are rather in the minority. The table of contents and the group headings are in four languages, including English. There are also lists of suppliers' addresses (with keys), comparisons of various standards referred to in the tables, and a substantial subject index for trade names and descriptions.

The work is as thorough as before, neatly and clearly arranged. Dr Karsten has again performed a useful job.

M. HESS



## **Manchester Section**

### **Corrosion theory for the paint technologist**

Some 45 Members and guests attended the Manchester Literary and Philosophical Society, George Street, Manchester, on 11 October 1972 to hear Dr J. Wolstenholme of the Department of Metallurgy of Flintshire College of Technology present a paper entitled "Corrosion theory for the paint technologist." Dr Wolstenholme dealt with the fundamental mechanisms of corrosion and showed various

slides of a practical nature to demonstrate the points. He then gave some consideration to ways of inhibiting corrosion and finally speculated on how corrosion processes were modified by the presence of organic surface coatings.

The lecture was followed by a lively discussion period and a hearty support of the vote of thanks was shown in the usual way.

A.McW.

# Information Received

## White Sea & Baltic gain Union Camp agency

As from 1 January, the White Sea & Baltic Company has been appointed sole distributors in the UK for a range of chemicals manufactured in the US by Union Camp Corporation. The product range will include pine oils, dipentene, alpha- and beta-pinene and other terpene chemicals.

## Hardman increases varnish capacity

E. Hardman, Son & Company Limited has recently installed two new stainless steel enclosed reaction vessels to increase media manufacturing capacity. The vessels are equipped for operation under vacuum or inert gas, and azeotropic refluxing can be incorporated when required. Each vessel has a capacity of two tons resin solids, and will enable Hardman to meet increasing demand for varnishes and media.

## General Electric investigates UV curing

Details of a development programme on the ultra-violet curing of coatings were released by General Electric Company of the USA at a recent meeting of the American Chemical Society. Although mainly concerned with the plant and UV source, the GE (USA) work includes the development of a series of solventless formulations for UV curing. The environmental advantages of such a coating, which was found to release less than two per cent of its weight into the atmosphere on curing, were stressed.

## Emulsion captures 54 per cent of the DIY market

A recent independent survey commissioned by Berger Paints shows that emulsion paint sales have grown from a 38 per cent share of the decorative market in 1967 to 54 per cent in 1972—almost twice those of gloss paint. Volume sales of emulsion have also increased, by 40 per cent since 1967, while gloss paint sales have dropped 15 per cent over the same period.

The report also shows Berger's Magicote Vinyl Silk Finish to be brand leader in the silk finish sector, and that Magicote and Brolac are increasing their market share. An interesting point brought out was the growth in specialist outlets for paint—particularly the "paint and wallpaper shop"—an increase in sales volume of 20 per cent being recorded through such outlets in the second quarter of 1972.

## BS 4800 now in operation

The new British Standard "BS 4800 Paint colours for building purposes," which was introduced in early 1972, officially came into operation on 1 January 1973, and the Paintmakers' Association has requested its members to service these colours as from that date. The previous standard range to BS 2660 was withdrawn at the same date, and manufacturers will cease making any general supply of the colours from 31 March.

## Croda buys Wm. Sim

The latest acquisition of the Croda Polymers Group Limited, one of the fastest-growing enterprises in the paint and ink industries in recent years, is that of Wm. Sim & Sons (Paints) Limited, the Edinburgh manufacturer of metal finishing enamels, including the *Tractol* range. The factory at Edinburgh

will continue to operate under the day to day management of Dr W. S. Sim, but its commercial activities will be integrated with those of the Croda group. The *Tractol* brand name will be retained.

## New paint waste disposal plant

A new incinerator plant is now in operation at the Four Ashes (Staffordshire) plant of Effluent Disposal Limited. Based on a Fraser unit designed for burning pitch, the plant is intended for 24 hour operation, handling large tonnages of paint waste. The facility is available to customers throughout the country; waste can be collected by Effluent Disposal's road tankers, or sent to the company's Four Ashes railhead. Volume storage facilities are available, so that every demand can be handled immediately.

## Bush Beach move into natural products

Bush Beach & Segner Bayley Limited is to diversify into the marketing of a wide range of naturally occurring materials, it was recently announced. The first product to be launched will be a high quality kieselguhr for use in paints and printing inks and as a filter aid.

## New Products

### Acrylic thickener for emulsion paints

*Viscalex EP 30* is a new acrylic copolymer emulsion thickener from Allied Colloids Manufacturing Company Limited, intended as a replacement for cellulose derivatives in water-based paints. In particular, it is considered by Allied Colloids to be ideally suitable for high PVC whitening extended systems, in which it is claimed to impart improved wet scrub resistance. Supplied as an emulsion with an active content of 30 per cent, *Viscalex EP 30* is claimed to give excellent flow, application and storage properties, and to resist bacterial attack.

### New instruments from Elcometer

Two instruments for pin-hole and porosity measurements on conductive substrates have been introduced by Elcometer Instruments Limited. The two instruments, one a restyled version of an existing model, are basically similar in design, operating by two leads, one earthed to the substrate and the other consisting of a probe connected to a sponge. The sponge is moistened and passed over the paint surface; moisture from the sponge penetrates any pin holes, completes the circuit to the earth lead and causes an audible warning. The restyled Pin Hole Detector Model 104 operates on a 9V DC supply, and is suitable for coatings up to 300 microns in thickness. The new Portector Model 169 operates on 90V DC, and can be used on coatings up to 500 microns in thickness.

### New black for carbon papers from Degussa

A new carbon black with a blue tone and very low structure has been developed by Degussa. The product, *Printex 200*, is claimed to have extremely low oil requirement and good flow properties, and is mainly recommended for the production of carbon papers.

## Literature

### Kinder Janes pumps

A new six-page leaflet giving details of the full range of equipment for which the company is UK agent has been produced by Kinder Janes Engineers Limited. In particular, the leaflet shows the range of pumps available from Kinder Janes, including those from Sundstrand, J. & S. Pumps, Borneman, Lewa, and Gilkes.

### Hoechst pigment range

Farbwerke Hoechst AG has recently issued "Organic pigments for the paint industry," a thick ring-file containing data sheets on all Hoechst organic pigments for the industry, and including suggested formulations. The publication is being distributed by Hoechst-Cassella Dyestuffs Limited.

### Adhesives Directory 1972/73

The fifth edition of "Adhesives Directory," the well-known handbook produced by A. S. O'Connor & Company Limited, publisher of Resin News, is now available. The book will need little introduction to the adhesives manufacturer and user. Suffice it to say that the directory classifies adhesives by basic materials and end use, and that raw materials, plant and equipment, trade names, a who's who and a section on adhesive tapes are also included. Copies are available from the publisher at £2.00 per copy.

### BS publication on nomenclature in chemistry, crystallography and mineralogy

A recent publication from the British Standards Institution is BS 1000 (54) 1972: Universal Decimal Classification, Second English full edition FID Publication No. 483 UDC 54 "Chemistry, crystallography, mineralogy." The Association was pleased to be of help in the preparation of this work; copies are available from the BSI.

### Hadfields primers and enamels

New literature has been published by Hadfields (Merton) Limited, describing the company's high- and low-bake stoving enamels and zinc-rich primers. Copies are available from Hadfields on request.

### Ceramic fibres in petrochemical production

Morganite Ceramic Fibres Limited has produced a 26-page handbook on its products. Entitled "*Kaowool* in the international petrochemical industries," the booklet has been produced by Morganite on behalf of the world's five *Kaowool* producers; Morganite itself makes *Triton Kaowool*, and the other four manufacturers are The Babcock & Wilcox Company, USA, Isolite Babcock Refractories Company Limited, Japan, Fibras Ceramicas, Puerto Rico, and Morganite Ceramic Fibres SA, Belgium. The basic properties of *Kaowool* are listed, together with many case histories of its use in petrochemicals manufacture; names and addresses of *Kaowool* suppliers throughout the world are appended.

### Take the Allopren line

"Take the *Allopren* line" is the title of a new film by ICI Limited on the use of *Allopren* in road marking paints, showing the advantages of such paints over thermoplastic materials. ICI has also produced

material supporting this promotion, including a colour brochure in English, French and German showing the various uses of *Alloprene*-based paints.

### **Conferences, courses, symposia**

#### **Heterophase and emulsion polymers**

A two-day international symposium under the above title is to be organised by the Society of Chemical Industry on 24-25 January at the Scientific Societies' Lecture Theatre, London W1. There will be four sessions, the principal review lectures being "Some aspects of the emulsion polymerisation of vinyl acetate and related monomers"

by Professor V. T. Stannett of North Carolina State University, "Particle nucleation in polymeric colloids" by Professor R. M. Fitch, University of Connecticut, "Dispersion polymerisation" by Mr K. E. J. Barrett of ICI Limited, "Proliferous polymerisation" by Professor J. W. Breitenbach, of Vienna University. Full details from Dr E. F. T. White, Department of Polymer and Fibre Science, UMIST, Manchester M60 1QD.

#### **Emulsion polymerisation**

A series of five evening lectures on selected practical and theoretical aspects of free-radical emulsion polymerisation will be given at the National College of Rubber Technology on consecutive Thursdays from

1 March 1973. The lectures are aimed at graduate level participants with a specialised interest in emulsion polymerisation. Course fee is £2.50, and further details may be obtained from Dr D. C. Blackley at NCRT, Polytechnic of North London, Holloway, London N7 8DB.

#### **External wall finishes**

A one-day symposium on external wall finishes is to be held jointly by the Road and Building Materials Group of the SCI and the Agrément Board. To be held in London on 15 February 1973, the symposium will be of interest to designers, specifiers, builders and manufacturers. Full details from the Assistant Secretary, SCI, 14 Belgrave Square, London SW1X 8PS.

## Section Proceedings

### Bristol

#### Systems of appraisal

A joint meeting of the Birmingham PVL Club and Bristol Section was held at the Hawthorns Hotel, Bristol, on Friday 27 October. The speakers for the evening were Mr N. R. Felstead and Mr C. Sibley of Berger, Jenson & Nicholson Ltd., who presented their joint lecture to some 40 Members and guests.

Mr Felstead began his lecture by referring to the lecture he had given to the section in November 1969: "Management by objectives in research and development." "Systems of appraisal" was the logical extension to that lecture. The advantages of, and the need for, appraisal were outlined and the necessity of paying fairly for performance was stressed.

All appraisal systems should be coupled to the individual's performance, and thus some definite method of measuring performance was essential. The setting of targets (objectives)

was considered very effective in this aspect. Appraisal interviews often highlighted the need for training and, at this point, Mr Felstead handed over to Mr C. Sibley, who showed a film strip by the Industrial Society, illustrating different appraisal interview techniques.

The importance of development of personnel was stressed, and the need to analyse training requirements illustrated. From an individual's training needs analysis, a section or departmental training plan could be formed, aimed to improve the overall performance of the company.

The lecture was well supported by literature, and the lively discussion that ensued was opened by Mr R. J. Woodbridge.

The vote of thanks, proposed by Mr Gilbert of the Birmingham PVL Club, was warmly supported by all present.

R.F.N.

### Hull

#### Stainless and low alloy steels

The second meeting of the present session was held jointly with the Institution of Chemical Engineers, South Humberside Group, at the Barrow Haven Inn, Lincolnshire, on 6 November. The Section chairman, Mr F. D. Robinson, introduced Mr J. E. Truman of Firth Brown Limited who gave a lecture with the above title.

Mr Truman began by outlining the conditions favouring the atmospheric corrosion of mild steel. The quantitative influence of these conditions, namely high humidity and sulphur dioxide, was illustrated in a series of curves representing the weight loss of mild steel panels as a function of time. Further curves were presented to show that the corrosion rate of mild steel could be reduced to about a quarter in some instances by alloying it with small quantities of certain elements. The effect of these elements was additive rather than synergistic and they conferred protection by providing an unstable but self-healing oxide barrier film. Work undertaken by Mr Truman's company had revealed that the most effective elements were beryllium, bismuth, phosphorus, antimony and chromium, whilst aluminium and manganese were only about one-tenth as effective as chromium.

A tremendously increased performance was obtained from

high alloy steels containing between 12 and 24 per cent of chromium with optional additions of nickel and molybdenum. The mechanism of protection was stated to be the formation of a stable oxide barrier film. The rates of corrosion of one particular cheap stainless steel and of a very good quality stainless steel were quoted as 270 and 1 respectively, relative to 4,000 for a carbon steel.

Referring briefly to painting, the lecturer said that good results had been obtained with both cheap stainless steels and with low alloy steels. Corrosion of unpainted low alloy steels set in a concrete or masonry matrix could cause unsightly iron staining. The present solution appeared to be to avoid direct contact between the steel and the concrete, or alternatively to paint the latter.

Mr Truman concluded his lecture with a short discussion of the behaviour of stainless steels when immersed in acids, alkalis and salt solutions, emphasising the virulent action of the halogen anion.

After a fruitful discussion period a vote of thanks was proposed by Dr G. Eveson who complimented the speaker on his interesting lecture and attractive presentation. The meeting was attended by 16 members, 12 chemical engineers and nine visitors.

J.A.H.

### Irish

#### Institute for Industrial Research and Standards

A very interesting talk was given to the Irish Section on Friday 20 October by Mr D. Carroll of the Institute for Industrial Research and Standards, describing the Institute itself.

Although the origins of the Institute went back more than 35 years, its effective history as a national agency for technological advance dated from a Government Act of 1961. In that year the Government acknowledged the essential role of the Institute, a semi-State body with a Board appointed by the Minister for Industry and Commerce.

Some idea of its growth could be seen from the following figures:

		<i>Government grant</i>	<i>Fees received</i>	<i>Staff</i>
1963	..	£50,000	£2,000	55
1972	..	£948,000	£209,000	418

Today it offered modern equipment and trained staff to help a wide range of industries, and many members present realised that there was in Ireland an organisation which could be of immense benefit to many companies in these days of intense and growing competition.

A vote of thanks to Mr Carroll, and to Mr L. O'Halloran who assisted him, was given by Mr D. Power.

A.R.

## London

### Permeability properties of surface coatings towards ions

The first technical meeting of the 1972-73 session was held on 11 October 1972 at the Polytechnic of the South Bank, London SE1, with Mr D. E. Eddowes in the chair. Dr J. D. Murray of Mats QAD gave a lecture with the above title.

Dr Murray referred to methods of detection of small quantities of ions which were now available and which could be used to detect ionic penetration through surface coating films. There were two theories of the penetrations of ions through films, the capillary penetration theory, in which the film was considered to be traversed by pores which had charged groups attached to their walls, e.g.  $-\text{COOH}$  which could ionise into  $-\text{COO}^-$  and  $\text{H}^+$ . Certain ions, e.g.  $\text{Fe}^{2+}$ , could penetrate under the influence of the charges on the walls. The second theory was that of activation energy in which diffusion of ions was analogous to the transfer of heat.

There had also been confusion over terms on the subject of pores; there was no standard laboratory method for preparation of films free of "wicks," and some authors used single coat films, while others may use three coats to prevent such pinholes having an effect. The speaker considered it was unrealistic to speak of any film as being "non-porous."

In the work described, an ion sensitive electrode (Selectrode) was used as a detector. This was not as sensitive as radio-tracers but was easier in use. The special permeation cell used was described. The polymer films examined were cellulose acetate, a polyamide-cured epoxy system, and a polyvinyl chloride/acetate copolymer. All of these were applied by spinning; two coats were used to overcome any imperfections which would invalidate permeability measurements, and the films were aged at  $40^\circ\text{C}$  to remove any entrained solvent. Film thickness measurements, which were very important, were made using a dial gauge with rounded contacts, which were applied under minimum pressure.

The cellulose acetate film was highly permeable to  $\text{Cl}^-$ , and the same film could be used repeatedly and reproducibly, after leaching, and Fick's law applied. This was an example of little interaction between the polymer and the aqueous solution. With the epoxy/polyamide films the behaviour was entirely different. At a 1/1 weight ratio the permeation was slow, and no  $\text{Cl}^-$  appeared for two days. The rate then increased with time and was still increasing after two weeks, then being similar to that with cellulose acetate. At this ratio there was about 14 per cent excess polyamide above the stoichiometric ratio, which meant that there were free primary amine groups as well as amide groups. The result was a hydrophilic film with polar sites which attract aqueous ions. As this occurred there was also an increasing plasticisation of the films where movement of the polymer segments became easier through diminished intermolecular bonding. This facilitated ionic progress through the film.

With the pvc/pva copolymer films, the  $\text{Cl}^-$  permeation rate decreased with time. At the start it was as fast as with cellulose acetate, but it reduced markedly and eventually ceased. This was a hydrophobic system and offered no attraction to aqueous species. Consequently they formed clusters in the pores and eventually blocked transport completely.

## Manchester

### Symposium: "Paint performance and the microbiological environment"

A total of 187 delegates attended the Manchester Symposium, held at Renold Building at UMIST on 19-20 September.

Following the welcoming address by the section chairman, Mr S. Duckworth, Mr H. R. Touchin took the chair for the

The lecture was followed by a lively discussion, after which a vote of thanks to the speaker was proposed by Dr W. Morgans.

V.T.C.

### European Liaison Lecture: Painting concrete

The European Liaison Lecture was held on 8 November 1972 at the Polytechnic of the South Bank, with Mr D. E. Eddowes in the chair. Mr B. Lindberg of the Scandinavian Paint and Printing Ink Institute gave a lecture with the above title.

Mr Lindberg referred to reasons for painting concrete, which may be simply decorative, or protective against certain environments, e.g. water, which could leach out materials, or attack by gases such as  $\text{SO}_2$  and  $\text{CO}_2$ . The structure of concrete was described, and details of its porous nature were given; the full strength of concrete was not reached until after about ten weeks' curing, during which period it should be kept damp.

In painting concrete, much depended upon the nature of the top layer; the pressure of water caused weaknesses, and the wet surface was alkaline and reacted with  $\text{CO}_2$ . Various methods of preparing concrete surfaces for testing were described and a modified pull-off type of instrument was described for measuring the surface strength. It was found that the surface strength increased on curing, and the effects of different atmospheres were studied. Thus, with  $\text{CO}_2$  the strength was about  $1\text{N mm}^{-2}$ , while with  $\text{N}_2$  it was about  $4\text{N mm}^{-2}$ . The effects of water/cement ratios was studied.

The pull-off test was not completely satisfactory, as the adhesive used penetrated the top layer. A tape peel-off method of test was described; this gave information about loosely held material on the surface. The surface was alkaline owing to the formation of  $\text{Ca}(\text{OH})_2$  in a thin layer, but even after brushing this off there was more alkalinity, pH 10-11, which gradually decreased with time.

The adhesion of five types of paint was examined: two latex paints, a chlorinated rubber, an epoxy, and an alkyd paint. Films about  $80\mu\text{m}$  thick were used, and measurement of adhesion was made after nine days' curing. The age of the concrete (up to three years) had no effect on the adhesion. Provided that the paint was alkali-resistant, good adhesion was obtained only 18 hours after painting. Diluting the paint to increase penetration, or priming with a lacquer, gave no increase in adhesion. An epoxy/urethane paint gave extremely good adhesion.

Mr Lindberg then showed some colour slides of cross-sections of painted concrete blocks, in which the medium had been dyed to show the degree of penetration into the surface. In general, pigmented paints did not penetrate; unpigmented media were then used, to show penetrations of up to 2mm. A vinyl lacquer gave very deep penetration, and a layer-type structure was seen, while other media, e.g. chlorinated rubber, gave very little penetration.

The lecture was followed by a lively discussion, after which a vote of thanks was proposed by Mr P. Whitely.

V.T.C.

opening session: "Microbiology—fundamental aspects." This session consisted of three papers:

"Microbiology" by Professor D. E. Hughes of University College, Cardiff,

"Micro-organisms, the environment and materials" written by Dr H. O. W. Egging of the Biodeterioration Centre,

University of Aston, and presented by Dr Allsop of the same establishment,

"Laboratory techniques employed in the study of surface colonisation of material by micro-organisms" by Mr B. S. Hollingsworth of the Biodeterioration Centre, University of Aston.

Following a pleasant buffet lunch the second session was chaired by Mr T. E. Johnson.

In this session, on "Preservation and stabilisation," six speakers presented seven papers:

"Effects of timber micro-organisms on paint performance" by Mr J. G. Savory of the Building Research Establishment,

"Water repellent preservative finishes" by Mr R. R. Hill of the Timber Research and Development Association,

"Microbiological attacks on paint: (a) anti-mildew coatings, (b) the migration of preservatives in latex paints" by Dr O. Pauli of Farbenfabriken Bayer AG,

"Chemical preservatives for coatings and a realistic procedure for evaluating their effectiveness" by Mr L. A. Wienert and Mr W. W. Vanderstraeten of Buckman Laboratories,

"Some aspects of the prevention of microbiological attacks on emulsion paint systems" by Mr G. Carter of ICI Limited, Organics Division,

"The incidence of microbiological contamination during manufacture of emulsion paints" by Mr W. C. Miller of Nuodex Limited.

The evening was occupied by an informal dinner. Professor Hughes gave a short but witty speech after which the party concentrated on the serious business of socialising. Some slight excitement was engendered by the news that the police were searching the building for a bomb, but this fortunately turned out to be a false alarm.

Starting the second day Dr F. M. Smith chaired the third session, on "Antifouling and corrosion resistant coatings." Four papers were presented:

"Developments in antifouling paints" by Dr J. Lorenz of CIBA-GEIGY Marienberg GmbH,

"Vinyl weed resistant antifoulings: a practical approach to laboratory formulation" by Mr R. D. Mearns of Goodlass Wall & Co. Ltd.,

"Sulphate reducing bacteria, surface coatings and corrosion" by Dr J. P. A. Miller of UMIST,

"Protective coatings and wrappings for buried pipes" by Dr E. S. Pankhurst of the London Research Station, The Gas Council.

Following the lunch break, the fourth and final session: "Architectural and environmental aspects," was chaired by Mr S. Duckworth. The four papers were:

"The occurrence and control of moulds and algae on paints" by Mr P. Whitely of the Building Research Station,

"A practical study of microbiological fouling of exterior emulsion paints" by Mr J. A. Wall of BP Chemicals and Mr E. C. Hill and Mr R. Edwards of University College, Cardiff; the paper was presented by Mr Wall,

"Ecological considerations of biodeterioration of interior surface treatments" by Mr D. J. McGlown, biological consultant to The Walpamur Co. Ltd.,

"Practical aspects of overcoming biological attack in buildings" by Mr G. Old, of The Walpamur Co. Ltd.

The two latter papers were complementary, and were presented together as a team effort.

Mr Duckworth closed the proceedings thanking all those concerned, and commenting on the discussion periods, which had yielded many lively exchanges.

No report of the content of the papers has been given here, since it is hoped that they will be published in *JOCCA* in due course.

A.McW.

## Midlands

### Colour passing, visual or instrumental

The 159th meeting of the Midlands Section was held at the Birmingham Chamber of Commerce on Friday 20 October 1972, with the Section Chairman, Mr A. S. Gay, presiding. A total of 49 members and guests heard a lecture by Mr K. McLaren of ICI entitled "Colour passing, visual or instrumental."

Mr McLaren began with a basic introduction to colour measurement, explaining the use of colour differential equations and the adaptation of plots of  $\delta E$  versus commercial colour match acceptance. It was considered that the Adams-Nickerson equation gave the best fit, being the most uniform, and the ANLAB system of co-ordinates was

discussed. Mr McLaren concentrated his discussion on the problems associated with the development of a system of using the colour difference equations for a satisfactory yes-no industrial shade passing. The aim of instrumental methods was to cut down the number of tinting operations required for a commercial match.

A number of instruments available to the paint industry were discussed in terms of their merits and the advantages being built into the more modern machines. The lecture was very well received by the audience and a vote of thanks was proposed by Mr D. Kimber before the Chairman closed the meeting.

R.J.K.

## Newcastle

### The money game

At the October meeting of the Newcastle Section a paper was presented by Mr S. M. Jennings of Thomas de la Rue, with the title "The money game." This was a very interesting and informative evening and covered some aspects of the design and other requirements of various types of security documents with particular reference to banknotes.

The talk was well illustrated by slides and examples of a selection of banknotes from many parts of the world. These showed the increasing use of paper money as a means of advertising, or promoting national interests such as tourism and national heroes. The discussion covered the type of paper and the requirements of the ink and included some examples of the many special features that are necessary for security printing. It illustrated the very high standard and



quality of engraving work needed in this industry. The special needs of the ink with regard to chemical and physical security of the total documents were also demonstrated.

## South African

### Transvaal Branch

#### Trouble shooting for the paint industry

A meeting of the Transvaal Branch was held at the Sunnyside Park Hotel on 27 September, with Mr P. C. Gelmacher in the chair. Dr Alison Milner, of the South African Paint Research Institute, spoke on "Trouble shooting for the paint industry."

Dr Milner described trouble shooting for the paint industry as a fascinating, non-routine job which required a wide knowledge of chemical and instrumental techniques, together with a lively imagination and a flair for detective work. Besides trouble shooting, laboratory services offered to members of the South African Paint Research Institute included physical and chemical tests to various specifications and analysis of coatings and raw materials. The experience gained from the necessarily wide range of analytical methods used had proved invaluable for the successful solution of many problems met by the trouble-shooting department.

A number of recent problems were described to illustrate the diversity of problems and some of the methods used for their solution. Three such problems are shown below.

Rolls of laminated, printed paper used for food wrappers were suspected of adversely affecting flavour. The complaint passed back through various channels to the manufacturers of the printing ink, the paper coating and the adhesive used for laminating. One of these, as a member of the Institute, handed the problem over for investigation. It was considered that the most probable causes were either substances soluble in the foodstuff or volatile materials which could permeate it, the latter being more likely since the food was solid.

A special technique was used for obtaining gas chromatograms of any volatile matter in the paper. The carrier gas flow was stopped and paper sections placed in a small heated tube between the injection port and the end of the column. About a minute was allowed for complete vaporisation, the gas flow was restored and the recorder switched on. Clear chromatograms were obtained showing the presence of considerable quantities of retained solvent. This was considered to be the probable source of the flavour, but, without comparative samples of printing ink, paper coating and laminating adhesive, it was not possible to determine which step in the paper processing was at fault.

This technique of interrupted gas flow could also be used to allow introduction of dry or wet paint samples for analysis of the solvents present.

Secondly, a complex lacquer, containing eight resinous components made under licence to an overseas formulation, became cloudy on ageing, but could be permanently cleared by filtering. The Institute was asked to show whether the whole or a significant part of any component was lost on the filter. There are no published methods for analysis of several of the resins present and separation of so complex a mixture was considered impracticable. However, the formulation was disclosed and all raw materials were supplied for comparison so that useful information could be obtained from infra-red spectra.

Spectra were recorded of a new clear batch of lacquer, a batch cleared by filtering, the cloudy residue and each individual component. Careful comparison and consideration of the absorption bands contributed by each resin led to the conclusion that the "cloud" contained all the components,

The success of the evening can be judged by the need for the chairman, Mr A. A. Duell, to halt the questions after 45 minutes and propose the vote of thanks.

F.H.

with a slight excess of one, but that the amount so removed from the system was not significant.

In the third case, very small scrapings of dried emulsion paint were submitted for identification of binder, since adulteration or substitution was suspected. The obvious means of recognition was infra-red spectroscopy, but preliminary separation of pigment was necessary since emulsion paints contained extender pigments which obscured the "fingerprint" region of the spectrum. For this separation, a semi-micro analytical technique, the use of a filter stick, was adopted. The flakes were soaked in two or three drops of solvent and the solution sucked into a tube through a firm wad of cotton wool which held back the pigment. The solution was evaporated on a potassium bromide disc leaving a clear film of resin for spectroscopy. The resulting spectra clearly showed one sample to be acrylic, as specified, one to be polyvinyl acetate, and one a mixture of the two, justifying the sender's suspicions.

During a lively question time, Dr Milner said that it was not possible, with these fragmented samples, to determine whether the above mixture resulted from the presence of two different coats or from stirring together two different emulsions. To further questions she replied that the great majority of defects in coatings could be traced to poor application and preparation rather than to inferior materials.

Finally, Mr E. A. Daligal proposed a vote of thanks, which was carried with acclamation.

P.A.J.G.

#### The CIE system of colour specification

A meeting of the Transvaal Branch was held on 7 September at the premises of Tæuber & Corssen (Pty) Ltd. Mr P. C. Gelmacher, in the chair, introduced Mr G. Chamberlin of The Tintometer Ltd., who described the working of the Commission Internationale de l'Éclairage, and the agreed system for the measurement and numerical specification of colour that it had produced. The actual instrumentation was not defined by CIE, but whatever approach was used must be capable of expressing results in an agreed mathematical form. The system defined: conditions of observation and exact form of illumination; mathematical treatment of results; and tabulated statement of the relationship of the visual response of an agreed "standard observer" to objective measurements made at each wavelength in the visible spectrum.

Mr Chamberlin stressed that the system relied on the invariability of response of a "normal observer" to a given light stimulus; the published data were the result of repeated observations on real people who had been checked for freedom from colour vision abnormalities.

He went on to outline the three standard illuminants: a tungsten lamp, sunlight, and diffuse daylight with the observer's back to the sun (i.e. south light in the southern hemisphere), and to show their precise characteristics.

The CIE defined conditions of observation—angle of illumination and viewing, size of viewing field, background—were next considered, and the speaker continued with a treatment of the way the human eye responds to light stimulation, using the Young-Helmholtz and Maxwell theories. Careful observation on a number of real observers

had produced tables defining what mixture of the three primaries sufficed to produce a visual match to each wavelength in the visible spectrum. However, as the eye was more sensitive to some wavelengths than to others, the figures were weighted from the  $V\lambda$  curve, which showed the relative sensitivity of the eye.

The adoption of the three "unreal" primaries (X, Y and Z) were defined in terms of mixtures of the red, green and blue lights and of greater saturation than could be achieved physically, was detailed, and the way in which negative

values were thus avoided shown, followed by a description of the distributive functions  $x$ ,  $y$  and  $z$ .

Mr Chamberlin closed by demonstrating the derivation and use of the trichromatic co-ordinates in practice, using the simple visual Tintometer colorimeters, employing nomograms and the Lovibond colour scale.

Mr P. A. J. Gate proposed the vote of thanks for a well-informed and interesting lecture.

P.A.J.G.

## West Riding

### Micronised iron oxide pigments

The first meeting of this session of the West Riding Section was held on Tuesday 12 September at the Griffin Hotel, Leeds. The speaker was Mr W. Schleusser of Bayer AG, who presented a paper dealing with various aspects of micronised iron oxide pigments.

He first described the micronisation, or jet milling, process in which a jet of steam at about 200°C caused the pigment particle to move in a grinding sphere and become ground by collision with each other rather than by contact with the walls of the mill. This resulted in a pigment which was less agglomerated than a non-micronised pigment. It was found that there was little difference in specific surface or particle size distribution (as determined by electron micrography) between micronised and normal iron oxide pigments. Since the grinding of paint aimed to reduce agglomeration the use of jet milled pigment obviously rendered dispersion easier. If dispersion is carried out with high shear forces (e.g. triple-roll mill) the difference between the two types of pigment is less pronounced.

Extensive research on primers pigmented with iron oxide pigments had shown that the degree of corrosion was related to the amount of water penetrating the paint film. Water diffusion was proportional to the binder content of the film if good wetting of the pigment occurred, provided the pigment binder ratio was below the critical pigment volume concentration.

Mr Schleusser then described some work comparing the salt spray resistance of paints containing either micronised or non-micronised iron oxides prepared by different dispersion techniques.

It was sometimes supposed that micronised natural iron oxide produced a more pronounced anticorrosive effect than

synthetic material owing, it was postulated, to the wide range of particle size giving better packing of pigment particles. By mixing synthetic iron oxides of various shades it was possible to obtain pigments with a wide range of particle distribution for comparison with natural material. The mixtures did not appear to have any superiority.

A series of micronised synthetic iron oxides of varying particle size was examined using the same binder system and salt spray time. This showed that the finest pigments gave the worst anticorrosive effect and the coarsest the best.

Mr Schleusser discussed these results and concluded that anticorrosive behaviour was a question of the distance between pigment particles in the dry film rather than critical PVC. The anticorrosive effect diminished rapidly as the distance between pigment particles fell. Natural pigment contained a high proportion of large diameter particles and a different effect was observed between large primary particles and agglomerates. The larger the primary pigment particle the lower the water diffusion through the paint film and the lower the corrosion.

During question time it was suggested that an iron oxide pigment of larger particle size than currently available would be desirable for anticorrosive paints. In reply Mr Schleusser said that, in practice, wetting was very difficult and sedimentation was a major problem. Furthermore, very large particles would protrude from a paint film and water could penetrate it. Such pigments also had very poor tinting strength.

Questioned about the relative tinting strengths of normal and jet milled pigments, Mr Schleusser replied that generally micronised pigments had 5-10 per cent higher tinting strength.

Mr D. Morris proposed a vote of thanks for a most interesting and well-presented paper.

R.A.C.

# OCCA Biennial Conference

## towards 2000

### eastbourne 19-23 june 1973

The final programme for the Association's biennial conference to be held at the Grand Hotel, Eastbourne, from 19-23 June 1973, has now been arranged, and is shown below.

#### Wednesday 20 June

##### Session 1 (09.30-12.30)

Chairman: Mr A. R. H. Tawn (Hon. Research & Development Officer)

Plenary Lecture: "Polymers—Developments for the future" by Professor C. E. H. Bawn (University of Liverpool)

"The influence of supermolecular structure on the properties of polymer films" by Professor W. Furke and Dr V. Zorll (Forschungsinstitut für Pigmente und Lacke eV)

"The hardening of organic coatings with infra-red lasers" by Dr E. Ladstädter and Dr H. D. Hanus (Vianova Kunstharz AG)

##### Session 2 (14.30-16.30)

Chairman: D. E. Eddowes (Chairman, London Section)

"The American Paint Industry—present trends and future prospects" by Mr F. Armitage (Sherwin Williams Co. Inc.)

"Britain and the EEC—technico-commercial aspects." Panel discussion

"Relations with the developing countries" by Mr A. T. S. Rudram (Donald Macpherson & Co. Ltd.)

#### Thursday 21 June

##### Session 3 (09.30-12.30)

Chairman: Dr J. B. Ley (Coates Brothers Ltd.)

Plenary Lecture: "The challenge of metal finishing" by Mr G. Isserlis (Polytechnic of the South Bank)

"The role of coatings in corrosion prevention—future trends" by Dr R. P. M. Procter and Dr V. Ashworth (UMIST)

"Electrochemical activity of conductive extenders for zinc rich coatings" by Dr V. P. Simpson and Mr F. A. Simko Jr. (Hooker Research Center, Niagara Falls)

"The chloride ion in epoxy/polyamide films and the substrate effect" by Dr J. D. Murray (Materials Quality Assurance Directorate, Woolwich)

##### Session 4: Techno-economic aspects (14.30-16.30)

Chairman: Dr F. M. Smith (CIBA-GEIGY UK Ltd.)

Plenary Lecture: "Environmental problems of the chemical industry" by Dr H. Gysin (CIBA-GEIGY, Basle)

"Tomorrow's packaging" by Professor G. Scott (University of Aston-in-Birmingham)

"The future for colour measurement" by Mr K. McLaren (ICI Ltd.)

#### Friday 22 June

##### Session 5 (09.30-12.30)

Chairman: Dr S. H. Bell

"100 years of standards" by Mr C. Meredith (BSI)

"The characteristics of mechanical properties of paint films" by Mr R. L. J. Morris (Materials Quality Assurance Directorate, Woolwich)

"Finish first—fabricate later. The continuing story of coil coating" by Mr D. S. Newton (British Steel Corporation)

"A consideration of powder coating problems" by Mr S. T. Harris (Arthur Holden & Sons Ltd.)

##### Workshop Sessions (14.30)

Sales forecasting

The changing influence of "do-it-yourself" on the retail paint market

The measurement of research output

#### Registration forms

Full details of registration fees, and a registration form, will be sent to all Members of the Association at the turn of the year. Non-members wishing to receive these items, when available, should apply to the Director & Secretary at the Association's offices.

## Conference authors

### Summaries and biographies

Summaries of all the papers were given in the December issue; photographs and short biographies of the authors appear below.



C. E. H. Bawn

Professor Bawn, a graduate of the University of Bristol, was a reader in chemistry at that university at the time of his appointment to the Grant-Brunner chair of inorganic and physical chemistry\* at Liverpool in 1948. During the war he had been in charge of a branch of the Armament Research Department set up in the

Chemistry Department at Bristol, and towards the end of 1943 he was asked to organise the new physico-chemical laboratories at the ARD, Fort Halstead, Kent. During this period he also acted as secretary to the Propellants Research Committee and the Pyrotechnics Committee of the Ministry of Supply. Soon after the war his book on "The chemistry of high polymers" was published. He was elected to the Fellowship of the Royal Society in 1952 and made a Commander of the Order of the British Empire in 1956. His appointments have included the Presidency of the Faraday Society, service as a Vice-President of the Chemical Society and President (1967-68) of the chemistry section, British Association for the Advancement of Science. He also served as Chairman of the Joint Services Non-metallic Materials Research Board (and sub-committees), of the Ministry of Technology. He is a member of the University Grants Committee and chairman of several of its sub-committees. He is also chairman of the Science Research Council's polymer committee. His main research interests are in the synthesis and study of properties of high polymers, and the oxidation and reaction of hydrocarbons. He is an honorary DSc of the Universities of Bradford and Birmingham.

\* Now separate chairs of inorganic and physical chemistry, of which Professor Bawn holds the Brunner chair of physical chemistry.



G. Isserlis

Graduating from London University, Mr Isserlis entered industry in 1943, and after eight years' specialising in metal finishing he switched to teaching, joining the staff of the applied chemistry department of the Northampton Polytechnic, London (now the City University).

In 1957 he transferred to the Kingston College of Technology as lecturer-in-charge of the metallurgy section, and in 1960 to the then Borough Polytechnic (now Polytechnic of the South Bank) in his present position as head of the division of metal science. There he developed courses in metal finishing and corrosion science, as well as in materials science.

In 1965 he became more deeply involved in the affairs of the Institute of Metal Finishing, when he was elected chairman of the education committee. He became president of the Institute during 1969-71, and is a member of the council, of the membership committee and of the publications committee. He is also a member of the education and membership committees of the Institution of Corrosion Technology.



**H. Gysin**

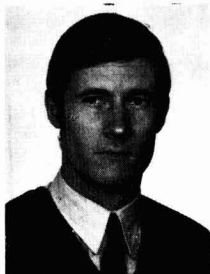
After completion of the chemical studies at the University of Basel, Dr Gysin joined the former J. R. Geigy Ltd. in 1938 as a research chemist in the pharmaceutical department. From 1947 on he became primarily concerned with the synthesis of insecticides, being responsible for the chemistry department of the Agchem Division in 1955. In 1959 he was elected head of agricultural research, becoming in 1968, executive head of the Agricultural Chemicals Division responsible for research, development and marketing and member of the managing committee of the former J. R. Geigy Ltd.

After the merger of J. R. Geigy Ltd. with Ciba Ltd., Dr Gysin was appointed deputy head of the Division of Agricultural Chemicals and a member of the management co-ordination committee, and since 1970 has been head of the Central Office for Environmental Protection, reporting directly to the executive committee.



**U. Zorll**

Dr Zorll received his Doctorate at the Martin-Luther-University of Halle (Germany), where he also worked as a scientific assistant for four years. He joined the Research Institute of Pigments and Paints in Stuttgart in 1960 after five years of activity in industry. Now in charge of the Physics Department of the Institute, Dr Zorll's fields of interest include the viscoelastic behaviour of polymers, electron microscopy and measurement techniques of mechanical and optical paint film properties.



**E. G. Ladstädter**

Dr Ladstädter studied experimental physics at the University of Graz from 1962 to 1970. Since completion of his studies he has worked at the Research Centre of Vianova Kunstharz Aktiengesellschaft in Graz, at present controlling the applied physics laboratory. His special areas of interest include new experimental methods for the examination of resins and varnish films, and the hardening of organic coatings with CO<sub>2</sub> lasers.



**H.-D. Hanus**

Dr Hanus studied chemistry at the University of Graz and graduated in 1964, when he joined Vianova Kunstharz Aktiengesellschaft. During his service with the company, he was seconded for six months to a German car manufacturer and for two years to a French varnish factory. In particular he has specialised in technical work on the French market, with emphasis on developments for the automobile industry.



**R. P. M. Procter**

Graduating with a first-class honours degree in Metallurgy from Cambridge

University in 1963, Dr Procter went on to a PhD in the Department of Metallurgy at Cambridge in 1966. He then spent about five years in the United States as a post-doctoral research fellow at Carnegie Institute of Technology in Pittsburgh, and as a research engineer in the Corrosion Division of the Olin Corporation Research Laboratories in New Haven, Connecticut. Currently a lecturer in corrosion science in the UMIST Corrosion and Protection Centre, his major research interests are stress-corrosion, corrosion-fatigue and other mechanical aspects of corrosion.



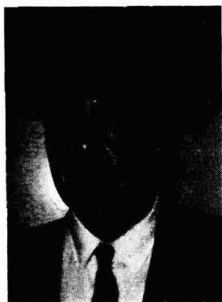
**V. Ashworth**

Dr Ashworth became a Graduate of the Royal Institute of Chemistry in 1962 and, following the award of an Electricity Council Research Scholarship in 1967, received a PhD from Nottingham University in 1970. He spent fourteen years in industrial research, initially with Johnson, Matthey & Co. Ltd., subsequently with CEBG. Currently a lecturer in corrosion science in the UMIST Corrosion and Protection Centre, his major research interests are inhibition and the effect of temperature on aqueous corrosion.



**D. S. Newton**

After qualifying from Bristol College of Technology in the mid-1950s, Mr Newton spent some years in the pvc industry with British Geon at Barry, South Wales. Joining the steel company Richard Thomas & Baldwins in 1960, he soon became involved in the emergent process of coating steel strip with paint and plastics, and was technically responsible for the commissioning of the first wide coil-coating line for steel in the UK at Gorseimon in West Wales. He is now head of organic coatings development at the research centre of the British Steel Corporation's Strip Mills Division, Port Talbot.



S. T. Harris

A graduate of the University of London, Mr Harris was for many years involved in all aspects of synthetic resin research and development with Arthur Holden, eventually transferring to the development of specialised industrial finishes. In this area he is now fully engaged with the problems of powder coatings, covering fundamental research, day-to-day development and technical service assistance to customers and equipment suppliers.



G. Scott

Professor Scott joined ICI, Billingham Division, as a laboratory assistant in 1943. Gaining entrance to Balliol College, Oxford, in 1948 he graduated with first class honours in chemistry, and, after a short period of research with Dr W. A. Waters on the chemistry of free radicals, returned to ICI (Dyestuffs Division) where he applied his knowledge of free radical chemistry in the field of auxiliaries for the polymer industry (free radical initiators, antioxidants, rubber accelerators, etc.).

Professor Scott became head of polymer auxiliaries section and was subsequently appointed head of the Grangemouth works research and development department. During this period his book "Atmospheric oxidation and antioxidants" was published.

Appointed professor of chemistry at Aston in 1967, Professor Scott is a strong supporter of the interdisciplinary PhD designed to provide industry with people of broader research background than the traditional specialist PhD. In accordance with these principles, his research activities in the University of Aston are concerned with industrial and social objectives. An interesting example of this is the work in progress on the development of photo-degradable plastics for the packaging industry, only one example of a number of socially directed research projects in progress in Aston's polymer research school.



K. McLaren

Mr K. McLaren graduated in Colour Chemistry at Leeds University in 1943 and since then has worked in the technical service department of ICI Dyestuffs (now Organics) Division; he is currently manager of the identification and colour measurement section. The author of more than thirty papers, mainly concerned with colour fastness, abnormal colour vision, lighting for colour matching and colour measurement, he is chairman of both the Fastness Tests Co-ordinating Committee and the Colour Measurement Committee of the Society of Dyers and Colourists. In 1957 he was awarded the Research Medal of the Worshipful Company of Dyers for his work on the fading of dyes and in 1970 was elected an Honorary Fellow of the Society of Dyers and Colourists for his notable contributions in the fields of colour measurement and colour fastness; this was only the second time the SDC had elected an Honorary Fellow.



C. Meredith

Mr Meredith graduated with a first class BSc in botany from the University of London, and subsequently joined BSI as a technical officer in 1949. Since 1970 he has been group manager of the chemical department. During his service with the BSI, Mr Meredith has acted as secretary of the pigments, paints and varnishes industry Standards Committee; he helped to initiate work on test methods for paints, following a request from the Paintmakers Association, and supervised the initiation of corresponding ISO work, acting as secretary for ISO/TC 35/SC 9 meetings for seven years. He is currently BSI representative on the PSSST product group on paints.



V. P. Simpson

Dr Simpson graduated from the University of Manchester Institute of Science and Technology in 1968 with a PhD in Chemical Engineering. For the past four years, he has held the post of project co-ordinator at the Central Research Laboratories of Hooker Chemical Corporation, where he has been engaged in studies of corrosion science, electrochemical cells, and surface chemistry

F. A. Simko, Jr.

Mr Simko graduated from the State University of Ohio with a BChE. His research studies have included colloid and surface chemistry, work conducted at the DuPont Experimental Station in Wilmington, Delaware. Since joining Hooker Chemical Corporation, he has held the post of manager, new chemical development, in which he has made many contributions in the areas of surface chemistry, corrosion science, and speciality chemicals.



R. L. J. Morris

R. L. J. Morris graduated from Cambridge University and joined the Materials Quality Assurance Directorate in 1959 to work on emission spectroscopy and the analysis of high purity metals. After working for seven years in the fields of explosives, plastics and rubbers, he joined the paints branch. Now appointed as a principal scientific officer and a research group leader, he has been seeking for the last three years to establish the technique of micro-indentation as a means of characterising the properties of paint films.



A. T. S. Rudram

Mr Rudram was educated in Wales where he developed his continuing enthusiasm for rugby football. After periods in resin, paint, leather finishes and printing ink manufacture, he saw service with the RAF, the last involving widespread travel across the world. He later joined the Paint Research Association and headed its technical division, as a principal research officer, for sixteen years. On his return to industry, his company's interests in overseas territories has continued to necessitate a great deal of overseas activity, particularly in the Caribbean and Far East.

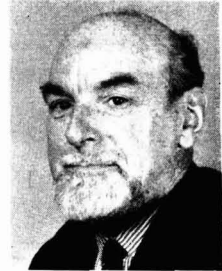
Mr Rudram is a past Honorary Research and Development Officer of OCCA and a past Chairman of the London Section. He is, at present, a member of Council, the Professional Grade Committee and the Technical Committee. He is the present Chairman of the Technical Advisory

Committee of the Paint Research Association and a member of its Executive Committee. In the Paintmakers Association he is a member of its Technical Committee and the present Chairman of the Health and Safety Sub-Committee. Much of his work with British Standards has involved paint testing methods and codes of practice on painting methods and prevention of corrosion.



J. D. Murray

In 1966, following six years' tertiary education at the University of Bristol, Dr Murray took up a lectureship in inorganic chemistry at the University of Malaya, Kuala Lumpur. Together with the normal teaching and administrative duties he was able to investigate an area of organotin chemistry.



F. Armitage

Mr Armitage, an Associate of the Royal Institute of Chemistry, worked for twenty years with Lewis Berger & Co., in the position of research manager for the latter half of this period. He contributed towards the introduction and establishment of monomer-modified oils and alkyds, and of water-based paint vehicles in this country. For the past ten years he has been attached to the Sherwin Williams Co. as European technical representative, in which position he has had wide experience of technical development on both sides of the Atlantic.

## News of Members

At the recent AGM of the British Colour Makers' Association, the following OCCA Members were elected to office:

*Chairman:* Mr P. Perkin, an Ordinary Member attached to the Manchester Section.

*Hon. Treasurer:* Mr F. B. Mortimer-Ford, an Ordinary Member attached to the London Section.

*Council Members:* Mr J. Bradley, an Ordinary Member attached to the Manchester Section; Mr H. G. Clayton, an Associate Member attached to the Manchester Section; Mr W. B. Cork, an Ordinary Member attached to the Hull Section; Mr V. Watson, an Ordinary Member attached to the Manchester Section.

The recently elected Officers and National Committee of the Paint Manufacturers and Allied Trades Association include the following OCCA Members:

*President:* Dr B. S. Gidvani, an Ordinary Member attached to the London Section.

*Vice-President:* Dr E. Bohringer, an Ordinary Member attached to the

London Section and a Fellow in the Professional Grade.

*Committee Member:* Mr R. J. Buckland, an Associate Member attached to the London Section.

Mr D. G. Dowling, an Ordinary Member attached to the Newcastle Section, has been appointed chief chemist/technical director of the Humbrol Paint Company Limited. Mr Dowling was previously with Toixide International Limited.

Mr J. P. Davidson and Mr F. Hellens, Ordinary Members attached to the Newcastle Section, have both taken up positions with the Universal Bedding & Upholstery Company Limited. Both were previously with Berger Chemicals Limited.

Mr T. E. Johnson, an Ordinary Member attached to the Manchester Section, has recently relinquished his position as technical director of John Matthews & Company Limited.

Mr J. Cain, an Ordinary Member attached to the London Section, has been appointed technical manager of Berger Paints, with responsibility for the automotive and electrocoat laboratories. Mr Cain was previously manager of the electrocoat laboratory.

Mr H. Smith, an Ordinary Member attached to the Manchester Section, has been appointed general manager of Robbialac Paints (Tanzania) Limited, and, in taking up the appointment, will transfer to the General Overseas Section.

Mr E. H. A. Bishop, an Ordinary Member attached to the Thames Valley Section, and an Associate in the Professional Grade, has been appointed to the board of E. & F. Richardson Limited as technical director.

## New FSPT President

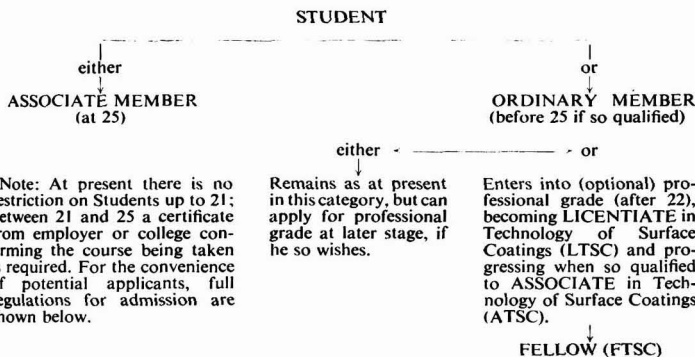
The recent Annual Meeting of the FSPT reported on p.12, was Mr. J.P. Teas' last function as president of the FSPT. Mr A. G. Rook, an ex-President of the Golden Gate Society, took office as President during the meeting.



# Professional Grade for Ordinary Members

The first year's working of the Professional Grade has proved to be most successful, as evidenced by the impressive list of names in the December issue of the *Journal*. As stated when the Grade was introduced, the

main emphasis is on younger Members at the Licentiate level, and the following diagram shows the suggested progression of a Student (up to 25 years of age)\*.



## Regulations for admission to the Professional Grade

Note: For the sake of simplicity, reference is made only to UK examinations etc., but equivalent qualifications overseas will naturally be accepted.

### A. Licentiate

1. Shall be an Ordinary Member of the Association and have been an Ordinary Member or Student of the Association for not less than one year.
2. Shall have attained the age of 22.
3. (a) Shall be a Licentiate of the Royal Institute of Chemistry in Coatings Technology (*viz.* Higher National Certificate + Endorsement in coatings technology + 1 year approved experience in the science or technology of coatings after passing the endorsement examination).

OR (b) Shall be a Licentiate of the Royal Institute of Chemistry in another relevant subject such as advanced analytical chemistry, colour chemistry or polymer science, and shall have two years' approved experience of coatings since so qualifying.

OR (c) Shall hold the Full Technological Certificate of the City & Guilds of London Institute in a relevant subject as approved by the Professional Grade Committee and shall have two years' approved experience in the science or technology of coatings since gaining the FTC.

OR (d) Shall have passed Higher National Certificate or Higher National Diploma with three years' approved experience in the science or technology of coatings since qualifying, but two years' approved pre-qualification experience shall be deemed equivalent to the third post-qualification year.

OR (e) Shall be graduate in relevant subject with not less than 1 year's approved experience.

OR (f) Shall have passed such other qualifications as approved by the Professional Grade Committee from time to time.

4. Shall be required to satisfy the Professional Grade Committee, or some other body approved by the Professional Grade Committee in a viva voce examination and submit a dissertation on a topic previously approved by the Professional Grade Committee.

5. Shall normally be sponsored by three Ordinary Members of the Association in the professional grade (either Associate or Fellow) at least one of whom must be a Fellow.

6. Shall have paid the fee stipulated by the Council and have paid the current subscription payable by an Ordinary Member.

### B. Associate, being already a Licentiate

1. Shall, since his election to the Licentiate-ship, have practised the science or technology of coatings for not less than three years.

2. Shall provide evidence acceptable to the Professional Grade Committee of his superior professional skill and maturity.

3. Shall hold the City & Guilds of London Institute Insignia Award OR shall submit a thesis or dissertation of comparable level on a topic previously approved by the Professional Grade Committee OR shall have published work which, in the opinion of the Professional Grade Committee, is of comparable merit.

4. MAY be required to satisfy the Professional Grade Committee or some other body as approved by the Professional Grade Committee in a viva voce examination.

5. Shall normally be sponsored by three Ordinary Members of the Association in the professional grade (either Associate or Fellow) at least one of whom must be a Fellow.

6. Shall have paid the fee stipulated by the Council and have paid the current subscription payable by an Ordinary Member.

### C. Associate, not already a Licentiate

#### EITHER

1. Shall be not less than 24 years of age.

2. Shall be an Ordinary Member of the Association and have been an Ordinary Member or Student of the Association for not less than two years.

3. Shall hold the Graduateship of the Royal Institute of Chemistry or Institute of Physics or a University or Council of National Academic Awards degree recognised by the Royal Institute of Chemistry or Institute of Physics as giving full exemption from the Graduateship examination.

4. Shall have not less than two years' approved post-graduate experience in the science or technology of coatings.

5. Shall normally be required to satisfy the Professional Grade Committee or some other body approved by the Professional Grade Committee, at a viva voce examination.

6. Shall normally be sponsored by three Ordinary Members of the Association in the professional grade (either Associate or Fellow) at least one of whom must be a Fellow.

7. Shall have paid the fee stipulated by the Council and have paid the current subscription payable by an Ordinary Member.

#### OR

8. Shall be not less than 30 years of age.

9. Shall be an Ordinary Member of the Association and have been an Ordinary Member of the Association for not less than three years.

10. Shall have been engaged in practising the science or technology of coatings for not less than seven years and shall have attained a position of considerable standing in the industry.

11. Shall normally be required to satisfy the Professional Grade Committee in viva voce examination of his professional competence.

12. Shall normally be sponsored by three Ordinary Members of the Association in the professional grade (either Associate or Fellow) at least one of whom must be a Fellow.

13. Shall have paid the fee stipulated by the Council and have paid the current subscription payable by an Ordinary Member.

### D. Fellow

Note: This is the senior award of the professional grade and signifies that the holder has made outstanding contributions to the science or technology of coatings or has reached a position of eminence in the industry through the practice thereof. The Professional Grade Committee will require substantial evidence of professional maturity in the science or technology of coatings although commercial experience will be

taken into account in assessing the merits of candidates.

1. Shall be not less than 33 years of age.
2. Shall have been an Ordinary Member of the Association for not less than five years.
3. Shall be engaged in a position of superior responsibility in the coatings industry.
4. EITHER (a) shall have been an Associate of the professional grade for at least eight years;  
OR (b) shall have not less than fifteen years' experience of the science or technology of coatings in a position of superior responsibility.
5. Shall submit, with his application, an account of his experience, with due reference to scientific and technological interests, achievements and publications.
6. Shall normally be sponsored by three Ordinary Members of the Association in the professional grade, all of whom must be Fellows.
7. Shall have paid the fee stipulated by the Council and have paid the current subscription payable by an Ordinary Member.

The fees payable with applications are as follows:

Fellow—£10.00 Associate—£6.00  
Licentiate—£3.00

#### Application

Completed application forms should be returned, together with the appropriate

remittance, to the Director & Secretary at the Association's offices (except in the case of those Members attached to the Auckland, South African and Wellington Sections, who should address their forms to their Section Hon. Secretaries).

The Committee wishes it to be known that Members rejoining the Association after a period in other industries may include length of service as an Ordinary Member before their resignation as part of the qualifying periods for entry into the Grade.

Students wishing to apply for entry into the Professional Grade must first make application in writing for upgrading to Ordinary Membership, giving the reasons for their eligibility for such regrading. Applications, together with the appropriate remittance, should be addressed as for application for admission to the Professional Grade.

Potential applicants are recommended to give the fullest possible details of their appointments, including the number and type of staff under their control, and indicating to whom the applicant is responsible, as this aids the committee considerably in its deliberations.

It is felt that applicants for admission to the Licentiate grade might wish to have further information on the pattern which it is suggested should be followed for dissertations, the subjects of which have first to be approved by the Professional Grade Committee.

The dissertation should be preceded by a short summary and commence with a brief introduction and some account of the

current state of knowledge. Where practicable it should follow the general format of a paper in *JOCCA*.

The dissertation may be a review of a subject, a theoretical treatment, descriptive of practical work or a combination of these. It must indicate that the candidate has a reasonably wide and up-to-date knowledge of his chosen subject and understands the basic scientific principles underlying it, and that he is able to think critically and constructively.

Where practical work is described, some attempt should be made to draw theoretical conclusions or to form some provisional hypothesis, together with an outline of what further work would be required to confirm the views put forward or further to advance the knowledge of the subject.

Where the dissertation is a review or a theoretical treatment, there should be an attempt to contrast and compare any opposing views expressed in earlier works, to examine the views critically, to suggest any compromise interpretation, to account for all the known facts and to outline any further work by which the opposing views could be tested.

Where applicable, references should be given to published work, graphs, diagrams etc. to be appended.

Length: Text should be approximately 5,000 words.

Applicants should refer to the paper by Moss which appears in this issue: the Professional Grade Committee feels that candidates for the Licentiate grade could with advantage use this paper as a model for their dissertations.

## London Section

### Ladies' Night

The London Section Ladies' Night was held on Friday 6 October in the Du Barry and Dauphine Rooms at the Café Royal, London W1. The guest of honour, M. M. Tapin, Past Chairman of the Paris Section of the Fédération Nationale des Fabricants de Peintures, Vernis et Encres d'Imprimerie, in proposing the toast to the London Section, referred to the even closer contact between the French and British Associations that will be necessitated when Britain joins the Common Market. He suggested a London-Paris Ladies' Night.

Mr D. Eddowes, Section Chairman, commenced his reply in French and said that the committee would certainly bear the trans-Channel suggestion in mind. He welcomed M. and Mme. Tapin, Chairmen of Sections and Branches as follows: Mr and Mrs Coates (Southern Branch), Mr and Mrs Burns (Scottish Section), Mr and Mrs Gollop (Bristol Section), Mr and Mrs Gay (Midlands Section), Mr and Mrs Duell (Newcastle Section), and Mr and Mrs Duckworth (Manchester Section), together with Dr S. H. Bell (Past President), Mr R. H. Hamblin (Director & Secretary) and Miss Joyce Varney. Mr Eddowes read a telegram received from Mr R. Munn and then amused the gathering with a series of humorous stories, finally thanking the ladies for being so helpful to the Section and for attending the function.

Mr Duckworth, replying on behalf of the Guests and Ladies, wished the committee and section every success.

Following the dinner there was a tombola, and dancing continued to the Billy Wells band until 1 a.m. D.F.A.J.



## 25th Technical Exhibition

### 21-24 May 1973

### Empire Hall, Olympia, London

#### Another exhibitor at OCCA 25

There will now be 94 stands featuring products from more than 100 companies from the UK, Belgium, Finland, France, Germany, Holland, Italy, Norway, Sweden, Switzerland and the USA.

In addition to the companies listed in the December issue, space has now been allocated to Jacobson Van Den Berg & Co (UK) Ltd, a company that has never shown at an OCCA Exhibition.

#### Official Guide

It is hoped that copies of the Official Guide will be available early in March when they will be circulated free of charge to Members of the Association and other scientists and technologists so that they can plan their itineraries well in advance of the Exhibition.

#### Proposed coach parties

It is hoped to organise coach parties from the UK Sections and Branches in order to keep travel costs to a minimum and thus encourage younger technical personnel to

visit the Exhibition in even greater numbers. Although priority will naturally be given to Members of the Association, any company or individual wishing to obtain places on these coaches should write to the Association's offices so that he may be put in touch with the local Section who will inform him of the appropriate charge and the day of the visit.

#### Facilities available for visitors and exhibitors

To provide services for the cashing of cheques and foreign drafts for both visitors and exhibitors, the National Westminster Bank has been allocated a stand in the Hall. A Post Office self-service suite will be available for the purchase of stamps and the posting of mail.

The services of interpreters will again be available free of charge to exhibitors and visitors and a Press Room will be provided for exhibitors to display material. There will be two buffets for general use, and in addition a special bar will be set aside for the use of exhibitors only.

## Forthcoming Events

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

### Thursday 4 January

*Newcastle Section:* "Business planning; only a numbers game?" by Mr J. Willey of Berger, Jenson & Nicholson Ltd., to be held at the Royal Turks Head Hotel, Grey Street, Newcastle, at 6.30 p.m.

### Monday 8 January

*Hull Section:* "Is the paint industry on the move?" by Mr D. E. Eddowes of Polymers Paint & Colour Journal, to be held at the Queens Hotel, George Street, Hull, at 7.00 p.m.

### Tuesday 9 January

*West Riding Section:* "High speed milling" by Mr D. W. Wilkinson of Augusts Limited, to be held at the Griffin Hotel, Leeds, at 7.30 p.m.

### Wednesday 10 January

*London Section:* "The inkmaker and heat transfer inks" by Mr D. W. Woods of Coates Bros. Inks Ltd., to be held at the South Bank Polytechnic at 7.00 p.m.

*Newcastle Section—Student Group:* "Fields of application of synthetic resins" by Dr A. F. Everard of Berger Chemicals Ltd., Resinous Chemicals Division, to be held at Newcastle Polytechnic, Ellison Place, Newcastle, at 3.00 p.m.

### Thursday 11 January

*Thames Valley Section—Student Group:* "Marine coatings" by a speaker to be announced. To be held at the Main Lecture Theatre, Slough College, at 4.00 p.m.

### Friday 12 January

*Manchester Section:* "Design is where you find it," a talk on the psychology of colour and design, by Mr E. Pond of WPM Ltd., to be held at the Manchester Literary and Philosophical Society at 6.30 p.m.

*Scottish Section:* Annual Dinner Dance, to be held at the Central Hotel, Glasgow.

### Wednesday 17 January

*Manchester Section—Student Group:* "Solvents in paints and printing inks" by Mr C. J. Nunn of Shell Research Ltd., to be held at the Manchester Literary and Philosophical Society at 4.30 p.m.

*Scottish Section:* Joint meeting with BPBMA, Technical Section. "Adhesives," speaker and venue to be announced.

*Scottish Section—Eastern Branch:* "Modern analytical techniques" by Mr P. S. Nisbet of A. B. Fleming & Co. Ltd., to be held at the Carlton Hotel, North Bridge, Edinburgh, at 7.30 p.m.

### Friday 19 January

*Irish Section:* "Marketing in the context of the small firm" by Mr Rock of the Irish Management Institute, to be held at the Clarence Hotel, Dublin, at 8.00 p.m.

*Midlands Section:* Annual Dinner Lecture "Purchasing in the paint industry" by Mr D. F. Brocklehurst of BJN Group Supplies Ltd., to be held at the Birmingham Chamber of Commerce and Industry, 75 Harborne Road, Birmingham 15, at 6.30 p.m.

### Saturday 20 January

*Scottish Section—Student Group:* "Pigments for plastics" by Mr S. T. Bebbington of ICI Ltd., to be held at the St. Enoch Hotel, Glasgow, at 10.00 a.m.

### Wednesday 24 January

*London Section:* Whole day symposium at Thameside Polytechnic, Woolwich. "Implications of pollution for the coatings industry." Papers: "Printing waste" by Mr K. Pond of Shuck MacLean & Co. Ltd.; "Pollution, industry and responsibility" by Dr R. C. Denney of Thameside Polytechnic; "Pollution of water by industrial waste products" by Mr M. D. Longstaff of the Trade Effluent Section, GLC; "Solvents" by Mr A. J. Goodfellow of Carless, Capel & Leonard Ltd. Film "The yellow trail".

### Thursday 25 January

*Thames Valley Section:* "Recent aspects of anti-corrosive pigments" by Mr M. Rendu of Imperial Smelting Corp. (Alloys) Ltd., to be held at the Beech Tree Hotel, Maxwell Road, Beaconsfield, Bucks, at 7.00 p.m.

### Friday 26 January

*Bristol Section:* "Gravure printing", speaker to be announced. To be held at the Royal Hotel, Bristol, at 7.15 p.m.

### Thursday 1 February

*Newcastle Section:* "Polyurethane systems in the marine industry" by Dr F. Blomeyer of Farbenfabrieken Bayer, to be held at the Royal Turks Head Hotel, Grey Street, Newcastle, at 6.30 p.m.

### Friday 2 February

*Scottish Section—Eastern Branch:* Annual Burns Supper, to be held at the Royal Mile Centre, Edinburgh.

### Monday 5 February

*Hull Section:* "Are emulsion paints good enough?" by Mr J. Cruden of Harco Ltd., to be held at the Queens Hotel, George Street, Hull, at 7.00 p.m.

*London Section:* "Zinc phosphate, its history, development and future" by Mr M. Rendu of Imperial Smelting Corp. (Alloys) Ltd., to be held at the East Ham Technical College at 7.00 p.m.

### Wednesday 7 February

*Newcastle Section—Student Group:* A talk by a member of the staff of Camrex Ltd., to be held at the Newcastle Polytechnic, Ellison Place, Newcastle, at 3.00 p.m.

### Thursday 8 February

*Midlands Section—Trent Valley Branch:* "Vinyl resins" by Mr D. J. Silsby of Bakelite Xylonite Ltd., to be held at the British Rail School of Transport, London Road, Derby, at 7.00 p.m.

*Scottish Section:* "Powder coatings" by Mr D. W. Brooker of Shell Research Ltd., to be held at the St. Enoch Hotel, Glasgow, at 6.00 p.m.

*Thames Valley Section—Student Group:* Works visit, to be arranged. Leaving Slough College car park at 1.00 p.m.

### Friday 9 February

*Manchester Section:* "Technological forecasting and its potential in the surface coatings industries" by Mr H. Jones, to be held at the Manchester Literary and Philosophical Society at 6.30 p.m.

*Thames Valley Section:* Buffet Dance, to be held at "Great Fosters," Egham, at 7.00 p.m.

### Saturday 10 February

*Scottish Section—Student Group:* "Selection of pva emulsions for water based paints" by Mr D. Wallace of Vinyl Products Ltd., to be held at the St. Enoch Hotel, Glasgow, at 10.00 a.m.

### Tuesday 13 February

*London Section—Southern Branch:* "SeaIants" by Mr L. D. Hall of Ralli-Bondite Ltd., to be held at the Pendragon Hotel, Southsea, at 7.00 p.m.

*West Riding Section:* "Functional extenders in emulsion paints" by Mr S. E. Maskery of Crosfield Chemicals Ltd., to be held at the Griffin Hotel, Leeds, at 7.30 p.m.

### Wednesday 14 February

*Manchester Section—Student Group:* "Synthetic stoving finishes" by Mr D. Pountain of WPM Paint Division, to be held at the Manchester Literary and Philosophical Society at 4.30 p.m.

### Friday 16 February

*Irish Section:* "Organic pigments for the paint industry" by H. Biffar of Fabwerke Hoechst AG, to be held at the Clarence Hotel, Dublin, at 8.00 p.m.

### Wednesday 21 February

*West Riding Section:* Joint meeting with the Northern Section of the Colour Group; subject "Whiteness."

*Scottish Section—Eastern Branch:* "Testing of raw materials for paint and varnish" by a speaker from ICI Ltd., to be held at the Carlton Hotel, North Bridge, Edinburgh.

### Thursday 22 February

*Thames Valley Section:* "Recent developments in vapour phase transfer printing" by Dr C. E. Vellins of L. B. Holliday & Sons Ltd. A short introductory talk on textile printing in general will be given by Mr M. Roberts of Pasolds Ltd. To be held at the Beech Tree Hotel, Beaconsfield, Bucks, at 7.00 p.m.

### Friday 23 February

*Bristol Section:* "Modern dispersion techniques" by Mr I. Berg of Berger Paints Ltd., to be held at the Angel Hotel, Cardiff, at 7.15 p.m.

*Newcastle Section:* Dinner Dance, to be held at the Five Bridges Hotel, Gateshead.

## Register of Members

The following elections to Membership have been approved by Council. The Section to which new Members are attached is given in italics.

### Ordinary Members

- HOLMES, MICHAEL JOHN, BSc, 22 Glamis Drive, Chorley, Lancashire. (*Manchester*)  
 HOWE, BRIAN RUSSELL, BSc, ARIC, Burrell & Co. Ltd., 262 West Ferry Road, Millwall, E14 9AQ. (*London*)  
 JEGHER, GAUDENZ JURG, Birmannsgasse 14, CH-4052 Basel, Switzerland. (*General Overseas*)  
 MANLEY, DAVID, 47 Glebe Road, Stoneferry Road, Hull, HU8 0DX. (*Hull*)  
 McMAHON, BARRY JOHN, BSc, Ciba-Geigy (UK) Ltd., Hawkhead Road, Paisley, Scotland. (*Scottish*)  
 McNEIL, DAVID CALDER, 21 Alloway Road, Newlands, Glasgow S.3. (*Scottish*)  
 NELSON, COLIN, GRIC, 26 Tannahill Crescent, Johnstone, Renfrewshire. (*Scottish*)  
 NG, WING YAN, BSc, PhD, 1st Floor, 92A Pokfulam Road, Hong Kong. (*General Overseas*)  
 NICKLIN, GILES, BSc, PhD, 6 Short Street, Pangbourne, Berkshire. (*Thames Valley*)  
 RAMPLEY, DENNIS NEIL, 28 Lower Herne Road, Herne Bay, Kent. (*London*)  
 SEMPLE, JAMES WILLIAM, BSc, 119 Cranworth Gardens, London SW9 0NU. (*London*)  
 TAILOR, M. RAMANLAL, BSc, PO Box 1277, Ndola, Zambia, Central Africa. (*General Overseas*)  
 THOMSON, JAMES, LRIC, 4 Ben Wyvis Drive, Hawkhead, Paisley, Scotland. (*Scottish*)  
 WONG, HO-KWONG, BSc, 223 Block 20, Tung Tau Village, Kowloon, Hong Kong. (*General Overseas*)

### Associate Members

- SEDDON, GARRY CHARLES, Buckley & Young Ltd., PO Box 30148, Lower Hull, Wellington, New Zealand. (*Wellington*)

### Student Members

- BAINES, KENNETH, Blundell Permoglaze, Sculcoates Lane, Hull, East Yorkshire. (*Hull*)  
 EDMONSON, JOHN MARK, Department of Colour Chemistry and Colour Technology, University of Bradford, Richmond Road, Bradford 7. (*West Riding*)  
 FINN, JOHN ROBERT, 6 Ashmore Street, Sunderland, Co. Durham (*Newcastle*)  
 HALL, JOHN, 121 Lynthorpe, Ryhope, Sunderland, Co. Durham. (*Newcastle*)  
 ROCK, JOHN WILLIAM, 80 Hartfield Crescent, Wimbledon, London SW19. (*West Riding*)  
 ROMANIUK, OLGA CATHERINE, 23 Pemberton Drive, Bradford 7, Yorkshire. (*West Riding*)  
 ROONEY, ROBERT LAMBIE, 18 Clarence Street, Paisley PA1 1PU, Scotland. (*Scottish*)  
 ROWELL, IAN STEWART, 2 Rydal Mount, Fulwell, Sunderland, Co. Durham. (*Newcastle*)  
 SOUTHALL, GARFIELD JEFFEREY, 27 Boothferry Road, Hull. (*Hull*)  
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 UNDERWOOD, ROSS WALTER, Botley Road, Bishop's Waltham, Southampton, Hants. (*West Riding*)

## Paint Technology Manuals Part Seven: Works Practice

The final chapter and index of "Works Practice," the seventh of the Association's series of Paint Technology Manuals, appeared in the December issue of the *Journal*. As already announced, reprints of each section of the Manual have been prepared every month, and the complete set is now to be collated, bound, and offered for sale.

The articles appearing under Student Reviews each month have been so designed

that the bound volume will form a complete, coherent book with full continuity. Apart from the necessary typographical changes in maintaining consistency in the *Journal*, "Works Practice" will be exactly the same as those parts of the Paint Technology Manuals already published by Chapman & Hall Ltd.

The price of "Works Practice," which will contain 218 pages and be bound in limp covers, will be £2.40, post free in the

UK. Registered Students of the Association may purchase the book at a special price of £1.40; however, this concession will be restricted to one copy per student, who should in all cases apply directly to the Association quoting his or her registered number.

It is anticipated that the book will be available towards the latter part of January orders can now be accepted.

# FAT-FAX 8

FAT-FAX... in which Price's Chemicals Ltd — Britain's largest oleochemical manufacturer — talks a bit about its raw materials, its processes, its products and their applications. FAT-FAX 8 is the first in a series of FAT-FAXes devoted entirely to SURFACE-COATING applications. Price's might prove relatively immodest when it comes to knowledge of oleochemicals; on second thought, read 'does' for 'might', and 'thoroughly' for 'relatively'. But when it comes to oleochemical applications, Price's modesty is real, and fully justified. You, gentle reader, are the expert here and we'll not forget it. Yet we dare hope that the information in this series will prove useful, thought-provoking, readable or (with luck) all three.

## Four whys and a wherefore re fatty acids

As far as alkyds are concerned (and that's pretty far, in fact), the important chemical characteristics of fatty acids are four in number. *First*, their reactive acid carboxyl groups: these make possible reactions with alcohols to produce the big molecules.

*Second*, their long straight carbon chains: these provide solubility in hydrocarbons, plus general flexibility. *Third*, their methylene groups activated by double bases: these permit the autoxidation that produces the necessary drying. *Fourth*, their unsaturation, making double bonds available for polymerisation and forming adducts via Diels-Alder reactions. When it comes to *Fatty Acids v. Whole Oils*, the wherefore that overcomes the slight price disadvantage of fatty acids is their whole spectrum of significant advantages. Some useful parts of the spectrum: more alkyd resin throughput from a given plant, easier process control, better quality control, superior resin properties, a more uniform molecular-weight distribution, freedom from contaminants, harder paints, glossier enamels, better exterior durability. But perhaps most important of all — with a fatty acid, you get exactly what you want every time, and your choice of polyol is unrestricted too.

## Not every good backbone's an alkyd

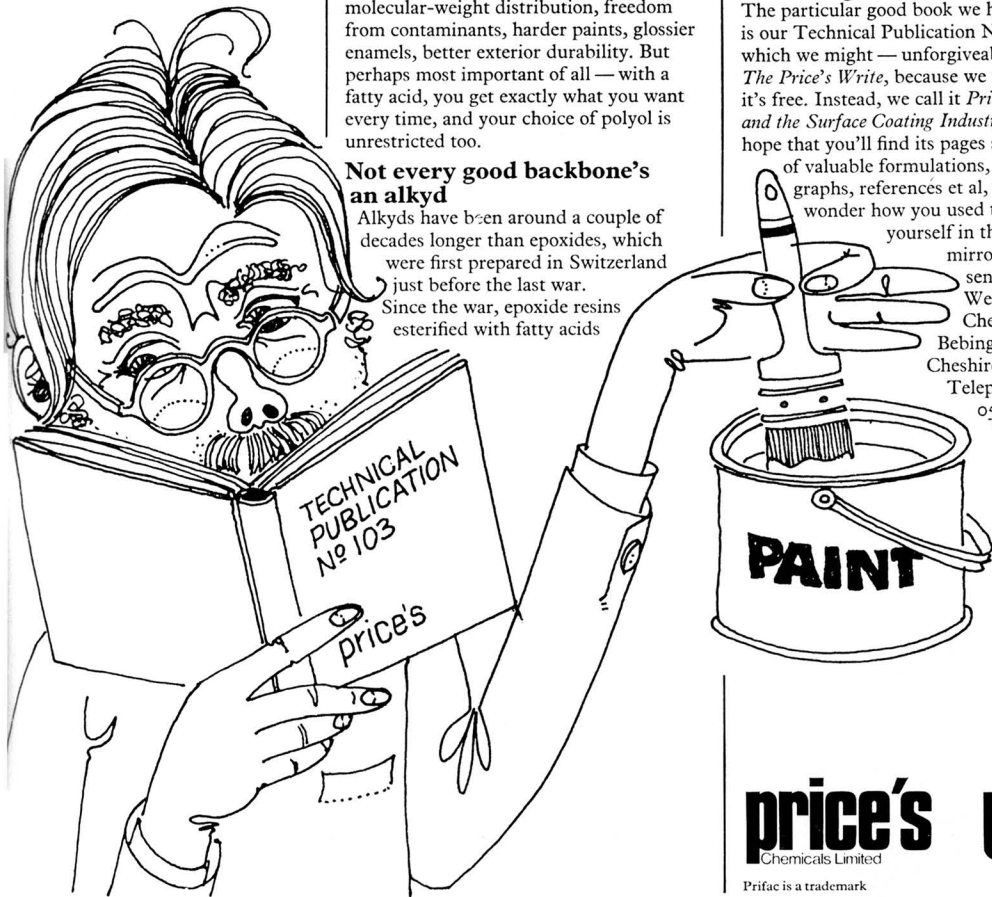
Alkyds have been around a couple of decades longer than epoxides, which were first prepared in Switzerland just before the last war. Since the war, epoxide resins esterified with fatty acids

have provided an increasingly popular backbone for surface coatings. This backbone, formed by ether and carbon-carbon links, isn't as easily saponified as its alkyd counterpart (which has ester links). So it exhibits excellent resistance to water and other chemicals. Both the fatty acid chosen and the degree of esterification affect the specific properties of the final coating. For example, a well-known ester suitable for air-drying brushing enamels is based on Prifac 007 White Distilled Soya Oil Fatty Acids, 80% esterified. In practice the theoretical quantity of fatty acid is not used and the degree of esterification or 'oil length' is selected according to the intended use of the ester: anything over 70% esterification is considered a long oil length. A typical short length (40%) esterification ester, based on Prifac 066 (12-hydroxystearic acid), makes a useful stoving finish with melamine/formaldehyde resin.

## But to get below the surface, read the good book

The particular good book we have in mind is our Technical Publication No. 103, which we might — unforgivably — call *The Price's Write*, because we wrote it and it's free. Instead, we call it *Price's Products and the Surface Coating Industry*, in the hope that you'll find its pages so chock full of valuable formulations, comments, graphs, references et al, that you'll wonder how you used to face

yourself in the shaving mirror. May we send you one? We're Price's Chemicals Ltd, Bebington, Wirral, Cheshire L62 4UF Telephone 051-645 2020. Telex 62408.



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# Paint Technology Manuals



## Part Seven: Works Practice

The seventh volume of the Association's series of Paint Technology Manuals, entitled "Works Practice," is now being collated, bound and offered for sale.

The articles which have appeared under Student Reviews each month have been so designed that the bound volume will form a complete, coherent book with full continuity. Apart from the necessary typographical changes in maintaining consistency in the *Journal*, "Works Practice" will be exactly the same as those parts of the Paint Technology Manuals already published by Chapman & Hall Ltd.

It is anticipated that the book will be available towards the end of January 1973; orders can now be accepted.

### Contents include:

*Chapter 1—The factory. Siting and layout*

*Chapter 2—Raw materials. Storage and handling*

*Chapter 3—Varnish and media manufacture:*

- (a) Cold processes*
- (b) Manufacture in open vessels*
- (c) Manufacture in closed vessels*

*Chapter 4—Paint manufacture:*

- (a) General*
- (b) Processes involving premixing*
- (c) Processes without premixing*
- (d) Layouts, maintenance, filling and warehouse*

*Chapter 5—Factory organisation and personnel*

*Chapter 6—Legal requirements and regulations*

*Chapter 7—Factory hazards and safety precautions*

To: Oil and Colour Chemists' Association, Priory House, 967 Harrow Road, Wembley, Middx. HA0 2SF.

Please send . . . copies of Paint Technology Manual Part Seven, "Works Practice," @ £2.40 per copy, post free in U.K. I enclose a cheque/P.O. for £. . .

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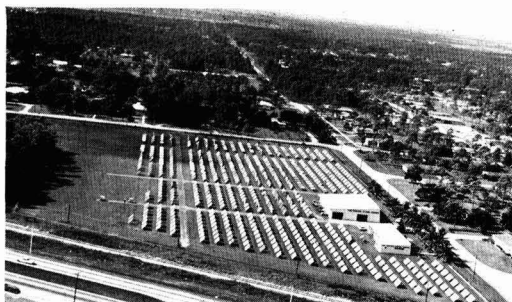


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# TECHNICAL EXHIBITION

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Further information in Notes and News p. 72 of this issue