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JOURNAL OF THE IL & COLOUR CHEMISTS' ASSOCIATION

Kinetics of film formation of alkyl silicate zinc-rich coatings

T. Ginsberg, C. N. Merriam and L. M. Robeson

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D. A. St. John and K. R. Markham

Polyester resins and glass reinforced polyesters, with particular emphasis on chemical plant

J. A. Raymond

Prospects for surface coating resins in the European market

Ch. P. Martin

The Health and Safety Act – a student review

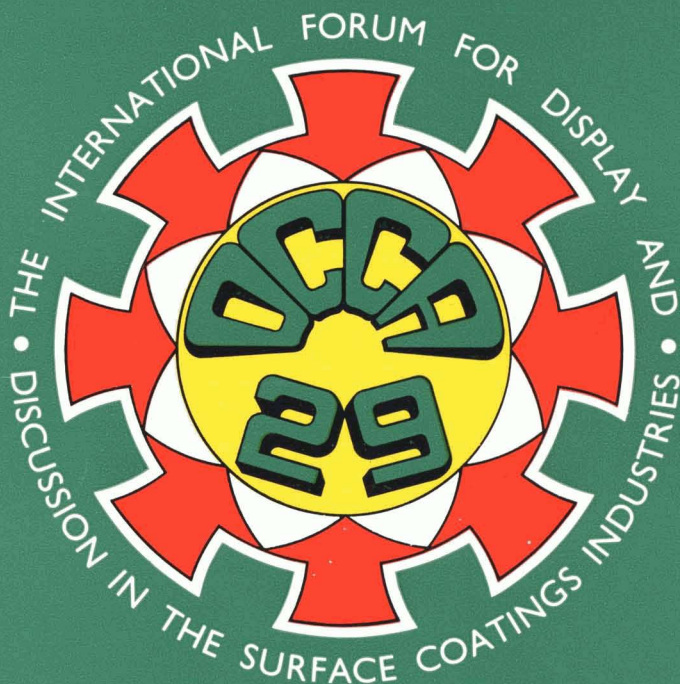
D. M. Wilson

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22-25 MARCH 1977

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OIL & COLOUR CHEMISTS' ASSOCIATION

CURRENT PUBLICATIONS

Introduction to Paint Technology

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

It is expected that the new edition of "Introduction to Paint Technology" will be available in the late autumn of 1976 and advance orders are now being accepted.

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

Paint Technology Manuals: Part Seven "Works Practice"

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

Price: £3.00

Paint Technology Manuals: Part Three "Convertible Coatings"

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

Price: £2.80

Ultraviolet Polymerisation

A volume, bound in limp covers, comprising papers presented at a symposium of the Newcastle Section of the Association in 1975 and subsequently published in the *Journal*. The titles and authors of the papers are given in an advertisement on page iv of this issue.

Price: £5.00

Conference Preprints

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

The 1973 Conference was held at Eastbourne with the theme "Towards 2000" and the bound set of preprints for this Conference includes 16 papers all dealing with various

aspects of the surface coatings industry towards the year 2000.

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

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

The Association's 1977 Conference will be held once again at Eastbourne; further details are given on page 349 of this issue.

History of the Association

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

Price: £1.50

Journal of the Oil and Colour Chemists' Association

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

Price: £20.00 per year

Back issues

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

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Consolidated Indexes

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

This invaluable "quick reference" guide to the literature of the surface coatings industries has been produced in the international A4 size and it can, therefore, be bound with copies of the *Journal*. (Binding facilities for JOCCA are available through the Association's offices.)

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

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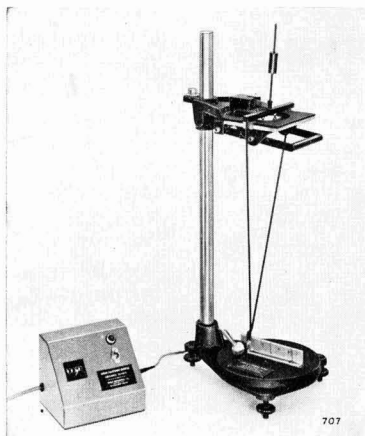
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UV polymerisation

The twelve papers in this volume were originally published in the *Journal* earlier in 1976. They are based on lectures given at a Symposium of the Newcastle Section of the Association, held at Durham University on 10 and 11 April 1975. Titles and authors are listed below:

Photochemical excitation and its consequences—a review by *R. B. Cundall*

The application of UV-curing materials and technology to packaging by *A. D. Lott*.

Photoinitiation by aromatic carbonyl compounds by *A. Ledwith*.

The technological literature relating to polymerisation photoinitiators by *A. Pryce*.
Photopolymerisation: the kinetics of a-type photopolymerisation by *F. C. de Schryver and N. Boens*.

The use of polyfunctional monomers in UV curing by *J. R. Younger*.

Sensitisation and stabilisation in monomer/polymer systems by *D. Phillips*.

Photodegradation of polymers by *I. C. McNeill*.

UV-drying equipment, design and installation by *R. E. Knight*.

Ultraviolet curing inks by *A. A. Gamble*.

Some aspects of the pigmentation of UV-curable systems by *B. E. Hulme*.

Some aspects of the formulation of UV-curable polyester based systems by *A. Laws, S. Lynn and R. Hall*.

To obtain this book, complete the order form on page (ii) of this issue and send with the necessary remittance to the Association's offices.

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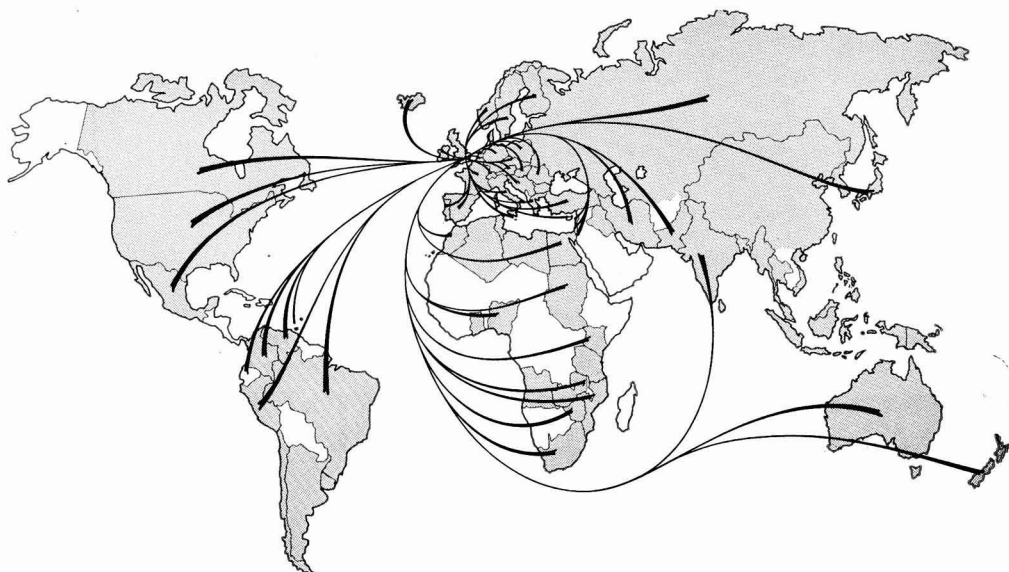
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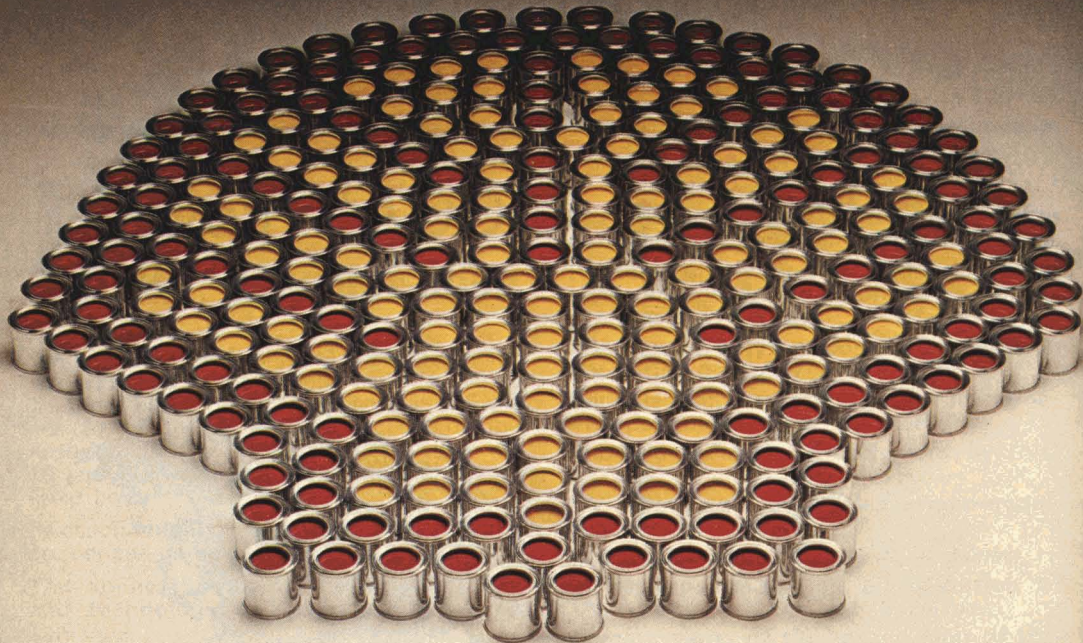


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For further information concerning advertisements in these or other OCCA publications, contact C. A. Tayler, JOCCA, Priory House, 967 Harrow Road, Wembley, Middlesex, HA0 2SF England. Tel: 01-908 1086. Telex 922670 (OCCA Wembley).



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The optional Professional Grade for Ordinary Members of OCCA was instituted by Council in 1971. Since that time, over 400 candidates have been successful in their applications to join the Professional Grade and a full list was last published in the December 1975 issue of the *Journal*. The various routes to the three Grades (see above) in diagrammatic form and the full regulations for admission are published in the July 1976 issue.

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

OCCA-29 Exhibition

The Association's twenty-ninth Technical Exhibition will be held at Alexandra Palace, London N22, from 22 to 25 March 1977. Further details regarding the arrangements for OCCA-29 and the many facilities offered at Alexandra Palace appear on page 346 of this issue of the *Journal*.

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

Intending exhibitors are reminded that their completed application forms and remittance for stand space must be returned to the Director & Secretary of the Association not later than **Friday 1 October 1976**.

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

"Official Guide"

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

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Transactions and Communications

Kinetics of film formation of alkyl silicate zinc-rich coatings

By T. Ginsberg, C. N. Merriam and L. M. Robeson

Union Carbide Corporation, Bound Brook, New Jersey, USA

Summary

The scientific study of the kinetics of film formation of alkyl silicate zinc-rich coatings has been hindered in the past by the lack of an accurate and sensitive method to monitor the changes occurring in the film. Application of the visco-elastic theory to the process of drying suggests the measurement of the elastic modulus as a particularly appropriate method for monitoring the changes occurring during film formation. This has been performed using two techniques of dynamic mechanical measurement: the torsion pendulum, which provides a measure of the shear elastic modulus; and the "Rheovibron", which yields values of tensile elastic modulus.

Keywords

Types and classes of coatings and allied products

zinc-rich coating
silicate paint

Raw materials: binders (resins, etc)

alkyl silicate

Both methods have been used to study the kinetics of film formation of 80 per cent hydrolysed ethyl silicate, ethyl-ethoxyethyl silicate, and ethoxyethyl silicate zinc-rich coatings under a variety of conditions. The data obtained show that the rate of film formation is independent of the humidity in the surrounding atmosphere, indicating that residual hydrolysis takes place at a very slow rate. The kinetics of initial film formation are determined primarily by the rates of evaporation of the solvents present and the consequent shifting of the equilibrium conditions, so that hydrolysis may proceed by a lacquer-type mechanism.

Processes and methods primarily associated with drying or curing of coatings

drying
film formation

Properties, characteristics and conditions primarily associated with materials in general

elasticity
mechanical property

La cinétique de la formation des films de revêtements de surface riches en poudre de zinc et à base d'un alkyl silicate

Résumé

Dans le passé l'étude scientifique de la cinétique de la formation des films revêtements de surface riches en poudre de zinc et à base d'un alkyl silicate a été retardée par l'absence d'une méthode précise et sensible pour contrôler les changements qui se produisent dans le film. L'application de la théorie de visco-élasticité au processus de séchage évoque la possibilité que la détermination du module d'élasticité pourrait offrir une méthode particulièrement apte pour contrôler les changements qui se produisent lors de la formation du film. On a effectué cette détermination en utilisant deux techniques de mesure mécano-dynamique: le pendule de torsion qui donne par cisaillement une mesure du module d'élasticité; et le "Rheovibron" qui rend les valeurs à la limite du module d'élasticité par torsion.

On a utilisé toutes les deux méthodes pour étudier sous une gamme de diverses conditions la cinétique de la formation des films de revêtements de surface riches en zinc à base respectivement de silicate d'éthyle hydrolysé à 80%, de silicate d'éthyl-éthoxy-éthyle, et de silicate d'éthoxy-éthyle. Les données ci-obtenues démontrent que la vitesse de formation du film est indépendante de l'humidité ambiante, et que l'hydrolyse résiduelle se produit très lentement. La cinétique de la formation du film initial se dépend en premier lieu des vitesses d'évaporation des solvants qui sont présents. Cela suggère un mécanisme du même type de ceux qui sont valables pour les vernis clairs.

Kinetik der Filmbildung von Zinkreichen Alkylsilikatfarben

Zusammenfassung

Bisher wurde die wissenschaftliche Untersuchung der Kinetik der Filmbildung von zinkstaubreichen Alkylsilikatfarben dadurch behindert, dass keine genaue und empfindliche Überwachungsmethode für die im Film vor sich gehenden Veränderungen existierte. Die Anwendung rheologischer Theorie auf den Trocknungsprozess legt die Messung des elastischen Moduls als besonders geeignete Überwachungsmethode nahe, um die während der Filmbildung vorkommenden Veränderungen zu messen. Zwei Techniken dynamisch-mechanischer Messung wurden hierfür angewandt: Benutzung des Torsionspendels, welcher ein Mass für den Scher-Streckungs-Modul gibt, und Benutzung des "Rheovibron", welcher Werte für den Zug-Streckungs-Modul aufzeigt.

Beide Methoden wurden zum Studium der Kinetik der Filmbildung von zinkstaubreichen, zu 80% hydrolysierten Äthylsilikat-, Äthoxyl-Äthylsilikat- und Äthoxyläthylsilikat-Farben, unter verschiedenen Bedingungen angewandt. Die erhaltenen Resultate zeigen, dass die Geschwindigkeit der Filmbildung von der in der umgebenden Lufthülle herrschenden Feuchtigkeit unabhängig ist, ein Anzeichen dafür, dass restliche Hydrolyse sehr langsam vonstatten geht. Die Kinetik der anfänglichen Filmbildung wird in erster Linie durch die Verdampfungszahlen der anwesenden Lösungsmittel bestimmt, was auf den Mechanismus eines durch Verdunstung trocknenden Lackes hinweist.

Introduction

Despite the proliferation of patents, review articles and symposia, the field of alkyl silicate zinc-rich coatings is still clouded by the lack of a basic understanding of the mechanisms involved. This is due partly to the difficulty in defining tests and making accurate measurements of the relevant parameters. For example, the measurement of drying time, which is elementary and routine for most other types of paint, becomes so complex in the case of alkyl silicate zinc-rich coatings that, to date, there is no good method by which it may be determined. There is a need to measure it, however, to provide answers to everyday practical questions, such as: What is the minimum time for placing the coating in service? and: What is the optimum time for topcoating? In addition, for the chemist, drying time is a gauge by which to monitor the response to changes in the formulation.

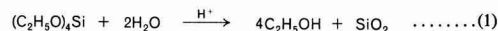
In most other types of zinc-rich coatings, the drying time can be determined with relative ease. For example, if the binder is a thermoplastic polymer, such as a phenoxy resin, the coating will dry by solvent evaporation only, which is a process easy to monitor; complete solvent evaporation can be ensured by baking. In addition, useful information can be obtained by studying the rate of film formation of the clear phenoxy resin solution.

If the binder used is a reactive polymer, such as an epoxy, complete cure can also be ensured by baking or alternatively, the curing properties of films of the clear resin will again provide information applicable to the pigmented coating. This cannot be done with alkyl silicates.

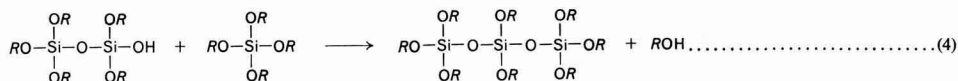
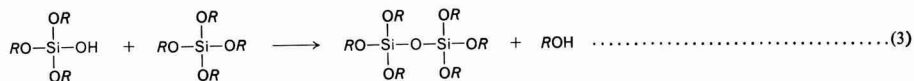
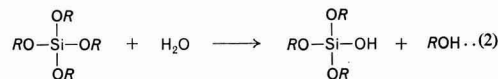
Alkyl silicates

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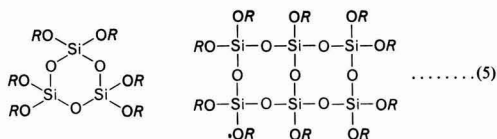
The case of the alkyl silicates is more complex, and is based largely on the fact that in contact with water, hydrolysis of the silicate takes place¹⁻⁷. For example, tetraethyl silicate reacts with the stoichiometric amount of water under conditions of acid catalysis thus:



The most common and commercially important alkyl silicates are those where the organic radicals are ethyl or ethoxyethyl (for example, the Cellosolve products of the Union Carbide Corporation, USA). Although equation (1) appears simple, complications will arise if less than the stoichiometric amount of water is used in the hydrolysis. In such a case, a series of reactions may occur:



These are merely simplifications for the purposes of illustration; in practice, complex mixtures of tri-dimensional structures are likely to coexist:



Partially hydrolysed alkyl silicates, therefore, cannot be identified as a simple chemical species, and are characterised by the concentration of water used in their preparation, expressed as a percentage of the stoichiometric amount needed for full hydrolysis. Table 1 shows the most common types of partially hydrolysed alkyl silicates commercially available. A zinc-rich coating could be prepared simply by mixing an alkyl silicate with zinc dust.

Table 1
Typical partially-hydrolysed alkyl silicates

Type	Alkyl group	Available SiO ₂ (%)	Solvent	Approximate level of hydrolysis (%)
ES-18	CH ₃ -CH ₂ -	18	Ethanol	80-85
ES-40	CH ₃ -CH ₂ -	40	Ethanol	40
MCE	CH ₃ -CH ₂ -	18	Ethanol	80-85
	CH ₃ -CH ₂ O-CH ₂ -CH ₂ -		Ethoxy-ethanol	
X-8018	CH ₃ -CH ₂ O-CH ₂ -CH ₂ -	18	Ethoxy-ethanol	80-85

A study of the film-forming properties of these various products reveals that in order to obtain films which dry in an acceptable time, products pre-hydrolysed to at least 80 per cent must be used. Other products, such as those hydrolysed to about 40 per cent, remain uncured for long periods of time.

A current and widely held theory maintains that the reason why the 80 per cent hydrolysed products provide acceptable drying times is that a smaller number of molecules must react to achieve the 100 per cent hydrolysed condition (SiO₂)_x than for a product pre-hydrolysed to only 40 per cent. Therefore, the time required to complete the cure of a zinc-rich coating based on an 80 per cent hydrolysed alkyl silicate would depend on the kinetics of hydrolysis of the remaining 20 per cent by atmospheric moisture. This represents a small change in a component which is in low concentration in the film; accurate measurements are, therefore, difficult.

Since, according to this mechanism, curing is a moisture-induced process, it is clear that baking is not an appropriate technique to ensure the completeness of the reaction (as it is for the other types of zinc-rich coatings discussed above). The reaction with atmospheric moisture also precludes the study of the kinetics of film formation in clear coatings, because the surface-to-mass ratios of clear films suitable for physical measurement will be several orders of magnitude smaller than those which actually exist in pigmented films.

Finally, the possibility that zinc itself may somehow affect, catalytically or otherwise, the kinetics of hydrolysis should not be disregarded.

It is concluded, therefore, that a valid study of the kinetics of film formation of alkyl silicate zinc-rich coatings should be performed only on the complete formulation.

Methods for the determination of drying time

Refs. 8-13

A wide variety of methods has been developed and used over the years to measure the drying time of coatings. The most elementary, those involving finger touch by different techniques such as tack or print, are not applicable to highly-filled systems, such as zinc-rich coatings, because these do not have the "wet" feel typical of conventional paints.

Existing instrumental techniques⁸⁻¹² include measurements of refractive index and several procedures relying largely on surface conditions, such as the siccometer, the rolling friction method, the Sanderson drying time meter, Gardner circular dry time recorder, Sward rocker, Parks Dry-O-Graph, the Walker-Steele beam, and others. Most of these methods have a fairly narrow range of applicability; the information is rather empirical, and accuracy and reproducibility are not adequate for resolution of the drying process.

This situation has led to the use of some unusual tests for alkyl silicate zinc-rich coatings. For example, the method of "dripping water" has been developed in order to provide a measure of the early stages of drying. It involves simply submitting the freshly applied coating to water dripping from a given height, and determining the time required to render the coating strong enough so that the falling water will not wash it away. Probably the origin of this method lies in the fact that alkyl silicate zinc-rich coatings have the virtue of quickly developing resistance to rain, a major advantage over water-borne alkali silicate zinc-rich coatings, which sometimes remain water-sensitive for up to 24 hours, with the danger of being washed away by an unexpected shower. Rain is actually beneficial for accelerating the hydrolysis of the alkyl silicate binder, and many paint manufacturers recommend the hosing down of coatings a few hours after their application.

Another yardstick to determine a more advanced stage of drying involves hardness measurements, whether by fingernail, a coin, a pencil, or a more sophisticated means, such as the Hoffman or similar tests. The problem here is not only the rather inaccurate nature of the tests, but the fact that the hardness of a zinc-rich coating is dependent on its apparent density. Zinc-rich coatings are porous¹³, but variations in porosity can result from different application techniques, and the rigidity of the porous structure is dependent on the concentration of void space. In addition, practically any physical indentation, however light, in a zinc-rich coating produces compaction of the zinc particles into a shiny metallic surface which does not allow any distinction

between a superficial indentation, a break-through, and even the metallic substrate itself.

Dynamic mechanical testing

Refs. 14-23

Whilst the methods involving mechanical testing described above are destructive, dynamic testing enables the investigator to monitor continuously the changes in mechanical properties of the same specimen by measuring the response of the material to small periodic forces. The response to these forces provides insight into a variety of the changes taking place, such as glass transition, crystallinity, cross-linking, and molecular rearrangements.

Coatings which are in the process of drying are typical examples of viscoelastic materials and, as such, their properties can be described by the Voigt spring-and-dashpot model, shown in Fig. 1. The spring represents the recoverable energy

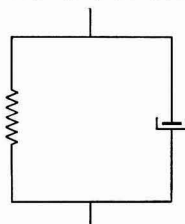


Fig. 1. Voigt element

stored in the material during a deformation, whilst the dashpot represents damping, a measure of the work spent in overcoming viscous forces.

The changes in the mechanical properties of a coating during drying can be represented graphically as shown in Fig. 2: here, qualitative charts show that the elastic modulus increases with time, whilst the damping decreases simultaneously. These two parameters are incorporated in the accompanying series of Voigt elements, which represent the viscoelastic characteristics of the coating at various stages of the drying process. Initially, the major characteristic of the coating is viscous; this is indicated by a large damping (big dashpot) and small elastic component (small spring). As the coating dries, it gains mechanical strength (bigger

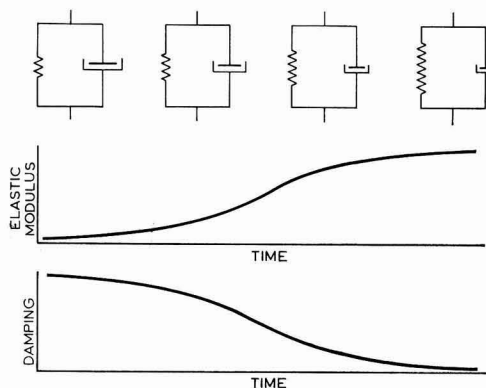


Fig. 2. Viscoelastic changes in a coating during drying

spring), whilst it tends to flow less and less (smaller dashpot).

Alkyl silicates are liquids of very low viscosity, and therefore their initial elastic component is negligible. As the coating dries, the binder undergoes chemical changes, approaching a $(\text{SiO}_2)_x$ structure, which in mechanical terms is a glassy condition characterised by very high elastic modulus. Measurement of the modulus of the coating, therefore, is a particularly appropriate method by which to monitor the kinetics of film formation of alkyl silicate zinc-rich coatings.

Among the many types of instrument which have been developed to determine mechanical dynamic properties¹⁴⁻¹⁹, some are intended primarily for soft solids, others for hard solids, whilst others are designed to measure liquids. However, the torsion pendulum developed by Nielsen²⁰ has proved to offer versatility over a wide range of systems, particularly those changing from liquids to solids either as thermoplastics in solution or chemically curing polymers²¹⁻²³, and this instrument was therefore selected for this study.

Torsion pendulum

Refs. 20, 24

When a torsion pendulum apparatus similar to that described by Nielsen²⁰ is used, an initial distortion of the inertial mass yields a damped sinusoidal wave, from which the damping coefficient and the relative modulus of the specimen can be determined. This arrangement is adequate for structural plastics, for which the instrument was originally intended, but not for coatings. A modified version is shown in Fig. 3, in which the inertial disc is suspended from a counterweight so that the specimen is not subject to tensile stress. The axle of the disc has a clamp which secures the upper end of the specimen, whilst the lower end is held by a clamp attached to a spring loaded shaft, mounted on bearings to minimise losses due to friction.

Testing with this modified instrument is performed by imparting a manual displacement to the disc, which then

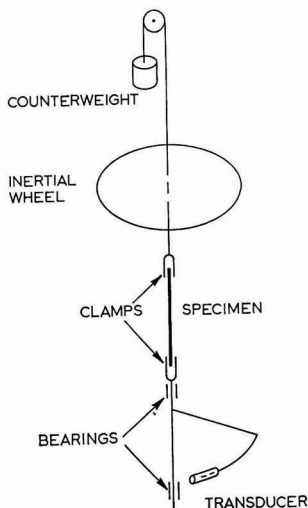


Fig. 3. The torsion pendulum

oscillates, transmitting the cyclic deformation to the lower axle through the specimen. As the oscillation occurs, the lateral angular arm attached to the lower axle swings in and out of a motion transducer, generating a signal which is amplified and recorded in a Sanborn recorder; the tracing produced is similar to that shown in Fig. 4, where amplitude and frequency can easily be measured.

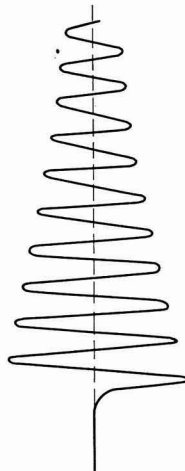


Fig. 4. Typical torsion pendulum output

The apparatus has a chamber surrounding the specimen, so that environmental conditions during testing can be controlled.

The specimen is prepared by dipping a strip of a support material, such as a cellulose blotter, which is rheologically inert, into the freshly-mixed liquid paint. The sample is then attached to the clamps, and measurements are started immediately.

From the data obtained, the mechanical properties of the specimen can be calculated²⁴ by the following analysis:

The equation of motion for free oscillations is:

$$I \frac{d^2 \gamma}{dt^2} + k(G' + iG'') \gamma = 0 \quad (6)$$

where I is the moment of inertia of the system, γ is the angle of oscillation, k a constant related to the geometry of the specimen, G' the shear modulus, and G'' the loss modulus. The solution to equation (6) is:

$$\gamma = \gamma_0 \exp((i\omega - \alpha)t) \quad (7)$$

where α is the attenuation factor, and the frequency ω indicates the vibratory nature of the motion. Substituting (7) in (6) gives:

$$I(\alpha^2 - \omega^2 - 2i\omega\alpha) + ikG' + kG'' = 0 \quad (8)$$

and separating G' and G'' produces:

$$G' = (I/k)(\omega^2 - \alpha^2) \quad (9)$$

$$G'' = 2\alpha I \omega / k \quad (10)$$

which indicates that the shear modulus is proportional to the square of the frequency. Since frequency in cycles per second can be calculated easily from the Sanborn chart, the data obtained are plotted as a relative modulus (cycles per second squared) versus time.

Fig. 5 shows typical curves obtained under average atmospheric conditions (25°C, 50 per cent RH) with zinc-rich coatings formulated with the three types of 80 per cent hydrolysed alkyl silicates shown in Table 1, at a theoretical ratio Zn/SiO_2 of 90/10. The shape of these curves is typical

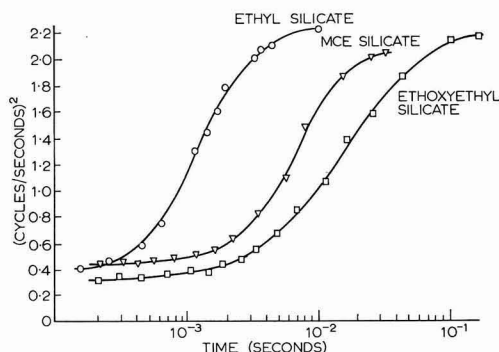


Fig. 5. Torsion pendulum curves of zinc-rich coatings made with various alkyl silicates

of reactive systems. In such cases, an interpretation of the various segments of the curve would indicate that the first inflexion point represents the induction time of the reaction; the slope of the mid-section indicates the rate of reaction (a steeper slope indicating a faster rate), the second inflexion point denotes the end of the reaction, since the ensuing plateau indicates no further increase in modulus. The graph, therefore, serves primarily to determine the time dependence of changes, not absolute values, of modulus.

It is important to note that several of the experiments were continued for extended periods of time, but no increases were found beyond the values obtained overnight. Thus, the data are plotted in semi-logarithmic coordinates up to 10^6 seconds (about 28 hours) to increase the resolution of the inflexion points.

Fig. 5 indicates that ethyl silicate dries fastest, followed by MCE, ethoxyethyl silicate being the slowest to dry. These results might be explained by the retarding effect of steric hindrance of the larger ethoxyethyl group to the approach of the water molecules.

The effect of atmospheric moisture on the rate of modulus increase was studied with an MCE silicate zinc-rich coating. The data plotted in Fig. 6 show the surprising result that the kinetics of film formation are not influenced by the ambient humidity. Because this result was contrary to theories on the curing of alkyl silicates described in the literature, confirmation by another method was sought (see below).

Vibratory measurements

Another way of measuring the properties of materials by dynamic testing is to subject them to a cyclic tensile strain²⁵⁻²⁸.

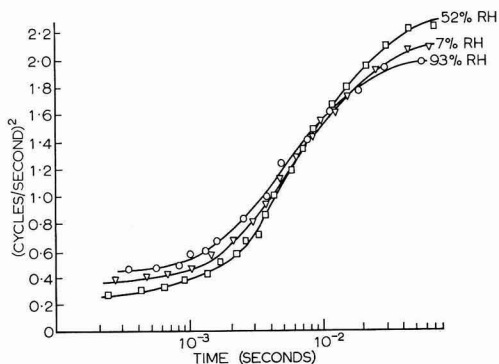


Fig. 6. Torsion pendulum curves of an MCE silicate zinc-rich coating at various humidities

Therefore, the data has the connotation of tensile modulus, rather than, as is the case with the torsion pendulum, of shear modulus.

In the Rheovibron viscoelastometer²⁹⁻³², manufactured by the Toyo Measuring Instruments Co., the principle of operation is based on the fact that if a sinusoidal tensile strain is applied to one end of a specimen of viscoelastic nature, the sinusoidal stress generated at the other end of the sample will be displaced from the strain by a phase angle δ .

The arrangement of the various components of the instrument is shown in Fig. 7. The specimen is held horizontally by two clamps (Fig. 8), one of which is attached to the driver unit providing the oscillatory motion; the other is connected to a load transducer. Outputs from both the strain and the stress transducer are combined to provide direct readings of $\tan \delta$.

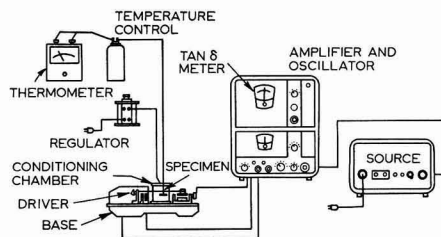


Fig. 7. Rheovibron viscoelastometer

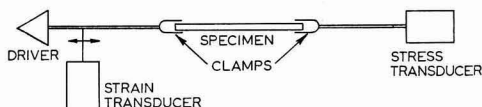


Fig. 8. Specimen disposition in the Rheovibron

The procedure for sample preparation is similar to that described for the torsion pendulum, except that a flexible nylon fabric is preferred as the support medium. Calculations

based on Rheovibron data provide modulus values in dynes/cm² (10^{-1} Pa). The apparatus has a chamber, which enables the operator to control the environmental condition of the test, including the passage of wet or dry air.

The absolute value of the complex tensile modulus E^* is given by:

$$E^* = FL/(\Delta L) \quad \dots\dots\dots(11)$$

where

F = tensile force

A = cross-sectional area of the specimen

L = length of specimen

ΔL = amplitude of elongation

from which E' , the real part of tensile complex modulus E^* , and the imaginary part, or loss modulus, E'' can be calculated by:

$$E' = E^* \cdot \cos \delta \quad \dots\dots\dots(12)$$

$$E'' = E' \cdot \tan \delta \quad \dots\dots\dots(13)$$

Since the computations become laborious, in the author's laboratory the experimental data is fed to an IBM 1130 computer, where a Fortran program calculates the various parameters and automatically generates graphs of E' and E'' versus time on a linear graph. For the present purposes, however, the data was replotted in semilogarithmic coordinates, because it shows more clearly the inflexion points and permits rapid comparisons with the torsion pendulum results.

Fig. 9 shows the data obtained with a zinc-rich coating based on ethyl silicate, at 7, 52 and 93 per cent relative humidities. The shape of the curves shows a remarkable similarity to those obtained with the torsion pendulum, although the inflexion points appear somewhat later, when compared with the corresponding curve in Fig. 5. It is noteworthy and very surprising that the curves obtained at both high and low humidity show a rate of drying faster than the determination made under ambient conditions; this phenomenon will be discussed later.

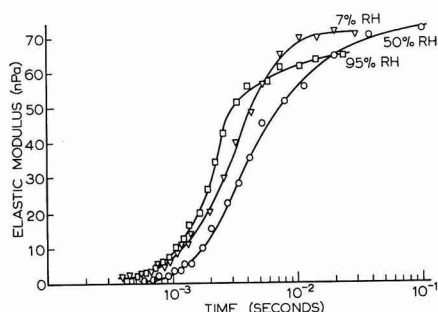


Fig. 9. Rheovibron curves of an ethyl silicate zinc-rich coating at various humidities

Fig. 10 shows values obtained with a zinc-rich primer formulated with MCE silicate. The inflexion points appear somewhat later than for ethyl silicate, confirming the data in Fig. 5. Again, both the 7 and 94 per cent RH curves initially show faster modulus increase than the one corresponding to 50 per cent RH.

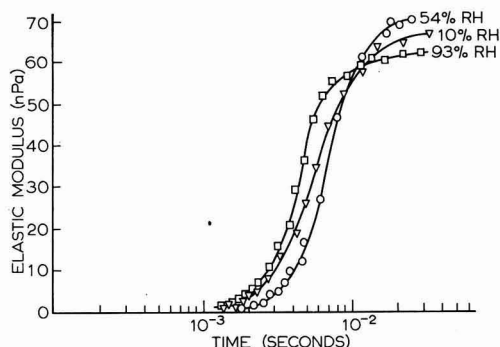


Fig. 10. Rheovibron curves of an MCE silicate zinc-rich coating at various humidities

This pattern is repeated in Fig. 11, showing results with an ethoxyethyl silicate, where there is faster modulus increase at high and low relative humidities than at medium humidity values.

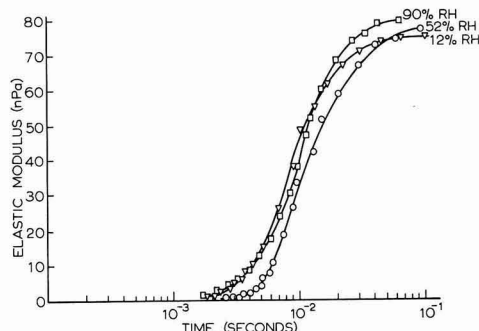


Fig. 11. Rheovibron curves of an ethoxyethyl silicate zinc-rich coating at various humidities

As previously indicated with the torsion pendulum, several of the experiments in the Rheovibron were allowed to age for prolonged periods of time without showing measurable increases in modulus. The data, therefore, are plotted up to only 10^5 seconds to increase resolution and facilitate comparisons.

Discussion and conclusions

Rheovibron data confirmed earlier torsion pendulum findings that the rate of film formation of alkyl silicate zinc-rich primers is not dependent on the humidity level under which the process takes place, within the relative humidity range 7-93 per cent. If the chemical reaction with moisture is not the primary factor, only a lacquer-type process of solvent evaporation can be accountable for film formation. This hypothesis is supported by the fact that the relative position of the curves in Fig. 5 corresponds with the evaporation rate of solvent from the respective silicates. The relative position of the families of curves in Figs. 9, 10 and 11 confirms the data in Fig. 5, but the most compelling support for the theory of film formation through solvent evaporation comes from the rather surprising fact that the Rheovibron curves at both high and low humidity show faster cure than

the rate obtained for ambient, medium humidity. The explanation of this is thought to be that the experiments made at high and low humidity were performed by passing air (either saturated or dried, respectively) through the environmental chamber of the instrument, so aiding the removal of solvent from the sample and accelerating its drying. The experiments made at medium humidity were carried out at the normal ambient atmospheric conditions in the laboratory. The fact that the Rheovibron curves show this effect whilst the torsion pendulum results do not (Fig. 6) is due to the different geometry of the instrument's conditioning chambers. The chamber of the Rheovibron is much smaller than that of the torsion pendulum, resulting in a higher number of chamber volumes changed per unit time.

The theory of film formation by solvent evaporation would also explain why in this type of acid-catalysed, two-component zinc-rich coating, it is necessary that the alkyl silicate be hydrolysed to a relatively high level (75-85 per cent) in order to form dry films, whilst other alkyl silicates, hydrolysed to a medium level (40 per cent) do not form dry films. At about 80 per cent hydrolysis, the silicate polycondensed structure has reached a balance: it is low enough in steric complexity and has sufficient organic radicals still attached to it to provide solubility in alcohols, but the molecules are large enough to form coherent films upon solvent evaporation. The presence of a high concentration of alcohol in the alkyl silicate prevents reaction (1) from proceeding. As the alcohol evaporates from the film, the equilibrium between alcohol and water shifts, so that water may react. This reaction is slow, however, so that it does not influence the initial stages of film formation, which are the most critical from a practical standpoint. This slow rate of hydrolysis explains why a 40 per cent hydrolysed silicate will remain wet indefinitely. If film formation were indeed due to hydrolysis, then it could be expected that the 40 per cent hydrolysed species would react relatively quickly to reach the 80 per cent level, after which it would continue at the same rate as an 80 per cent hydrolysed material. This, of course, is not the case.

The first inflexion points in the modulus curves evidently represent the condition of critical packing of spheres, where particles can no longer flow past one another. From this point on, the increase in modulus is due to a process of diffusion and evaporation, much the same as occurs with a fugitive plasticiser in a thermoplastic film. The rather smooth transitions to a plateau indicate a lowering in the diffusion-evaporation rate caused by solvent depletion.

Acknowledgments

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Prospects for surface coating resins in the European market*

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Summary

The current situation and likely developments in the surface coating industry are reviewed in the first part of the paper. The new trends which are expected to affect this market are illustrated by means of figures and statistics. A review of EEC resin production is made, with special reference to the situation in the UK. The major areas of application for paints have been defined in order to try to give as reliable a picture as possible of the different outlets and their relative importance.

In the second part of the paper, the technical aspects of a number of binders and enamels, such as powder coatings, water soluble resins, polyurethanes and high solids, are discussed. The proper selection of aromatic polybasic acids and anhydrides is shown to be of great importance in obtaining the properties required by these different systems. The conclusion is drawn that important changes are underway in the surface coating industry, despite the slow growth rate of the market for surface coating resins.

Keywords

Raw materials:

binders (resins, etc)

alkyd resin
polyester resin
polyurethane
titanium ester
water-soluble resin

catalysts, accelerators, inhibitors

catalyst

used in the manufacture or synthesis of ingredients for coatings

isophthalic acid
phthalic anhydride
trimellitic anhydride

Types and classes of coatings and allied products

corrosion resistant coating
high build coating
powder coating

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

corrosion resistance
flexibility
yellowing

Les perspectives à l'égard des résines pour revêtements de surface dans le marché européen

Résumé

Dans la première partie de l'exposé on passe en revue la situation actuelle et l'évolution éventuelle de l'industrie des revêtements de surface. On indique au moyen des chiffres et des statistiques les nouvelles tendances qui vont probablement exercer une influence sur ce marché. On donne un aperçu de la production de résines dans la C.E.E., et, en particulier, à la situation dans le Royaume Unie. On a défini les domaines d'utilisation des plus importantes peintures afin que l'on puisse fournir l'impression la plus valable que possible des divers débouchés et de leur importance relative.

Dans la deuxième partie de l'exposé sont discutés les aspects techniques d'un certain nombre de liants, peintures-émaux, résines solubles dans l'eau, résines polyuréthanes, résines "high-build" et revêtements en poudre. On démontre que la sélection rationnelle des acides et des anhydrides aromatiques polybasiques est d'une grande importance pour obtenir les caractéristiques requises de ces différents systèmes. On conclut que malgré le faible taux de croissance du marché des résines pour revêtements de surface, des changements importants sont déjà en train au sein de l'industrie de revêtements de surface.

Die Aussichten für Lackharze im Europäischen Markt

Zusammenfassung

Im ersten Teil der Abhandlung werden die gegenwärtige Situation und die voraussichtlichen Entwicklungen in der Lackindustrie betrachtet. Mittels Zahlenwerk und Statistiken werden die neuen, den Markt voraussichtlich beeinflussenden Trends aufgezeigt. Eine Übersicht der EWG-Harzproduktion wird mit besonderem Bezug auf die Lage im UK gegeben. Die wichtigeren Anwendungsgebiete für Lacke wurden definiert, um zu versuchen, ein so verlässliches, wie mögliches Bild der verschiedenen Absatzmöglichkeiten und ihrer relativen Wichtigkeit zu geben.

Im zweiten Teil der Abhandlung werden technische Gesichtspunkte einer Anzahl von Bindemitteln und Emaillelacken, wie z.B. von Pulverlacken, wasserlöslichen Harzen, Polyurethanen und Lacken mit hohem Festgehalt besprochen. Es wird gezeigt, dass die richtige Wahl aromatischer, mehrbasischer Säuren und Anhydride von grosser Wichtigkeit für die von diesen verschiedenen Systemen geforderten Eigenschaften ist. Es wird geschlossen, dass trotz des langsamen Wachstums des Marktes für Lackkunstharze wichtige Veränderungen in der Anstrichmittelindustrie im Gange sind.

*Paper presented to a meeting of the West Riding Section in Leeds on 11 November 1975.

Introduction

It is difficult at the present time to predict the future of the surface coating industry. The shortage of raw materials and energy, inflation and high interest rates have already badly affected areas such as the automotive industry, where a drop in output of 40 per cent was recorded during the last month of 1974 when compared with the same period in 1973.

The building sector, like the appliance industry, is also ailing and no major change is expected to ease the situation this year. As production and labour costs increase, a greater demand for long-life paints may be expected. Compared with the cost of application, the price of the paint is relatively small and this is why emphasis is likely to be placed on the development of paint films of great durability in order to reduce costly periodic maintenance. Since the petroleum crisis, some effort has also been made to decrease the use of organic solvents.

The growth rate, if any, of the paint industry is likely to be very small. Even during 1976, most areas are not expected to show significant improvements. Most of the statistics resulting from data collected at the beginning of 1974 have had to be revised downwards.

Even if, economically, the overall picture for the coming years looks a little disheartening, some significant changes will occur, which will affect the whole of the surface coating industry.

What are the areas in which these changes will occur?

First, air-drying paints in the trade and "Do-it-yourself" sector and, to a larger extent, those for industrial finishes will adopt water-soluble products. As far as industrial finishes are concerned, major development efforts will be directed towards: powder coatings; water-soluble resins and aqueous dispersions; polyurethanes; and high solids coatings.

The present situation and a prediction for the future will be given in the first part of the paper by means of figures and statistics, and an attempt will be made to define the new trends affecting the surface coating market.

In the second part, the technical aspects of new binders and enamels will be emphasised. How the selection of suitable dicarboxylic aromatic acids and anhydrides can lead to the successful development of such systems will also be discussed.

Market survey

Early in 1974, surface coating resin and enamel producers faced an embarrassing situation due to a severe shortage of raw materials and an upsurge in production costs. Research and development work was in many instances stopped, and effort turned instead to the search for materials which could be substituted for those in short supply.

A market survey was carried out with the co-operation of many of the most important resin and paint producers. This work was also assisted by information obtained from different national organisations. It was obvious from the discussions with people at that time, that much of the industry was conscious of the fact the surface coatings market had reached a turning point and that significant modifications within the different classes of binders were to be expected during the coming years.

Table 1 shows a breakdown of coating resins production in the EEC. Unfortunately, figures for the Scandinavian countries could not be collected in time for inclusion in these statistics.

Table 1
Coating resins production in the EEC*
(tonnes)

	1974	1976 (Forecast)	1980 (Forecast)
Alkyds (solvent borne)	302 000	296 000	272 000
Alkyds (water borne)	7800	15 300	115 000
Acrylics†	15 400	13 900	15 000
Amino resins	40 100	41 700	46 700
Cellulosics	22 000	20 200	18 500
Dispersions	209 200	226 100	263 200
Epoxy resins	Figures not available		
Epoxy resins (water borne)	21 200	18 200	10 500
Oil-free alkyds†	6290	10 270	18 860
Phenolics	12 600	12 500	12 100
Polyurethanes	51 700	59 900	77 300
Unsaturated polyesters (for coatings only)	Figures not available		
Vinyl resins	5800	6010	6340

*Does not include Scandinavian countries

†Including powders and water-soluble resins

Even bearing in mind the economic uncertainty prevalent when these figures were collected, some interesting considerations emerge from a study of this Table.

A significant downward move is forecast for solvent-borne alkyds and, conversely, a steady growth of water-soluble products can be seen. Water-soluble resins are expected to account for at least 30 per cent of all alkyds produced in 1980.

Negative growth rates have also been recorded for water-soluble epoxy (mainly used in electrodeposited primers for the automobile industry) and phenolic resins. Polyurethanes and oil-free alkyds show an interesting progression, most likely linked with the soaring usage of water-soluble resins and powder coatings. Similar statistics are given for the UK in Table 2.

Table 2
Resin production in the UK
(tonnes)

Type	1974	1976 (Forecast)	1980 (Forecast)
Alkyds (solvent borne)	60 400	61 600	67 800
Alkyds (water borne)			
Acrylics*	4300	4000	4200
Amino resins	6300	6500	7600
Cellulosics	3000	2900	3200
Dispersions	46 100	48 500	56 000
Epoxy resins	13 300	14 100	15 800
Epoxy resins (water borne)	4600	4100	2100
Oil free alkyds*	450	700	1400
Phenolics	6600	6400	6400
Polyurethanes	5400	6100	7600
Unsaturated polyesters (for coatings only)	No figures available		
Vinyl resins	820	800	870

*Including powders and water-soluble resins

The different application areas for paints in the EEC were examined and the results are shown in Table 3. Decorative enamels account for the bulk of paint production (expressed here as a percentage). This has been further split up into trade paints (sold to professional painters) and retail paints, which are sold directly to customers for do-it-yourself applications.

Table 3
Application areas for paints in the EEC* (%)

Area of application	1973	1974	1976‡	1980‡
Trade paints	36.85	36.50	36.40	35.60
Retail paints	16.00	16.15	16.55	16.80
Industrial paints†	22.60	22.70	22.80	22.25
Motor vehicles (line paints)	5.90	5.30	4.45	4.40
Motor vehicles (refinishing)	3.05	3.30	3.55	3.75
Coil-coatings	0.85	0.80	0.90	1.10
Powder coatings	0.45	0.60	0.90	1.60
Appliances	1.35	1.30	1.20	1.20
Marine paints	3.10	3.05	3.05	3.20
Wood finishes	9.85	10.30	10.20	10.10
	100.00	100.00	100.00	100.00

*Does not include Scandinavian countries

†Other than motor vehicles, marine appliances, powder and coil coatings

‡Estimated figures for these years

The heading "Industrial enamels" groups together all industrial applications, other than automotive, marine and appliances (coil coating and powder coating are also considered separately).

As expected, the share represented by car enamels will diminish during the next few years if line paints are considered; conversely, the share will increase slightly for car refinishing.

Coil and powder coatings show a constant rate of progression on the total market share, but the final percentages are still small when compared with the other fields. Appliances are likely to lose part of their present position, whilst marine paints and wood finishes will stay almost at the same level. Similar information for the UK is shown in Table 4.

Table 4
Application areas for paints in the UK (%)

Area of application	1973	1974	1976‡	1980‡
Trade paints	37.70	37.65	38.00	37.40
Retail paints	19.80	19.90	19.90	19.60
Industrial paints†	24.20	24.20	24.30	24.00
Motor vehicles (line paints)	6.10	5.60	4.80	4.70
Motor vehicles (refinishing)	4.50	4.75	4.85	5.10
Coil coatings	0.90	0.85	1.05	1.30
Powder coatings	0.30	0.50	0.60	1.65
Appliances	1.00	1.00	1.00	0.95
Marine paints	3.00	3.10	3.00	2.95
Wood finishes	2.50	2.45	2.50	2.35
	100.00	100.00	100.00	100.00

†Other than motor vehicles, marine appliances, powder and coil coatings

‡Estimated figures for these years

Returning to the three most important areas as illustrated in the previous Table (trade, retail and industrial paints), trade paints in 1973 and 1974 (Table 5) do not appear to show any breakthrough by water-borne resins. Although the situation looks as if it might change in 1976 to reach a level of 10 per cent penetration in 1980, this would be to the detriment of solvent-borne alkyds.

Table 5
Breakdown in % of binders used (EEC countries*) in trade paints

	1973	1974	1976‡	1980‡
Solvent-borne resins	66.3	66.2	66.0	56.1
Water-borne resins	Traces	0.1	1.1	10.2
Dispersions (all types)	33.7	33.7	32.9	33.7
	100.0	100.0	100.0	100.0

*Does not include Scandinavian countries.

‡Estimated figures for these years

For retail paints, the trend follows almost the same lines (Table 6).

Table 6
Breakdown in % of binders used (EEC countries*) in retail paints

	1973	1974	1976‡	1980‡
Solvent-borne resins	59.7	59.0	58.2	52.5
Water-borne resins	—	0.7	1.5	7.3
Dispersions (all types)	40.3	40.3	40.3	40.2
	100.0	100.0	100.0	100.0

*Does not include Scandinavian countries.

‡Estimated figures for these years

In the industrial field, however, the picture appears to be different (Table 7). Water-borne resins have successfully penetrated the industrial market and even in 1973, a sizeable market had already developed. Their progression since then has been steady and a market share of approximately 32 per cent is expected in 1980.

Table 7
Breakdown in % of binders used (EEC countries*) in industrial paints

	1973	1974	1976‡	1980‡
Solvent-borne resins	92.5	92.0	83.0	66.0
Water-borne resins	7.5	8.0	16.4	31.5
Dispersions (all types)	—	—	0.6	2.5
	100.0	100.0	100.0	100.0

*Does not include Scandinavian countries.

‡Estimated figures for these years

The majority of the binders currently used consist of medium oil length alkyds, but the recent development of thermosetting dispersions might cut down the share represented by solvent-based products still further.

Finally, a review of the market for powder coatings is given in Table 8. This Table shows the total production of thermosetting powders in the EEC, together with a breakdown (in per cent) of the different types used. It is interesting to note the steady increase in the market share obtained by polyester and acrylic systems at the expense of the epoxy powders. In

order to complete the Table, the production of powder coatings by country (in per cent) is also listed.

Table 8
Thermoset powder coating production in EEC*

	1973	1974	1976‡	1980‡
Total production (tonnes)	12 600	16 400	27 500	54 300
Growth rate/year (%)	—	30.0	33.8	24.4
Breakdown by type (%)				
Epoxy	90.9	87.7	58.4	52.0
Polyesters	6.2	8.6	36.3	40.8
Acrylics	2.9	3.7	5.3	7.2
	100.0	100.0	100.0	100.0
Production by country (%)				
Germany		37.6		
Italy		22.1		
France		16.6		
Benelux countries		14.0		
UK		9.7		
		100.0		

*Does not include Scandinavian countries.

‡Estimated figures for these years

Aromatic acids in powder coatings

The trend in powder coatings, already mentioned, where polyesters are gaining acceptance at the expense of epoxy resins, is not only a result of the economics of using the latter type of resin, but also a consequence of the performance characteristics obtained with the polyesters.

Polyesters based on terephthalic acid or blends of terephthalic and isophthalic acids are polar polymers, which show excellent electrostatic charge and spray characteristics. The addition of trimellitic anhydride allows the formulation of carboxylated polymers, which may be crosslinked with either triglycidyl isocyanurate or epoxy resins. The polymer backbone thus allows a variety of curing mechanisms. The resins contain either free hydroxyl groups, free carboxyl groups or both. The different possibilities are illustrated in Table 9.

The use of terephthalic acid greatly affects the crystalline structure of the polymer, increasing the glass transition temperature T_g , which in turn gives a better storage stability of the powder coating enamel. However, it is generally recommended that both terephthalic and isophthalic acid should be included in the formulation. This improves the flow properties of the enamel and results in a smoother film surface. It is also worthwhile mentioning that ortho-phthalic polymers have considerably lower T_g s than either the isophthalic or terephthalic resins at equivalent molecular weight. This is probably due to the presence of low molecular weight material in the phthalic products, indicated by a large weight percentage of material smaller than 420 nm, as determined by gel permeation chromatography (GPC). Moreover, the increasing branching value of a polymer also causes a large decrease in its glass transition temperature.

The trifunctionality of trimellitic anhydride is often used to obtain reactive systems with triglycidyl isocyanurate. These can be cured in as little as 15 minutes at 180°C. One of the major drawbacks of such a system is that the gelation time is too short and does not allow complete flow-out of the paint film. Nevertheless, all the other properties (apart from a borderline storage stability) have proved to be most acceptable, and trimellitic anhydride is widely used for such applications.

As indicated in Table 9, melamine-crosslinked powder coatings have the disadvantage of liberating water as a by-product of the condensation reaction which takes place on curing.

Sometimes this water evolution has an adverse effect on the appearance of the paint film, at least in film thicknesses greater than 50 μm : a suitable resin for saturated polyesters to be crosslinked with a melamine resin of high T_g is shown in Table 10.

The hot melt viscosity (determined with a Shirley-Ferranti cone-and-plate viscometer at a shear rate of 80.5 sec^{-1}) indicates that terephthalic acid based polymers tend to have very high viscosities, which may hinder the flow. The partial replacement of terephthalic acid by isophthalic acid is expected to improve the flow properties by lowering the viscosity of the polymer.

Table 9
Polyester powder coating systems

Curing system	TGIC	Blocked isocyanate	Melamine	Epoxy	Autocross linkable
Reactive groups	$\begin{array}{c} \text{—COOH} \\ \\ \text{O} \\ \\ \text{—CH—CH}_2 \end{array}$	$\begin{array}{c} \text{—OH} \\ \\ \text{—NCO} \end{array}$	$\begin{array}{c} \text{—OH} \\ \\ \text{—OH} \end{array}$	$\begin{array}{c} \text{—COOH} \\ \\ \text{O} \\ \\ \text{—CH—CH}_2 \end{array}$	$\begin{array}{c} \text{—COOH} \\ \\ \text{—OH} \end{array}$
Acid number	60	<10	10	100	120
OH number		70	50		100
Storage stability	⊕	+	+	+	⊕
Type of reaction	Addition	Addition (Caprolactame ↑)	Condensation (Water ↑)	Addition	Condensation (Water ↑)
Stoving conditions	+	+	⊕	+	0
Flow	⊕	+	+	+	+
Mechanical properties	+	+	+	+	0
Weatherability	+	+	+	0	⊕
Detergent resistance	+	+	⊕	+	0
Heat resistance	+	+	+	⊕	+
Corrosion resistance	+	+	+	+	+

Key: + Excellent; ⊕ Fair; 0 Poor

Table 10
Polyterephthalate resin for powder coatings (resin PC-600)

Water	46
Neopentylglycol	465
Terephthalic acid*	675
Dibutyl tin oxide	6
	1192
Less: Water	192
	1000
Properties:	
Acid number	9.5
Hydroxyl number	50.0
Gardner colour	1.0
T _g (°C)	65.0
Softening point (°C)	108.0
Hot melt viscosity at 120°C	3750 poises

*Amoco TA 33 MP

The development of new amino crosslinkers with high T_g have made possible the formulation of enamels which show excellent storage stability. Fig. 1 illustrates the influence of the T_g of an amino resin upon the maximum permissible percentage of crosslinking agent, when combined with the PC-600 resin (T_g 65°C). A stable powder can then be produced, providing that low molecular weight ends, the percentage of "fines" and the moisture pick-up of the powder, are kept to a reasonably low level. This polyester resin can also be adapted for further crosslinking with blocked isocyanates simply by increasing its hydroxyl value.

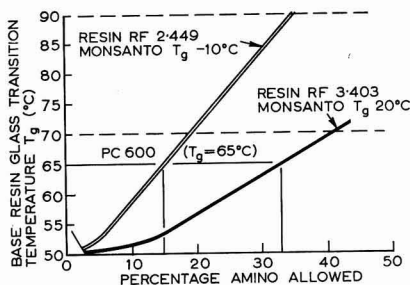


Fig. 1. Maximum allowed melamine percentage to produce a stable powder (T_g = 50°C) whose base resin T_g is known

At present, it seems that the powder coating market is moving towards polyester systems either in combination with epoxies or alone. Without doubt, the best systems in terms of performance are carboxylated polyesters crosslinked with triglycidyl isocyanurate or hydroxyl terminated polyesters combined with blocked isocyanates. Both systems still have some weak points, however, such as limited storage stability and flow on the one hand and the evolution of caprolactame on the other. Acrylic powders have not shown yet the great breakthrough expected of them two or three years ago and, despite some interesting results obtained in the United States in the automotive industry, it would seem that progress is still limited. Indeed people are becoming more concerned about both the toxicity of the acrylic monomer and the technical problems, such as contamination of the equipment and incompatibility with other types of powders leading to "fish-eyes".

Aromatic acids in water-soluble resins

Refs. 1, 2

The development of water-borne systems is expected to bring a major change in the surface coating industry, as has already been indicated in the breakdown of resin production. Aromatic

acids or anhydrides are an important part of the resin and greatly influence such final properties as molecular weight, viscosity, hydrolytic resistance and so forth.

Trials carried out with the neopentyl-glycol esters of several aromatic acids have shown a significant difference in hydrolytic resistance. The behaviour of isophthalic acid compounds is remarkable compared with ortho-phthalic esters, where the rates of hydrolysis were 1 per cent and 8 per cent, respectively. Trimellitic acid was rated at 3.7 per cent. Turpin¹ recently demonstrated that these figures were related to the so-called "anchimeric effect". This effect is described as the intramolecular catalytic action of a neighbouring atom or group on the same molecule, for example, hydroxyl groups or carboxylate ions situated close to the ester link. The anchimeric effect is very important in the case of 1,2-dicarboxylic acid or anhydride.

Water solubility of polymers is usually obtained by processing the resin to a high acid number, the free carboxyl groups then being neutralised with an amine.

Trimellitic anhydride, widely used as a source of free carboxyl groups, has the advantage of giving not only polymers exhibiting excellent water solubility, but also paint films having excellent mechanical properties and corrosion resistance.

A series of salt spray tests has been carried out on water-borne polyesters, all of which employed trimellitic anhydride

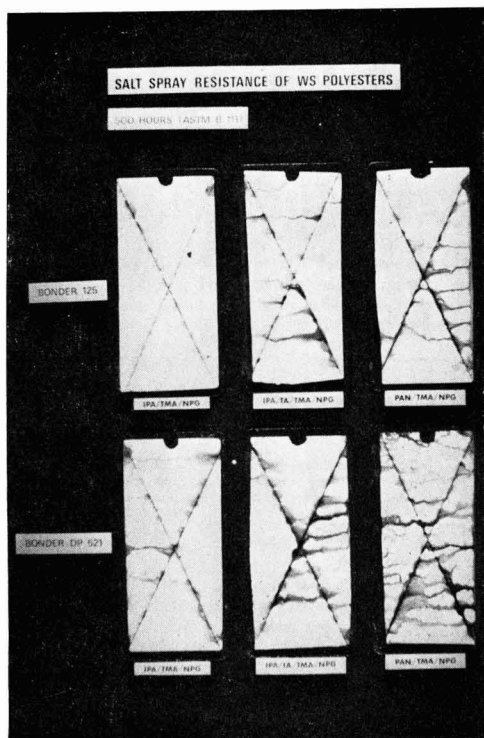


Fig. 2. Salt spray test panels coated with water-borne polyesters, incorporating trimellitic anhydride to promote water solubility and showing the effect of partial replacement of isophthalic by terephthalic acid and total replacement by phthalic anhydride

to promote water solubility. Polyols and flexibilising agents were also similar terms of both type and equivalent weight. The only variable was the replacement of isophthalic acid by terephthalic acid (only a partial replacement for reasons of resin stability) or by phthalic anhydride. It is apparent from Fig. 2 that the best performance was obtained either with the isophthalic acid based formulation or the isophthalic/terephthalic acid blend.

A comparison of these water-soluble polyesters with solvent-borne systems is shown in Table 11. Physical properties have been measured and, apart from a lower Erichsen flexibility, the water-soluble polyesters show almost similar results for hardness, impact resistance and adhesion. Even after a 500 hours salt-spray test, no significant difference could be seen between the two systems.

Table 11
Comparative properties of water-borne and solvent-borne oil free alkyl resins

Cure cycle	Water-borne	Solvent-borne
	20 minutes at 175°C	20 minutes at 175°C
Gloss 60° (%)	93	93
Sward hardness	46	41
Pencil hardness	3 H	2 H
Gardner impact resistance (reverse)	160	160
Erichsen flexibility (mm)	7.2	9
Adhesion (% pass)	100	100
Salt spray* (500 hours)	Pass	Pass

*Bonder 1000—Parker

Water-soluble resins with very fast air-drying properties can be formulated by combining a suitable fatty acid with trimethylol propane and aromatic acids, like isophthalic acid and trimellitic anhydride (Table 12).

Table 12
Fast air-drying, water-soluble resin (resin WS 549)

Linoleic fatty acid*	357
Trimethylol propane	335
Isophthalic acid†	299
Trimellitic anhydride (abbrev. TMA)	99
Less: Water	90
	1000
Properties	
Acid number	50
OH excess (%)	15
NVM (%)	75
Solvent	Propoxy propanol
Gardner Holdt viscosity	Z6
Gardner colour	6

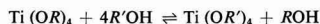
*Pamolyn 200, Hercules Powder Co. Ltd

†Amoco IPA 99

A dust-free time as short as 15 minutes may be easily obtained, with a total drying time of about two hours. When ammonia is used as the neutralising base, a faster drying speed is favoured; on the other hand, it has the disadvantage of forming a coloured complex with cobalt driers, which results in serious yellowing of the paint film and poor storage stability of the enamel. The introduction of a new curing concept, however, has overcome these problems.

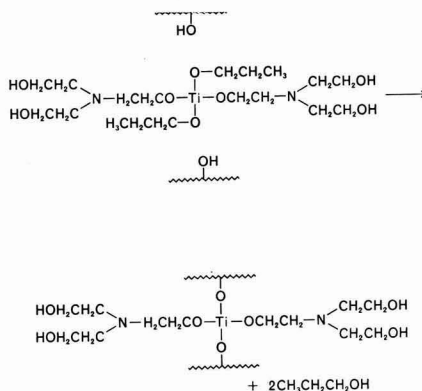
When an organic titanate is added to the polymer, crosslinking can occur through the available —OH groups, even

at room temperature³. The crosslinking depends upon the alcoholysis reaction occurring between the hydroxyl groups of the polymer. It is shown schematically as follows:



Two major problems are encountered with organic titanates: firstly, a very fast hydrolytic decomposition in aqueous media and secondly, a reaction which starts immediately they come into contact with available hydroxyl groups. The storage stability would obviously be adversely affected and there would be no practical use of such a curing mechanism.

Titanium chelates formed from an amino alcohol, for example triethanolamine, are stable, however, at above pH 8 (there is a co-ordinate bonding between titanium and the electron donating atom of nitrogen). Triethanolamine titanate gives the same type of reaction as previously described, but in this instance it is pH dependent (Fig. 3).



(where  represents a polymer which contains hydroxyl groups.)

Fig. 3. Crosslinking of a polymer with an organic titanate

Once the paint has been applied, the neutralising amine evaporates from the film, thus decreasing the pH, and the crosslinking reaction can start. The high initial pH of the paint guarantees good can stability. The final properties of paint films crosslinked in this manner are compared in Table 13 with those of conventional systems, in which driers have been included.

Table 13
Paint film properties (resin WS 549)

	Enamel I (with driers)	Enamel II (with organic titanate)
Drying time (hours):		
Set to touch	0.15	0.15
Tack free	0.45-0.60	0.45-0.60
Dry hard	1.50-2.00	1.50-2.00
Gloss 60° (%)	91	92
Sward hardness	32	24
Pencil hardness	H	HB
Impact resistance	10	80
Adhesion (% pass)	100	100
Yellowness ((A-B)/G) × 100	+8.81	-0.50
Salt spray resistance* (450 hours)	Fail-240 hrs	Pass
Weather-Ometer resistance (500 hours) Gloss 60°	67	76

*Parker, Bonderite 37

The impact resistance, tendency to yellowing, salt spray resistance and weatherability have all been improved to a large extent. The only exception is the surface hardness, which remains slightly below the value obtained with an enamel containing driers. (All mechanical tests were carried out after seven days drying.) The "yellowness" values vary from 0 (for a calibrated standard based on magnesium oxide) to positive values for yellowish surfaces or to negative values for bluish surfaces.

Polyurethane coatings

Considerable quantities of polyurethane coatings are already used in industrial outlets, such as the aircraft industry, marine coatings, railway rolling stock, and also for wood coatings, where they are very popular. According to the most pessimistic market surveys, the production of two-pack polyurethane systems is expected to more than double in the next 10 years.

A combination of saturated polyesters and aliphatic isocyanates generally results in films exhibiting excellent overall properties. The drying rate, however, is rather slow compared with formulations based on aromatic isocyanates. The influence of the use of aromatic acids in the polyester formulation was examined not only in terms of drying properties, but also for mechanical performance, solvent resistance, weathering and corrosion resistance. Three different aromatic acids were studied: phthalic anhydride, isophthalic acid and terephthalic acid, with and without addition of trimellitic anhydride, at three different hydroxyl contents (Table 14).

Resin molecular weights and hot melt viscosities vary broadly with the type of aromatic acid used and the —OH excess, as shown in Figs. 4 and 5.

Enamels based on these resins were examined initially for their drying characteristics, mechanical performance, ageing and corrosion resistance. The enamels based on the terephthalic acid resin gelled in the can 10 minutes after the addition of the isocyanate. This was probably due to a small percentage of esterification catalyst (di-butyl tin oxide) present in the terephthalate polyester.

Drying and through-drying times (Fig. 6) are shorter for isophthalic acid based resins than for phthalic anhydride products at similar —OH excess. The addition of trimellitic anhydride improves still further the speed of drying.

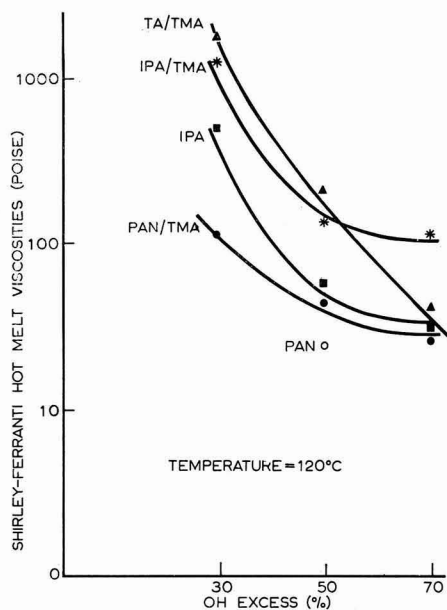


Fig. 4. Variation of viscosity with —OH excess

The final properties of an isocyanate-cured polyester depend on several variables: the structure of the polyester and its degree of branching, the increasing —OH content, the type of polyol, and so forth. Nitrogen containing polyols, such as tris-hydroxy ethyl isocyanurate, also contribute to faster drying rates and better heat resistance. The mechanical properties have been tested 72 hours after application and the results are recorded in Table 15.

It can be seen that the best properties were obtained at low —OH excess, with the isophthalic/trimellitic anhydride combination. Accelerated ageing and corrosion resistance testing completed this evaluation, which again demonstrated the excellent behaviour of the isophthalic acid/trimellitic anhydride based polyurethanes (Figs. 7 and 8).

Table 14
Polyester formulations

Equivalents				Equivalents			
Di-basic aromatic acid	0.60	0.60	0.60	Di-basic aromatic acid	0.80	0.80	0.80
TMA	0.20	0.20	0.20	—			
Adipic acid	0.20	0.20	0.20	Adipic Acid	0.20	0.20	0.20
THEIC	0.35	0.40	0.45	THEIC	0.35	0.40	0.45
NPG	0.95	1.10	1.25	NPG	0.95	1.10	1.25
OH excess (%)	30	50	70	OH excess (%)	30	50	70

Di-basic aromatic acid = Phthalic anhydride, isophthalic acid, or terephthalic acid
 TMA = Trimellitic anhydride
 THEIC = Tris- (2 hydroxyethyl) isocyanurate
 NPG = Neopentyl-glycol

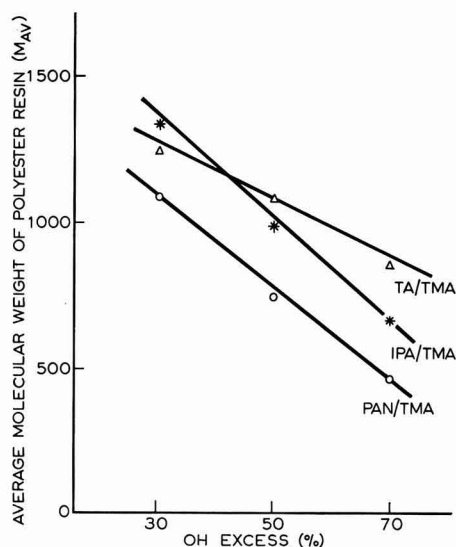


Fig. 5. Variation in molecular weight with —OH excess

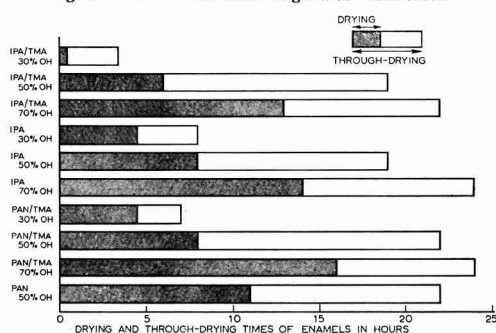


Fig. 6. Variation of touch-dry and through-drying times with —OH content for both isophthalic acid (IPA) and phthalic anhydride (PAN) based products, with and without the addition of trimellitic anhydride (TMA)

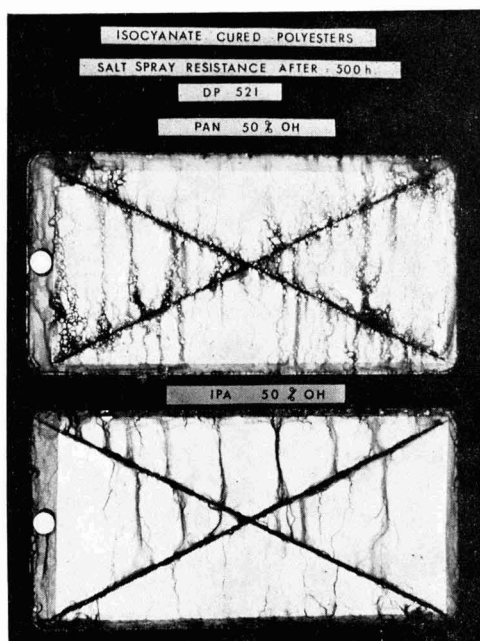


Fig. 7. Accelerated ageing of test panels with a polyurethane coating

High solids

Ref. 3

Water-soluble and high solid enamels are expected to have an important future in industrial coatings. In the United States, the National Paint and Coating Association assumes that 11 per cent of all enamels might be high solids by 1980. On the other hand, water-soluble resins and powder coatings might account for 34 per cent and 7 per cent, respectively. At present, high solids paints do not exceed 1 per cent of the market. In general, the term "high solids" refers to 70-80 per cent volume solid enamels. Enamels of up to 75 per cent

Table 15
Mechanical properties of isocyanate-cured enamels after 72 hours

	Hardness			Erichsen flexibility ¹ (mm)	Cross-hatch adhesion (%) ¹	Gardner impact (rev)		24 hours ageing at 205°C Gardner impact	
	Persoz ¹	Sward ¹	Pencil ¹			¹ CRS	UA ²	¹ CRS	UA ²
IPA/TMA 30% OH	238	30	2H	9.8	100	160	>50	160	>50
IPA/TMA 50% OH	307	49	3H	8	100	160	>50	160	>50
IPA/TMA 70% OH	287	40	3H	7.6	100	50	50	50	<10
IPA 30% OH	241	29	2H	>10	100	160	>50	<10	<10
IPA 50% OH	305	32	3H	7	100	40	50	<10	<10
IPA 70% OH	264	32	2H	9.5	100	100	40	<10	<10
PAN/TMA 30% OH	243	32	2H	8.7	90	90	50	<10	<10
PAN/TMA 50% OH	236	32	2H	7.9	90	10	50	<10	<10
PAN/TMA 70% OH	305	49	2H	6	90	30	50	<10	<10
PAN 50% OH	284	30	2H	7.6	100	40	50	<10	<10

¹Cold rolled steel (CRS). ²Untreated aluminium (UA)

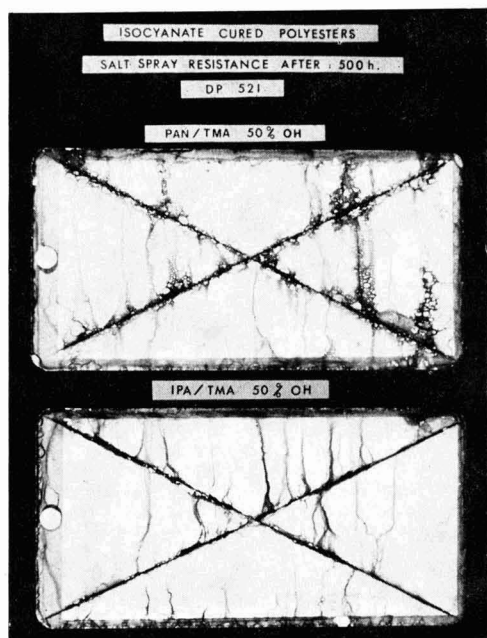


Fig. 8. Corrosion resistance of test panels with a polyurethane coating

solids can be applied at room temperature, whilst above this value it is necessary to heat the paint.

A large fraction of the non-aqueous dispersions by the end of 1976 used in the automotive industry are expected to have been replaced by high solids NADs (in the UK, NADs represent about 10 per cent of the enamels used for car coatings).

High solids allows for a significant reduction in organic solvent emission. Antonelli³ has quoted the following example:

Assuming a coated surface area of 10⁶ square feet and a film thickness of 1 mil. and assuming also that the application efficiency is 100 per cent, the solvent emission from an 80 per cent volume solids enamel shows a drop of some 83 per cent when compared with that from a conventional 40 per cent solids industrial enamel. (See Table 16.)

Table 16
Solvent emission as function of volume solids

Enamel solids content (%)	Total volume (litres)	Volume of solvents (litres)	Reduction in solvent emission (%)
100	2830	—	—
80	3540	707	83.3
70	4070	1220	71.4
40	7070	4250	—

High solids enamels based on alkyds which contain isophthalic acid are currently used in the car industry whenever an excellent performance is needed. Other systems include the high solids polyurethane enamels, where a part of the solvent is replaced by a low viscosity hydroxyl-bearing product, such as 2 ethyl-1-3-hexanediol or 1-4 butanediol. These diluents react with the isocyanate and become a constituent of the paint film.

Further studies are now being carried out to develop the use of high solids in other areas, including an investigation into the possibility, for example, of formulating a suitable water-borne system.

Conclusions

Despite the general slow-down in the economy, large changes are underway in the surface coating industry. Even if the growth in total resin production has come to a standstill, the outlook for some areas is very promising. Water-soluble polymers, powder coatings, polyurethanes and high solids are all expected to achieve major penetration in the coatings market at the expense of conventional products.

Isophthalic acid or terephthalic acid and trimellitic anhydride have been successfully used in each of these four different systems. Their use has resulted in enamels exhibiting such interesting and desirable properties as better mechanical characteristics, improved corrosion and outdoor resistance, and shorter drying times in air-drying resins.

The author firmly believes that more drastic antipollution regulations and the requirement in a number of application areas for higher quality products (together with the need to cut down labour and maintenance costs) will modify the whole pattern of the surface coatings industry in the near future.

[Received 4 December 1975]

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Next month's issue

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

A comparison of the DC and AC methods for measuring the electrical resistance of polymer films by M. Buller, J. E. O. Mayne and D. J. Mills

Autoxidation and antioxidants by D. N. Rampley and J. A. Hasnig

Blast cleaning in inflammable atmospheres by D. W. Singleton

The photo-oxidation of normal primary alcohols by anatase titanium dioxide by A. D. Buss, M. A. Malati and R. Atkinson

The role played by water-soluble polymers in paint performance. Part III Molecular weight and concentration effects by W. C. Arney Jr and J. E. Glass

Paint disfiguration by migration of antioxidant from wall-board adhesive

By D. A. St. John and K. R. Markham

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Summary

Paint "spotting", which occurs sporadically on the face of fibre-reinforced gypsum plaster board linings fixed with adhesive, is shown to be due to migration of the adhesive antioxidant (in this case 2,6-di-*tert*-butyl-4-methyl-phenol) through the board on to

the finished face. The antioxidant residue retards the oxidation of paints or causes yellow coloured spots on water-based paints. It is concluded that for porous lining materials of this type, adhesives should be formulated to prevent migration of antioxidant.

Keywords

Types and classes of structures or surfaces to be coated

plaster board

Raw materials: catalysts, accelerators, inhibitors

antioxidant

retarder

Processes and methods primarily associated with service or utility

adhesive migration

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

discolouration

staining

La décolorisation des peintures provoquée par la migration des antioxydants à partir des adhésifs utilisés pour le posage des plaques de plâtre

Résumé

On a démontré que les mouchetures qui se manifestent par-ci par-là sur la surface d'une peinture appliquée à la face des plaques de plâtre renforcé par fibres est due à la migration, à travers la plaque jusqu'à la surface peinte, de l'antioxydant (2,6-di-*tert*-butyl-4-méthyl-phénol) présent dans l'adhésif qui a été utilisé pour poser les plaques. Les restes de l'antioxydant retardent

l'oxydation des peintures ou ils provoquent la formation des plages jaunâtres sur les surfaces de peintures à base d'eau. On conclut que dans le cas des revêtements poreux en plaques de genre, les adhésifs utilisés devraient être conçus en vue d'empêcher la migration de l'antioxydant.

Anstrichverschmutzung durch Antioxidantienwanderung aus Faserplattenklebstoffen

Zusammenfassung

Es wird gezeigt, dass sporadisch auftretende Fleckenbildung in Anstrichfilmen auf faserverstärktem, mit Klebstoff befestigtem Gipsmörtelplattenfutter die Folge der Wanderung von Antioxidantien aus dem Klebstoff (in diesem Falle 2,6-di-*tert*-butyl-4-Methylphenol) durch die Platte hindurch auf die Filmoberfläche

ist. Der Antioxidantienrückstand verzögert die Oxidation von Anstrichen und verursacht gelbgefärbte Flecken auf wasser emulsions-Farben. Es wird gefolgert, dass die Klebstoffe für poröse Fütterungsmaterialien dieser Art so zusammengesetzt sein müssen, dass die Wanderung von Antioxidantien verhütet wird.

Introduction

Refs. 1, 2

Fibre-reinforced gypsum plaster board, locally known as "fibrous plaster", is mainly restricted to Australasia as an interior lining¹. The common method of fixing is with adhesives rather than by wadding or nailing. In the past, staining of decorative paint systems on this board has been a problem and because of this it has been a standard recommendation² that a solvent-thinned type of sealer be used irrespective of the subsequent paint system. However, since the start of adhesive fixing, a new type of problem became evident, even in cases where the recommended type of sealer was being used. Sporadic cases of paint "spotting" were reported to occur in a pattern which directly related this spotting with the fixing adhesive, and information supplied on the composition of the adhesive indicated that antioxidant in the adhesive could be the cause of spotting.

This paper describes the methods used to detect antioxidant in such paint spots and the experiments used to reproduce paint spotting under laboratory conditions.

Experimental

Description of materials used in construction

Ref. 3

The lining material used was 12mm cast gypsum plaster board reinforced with sisal fibre in case 1 and glass fibre in case 2. The use of sisal is unusual, and glass fibre is now the common reinforcing fibre used. However, the presence of sisal was not considered to be significant. Both types of lining board are fairly porous, each having a voids volume of 30-40 per cent.

The adhesive is applied in patches to the timber framing at 230mm centres and the lining board positioned preferably within 10 to 15 minutes to give adequate wetting. Some support for the board is required for at least 24 hours to allow setting to take place. The composition of the wall board adhesive used consists of a lightly crosslinked polychloroprene with phenolic resin tackifier and filler, together with magnesia, zinc oxide, toluene as solvent and 0.6 per cent by weight of 2,6-di-*tert*-butyl-4-methylphenol as antioxidant.

The adhesive is similar to formulations mentioned in the literature³.

Details of the paint systems used were as follows:

In case 1, one coat of solvent-thinned sealer, one layer of undercoat, followed by two coats of a semi-gloss alkyd resin type paint. The paint was applied by brush two to four weeks after the fixing of the linings.

In case 2, the paint system consisted of only two coats of white PVAc flat paint applied with a roller a few weeks after fixing of the linings.

Description of spotting

Case 1

In the ceilings affected, the paint spotting was sporadic and varied in extent from room to room. In all cases the spots appeared to correspond directly with the points where patches of adhesive had been applied on to the back of the lining. The worst spots were yellowish to brown in colour and the surface of the paint was soft, almost tacky and wrinkled. Other less noticeable spots were visible mainly by specular reflection as a "flash" of higher gloss slightly raised above the surrounding paint. The spots became noticeable within a few days of completion of the painting operation.

Case 2

The ceilings were more uniformly affected than in case 1, in that the spots were more widely distributed. However, the spotting was of a different character, being present as brightly yellow coloured spots superimposed directly over adhesive points. Surface textural effects were absent. This type of spotting was more noticeable than in case 1 because of the amount of colouration involved. It did not appear until at least three months after completion of decoration and increased in intensity over a period of approximately six months. Samples were taken approximately 18 months after completion of painting.

Results

Sampling

Samples were removed from the spotted areas (samples A) and from closely adjacent unaffected areas (samples B) in both case 1 and case 2. In case 2 the more prominent spots had been removed by the painter and only two faintly coloured spots could be sampled—which was barely adequate for analysis. In addition, samples were obtained from spotted paint areas produced in the laboratory as detailed below. These are designated case 3 samples A₃₀ and A₆₀. The adhesive manufacturer supplied samples of the wall-board adhesive and of the antioxidant (2,6-di-*tert*-butyl-4-methyl-phenol), which is incorporated in the adhesive.

Laboratory paint experiments (case 3)

An attempt was made to reproduce the spotting under laboratory conditions for case 1. Samples of fibre reinforced gypsum wall-board were spotted with antioxidant in toluene at concentrations of 30 and 60mg per spot (the average antioxidant content of one adhesive blob was estimated at 60mg). After the spots had been allowed to dry, one coat of white semi-gloss alkyd resin type paint was applied by

roller. It was noted that the spotted areas did not cover well, but no attempt was made to roll out these areas. After a few days it was found that the spotted areas were showing visible textural differences. The spots were similar to many of the less obvious spots noted in case 1, which were more evident by flashing of the surface than by actual wrinkling of the film. The spotted areas were chalky when scratched and this chalky area extended well beyond the boundaries of the original spot of antioxidant, indicating that the antioxidant had been spread out by painting over it. Differences between the two concentrations used were not visible.

Antioxidant detection

Paint samples 1 and 2A, 1 and 2B and 3A₃₀ and 3A₆₀, and the adhesive and the antioxidant were separately extracted at 20°C with petroleum ether (60–80°C bp). The extracts were analysed by thin-layer chromatography (TLC) on silica-gel G with hexane as eluant, and the completed plates were sprayed with a dilute aqueous solution of potassium permanganate.

The antioxidant showed up clearly at R_f 0.9 as a $KMnO_4$ -sensitive spot (white on a pink background) and this same spot was also observed as the only mobile component of the adhesive extract. Paint samples 2A, 3A₃₀ and 3A₆₀ also showed this spot, but sample 1A did not. However, in 1A (and to a lesser extent in 2A, 3A₃₀ and 3A₆₀) two other $KMnO_4$ -sensitive spots were visible at R_f 0.4 and c. 0.3 (smeared), together with one visibly yellow spot at R_f c. 0.15 (smeared). The paint samples 1B and 2B showed no spots at all on TLC.

Oxidised antioxidant detection

In $KMnO_4$ -treated antioxidant: A sample of the antioxidant in petroleum ether was shaken manually with a dilute aqueous solution of $KMnO_4$ for about five minutes and warmed occasionally in a water bath during the course of the oxidation. TLC analysis of the products revealed, in addition to the unchanged antioxidant (at R_f 0.9), $KMnO_4$ -sensitive spots at R_f 0.75 and 0.4 and one visibly yellow spot at R_f 0.16. The spot at R_f 0.4, if left unsprayed overnight, turned yellow. This new yellow compound was again tested by TLC and showed an R_f of 0.5.

In paint sample 1A: Co-chromatography (TLC, SiO_2 /hexane) of the oxidised antioxidant with the petroleum ether extract of paint sample 1A, revealed that both contained the $KMnO_4$ -sensitive compound at R_f 0.4, and its conversion in both cases to the yellow compound (R_f 0.5) confirmed its identity. The yellow, R_f 0.16, spot was present in the paint extract, but it was necessary to isolate this from other low mobility compounds before an accurate chromatographic comparison could be made.

Mass spectrometric identification of the oxidation product

Ref. 4

Samples of the R_f 0.4 compound were obtained from both the oxidised antioxidant and paint sample 1A by chromatography of a band of extract on a silica thin-layer plate. The compound was washed from the plate with diethyl ether. In the course of this isolation, the compound turned slightly yellow. The mass spectra were measured on an AEI-GC MS-30 mass spectrometer by solid insertion using a probe temperature of 80°C.

Although both mass spectra showed the same key ions (see below), the spectrum of the sample from the oxidised antioxidant was cleaner and is quoted here. In this, major ions were present at m/e 436 (100%), 434 (76%) and 219 (76%) but relatively minor ions were also present at m/e 438 (10%), 437 (36%) and 435 (32%). This data is consistent with the sample being the product of oxidative coupling of the antioxidant⁴, that is, a dimer of molecular weight 438 (Fig. 1). However, yellow oxidation products of molecular weight 436 and 434 may also be present, since the sample appeared yellow. Successive losses of one, two, three and four hydrogen radicals from the parent ion would give rise to ions at m/e 437, 436, 435 and 434, represented by the sequence of structures outlined in Fig. 1. The fragment at m/e 219 is accounted for by the expected fission, under electron impact, of the parent molecule at its central bond.

Discussion

There is little doubt from the results obtained that the antioxidant present in the adhesive is responsible for the paint

spotting. In the two dwellings studied (cases 1 and 2), either the antioxidant or its oxidation products were detected in the spots on the paint film. The antioxidant is carried through the porous board by the adhesive solvent (toluene) and is deposited as the solvent evaporates from the lower surface of the ceiling. The deposit so formed can cause spotting by two different mechanisms: with air drying alkyd paints it hinders the drying process, which causes wrinkling or flashing almost immediately, followed by discolouration at a later date; however, with PVAc latex finishes, it appears to migrate into the film and subsequent oxidation produces highly coloured spots after a period of several months.

It is considered that the observations and results reported in this paper confirm the first mechanism, whilst the second mechanism must be considered no more than a tentative suggestion because of the unsatisfactory nature of the samples obtained. It is clear that these types of adhesive must be formulated to prevent migration of the antioxidant, if the problem paint spotting is to be prevented.

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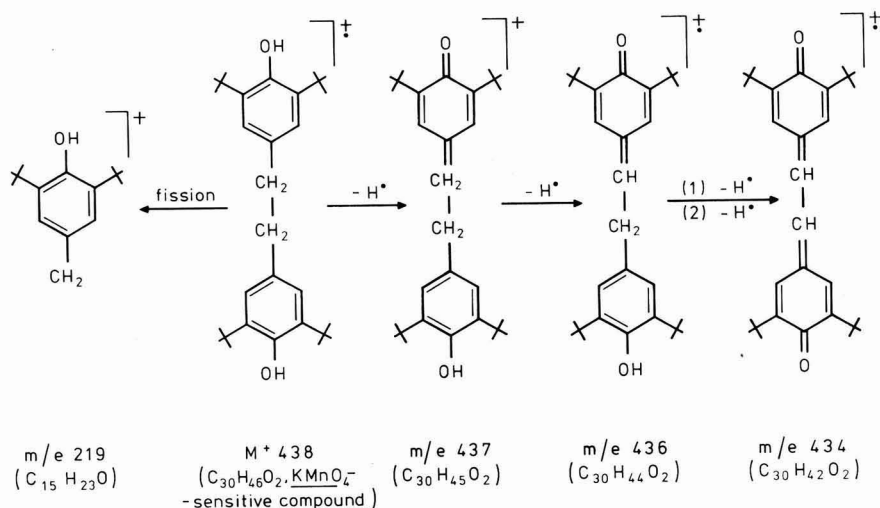


Fig. 1. Proposed structure for the major ions observed in the mass spectrum of the potassium permanganate-sensitive compound, R_f 0.4

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Polyester resins and glass reinforced polyesters, with particular emphasis on chemical plant*

By J. A. Raymond

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Summary

A general description of unsaturated polyester resins and glass-reinforced polyesters (GRPs) is given in which the more important methods of application, properties and markets are outlined.

In the main body of the paper, several polyester resin systems are outlined and their performances as applied to chemical plant are compared in terms of the maximum working temperature of each

system when it is immersed in chemicals representative of the general types (for example: aqueous salt solutions, acidic, alkaline and oxidising environments, alcohols, etc). These maximum working temperatures are derived from a number of sources, including case histories and laboratory tests, in order to make the recommendations meaningful in practical terms.

Keywords

Types and classes of coatings and allied products

glass fibre polyester coating

Raw materials: binders (resins, etc)

polyester resin

Properties, characteristics and conditions

primarily associated with dried or cured films

chemical resistance

solvent resistance

water resistance

Les résines polyesters et les polyesters renforcés de fibre de verre destinés tout particulièrement aux installations de produits chimiques

Résumé

L'auteur donne dans cet article une description générale des résines polyesters non saturées et des polyesters renforcés de fibre de verre (GRP) et en souligne les méthodes d'applications, les propriétés et les débouchés les plus importants.

Dans le corps principal de cette communication, il décrit en détail plusieurs types de résines polyesters et compare leurs performances

dans le domaine des installations de produits chimiques, du point de vue température maximale de fonctionnement pour chaque type, lorsque celui-ci est immergé dans des produits chimiques d'un ordre général (par exemple: solutions aqueuses salines, milieux oxydants, acides et alcalins, alcools etc.). Ces températures maximales de travail sont dérivées d'un certain nombre de sources. Il donne également quelques exemples d'usages courants des résines polyesters et des GRP.

Polyesterharze und glasfaserverstärkte Polyester mit besonderem Augenmerk auf chemische Verarbeitung

Zusammenfassung

Eine allgemeine Beschreibung ungesättigter Polyesterharze und glasfaserverstärkter Polyester wird gegeben, in der die wichtigeren Methoden der Anwendung, die Eigenschaften und die Marktgebiete umrissen werden.

Im Hauptteil der Abhandlung werden mehrere Polyesterharzsysteme kurz beschrieben, und ihr Leistungsverhalten in bezug auf ihre

chemische Verarbeitung verglichen, wobei die Höchsttemperatur jedes Systems untersucht wird, wenn es in Chemikalien eingetaucht wird, die für allgemeine Harten repräsentativ sind (z.B. wässrige Salzlösungen, Verunreinigung durch Säuren, Basen und Oxidationsmittel, Alkohole usw.). Diese Höchstarbeitstemperaturen gehen auf mehrere Quellen einschliesslich spezifische Fälle und Laboratoriumsversuche zurück. Auf diese Weise sollen die Empfehlungen für Anwendung in der Praxis brauchbar gemacht werden.

Introduction

Polyester resins or, more correctly, unsaturated polyester resins, are generally supplied as liquids which can be cured to a solid form by adding small quantities (typically 1 to 4dm³ per kg of resin) of certain activating agents: these may be organic peroxides (initiators) or cobalt compounds (promoters), although frequently cure is effected by just the use of an initiator (commonly called a "catalyst") and the application of heat.

Unsaturated polyesters are thermosetting resins and can be moulded, therefore, into a variety of forms, with or without the use of heat and pressure. Applications are diverse and include surface finishes, shirt buttons, specimen encapsulation and, when mixed with suitable fillers, car body stopper compounds, imitation marble, and self-levelling floors.

By themselves, unsaturated polyester resins are too brittle for large unsupported structures, and require some type of

reinforcement. Glass fibre reinforced polyesters (GRPs or FRPs) make a particularly effective combination, and glass fibres in the form of randomly chopped strand mats (CSMs) and woven rovings (WRs) are readily available.

A simple method of producing a GRP moulding is to coat a mould, previously treated with a release agent, with some accelerated and catalysed resin, pigmented if desired, to form a gel-coat. When this has reached a suitable stage of cure, successive layers of resin and reinforcement are added, each layer being "consolidated" with a brush or roller so that the mat is impregnated completely. Suitable rollers are available to make the wet reinforcement conform to the contours of the mould, and to squeeze out as much of the entrapped air as possible. After it has cured (often overnight for convenience), the moulding may be released. A variation of this process is to spray the resin and chopped glass fibres on to the mould. These methods are widely used in the boat-building industry.

*Paper presented at a joint meeting of the Hull Section of OCCA and Institute of Chemical Engineers on 6 October 1975.

Hollow cylindrical shapes, such as pipes and tanks, can be fabricated by winding glass rovings which are impregnated with resin on to a rotating mandrel. Known as "filament winding" this technique results in products which can be designed to have considerable strength. The manufacture of corrugated roof sheeting is an example of the continuous production of GRP, in which the chopped fibres and resin, combined between two sheets of film, pass through rollers into an oven for curing, and emerge ready for cutting to length. Another process which is often used for producing mouldings, is resin injection into a closed mould already containing the glass reinforcement. A further development is to supply the glass mat preimpregnated with a filled and catalysed resin, sandwiched between film and wound into rolls: this "sheet moulding compound" (SMC) has only to be removed from the protective wrapping (polyethylene film, for example) and hot pressed to form such articles as lorry cabs, car bumpers, filter plates and casings for electrical switchgear.

In the examples already given, GRP is used as a material in its own right, but in many instances it is used to complement more traditional materials. Steel tanks may be lined with GRP to provide insulation from an aggressive environment; alternatively, PVC or "Celmar" linings, sometimes used to contain chemicals less suitable for storage in straight GRP, are backed by GRP to provide the necessary strength. GRP panels are produced in a wide variety of colours for cladding

the exterior of buildings and they provide both protection against the weather and good appearance.

Fig. 1 summarises the distribution of unsaturated polyesters in the UK market in 1974/5.

Typical mechanical properties

Ref. 1

These are summarised in Table 1. GRP has a lower modulus than steel or aluminium alloy and is elastic up to its point of failure. These are both factors which need to be taken into account at the design stage.

The fatigue properties of polyester typically show a 23 per cent retention of strength after 10^6 cycles, compared with 32 per cent for epoxides, 12.5 per cent for aluminium and 8 per cent for titanium. More detailed information concerning GRP under tensile fatigue and creep conditions (in air at 20°C) is given by Howe and Owen¹.

Chemical resistance

Refs. 2-9

This paper is concerned, however, with dealing in greater depth with chemical resistance, a property more sensitive than most to the type of resin used: for this reason, a comparison is made between a selection of "Crystic" resins (Scott Bader Co. Ltd), some of them specially formulated for use in the chemical processing industry. The brief description given in Table 2 includes the heat deflection temperature (HDT) of each resin, which is the temperature at which a cast begins to lose its rigidity². Whilst reinforcing raises the HDT to 200°C or more, the creep of the laminate becomes an appreciable factor, thus making the HDT of the resin alone a more meaningful basis for design data³.

For environments, such as uncontaminated long chain hydrocarbons, in which polyester resins are inert, the maximum working temperature (MWT) at which the GRP material should give satisfactory use is closely related to the HDT. However, in aggressive environments other aspects of the chemical composition are critical, which makes the study of chemical resistance of different polyester resin systems a complex and wide subject. A convenient method of assessment is to compare resins in terms of their MWTs in environments which are representative of various classes of chemicals. It does not follow, however, that in a particular environment, a resin with a higher MWT will provide more long term satisfactory performance than a resin with a lower MWT,

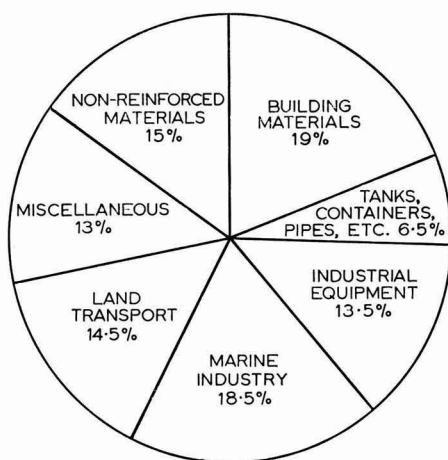


Fig. 1. The UK market for unsaturated polyester resins during 1974/75 (100 per cent = 50 000 tonnes). EEC usage at that time equalled 226 000 tonnes

Table 1
Typical mechanical properties

Material	Fibre (% by weight)	Specific gravity	Ultimate tensile strength (N mm ⁻²)	Tensile modulus (N mm ⁻²)	Specific strength	Specific modulus	Percentage strain at elastic limit
Cast polyester	—	1.2	55	4 000	—	—	2.0
E/glass	—	2.56	2000	77 000	—	—	2.5
Steel HTS 98	—	7.87	1360	200 000	165	25 400	0.2
Aluminium alloy	—	2.75	420	70 000	150	25 000	0.4
CSM laminates	30	1.47	100	7 000	68	4 760	2.0
WR laminates	50	1.67	250	15 000	150	8 980	2.0
Unidirectional roving	70	1.94	700	45 000	360	23 200	2.5
SMC	25	1.81	95	11 000	53	6 100	1.5

Table 2
A selection of polyester resins and their uses

Resin	Description	Cast HDT	Typical application
Crystic 189	Tough orthophthalic	60°C	Boatbuilding
Crystic 198 (thixotropic version Crystic 474)	Highly crosslinked, heat resistant orthophthalic resin	110°C	Chemical plant. Backing resins for "Celmar"
Crystic 199	Highly crosslinked, heat resistant isophthalic resin	130°C	Heat resistant resin
Crystic 272 (thixotropic version Crystic 491)	Tough isophthalic resin	75°C	Filament winding resin for pipes (hand lay-up version-C491). Recommended resins for wines and foodstuffs
Crystic 392	Tough isophthalic, neopentyl glycol resin	88°C	Chemical plant, and backing resin for PVC
Crystic 600 and 600 PA	Tough bisphenol resins	120°C	Aggressive chemical environment, particularly alkaline ones.

when both are used at working temperatures below the MWT of both resins. Table 3 gives values of MWTs at which plant and vessels designed to BS 4994 will operate continuously for several years without serious deterioration³. The surface in contact with the environment should be of the following type:

Greater than 0.125 mm thickness of gelcoat or resin rich surface.

Suitable surface tissue (synthetic for environments which are particularly aggressive to glass).

Chopped strand mat 300 gm⁻² } Resin: glass ratio
Chopped strand mat 450 gm⁻² } 2.5:1 by weight.

There should be no voids or blisters of substantial size in the surface, since these can form sites for blister formation, particularly when immersed in aqueous solutions⁴.

The MWTs are derived from a number of sources. These range from simple screening tests on totally immersed castings and laminates, to case histories, and include "bridging" tests (of varying complexity) in selected environments: for example, one-sided exposure tests under stressed and unstressed conditions. Whilst the comparative performance of the resin types in each of the chosen chemical environments is being discussed, a selection of the test results considered in arriving at the MWT and the way in which these relate to the recommended MWT will be outlined.

The first row in Table 2 shows marked differences between the MWTs of the resins, which are reflected in the appearance of the cast resins shown in Fig. 2. The most significant sign of

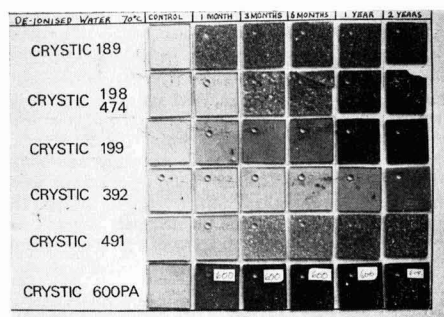


Fig. 2. Castings after total immersion in water at 70°C showing the better resistance of C.392 and C.600 to "disc cracking"

failure in Fig. 2 is considered to be the appearance of disc-shaped cracks rather than discolouration. These are first visible only under a microscope. When reinforcements are present, the process is considerably delayed, but still occurs in resin-rich areas with some laminates⁵. These cracks increase in size and number until they are readily visible with the naked eye; some finally merging to cause complete cracking of the casting. The processes involved are permeation, solution of water soluble material, osmosis, swelling and stress relief by internal cracking (in laminates there is also blistering). Further investigations in the author's laboratories

Table 3
Examples of maximum working temperatures for GRP plant and vessels

Chemical type	Characteristic	Chemical	Maximum working temperatures (°C)					
				C.198 C.474	C.199	Resin C.272 C.491	C.392	C.600 C.600PA
Aqueous	"Neutral"	Water, Salt solutions		55	70	50	75	95
Alkalis		Sodium hydroxide	50%	NR*	NR	NR	55	80
		Ammonia	5%	NR	NR	25	35	60
Bleach		Sodium hypochlorite Cl	14%	NR	NR	NR	60	55
Acids	Mineral	Sulfuric	50%	80	85	60	80	100
		Phosphoric	85%	55	70	50	75	95
	Oxidising	Chromic	5%	45	55	45	75	65
		Nitric	10%	50	50	25	60	60
	Organic	Acetic	60%	NR	35	25	55	65
Inert		Gas/fumes dry	100%	100+	120+	70+	80+	110+
Organics	Long chain hydrocarbon	Lubricating oil	100%	95	100	50	45	90
	Glycol	Ethylene glycol	100%	70	80	55	75	95
	Alcohol	Methanol	100%	35	35	25	30	30
	Aromatic	Toluene	100%	25	30	NR	NR	NR

*NR—Not recommended

have shown that, by suitable treatment, disc cracking can be made reversible in its early stages in one of the Crystic resins. Since osmosis plays an important part in this process, the more concentrated the salt solution of an environment, the lower will be the internal osmotic pressure. However, even a saturated solution of, for example, potassium chloride at 108°C (5.99N) will not balance the osmotic pressure, which requires a solution of 16N potassium chloride⁶. In fact, disc-cracking can be accelerated by doping the liquid resin with water soluble salts⁶. De-ionised or distilled water is clearly one of the most aggressive environments causing this mode of attack, and hence it is recommended that the temperatures given in the first row of Table 2 be reduced by 5°C for distilled water. The MWTs given for salt solutions are, generally, conservative, although allowance should be made for the changes in concentrations, and "washing out" etc, which usually occur in industrial applications. A few salt solutions, such as ammonium and sodium carbonates, are more aggressive towards GRP and have lower MWTs. For drinking water, food applications, and wine storage, tainting of the contents can result in the need for lower MWT. Crystic 491 is a better choice of resin and has been approved for such use because of the low residual styrene concentration after appropriate curing, which results in taint-free storage vessels⁷.

Under alkaline conditions, most isophthalic and orthophthalic resins are attacked, and Fig. 3 shows why Crystic 491 is not recommended for contact with sodium hydroxide



Fig. 3. Crystic 491 laminate after exposure to sodium hydroxide solution

(certainly at concentrations from one per cent upwards). In the new automated Whitbread Brewery at Salmesbury, Lancashire, for example, the tank tops originally made in an isophthalic resin, had to be replaced by Crystic 600PA since the cleaning process involved a 4 per cent sodium hydroxide wash. Glass is also attacked by alkaline solutions, and synthetic surface tissues are recommended in place of glass for surfaces in contact with alkaline fluids. Although the bisphenol resin "Crystic 600" exhibits excellent resistance to sodium hydroxide in the cast form, the MWTs have been reduced to give the values shown in Table 3 for laminated constructions. It should be noted, however, that the rate of attack varies with the concentration of sodium hydroxide, the lower MWT (50°C) being in the 25 per cent (by weight) range, whilst higher MWT can be obtained at higher and lower concentrations⁸.

Bleaching solutions, such as sodium hypochlorite, readily attack most polyester resins, etching 1.5 mm thick castings to wafer thinness in a matter of months. Accelerated tests of this kind highlight the superiority of Crystic 392 and Crystic 600 under these conditions.

In mineral acids, most polyester resins, such as those given in Table 3, are resistant, however, they are sensitive to higher concentrations of sulfuric and nitric acids. Crystic 600, which is one of the most resistant polyester resins available, although offering good resistance to 77 per cent sulfuric acid at 60°C, deteriorates rapidly at concentrations above 90 per cent at ambient temperatures. The flexural retention values in Table 4, however, demonstrate the modest effects on CSM laminates of total immersion in a weaker phosphoric acid based environment.

Table 4
Retention of CSM laminate properties after immersion in 11% orthophosphoric acid and 0.3% butyl cellulose at 50°C

	Resin		
	Crystic 199	Crystic 272	Crystic 600
Flexural strength (Nmm ⁻²) Control	130	137	125
4 months immersion	119	103	114
Retention (%)	92	75	91
Flexural Modulus (Nmm ⁻²) Control	4200	4200	4000
4 months' immersion	4400	3400	4200
Retention (%)	105	80	105

It is when dealing with the highly concentrated mineral acids, the oxidising acids, and particularly the organic acids, that the better resistance of Crystic 392 and Crystic 600 becomes more pronounced (Figs. 4 and 5).

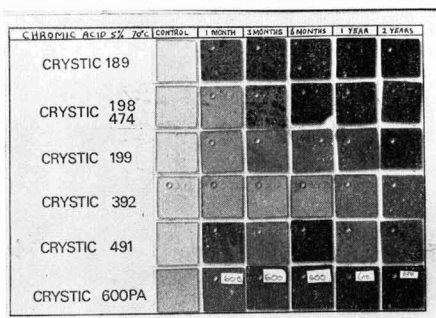


Fig. 4. Castings after total immersion in 5% chromic acid at 70°C

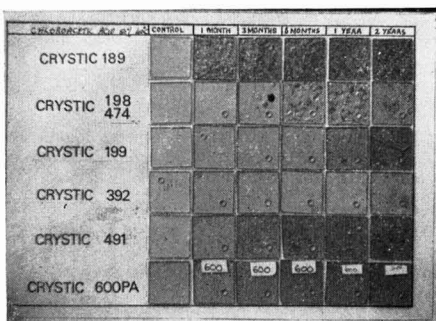


Fig. 5. Castings after total immersion in 50% chloroacetic acid at 40°C

When gases and fumes are involved, it is often possible to tolerate a greater degree of deterioration. Fume stacks continue to perform satisfactorily even when the surface is pitted beyond a state which may not be tolerable for containing liquid chemicals. The MWTs in Table 3 for inert dry fumes and gases are determined, therefore, by the structural limitations imposed by the heat deflection temperatures, as previously discussed. However, when support is provided by other means, higher MWTs are possible (as indicated by the +s in Table 3), and in fact one application involving Crystic 199 has been reported where the temperatures range from 260°C to 315°C. From a structural point of view, it is also worth mentioning that the lower thermal conductivity of GRP compared with metals enables fume stacks to be operated at temperatures higher than might otherwise be expected. GRP exposed to air at 150°C does gradually darken and after six months the surface generally ranges from a golden to a dark brown colour. Laboratory tests have shown that there is a loss of weight, and an embrittlement resulting in 40 to 50 per cent losses in flexural strength when tested at ambient temperature, after a three year exposure at 150°C. This loss is less, however, when tested at elevated temperatures, since the embrittling effect is reduced.

Organic liquids can present a problem to GRP, but generally, those consisting of long chain hydrocarbons with no low molecular weight or low boiling point fractions, are inert with regard to polyester resins. Glycols and alcohols, particularly at the lower molecular weight end of the series, do produce a softening effect which restricts the MWT. In aromatics, such as benzene and toluene, Crystic 392 and Crystic 600 are attacked badly, and are not recommended, therefore, for containing such solvents. However, the highly crosslinked resins perform more satisfactorily and may be used at ambient temperatures⁹.

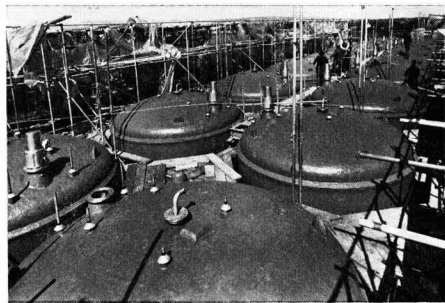


Fig. 6. GRP tank top at Whitbread's new brewery at Salmesbury, Lancashire. Originally made with an isophthalic resin, these have now been replaced with Crystic 600 PA to withstand the caustic wash

To conclude, it is necessary to state that the values recommended for various environments, of which Table 3 forms a brief summary, are periodically placed under review in the light of the latest information generated both from within the author's laboratories and from case histories world-wide.

Future trends

Ref. 10, 11

It is considered that during the next decade, a better appreciation of the mechanism of failure will be one important prerequisite to the development of better resin systems and the more effective use of GRP in chemical plant.

To this end, stress wave emission devices¹⁰ have been used as aids to a fuller appreciation of the mechanisms of internal breakdown. These involve micro- and macro-cracking under stressed conditions, which can then be related to the way GRP behaves in different chemical environments under stressed conditions. The author is concerned with studying polyester resins as they are used in their final form; that is, how the gelcoat behaves when backed up with different laminates and why it behaves in such a manner¹¹. The most important need is to identify those failure mechanisms occurring in the laboratory specimens, which are relevant to chemical plant, and vice versa. For this purpose, information is being collected on chemical plant in great detail. Through the selection of meaningful criteria, critical factors affecting the performance of chemical plant will be identified and future requirements more confidently forecast and met.

Acknowledgments

The author wishes to acknowledge the many workers in this field, particularly Mr L. Cory and Mr B. Stephenson, who over the years at Scott Bader have built up the background data on which this paper is based. A special acknowledgement is due, however, to Mr C. Barrett, the person now directly responsible for this work, who played a major part in arranging the background data into its present form.

[Received 8 March 1976]

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Next month's issue—see page 330

Student Review

The Health and Safety Act*

By D. M. Wilson

"Charnwood", Chapel Lane, South Cockerington, Louth, Lincs.

Keywords

Specifications, standards and regulations
government specifications

Miscellaneous terms
safety

La loi sur la santé et la sécurité au travail—une revue pour stagiaire

Das Gesundheits- und Sicherheitsgesetz—ein Studentenreferat

The purpose of the Health and Safety at Work Act is to provide the legislative framework to promote, stimulate and encourage high standards of health and safety at work. The aim is to promote safety awareness and effective safety organisation and performance, by schemes designed to suit the particular industry or organisation; and by the accumulation of influences and pressures, operating at many levels in a variety of ways. The Act consists of four parts: Part I relates to health, safety and welfare in relation to work; Part II relates to the Employment Medical Advisory Service; Part III amends the law relating to building regulations; Part IV contains a number of miscellaneous and general provisions.

The Act is an enabling measure superimposed over existing health and safety legislation. The existing duties under, for example, the Factories, Offices, Shops and Railway Premises and the Mines and Quarries Acts remain in force, the main provisions of which, for the time being, continue. This makes one comprehensive and integrated system of law to deal with the health and safety of virtually all people at work, and the protection of the public where they may be affected by the activities of people at work. The Health and Safety Commission and the Health and Safety Executive will administer the legislation and become a focus of initiative for all matters relating to health and safety at work. In addition to placing duties of a general nature on employers, manufacturers, employees and the self-employed and others, the Act provides a wide regulation-making power. Part I provides for securing the health, safety and welfare of people at work; protecting other people against risks to health or safety arising from the activities of people at work; controlling the storage and use of dangerous substances and preventing unlawful acquisition, possession and use; and controlling the emission into the atmosphere of noxious or offensive substances from prescribed premises.

All "persons at work", whether employers, employees or the self-employed, will be covered—with the exception of domestic servants in a private household. About five million people, such as those employed in education, medicine, leisure industries and in some parts of the transport industry who have not been covered by previous health and safety legislation, will now be protected for the first time.

The new Act provides for the gradual replacement of existing health and safety requirements by revised and up-

dated provisions, in the form of a system of regulations and approved codes of practice, prepared in consultation with industry to create an integrated body of requirements enforced on a common basis. The new provisions made in regulations and approved codes of practice are designed to maintain or improve the health and safety standards established by existing legislation, the objective being not only to rationalise and update the law, but also to improve the standards of protection which it affords to people at work and the public.

The regulations will be made by the appropriate minister, normally on the basis of proposals submitted by the Health and Safety Commission after consultation with appropriate organisations. Most of the regulations will be made by the Secretary of State for Employment. In the case of regulations for mines, these may be made jointly with the Secretary of State for Energy. Where appropriate, the regulations will be supplemented by approved codes of practice, which will have a special legal status. They will not be statutory requirements, but they may be used in criminal proceedings as evidence that the statutory requirements have been contravened.

Employers must safeguard as far as is reasonably practicable, the health, safety and welfare of the people who work for them. This applies in particular to the provision and maintenance of safe plant and systems of work, and covers all machinery, equipment and appliances used. It is intended that there will be a statutory requirement for the employer of five or more people to prepare a written statement of his general policy, organisation and arrangements for health and safety at work, to keep it up to date by revision and to bring it to the notice of his employees. It is now the duty of employers to provide any necessary information and training in safe practices, including information on legal requirements. Employers will need to consider the specific training needs of their firms with particular reference to processes with special hazards. Employers must, so far as is reasonably practicable, have regard for the health and safety of self-employed or contractors' employees, who may be working close to their own employees, and also for the health and safety of the public, who may be affected by their firm's activities. This covers, for example, the emission of noxious or offensive gases or dusts into the atmosphere or danger from machinery or plant where the public have access.

Designers, manufacturers, importers or suppliers of articles or substances for use at work must ensure that they are

*Based on a lecture "The factory inspector and industry: partnership is preferable to imposition" given by Mr R. V. Souter, of HM Factory Inspectorate, to a meeting of the Hull Section on 3 November 1975.

safe when properly used. They must test articles for safety in use or arrange for this to be done by a competent authority. They must also supply information about the use for which the article was designed and include any conditions of use regarding its safety. Anyone who installs or erects any article for use at work must ensure that it does not constitute a risk and is safe for use.

Employees have a duty under the Act to take reasonable care to avoid injury to themselves or to others by their work activities and to co-operate with employers in meeting statutory requirements. The Act also requires employees not to interfere with or misuse anything provided to protect their health, safety or welfare in compliance with the Act.

If an inspector discovers a contravention of one of the provisions of the existing Acts or regulations or a contravention of a provision of the new Act he can:

(a) Issue a prohibition notice if there is a risk of serious personal injury, to stop the activity giving rise to the risk until the remedial action specified in the notice has been taken. It can be served on the person undertaking the activity or on the person in control of it at the time the notice was served.

(b) Issue an improvement notice if there is a legal contravention of any of the relevant statutory provisions to remedy the fault within a specified time. This notice would be served on the person who is considered to be contravening the legal provision or it can be served on any person on whom responsibilities are placed—he may be an employer, an employed person or a supplier of equipment or materials.

(c) Prosecute any person infringing a relevant statutory provision—instead of, or in addition to, serving a notice. On conviction, the maximum fine for most offences will be £400; imprisonment for up to two years can be imposed for certain offences.

(d) Seize, render harmless or destroy any substance of article that he considers to be the cause of imminent danger or serious personal injury.

[Received 20 November 1975]

Appendix

A one-day symposium of the London Section on the subject "Health and Safety" was held at the Thames Polytechnic, Woolwich, London SE18 on 18 February 1976. A report of the symposium has been prepared by Mr A. J. Newbould, Honorary Publications Secretary of the London Section, and it is published here as an Appendix to Mr Wilson's Student Review.

After an opening address to delegates by Dr B. R. Currell of the Thames Polytechnic, Mr J. Hatto of HM Factory Inspectorate, Health and Safety Executive presented a lecture entitled "The Health and Safety at Work etc Act". (The subject of Mr Hatto's lecture is reviewed in the main body of this paper.)

Mr B. Sykes of Goodlass Wall Ltd in his lecture reviewed the measures taken to implement the requirements of the Health and Safety at Work Act at Goodlass Wall's Liverpool factory. The safety procedures put into operation following a "selected survey" by the Factory Inspectorate were discussed and the way in which some processes had to be changed to enable them to be used safely were described.

Safety audits were regularly undertaken to ensure that no new processes or procedures were being used without full assessment of the health and safety implications. Production workers were made aware of hazardous materials in a batch of paint by a "hazardous" marking on the batch card and had to refer to safety manuals for the appropriate handling and safety precautions. Jobs requiring protective equipment and clothing were readily identified and this was provided. All employees were being trained and retrained constantly to make sure that the general requirements of the Act and the responsibilities placed on the individual to enable him to carry out his job safely were fully understood.

The development laboratories, as well as having to implement safety procedures in their own areas, were also involved in evaluating raw materials for potential hazards and issuing procedures for their safe usage. Only then could materials be ordered for works production. A number of coatings submitted by the development laboratories had been found too hazardous for use at the factory and these were rejected.

Mr K. Safe of Vinyl Products Ltd spoke on "Smoke emission from polymer and paint films". The loss of life which occurred when fire broke out in homes, factories, offices and warehouses had resulted in higher standards for building regulations, with particular regard to a reduction in the flammability of materials of construction. It had recently been established that smoke led to a greater number of deaths than the fire itself, either by asphyxiation or by reducing visibility, effectively causing blindness. The primary concern of the various bodies examining smoke generation had been this loss of visibility and a number of ways had been developed to measure it. The American National Bureau of Standards' Smoke Density Chamber was gaining wider acceptance than the rest, and this device had been used by Vinyl Products to measure the smoke emission of a range of emulsion paint films. Smoke emission was determined by measuring the attenuation of a light beam.

The smoke emission, under both flaming and non-flaming conditions, of various polymer systems was discussed and it was shown how styrene-containing polymers generated more smoke under both conditions than the other types and how the level was related to the styrene content. The 100 per cent acrylics generated more smoke under non-flaming rather than flaming conditions, whereas vinyl acetate polymers showed the reverse trend. Vinyl acetate polymers generated less smoke than other types. Increased levels of pigmentation reduced the emission of smoke and the rate at which it was produced. In the course of the work, it had been found that fire retardant paint films produced a lot of smoke, rapidly creating a danger of death from suffocation rather than burning.

The subject of Dr L. A. O'Neill's paper was "Atmospheric pollution from industrial stoving paints" and described the work carried out at the PRA relating to the requirements under paragraph 5(1) of the Health and Safety at Work Act that "there is a duty to prevent the emission into the atmosphere of noxious or offensive substances and render harmless any that are emitted".

Atmospheric pollution around the world varied from country to country. In the USA the anti-pollution legislation required that eye irritating photochemical smogs were prevented. Germany prohibited the emission of solvents into the atmosphere above a certain concentration depending on the toxicological effects of the particular solvent. In the

UK the main complaints were about the odours from stoving lines caused by breakdown products of the paint binders during stoving.

The PRA's programme had been set out to identify the breakdown products from various stoving paints and to assess their odours and then suggest ways of avoiding the production of these by-products. Paint films were stoved and the gases collected and analysed by gas/liquid chromatography (GLC), infrared and mass spectroscopy and by scatter spectroscopy. The odours were assessed by stoving the films on to aluminium foil in a flask and using a panel of volunteers to grade the smells. By sealing the flask it was possible to retain a record of the odour.

The worse offenders were found to be automotive electrocoat primers, primer surfacers and industrial acrylic paints. Polybutadiene primers gave up to 100 breakdown products, which were identified by GLC. Different binders gave different breakdown products and offensive odours. Acrylics produced free monomers either in the resin or from resin decomposition; epoxy-ester paints saturated and unsaturated aldehydes and alkyd-amino coatings gave formaldehyde and butanol. No compounds of high toxicity were discovered in any of the emissions even though a very careful study was made of the breakdown products.

Reformulation of the paint, a stoving cycle at lower temperatures, catalytic or thermal incineration, scrubbing and reodourising were all suggested as possible ways of eliminating any noxious odours: the relative usefulness of each method was discussed.

Mr W. Rees, BP Chemicals International Ltd, presented a lecture on "The marking and labelling of hazardous chemicals". In recent years, a large number of agreements and some legislation on the transportation of dangerous materials had been introduced, which have overlapped to a certain extent and caused confusion. The labelling and identification, handling and storage of materials had also been covered in a number of ways.

Mr Rees concentrated on the movement of dangerous materials by road, and he explained two of the warning systems used. The "Hazchem" system used in the UK was a composite warning label, which combined legal and voluntary markings: the information given in the statutory diamond symbol indicated the class of hazard (and on tankers the material in each compartment) and was supplemented by an agreed voluntary scheme, which indicated the product carried in all loads, gives guidance on emergency action to be taken and a telephone number for more detailed information.

Deliveries by road between countries were covered by the "Tremcard" system, whereby a card fixed to the vehicle outlined the action to be taken in the event of an accident or spillage. This information was given in the languages of the countries through which the load would be passing. Most materials were described on individual cards, the remaining small percentage being covered by a group or "class" card.

The labelling regulations were due to change in 1976 when the EEC Dangerous Substances Directive became part of UK law. This change would require more detailed information about the risk and safety aspects of the material in the container.

Mr G. Holt of ICI, Paints Division spoke on "The role of the toxicologist in the paint industry". Because of the increasing sophistication of science and technology, it was considered (i) that engineers and scientists had to do more than ever before to ensure the health and safety of process workers and (ii) that the toxicity, handling hazards and precautions and the action to be taken in the case of an accident had to be documented and fully understood by all who were likely to come into contact with them.

A complex, expensive programme of work, including animal feeding and skin tests was needed fully to evaluate potential raw materials and identify any hazardous components.

ICI separated dangerous materials into three classes in their hazardous products manual, based broadly on the speed at which first aid had to be administered. Mr Holt stressed that skin absorption of liquids, inhalation of dusts and vapours and splashes in the eyes were more common causes of injury and poisoning on the factory floor than the more widely held cause of swallowing.

In common with Goodlass Wall, ICI did not purchase any new raw material for use in the factory until the health and safety hazards of the product had been determined and the precautions fully documented. All materials were clearly labelled and where necessary new labels were designed and used.

Dr R. C. Denney of Thames Polytechnic presented a short lecture on "The implications of the Health and Safety at Work Act on university and polytechnic laboratories". The range of laboratories in higher education had to be made safe for postgraduates, visitors, technicians, supervisors and undergraduates—all of differing abilities. Because of this requirement a safety audit was carried out in the laboratories at Thames Polytechnic. A list of the most common hazards were presented and these included:

1. Carcinogens in the laboratory
2. Smoking in the laboratory
3. Eating and drinking in the laboratory
4. Poor eye protection
5. Few laboratory coats worn.

Dr Denney suggested that delegates working in industry should examine how many of these occurred in their laboratories before criticising academics for a complacent attitude to health and safety.

The symposium was concluded with a vote of thanks to the lecturers by Mr A. Hipwood.

[Received 21 May 1976]

Information Received

A/S Norwegian Talc appointed agent for Iberica de Talcos

A/S Norwegian Talc, Bergen, Norway has been appointed world-wide agent for the sale of talc produced by Iberica de Talcos, Leon, Spain. The material is of laminar structure and of high quality and whiteness. Two grades are produced by Bergen for the paint industry: "ST1" (99 per cent passing through a 30µm sieve) and "ST Extra" (98 per cent passing a 10µm sieve). Coarser grades are available direct from Spain, which are well suited for the cosmetic and plastics industries. In addition, a special grade low in copper and manganese is produced for the rubber industry.

BASF "Heliogen" plant on stream

BASF has erected a plant for the production of "Heliogen" pigments at its Ludwigshafen works. The plant, which produces blue and green pigments, has a capacity of 12 000 tonnes a year. Capital investment costs amounted to over DM 50 million. "Heliogen" pigments have a very wide field of application, including the production of printing inks, paints, automobile finishes, and the colouring of plastics.

Berger travel scholarships

This year's Berger "Travelling Scholarships" at the Royal College of Art were presented recently by the Berger Group Director, Mr V. C. Thompson, who is an Ordinary Member of the Association (attached to the Newcastle Section), a former member of Council and was a Vice-President of the Association from 1966 to 1968. The awards were made to three students at RCA, who received cheques for £600 to help them broaden their experience with travel overseas.

Change of address—Arakawa

Arakawa Forest Chemical Industries Ltd, manufacturer of hydrogenated hydrocarbon resins and various other products, has moved to Chicago's "Magnificent Mile". The company's new address is: 625 North Michigan Avenue, Chicago, Illinois 60611, USA.

Common Market Telephone Directory—are paint manufacturers really interested?

The first major attempt to produce a directory, which may be used throughout the member countries of the EEC as a marketing and purchasing tool for exporting and importing companies, has now been published under the title "Common Market Telephone Directory".

Under the heading "Paint", the total of UK companies represented is 42, whereas the Paint Manufacturers and Allied Trades Association alone lists 110 companies as members. As there is no charge for the listing of company names, addresses, telex, and telephone numbers in the directory, it would seem that many manufacturers are either unaware of its existence, or are not concerned that many of the competing organisations in Belgium, Denmark, France, Germany, Ireland, Italy and the Netherlands are already listed.

Printed in five separate editions in the major languages of the EEC, the Common Market Telephone Directory includes details of over 96 000 European companies who either import or export products or services, and compilation of the second

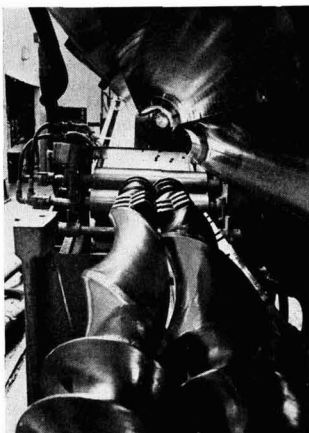
edition, which will be published annually, is now well in hand.

Drynamels expands powder coating production capacity

Drynamels Ltd, a Tube Investments company, is expanding production capacity for thermoset powder coatings with a £0.25 million investment in additional plant and equipment at its Birmingham factory. When completed, the new Drynamels installation will more than double the company's production capacity for powder coatings.

A major feature of the plant is a new twin-screw extruder developed by Baker Perkins Chemical Machinery Ltd, which is the biggest machine of its kind in Britain and capable of 600 kg per hour throughput. It is used to link into a flow-line powder manufacturing system and is said to achieve a new degree of powder quality and consistency.

The new machinery will enable Drynamels to meet the greatly increased demand in the field of powder coatings for domestic appliances and for the automotive industries.



A view of the new thermoset powder coatings extruder installed at Drynamels's Birmingham factory (see above)

Formation of Cadmium Association

The inaugural meeting of the Cadmium Association was held in London on 15 July 1976. Mr Jean-Louis Blain of CR Asturienne des Mines was elected its first Chairman. The new Association will cover all aspects of the use of cadmium, and planned activities include technical publications, environmental work and market surveys. It will form part of the Zinc Development Association/Lead Development Association group with offices at 34 Berkeley Square, London W1.

Instem control system for BTP

Three "Instep Plus" sequencers, manufactured by Istem Ltd., Staffs., are to be used to control a water treatment plant which will provide deionised water for the Grimby titanium pigments plant of BTP Tioxide

Ltd. The order, which with spares is worth over £23 000, was placed with the Industrial Products Group of Instem Ltd by Degremont Laing Ltd, following the successful operation of Instep sequencers in other BTP Tioxide process plants.

Laporte to distribute Solvay products in UK and Eire

Laporte Industries Ltd has announced that arrangements have been concluded for Laporte to market a number of Solvay products in the UK and Eire.

Offshore contracts for ICI

The ICI subsidiary, Imperial Metal Industries Ltd, and the Paints Division of ICI have announced orders worth £300 000 following a recent Offshore Technology Conference in Houston, Texas.

Seamless IMI titanium tubing for high integrity titanium tubular heat exchangers, valued at £212 000, has been ordered from the New Metals Division of IMI for use on the Phillips Norway Group Ekofisk project in the North Sea. The heat exchangers will be used for high pressure direct cooling—an economical alternative to indirect cooling systems which incorporate titanium sheet in plate heat exchangers (an area of business in which IMI is already established).

ICI Paints Division in conjunction with Metallisation Service Ltd has won a contract for the supply of high durability paints for six modules being fabricated for Ninian Field production platforms.

In association with an Aberdeen company Salamis Marine & Industrial Ltd, it has sold protective coatings for 25 000m² of immersed steelwork on the Ninian central platform now under construction at Loch Kishorn in Scotland.

The two Paints Division contracts together are valued at about £100 000.

R. H. Cole investment at Milton Keynes

R. H. Cole Ltd is planning to spend £2.5 million to re-locate and modernise the compounding and colouring operations of Cole Plastics Ltd and East Anglia Plastics Ltd on a new site at Milton Keynes.

The new premises will have a floor area of 10 500m² and an option on an adjacent site has been taken to cover future needs. Loan facilities for the major proportion of the redevelopment cost have been negotiated with the Finance Corporation for Industry Ltd and the Midland Bank Ltd. Assistance in meeting the cost of servicing these loans will be provided by an Interest Relief Grant from the Department of Industry.

Safety award for Burrell & Co

Chemical colour maker, Burrell & Co Ltd, was among the 450 recipients of the 1975 British Safety Council Award.

The award is staged annually to honour and encourage companies who have by their efforts saved lives, resources and profits by reducing their accident rate to below the national average for their industry.

For 1975 the measure of reportable accidents, involving more than three days absence, was 30.3 points. At 14.1, Burrell's return was under half the national average and this is the first time the company has earned the award.

Sonneborn products in the UK

Witco Chemical Corporation of the USA has recently announced new plans for its Sonneborn line of products in the United Kingdom.

Witco Chemical Ltd, a wholly-owned UK subsidiary of the parent company, will be responsible for the marketing, sales and distribution of Sonneborn special purpose petroleum products, which include white oils, natural and synthetic petroleum sulfonates, petrolatums, microcrystalline waxes and rust preventatives.

Witco has established bulk storage facilities for white oil at Birkenhead and will be providing immediate deliveries on an "ex-stock" basis in tankers and drums.

Tioxide Group developments

The Tioxide Group is amongst the world's leaders in the development of titanium pigments and in the design and construction of plants using modern technologies for the manufacture of these pigments.

A large and efficient new plant has been built at Huelva in Spain during the last two and a half years using Tioxide International's "sulfate" process technology. This new plant is being started up by Titanio S.A., a Spanish company owned 55 per cent by Explosivos Rio Tinto S.A., and 45 per cent by Tioxide Group Ltd.

Tioxide uses the newer "chloride" process as well as the "sulfate" process. The Group has been a pioneer in the use, development and exploitation of plasma technology in the manufacture of titanium pigments from titanium tetrachloride and has operated this process on Teesside for a number of years.

The company has now established further major technological advances in the design and operation of these processes and is now seeking planning approval for a major programme of modification and improvement for its Greatham Works "chloride" process plant. Included in this programme will be the replacement of original units for the production of titanium tetrachloride by newly-developed large-scale chlorination plant and the installation of the most recent design of plasma oxidation equipment in place of the existing units. Some other items of re-equipment are also envisaged and will be included in the work, which will be undertaken for the UK company, BTP Tioxide Ltd, by Tioxide International Ltd and by Humphreys & Glasgow Ltd.

New products

Pigments for process printing inks

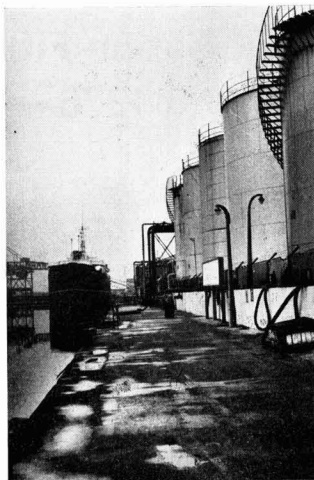
KVK A/S has issued a data sheet describing three new litho pigments:

"ISOL Diaryl Yellow GR"
(C.I. Pigment Yellow 13, C.I. Constitution No. 21100);

"ISOL Bona Rubine 4BK"
(C.I. Pigment Red 57:1, C.I. Constitution No. 15850);

"ISOL Phthalo Blue GB"
(C.I. Pigment Blue 15:3, C.I. Constitution No. 74160).

These pigments are claimed to be ideally suited to multicolour printing and to



Part of the facility at Birkenhead now being used by Witco Chemical Ltd for the bulk storage of white oil in the UK

require only the minimum of processing on a triple-roll mill. Further information in the UK may be obtained from KVK's agent, Charles Tennant & Co. (London) Ltd.

Silicone resin from Wacker-Chemie

Wacker-Chemie GmbH, Munich, has announced a new solvent-free, alkoxy functional silicone intermediate "Silicone Resin SY 231" (previously, trial product VP 2231).

This new intermediate will react with a variety of coating resins, such as polyesters, acrylics, epoxies and other resinous materials containing hydroxyl groups. Silicone polyesters for coil coatings based on this material are claimed to show remarkably fast cure and high surface hardness, whilst still retaining the required flexibility. Formulations for such resins as well as heat resistant polyesters and other modifications are available from Wacker or in England from Bush Beach & Segner Bayley Ltd, Cheshire.

Literature

Colour co-ordination for building

The British Standards Institution has published the first standard for colour co-ordination for buildings, based on the important BRE work of four years ago published as a Draft for Development on colour co-ordination.

BS 5252 "framework for colour co-ordination for building purposes" provides 237 systematically related colours, from which specific ranges may be chosen. At the same time, three new standards actually utilizing the framework are issued: BS 4900, BS 4901 and 4902, giving ranges for vitreous enamel, for plastics and for sheet

and tile flooring. BS 4800 for paint colours already exists.

Floor coverings

Shell Composites Ltd has published a new four-page illustrated colour leaflet describing five different flooring systems and four floor coatings for areas such as warehouses, machine tool shops and industrial assembly plants, where hard-wearing and chemically resistant, easily cleaned surfaces may be required.

Oils and fats statistics

Copies of "World Oils and Fats Statistics 1972/1975", prepared by the Economics and Statistics Department of Unilever Ltd for the International Association of Seed Crushers, June 1976, are now available at £5.00 per copy.

Rheometer brochure

A neat two-colour brochure entitled "Viscosity measurement and recording with Contraves rheometers" is now available, free of charge, from Contraves Industrial Products Ltd.

As well as providing details of the entire range of Contraves laboratory viscometers, the leaflet answers the question, "Why measure viscosity?" and gives information on some of the applications of viscometry. The 16-page booklet is copiously illustrated and provides details of all the different measuring systems, accessories and services offered by the company.

Included with each brochure is information describing the recently introduced Rheomat Programmer, which greatly extends the versatility of the Rheomat 30 instrument.

Conferences, courses etc

Colour Education Symposium

The Colour Group of Great Britain Ltd will hold a symposium with the title "Colour Education" at the Normandy Hotel, Renfrew, Scotland on 1 and 2 October 1976. Further details and application forms are available from Dr A. R. Hill, at Glasgow College of Technology.

From powder to print

The Annual Lecture of the Technical Training Board of the British Printing Ink & Roller Making Industries will be presented by Dr W. Carr at the Royal Institution, Albemarle Street, London, W1 on 11 October 1976. The title of Dr Carr's talk will be "From powder to print—a pigment's progress". Tickets are available at no charge from The Society of British Printing Ink Manufacturers.

SLF Congress

The 8th Congress of the Scandinavian Federation of Paint and Varnish Technologists will be held at Hotel Kalastajatorppa, Helsinki, Finland from 29 September to 1 October 1976. Enquiries should be addressed to Box 52, 01301 Vantaa 30, Finland.

Paint Research Association Fiftieth Anniversary

1926-1976

The Paint Research Association this year celebrates its fiftieth anniversary. The Oil and Colour Chemists' Association has paid tribute to the Paint RA on this achievement by presenting a Congratulatory Address—see Report of Council Meeting on page 347 of this issue.

The Paint Research Association has produced a commemorative booklet based on the book "The first half century—a history of the PRA" by Dr S. H. Bell and we are pleased to publish the following selected extracts (by kind permission of the Director, Dr G. de W. Anderson) in recognition of the anniversary:

Our close connections with OCCA have continued throughout our history. Paint RA staff have served in their Sections, Council and Committees, and given lectures at home and abroad, with many of our "open" papers in JOCCA. The Paint RA has provided five OCCA Presidents from past or current staff—Dr G. F. New (1936-38), Dr L. A. Jordan (1947-49), Mr P. J. Gay (1961-63), Dr S. H. Bell (1967-69) and the present President, Mr A. T. S. Rudram.

The Beginning

Immediate corrective measures do not always follow the recognition of serious deficiencies, national, industrial or personal, but urgent action was taken by Government towards curing the technical and industrial deficiencies starkly revealed by the cataclysmic first World War in an age of rapidly increasing scientific knowledge. Amid the stress of war there emerged the idea of co-operation among companies, industry by industry, for research with Government backing, with grants from the "million pound fund" equal to the collective industrial contribution paid through the newly-formed Department of Scientific and Industrial Research.

Not all industries were sufficiently closely knit for immediate co-operative action, but twelve "Research Associations" were formed between 1918 and 1920 and we were sixteenth in 1926. The initial impetus in our industry came from men who were "concerned to put some scientific muscle into the body of empirical and craft technology of a long-established industry". Many industrialists were involved in the preparatory discussions, but the undoubted leader was Mr S. K. Thornley. At a meeting in April 1926 he proposed the formal resolution to establish our RA, significantly and strongly supported by Mr A. Selby Wood, President of the National Paint Federation. On the technical side, the records emphasise the prominent parts played by Dr H. H. Morgan and Mr C. A. Klein. Both were leaders in the Oil and Colour Chemists' Association (Presidents in 1924-26 and 1926-28 respectively) which had been formed in 1918 indicative of the post-war urge towards less "trade secrets" and more co-operating in the application of science in our industry.

Early Growth

In mid-1929 there was an appeal for increased industrial income; many more were to come. Plans were prepared for a major building extension along the Waldegrave Road frontage; the foundation stone was laid in March 1930 and the building was officially opened in November 1930—an early example of rapid movement. By that time the total income had become £11 500 (still with half from Government), floor space increased to 13 000 sq ft—Membership 156 and number of graduates 10. To quote from the record: "It was felt that the research concept for the industry was safe. Nursery days were over, but unfortunately the world depression was imminent."

Post-depression Advance

In 1933 came the first Member research contract at £500 pa, in those days a major amount, for which 2 post-graduates were recruited; it continued for 5 years. The aim was to improve the outdoor performance of zinc pigments, particularly lithopone, including studies of hiding power and dispersion—effectively the beginning of our school of research on pigment/medium relationships. It was soon to be followed by others on assessment of hydronaphthalene solvents, "molecular-oils" (stand oils treated in a molecular still) and "glue in resins" for which staff was seconded from Member companies. Such contracts with Members, frequently qualifying for Government grant and those directly sponsored by Government Departments, continue to the present.

The Second World War

The intensive war-time laboratory work and associated larger-scale field work was almost exclusively under contract with the Services, Ministries and Departments—the records show 800 specific items requiring experimental work. They included camouflage of buildings, airfields and ships, and areas of concrete, water, glass and grass. The RA became the standardisation and certifying authority for many camouflage paints.

We were also active in the industry's efficiency campaign. Members were kept promptly informed by some 114 war-time Research Memoranda.

Post-war

Relief doubtless played its part but our income from industry doubled to £24 000 and in July 1946 we returned successfully to the DSIR, having exceeded the maximum grant-earning industrial income. By the time of our 21st Birthday celebration in September 1947 the total annual income had reached £48 000 with 238 Members; 24 graduates including two liaison officers for visits to Members; and the additional buildings on the rear of the site had increased the floor space to 26 500 sq ft.

The Middle Years

The major researches had been directed towards a thorough understanding of materials and processes, but as a basis for improvement and background for a growing technical enquiry service. The war had demanded rapid solution of practical

problems, and immediately after the war various new materials received practical appraisal, including rutile titanium oxide (sponsored by a Member). Also, this was a period of great attention to developments in USA. The Director reported in 1948 and 1951 on extensive tours in two "American Journey" Bulletins covering the US paint industry in war-time, industrial research at the Mellon and Battelle Institutes, instrumentation, corrosion and motor vehicle finishing and "new" materials including "latex" paints and organosols.

Widening Horizons

During the latter part of his service Dr Jordan was keenly involved in the planning and erection of a substantial building extension to rehouse the rapidly growing library facilities and provide additional chemical and microbiological laboratories increasing the total floor space to 35 000 sq ft. It was opened on 20 September 1960 with due ceremony by Mr E. B. Calvert, Chairman of the Paint Manufacturers Joint Executive Council in the presence of Dr Jordan and the new Director, Dr L. M. Valentine who took up his duties in April 1960. It was called the Jordan Laboratory "in appreciation of his long service and his establishing an organism (not just an organisation) with a character of its own".

Dr Valentine soon completed the transfer of organic chemistry and microbiology to the new building, whilst rearranging the physics, surface chemistry and technology activities in the old buildings, followed by the addition of instrument rooms and other improvements in the main technical laboratory. He was also keen to continue the tradition of applying the latest research methods to paint problems with adequate equipment.

Modern Times

Dr G. de W. Anderson took up his duties as Director in April 1968. In his address to the Annual General Meeting in November he surveyed the first fifty years of the RA Movement from 1918 stressing the inventive achievements of various RAs and the increasing need for obtaining best value in increased industrial profitability from that expensive commodity called "research". There was no doubt of his energetic desire to ensure that "industrial research is to make money", reiterated in his later Scandinavian address with the challenging title "Don't Waste Your Money on Research", and to give the RA itself a strong business-like image.**

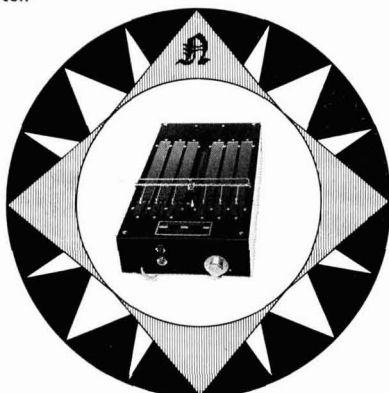
At the end of the booklet Dr Anderson, commenting on the future, states:

In its ideas and internal research programme a research institute must anticipate client needs; consequently, work is already well advanced towards many of tomorrow's major targets. The first fifty years at the Paint RA produced many valuable achievements. We are now poised to serve Members and clients for the next century.

*Published in a shortened form in the Journal during 1974 (JOCCA, 1974, 57, 293)

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The Health and Safety Act

A one day symposium was held at Thames Polytechnic, Woolwich on 18 February 1976 with the theme "Health and safety". Further details are published as an appendix to the Student Review on page 339 of this issue.

Works visit

A party of 13 Members visited the Motor Insurance Repair Research Centre at Thatcham, Berks., on 10 June 1976.

An introduction was presented by Mr B. F. Rand, the Public Relations Officer, who explained that the Centre had been set up following the McKinsey Report seven years ago into insurance claims. The investigation had found that insurance companies were not sufficiently cost conscious when settling claims and that repairs were not necessarily being carried out economically. The Centre, financed by the insurance companies, was set up to examine repair research and development. The Centre did not carry out repairs itself but passed on its findings to the trade. From the results of works study analyses on new and crashed cars, several manuals had been prepared listing standard times for any repair job on a wide range of models. These were used by the trade and insurance assessors in agreeing repair costs. The Centre worked in close cooperation with motor manufacturers and was able to work on new models before they were released. In this way it was able to have repair manuals available for the trade as soon as the new model was released to the public. Occasionally, the Centre was able to influence the design so that repairability of particular parts could be improved. The Centre considered that improving repairability would make the major contribution to reducing insurance costs. To further its aims, it was building its own crash track to ensure an adequate supply of damaged cars. Spare parts was another area where the Centre was involved.

The Centre had evaluated all the refinishing paint systems on the market and had found variations both in the time taken to apply the coatings and in the finish obtained. Final shades from different suppliers were found often to be dissimilar and the Centre was able to take up these points with the paint manufacturers.

The party was then shown around the workshops and saw demonstrations of the latest cutting, shaping and sanding pneumatic tools and welding gear. A tour of the spray booths, equipped with various spray systems, and ovens big enough to take a whole car, completed the visit to the Centre.

A.J.N.

European Liaison Lecture—Protective systems for structural steel

The annual European Liaison Lecture of the Section was presented in March this year in Copenhagen to the Danish Varnish and Paint Chemists' Association and the Malmö Section of the Swedish Paint and Varnish Technologists' Association. Mr E. A. Watson, an Ordinary Member attached to the Newcastle Section, presented a paper on behalf of the London Section entitled "Modern paint coatings in theory and practice", which concentrated on the developments in protective systems for structural steel. The high cost to the community of metal corrosion (£1365 million per annum in the UK alone) had created the urgent need for cooperation in such areas as structural design, substrates, surface preparation, coatings and the environment, to ensure that this figure was reduced.

An efficient anti-corrosive coating needed to (i) contain an active corrosion inhibitor, (ii) be capable of high film build as a barrier to water permeation, and (iii) be based on a resin system which prevented water/ion movement through the film.

The importance of correct surface preparation was stressed. Structural steel was becoming more commonly cleaned by grit blasting in the UK, with chemical treatment and flame cleaning still being used. It was shown how the blast profile and surface cleanliness influenced the corrosion rate: film build had to be sufficiently deep to cover the surface peaks, and retained salts on cleaned steel had been found to draw moisture through the film and cause premature failure.

To protect freshly blast-cleaned surfaces, a blast or 'holding' primer was required. Such a coating needed to have a short surface setting time, to be hard, to be capable of welding through without hazard or effect on weld strength and to have up to six months corrosion resistance before overcoating. Low volume solids polyvinyl butyral resins pigmented with zinc phosphate or zinc chromate/iron oxide and two-pack epoxy resins pigmented with zinc flake or phosphate pigments met all these requirements.



European Liaison Lecture guest speaker, Mr E. A. Watson (left) is welcomed on behalf of the SLF and the Danish Varnish and Paint Chemists' Association by Mr H. Meyer

Where steel was cleaned by methods other than blasting, for example wire brushing or mechanical means, more conventional primers were required, often applied at up to 75 micrometres in one pass. The use of lead-based, chromate, phosphate and molybdate anti-corrosive pigments in the various alkyd and oleoresinous primer resin systems was discussed in detail. Phosphate pigments gave acceptable resistance at low toxicity and could be used in all types of media. The optimum pigment concentration, however, had still to be determined.

Build coats and finishes applied over the primers afforded complete protection to the substrate. Since application costs were often higher than paint manufacturing costs, the maximum film build from the minimum number of coats was most desirable. The choice of coating depended on the environmental requirements and on the primer used. Oleoresinous, oil-modified alkyd, chlorinated rubber, and two-pack epoxy and polyurethane were suitable media when pigmented with micaceous iron oxide (MIO) or with titanium dioxide and extenders. MIO was widely used, although high concentrations were required otherwise resistance properties were reduced. The different properties and applications of each resin type were discussed in detail.

In conclusion, Mr Watson said that there would be future demands for coatings with improved handling, safety and hygiene features and for improved application and drying properties. Higher build coatings with reduced application costs were still being sought.

A.J.N.

OCCA-29 Exhibition

22-25 March 1977 at Alexandra Palace, London

The continuous dialogue between suppliers and manufacturers

Closing date for applications for stand space: 1 OCTOBER 1976

Many enquiries received from home and overseas



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

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

Motif of the Exhibition

The motif, designed by Robert Hamblin, uses red arrows to symbolise how exhibitors and visitors are drawn from all points of the compass to the exhibition. The heads of the red arrows form white arrows in the opposite direction showing the subsequent spreading of knowledge of technical advancements from this unique annual focal point for the surface coatings industries.

Invitation to Exhibit

Copies of the Invitation to Exhibit have been despatched to companies and organisations in the United Kingdom and overseas which have shown at previous OCCA exhibitions or have requested information for the first time for the 1977 exhibition. Completed application forms for stand space must be returned to the Director & Secretary of the Association not later than Friday 1 October 1976.

The Exhibition Committee was particularly pleased to see the number of overseas companies showing at OCCA-28, either directly or through their British associates, as this emphasises the international character of the function.

Any organisation which has not previously exhibited and wishes to obtain an Invitation to Exhibit should contact the Association's offices immediately.

Facilities at Alexandra Palace

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

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

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

In pursuance of this idea, the Committee has decided to amend the regulation which has previously precluded exhibitors from offering alcoholic refreshments to visitors on their stands. The Committee feels certain that the relaxation of this rule will be welcomed by exhibitors and visitors alike.

"Official Guide"

This unique publication will contain descriptions of all exhibits and advertising space is available both to exhibitors and those organisations not able to show at the 1977 Exhibition. The "Official Guide" will be published at the end of January 1977, so that intending visitors can obtain copies and plan their itineraries.

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

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

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

For the last two exhibitions, it was decided that a small charge should be made both for the "Official Guide" and for season admission tickets to the exhibition. This policy undoubtedly deterred casual visitors who might otherwise be attracted to exhibitions for which no admission charge was made and who gathered quantities of technical literature from the stands. The innovation was welcomed by many exhibitors and in no way acted as a deterrent to visitors to this exhibition. A similar charge will be made for the "Official Guide" to OCCA-29.

Information in foreign languages

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

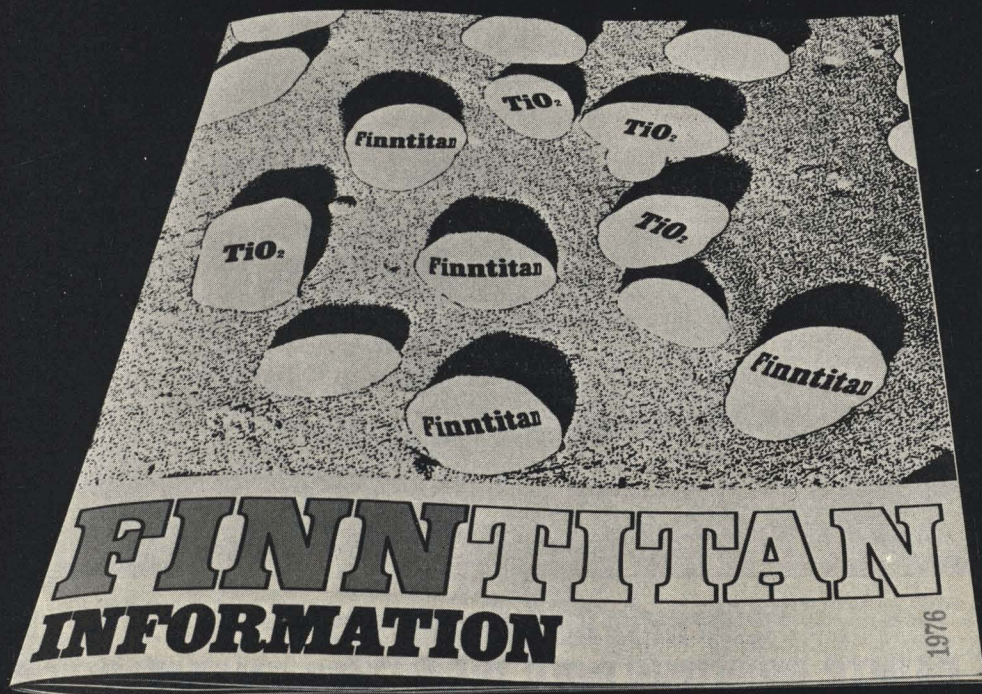
Arrangements for overseas Members visiting the Exhibition

Members attached to the six Sections overseas and those in the General Overseas Section resident outside the continent of Europe, who are planning to visit the Exhibition, are reminded that upon application to the Director & Secretary, the "Official Guide" and admission ticket will be sent to await their arrival at their temporary United Kingdom address.

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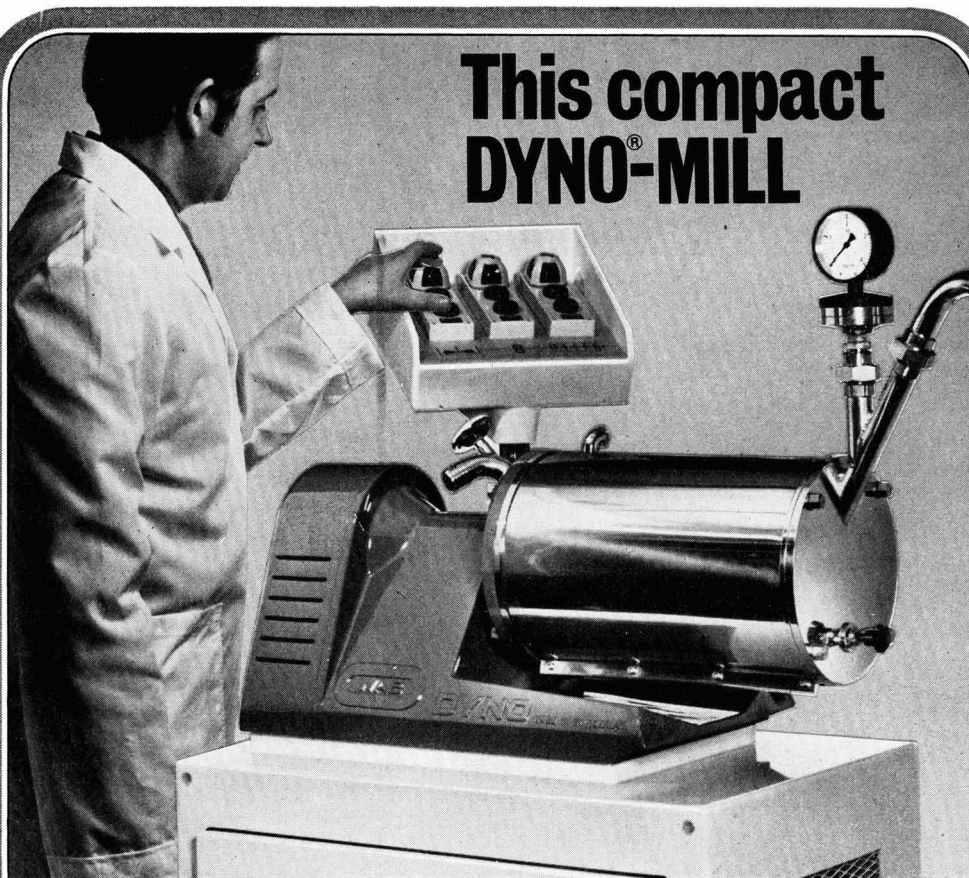
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Report of Council Meeting

A meeting of the Council took place at 2.00 pm on Thursday 8 July 1976, at the Great Northern Hotel, London N1 9AN. The President, Mr A. T. S. Rudram, was in the chair. There were 26 members present.

Before commencing the meeting, the President reported to the Council the tragic death at the weekend prior to the Annual General Meeting of Mr A. A. Duell, who had been Council's nomination for appointment as President Designate at the Annual General Meeting on 25 June 1976. The Council stood in silence in tribute to the memory of Mr Duell.

It was explained to the meeting that in order to fulfil the provisions of Article 65(A) it had been necessary to adjourn the Annual General Meeting until Wednesday 13 October 1976, when it would be reconvened at 4.00 pm at the Great Northern Hotel, London; the sole business transacted on that day would be to receive notification of the Council's appointment of a Member as President Designate. It would thus be necessary for Council to proceed with the election of a President Designate, following the pattern agreed at the Council meeting on 10 May 1968, before the next Council meeting on 13 October.

The President welcomed all Members who were serving on Council for the first time. Dr H. R. Hamburg was present for the first time as Hon. Treasurer since his election at the Annual General Meeting. The President pointed out that there were now six Chairmen overseas who served *ex officio* on the Council and it was also very pleasing to record that Council had nominated as one of the Vice-Presidents a Member from the General Overseas Section, Dr H. Rechmann, who was a Past-President of FATIPEC and a Fellow in the Association's Professional Grade. There were only rare occasions when Members from the General Overseas Section were nominated in this fashion, but it was felt that this would help to strengthen ties with the sister society and at the same time show the high regard in which Members of the General Overseas Section were held.

It was noted that two ladies were now serving on the Council—Miss P. Magee (Chairman, Irish Section) and Mrs E. N. Harper (Representative, Bristol Section).

It was reported that the three Members elected at the Annual General Meeting were:

Mr J. Smethurst
Mr F. B. Redman
Mr J. R. Bourne

The dates of the Council meetings for the forthcoming session were agreed and the appointment of Members to serve on Committees of the Council and to represent the Association on other organisations was confirmed.

Reports were received on the 1976 Dinner Dance, the forthcoming Council Reunion Dinner, the Annual General Meeting, and the arrangements for the 1977 Ex-

hibition. The Council feels that the value of the Exhibition to exhibitors and visitors alike should be constantly stressed by all Members in conversation with both colleagues and suppliers. The Exhibition was a unique achievement for a learned society and had become the annual international meeting place for those concerned with the surface coatings industries; every effort should be made by Members to foster the continued well-being of this important Association activity. Section Chairmen were asked specifically to ensure through Section Committees that where Members had any points which they wished to raise on Exhibition matters they should write to the Director & Secretary. Full replies were always sent to those interested in various aspects of this activity and specific points were referred to the Exhibition Committee.

The present position concerning the "Introduction to Paint Technology" (fourth edition) and the publication of papers from the Newcastle Symposium "Ultraviolet polymerisation and the surface coatings industry" were reported to Council. The fourth edition of "Introduction to Paint Technology" should be available by the autumn and the book on "Ultraviolet polymerisation and the surface coatings industry" was now available. Sales of the Decennial Index were steady, but not yet of the order which Council had felt would be forthcoming, particularly bearing in mind the very low rate charged to Members compared with that to libraries. Members were urged to ask their companies to ensure that copies of this important Association publication were purchased for use in company libraries.

Details of the titles of papers submitted for consideration to the Honorary Research and Development Officer for the 1977 Conference were provided, it being pointed out that these came not only from the United Kingdom but from the United States, Germany and Japan.

Consideration was given to the question of Section Symposia and the need to ensure that these were correlated.

A report of the meeting of the Professional Grade Committee held earlier that day was received and it was stated that one Fellow and five Associates had been admitted. (Following the Council meeting, a *viva voce* examination was held and another Member was admitted to the Licentiate Grade.)

Council was pleased to learn that Dr M. L. Ellinger had agreed to present a paper on behalf of the Association at the Convention in Washington in October of the Federation of Societies for Coatings Technology. It was reported to the Council that the Paint Research Association was celebrating its 50th Anniversary, and Council agreed that a commemorative scroll should be sent to the Paint Research Association, the wording of which is shown in the next column.

OIL & COLOUR CHEMISTS' ASSOCIATION



The Council of the Association at its meeting this day sends greetings and congratulations to the Paint Research Association on the occasion of the Fiftieth Anniversary of its foundation.

The Council recalls with pleasure the close liaison which has existed between the two Associations since the foundation of the Paint Research Association.

Members of the staff of the Paint Research Association have distinguished themselves in our activities by the continual service which they have rendered, in that many have served on the Council, its Committees and Section Committees, have presented numerous papers to Conferences, Symposia and other meetings of this Association, together with papers, reviews and other articles in our monthly Journal.

It is a matter of particular pride that no less than five Presidents of this Association were serving on the staff of the Paint Research Association, or had previously served thereon.

The Oil & Colour Chemists' Association looks forward to maintaining and strengthening this liaison in the years to come.

A. T. S. RUDRAM
President

R. H. HAMBLIN
Director & Secretary

8 July 1976

The President reported on the Liaison Committee meeting held at Cannes during the FATIPEC Congress on the 4 May and the preparations for a further meeting at the SLF Congress at Helsinki at the end of September.

The Director & Secretary reported on the possibilities of a closer liaison with the Worshipful Company of Painter-Stainers and of using Stratford-upon-Avon as a further Conference venue.

Reports were received of Section activities in the United Kingdom and Overseas and the rules of the new Ontario Section were approved. Suggestions for Branch activities in various parts of the world were also mentioned as requests had been received from Members in several areas.

The Council were informed that the "Notes for Guidance" for new members of Council and its Committee had been brought up to date. It was noted that the British Standards Institution had requested comments on certain standards covered by Committee on which the Association was represented.

A report on negotiations for the rent review at the end of 1976 under the terms of the lease for Priory House was received.

There being no other business, the President thanked members for their attendance and declared the meeting closed at 3.55 pm.

ANNUAL GENERAL MEETING

Notice of Adjournment

(See Classified Advertisements on page xvi)

Arthur Albert Duell, c.CHEM, MRIC, FTSC



1921-1976

AN APPRECIATION BY FRANK HELLENS

It is with deep regret that I write of the sudden death of Arthur Duell whilst on holiday in Malta on Sunday 20 June 1976.

Arthur had been a staunch supporter of OCCA since he was first employed in the paint industry. He became a Member in 1945 and, apart from the period 1963-67 when he was attached to the London Section, was always a member of the Newcastle Section. During this period he held the offices of Newcastle Section Honorary Secretary from 1954 to 1960 and Section Chairman from 1971 to 1973. His interest was also national as he was a Representative on Council 1960-63 and also served continuously on Council in various capacities from 1969 until his death. He was the Council's appointment as President Designate, to have been notified at the Annual General Meeting on 25 June 1976.

His death is a great loss to the Association; Arthur Duell would have made a tremendous contribution as its President. This dedication to OCCA is even more remarkable when it is realised that in 1956, Arthur set up the Sealants and Elastomers Division of Berger Chemicals and was subsequently Director and General Manager.

In addition to his involvement with OCCA he was an active member of the Sealants Manufacturers Association and was Chairman in 1970.

Everyone who met Arthur could not help but be conscious of the tremendous enthusiasm and energy which he brought to every aspect of living.

He was a keen sportsman, his overriding passion being golf, and also a dedicated bridge player, representing Newcastle in area competition.

Arthur was ably supported by his wife Gwen in all activities and greatly enjoyed the company of other people. He particularly enjoyed the company of young people and had five sons of his own.

To everyone who was associated with Arthur he was an inspiration as well as a leader and friend.

Our heart-felt sympathy is extended to Gwen and her sons. She has lost a fine partner and we have lost a dear friend and worthy business associate.

New publications

In July this year the Association published "Ultraviolet polymerisation and the surface coatings industries", a volume comprising papers presented at a symposium of the Newcastle Section of the Association in 1975 and subsequently published in the *Journal*. The twelve papers form a comprehensive 'state of the art' review of this new and exciting field, with particular reference to the surface coatings industries.

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

of chemistry and chemical symbols, formulae and equations is included as an introduction to the glossary itself.

It is expected that the new edition of "Introduction to Paint Technology" will be available in the autumn of 1976 and advance orders are now being accepted.

Jordan Award

This award was instituted by the late Mrs M. R. Jordan in memory of her husband Dr L. A. Jordan, who was President of the Association 1947-49 and an Honorary Member, and who died in December 1964. The Committee invites applications for the fourth award of £100.

The rules of the Award are:

1. The Award will be made for the best contribution to the science or technology of surface coating by a Member of any nationality working in either the academic or industrial field who is under the age of 35 at the date of application.

2. The final date for submission of applications will on this occasion be 31 December 1976 and it is hoped to present the award at the 1977 Association Conference.

3. The selection of the recipient of the Award will be made by a Committee under the chairmanship of the Association's Hon. Research and Development Officer.

4. There will be two methods of application. First, by the submission of a paper describing original work by the candidate which is offered for publication in the *Journal* or has been so published during application. The alternative method will be by recommendation by a superior for work which for reasons of commercial secrecy cannot be published; in this case the candidate will be expected to submit a dissertation on a topic relating to his work and demonstrating his superior knowledge of the principles thereof. The Award is for individual merit and clear evidence of the candidate's own contribution will be required if a paper is offered under joint authorship.

5. Applications should be addressed to the Director & Secretary at the Association's offices.

Bristol Section

Annual Dance

The Section's Annual Dance was held on Friday 2 April in the Mayfair Suite of the New Bristol Entertainment Centre. The Chairman, Mr L. J. Brooke, welcomed Members and the guests, who included the President Mr A. T. S. Rudram and Mrs Rudram; the President of the Birmingham Paint, Varnish and Lacquer Club Mr R. Arnold and his wife; the Chairman of the Thames Valley Section Mr J. Inshaw and his wife; and Mr W. McWaters.

In his speech, the Chairman paid tribute to the work of the Section's Social Secretary, Mrs E. Harper. This was the last Annual Dance she would organize due to her nomination as Chairman Elect. Mrs Harper's hard work on this occasion was well rewarded by a record attendance and a most enjoyable evening, which came to an end all too soon.



The President, Mr A. T. S. Rudram (right), and the Chairman of the Bristol Section, Mr L. J. Brooke, with their ladies

Professional Grade

Courses for Licentiatehip

Several colleges of further education are willing to help Registered Students and Ordinary Members of the Association with courses and the preparation of dissertations in respect of Licentiatehip in the Technology of Surface Coatings (LTSC), and these include:

East Ham College of Technology, High Street South, East Ham, London E6 4ER. "Paint technology and courses related to the surface coating industry". Short courses: "Coatings technology—the essentials" (September 1976); "Packaging material coatings" (Spring Term 1977). *Note:* The last two mentioned short courses are intended for those already engaged in the industry who possess adequate scientific knowledge and experience of the technology.

John Dalton Faculty of Technology, Manchester Polytechnic, Chester Street, Manchester M1 5GD. The following courses are available:

The Polytechnic Certificate for Paint Technicians Parts I and II.

The Higher National Certificate endorsement subject (for LRIC purposes) "Chemical technology with special reference to polymers and surface coatings".

The Associateship of the Society of Dyers and Colourists (including Branch 5, relevant to paints, lacquers and printing inks).

The City and Guilds Chemical Technicians Certificate (subject 087) Paint Option Part II (in conjunction with local colleges), and Part III leading to the award of a Full Technological Certificate.

The Polytechnic Certificate is a relatively recent development which provides a course of studies for technicians involved in such areas as quality control, raw material testing, technical service and product development within the paint manufacturing and allied industries. The course is arranged on a modular/credit basis and necessitates a total of three years of part time study (one day per week) to the Part II level, giving a standard of attainment similar to the former City and Guilds Full Technological Certificate in Paint Technology.

In addition to the above courses the Department will offer short courses in Surface Coatings Technology and make available evening facilities for persons wishing to produce a dissertation for submission for the award of LTSC.

Enrolment for the above courses will take place at the John Dalton Faculty of Technology on the 13, 14, 15 September 1976, during the periods 10.00–12.00 noon, 2.00–4.00 pm and 6.00–8.00 pm. However, students wishing to take the Chemical Technicians Paint Option should initially enrol at a local

technical college operating the Chemical Technicians Part II scheme.

Any persons requiring further information should contact the Head of Department, Dr V. G. Bashford, at the above address or discuss the matter with staff during enrolment week.

Polytechnic of the South Bank, Borough Road, London SE1 0AA. Courses leading to MSc in Polymer Science and Technology.

Slough College of Higher Education, Wellington Street, Slough SL1 1YG. During the academic year 1976/77 the College will offer City and Guilds Chemical Technician 086 Paint Option—Part II and the Full Technological Certificate. In addition, a short course (approximately nine weeks' duration) entitled "The protection of steel against corrosion" will be organised.

Thames Polytechnic, 13–40 Wellington Street, London SE18 6PF. Several degree courses in chemistry are organised.

Biennial Conference 1977

The next biennial Conference of the Association will be held at the Grand Hotel, Eastbourne, from Thursday 16 to Sunday 19 June 1977 and the theme for the Conference will be:

The conservation of energy, materials and other resources in the surface coatings industry

It is intended that as on previous occasions, full preprints will be published for despatch to delegates in advance of the Conference. It is important, therefore, that any person, whether or not a Member of the Association, who feels that a report of his work could form the basis of a suitable paper should contact immediately the Honorary Research and Development Officer by writing in the first instance to the Director & Secretary of the Association at the address on the Contents page of this issue.

Sixtieth Anniversary celebrations

In May 1978 the Association will celebrate the sixtieth anniversary of its foundation and it is felt that Members (and others who wish to take part in the celebrations) would like to have as much notice as possible of the dates. Following the pattern successfully used at the Association's Fiftieth Anniversary in 1968, the main events will be on two consecutive days. On the evening of Thursday 11 May it is planned to hold at a City Livery Hall a Commemorative Lecture, followed by a Dinner to which Past Presidents, Past Honorary Officers of the Association, Honorary Members and the surviving Founder Member will be invited as guests. On Friday 12 May the Association's Dinner and Dance will be held at the Savoy Hotel, London WC2 and Presidents of other societies, together with their ladies, will be invited to attend. Full details will be published in the *Journal* from time to time and forms of application for the celebrations will be sent to all Members (and others who have made application to the Director & Secretary) at the beginning of 1978.

Register of Members

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

Ordinary Members

ASHTON, RONALD, Walpamur Co (Kenya) Ltd, Likoni Road, Box 18141, Nairobi, Kenya. (*General Overseas*)
 BAXTER, DAVID, 175–4 Elm Ridge Drive, Kitchener, Ontario, Canada. (*Ontario*)
 BHIMJANI, NARENDRA, BSC, Giant Paints Ltd, PO Box 1772, Wellington, New Zealand. (*Wellington*)
 BOLL, SIEVARD RUDOLF, Croda Chemicals SA (Pty) Ltd, PO Box 1097, Parow East, Cape Province, South Africa. (*Cape*)
 BRITAIN, DAVID LEONARD CHARLES, 34 Hereford Street, Te Atatu, Auckland 8, New Zealand. (*Auckland*)
 CLARK, PAUL REGINALD, BSC, 58 Evertop Road, Potton, Sandy, Beds SG19 2PB. (*London*)
 DASGUPTA, DIPJYOTI, BSC, MSc, Paint Research Association, Waldegrave Road, Teddington, Middlesex. (*London*)
 FAWCETT, JOHN PAUL, BSC, PhD, Shell Oil (NZ) Ltd, Shell House, Albert Street, Auckland, New Zealand. (*Auckland*)
 FORTH, THOMAS FRED, 118 Applefield Drive, Scarborough, Ontario, Canada. (*Ontario*)
 JAMIL, EMAD TAMMY, BSC, Rafidain Paint Co, PO Box 2145, Baghdad, Iraq. (*General Overseas*)
 LAMING, CHRISTOPHER DEREK, 2 Sheffield Street, Toronto M6M 3E7, Ontario, Canada. (*Ontario*)

MITCHELL, COLIN MURRAY, BSC, Craig & Rose Ltd, 172 Leith Walk, Edinburgh 6. (*Scottish-Eastern Branch*)
 RUSSELL, GRAHAM JOHN, BSC, 74 Beechdale Crescent, Pakuranga, New Zealand. (*Auckland*)
 SMITH, ALEXANDER FERGUSON, NCP Marketing Corporation, Box 4113, Cape Town, South Africa. (*Cape*)
 STEMPA, KEVIN, 65 Jillett Street, Titahi Bay, New Zealand. (*Wellington*)
 STEVENS, ALFRED JAMES, BSC, 34 Ednam Road, Rondebosch, Cape, South Africa. (*Cape*)
 WALTERS, PETER, MSc, 21 Norrie Avenue, Mt. Albert, Auckland 3, New Zealand. (*Auckland*)
 YARNELL, TERENCE, London Electric Wire Co & Smiths Ltd, Church Road, Leyton, London E10. (*London*)

Associate Members

BOHMAN, JOHN, 251 Wincott Drive, Weston, Ontario M9R 2R6, Canada. (*Ontario*)
 CRAIG, ROBERT PETER, PO Box 1521, Durban 4000, South Africa. (*Natal*)
 FERGUSSON, DARRELL DESMOND, PO Box 75077, Gardenview, Transvaal 2047, South Africa. (*Transvaal*)
 HAYCOCK, RAYMOND JAMES, 25 Devon Road, Bucklands Beach, Auckland, New Zealand. (*Auckland*)
 HENTON, JOHN EDWARD PALMER, 36 Wright Street, Newtown, Wellington, New Zealand. (*Wellington*)

Forthcoming Events

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

September

Wednesday 8 September

Manchester Section: Golf Tournament, to be held at Stockport Golf Club.

Tuesday 14 September

West Riding Section: "The Health and Safety Act", by Mr P. F. Neal, H.M. Factory Inspectorate, to be held at Griffin Hotel, Board Lane, Leeds, at 7.30 pm.

Wednesday 15 September

Ontario Section: Plant tour—Reed Wall Coverings.

Friday 17 September

Irish Section: "Polymer emulsions and their effects on paint properties" by Mr K. R. Geddes, Crown Decorative Products Ltd, to be held at Clarence Hotel, Dublin, at 8.00 pm.

Manchester Section—Student Group: Symposium: "The theory and practice of dispersion" to be held at the Woodcourt Hotel, Sale, Cheshire.

Midlands Section: Ladies' Night at the Botanical Gardens, Edgbaston, Birmingham B15, at 7.00 for 7.30 pm.

Monday 20 to Wednesday 22 September

London Section: "Current trends in industrial finishing". Joint Conference with the Institute of Metal Finishing at Warwick University.

Thursday 23 September

Midlands Section: "Litho and letterpress inks: present and future" by Mr F. Whitfield, Mander Kidd Ltd. To be held at Birmingham Medical Centre, 36 Harborne Road, Edgbaston, Birmingham B15 commencing at 6.30 pm.

Friday 24 September

Bristol Section: Ladies' Evening, with wine tasting and a film to be presented by a member of Harveys Ltd. To be held at the Royal Hotel, Bristol, at 7.15 pm.

October

Scottish Section—Eastern Branch: Annual skittles match vs Student Group. Date and venue to be announced.

Friday 1 October

Hull Section: Annual Dinner Dance to be held at the Willerby Manor Hotel, Willerby, Hull, commencing at 8.00 pm.

Monday 4 October

Hull Section: "Safety and pollution effluent disposal" by Mr J. Alexander, PD Pollution Control Ltd. Joint meeting with the Institution of Chemical Engineers at the Haven Inn, Harrow Haven, Lincs, commencing at 6.30 pm.

Thursday 7 October

London Section: "Water repellent preservative stains" by a speaker from the British Wood Preserving Association. Evening meeting at the Royal Society of Tropical Medicine & Hygiene, 26 Portland Place, London W1 commencing at 7.00 pm.

Midlands Section—Trent Valley Branch: "North Sea oil—inspection" by Mr J. D. Griffiths, RJP Nicklin & Co, to be held at the Crest Hotel, Pastures Hill, Littleover, Derby at 7.00 p.m.

Newcastle Section: Joint Meeting with the Institution of Corrosion Science and Technology. Two papers will be presented: "The integration of the protection function when building a ship", by Mr J. P. Jemitus, Cammell Laird Shipbuilders Ltd, and Mr A. N. McKelvie, Paint Research Association and "Steel cleaning standards—a case for their reappraisal" by Mr McKelvie. To be held at Newcastle Polytechnic.

Scottish Section: "Recent developments in polyurethanes" by Mr R. M. Entwistle, Bayer (UK) Ltd, to be held at the Beacon's Hotel, 7 Park Terrace, Glasgow G3 at 6.00 pm

Thames Valley Section: "Health and Safety at Work Act". A panel meeting and discussion with speakers from Government and industry, to be held at the Beaconsfield Crest Motel (White Hart), Aylesbury End, Beaconsfield, Bucks at 6.30 for 7.00 pm.

Friday 8 October

Manchester Section: "Films from particles" by Mr M. J. Waghorn, ICI Paints Division, to be held at the Manchester Literary & Philosophical Society, George Street, Manchester commencing at 6.30 pm.

Tuesday 12 October

West Riding Section: "Water-based stoving finishes" by Dr K. Sellars, Harlow Chemical Co Ltd, to be held at the Griffin Hotel, Board Lane, Leeds commencing at 7.30 pm.

Wednesday 13 October

Reunion Dinner for past and present Members of Council. To be held at the Piccadilly Hotel, London W1 at 6.30 for 7.00 pm. *Informal dress.*

Friday 15 October

Irish Section: "Implementation of EEC directives on the packaging in paint, printing ink and adhesive industries" by Mr F. Shaughnessy, Industrial Inspector, Department of Labour, to be held at the Clarence Hotel, Dublin at 8.00 pm.

Wednesday 20 October

Ontario Section: "Fluorescent pigments" by Mr H. Lavel.

Thursday 21 October

Midlands Section: "The use of aromatic acids in modern coatings technology" by a speaker from Amoco Chemicals UK Ltd.

Friday 22 October

Manchester Section: Annual Dinner Dance to be held at Peacock Suite, Hotel Piccadilly, Manchester.

Friday 29 October

Bristol Section: "North Sea oil and gas—their effect on industry" by Mr J. Tooke-Kirby, FTSC, Chairman of the London Section. Joint meeting with the Birmingham Paint, Varnish and Lacquer Club to be held at the Royal Hotel Bristol at 7.15 pm.

Midlands Section—Trent Valley Branch: Social Buffet and Dance at the Cross Keys Inn, Turnditch, commencing at 7.30 pm.

November

Wednesday 17 November

Ontario Section: "The ultraviolet screening behaviour of pigments" by Dr O. Kennedy.

News of Members

The following appointments have been announced by the International Paint Company:

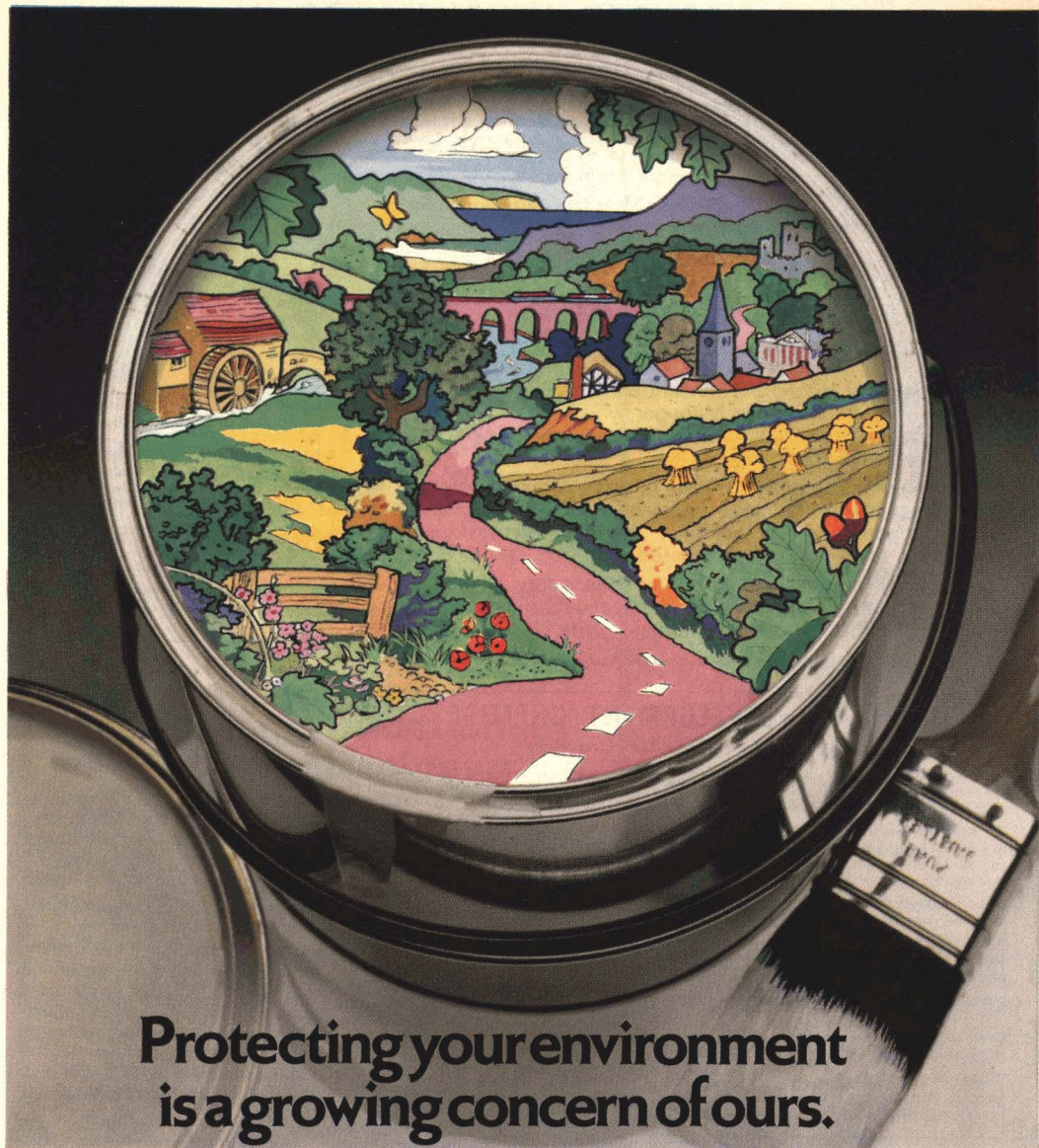
Mr T. B. Lemmon, an Ordinary Member attached to the Newcastle Section, has been appointed Managing Director of International Marine Coatings.

Mr B. R. Sykes, who is also an Ordinary Member attached to the Newcastle Section, has been appointed Technical Director of International Marine Coatings.

These and other International Pinchin Johnson board appointments have been made to meet long-term plans to cover future demand in Britain and Europe, and are followed by a number of senior management appointments and expansion of the company's marketing structure.

George R. Lester

With regret, we record the death of Mr G. R. Lester, who was an Ordinary Member attached to the Newcastle Section and Commercial Director of Durham Chemicals Ltd, and formerly Technical Manager of Durham Raw Materials Ltd. George Lester, who was 64, had been associated with the paint and allied raw material industries for many years.



Protecting your environment is a growing concern of ours.

Preservation without pollution for emulsion paints and latex emulsions. After using Proxel you'll never again go back to those old mercurial, phenolic and formaldehyde preparations.

Based on the unique preservative (1:2 benzisothiazolin-3-one), Proxel biocides combine chemical and heat stability with low toxicity and maximum protection against a wide range of spoilage organisms.

The range includes:

Proxel XL (latest liquid formulation, free from amines, completely non-yellowing in all systems);

Proxel CRL (liquid, based on ethylene-diamine solvent);

Proxel HL (liquid, based on mixed amine solvents) and

Proxel AB (original fluid dispersion).

Get Proxel protection now—for further information contact your local ICI Sales Office or



Organics Division

Hexagon House, Blackley, Manchester M9 3DA



CLASSIFIED ADVERTISEMENTS

Classified Advertisements are charged at the rate of £3.00 per cm. Advertisements for Situations Wanted are charged at 75p per line. A box number is charged at 50p. They should be sent to C. A. Tayler, BSc, Assistant Editor, Oil & Colour Chemists' Association, Priory House, 967 Harrow Road, Wembley, Middlesex HA0 2SF. JOCCA is published EVERY month and Classified Advertisements can be accepted up to at least the 12th, and in exceptional circumstances the 20th of the month preceding publication. Advertisers who wish to arrange for an extension of the copy deadline should contact the Assistant Editor, Mr C. A. Tayler, at the address given above (telephone 01-908 1086, telex 922670 OCCA Wembley).

SITUATIONS VACANT

**MANAGER
PAINT
SALES SERVICE LABORATORY**

An international group of companies with world wide interests in the paint industry has plans to develop its business in the U.K. As a consequence there is a need to recruit a Technical Service Manager who is experienced in all aspects of industrial and decorative paints and their formulation and application.

The main emphasis of the job will be to provide a laboratory support service for the sales organisation and the individual will be concerned with technical liaison with major paint companies to exploit company products.

As this is an important appointment the salary will be by negotiation at a managerial level. Applications should be sent to the Group Personnel Manager, Box No. 427.

SITUATIONS VACANT

**Chief Chemist
CANADA**

A modest sized, privately owned paint company, in Winnipeg, has a Chief Chemist position open for a person with strong industrial paints background and experience. The Chief Chemist in this new position, will direct formulation and laboratory work, as well as field testing and evaluations in a new area of coating technology. Salary commensurate with knowledge and experience in industrial coatings.

Contact: Guertin Bros. (Paint) 1976 Limited
270 Assiniboine Avenue at
Garry Winnipeg, Manitoba,
Canada R3C 0X6.

**LIQUID INK DEVELOPMENT CHEMIST
for Swale Chemicals, Nr. Croydon**

We are a major supplier of inks and coatings to specialised sectors of the paper, film and foil converting market. Our newly-built research laboratory deals with the development of coatings and inks for the UK and our world-wide overseas associates.

We require an experienced ink technologist or development chemist who is capable of working on his/her own initiative in the development and laboratory production of gravure and flexographic inks to meet new product specifications and to upgrade existing formulations when necessary.

It is anticipated that the successful applicant will have a technical qualification and at least five years experience with a liquid ink manufacturer.

Although the successful applicant will be based at Croydon, short periods away from base may sometimes be necessary for technical service requirements.

Swale Chemicals is a rapidly expanding company with specialised outlets and the present vacancy presents an opportunity to join a small, expert team with excellent long-term prospects.

Salary will be commensurate with experience, and benefits include four weeks holiday and pension/life assurance after one year.

Please write for application form to:—

BRIAN J. GREEN, SWALE CHEMICALS LIMITED, SWALE HOUSE, 164 THORNTON ROAD, THORNTON HEATH, SURREY CR4 6BB.

MISCELLANEOUS

**OCCA Annual General Meeting
NOTICE OF ADJOURNMENT**

Owing to the sudden death of Arthur Duell at the weekend preceding the Annual General Meeting, it was not possible to make the Council appointment of President Designate in accordance with Article 65(A). All other items on the Agenda were, therefore, completed and the Annual General Meeting was then adjourned until 4.00 p.m. on Wednesday 13 October 1976 at the Great Northern Hotel, London N1 9AN, when the only item on the agenda will be to receive notification from the Council of the appointment of a Member as President Designate.

SYMPOSIA

**Technical Education in the
Paint and Ink Industries**

The Borough Surface Coatings Association will hold a one-day symposium "Technical education in the paint and ink industries" at the Polytechnic of the South Bank on Tuesday 5 October 1976.

This symposium has been designed to acquaint those in the surface coatings industries concerned with further education of personnel with the changes in technological courses which are soon to take place under the aegis of the Technical Education Council (TEC).

The fee for the symposium will be £7.50 (£7.00 for members of the Borough Surface Coatings Association), which will include morning and afternoon refreshments and lunch. For further details write to:

P. J. Barnes, Esq.,
Department of Chemistry &
Polymer Technology,
Polytechnic of the South Bank,
Borough Road,
London SE1.

CLASSIFIED ADVERTISEMENTS

SITUATIONS VACANT



Join the Perfectionists at Rolls-Royce Motors

As well as Rolls-Royce and Bentley Motor cars, Rolls Royce Motors produce diesel engines, petrol and gas engines, light engines, light aircraft engines and investment castings. General engineering is another major activity.

These Rolls-Royce products enjoy an unequalled reputation for excellence and the people who make them are encouraged to use their many skills to the full.

Employee facilities include a company pension scheme, a comprehensive welfare society and a company subsidised canteen. Holiday entitlement starts at 27 days per year.

Senior Production Paint Technologist

The successful applicant will report directly to the Chief Paint and Plastics Technologist and will be responsible for the day-to-day process control in our Production Paint Shops. In addition he or she will be responsible for allied development work on new materials, methods and processes.

Applicants should hold Full Technological Certificates in paint technology or an equivalent degree and should have had relevant experience either with a paint manufacturer or within the automobile industry.

The factory is situated in Cheshire in a rural location and yet within easy access of the M6. The area caters for a wide variety of residential preferences and generous relocation allowances will be given. Salary will be not less than £4,100 per annum.

Reply in confidence to:-

John Williams, Staff Personnel Officer
with details of career history and salary.
Rolls-Royce Motors Limited,
Car Division, Crewe CW1 3PL.



Oil & Colour Chemists' Association

Paint Technology Manuals

Part 7: Works Practice

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

Price: £3.00 per volume
limp covers

★ See Order Form on page (ii) ★

Oil & Colour Chemists'
Association, Priory House,
967 Harrow Road, Wembley,
Middlesex HA0 2SF England

Send your Classified Advertisement for JOCCA* by telex 922670 (OCCA Wembley)

*Published 1st week of month. Copy date 12th of preceding month; extensions usually possible, by special arrangement

CLASSIFIED ADVERTISEMENTS

SITUATIONS VACANT

SURFACE COATINGS CHEMIST

Harco

The Surface Coatings Department of Harlow Chemical Co Ltd, an established and expanding joint venture of Hoechst and Revertex Ltd, manufacturing synthetic resin emulsions, requires a graduate chemist with experience of paint manufacture, surface coatings technology and related applications.

A suitable candidate (male or female) will probably be over the age of 28 years, be able to initiate and bring to fruition various projects with a minimum of supervision, and be sufficiently knowledgeable and authoritative to make technical visits to major companies.

A competitive salary is offered, together with re-location assistance where necessary, an excellent pension scheme and accident/life insurance.

Please telephone or write for application forms to:

Peter Shadbolt
Personnel Officer
Group Personnel Department
Harlow Chemical Company Limited
West Road
Templefields
Harlow, Essex. Tel: Harlow 29555

ORGANIC CHEMISTS

As part of a general expansion programme, Warwick Chemical Limited are seeking qualified chemists to staff their new laboratories.

The Company, part of a multi-national organisation and a leader in its own field, manufactures synthetic resins, (maleics, phenolics, alkyds, etc.) based on polymers formed from phenol formaldehyde condensates, vinyl monomers, esterification of fatty acids, etc. for the printing ink and paint industries. Salary will depend on age and experience but will be *circa* £3,850 per annum.

Warwick Chemical Ltd is well established, highly progressive and continually expanding the range of its products, so if you are prepared to work on the bench, aged about 30, and possess a University degree or Membership of the Institute of Chemistry, and have some experience in the above areas of organic chemistry, please write giving full details of academic qualifications and industrial experience to:

The Director of Surface Coatings,
Warwick Chemical Limited,
54, Willow Lane,
Mitcham, Surrey,
CR4 4NA.

A unique opportunity in Resin Far East

The Company manufactures synthetic resins for the Surface Coatings Industry internationally and already, through over 12 plants. Its growth record, profitability and present major size are impressive.

It is to set up a new plant in a climatically agreeable and low tax area of the East, and is to appoint an entrepreneurial Manager to establish and run its sales, marketing and production.

This is an exciting 'green field' situation.

It will be negotiated probably with a single Graduate Chemist certainly with in-depth experience of resins.

Salary will not be a bar to this senior appointment.

Candidates for it are invited to write, in absolutely guaranteed confidence, to:



Gordon Birtles, Managing Director, S.I.T.A. Ltd., 3 Lauriston Road, London SW19 4TJ. Tel: 01-946 0915 who has been retained to advise on this appointment.

OCCA-29 EXHIBITION MARCH 1977



Intending exhibitors are reminded that the closing date for returning application forms is

FRIDAY 1 OCTOBER 1976

Any organisation which has not yet received an Invitation to Exhibit and wishes to do so, should contact the Director & Secretary at the Association's offices

Telephone 01-908 1086
Telex 922670 (OCCA Wembley)

NEW OCCA TIE

Members are reminded that in addition to the original tie with a blue background, the Association now offers an alternative maroon tie with a single gold motif.

The maroon ties cost £1.85 each, including Value Added Tax, packing and postage by surface mail, and may be obtained (prepayment only) by sending the necessary remittance to the Association's offices at the address on the contents page of this issue, quoting Section, Membership number, and full postal address.

CLASSIFIED ADVERTISEMENTS

SITUATIONS VACANT

More jobs in paint

£3,500 — £6,500.

As Selection Consultants working especially in the Surface Coatings Industry, we have been retained to advise on the appointments of:—

1. Senior Chemist for General Industrial Paints

This is a major opportunity to lead several teams covering the development and formulation of domestic appliance and other specialised industrial finishes for UK and Overseas markets.

A chemist of the highest calibre and with substantial experience of Laboratory management and sophisticated product development is required.

Location: SOUTH *Salary to* £6,500+ *Reference:* SCGIP/OCCA/Sept.

2. Vehicle Refinishes Chemist

This is a senior opportunity for a chemist qualified at least at HNC level and with 'in-depth' experience of Refinishing Paints and/or General Industrial Coatings.

Responsibility is for product development, formulation and T/S through a small team backed by sophisticated and advanced technology.

Location: MIDLANDS *Salary up to* £6,000 *Reference:* VRC/OCCA/Sept.

3. Team Leader for Metal Decorating Products

The Company is a leading manufacturer of can and coil coatings. It is to appoint an experienced technologist qualified at least at HNC level to lead a team in product development and technical service. Experience of UV is a particular advantage.

Location: LONDON *Salary up to* £5,500+ *Reference:* TLM DP/OCCA/Sept.

4. Senior Chemist

For Electrodeposition Development work as a Graduate or HNC Chemist, aged 35-40. Responsibility within a leading Company is for R&D, Product Development, Trials and Q.C. Liaison.

Location: MIDLANDS *Salary up to* £5,500 *Reference:* SCED/OCCA/Sept.

5. Wood Finishes Chemist 30+

To lead a small team within a major organisation with responsibility for technical sales support, product development and formulation control.

Location: SOUTH *Salary up to* £5,000 *Reference:* WFC/OCCA/Sept.

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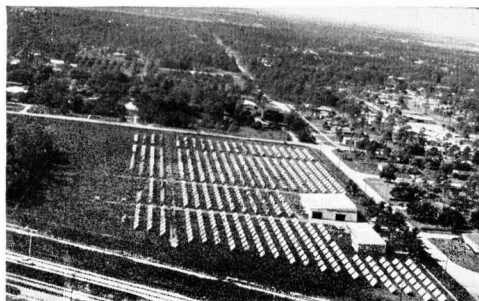
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