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# JOURNAL OF THE OIL & COLOUR CHEMISTS' ASSOCIATION

Thermal cure of acrylic finishes in a laboratory gradient stove

*J. F. A. Hazenberg*

Preparation and properties of paint films with special morphological structure

*W. Funke*

The effect of wet time on accelerated outdoor exposures

*J. A. Scott and T. E. Anderson*



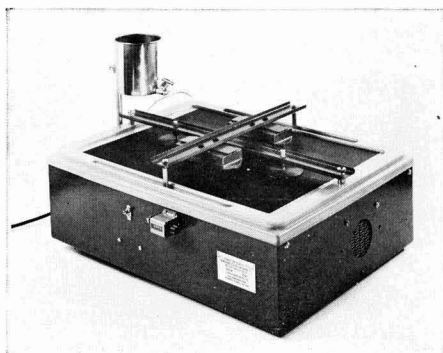
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Full details, including technical programme, summaries of papers and authors' biographies on pages 419-425



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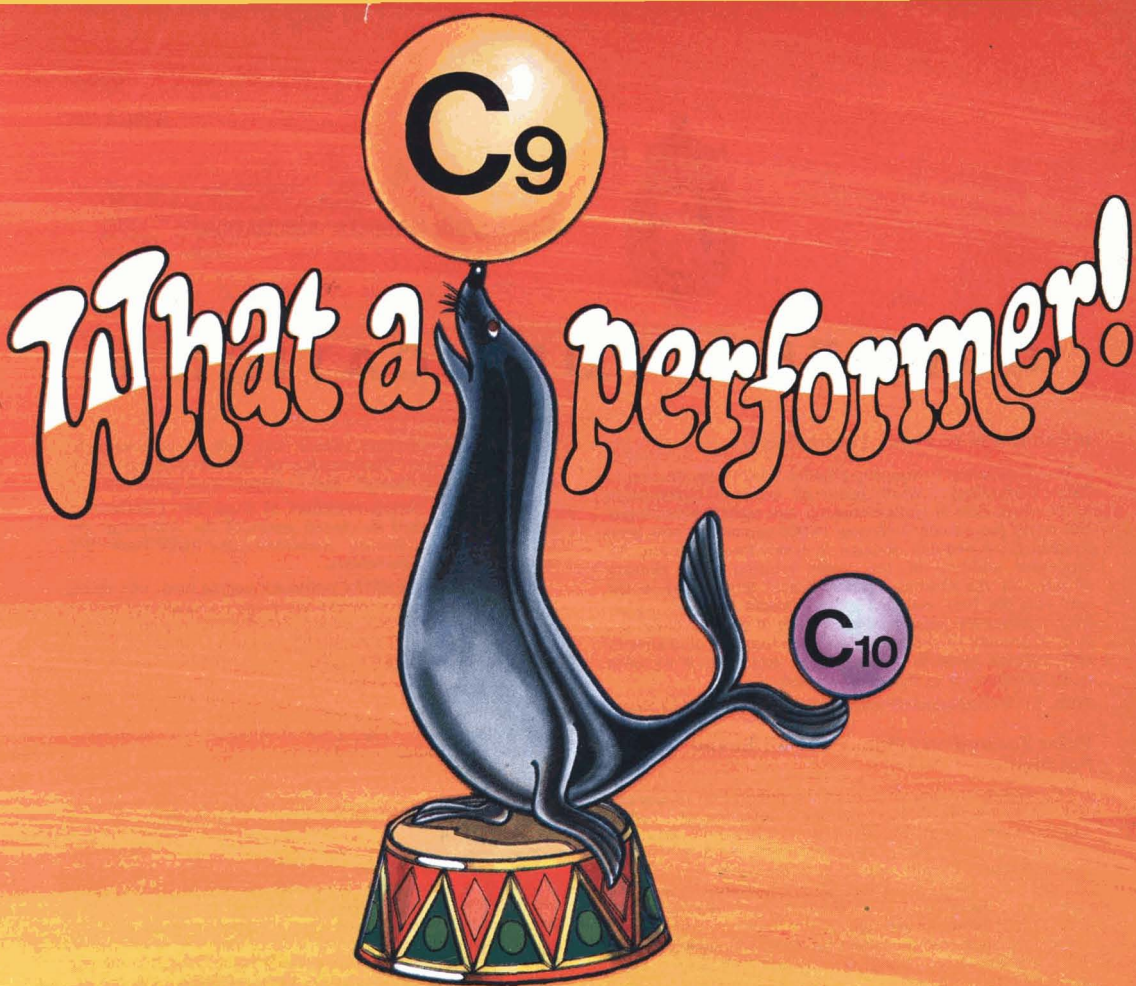
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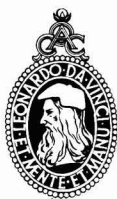
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The Association has sponsored the publication of a series of Paint Technology Manuals primarily intended for students and those entering the industry but already acknowledged to be invaluable to the practical man within the industry. "Works Practice" 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.

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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 pages 419 to 425 of this issue.

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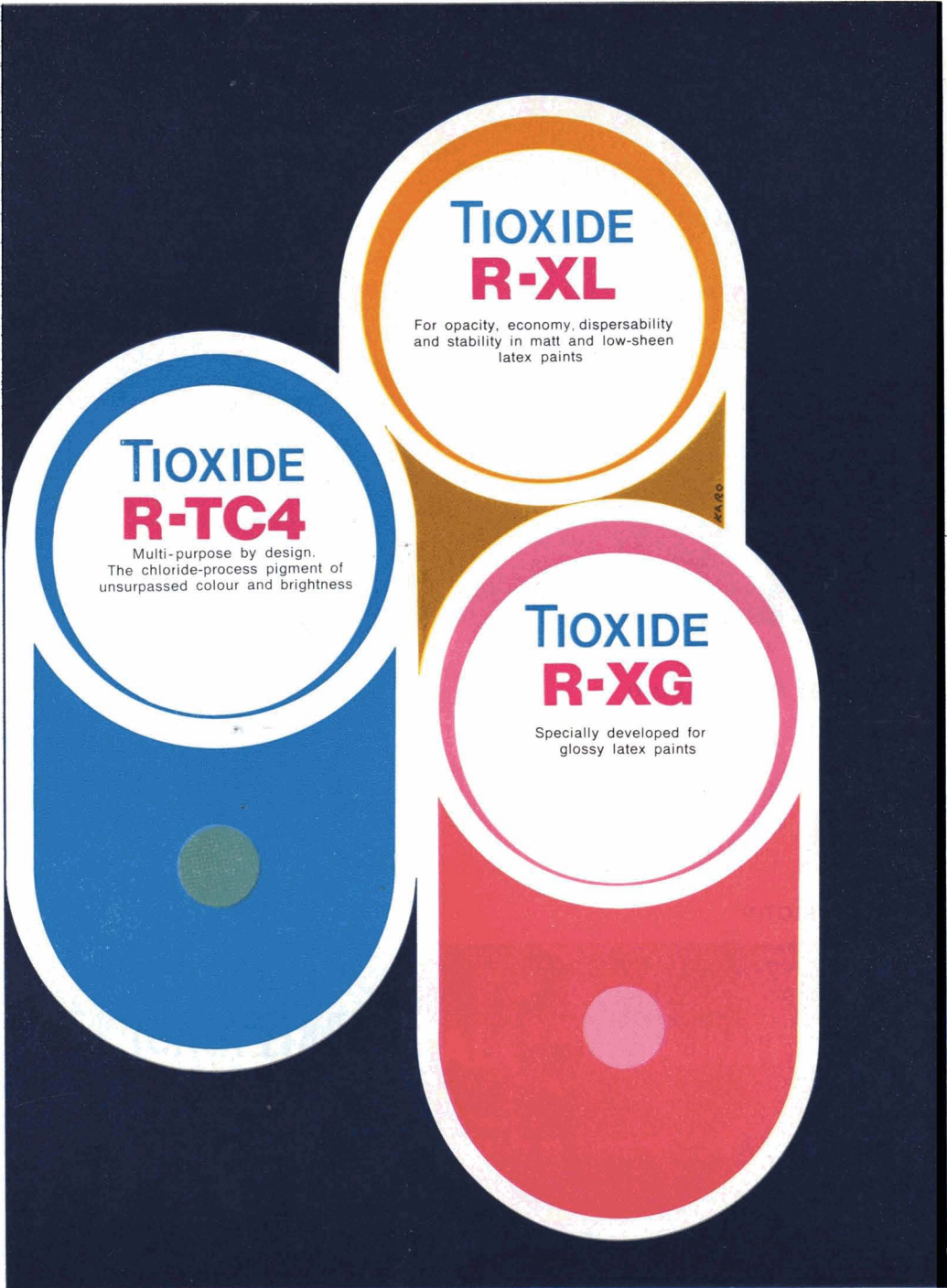
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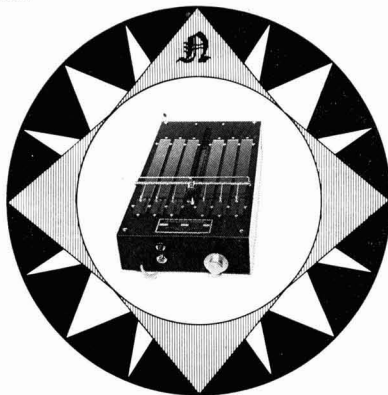
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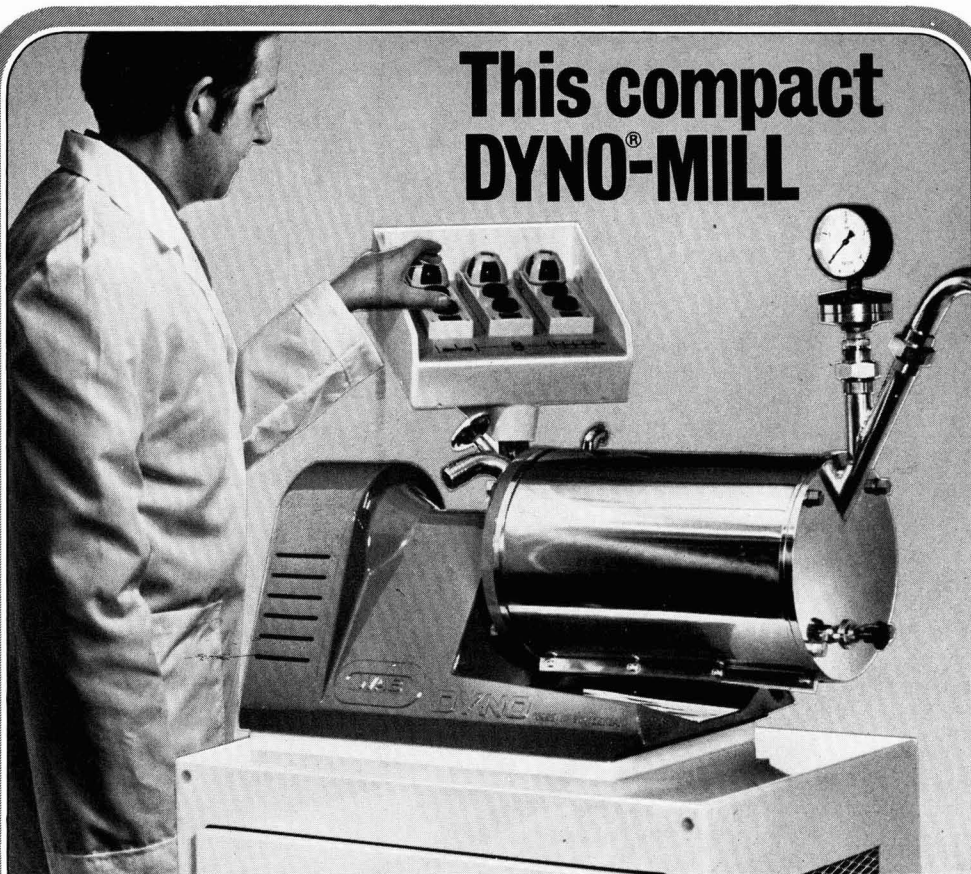
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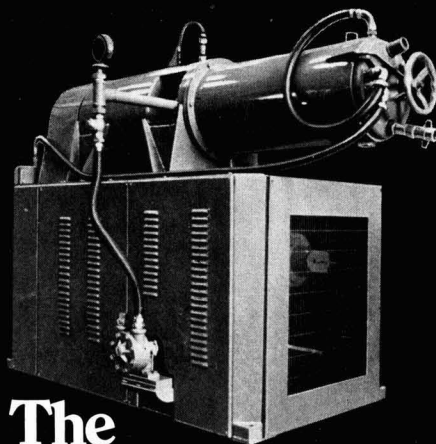
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Vol. 59 No. 11

November 1978

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## Transactions and Communications

# Thermal cure of acrylic finishes in a laboratory gradient stove\*

By J. F. A. Hazenberg

Verfinstituut TNO, Delft, Netherlands

### Summary

A special laboratory stove has been developed at Verfinstituut TNO (Paint Research Institute, Holland). Features of this gradient stove are an almost linear temperature gradient in one direction and a constant temperature in any cross section.

With this device, all kinds of storable materials which can be applied in an even coat on a metallic substrate, may be stoved at strictly controlled temperatures. Moreover, only one baking test

has to be made for the measurement of film properties, such as hardness, extensibility, adhesion, gloss and yellowing, as a function of temperature.

The principle of operation, method of construction and reliability as well as the various applications of the apparatus are outlined, and its use is illustrated by describing the results obtained with two commercial acrylics of the thermosetting type.

### Keywords

*Types and classes of coatings and allied products*

acrylic coating

*Equipment primarily associated with analysis, measurement or testing*

electric oven

*Processes and methods primarily associated with:*

analysis, measurement or testing

laboratory test

thermal analysis

*drying or curing of coatings*

stoving

*Properties, characteristics and conditions primarily associated with materials in general*

temperature

## Le durcissement thermique des peintures acryliques dans une étuve laboratoire à gradient de température régulière

### Résumé

On a élaboré à la Verfinstituut TNO (Centre de Recherches sur Peintures, Hollande,) une étuve laboratoire de conception exceptionnelle. Les caractéristiques de cette étuve comprennent un gradient de température quasi-linéaire dans une certaine direction et une température constante à chaque section transversale.

Au moyen de ce dispositif, toutes sortes de produits qui sont capables à être cuits au four et aussi à être appliqués sous forme d'une couche uniforme sur un subjectile métallique, peuvent être cuits au four aux températures rigoureusement contrôlées.

D'ailleurs, on n'est obligé de mettre à l'étuve qu'une seule plaque pour déterminer, en fonction de température, les caractéristiques de film telles que, la dureté, l'extensibilité, l'adhérence, le brillant et le jaunissement.

On trace les grandes lignes des principes opératoires, la méthode de construction et la fidélité, de même que les diverses applications de l'appareil, et en tant qu'indication de son utilisation, on décrit les résultats obtenus avec deux revêtements acryliques auto-durcissables de commerce.

## Wärmehärtung von Akrylharzlacken in einem Laboratoriums-Gradientofen

### Zusammenfassung:

Im Verfinstituut TNO, Holland wurde ein besonderer Trockenofen entwickelt. Charakteristika dieses Gradientofens sind der beinahe lineare Gradient in einer Richtung und die konstante Temperatur in jedwedem Querschnitt.

Mit diesem Gerät können ofentrocknende Materialien aller Art, welche sich in gleichmässiger Schicht auf ein metallisches Substrat auftragen lassen, unter streng kontrollierten Temperaturen getrocknet werden. Ausserdem braucht nur eine Trocknungsprobe

zur Messung der Filmeigenschaften, wie Härte, Dehnbarkeit, Haftung, Glanz und Vergilbung als Funktion der Temperatur, gemacht werden.

Das Arbeitsprinzip, Konstruktion und Verlässlichkeit werden, ebenso wie die verschiedenen Anwendungsmethoden des Apparates skizziert, und durch Beschreibung der mit zwei, zum selbsthärten Typ gehörenden Akrylaten des Handels, erhaltenen Resultate wird die Benutzung klargemacht.

### Introduction

*Refs. 1-7*

A coating is generally applied to a metallic substrate to impart better visual appearance and increased protection against all kinds of environmental influences. Full develop-

ment of the coating's properties is obtained either by normal air drying or by baking. Some of the important properties of stoved paint films are hardness, extensibility, adhesion, impact resistance, gloss and often resistance to solvents, chemicals and weathering. The dependence of these properties on paint composition and stoving conditions, for example

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

temperature and time, is studied in the paint laboratory by means of well-known laboratory stoves.

It is known that in many cases the reproducibility of the results obtained and also their agreement with practical performance leaves much to be desired. Some reasons for the problems encountered have been given by Boers<sup>1</sup> and by Bannink and Ribberink<sup>2</sup>. The most important shortcomings of the apparatus were considered to be a slow or retarded heating up of the object and a non-homogeneous temperature distribution throughout the stove. The importance of measuring continuously the temperatures of the stove and the object has been stressed, therefore, by various authors<sup>3-6</sup>.

In earlier publications<sup>3,7</sup> the author has shown that the results of stoving experiments can be made more reliable by using either a normal laboratory stove in which the temperature distribution is really homogenous, or a so-called "gradient stove", based on the principle of the well-known Kofler heating bench.

Based on the experience gained with the already described experimental gradient stove<sup>7</sup>, a prototype of far more flexible design has been built by the Engineering Department of TNO. This apparatus permits one test panel to be stoved at a continuous range of constant temperatures at the same time.

In this paper information about the construction, stoving procedure, measurement of paint film properties and fields of application will be given. Moreover, the use of the new apparatus will be illustrated by some results obtained with two commercially available thermosetting acrylics.

With a few exceptions, the experiments involved formed part of a research programme sponsored by the VVVR (Research Organisation of the Dutch Paint Industry).

## The TNO gradient stove

### Construction

A general impression of the construction can be obtained from Fig. 1. The proper stoving space is located in the heat isolated upper part, whereas all necessary control mechanisms are contained in the lower part of the apparatus.

Thermal equilibrium is reached in about three hours by carefully balanced heating and cooling; for this purpose the gradient stove is provided at both ends with a thermostatically controlled heating element and at the low tempera-

ture side with a cooling unit. As a cooling agent, air or a liquid of constant temperature can be used. For gradient ranges lying partially below room temperature the heating element at the low temperature side is switched off. Stove temperatures which are approximately equal to the object temperatures, are measured by means of ten thermo-elements embedded in the wall at regular distances of 10 cm and the results recorded.

At equilibrium, a nearly linear temperature gradient exists along the apparatus and a constant temperature in any cross section. With certain restrictions, gradient ranges between  $-10^{\circ}$  and about  $300^{\circ}\text{C}$  can be chosen. Gradient ranges smaller than  $10^{\circ}$  or greater than  $200^{\circ}\text{C}$  cause certain problems with regard to conservation of thermal equilibrium and maintenance of linearity. A few examples of possible gradient ranges are shown in Fig. 2.

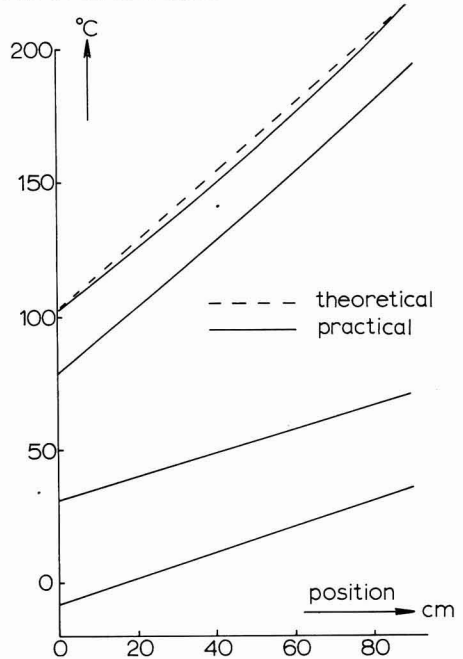


Fig. 2. Temperature distribution in gradient stove from low to high temperature side

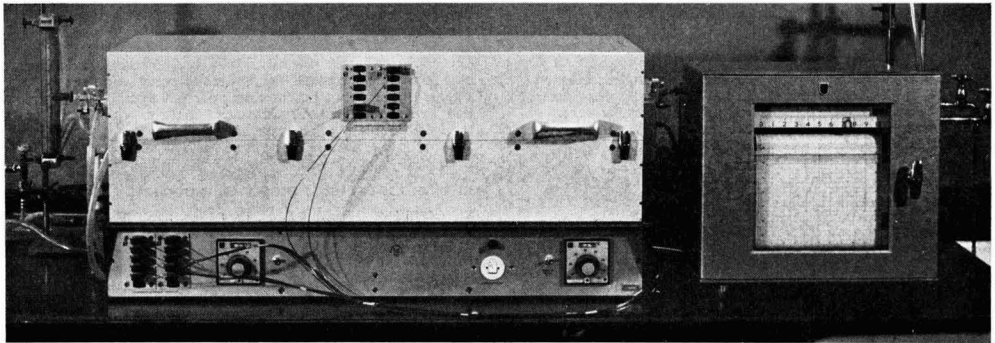


Fig. 1. Photograph of the gradient stove apparatus

Specially designed doors at both ends are used to insert a test panel,  $1000 \times 100$  mm. To prevent the low temperature side of the coated test panel from being exposed to high temperatures, loading at the low temperature side is preferred, especially when powder coatings have to be stoved.

The temperature equilibrium of the gradient stove is hardly affected by insertion of the test panel, which is normally at room temperature. Heat transfer between gradient stove and test panel is very rapid as a consequence of direct metallic contact with the bottom side of the stove. Fig. 3, showing the temperature/time relationship for a number of spots on the top of a 1 mm steel panel, gives an indication of this rapid heat exchange. This Fig. further shows that every point of the test panel (object) has a nearly constant temperature throughout the whole thermal treatment.

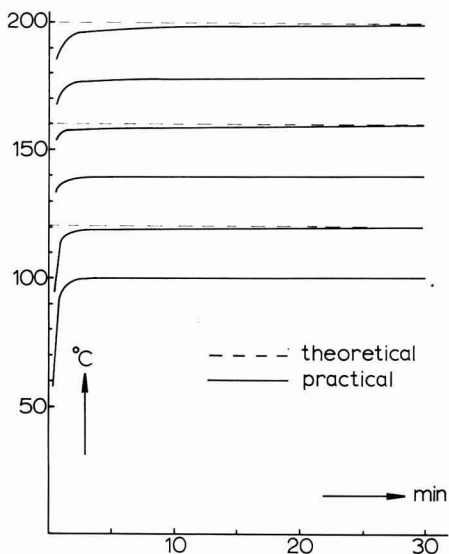


Fig. 3. Temperature-time relation for a number of places on a test panel in the range 80–200°C

Gases such as air can be passed through the oven space to remove any evaporated solvents and gaseous products set free during the thermal treatment of a coated substrate. A moderate stream of gas flowing from the low to the high temperature region does not disturb appreciably the heat equilibrium.

#### General stoving procedure

It should be stressed that treatment of any material in the gradient stove does not mean anything other than a thermal treatment with accompanying physical and possibly chemical changes in the material. In principle, this means that all kinds of paint may be stoved in the apparatus and further that all known methods of paint film characterisation may be applied. Nevertheless, in using the technique of gradient stoving some special requirements have to be fulfilled. The most important are indicated below.

All kinds of metallic substrate may be used. For most problems in the paint field, test panels of cold rolled steel ( $1000 \times 100 \times 0.5$  mm) are convenient. For special purposes, as for instance swelling experiments and weathering studies,

panels of tin-plated steel or aluminium may be used, and detached films can be obtained, for example by the usual method of immersing the tin-plate panels in mercury.

Common pretreatments, such as phosphating, degreasing and sanding with abrasive paper, may be applied. As the interest was primarily in the behaviour of single paint systems, phosphating was omitted from the author's tests in nearly all cases.

Paint material may be applied by brushing, spraying or by means of special instruments. Solvent based paints, which were used in most of the experiments, were always applied by means of a specially designed applicator, enabling a wet film of constant thickness and about 6 cm wide to be obtained, whilst leaving the edges of the test panel uncovered.

Powder coatings were normally applied by means of electrostatic spray equipment. In cases where only a small sample was available, a powder slurry in a non-solvent could be made and applied by a doctor blade, in a manner similar to that used when producing plates for thin layer chromatography. In all cases special attention should be paid to possible pollution of the bottom side and edges of the test panel, since attached material may give rise to clogging and contamination of the oven. Polluted test panels can be cleaned quite satisfactorily by wiping with a cloth.

Once the oven has reached equilibrium the coated test panel is inserted into a fixed position in the stove via one of the unlocked doors, treated for the time chosen and then taken out the same way.

After stoving, the test panel is marked at regular distances of 10 cm in accordance with the positions of the thermoelements in the stove. Object temperatures equal to the corresponding stove temperatures are attributed to these marks with the aid of the recorded temperature data.

Stoving temperatures at various points between the fixed marks may be estimated by linear interpolation. It is thus evident that the accuracy of interpolation increases with the linearity of the gradient. In the extreme case of exact linearity, measurement of the temperature at only two places in the gradient stove will be sufficient.

#### Measurement of paint film properties

Before a discussion of the quantitative aspects, it should be observed that gradient test panels once stoved, give a lot of qualitative information simply from a visual inspection. Normal solvent-containing industrial finishes, for example, very often show the influence of temperature on flow, gloss and yellowing. The results of stoving powder coatings in this manner are quite impressive. The technique of gradient stoving allows the entire pattern of flow behaviour to be visualised easily. When, for instance, a test panel which has been sprayed electrostatically and stoved, is carefully inspected, four distinct areas can be observed at increasing temperature:

- (i) unchanged powder layer, easily removed by wiping with a cloth
- (ii) slightly sintered powder layer without coherence, which cannot be removed by wiping
- (iii) area of flow with some gloss, insufficiently cured
- (iv) area of optimum cure, followed in some cases by a region of obvious overbake.

Before measurement of the usual paint film properties, the stoved products were always brought to equilibrium at 23°C and 50 per cent relative humidity in accordance with the latest standardisation requirements (ISO 3270-1974). It should be remembered that each test panel represents paint material cured at an infinite series of stoving temperatures according to the gradient chosen; that is, the test surface for any specified temperature is reduced to a straight line across the panel.

In theory, this means that measurements requiring an extended test surface are not suitable for the gradient stoving technique. In practice, however, paint film properties such as König pendulum hardness, extensibility according to Erichsen, cross-cut adhesion, gloss and yellowing can be measured without any difficulty. For reasons of convenience, the relatively large test panel is cut into appropriate sections.

The number of separate properties which can be determined from one test panel depends obviously on whether or not the chosen method of test damages or destroys the film. It is evident that the non-destructive tests should be performed first. In this way the above-mentioned properties can all be determined from one test panel.

Determination of the impact resistance, basically a statistical and destructive method, demands a number of test panels. Due to the technique of gradient stoving, this property can be determined in a modified way. It is performed by testing the panel at a fixed impact value (constant weight and height) at regular distances (typically 5 cm) along the whole length of the panel (gradient range). The panel is then tested in the same way at another impact value, sensibly chosen on the basis of the first results, and so on, if necessary, on other identically stoved test panels. The temperature dependency of the impact resistance is then found by recording for each individual impact whether damage has occurred or not, and then determining the boundary of damaged and undamaged areas.

When using this new technique it was found that three test panels was a sufficient number in most cases. Fig. 4 shows an example of the method described for an arbitrarily chosen, water soluble acrylic finish.

Structural properties, such as swelling and modulus of elasticity in the rubbery state, are determined on the basis of appropriate sections of free paint films. From the description given above, it will be clear that paint film properties can be measured either directly or in a slightly modified manner;

after determination of the property at a series of points along the test panel, it can easily be plotted against temperature of stoving.

**Reliability of gradient stoving**

When introducing the new method, the question will arise whether the results are consistent with those obtained by stoving in a normal laboratory oven operating at a series of constant temperatures. Fig. 5 shows the hardness of some alkyd/melamine systems similar in composition but differing in reactivity of melamine component, and this serves to demonstrate that the curing behaviour is the same in both cases.

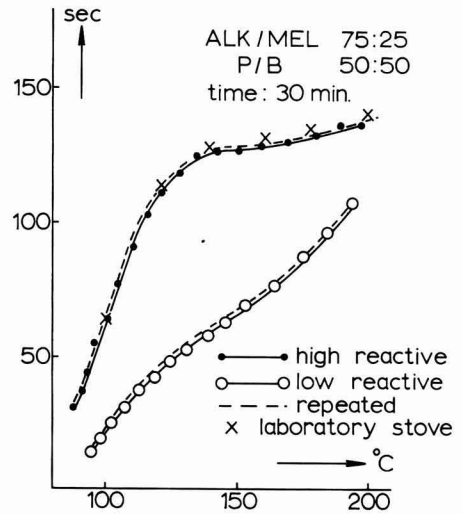


Fig. 5. König pendulum hardness of alkyd/melamine combinations

Another important factor in gradient stoving is the reproducibility of stoving experiments. Fig. 5 gives a good indication that there is very little scatter in hardness measurements; it implies that rather large deviations from the smooth curve through the measured points can be discarded as obvious errors. In this connection it must be remarked that the scatter found in the results of other properties, for instance

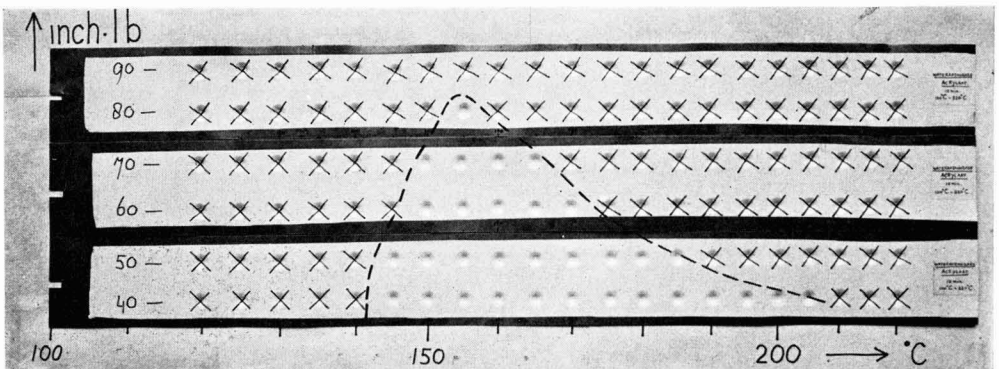


Fig. 4. Method of determining the temperature dependency of impact resistance

extensibility and impact resistance, are somewhat larger. Nevertheless, it is still possible in these cases to draw accurate curves through the points of measurement. Fig. 5 also shows that the repeatability of a gradient stoving experiment is quite satisfactory.

It is believed that the good reproducibility of the experiments is a direct benefit of the new technique of stoving. Working with a single, comparatively large, test panel in a temperature gradient ensures optimum conditions for good results. Important factors in this respect are:

- (i) temperature equilibrium in the oven,
- (ii) very fast heat equilibrium being attained between the oven and the test panel, leading to a nearly constant temperature gradient during the whole stoving process,
- (iii) the same substrate, paint composition and thickness of paint can be ensured,
- (iv) the same flash off and stoving times are ensured.

#### Fields of application

The gradient stove was developed originally for applications in the paint field, particularly as a convenient means of studying the influence of a systematic variation in composition and stoving time on the ultimate properties of a paint film. Paint formulations and stoving conditions can be optimised in this way.

Emphasis should be given to the time saving character of the method. The saving of time is due to the whole temperature range being studied with only one test panel; the numerous experiments required in conventional laboratory stoving become unnecessary. The new method will undoubtedly find application in other fields, for example the adhesives and plastics industries. The melt, flow and decomposition behaviour of powdery materials can also be studied. The following are only a few examples of possible new areas of application within the paint industry:

1. To study the influence of time and relative humidity on the air drying of alkyds and auto-repair enamels or on the minimum film formation temperature of unpigmented latices.
2. To study solvent containing finishes, such as alkyd/melamines, oil free polyester combinations, epoxy/novolac systems and acrylics under a variety of conditions.
3. To study high solids, water soluble or dilutable paints, plastisols and non-aqueous dispersions.
4. Quality control of new materials and so forth.

When a paint system has been stoved, the end product can be investigated by various test methods. Apart from the mechanical and structural properties discussed above, others which can be investigated include electrical properties, such as resistance and dielectric constant, and those properties influenced by physical or chemical attack on the coating, such as solvent and chemical resistance, salt spray and weathering.

Information on the chemical reactions taking place during stoving can be obtained by determining the degree of conversion via infrared spectroscopy of the clear film.

In addition to the above mentioned applications, the gradient stove technique offers some additional possibilities:

1. Measurement of paint film properties on panels which have previously been stoved at a fixed temperature. Properties showing a drastic change over a restricted temperature range may be determined in this way, for example, glass transition temperature, cold check, attack on the paint film by all kinds of material (oils, fats, etc).
2. Collection of effluent gases, for example, by condensing with liquid air. Subsequent analysis of the condensed material offers the possibility of gaining a deeper insight into the mechanism of curing and/or a better knowledge regarding the products which are possibly harmful to the environment.

#### Acrylic finishes

##### General

*Ref. 8*

Acrylics are synthesised by simple addition polymerisation or copolymerisation of unsaturated monomers. In general they are characterised by a higher molecular weight than normal condensation polymers, such as alkyds, phenolics and epoxies. According to the absence or presence of pendant reactive groups in the molecule, they may be thermoplastic or thermosetting in nature. The thermosetting acrylics which do not need a hardening component are termed "self-hardening". There are many types of possible systems and these have been reviewed by Solomon<sup>8</sup>.

Some of the properties of the self-hardening acrylics are good adhesion, an attractive compromise between hardness and elasticity and good resistance to hydrolytic agents and natural weathering. They may be used for domestic appliances, automotive finishes and industrial sidings.

##### Starting materials and paint preparation

Two commercially available self-hardening acrylic resins dissolved in a 1:1 mixture (by volume) of xylene and butanol were investigated; one of these gave rise to relatively hard and the other to relatively flexible coatings and, based on this difference, the resins were coded *H* and *F*, respectively. Solids content, acid number, viscosity and molecular weight are given in Table 1.

Table 1  
Properties of the acrylic resins studied

Property	<i>H</i>	<i>F</i>
Solids content, 16 hours at 100°C (%)	54	52
Acid number (mg KOH/g dry)	14	34
Viscosity (poises)	16	8
Molecular weight at peak maximum	14 000	13 000

Molecular weight distribution of the resins was determined by means of gel permeation chromatography (GPC), with tetrahydrofuran as eluent. Molecular weight at peak maximum was attributed to the resin by direct comparison with the calibration curve for polypropylene glycol.

A rutile titanium dioxide coated with a mixture of aluminium and silicon oxides was used as pigment. This was dispersed in part of the acrylic resin and ball milled for 20

hours to a fineness of 7  $\mu\text{m}$ . The remainder of resin and solvent was then added in the usual way to the ground mill base. A pigment/binder ratio of 50 : 50 (by weight) was used in all cases.

#### Application, stoving and conditioning

Steel panels (1000  $\times$  100  $\times$  0.5 mm) were used as substrates in most cases. These panels were first degreased with perchloroethylene, sanded and again degreased. The tinplate panels (720  $\times$  100  $\times$  0.3 mm) intended for making free paint films, and the aluminium panels (1000  $\times$  100  $\times$  1.0 mm) for weathering experiments were degreased only once. The clear and pigmented paints were applied by means of an applicator bar as already described and the thickness of the dried layers were found to be 36  $\mu\text{m}$  for the clear films and 33  $\mu\text{m}$  for the pigmented ones. The general stoving procedure was followed. Experimental data in abbreviated form were as follows:

Flow of air:	0.2 m <sup>3</sup> /hour
Gradient range:	80–200°C
Flash-off time:	20 minutes
Stoving times:	10, 20, 30 or 40 minutes
Conditioning:	48 hours at 23°C and 50 per cent RH.

The paint film properties tested are summarised in Table 2.

Table 2  
Methods used to measure the properties of substrate-bonded and free paint films

Property	Method
Thickness	Elcometer
Hardness	König pendulum
Extensibility	Ericksen cupping test
Impact resistance	Gardner (modified)
Adhesion	Cross cut and pulling off with tape
Gloss	Langé (45°)
Yellowness	Colour index (R–B/G)
Weathering	Langé (45°) and chalking (TNO)
Linear swelling in acetone	Measurement of length
Modulus of elasticity in acetone	Instron dynamometer (0.4 cm/min).

#### Experimental results

It will firstly be shown how the individual paint film properties depend on stoving conditions and pigmentation, and secondly it will be shown how for a fixed stoving time a set of important mechanical properties can be optimised. Lastly the temperature dependency of some structural properties will be described.

#### Temperature dependence of paint film properties

König pendulum hardness of the clear finishes *H* and *F* for various stoving times are given in the Figs. 6 and 7. The curves relating hardness to stoving temperature all show a plateau level of hardness after an initial steady increase. As expected, the hardest coating is acrylic *H*. Increasing the stoving time from 10 to 40 minutes has relatively little influence on hardness, especially in the high temperature range. The most remarkable fact to emerge from the figures is that acrylic *H* becomes very hard even at low stoving temperatures. The reason for this is not clear since both basic resins differ in chemical composition, molecular weight and acid number, and all these factors may influence hardness. For pigmented

paint films (also shown in the Figs. 6 and 7), the same trends have been found.

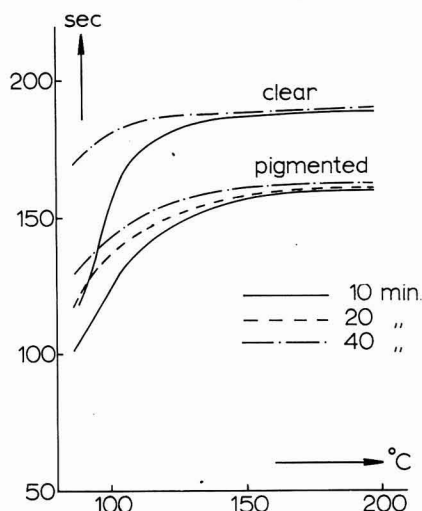


Fig. 6. König pendulum hardness of acrylic finishes H

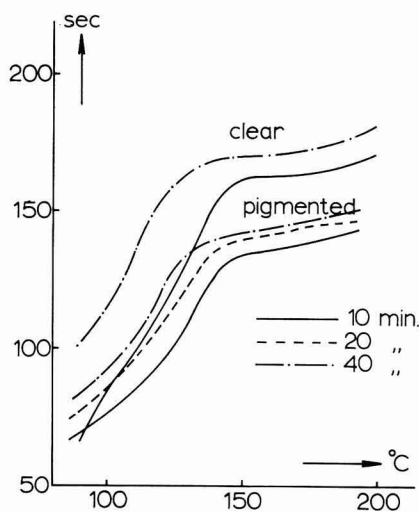


Fig. 7. König pendulum hardness of acrylic finishes F

This leads to the conclusion that the general form of the hardness curve is primarily determined by the nature of the acrylic resin. The decrease in hardness caused by pigmentation is rather unexpected. This decrease is absent, or rather restricted, at relatively low stoving temperatures, but becomes significant at higher temperatures. Indentation hardness according to the Wallace method has confirmed the decrease in hardness due to pigmentation. It is assumed that acrylics, like alkyd/melamines, exhibit this decrease in hardness as a consequence of a partial loss in catalytic activity. Carboxyl groups may be neutralised by interaction with the coating on the pigment.

Extensibility of the acrylic finishes *H* and *F* is shown in the Figs. 8 and 9. The hardest, clear finishes show very good extensibility. Resin *F* is even perfectly flexible since metal fracture occurs before crack formation in the paint film can be detected. The extensibility of the pigmented coatings is also excellent and somewhat independent of time and temperature of stoving. Finish *H* (Fig. 8) shows very remarkable behaviour when stoved at low temperatures, for a temperature range can be found in which the stoved finish has absolutely no extensibility; at higher or lower temperatures this strange effect of brittleness suddenly disappears.

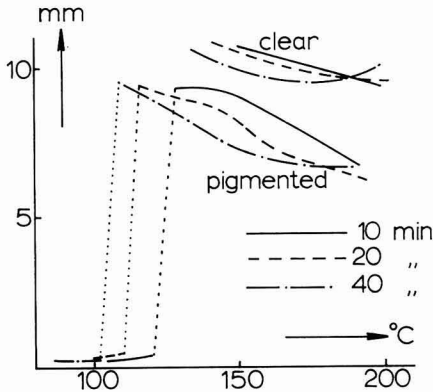


Fig. 8. Erichsen extensibility of acrylic finishes H

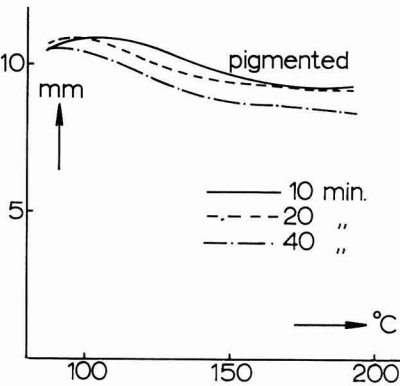


Fig. 9. Erichsen extensibility of acrylic finishes F

Results of the cross-cut adhesion tests for the acrylics *H* and *F* are shown in the Figs. 10 and 11. Both clear finishes show good adhesion when stoved at elevated temperatures, but fair to bad adhesion at lower stoving temperatures. Finishes based on acrylic resin *H*, which has the highest molecular weight and hardness, show the least adhesion, possibly as a consequence of a lower degree of surface wetting during flowout.

The effect of pigmentation on adhesion can be described as favourable in the lower temperature range and as unfavourable at higher stoving temperatures. All pigmented systems show an optimum stoving schedule for good adhesion, which moves to a lower temperature range when the stoving time is extended. With regard to adhesion it should

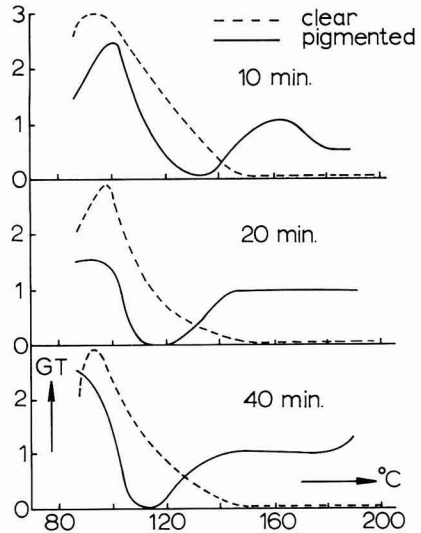


Fig. 10. Cross-cut adhesion of acrylic finishes H

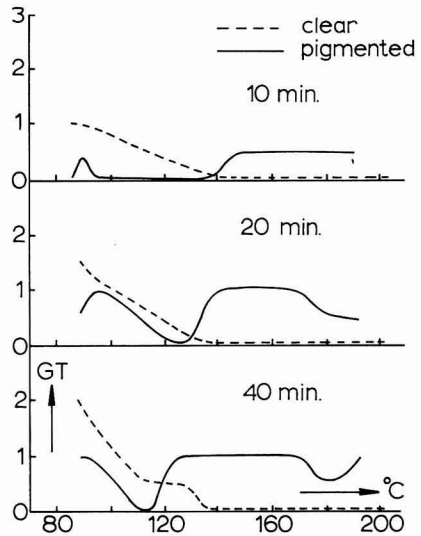


Fig. 11. Cross-cut adhesion of acrylic finishes F

be noted that the results obtained do not necessarily mean that the same effects will be found in a complete paint system.

Figures showing the temperature dependency of gloss are not given because the effect of temperature is relatively small. Generally, a slight tendency can be observed for a decrease in gloss at higher stoving temperatures and extended stoving times.

The relationship between the yellowness of both finishes (on a steel substrate) and temperature is shown in Fig. 12. There is a tendency for the film steadily to increase in yellowness with time and with temperature of stoving. It is evident

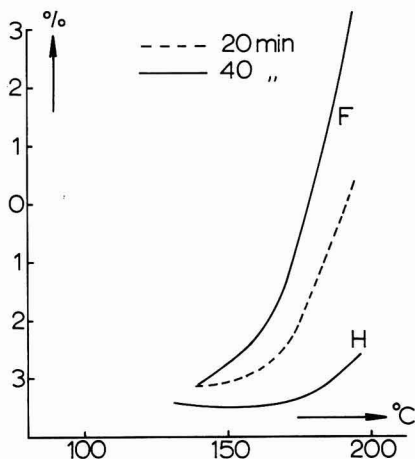


Fig. 12. Yellowness of acrylic finishes H and F

from Fig. 12 that finishes based on the flexible acrylic resin are the most susceptible to yellowing. Resistance to natural weathering after exposure for up to 24 months has been determined by measuring gloss retention and chalking. The gloss behaviour of the finishes *H* and *F* is shown in Figs. 13 and 14. Both acrylic finishes are first attacked in the range corresponding to higher stoving temperatures or obvious overcure and later in the range of low stoving temperatures or obvious undercure. After exposure for two years, the temperature for maximum weather resistance is found to be 135°C for acrylic finish *H* and 130°C for *F*. A certain tendency to recover gloss during winter has been noticed, especially for

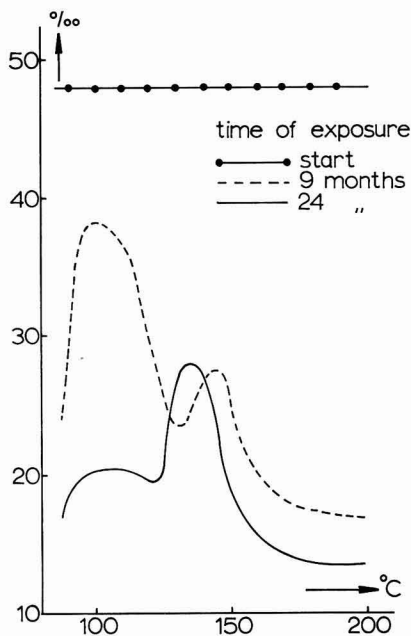


Fig. 13. Effect of stoving temperature on weathering of acrylic finishes H

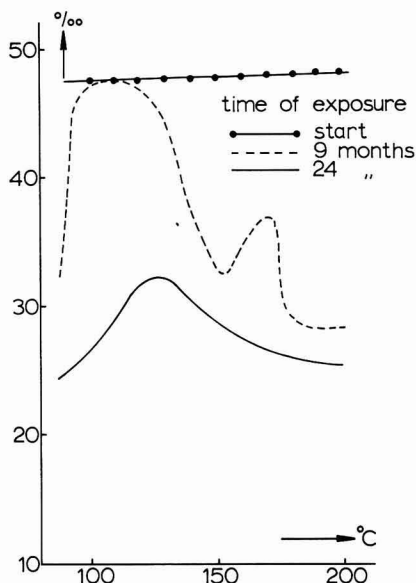


Fig. 14. Effect of stoving temperature on weathering of acrylic finishes F

the more flexible type *F*. This recovery corresponds to low stoving temperatures characterised by a low degree of cross-linking. From Table 3, which gives the chalking characteristics of both acrylic finishes, far less convincing conclusions may be drawn. It can be seen that weather resistance of type *F* is better than that of type *H* in most cases.

Table 3  
Chalking of pigmented acrylic finishes

Time of exposure (months)	90°C		130°C		200°C	
	H	F	H	F	H	F
6	0	0	0	0	0	2
9	4	2	4	3	6	3
15	6	2	6	3	6	4
18	9	9	6	8	9	8
24	9	9	8	9	9	9

#### Optimisation of stoving temperature

A new series of experiments with pigmented material was carried out. After stoving for a fixed time of 30 minutes, a number of mechanical properties was measured and the results plotted against temperature of stoving in one Fig. Such Figs offer a general view of the separate properties and can be used to optimise stoving conditions for a set of desired properties. As examples, data concerning hardness, extensibility, adhesion and impact resistance of acrylic finishes *H* and *F* are given in the Figs. 15 and 16. These will be analysed in more detail.

At lower stoving temperatures, acrylic finish *H* (Fig. 15) shows acceptable-to-good hardness and a fair adhesion; the flexibility and impact resistance, however, are entirely unsatisfactory. Hardness and adhesion improves simultaneously as the stoving temperature increases and the coating becomes



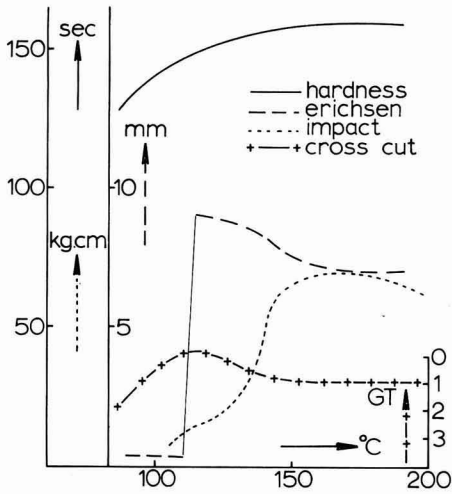


Fig. 15. Physical-mechanical properties of acrylic finishes H

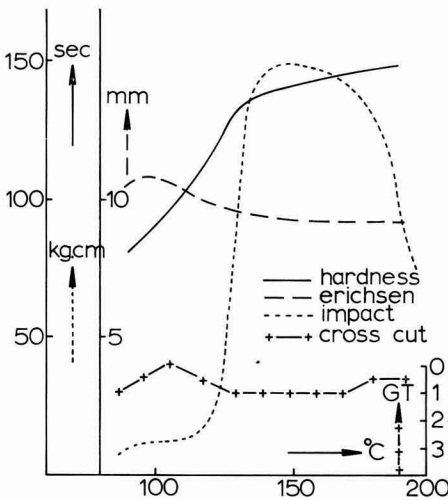


Fig. 16. Physical-mechanical properties of acrylic finishes F

more highly reacted. A sudden manifestation of flexibility is found at the optimum temperature for adhesion (110°C). At a still higher temperature (135°C) the impact resistance becomes manifest. It is apparent from Fig. 15 that all the properties considered remain nearly constant at temperatures between 140 and 200°C. This means that for the chosen stoving time (30 minutes), the stoving temperature can be chosen arbitrarily within this interval. In practice, of course, the operator will usually choose a stoving temperature at the lower end of the range to save energy, although not so low as to leave no margin for error. A slightly higher stoving temperature than the minimum will provide a safety factor against accidental fluctuations in object temperature.

From a comparison of the stoving results for the more flexible acrylic F (Fig. 16) with those for acrylic H (Fig. 15),

it can be seen that its hardness is poorer and its flexibility, adhesion and impact resistance are better. Another difference between the two acrylic finishes is the greater decrease in impact resistance for resin F at higher temperatures. The range over which the stoving temperature of acrylic F can be chosen at the fixed stoving time of 30 minutes is here restricted to 135-180°C.

The recommended stoving schedules for both these acrylics (30 minutes at 150°C) agrees rather well with the findings from experiments using the gradient stove.

**Structural properties**

The change in paint film properties during stoving can be considered to depend on the mode and extent of the underlying chemical reactions. A certain degree of chemical crosslinking forms part of this process. In order to gain some insight into the build up of structure in the acrylic paints, the degree of crosslinking and the modulus of elasticity in the rubbery state were determined, both after equilibrium swelling of the film in acetone. The results obtained are shown in the Figs. 17 and 18 for acrylics H and F. Comparison of these Figs. shows that the hard acrylic H crosslinks to a greater extent than the more flexible acrylic F. The swelling data, which can be considered as the counterpart of the modulus of elasticity, confirm this statement. From the swelling data in Figs. 17 and 18 and from the fact that the modulus of elasticity could not be determined, the conclusion can be drawn that clear finishes crosslink to a lesser degree than the corresponding pigmented ones. The curves showing the relation between degree of crosslinking and stoving temperature both show an optimum (at 175°C for acrylic H and at 145°C for acrylic F). The relationship discussed may be used to find out to what extent a paint film property depends on, or rather reflects, the network structure formed.

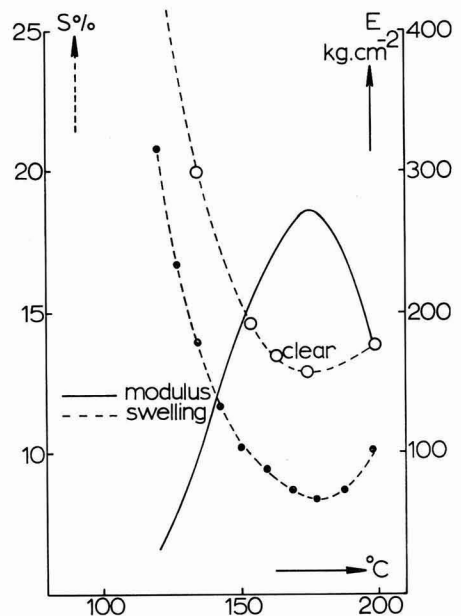


Fig. 17. Swelling and degree of crosslinking of acrylic finishes H

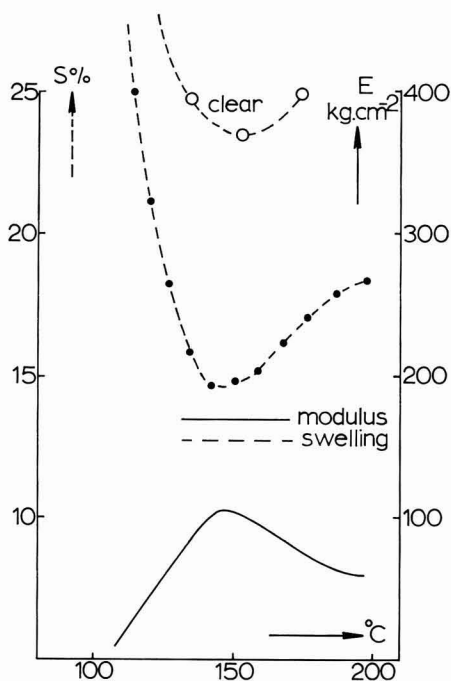


Fig. 18. Swelling and degree of crosslinking of acrylic finishes F

This may be done in two different ways:

- (i) by plotting the property to be investigated against the degree of crosslinking, thus eliminating the temperature, or
- (ii) by plotting both properties against the common temperature and comparing them.

As an example of the first method, the relationship between hardness and the degree of crosslinking for both acrylics is given in Fig. 19. For the sake of comparison, the same relationships for two alkyd/melamine finishes differing only in the reactivity of the melamine component are also shown. From Fig. 19, it can be seen that the acrylic finishes are characterised by a greater hardness at the onset of crosslinking (Young's modulus = 0 kg cm<sup>-2</sup>). Their higher molecular weight, compared with that of the alkyd/melamine mixtures, is well reflected in this Fig., as is the difference in molecular weight between the two acrylics.

In the same way, other paint film properties may be intimately connected with the degree of crosslinking, for example, extensibility and impact resistance. The occurrence of a maximum in crosslinking for acrylic finishes, however, causes in most cases rather complicated Figs. In such cases better and more useful results can be obtained by using the second method, and several interesting results have been obtained:

1. The onset of crosslinking corresponds, at the one hand to a termination of brittleness in resin type H and on the other, to a maximum of extensibility in type F.

2. The onset of crosslinking corresponds to greatest adhesion and to the start of a rapid increase in impact resistance for both types.

3. Maximum resistance to natural weathering occurs at a degree of crosslinking which is approximately half the maximum value.

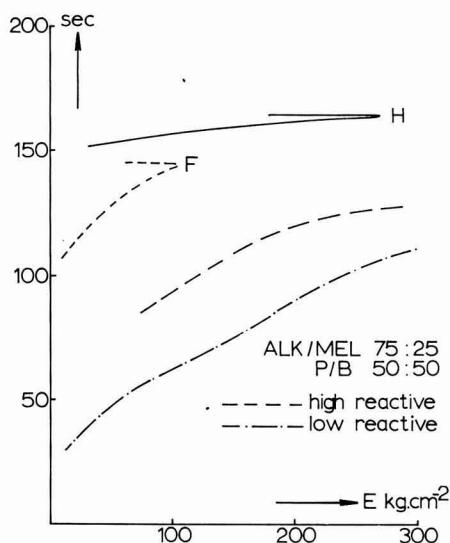


Fig. 19. Relation between hardness and degree of crosslinking

## Conclusions

A new type of laboratory stove developed at TNO has been introduced. Details of its construction, mode of operation, reliability and fields of application have been given. The gradient stove is recommended primarily as a convenient means of studying coatings which need thermal treatment to develop the desired properties. Possible uses of the stove outside the paint field have been indicated. The saving in time and the large amount of useful and reliable information which can be obtained from a very restricted number of stoving experiments are considered to be the most important advantages of the new apparatus.

A knowledge of the temperature dependency of various paint film properties can be used to formulate a paint of specific formulation so that stoving conditions are optimised. By elimination of the temperature factor, relationships between film properties can easily be obtained.

Some of the possibilities have been illustrated by examples of the results obtained with a hard acrylic and a flexible self-hardening finish. The most striking results of this investigation proved to be the constant nature of paint film properties over a large temperature range and the necessity for a certain amount of crosslinking to provide for the development of the various physical and mechanical properties of the film and resistance to weathering.

### Acknowledgments

The author wishes to thank Ir R. G. de Lange, Director of the Paint Research Institute TNO, as well as the members of the sponsoring research group of the Dutch paint industry, for their permission to publish this paper.

Thanks are also due to colleagues and members of the sponsoring group for valuable comments and to Mr M. Hoeflaak and Mr C. Roos for their able assistance in carrying out the experimental parts of the work described.

[Received 24 April 1976]

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# Preparation and properties of paint films with special morphological structure\*

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## Summary

By phase separation processes during film formation, paint films with special morphological structures are formed. The incompatibility attained during film formation may be caused by the increasing concentration of higher boiling non-solvents or by condensation of water at the surface of the liquid paint film. In the case of a mixture of polymer powders, the inherent incompatibility of the

resin components becomes obvious when the powders are melted during film formation. The onset of phase separation is reflected in the rate of evaporation of the solvent components. The morphological structure obtained depends on the time of phase separation and gel formation. Possible advantages of such paint films are indicated.

## Keywords

*Processes and methods primarily associated with drying or curing of coatings*

film formation  
solvent evaporation

*Properties, characteristics and conditions primarily associated with:*

*materials in general*

microporosity  
phase behaviour

*dried or cured films*

film discontinuity

## La préparation et les caractéristiques des films de peintures à structure morphologique particulière

### Résumé

Grâce aux processus de séparation de phase qui se produisent au cours de la formation du film, sont formés des films de peintures à structure morphologique particulière. L'incompatibilité mise en évidence lors de la formation du film pourrait être provoquée par la concentration croissante de diluants à point d'ébullition élevé, ou par la condensation d'eau à la surface du film de peinture liquide. Dans le cas d'un mélange de polymères en poudre, l'in-

compatibilité inhérente des constituants résineux devient évidente lorsqu'on fait fondre les poudres au cours de la formation du film. Le début de la séparation de phase se reflète dans le taux d'évaporation des constituants du solvant. La structure morphologique obtenue se dépend du temps de la séparation de phase et de la formation de gel. On indique les avantages éventuels de tels films de peinture.

## Herstellung und Eigenschaften von Lackfilmen mit spezieller morphologischer Struktur

### Zusammenfassung:

Lackfilme mit speziellen morphologischen Strukturen werden durch einen Phasentrennungsprozess während der Filmbildung erzeugt. Die während der Filmbildung erreichte Unverträglichkeit kann durch Erhöhung der Konzentration höher siedender Nichtlöser oder durch Kondensation von Wasser an der Oberfläche des flüssigen Lackfilms verursacht werden. Bei einer Mischung von polymeren Pulvern wird die den Harzkomponenten innewohnende

Unverträglichkeit deutlich, wenn die Pulver bei der Filmbildung geschmolzen werden. Der Beginn der Phasentrennung spiegelt sich in der Verdampfungszahl der Lösungsmittelkomponenten wieder. Die erhaltene morphologische Struktur ist von der Dauer der Phasentrennung und der Gelbildung abhängig. Angaben über die möglichen Vorzüge solcher Lackfilme werden gemacht.

## Introduction

*Refs. 1-6*

For many years it has been a common opinion in the paint field that binder materials, as used in paint formulations, lead to films which have a homogeneous amorphous structure. Consequently, it would be expected that film properties are constant throughout the film section from the surface to the substrate.

Of course, some exceptions have been recognised, for example it is well known that film formation by oxidative drying reactions is considerably retarded in the layer adjoining the substrate. This is shown by the tackiness of the lower surface of alkyd resin films when they are detached from the

substrate. Another indication is the upward bending as a result of internal stresses which develop during drying on a thin flexible substrate<sup>1</sup>.

More recently, it has been pointed out that the chemical composition and the packing density of those macromolecules in the film which are adjacent to the substrate may differ from that in the bulk and, of course, from that at the surface<sup>2</sup>. The different chemical structure of that part of the film which is at the interface with the substrate is considered to be caused by preferential adsorption of special binder molecules or of certain chemical groups of these polymers, which show a special affinity to the substrate. Some evidence exists that these adsorption processes may, in turn, cause a long range structure formation, which extends into the bulk film beyond the dimensions of the primary adsorption layer<sup>3</sup>.

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

Analogous to this, an adsorption-induced structure formation may also be initiated by the adsorption of polymer molecules at the surface of the pigment particles within the paint film<sup>4, 5</sup>. The relatively large surface area of the pigment particles, especially at higher PVCs, may require a major part of adsorbing sites present on the polymer molecules of the binder. As a consequence of this, the chemical composition would be expected to change over the distance between neighbouring pigment particles and, correspondingly, the morphological structure of the polymeric binder matrix would be expected to vary.

Since during paint production and film formation the pigment particles always have the first opportunity to adsorb, the vehicle to some extent may be depleted of adsorbable polymer molecules or groups. This depletion may have some effect on interfacial properties which depend on adsorption phenomena, such as adhesion to the substrate.

However, interesting as these observations may be, very little attention has been paid to the morphological structure of the polymer matrix of a paint film and its technological relevance. In order to define the structure of polymers, a micro- and macrostructure should be distinguished. The microstructure is represented by the conformations of the polymer chains and their arrangement to larger structural units or domains. The macro- or morphological structure consists of the size, shape and amount of such domains having special microstructures and of voids, as well as of their spatial arrangement and distribution.

Of course, this classification is best suited to characterise partially crystalline polymers. However, in non-crystalline polymers—which includes most binders for paint films—a large variety of less ordered domains of polymer molecules have been observed, and the spatial arrangement of these gives rise to many different morphological patterns.

Though being intrinsically related to the molecular structure and composition of the polymer molecules, the macrostructure is strongly influenced by the way a polymer passes from the liquid state in a melt or solution to the solid state or, in terms of paint technology, by the conditions and type of paint application and film formation.

The following discussion will be confined to structures in paint films which are formed during the evaporation of solvents and during the melting process of polymer powders. Even if allowance is made for the fact that in future air pollution problems will restrict the use of organic solvents, nevertheless the formation of organic coatings inevitably implies the liquid state as a starting point. It may be safely assumed, therefore, that a study of the fundamental mechanisms, that is how morphological structures are formed during the drying process of a film, will not lose its importance.

For a long time it has been an axiom of paint formulation that the binder material should be completely compatible in the solvent system of the paint. The main criterion for compatibility is a clear appearance of the solution and the absence of any turbidity. Moreover, no incompatibility or phase separation should occur during film formation.

More recently, with the development and application of water-borne organic coatings, this axiom has lost some of its validity. Many water soluble binders now consist of polymers, at least part of which are very close to incompatibility.

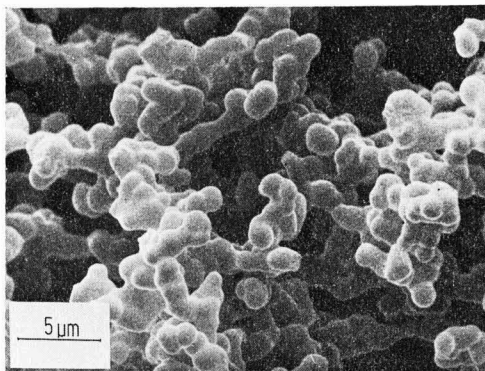
The appearance on the market of microcellular coatings<sup>6</sup>, which are also known as bubble, pitted or microvoid coatings, is another example that the occurrence of incompatibility does not necessarily lead to "pathological" coatings which are technically unsuitable for use. Moreover, microcellular polymer films have already gained great practical importance in other technical applications, for example as asymmetric membranes for desalination by reverse osmosis.

### Formation of films containing microvoids by phase separation during film formation

*Refs. 7-17*

Solvent evaporation phenomena have been extensively studied, more recently by many authors<sup>7-14</sup>. When solvents evaporate from polymer solutions containing a mixture of low-boiling solvents and high boiling non-solvents, incompatibility and phase separation occurs at some time during film formation, depending on the original solvent/non-solvent composition and on the temperature. The special type of microcellular structure resulting from phase separation depends on the time after which the system becomes incompatible during film formation.

If incompatibility occurs very early, phase separation presents itself as a precipitation of the polymer from the solution. If the polymer is in its glassy state after evaporation of the solvents, then finally a powdery incoherent polymer layer is formed (Fig. 1). On approaching the gel point, a transition to an open-porous but still coherent film is observed (Fig. 2). If phase separation takes place after gelation, the film becomes closed at its surface and a non-porous surface layer develops which is free from voids and which increases in thickness the later phase separation occurs (Fig. 3). If the film reaches the glassy state before incompatibility sets in, a normal clear film results. The sequence of these events is schematically illustrated in Fig. 4.



**Fig. 1.** Section of a PVCl/PVAc copolymer film (10% solution in MEK/n-butanol=2:3): intermediate stage between powder and open porous layer

The kinetics of evaporation has been studied by evaporation analysis in the case of unpigmented films of vinyl chloride/vinyl acetate copolymers<sup>15</sup>. For this purpose a gas chromatograph was used and a special cell for a controlled and well-defined evaporation process was developed. Experimental details will be given in a later publication. By means of this apparatus, the amount and composition of the evaporated

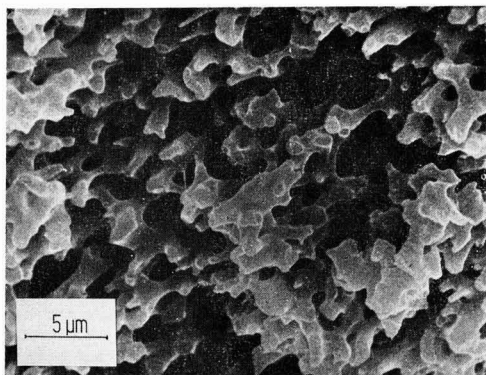


Fig. 2. Section of a PVCi/PVAc film (10% solution in MEK/n-butanol = 3 : 7): open porous structure

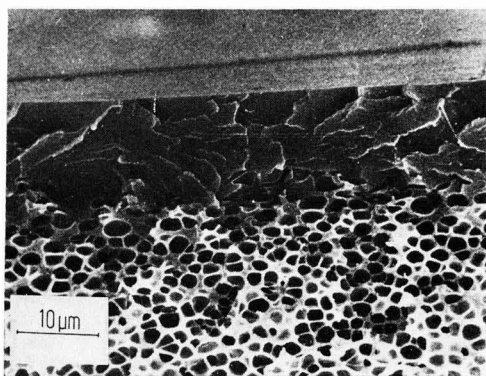


Fig. 3. Section of a PVCi/PVAc film (20% solution in MEK solution in MEK/n-butanol = 3 : 7): microvoid structure with clear, void-free surface layer

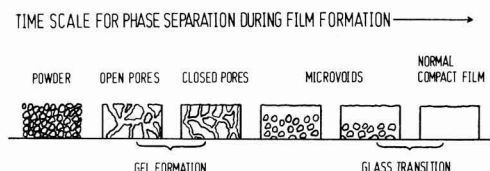


Fig. 4. Schematic diagram illustrating the sequence of structure formation at different times of phase separation

solvent/non-solvent mixture can be followed very accurately over the whole process of film formation.

In Figs. 5 and 6 the relative weight loss by solvent (methyl ethyl ketone) and non-solvent (n-butanol) evaporation, respectively, is plotted versus the drying time, which has been reduced to unit dry film mass. The initial polymer concentration chosen was 10 per cent by weight in each composition of solvent/non-solvent solution. The non-solvent fraction was varied from 0 to 40 per cent, corresponding to curves 0 to 4.

As may be seen in Fig. 5, the influence of phase separation after gelation, though not very distinctly revealed, is expressed by the increasingly reduced evaporation rate of the solvent

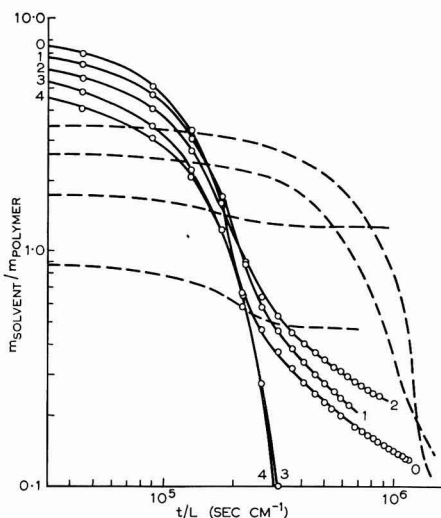


Fig. 5. Weight loss solvent versus drying time (reduced to unit binder mass)

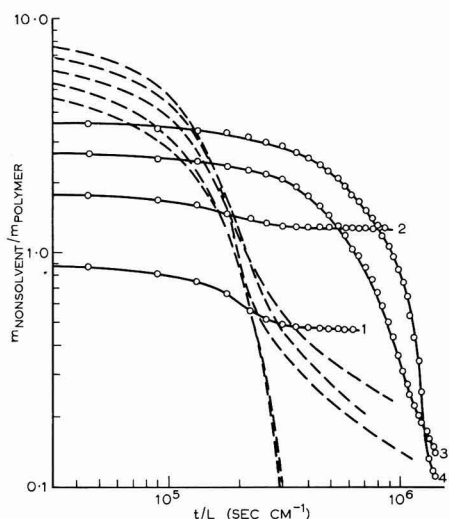


Fig. 6. Weight loss non-solvent versus drying time (reduced to unit binder mass)

at longer drying time (curves 1 and 2). This shows that by phase separation after gelation, which leads to a microcellular structure (Fig. 3), the residual solvent too evaporates at a reduced rate.

Curves 3 and 4 represent the evaporation of the solvent from a solution composed of higher fractions of non-solvent. Here phase separation sets in before or within the gelation range, resulting in an open-porous structure (Figs. 1 and 2), which allows unhindered solvent evaporation.

The evaporation of the non-solvent is presented in Fig. 6. When starting from an initial fraction of 10 or 20 per cent by weight of non-solvent, the evaporation of the non-solvent

practically ceases after phase separation has occurred (curves 1 and 2 after longer drying times). Obviously, the non-solvent is now retained within the incoherent liquid phase; this retention becomes even more pronounced when the initial fraction of non-solvent is increased. Finally, phase separation sets in before gelation and the open-porous structure thus formed greatly facilitates evaporation of the residual non-solvent. Due to the lower volatility and incompatibility of the non-solvent, the change of the microvoids to the open-porous structure still more distinctly enhances the evaporation than in case of the more volatile solvent.

It is not only the non-solvent which moves into the liquid phase when the two-phase system is formed, but at the same time a considerable amount of residual solvent still present in the polymer accompanies the non-solvent in order to attain phase equilibrium. Accordingly the polymer matrix is brought to a higher degree of drying within a short time and it becomes much more difficult for material, especially non-solvent, to leave the internal phase by diffusion to the film surface.

Phase formation and separation as a result of changing composition of the solvent/non-solvent mixture during film formation not only influences the bulk properties of the films, but also those properties which depend on the interface<sup>16</sup>. It can be conceived that if the affinity of the non-solvent for the substrate exceeds that of the polymer, some of the internal phase containing predominantly non-solvent comes into contact with the substrate and the non-solvent spreads at the substrate/film interface. As a consequence of this, adhesion is strongly diminished or even completely lost. This lack of adhesion may even be observed with films having no visual indication of being composed of two phases, that is when formed from solutions of a higher solvent/non-solvent ratio. Because of the low diffusion rates at this advanced stage of film formation, the solvents evaporate from the interface very slowly. This situation could be overcome by drying at higher temperatures, but if the polymers are thermoplastic this endangers the preservation of the microcellular structure.

A similar situation can arise when phase separation takes place in pigmented films. In this case, the pigment/binder interaction may be interrupted by a liquid layer which consists predominantly of non-solvent. It would be worthwhile to consider how often phase separation occurring at late stages of film formation is overlooked, perhaps due to pigmentation or to some other reason; inadequacies in the practical performance of paint films might be explained with reference to this effect.

Normal microcellular coatings have the drawback of being thermally unstable due to the thermoplastic character of the binder. Recently good thermal durability has been achieved by using binder compositions consisting of a thermoplastic and a crosslinked component. The microvoids can be incorporated into either one or both of these components<sup>17</sup>.

### Formation of paint films with a special surface structure due to presence of water

Refs. 18, 19

In a consideration of the influence of non-solvents on the formation of morphological structures in paint films which have been prepared from solutions of vinyl chloride/vinyl acetate copolymer, chlorinated rubber or cellulose nitrate in organic solvents, the non-solvent water takes a special position. A well-known film defect, known as "blushing" or "blooming", appears on evaporation of low-boiling solvents

from a polymer solution when the humidity of the surrounding atmosphere is not too far from saturation point. The effect is an opalescent or an even white appearance of the film. The reason for it is due to the latent heat of evaporation of the solvent, which causes the film to cool down and water to condense at the surface. An inspection of the film section by electron microscopy reveals that a thin layer of an open, porous void structure has been formed at the film surface (Fig. 7). Two mechanisms for the formation of this structure

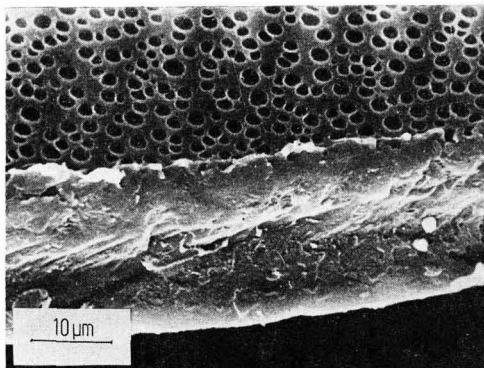


Fig. 7. PVC/PAc film with open porous surface layer (20% solution in MEK, wet film thickness 200  $\mu\text{m}$ , temperature 20°C, relative humidity (RH) 80%)

may be proposed: either by contraction of the condensed water film to droplets, which sink into the still liquid polymer film; or by dissolution of condensed water in a surface layer, followed by phase separation as a consequence of incident incompatibility. Another unsolved problem has been why in presence of atmospheric humidity, in some cases a microcellular structure could be observed not only at the surface but throughout the thickness of the film (Fig. 8).

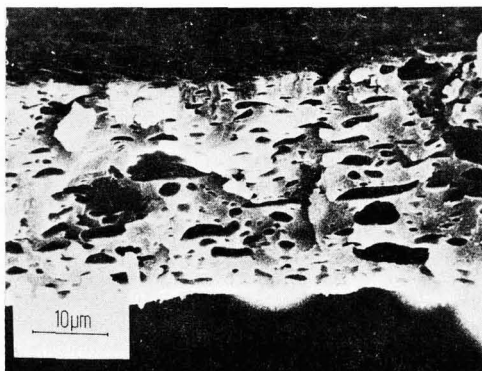


Fig. 8. Chlorinated rubber film with void structure throughout the film due to the Benard-mechanism (20% solution in dioxane, wet film thickness 200  $\mu\text{m}$ , temperature 20°C, RH 80%)

In order to find out how the surface structure is formed, a series of organic solvents, each of different compatibility with water, was used for dissolving the binder material. It could be shown that the characteristic structure which caused the white appearance of the films was formed only if

solvents with a certain degree of water compatibility were used. With hydrophobic solvents, even low-boiling ones, a structure formation could not be observed, despite a considerable temperature decrease due to evaporation. This result clearly implies that a dissolution and phase separation mechanism is responsible for structure formation at the film surface.

In order to find out why structure formation can also occur throughout the film, water was added up to the limit of compatibility in the formulation of the polymer solution. The film obtained after evaporation of the solvents showed a white appearance similar to that of a film dried without addition of water prior to film formation. An inspection of the film sections under the scanning electron microscope, however, showed that in the case of the film which had had water added to the formulation, the microvoid structure was formed throughout the film, in much the same way as with solvent/non-solvent mixtures (Fig. 9). This means that despite comparable optical effects, the internal structure of the films causing these effects may be significantly different.

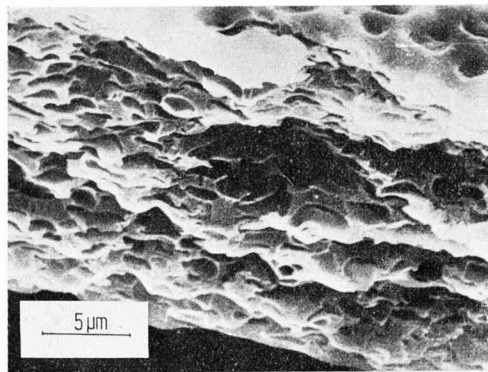


Fig. 9. Cellulose nitrate film with void structure throughout the film due to addition of water close to the limit of compatibility (20% solution in acetone and water, wet film thickness 200  $\mu\text{m}$ , temperature 20°C)

By variation of the film thickness and viscosity and selection of solvents of different volatility and also by using electron microscope techniques, it can be shown that depending on the above mentioned experimental parameters, the Benard cell mechanism becomes effective, by which the condensed water is distributed throughout the whole film<sup>18</sup>.

In contrast to polymer films, in which microvoid structure has been caused by incompatibility in the residual solvent/non-solvent mixture, films with microcellular surface layers caused by water condensation were always found to be open-porous. By analogy with the microcellular films, open-porous surface structures obtained under the influence of water may be simply considered as a consequence of incompatibility, starting first at the film surface and then proceeding into the interior during later stages of film formation. Thus a similar sequence of structures is observed, beginning at the film surface with open pores, followed by a small closed pore region and finally ending some way into the film with the normal void-free bulk material.

As has been indicated earlier with paint films having an open-porous surface structure, a very good adhesion of successive layers may be obtained by mechanical anchoring in the pores of the layer immediately below<sup>19</sup>. This opens the possibility of producing two-layer coating systems of films of different polymers, even when they exhibit very poor intercoat adhesion. The only requirement is that the solvents used when applying the second layer, must not dissolve the first layer.

In comparison with bubble coatings, which contain microvoids throughout their section, films having open-porous surface structures may provide a similar hiding power when applied to a substrate, but at the same time avoid the rather strongly reduced adhesion to the substrate, which can occur in the case of bubble coatings by non-solvent extending at the interface on phase separation.

### Formation of two-layered paint films on metallic substrates from mixtures of polymer powders

Ref. 20

The limited compatibility of polymers in the melt may be used for the preparation in one operation of powder coatings composed of several layers. Suitably formulated powder mixtures consisting of both crosslinkable and thermoplastic polymers will separate in two horizontal layers during heating. If a mixture of an epoxy and a polybutyl methacrylate powder is used, the epoxy resin layer, which has a good adhesion to the substrate, accumulates at the oxidised metal surface, whereas the weather resistant layer of the thermoplastic polymer separates to the surface.

Differences in the densities of the liquid phases may be responsible for the vertical sequence of layers. It was found, for example, that during the evaporation of a diluted solution of polystyrene and polyvinyl acetate in the same solvent, the direction of phase separation occurring at some critical concentration corresponded to the different specific weights of the two phases. During the separation of the molten powder mixture, however, no clear relation could be found between the direction of separation and the different densities of the polymer powders.

Infrared analysis<sup>20</sup> of a section of film which had been sandwiched between a tin foil and a glass substrate (Fig. 10) disclosed that the epoxide resin component was enriched at the interfaces to the tin (C) as well as the glass substrate (A). It may be concluded, therefore, that different affinities of the polymer components for the substrate surface are important. Obviously, in this case the viscosity is too high to allow the relatively small differences in density to become effective. The additional crosslinking of one of the polymer components promotes the separation; however, it should not occur too rapidly in order to leave sufficient time for the thermoplastic component to separate.

All the various possibilities discussed above for obtaining paint films with unusual morphological structures, and accordingly special film properties, are based more or less on paint defects long known in paint technology. An intentional application of these effects requires a better knowledge



Powder Mixture : Epoxide Resin/Acrylate Resin (1/1)

Film Forming Condition: 15 min/180°C

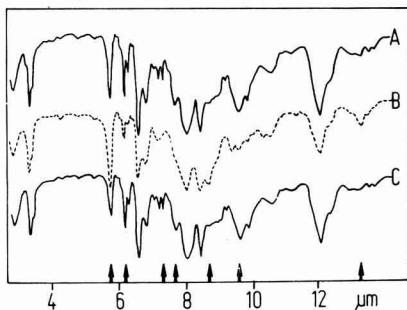
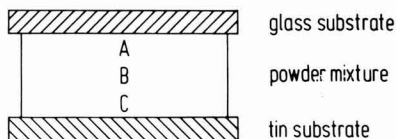


Fig. 10. Phase separation of a mixed powder polymer layer (epoxide/methacrylate resin) sandwiched between glass plate and a tin foil after film formation (15 minutes at 180°C): infrared analysis of upper, middle and lower sections of the film

of the mechanism involved and this again may help to avoid such paint defects when they are not wanted.

[Received 24 April 1976

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# The effect of wet time on accelerated outdoor exposure\*

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## Summary

A variety of materials has been exposed on "EMMAQUA" accelerated weathering machines using three different spray cycles in an attempt to determine the effect of wet time on those accelerated weathering results which correlate well with other test methods.

Preliminary results indicate that an extended total wet time does produce a colour change and loss of gloss more rapidly than standard spray or wet/dry cycling. The end results of long-term testing (320 000 langleys or about 64 days), however, reveal that

wet/dry cycling may be as important as total wet time in the degradation process.

This paper reports colour change and gloss retention results obtained from the exposure of several industrial and automotive finishes, and also the percentage total transmission of transparent free films. The results are correlated with data from other test methods, in cases where these data are available.

## Keywords

*Equipment primarily associated with analysis, measurement or testing*

weatherometer

*Processes and methods primarily associated with analysis, measurement or testing*

accelerated weathering

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

fade resistance

gloss retention

weather resistance

## L'effet du "wet time" sur les essais de vieillissement accélérés à l'extérieur

### Résumé

Une gamme diverse de matériaux a été exposée dans un appareil "EMMAQUA" de vieillissement accéléré qui comporte trois différents cycles d'arrosage, en vue de déterminer l'effet qu'exerce le "wet time", c'est-à-dire la durée où la plaque reste mouillée, sur les résultats des essais de vieillissement accéléré qui s'accordent bien avec d'autres méthodes d'essai.

Les résultats préliminaires indiquent, dans le cas où la durée cumulative est prolongée où la plaque reste mouillée, que l'altération de couleur et la perte de brillance se produisent plus rapidement que dans le cas où on utilise un arrosage ou un cycle sec/humide dont tous les deux sont normaux. Pourtant, les résultats

ultimes des essais à longue durée (320 000 langleys ou 64 jours à peu près) mettent en évidence qu'à l'égard du processus de dégradation, l'exposition aux cycles sec/humide pourrait être aussi importante que la durée cumulative où la plaque reste mouillée.

Cet exposé rend compte des résultats sur l'altération de couleur et sur la retention de brillance obtenus à partir de l'exposition de diverses peintures industrielles ou pour automobiles, et d'ailleurs on donne la transmission totale en pourcentage des feuil transparents. Les résultats sont mis en rapport avec les données rendues par d'autres méthodes d'essai, dans les cas où ces données sont disponibles.

## Die Auswirkung der Nasszeit auf Aussen-Knrzprüfung

### Zusammenfassung:

Eine Anzahl von Materialien wurden auf "Emmaqua" beschleunigten Bewitterungsapparaten unter Benutzung von dreierlei sich unterscheidenden Spritzzyklen exponiert, um den Einfluss der Nasszeit auf solche durch Kurzprüfung erzielte Resultate zu ermitteln, welche mit anderen Prüfmethode übereinstimmen.

Vorläufige Versuchsergebnisse deuten an, dass anhaltende, totale Nasszeit schneller Farbtonveränderung und Glanzverlust verursacht, als Standard Spritz- oder nass/trocken Perioden. Nach Dauerprüfung (320 000 Langleys oder etwa 64 Tagen) zeigen die

Endergebnisse jedoch, dass nass/trocken Perioden in dem Abbauprozess ebensolche Bedeutung haben können, wie totale Nasszeit.

In dieser Abhandlung werden nach Bewitterung verschiedenert Industrie- und Automobillacke erhaltene Resultate hinsichtlich Farbtonveränderungen und Glanzhaltung berichtet, ebenso wie der Prozentsatz der Gesamttransmission transparenter, freier Filme. Die Resultate werden mit den Zahlenwerten aus anderen Prüfmethode, soweit sie zur Verfügung stehen, in Beziehung gebracht.

## Introduction

Refs. 1-2

For more than three quarters of a century, scientists have been investigating the deterioration of materials exposed to the weather. During the last fifty of those years, accelerated weathering devices of one type or another have been used by the coatings industry in an attempt to reduce the time

required for obtaining weatherability data from real-time tests<sup>1</sup>.

The EMMAQUA (Equatorial Mount with Mirrors for Acceleration with water) illustrated in Fig. 1 has been used successfully since late 1959 as a test for determining quickly the durability of materials destined for outdoor use. Successful as this machine has been, and whilst good correlation with

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

real-time tests has been established<sup>2</sup>, it is known that the eight-minute-per-hour standard spray cycle is not the most effective one for all the various materials now being exposed on these machines. It was believed correlation would be better and results obtained more quickly if the samples experienced longer wet time during the exposure period, and a research project was initiated. Samples for the programme were supplied by seven companies located in four countries. A total of 297 specimens was examined for this report.

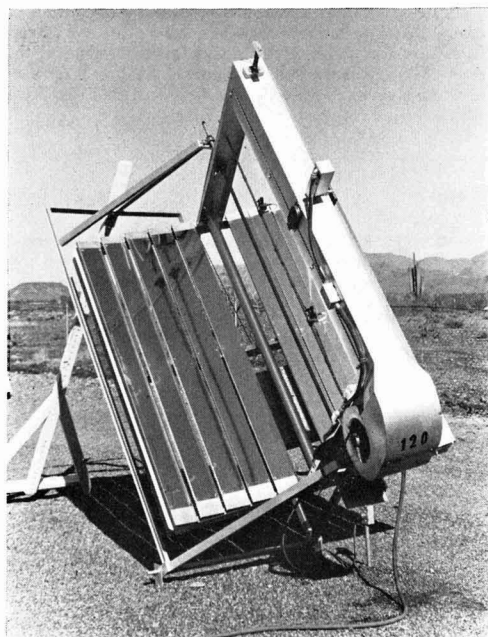


Fig. 1. The EMMAQUA solar concentrator

### Choice of spray cycle

The standard spray would naturally account for one of the cycles in order to determine the relationship between the experimental machines and the standard EMMAQUA. Samples exposed on the standard EMMAQUA are sprayed for eight consecutive minutes during each hour of operation, in all cases with steam-distilled water.

### Wet/dry cycling

Even though a great deal of importance has been placed on total wet time, it was believed desirable to determine in addition the effect of wet/dry cycling as a factor in accelerating the degradation process. The cycle used for this part of the programme would have to be timed so that the water spray thoroughly wetted the samples, and yet allowed sufficient time for the samples to dry completely and attain maximum surface temperature. The 12/15 cycle was selected for this purpose: samples exposed on the machines modified to accommodate this cycle were sprayed for twelve 15-second periods each hour, and were allowed to dry for four minutes and 45 seconds between sprayings. The actual surface wet time was about 30 seconds per cycle, or six minutes per hour.

### Wet/wet cycling

Another important objective of the programme was to establish the effect of extended wet time and to determine whether the results of EMMAQUA exposures could be correlated more closely with those obtained from real-time tests in wetter areas of the globe. Before such a cycle could be designed, it was necessary to know the climatic conditions in other areas. These data are presented in Table 1.

Table 1  
General climatological data (by courtesy of BTP Tioxide Ltd, Cleveland)

Location	Climate	Water (mm per year)	Temperature (°C)
Teesside	N. temperate Maritime	55	8.5
Durban	Sub-tropical Continental	101	20.5
Johannesburg	Sub-tropical	75	15.8
Melbourne	S. Temperate	66	14.8
Miami	Sub-tropical	152	23.8
Phoenix	Desert	16	20.8
EMMAQUA	Accelerated (Natural)	Sprayed for 8 minutes every hour	50.0

Weather reports from Teesside and Miami indicate that these areas average 5.4 and 4.2 hours of wet time daily. It was obvious from this information that samples exposed at those locations experienced considerably longer periods of wetness than on EMMAQUA (1.5 hours per day), and the experimental wet/wet cycle should increase the total wet time, therefore, to approximate the conditions at one of these stations. In addition, it was important that the dry period following spraying was short, so that the sample surfaces would not remain completely dry for more than a few seconds, and the surface temperatures would not return to maximum until the complete cycle had ended.

From a study of the 12/15 cycle, it was known that panel surfaces were saturated and experienced water run-off after 15 seconds of spray. Complete surface drying did not occur on average for another 15 seconds, and up to 30 seconds were required in some cases.

The 15/45 cycle was selected as the second experimental spray. Samples exposed on these EMMAQUAs were sprayed for thirty-two consecutive 15-second periods each hour, and allowed to dry 45 seconds between sprayings. An extended dry period of 28 minutes each hour allowed for complete drying and maximum temperature build-up.

### Characteristics examined

During the planning stages of the programme, it was decided to limit the sample characteristics monitored to those which could be measured instrumentally, in order to remove the possibility of errors from subjective visual evaluations. Fortunately, the first samples received in response to the authors' request were in the aesthetic category; that is, appearance was as important to the supplier as physical changes. This simplified the job of selecting the data to be reported; colour change and gloss retention were chosen. Inspections were conducted at 80 000-langlely intervals—roughly equivalent to six months' conventional exposure in Florida.

**Colour change**

*Ref. 2*

Colour measurements were made according to ASTM Standard Method D2244, using a Hunter D-25DA Color and Color Difference Meter. Colour difference ( $\Delta E$ ) was calculated by colour scale system B, that is:

$$\Delta E = [(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2]^{0.5} \dots \dots \dots (1)$$

However, for the industrial finishes it was necessary to compute the colour difference by the Glasser *et al.* cube-root colour coordinate system, as this was the system used to determine colour changes in the panels exposed in Florida. The algorithm for computation may be expressed as:

$$\Delta E = [(\Delta L_G)^3 + (\Delta a_G)^3 + (\Delta b_G)^3]^{0.5} \dots \dots \dots (2)$$

**Gloss**

Gloss readings were taken as specified in ASTM Standard Method D523 at 60° with Gardner Multi-Angle Gloss Meter and results presented as per cent retention.

**Exposure information**

Florida exposure data for the industrial finishes and data for all EMMAQUA tests are listed in Table 2. The panels of automotive finishes—Series I were exposed for a total of 18 months in Florida between 20 February 1973 and 19 January 1976 for approximately 220 000 langleys. The Xenon cycle used to obtain the results reported was 18 minutes wet, 102 minutes dry at 70–75 per cent constant relative humidity, with a 60°C black body temperature. The Xenon was equipped with a No. 7 cam. Florida data are not available for automotive finishes—Series II. However, the panels were exposed for 12 months at 5° south, and it may be assumed they received approximately 160 000 langleys. Average black panel (dial thermometer) temperatures for the DSET facility in New River, Arizona are given in Table 3.

**Results**

Colour difference and gloss retention data for individual panels are listed in Tables 4–16. Table 17 gives the total transmission data on the three transparent free films.

*Table 2*  
*Exposure data*

	Solar radiation 45°	5°	Wet time (hours)		Average panel (°F)	Black temperature (°C)
<b>Miami, Florida</b>						
(Industrial finishes)						
1st six months (16/2/72 to 16/8/72)	79 325	84 063	1341.0		104.8	40.5
2nd six months (29/12/72 to 29/6/73)	73 086	71 086	973.0		105.8	41.0
3rd six months (1/8/73 to 1/2/74)	65 580	66 139	1423.0		103.8	39.9
Eighteen month total	216 991	221 288	3737.0		103.8A	40.5A
(Automotive finishes: series I)			(Estimates based on above data)			
Eighteen months (between 20/2/73 and 19/1/76)		220 000	3737.0		103.8A	40.5A
	Solar radiation	Std	12/15	15/45		
<b>EMMAQUA (Summer)</b>						
(Industrial finishes—Automotive finishes: series I) (Free film)						
1st period 16 days (16/4/75 to 1/5/75)	80 000	24.0	14.4	76.8	144.5	62.6
2nd period 17 days (9/5/75 to 26/5/75)	80 000	25.5	20.4	81.6	150.5	65.9
3rd period 15 days (31/5/75 to 15/6/75)	80 000	22.5	18.0	71.8	165.5	74.2
4th period 16 days (19/6/75 to 5/7/75)	80 000	24.0	14.4	76.8	165.5	74.2
64-day totals	320 000	96.0	67.2	307.0	156.5A	69.2A
<b>EMMAQUA (Autumn)</b>						
(Automotive finishes: series II)						
1st period 15.5 days (15/8/75 to 30/8/75)	80 000	23.4	13.9	74.7	166.0	74.5
2nd period 21.5 days (4/9/75 to 22/9/75)	80 000	27.0	20.3	108.0	156.0	68.9
3rd period 16.5 days (24/9/75 to 10/10/75)	80 000	24.6	14.9	79.2	146.5	63.7
4th period 15.5 days (21/10/75 to 5/11/75)	80 000	23.4	13.9	74.7	134.5	57.0
69-day totals	320 000	98.4	63.0	366.6	150.8A	66.0A

Table 3  
Average black panel temperatures (New River Arizona)

Month	EMMAQUA		45°		5°		Ambient	
	(F°)	(°C)	(°F)	(°C)	(°F)	(°C)	(°F)	(°C)
Jan	117.5	47.5	120.0	48.9	108.0	42.3	66.5	19.2
Feb	126.5	52.5	119.0	48.4	115.0	46.1	70.0	21.1
Mar	118.5	48.1	127.0	52.8	125.5	52.0	66.5	19.2
Apr	144.5	62.6	132.5	55.9	135.5	57.5	81.5	27.5
May	150.5	65.9	136.0	57.8	151.5	66.4	82.5	28.1
Jun	165.5	74.2	151.0	66.2	161.0	71.7	106.0	41.1
Jul	157.5	69.8	141.0	60.6	147.0	63.9	106.0	41.1
Aug	166.0	74.5	148.0	64.5	156.0	68.9	106.5	41.4
Sep	156.0	68.9	128.5	53.7	132.5	55.9	99.5	37.5
Oct	146.5	63.7	123.0	50.6	123.0	50.6	79.0	26.1
Nov	134.5	57.0	121.5	49.8	105.5	40.9	75.0	23.9
Dec	126.5	52.5	120.0	48.9	102.0	38.9	69.5	20.9
Yearly average	142.5	61.4	130.6	54.8	130.2	54.6	84.0	27.3

Table 4  
Industrial finishes: panel description

Panel No.	Description
1	Light blue acrylic on steel
2	Medium dark brown silicone polyester on steel
3	Medium blue vinyl on steel
4	White silicone polyester on steel
5	White fluoropolymer on aluminum
6	Dark blue silicone acrylic on aluminum
7	Medium dark blue acrylic on steel
8	Medium dark green acrylic on steel
9	Medium green acrylic on steel
10	Light green melamine on steel

Table 5  
Colour difference for industrial finishes: EMMAQUA spray cycle comparisons

Panel No.	80 000 Langley (16 days)			160 000 Langley (33 days)			240 000 Langley (48 days)			320 000 Langley (64 days)		
	Std	12/15	15/45	Std	12/15	15/45	Std	12/15	15/45	Std	12/15	15/45
1	0.5	0.6	0.9	2.4	4.3	2.9	3.8	6.5	5.2	5.9	8.1	10.1
2	0.8	0.6	2.1	1.3	1.4	3.4	1.7	1.8	4.3	2.5	2.3	5.4
3	0.8	0.5	0.8	3.5	3.4	2.5	10.9	9.6	7.8	11.8	14.6	16.5
4	0.2	0.1	1.1	0.2	0.6	2.2	0.5	1.3	2.0	1.0	2.6	4.0
5	0.3	0.2	1.4	0.9	2.2	4.2	2.1	6.8	6.0	1.7	4.8	8.5
6	1.1	1.0	3.2	1.9	2.6	4.6	2.8	4.5	5.7	5.3	8.7	10.0
7	1.3	1.1	1.4	2.3	1.6	1.7	2.4	3.8	2.7	4.1	6.0	4.9
8	0.4	0.5	0.5	0.8	2.0	0.6	1.8	3.7	0.7	3.2	5.8	2.9
9	0.4	0.5	0.6	1.0	1.8	0.7	1.8	4.7	1.2	3.3	7.4	3.3
10	2.1	2.2	2.2	2.6	3.0	2.6	4.1	5.2	2.8	6.2	8.0	4.9

Table 6  
Industrial finishes: Colour differences for EMMAQUA/Florida correlation

Panel No.	80 000 Langley (16 days) Florida—6 Months						160 000 Langley (33 days) Florida—12 Months						240 000 Langley (48 days) Florida—18 Months					
	90s	5s	45s	Std	12/15	15/45	90s	5s	45s	Std	12/15	15/45	90s	5s	45s	Std	12/15	15/45
1	0.8	1.1	1.2	0.5	0.6	0.9	1.4	3.0	2.8	2.4	4.3	2.9	2.7	3.2	3.1	3.8	6.5	5.2
2	0.2	1.2	1.1	0.8	0.6	2.1	0.7	2.4	1.6	1.3	1.4	3.4	1.6	2.8	2.5	1.7	1.8	4.3
3	1.8	1.9	1.7	0.8	0.5	0.8	3.0	3.4	2.3	3.5	3.4	2.5	3.1	4.0	3.9	10.9	9.6	7.8
4	1.7	2.8	3.1	0.2	0.1	1.1	1.5	2.3	3.1	0.2	0.6	2.2	1.8	2.9	3.2	0.5	1.3	2.0
5	0.4	0.8	0.7	0.3	0.2	1.4	0.2	0.3	0.1	0.9	2.2	4.2	0.1	0.1	0.1	2.1	6.8	6.0
6	1.5	2.4	1.5	1.1	1.0	3.2	2.3	4.1	3.5	1.9	2.6	4.6	3.9	7.8	6.5	2.8	4.5	5.7
7	1.4	2.1	1.5	1.3	1.1	1.4	1.6	3.1	2.6	2.3	1.6	1.7	2.1	4.2	4.0	2.4	3.8	2.7
8	1.0	0.7	1.1	0.4	0.5	0.5	0.6	1.9	1.7	0.8	2.0	0.6	2.1	3.4	1.8	3.7	3.7	0.7
9	0.5	0.7	0.6	0.4	0.5	0.6	0.5	1.4	1.1	1.0	1.8	0.7	1.4	2.8	2.5	1.8	4.7	1.2
10	1.4	2.1	1.7	2.1	2.2	2.2	2.2	3.4	3.4	2.6	3.0	2.6	3.4	4.6	4.5	4.1	5.2	2.8

Table 7  
Percentage retention for industrial finishes—60° gloss: EMMAQUA spray cycle comparisons

Panel No.	80 000 Langleys (16 days)			160 000 Langleys (33 days)			240 000 Langleys (48 days)			320 000 Langleys (46 days)		
	Std	12/15	15/45	Std	12/15	15/45	Std	12/15	15/45	Std	12/15	15/45
1	108	113	101	73	47	84	41	23	68	24	17	44
2	87	83	61	52	57	39	65	65	30	57	52	22
3	57	71	57	57	33	43	14	14	19	10	10	10
4	88	83	54	83	71	42	79	63	33	63	50	25
5	110	107	90	107	100	90	103	97	84	97	84	71
6	75	75	58	75	71	54	67	42	42	50	13	13
7	72	67	47	53	47	36	36	22	25	19	14	14
8	85	87	72	70	48	63	50	11	50	30	9	35
9	96	98	83	86	70	74	68	28	63	47	17	53
10	114	108	111	94	67	103	69	33	94	36	14	57

Table 8  
Percentage retention for industrial finishes—60° gloss: EMMAQUA/Florida correlation

Panel No.	80 000 Langleys (16 days) Florida—6 Months						160 000 Langleys (33 days) Florida—12 Months						240 000 Langleys (48 days) Florida—18 Months					
	90°	5°	45°	Std	12/15	15/45	90°	5°	45°	Std	12/15	15/45	90°	5°	45°	Std	12/15	15/45
1	96	98	101	108	113	101	91	53	56	73	47	84	54	45	43	41	23	68
2	101	88	96	87	83	61	100	72	88	52	57	39	91	73	76	65	65	30
3	75	52	68	57	71	57	31	34	32	57	33	43	15	12	12	14	14	19
4	81	83	90	88	83	54	80	135	69	83	71	42	78	83	76	79	63	33
5	94	101	105	110	107	90	96	100	99	107	100	90	102	112	106	103	97	84
6	83	70	51	75	75	58	99	80	79	75	71	54	102	60	71	67	42	42
7	73	48	38	72	67	47	66	45	49	53	47	36	55	63	64	36	22	25
8	84	77	77	85	87	72	84	73	72	70	48	63	80	76	70	50	11	50
9	101	98	100	96	98	83	100	94	97	86	70	74	91	79	78	68	28	63
10	83	78	84	114	108	111	69	46	53	94	67	103	45	29	32	69	33	94

Table 9  
Panel description for automotive finishes: series I

Panel No.	Description*
1	Beige
2	Dark yellow
3	Dark blue metallic
4	Blue (purple)
5	Orange
6	Silver green metallic
7	Green metallic
8	Red
9	Burgundy metallic
10	Pewter metallic
11	Dark green metallic
12	Blue metallic
13	Gold metallic
14	Blue metallic
15	Copper metallic
16	Plum metallic

\*All systems non-aqueous dispersion acrylics on steel panels.

Table 10  
Colour difference for automotive finishes—series I: EMMAQUA spray cycle comparisons

Panel No.	80 000 Langleys (16 days)			160 000 Langleys (33 days)			240 000 Langleys (48 days)			320 000 Langleys (64 days)		
	Std	12/15	15/45	Std	12/15	15/45	Std	12/15	15/45	Std	12/15	15/45
1	0.2	0.2	0.2	0.3	0.2	0.2	0.2	0.5	0.5	0.6	0.2	0.4
2	0.6	0.5	0.4	0.7	0.7	0.6	0.8	1.0	1.0	1.5	1.6	1.6
3	0.6	1.0	1.9	1.1	2.1	2.8	2.3	3.3	4.0	3.8	6.0	6.0
4	0.8	2.0	1.8	1.7	3.9	3.5	3.3	6.1	5.1	6.5	11.6	9.9
5	0.5	0.7	1.6	0.7	1.5	2.1	2.2	3.0	3.7	3.8	4.6	4.9
6	0.2	0.4	0.7	0.7	0.8	1.1	0.3	0.5	0.9	0.9	1.2	1.6
7	1.2	1.2	1.6	1.6	1.8	2.1	2.6	2.7	2.5	3.7	4.2	3.1
8	1.2	1.4	2.5	1.5	1.9	3.7	3.0	3.6	5.8	5.0	5.8	7.1
9	0.3	0.5	2.4	1.8	2.5	3.6	6.6	7.6	7.8	9.1	10.5	8.4
10	0.2	0.4	0.3	0.6	0.9	0.7	1.0	1.2	0.4	1.5	1.7	1.3
11	1.1	1.0	1.4	1.6	1.8	2.3	2.6	2.6	2.8	4.3	4.2	3.6
12	0.5	0.1	0.6	0.9	0.8	1.4	1.4	1.3	2.0	2.6	3.4	3.6
13	0.3	0.4	0.3	0.5	0.5	0.6	0.9	1.0	1.2	1.2	1.2	1.6
14	0.4	0.4	0.7	1.0	0.9	1.3	1.4	1.5	1.9	2.6	2.7	3.2
15	0.4	0.4	1.2	0.7	0.8	1.8	2.3	2.2	3.2	3.5	3.7	4.7
16	1.8	1.2	3.0	2.6	2.4	4.4	4.8	5.4	6.6	8.3	9.0	9.3

Table 11  
Colour difference for automotive finishes—series I: EMMAQUA—XENON arc correlation

Panel No.	80 000 Langleys (16 days) Xenon—500 hours				160 000 Langleys (33 days) Xenon—1000 hours				Florida—18 Months 24 000 Langleys (48 days) Xenon—1500 hours					
	Xenon	Std	12/15	15/45	Xenon	Std	12/15	15/45	Florida 5°	Xenon	Std	12/15	15/45	
1	0.5	0.2	0.2	0.2	0.5	0.3	0.2	0.2	0.4	0.6	0.2	0.5	0.5	
2	0.7	0.6	0.5	0.4	0.6	0.7	0.7	0.6	1.3	1.4	0.8	1.0	1.0	
3	2.8	0.6	1.0	1.9	2.3	1.1	2.1	2.8	4.1	3.2	2.3	3.3	4.0	
4	1.3	0.8	2.0	1.8	1.2	1.7	3.9	3.5	4.4	2.6	3.3	6.1	5.1	
5	1.7	0.5	0.7	1.6	2.0	0.7	1.5	2.1	2.8	2.7	2.2	3.0	3.7	
6	0.9	0.2	0.4	0.7	1.0	0.7	0.8	1.1	1.5	1.3	0.3	0.5	0.9	
7	1.9	1.2	1.2	1.6	1.5	1.6	1.8	2.1	2.2	2.6	2.6	2.7	2.5	
8	1.7	1.2	1.4	2.5	1.6	1.5	1.9	3.7	2.8	3.1	3.0	3.6	5.8	
9	3.2	0.3	0.5	2.4	2.5	1.8	2.5	3.6	6.5	4.7	6.6	7.6	7.8	
10	2.3	0.2	0.4	0.3	2.0	0.6	0.9	0.7	1.1	1.5	1.0	1.2	0.4	
11	2.4	1.1	1.0	1.4	1.4	1.6	1.8	2.3	3.3	2.6	2.6	2.6	2.8	
12	0.9	0.5	0.1	0.6	0.9	0.9	0.8	1.4	2.2	2.1	1.4	1.3	2.0	
13	0.7	0.3	0.4	0.3	0.2	0.5	0.5	0.6	0.9	0.4	0.9	1.0	1.2	
14	1.5	0.4	0.4	0.7	1.8	1.0	0.9	1.3	2.6	2.1	1.4	1.5	1.9	
15	2.4	0.4	0.4	1.2	2.6	0.7	0.8	1.8	2.3	2.8	2.3	2.2	3.2	
16	3.1	1.8	1.2	3.0	2.6	2.6	2.4	4.4	6.0	3.3	4.8	5.4	6.6	

Table 12  
Percentage retention for automotive finishes—series I. 60° gloss: EMMAQUA spray cycle comparisons

Panel No.	80 000 Langleys (16 days)			160 000 Langleys (33 days)			240 000 Langleys (48 days)			320 000 Langleys (64 days)		
	Std	12/15	15/45	Std	12/15	15/45	Std	12/15	15/45	Std	12/15	15/45
1	96	93	74	92	78	72	86	74	69	70	65	62
2	98	93	88	93	78	80	82	59	66	60	43	46
3	96	96	80	92	81	69	85	70	58	74	56	56
4	98	94	84	93	80	76	79	57	62	52	34	45
5	105	102	87	100	87	79	85	68	70	58	38	64
6	95	93	81	87	76	64	73	59	50	56	40	32
7	96	94	80	88	82	65	63	40	51	34	20	34
8	98	96	81	94	88	69	82	76	62	67	53	49
9	98	97	78	93	85	71	55	43	65	25	19	51
10	93	87	76	81	73	62	58	51	51	33	29	36
11	96	95	77	87	84	67	65	60	64	40	37	53
12	94	91	77	86	84	67	76	74	61	60	56	50
13	87	84	76	73	74	61	63	66	53	56	53	43
14	93	85	82	82	79	70	71	70	62	56	52	48
15	90	95	77	87	88	58	67	65	55	41	40	39
16	91	92	76	84	84	67	65	66	55	39	43	35

Table 13  
Percentage retention for automotive finishes—series I, 60° gloss: EMMAQUA|XENON arc correlation

Panel No.	80 000 Langleys (16 days) Xenon—500 hours				160 000 Langleys (33 days) Xenon—1000 hours				Florida—18 months 240 000 Langleys (48 days) Xenon—1500 hours					
	Xenon	Std	12/15	15/45	Xenon	Std	12/15	15/45	Florida UW <sup>1</sup> W <sup>2</sup>		Xenon	Std	12/15	15/45
1	73	96	93	74	66	92	78	72	86	101	57	86	74	69
2	71	98	93	88	61	93	78	80	72	89	60	82	59	66
3	68	96	96	80	67	92	81	69	75	91	58	85	70	58
4	71	98	94	84	65	93	80	76	84	99	62	79	57	62
5	77	105	102	87	73	100	87	79	84	100	68	85	68	70
6	76	95	93	81	68	87	76	64	68	84	57	73	59	50
7	67	96	94	80	61	88	82	65	42	68	54	63	40	51
8	73	98	96	81	62	94	88	69	79	94	55	82	76	62
9	71	98	97	78	67	93	85	71	45	88	56	55	43	65
10	72	93	87	76	72	81	73	62	40	44	71	58	51	51
11	64	96	95	77	63	87	84	67	60	95	53	65	60	64
12	75	94	91	77	66	86	84	67	57	71	58	76	74	61
13	79	87	84	76	61	73	74	61	77	91	57	63	66	53
14	76	93	85	82	70	82	79	70	56	72	61	71	70	62
15	67	90	95	77	66	87	88	58	61	95	59	67	65	55
16	72	91	92	76	65	84	84	67	45	59	69	65	66	55

<sup>1</sup>UW = Unwashed; <sup>2</sup>W = Washed

Table 14  
Panel description for automotive finishes: series II (metallics)

Panel No.	Description*
1	Burgundy
2	Red-orange
3	Lavender
4	Lavender
5	Lime
6	Lime
7	Burgundy
8	Red-orange
9	Lavender
10	Lavender
11	Lime
12	Lime
13	Maroon
14	Maroon

\*Non-aqueous dispersion acrylics and alkyd melamines on steel panels.

Table 15  
Colour difference for automotive finishes—series II

Panel No.	EMMAQUA— 80 000 Langleys (15.5 days)		Florida—6 months EMMAQUA— 160 000 Langleys (37 days)			EMMAQUA— 240 000 Langleys (53.5 days)		EMMAQUA— 320 000 Langleys (69 days)	
	12/15	15/45	Florida	12/15	15/45	12/15	15/45	12/15	15/45
1	2.0	2.7	2.9	4.4	5.4	4.9	6.4	13.7	12.3
2	1.2	2.3	5.5	4.9	4.8	7.4	8.4	7.0	8.6
3	2.1	1.6	2.7	3.2	3.0	4.5	4.0	3.8	3.8
4	2.8	2.5	3.2	3.6	4.2	4.8	5.1	5.0	5.5
5	0.9	1.6	5.2	2.3	3.2	3.3	4.1	7.8	7.8
6	1.3	1.1	4.7	1.8	2.7	7.5	4.0	6.6	7.8
7	2.9	3.5	2.9	4.8	6.6	6.5	8.1	10.6	10.2
8	1.6	2.5	1.8	2.3	3.6	3.0	4.1	5.9	5.3
9	1.5	1.6	1.1	2.2	3.0	3.2	3.4	5.7	5.4
10	2.7	3.4	5.3	4.7	5.5	6.5	7.0	5.7	5.7
11	2.9	2.7	7.1	5.6	5.3	7.3	7.3	12.4	11.6
12	1.2	1.1	2.2	1.9	2.5	2.5	2.7	7.5	6.8
13	4.0	3.5	2.3	5.1	4.9	5.2	5.0	13.5	14.4
14	3.1	2.5	5.2	6.2	5.6	8.5	7.6	9.8	8.9



Table 16  
Percentage retention for automotive finishes—series II. 60° gloss

Panel No.	EMMAQUA—80 000 Langleys (15.5 days)		Florida—6 months EMMAQUA—160 000 Langleys (37 days)			EMMAQUA—240 000 Langleys (53.5 days)		EMMAQUA—320 000 Langleys (69 days)	
	12/15	15/45	Florida	12/15	15/45	12/15	15/45	12/15	15/45
1	90	81	93	83	69	73	62	63	56
2	91	84	72	63	62	37	38	12	22
3	94	88	99	85	75	77	74	72	69
4	81	82	95	80	70	73	68	69	64
5	88	82	86	82	68	74	66	68	64
6	88	86	85	82	68	71	65	63	59
7	87	84	84	74	60	62	51	37	38
8	90	85	92	82	69	74	66	67	62
9	88	84	89	78	70	69	68	62	66
10	88	82	87	78	70	67	67	57	64
11	85	81	75	67	61	58	55	46	46
12	86	80	91	79	65	67	62	53	55
13	88	83	75	78	67	70	55	43	49
14	92	92	78	68	61	45	40	22	27

Table 17  
Percentage transmission for free transparent film: EMMAQUA spray cycle comparisons

Film	80 000 Langleys (16 days)			160 000 Langleys (33 days)			240 000 Langleys (48 days)			320 000 Langleys (64 days)		
	Std	12/15	15/45	Std	12/15	15/45	Std	12/15	15/45	Std	12/15	15/45
Polyvinyl chloride <sup>1</sup>	89.3	88.6	89.2	87.4	85.4	86.4	84.9	84.6	83.3	82.6	82.5	79.5
Polypropylene <sup>2</sup>	91.1	88.5	90.4	88.8	84.3	86.4	87.9	82.1	85.9	86.9	80.3	83.7
Polyethylene <sup>3</sup>	89.5	89.0	89.1	87.1	87.6	86.7	86.5	86.4	86.7	86.9	85.6	83.9

<sup>1</sup>µm PVC initial transmission 87.9%      <sup>2</sup>µm PP initial transmission 92.2%      <sup>3</sup>µm PE initial transmission 90.6%

The plots presented in Figs. 2–13 are composites representing the average colour difference and percentage gloss retention values obtained at each inspection interval for each series.

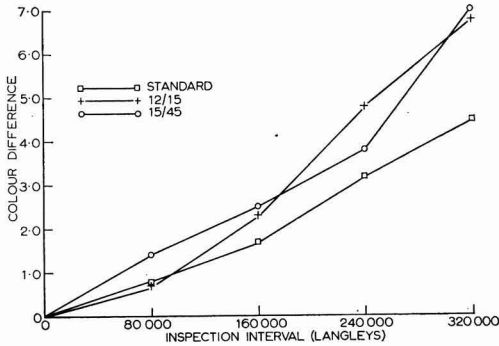


Fig. 2. Colour difference for industrial finishes: EMMAQUA spray cycle comparison

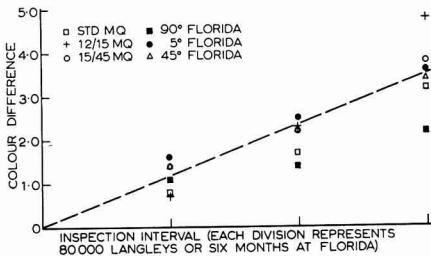


Fig. 3. Colour difference correlation for industrial finishes

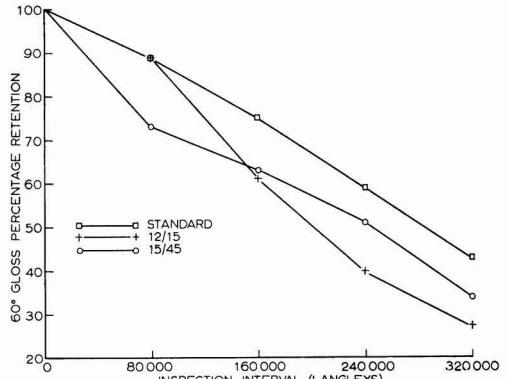


Fig. 4. Percentage gloss retention for industrial finishes: 60° gloss. EMMAQUA spray cycle comparison

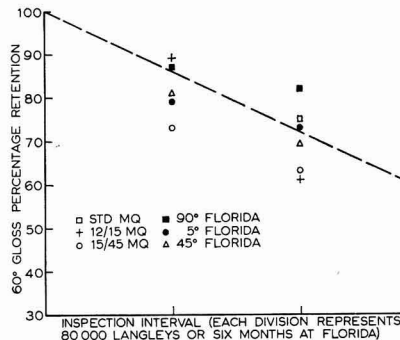


Fig. 5. Percentage gloss retention for industrial finishes—series I: 60° gloss. EMMAQUA/Florida correlation

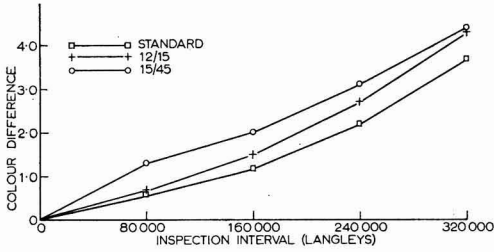


Fig. 6. Colour difference for automotive finishes—series I. EMMAQUA spray cycle comparison

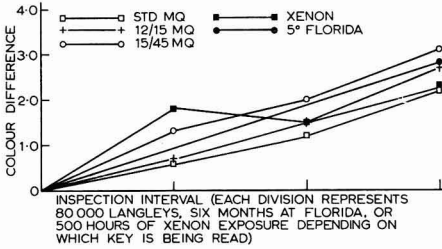


Fig. 7. Colour difference correlation for automotive finishes—series I.

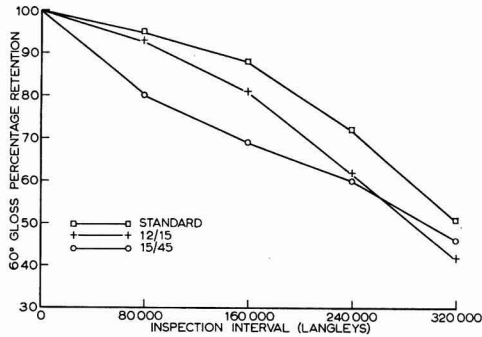


Fig. 8. Percentage gloss retention for automotive finishes—series 1: 60° gloss. EMMAQUA spray cycle comparison

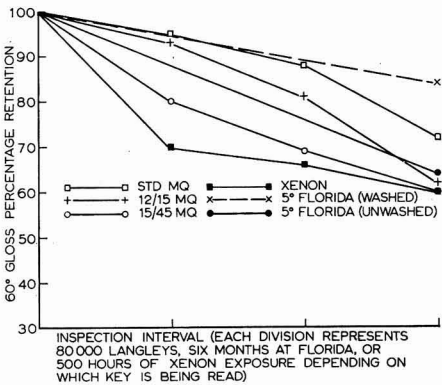


Fig. 9. Correlations of percentage gloss retention for automotive finishes—series I: 60° gloss

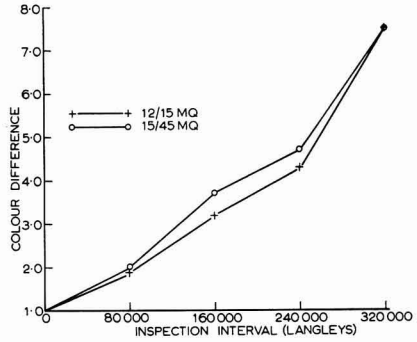


Fig. 10. Colour difference for automotive finishes—series II. EMMAQUA spray cycle comparison

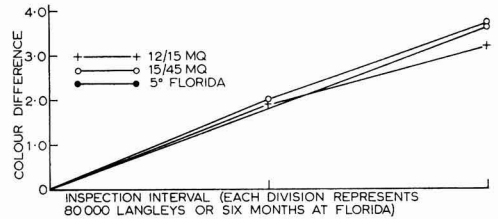


Fig. 11. Colour difference correlation for automotive finishes—series II.

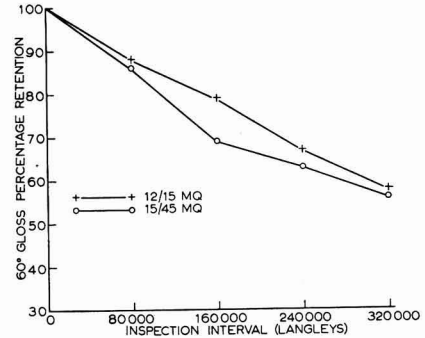


Fig. 12. Percentage gloss retention for automotive finishes—series II: 60° gloss. EMMAQUA spray cycle comparison.

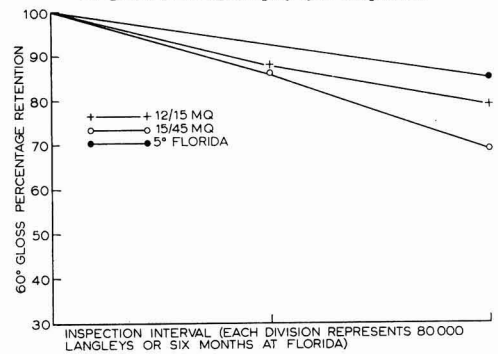


Fig. 13. Correlations of percentage gloss retention for automotive finishes—series II: 60° gloss.

Even though surface breakdown is not reported in these Tables and Figs., crazing/cracking, blistering, and chalking were observed. None of the panels involved exhibited signs of crazing/cracking or blistering at the completion of 320 000 langleys. All industrial finishes displayed chalk formation by the end of the test, regardless of the spray cycle. In most cases, chalking was heaviest on samples exposed to the 12/15 cycle, possibly because of wash-off on the Standard and 15/45 cycles. Chalk could be detected on only three of the automotive finishes at the completion of exposure. It is interesting to note that differences in colour, formulation, or substrate did not appear to affect the rate of degradation.

### Conclusions

In almost all instances, the data clearly indicate that the 15/45 experimental cycle dramatically reduces the induction period normally required for the onset of colour change and loss of gloss when compared with the 12/15 and Standard cycles. This is quite evident in the results given in the Tables for the 80 000-langley inspection, and is graphically illustrated when the plot shown in the Figs. for the same period is examined. However, at about 160 000 langleys, the 12/15 cycle is comparable with the 15/45 cycle in most instances

and sometimes even exceeds its performance. These results lead to the primary conclusion that wet/dry cycling may be as important a factor in the rate of deterioration as total wet time for the specimens used in this report. Tests with other materials are being continued and results will be made available as they are obtained.

### Acknowledgments

The authors would like to express their appreciation to the following companies for supplying panels and other test method data: American Motors Corp, Detroit, Michigan, USA; Goodlass Wall & Co. Ltd, Liverpool; Dr. Kurt Herberts & Co, Wuppertal, Germany; Motor Assemblies Ltd, Jacobs, Natal, South Africa; Tioxide International Ltd, Teesside; Volkswagenwerk AG, Wolfsburg, Germany.

[Received 3 June 1976]

### References

1. Stieg, F. B., *J. Paint Tech.*, 1975, **47**, (611), 54.
2. *Correlation Table* "Accelerated outdoor versus conventional outdoor weathering", Desert Sunshine Exposure Tests Inc., Phoenix, Arizona, USA.

## Next month's issue

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

Comparison of the effects of resin composition and pigments on the anticorrosive properties of powder coatings by *A. van de Werff*

Optical properties of films by *W. Carr*

Progress towards international agreement on gloss measurement of paint films by *U. Zorll*

Usefulness of the measurement of internal stresses in paint films by *A. Saarnak, E. Nilsson and L. O. Kornum*

## Section Proceedings

### Natal

#### Titanium dioxide air-drying gloss paints

A meeting of the Section was held at the Ocean Terminal Restaurant, Durban, on Tuesday 7 September 1976 when 34 members and two guests attended to hear a lecture given by Mr J. R. Gush of S.A. Tioxide (Pty) Ltd entitled "Titanium dioxide air-drying gloss paints".

Commercial, dry titanium dioxide pigments were sold in the form of naturally cohesive flocculated powder, with at least 75 per cent air spaces in the bags. In order to make a normal gloss paint, this air had all to be displaced and replaced by resin, ensuring that the pigment particles were separated and kept separate.

Calculations had shown that although primary pigment particles were cohesive, bulk pigment did flow in flocculates of about 50  $\mu\text{m}$  in diameter. These flocculates were easily broken

up during paint manufacture by hand stirring. However, particles which might be present in dry pigment were aggregates, which had to be broken up by the pigment manufacturing process, and agglomerates, which were formed by a cementing action occurring after calcination.

These particles had to be broken up by paintmaking equipment. In order to keep the pigment apart, a repulsive force had to be present. It had been estimated that in the case of alkyds only 20 per cent of this force was due to electrostatic repulsion, the remainder being due to steric repulsion between layers of resin stuck to the pigment surface.

High quality pigments for use in alkyd resins usually had a basic surface coated with alumina to form strong bonds at a few points with free carboxylic acid groups in the resin molecule.

The introductory talk was followed by the screening of a technically excellent film produced by Tioxide.

L.F.S.

# Information Received

## New synthetic resin emulsions plant to be constructed by Harlow Chemical Co.

Harlow Chemical Co. Ltd is beginning immediately the construction of a second works for the production of synthetic resin emulsions. This expansion has been sanctioned by its parent companies, Revertex Ltd and Hoechst UK Ltd, a subsidiary of Hoechst AG in Frankfurt.

The new works will be on a green-field site at Stallingborough, South Humberside. The initial phase of construction will create facilities for early expansion to 30,000 tonnes per annum of emulsions at Stallingborough, and the site will be suitable for further development in the 1980s.

The cost of the project through to initial production in 1978 will be at least £3 million. The investment, previously deferred, has now been brought forward because of a substantial grant from the Department of Industry under the Accelerated Projects Scheme.

Harco intends to award a contract for design, procurement, and construction to Matthew Hall Engineering Ltd, whose associated company, Norcain (Projects) Ltd, will be responsible for detailed design and project management. Engineers from Harco and from Hoechst AG will control the project.

## Carbon black dispersions

Colloids (Widnes) Ltd has announced an agreement with Runnymede Dispersions Ltd whereby both solvent and aqueous based, carbon black liquid dispersions formerly manufactured by Runnymede will be produced and marketed by Colloids (Widnes) Ltd.

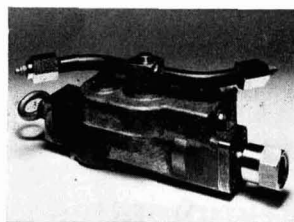


New headquarters for Jacobson Chemicals—the UK head office of R. S. Stokvis & Sons at East Molesey, Surrey, where Jacobson Chemical Division has now established its marketing organisation

## Inflatable air structure provides temporary covered storage space for major paint manufacturer

Goodlass Wall & Co. Ltd, manufacturer of decorative, industrial, automotive and marine paints and varnishes, has hired an 846m<sup>2</sup> inflatable air structure from Gott-hard Air Structures (UK) Ltd for installation at its Speke, Liverpool, works.

The structure, approved by the local fire and planning authorities, was chosen by Goodlass Wall to enable temporary covered storage space to be quickly provided. It's "skin" was tailor-made to the company's exact requirements from high frequency welded sheets of white, PVC coated, flameproof, light-stabilised fabric.



A new rotary cleaning head, model "TWK 18", from Reiss Engineering Co. Ltd, which is said to be particularly suitable for the cleaning of mixing tanks.

## Copper oxide rights

Mechem Ltd, London, has acquired the UK and Eire selling rights for the black copper oxide concentrate quarried and refined in Australia by the Poseidon Group.

Black copper oxide concentrate, now mined by open-cast techniques and refined by the specially developed "Samin" process is transported from Australia to Mechem's South Wales plant where it is a feedstock for chemicals produced by Mechem and used in the manufacture of pigments, ceramics, glass, electrical components, catalysts and also in animal foods and fertilisers. Mechem will also market the "Samin" product to other chemical manufacturers.

## UK distributors for ICI's non-textile

ICI Organics Division has appointed the Brico Pigment Sales Division of Keegan Brico Tetley Chemicals Ltd as UK distributor of ICI's non-textile colours for a range of customers in the paint, printing ink, plastics and rubber industries. This complements similar arrangements for textile colours between ICI Organics Division and the Keegan Textile Sales Division.

ICI will continue to be responsible directly for business with the larger colour users.



The DMC 26 colorimeter now available from Carl Zeiss. The company emphasises speed, high accuracy and reproducibility as features of the new instrument, which is claimed to be a "master instrument" for reflectance measurement and colour matching. Computer and digital plotter are from Hewlett Packard.

### New thinking in emulsion resins for the paint industry

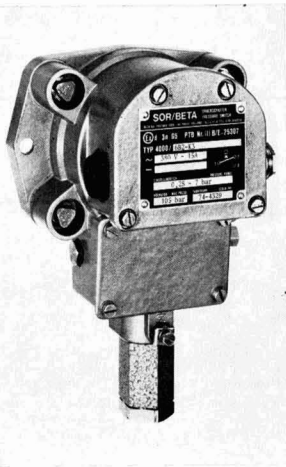
It is now one year since Kirklees Chemicals commissioned its emulsion resin plant, and already "Vinaking" has a reputation in the paint industry, not only in the United Kingdom but in other countries also, as being a highly versatile emulsion with uses in many different paint formulations from certain textured coatings to high PVC paints.

"Vinaking 80/20" is a copolymer containing 20 per cent VeoVa 10, and is offered in 50 and 55 per cent solids content form. The decision to produce a 20 per cent VeoVa type, although not marketed to any great extent before, has undoubtedly been a great success. Whilst the product has the usual good pigment binding properties to be expected from a VeoVa resin, the reduced level of VeoVa gives a harder film and this has been particularly useful to manufacturers of silk vinyls, who are looking for a tough and very glossy film in their paints.

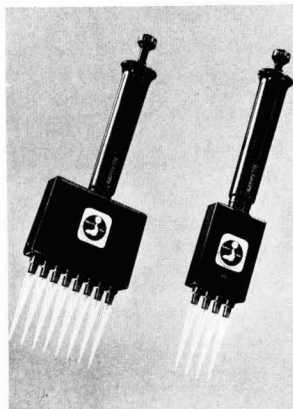
The new storage system for the finished product will ensure that Kirklees Chemicals keeps up with the increasing demand for its products and plant expansion is being planned for next year. This, coupled with BP Chemical's assurances of continued support in the supply of vinyl acetate monomer, will enable the company to serve a small but important part of the paint industry into the 1980s.

Development work in the company's own laboratories and the prospect of buying technology from overseas, mean that it is very likely that 1977 will see Kirklees Chemicals branch out into areas outside the paint industry.

### New products

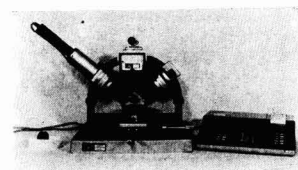


An explosion-proof pressure switch recently introduced to the UK by Loba (Industrial Products) Ltd



Two new multichannel pipettors with polypropylene tips: distributor in the UK is Jencons (Scientific) Ltd, Hemel Hempstead

### New ellipsometer for film thickness measurement



A new ellipsometer "Model LI17" from Gaertner Scientific Corporation for non-contact measurement of solid or liquid films.

The ellipsometer uses a laser light source and solid-state detector. Angles of incidence of source and detector can be quickly set at 30°, 50°, 70° and 90° by pin locations. The final thickness determination is by newly computed and corrected tables, but a mini-computer is also available for rapid print-out of results and a punch-card deck can be supplied for use with large computers.

### Specially coated vat heater

Eltron (London) Ltd has recently announced a new vat heater, which is both robust and specially coated to resist most commercial acids. It is believed that the heater will find particular application in the metal finishing industries. Ranges of the heater are from 1 kW to 4 kW (single phase) and from 3 kW upwards (three phase).

### Courses, symposia, etc.

#### Minerals processing

A symposium "Economic factors in minerals processing", organised by the Minerals Engineering Society, will be held on 29 and 30 March 1977 at the University of Nottingham.

### Colour course

Instrumental Colour Systems Ltd has announced details of its winter course in applied colour technology. This course is being held at the Aldermaston Court Conference Centre from Tuesday 30 November to Friday 3 December and is intended to be of interest to all those involved in the fields of colour management, colour specification and colour matching. The syllabus covers the subject from an introductory level through to the use of colorimeters and spectrophotometers linked to on-line computers. The fee for the course is £89.00 plus VAT (payment for accommodation is made separately).

### Literature

#### Wolfson Research Unit report

The Wolfson Organic Powders Research Unit, set up in the Department of Colour Chemistry, University of Leeds just over two years ago as the result of a grant from the Wolfson Foundation, has issued its second annual Research Report to subscribing members.

The report contains four papers on the non-confidential work of the Unit. Their titles are "Observations on aqueous dye dispersions at high temperatures—solubility and solution enthalpy of CI disperse yellow 1", "The use of a millipore filter as a support in scanning electron microscopy", "X-ray diffraction at elevated temperatures", and "Studies of the nucleation and growth of pigment dispersions using a laser light-scattering apparatus in a flow system". These originate from the main research programme of the Unit, which is concerned with the preparation of dyes, pigments and other organic powders in the size, shape and crystallographic form most suitable for their technological end-use.

In addition to this broad programme, the Unit also engages in short term investigations on a contract basis using the extensive range of facilities it possesses, to which has recently been added a Zeiss RFC3 colorimeter and an associated computer. This latter was acquired as the result of financial assistance from the Dyers' Company of London.



Two new brochures produced by Bee Chemical Co. to describe its "Injecta Color" system of liquid colourants for plastics

#### Powder coating review

R. H. Chandler Ltd has produced a 57-page booklet "Powder coating 1975" which summarises most of the articles and patents concerned with powder coating published during 1975.

# Reviews

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## Encyclopedia of Chemical Processing and Design. Volume 1

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Executive Director J. J. McKetta

New York and Basel: Marcel Dekker Inc.,

Pp XIII + 472. Price Sw. Fr. 300.00

The potential size of this mammoth project can be assessed when it is realised that this first volume covers only the initial letters *ab* to *ac*, in other words, from *abrasives* to *acrylonitrile*. However, it is possible to obtain an idea of the aim and scope of the work from this volume. The preface points out that this encyclopedia is not a dictionary nor a handbook, nor a series of specialised monographs. It is said to be written for readers planning to design or develop a process, but one wonders whether there are enough of these people and whether they really need such a vast work.

The subject matter of the first sixteen articles covers principles (*absorption*), chemicals (*acetaldehyde*), industrial raw materials (*acetal resins*), finished products (*acetate fibres*) and tools (*abrasives*).

The chapters on materials cover in the main, manufacture, properties and uses, as would be expected in an encyclopedia of industrial chemistry, but with the addition of a detailed account (with diagrams) of manufacturing plant design, and this is obviously to be the characteristic feature of this work. Two of the chapters are in the form of a student design problem (for the manufacture of acetic anhydride and acetone) with a model solution, and they seem a little out of place in a work otherwise so large. The remaining chapters have a number of original references, but not substantial bibliographies.

This work is obviously meant for the larger libraries and further volumes will be awaited with interest.

L. A. O'NEILL

## Handbook of analysis of organic solvents

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By V. Šedivec and J. Flek

(translation by H. Sommernitz)

Chichester: Ellis Horwood Ltd, 1976.

Pp. 455. Price £18.00

Some idea of the style of this book may be gathered from the fact that there is an appendix of 67 pages, giving lists of density, refractive index, optical dispersion, dielectric constant, vapour pressure, mutual miscibility and boiling points of solvents; this last mentioned list contains the boiling points of no less than 1662 compounds and azeotropes. The text itself, after some 90 pages of general principles, discusses the individual solvent classes; the various hydrocarbons, halogen derivatives, alcohols, ethers, ketones, aldehydes, esters, nitro-compounds, amines and sulfur compounds. It leaves nothing to chance and backs every statement with a reference—1012 of them in all. And here the book begins to get a little silly: the Lassaigne test gets two references—not to the original work, which would have been a charming piece of pedantry—but to two twentieth-century textbooks!

It is very difficult to know who is meant to use the book; very few laboratories will handle the whole range of solvents discussed, and those which do will not need the two pages spent on the setting-up of a distillation apparatus. No

doubt the appendices will be quite widely used, except for the jejune list of trade names. A serious omission is the lack of any mention of toxic hazard—not even phosgene!

The translation is good except for a few eccentricities, such as "digital index on a symbol" for "superscript".

M. H. M. ARNOLD

## Titanium dioxide production 1965-1975

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A patent review (TTIS Publication No. 6)

By R. H. Chandler Ltd., Braintree, Essex, 1976.

Pp. 138. Price £20.00

The reviewer feels it is doubtful whether this publication meets a real need for the information it contains.

The book is primarily of interest only to titanium dioxide producers (of whom there are relatively few) and, since the technical literature in this industry consists almost solely of patent specifications, those producers take care to ensure that they are aware of the content of all patent specifications published by their competitors as soon as possible after publication. It is not seen, therefore, how the publication under review can provide TiO<sub>2</sub> producers with information which they do not already possess, particularly since most of the specifications abstracted have been available to the public for a number of years.

It is always a problem when attempting to obtain useful information from patent specifications in any particular field, that only a minor proportion of such specifications disclose products or processes which are in use, or which are likely to be used, and it is not normally possible for an author or a reader who is not engaged in the industry concerned to select those specifications which disclose such useful information. The TiO<sub>2</sub> industry is no exception to this general observation.

However, bearing in mind the foregoing, the author of the publication under review has clearly gone to a great deal of trouble to bring together information from several sources to produce this publication and in so doing he has placed such information in a more readily available form for the very limited number of non-specialists in the field. Furthermore, the author appears to have been successful in including most of the relevant patent specifications in this review of patents which were published during the period 1965 to 1975 in the UK, Austria, Canada, France, Germany, Holland, Russia, Switzerland and the United States.

J. RUSSELL

## Introduction to Paint Technology

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Fourth Edition with glossary

London: Oil & Colour Chemists' Association, 1976

Pp. 219; illustrated. Price £5.00

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 up-dated in a new Fourth Edition. Further details are given in an advertisement on page xviii of this issue of the *Journal*.

(Hon. Editor)



# OCCA-29 Exhibition

22-25 March 1977 at Alexandra Palace, London

## attracts support from 14 countries

## The continuous dialogue between suppliers and manufacturers

Six months before the 29th annual Exhibition of the Oil & Colour Chemists' Association (OCCA-29) applications have been received from organisations in 14 countries, leaving no doubt of the value to exhibitors of this international meeting place for both suppliers and manufacturers in the surface coating industries.

### Technical display and discussion

Although the display of technical developments is naturally welcome, the Committee does not insist on this and it lays equal emphasis on the opportunity afforded by this annual event to personnel at all levels to meet and renew acquaintances so as to discuss common technical problems in an informal atmosphere.

### Refreshments on stands

This year in pursuance of this theme the Committee has amended the rule which had previously prohibited the serving of alcoholic refreshments on the stand. Exhibitors at previous Association Exhibitions at Alexandra Palace have commented that the provision of exclusive facilities, such as unlimited car parking, free bus shuttle service from the Underground station, two restaurants, a cafeteria and several bars has meant that visitors tend to spend longer time at the Exhibition.

### Venue

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

### The international forum for the surface coatings industries

The exhibition provides an excellent annual opportunity for the technical personnel in the supplying industries to meet their counterparts in the manufacturing industries and to discuss their common technical problems. The advantage both to exhibitors and visitors of meeting in an informal atmosphere needs hardly be stressed, since the cost to exhibitors sending representatives to all the countries from which the visitors are drawn might well be prohibitive, particularly to smaller companies. In pursuance of the idea of an informal atmosphere the Committee has amended the rule which had previously prohibited the serving of alcoholic refreshments to visitors on the stands. It is felt

that the opportunity now afforded to exhibitors to offer alcoholic refreshments if they so wish will not detract in any way from the technical content of the exhibition.

### Facilities at Alexandra Palace and travel arrangements

The facilities at Alexandra Palace, which the Association's exhibition will occupy on its own, include 2 licensed Restaurants, 2 bars, a Cafeteria and an Exhibitors' bar. Exhibitors have commented that an advantage of Alexandra Palace is that visitors tend to spend longer at the exhibition as all facilities are provided under one roof. Ample free car parking facilities are available for both exhibitors and visitors and Alexandra Palace is within easy access of the M1 Motorway and the flyover at the junction of the M1 and the North Circular Road was opened earlier this month. The Association organises a free bus service to and from Turnpike Lane Station on the Piccadilly Underground Line. The journey by Underground from Central London takes approximately 18 minutes and in due course the Piccadilly Line will be extended to Heathrow Airport terminal, which will give a direct link for visitors arriving at the Airport. 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.

### "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"

### News of Members

Mr R. C. Littlefield, an Ordinary Member attached to the London Section, has joined the board of Byrema Products Ltd as technical director. Byrema Products, based in Byfleet, Surrey, manufactures PVC plastisols and other speciality coatings and compositions. Mr Littlefield has been with the company since 1968.

Mr A. J. Goodfellow, an Ordinary Member attached to the London Section, having reached the age of retirement has resigned recently from the board of Carless Exploration. He is still retained by the company as a consultant and remains a director of the parent company, Carless Capel & Leonard Ltd in a non-executive capacity.

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.

## Obituary

### Edward McDougall

The Ontario Section deeply regret to announce the sudden death of Edward McDougall, Committee member, on Wednesday 15 September 1976.

Whilst he was elected to the Section Committee only in May 1976, Ed in his capacity as a member of the Programme and Publicity Sub-Committee had proved to be a very energetic and conscientious worker. He was deeply involved, at the time of his death, in making arrangements for the Section's first Annual Dinner Dance. His presence and enthusiasm will be greatly missed by all members in Ontario.

## Annual General Meeting

### Proceedings of the adjourned AGM

The adjourned 14th Annual General Meeting of the Incorporated Association was held on 13 October 1976 at 4.00 p.m. at the Great Northern Hotel, London N1 9AN with the President (Mr A. T. S. Nudram) in the Chair. There were 26 Members present.

Apologies for absence were received from Mr R. J. Woodbridge and Mr A. S. Gay.

The President called upon the Director & Secretary to read the notice of the adjournment of the Annual General Meeting as published on page 308 of the August issue of the *Journal*, in which it was explained that owing to the sudden death of Mr A. A. Duell on 20 June 1976 it had not been possible for Council to appoint a Member as President Designate at the Annual General Meeting held on 25 June 1976 at the Crown Hotel, Harrogate. Accordingly, in fulfilment of the provisions of Article 65(A) it had been agreed to adjourn the Annual General Meeting until 4.00 p.m. on 13 October 1976 when the sole business to be transacted would be to receive notification of the Council's appointment of a Member as President Designate.

### Appointment of President Designate

The President reported that since the Annual General Meeting on 25 June 1976 Council had held a further ballot, the result of which had been reported to its meeting held earlier that day. Accordingly, Mr A. McLean was the Council's appointment as President Designate for the Session 1976/77. (See also page 425.)

The President congratulated Mr McLean upon his appointment. Mr McLean stated that he was honoured to serve in this capacity and would do his utmost to further the activities of the Association.

The President thanked members for their attendance and declared the adjourned Annual General Meeting closed at 4.05 p.m.

## Professional Grade

### Admissions

At a meeting of the Professional Grade Committee held on 13 October 1976 the following admissions to the Professional Grade were authorised:

#### Fellows

Hoshidar Peshotan Kotwal—*General Overseas Section: Pakistan*

James Douglas Easton—*Ontario Section*

Chidanand Jayram Bhumkar—*General Overseas Section: India*

#### Associates

Jack Ian Fry—*Wellington Section*

Andrew Chi Kit Wu—*General Overseas Section: Hong Kong*

## Manchester Section

### OCCA Golf Tournament

The tournament was held this year on Wednesday 8 September and the venue for the competition was the Stockport (Torkington) Golf Club. The occasion was organised by Mr B. Falder of the Manchester Section, who was last year's winner.

Over 50 members and guests attended both the golf and the Dinner afterwards, and of these, 25 were from outside the Manchester area. In fact, most Sections appear to have been represented.

As well as the OCCA Trophy, this year the Manchester Section members also competed for the Manchester "Jubilee Cup". There were donations of other prizes, so that the chances of winning something were about 5:2.

The competition was run as an 18-hole Stapleford and the Trophy was won by Mr J. H. Sinclair (Manchester Section) with 39 points; second and third places required a play-off between Mr R. L. Wells (Hull Section) and Mr R. Hardy (West Riding Section), the former managing to secure second place (38 points). The highest scoring visitor was Mr M. Tennant of Carrs Paints Ltd, who equalled the winner's score. Manchester had the "honour" of taking the Booby Prize.

## Ontario Section

### Programme for 1976/77

Unless otherwise stated all meetings will be held at the Skyline Hotel, 655 Dixon Road, Ontario. A cash bar opens at 6.00 p.m. followed by a meal at 7.00 p.m.

**Wednesday 17 November** "The ultraviolet screening behaviour of pigments" by Dr R. J. Kennedy, of Hilton-Davis Co.

**Wednesday 15 December** "Cosmetics" by Mrs Hamly of Yardley Ltd. *Ladies' Evening.*

**Wednesday 19 January** "De-inking of paper" by Dr O. Sepal, of Reed Ltd.

**Friday 28 January** Dinner Dance to be held in "The Old Mill", 21 Old Mill Road, Toronto, Ontario. Reception 6.00 p.m.; Dinner 7.00 p.m.

**Wednesday 16 February** "Ultraviolet processes in the printing ink industry" by Mr W. Woodfine of Inmott Corporation, Canada.

**Wednesday 23 March** "Organic pigments for trade sales paints" by Mr R. E. Edelman, of Du Pont, U.S.A.

**Wednesday 20 April** Annual General Meeting.

**May To be announced.** A talk by V. Zlatareff of Ciba-Geigy (UK) Ltd. Joint meeting with the FSCT.

Any Member from another Section who is visiting Canada and could attend a meeting would be particularly welcome to do so. The Section would also be very pleased to receive offers of papers from European Lecturers. Those interested are advised to contact, in the first instance, the Section's Representative on Council, Mr H. C. Worsdall, Worsdall Chemical Company, Sole Street, Cobham, Kent. (Tel: 0474 812812).

## West Riding Section

### Notice of Dinner Dance

The Section's Dinner Dance will be held on 26 November 1976 at the Crown Hotel, Harrogate, Yorkshire. Those wishing to obtain tickets (price £7.00 each) should contact Mr N. Cochrane, 19 Rutland Road, Harrogate, Yorkshire HG1 2PY, before 9 November.

## 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

BATES, TIMOTHY JOHN, BSc, 37 Alexandra Road, Sale, Trafford, Manchester *(Manchester)*

CAMERON, GARY JOHN, AC Hatrick (NZ) Ltd, PO Box 51005, Tawa, New Zealand *(Wellington)*

GALATI, ROSS, 13346 Butternut Road, Burton, Ohio 44021, USA *(General Overseas)*

JOHNSON, PETER DAVID, 9 Woodville Drive, Sale, Cheshire M33 1NF *(Manchester)*

KIRSCH, HANS JURGEN, BSc, PO Box 207, Hammanskraal, Transvaal, South Africa *(Transvaal)*

KOPPL, DIETER, HENKEL SA (Pty) Ltd, PO Box 102, Isando 1600, Transvaal, South Africa *(Transvaal)*

POKLEWSKI-KOZIELL, Antoni Zenon, BSc, PO Box 43116, Industria 2042, Transvaal, South Africa *(Transvaal)*

SANDELL, ALAN JAMES, BSc, ARCS, 14 Crespiigny Road, London NW4 3DY *(Thames Valley)*

WELLS, RONALD JAMES, BSc, 146 Wainuiomata Road, Wainuiomata, New Zealand *(Wellington)*

WIDDOP, BRIAN, LRIC, 56 Mannheim Road, Heaton, Bradford 9, West Yorkshire *(West Riding)*

### Associate Member

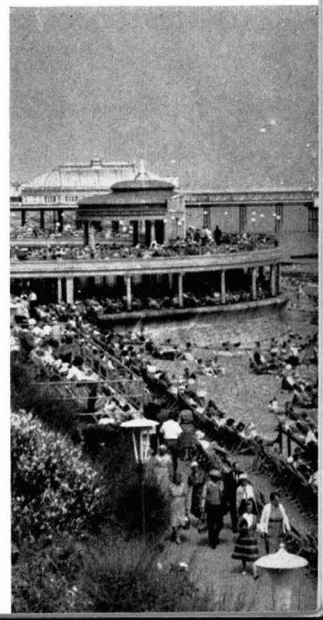
HERFST, GYSBERT FLORIS, BASF South Africa (Pty) Ltd, Box 11337, Johannesburg 2000, South Africa *(Transvaal)*



INCE 19

# Eastbourne

OCCA CONFERENCE 1977





# OCCA CONFERENCE 1977

The Grand Hotel, Eastbourne, Sussex, England

16—19 June 1977

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

As already announced in the *Journal*, the next Biennial Conference of the Association will take place at Eastbourne from Thursday 16 to Sunday 19 June 1977. The headquarters will be the Grand Hotel, with overflow accommodated at the Burlington Hotel. The title for the Conference will be "The conservation of energy, materials and other resources in the surface coatings industry". Summaries of most of the papers and biographies of the lecturers are given below. The programme for the technical sessions is as follows:

### Friday morning session

#### Painting energy policy

**Microvoid coatings—material and energy savers**

**Photocatalysts for combined radical and ionic curing systems**

**Recent developments in antifouling**

Chairman: C. N. FINLAY, ATSC (Hon. Research & Development Officer)

By Prof. G. R. Bainbridge (The Energy Centre, University of Newcastle)

By Mr J. A. Seiner (PPG Industries, USA)

By Dr T. J. van der Hauw (Akzo Chemie, Netherlands)

By Dr A. O. Christie (International Paint Co.)

### Friday afternoon session

Chairman: Dr L. VALENTINE, FTSC (Technical Director, Berger Jenson & Nicholson Ltd)

**Conserving human resources through innovation**

By Mr M. A. Glaser (Dexter Corporation, USA)

**The cost of flocculation**

By Mr J. G. Balfour (BTP Tioxide Ltd)

**Layer structures of absorbed polymers at pigment/solution interfaces and their influence on the dispersion stability of pigments in paints**

By Prof. K. Hamann and Dr G. Joppien (Research Institute for Pigments and Paints, Stuttgart)

### Saturday morning session

Chairman: Dr F. M. SMITH, FTSC (President of the Paint Research Association and a Vice-President of OCCA)

**The paint industry in a situation of diminishing availability of raw materials**

By Mr D. J. T. Howe (Consultant to the Paintmakers Association)

**Binders without environmental pollution and with good penetration properties on the basis of acrylic emulsions with very fine particle sizes**

By Dr J. L. Mondt (Hoechst AG, Germany)

**Save your paint, your money and your substrate**

By Mr A. F. Sherwood and Mr T. R. Bullett (Paint Research Association)

**Possible uses of reactive microgels in paint formulation**

By Prof. W. Funke (University of Stuttgart)

### Saturday afternoon session

Chairman: A. G. HOLT, FTSC (Former Chairman of the Thames Valley Section and a former Vice-President of OCCA)

**A study of the effects of thiol modification of alkyd resins on their ease of cure with aminoplasts**

By Mr K. Winterbottom, Mr N. S. Moss and Mr D. J. R. Massy (Ciba-Geigy Plastics & Additives Co.)

**Labelling of dangerous substances and preparations in the EEC**

By Mr R. W. Kay (HM Factory Inspectorate)

It is expected that a paper will also be given on behalf of the Scandinaviska Lackteknikers Forbund (SLF) and further details will be published in a forthcoming issue of the *Journal*.

As in previous years, the Association will organise a comprehensive programme of social events and further details will be announced in due course.

### Conference preprints

Preprints of the papers are now being prepared and it is expected that these, together with details of accommodation, badges, programmes, etc. will be sent to those who have registered in May 1977. It is a feature of the Association's Conferences that preprints are sent well in advance of the function, in order to enable delegates to read them before the Conference, at which the lectures illustrate

their topics but do not read the papers in their entirety. This allows for a much longer discussion period than at many other conferences, and has proved of immense value to those attending previous Association Conferences.

### Registration fees

Forms for registration will be despatched shortly to all Members of the Association attached to the Sections in the UK, Ireland and General Overseas Section, and it is expected that registration forms will also be included in the December issue of the *Journal* for the benefit of non-Members.

Council has fixed the registration fees for the Eastbourne Conference at £40.00 (plus Value Added Tax at the standard rate) for Members, £60.00 (plus VAT) for non-Members and £15.00 (plus VAT) for wives.

**Non-Members wishing to avail themselves of the preferential Conference fee for members should request application forms from the Association's offices and these should accompany the Conference registration forms.**

Further information may be obtained from the Director & Secretary at the address on the contents page of this issue (tel. 01-908 1086; telex 922670).

The papers to be presented, together with summaries and biographies of the authors are given below

### Painting energy policy

By G. R. Bainbridge

Energy policy in Britain matures slowly in the style of a picture ever subject to re-touching to suit the ideas of the painters transiently in command of the regulatory brushes. The basic scene is not altered radically as the years go by, though important aspects of it may be gradually modified or replaced. A feature or a colour may be enthusiastically tested and then obliterated upon reconsideration or in the light of experience. In this paper, consideration will be given to the prospect of getting the Energy Policy scene acceptably right reasonably soon.



G. R. Bainbridge

The author is at present the Professor of Energy Studies and Director of the Energy Centre in the University of Newcastle upon Tyne. He came to this post following teaching of various kinds and then an 18-year period with the United Kingdom Atomic Energy Authority working with the consultancy arrangements on safety and economic problems with the designers, constructors and operators of nuclear power stations. Later, whilst with the UKAEA as head of a Technical Assessments Section, he was responsible for evaluation of forward nuclear power development programmes.

### Microvoid coatings: material and energy savers?

By J. A. Seiner

Previous publications in the field of microvoid coatings have dealt with either theoretical light scattering, physical properties or manufacturing procedures. The main emphasis of this paper will be on a comparison between theory and measurements in practice; heat rejection and light scattering, in particular, will be studied. Questions to be answered include the extent to which it might be possible to reduce heat losses from houses and factories by painting these buildings with lighter colours (such as ultra-white microvoid coatings) and whether optimising combinations of microvoids and titanium dioxide pigments can afford appreciable savings in raw materials. At the end of the talk, if time permits, a summary of manufacturing procedures will be given.

Mr Seiner received his BSChE from Carnegie Mellon University at Pittsburgh, USA in 1954. He subsequently received a



J. A. Seiner

BSIM from the same University as a part-time student in 1960. He has been employed by PPG Industries as a coatings and plastics research scientist since 1954. His experience included periods as Senior Pilot Plant Engineer, Manager, Resin Products (plastics and foams) Research and his current position of Manager, Consumer Products Research. The author of more than twenty papers and thirty US patents, he has made major contributions to the fields of polyester processing, copolymerisation theory, microvoid coatings and plastics fire retardancy.

### Photocatalysts for combined radical and ionic curing systems

By T. J. van der Hauw

Within the range of known photoinitiators, chlorinated acetophenones are a special class. By radiation with ultraviolet light, they can produce both radicals, suitable for acrylic polymerisation, and also hydrochloric acid, which can be applied as an *in-situ* generated catalyst for acid-curing lacquers. A further development is the application of both actions in a suitably chosen binder system formulated from an alkyd polyol/aminoformaldehyde resin and acrylic monomer(s). Suggestions are given for the formulation of a 100 per cent-convertible coating system for application on substrates such as paper and wood.

Because the curing is effected by ultraviolet radiation at room temperature and with a high solids content, such systems show promise for the future from the point of view of conservation of energy and materials by avoiding volatile solvents. In comparison with 100 per cent acrylic



T. J. van der Hauw

systems, the price/performance relationship looks attractive as well.

Dr van der Hauw studied chemistry at the Free University of Amsterdam and graduated in 1954. Two years later he joined Kunsttharsfabriek Synthese and was active in the development of synthetic resins.

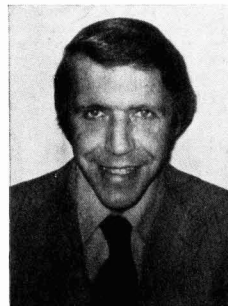
In 1969 he moved to the Akzo Chemie Research Centre at Deventer, where he is responsible for the application research in the field of prepolymers.

### Recent developments in antifoulings

By A. O. Christie

It is useful at the outset to define the purpose of antifouling coatings. Some years ago, it would have been sufficient to state that their purpose was to keep underwater hulls in a fouling-free condition. A more appropriate updated definition would be to keep frictional resistance as low as possible for as long a time as possible. The definition has suddenly become much more precise and scientific—as might be expected in an area which is currently under the spotlight of many scientific disciplines. The problem is biological, the consequences can be subjected to hydrodynamic interpretation and to economic evaluation, but the answer remains with the antifouling paint chemist.

The fouling problem on ships, and both traditional and current approaches to its control, will be discussed.



A. O. Christie

Dr A. O. Christie graduated from Aberdeen University in 1959. He held the Red Hand Compositions Research Fellowship at the Marine Science Laboratories, Menai Bridge, before joining Red Hand in 1965. At the merger with International Paint in 1968 he was transferred to the International's marine laboratory in Devon, before going to the Group's Felling Laboratory as Research Manager for Antifoulings.

The author of several papers on fouling topics, Dr Christie has a special interest in algal fouling problems and in conjunction with Dr L. V. Evans of Leeds University has pioneered research in this field.

## Conserving human resources through innovation

By M. A. Glaser

If we learn how to innovate with greater frequency and effectiveness in our organic coating laboratories, we can perform the new technological tasks required with fewer scientists or technologists. The human resources thus saved can then be utilised to perform other vital functions, which will be required of our evolving society—improvement in air and water quality, removal of toxic substances from human contact and improvement in the overall quality of life in all parts of the earth (including life without wars and terrorism). A model for "Innovation" has been developed which can be used as a "checklist" to see if there is a probability that innovation will take place in a given research or development project. The model was based on retrospective analyses, and communications, psychological and sociological principles as well. This model is now being used as a working tool in the author's laboratories and in others as well. The technique discussed has proven valuable in pointing out weaknesses in the organisation and execution of research and development projects and has suggested means of correcting these weaknesses. Designations of "Innovation potential", "Critical innovation potential" and "Fatal flaw factor" will be reviewed.

Mr M. A. Glaser received his BSc degree in chemical engineering from Tufts College Mass. in 1934. After serving in an engineering position with the Metropolitan District Commission of the Commonwealth of Massachusetts he moved to Chicago in 1937 to work as a chemist in the polymer research and development laboratory of Standard Varnish Works. He received several promotions and in 1940 became chief chemist and director of research.

Mr Glaser moved to Midland Industrial Finishes Co. in 1945 as technical director and has remained with the company since that time—as Vice-President and technical director and as a member of the board of directors. After acquisition of the company by the Dexter Corporation, he became Vice-President, Research and Development.



M. A. Glaser

Mr Glaser is a past-President of the FSCT; he is a registered professional engineer and for many years was the USA titular member of the Organic Coatings Section of IUPAC; he is also a member of the New York Academy of Sciences and a

Fellow of the American Institute of Chemists; he has presented lectures on paint technology to many Societies and is the author of a considerable number of articles, papers and patents.

## The cost of flocculation

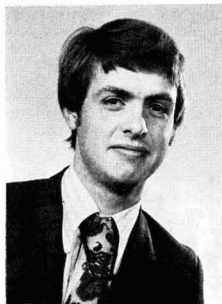
By J. G. Balfour

An empirical technique for assessing the extent of titanium pigment flocculation in decorative alkyd gloss paints was reported at the 1975 OCCA Conference.

Experience with the technique in the ensuing two years will be described.

The effect of flocculation on paint film properties such as opacity, gloss, colour and durability is discussed, particularly with reference to decorative alkyd gloss paints.

Indications as to the possible economic and technical benefits which could accrue from improvements in the states of dispersion of commercially available paints are discussed.



J. G. Balfour

Mr Balfour graduated from the University of Leeds in 1971 with an Honours degree in Colour Chemistry. He then joined the Group Research Laboratory of Berger, Jensen & Nicholson Ltd and was concerned with pigment evaluation.

In 1974 he joined the Technical Service Department of Tioxide International as a Technical Officer and is now primarily engaged in work concerned with the physical testing of pigmented materials.

In 1975 he was a joint winner of the Association's Jordan Award.

## Layer structures of adsorbed polymers at pigment/solution interfaces and their influence on the dispersion stability of pigments in paints

By G. Joppien and K. Hamann

Polymer molecules generally undergo considerable alterations in shape when only small external forces are applied. In particular, this is true for polymer adsorption, and such a situation arises whenever pigments are dispersed in binder solutions. During paint pigmentation, binder molecules make contact with the force fields of the pigment surfaces and build up adsorption layers, the structures of which are different from those of the polymers in solution. As the properties of polymers are determined mainly by their shape, and the internal pigment/binder surfaces in pig-

mented systems are so extremely high, significant effects of adsorbed polymer layer structures on various paint properties are to be expected.

The object of this paper will be to describe some of the structure/property interrelationships in order to emphasise their possible utilisation in future paint technology.

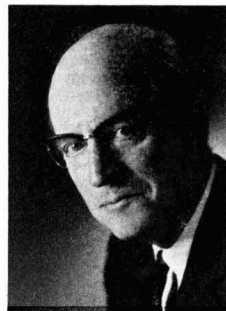
The main object of relevant investigations has to be the structure determination of adsorbed polymer layers. Although no direct methods are available, the structure can be deduced from the amount of adsorbed polymer and the fraction of polymer segments in direct contact with the substrate. Two different methods have been applied to study polymer layer structures in several different model adsorption systems.

By infrared analysis of pigment and filler ( $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{TiO}_2$ ,  $\text{Fe}_2\text{O}_3$ ) dispersions in organic polyester solutions, the fractions of bound carbonyl groups of the adsorbed molecules as well as the fractions of interacting surface groups of the substrates can be determined via shifts of their characteristic infrared absorption bands. Determination of the concentration of solid-free solutions before and after adsorption allows calculation of the adsorbed amounts. These investigations have shown the formation of adsorption loop structures on the surfaces. The size of loops protruding into the solutions is dependent on solid surface structure as well as sorbate composition.

Potentiometric acid-base titrations of pigments in aqueous polymer solutions in the presence of electrolytes, combined with interferometric concentration determinations, permits a study of adsorbed polymer layer structures at charged pigment surfaces. The fractions of bound polymer segments can be deduced from comparative adsorption experiments with low molecular weight model compounds since both behave similarly in the surface-adjacent layer. These experiments also indicate loop structure formation, the loop sizes being strongly dependent on pH and the state of dissociation of adsorbed polyelectrolytes.

From these findings, the possibility arises of synthesising polymers which on adsorption form layer structures with optimal dispersion properties. Several examples will be given as proof.

The significance of these experiments lies in a correlation of polymer loop structures with the physical properties of the systems, such as dispersion rate, wetting, and especially the dispersion stability of pigments in paints.



K. Hamann

Professor Hamann graduated from the University of Hamburg in 1931 with a doctorate in chemistry. After a two years postdoctoral fellowship at the University of Bonn, he entered the I.G. Farbenindustrie (now Bayer AG) in 1934. From 1944 to 1953 he was director of the scientific research laboratory of Bayer AG at Uerdingen. In 1953 he was appointed full professor of technical chemistry at the University of Stuttgart, and at the same time became director of the Research Institute of Pigments and Paints in Stuttgart.



G. Joppien

Dr Joppien graduated from the Technical University of Berlin with a doctorate in natural sciences in 1969. After working for two years as a postdoctoral research associate at the University of Wisconsin in Madison, USA, he joined the Research Institute of Pigments and Paints eV, Stuttgart in 1972. In addition, he was appointed scientific assistant to Professor Hamann's chair at the University of Stuttgart where he has recently submitted a "Habilitation" thesis in chemical engineering. His research interests are polymer conformations at interfaces with relevance to technical properties and surface properties of pigments.

### The paint industry in a situation of diminishing availability of raw materials

By D. J. T. Howe

As the end of the century approaches, the availability of many raw materials to the paint industry may diminish as a result of political, economic and social pressures and an acceleration in the depletion of some finite natural resources. The introduction of new or alternative materials may be delayed or even prevented by legislative requirements considered necessary to safeguard the health of the employee and customer and to protect the environment. The question how the industry should react to these problems both in the short term and in the long term will be posed.

After some years of research in photography with Barnet Ensign Ltd and with ICI, Mr Howe, a graduate in Chemistry in the University of London, joined what was then known as the Industrial Hygiene Research Laboratories of ICI in 1958. He was the member of the Management Group responsible for liaison with the manufacturing divisions of the company and with

governmental regulatory authorities worldwide. He became a consultant in 1973 on matters concerned with the environment and with the toxicology of industrial chemicals, and he is retained as such by the Paintmakers Association of Great Britain.



D. J. T. Howe

Committee work has occupied a great deal of his time in recent years. In particular, he has been a member of the Council of the Royal Institute of Chemistry 1972-75, and that of the Chemical Society 1974-75, a member of EEC working parties preparing the Directive on the Labelling of Dangerous Preparations (paints, printing inks, etc.), the working party concerned with "Works' Hygiene in the Chemical Industry" and the working group of the Department of the Environment concerned with the code of practice on the disposal of non-halogenated solvent waste under the Control of Pollution Act 1974.

### Binders without environmental pollution and with good penetration properties on the basis of acrylic emulsions with very fine particle sizes

By J. L. Mondt

The priming and impregnating of porous substrates is usually carried out with solutions of polymers in organic solvents. Efforts towards environmental protection, and also increased cost and uncertain availability of raw materials after the 1974 oil crisis, has speeded up a tendency for replacement of the conventional solvent-based paint materials. Water-based systems, especially aqueous emulsions, present a good alternative.

The particle size of dispersed high polymers in aqueous emulsions is an important factor with regard to the technological characteristics of emulsions. The emulsions presently on the market normally show latex particle sizes of diameter greater than or equal to 0.1  $\mu\text{m}$ . This paper summarises the synthesis and the field of application of emulsions with latex particles of diameter less than or equal to 0.05  $\mu\text{m}$ .

Emulsions with very fine particle size will provide technological advantages—in contrast to the known emulsions with a normal particle size distribution—especially in the field of priming and impregnation, where good penetration is an essential property. Optical brighteners have been

used as comonomers for the synthesis of small particle size emulsions in order to observe the penetration of polymer latices in comparison with polymer solutions. Vinylsulfonpyrazoline derivatives form a suitable class of comonomers for the preparation of optically brightened high polymer latices, which can easily be detected under ultraviolet light after application. The ease of penetration of very small particle sized latices into various substrates will be demonstrated.



J. L. Mondt

Dr J. L. Mondt studied chemistry at the Universities of Marburg, Munich and TH Darmstadt. He graduated from the TH Darmstadt Institute of Organic Chemistry in 1968 with a PhD in organic chemistry. After about two years in the United States as a research associate at the Department of Chemistry, University of Oregon, Eugene, he joined the Research Centre of Hoechst AG, Frankfurt, Department of Resins and Emulsions, where the major research interests are in the synthesis and study of the properties of acrylic emulsions.

### Save your paint, your money and your substrate

By T. R. Bullett and A. F. Sherwood

An effective paint system forms an invaluable aid in the conservation of materials, not only directly by protecting the substrate from deterioration but also by enhancing the environment—even the most starkly functional paint system, in good condition, can have aesthetic appeal.

Conversely, a misused paint system represents waste of material and effort, and results in substrate deterioration. Misuse can originate in wrong choice of paint or badly executed preparation and painting, but may equally result from bad design or poor specification or failure to consider painting operations in a general work plan.

The need will be demonstrated for designers to be aware of the factors important in effective protection, for the specifier, paint supplier and painting contractor to cooperate to ensure that the right paint is used in the right way, for the engineers to plan so that painting can be done at the right time and under favourable conditions and for the clerk of works and inspectors to have strong and effective teeth!



T. R. Bullett

Mr T. R. Bullett joined the Paint Research Association after graduating in Physics at University College London. He is now Head of Technology Division. He has presented papers to O.C.C.A., F.A.T.I.P.E.C., and the American Chemical Society and elsewhere on paint adhesion, optical and mechanical properties and durability testing. Long experience of paint failure consultancy has made him a crusader for the better use of paint.

Mr Sherwood joined the Paint Research Association in 1943. In the intervening years he has been involved, amongst other topics, in studies on oil polymerisation, varnish chemistry, molecular weight determinations, the protection of aluminium alloys and the use of tracer techniques. The latter were used extensively in his studies on the interactions between pigments and media. Now as Section Leader, Building Materials, he is extensively involved in the protection problems of the building industry.



A. F. Sherwood

### Possible uses of reactive microgels in paint formulation

By W. Funke

Reactive microgels are crosslinked polymer particles with diameters ranging from about 5 to 10 nm. They are usually prepared by the emulsion polymerisation of monomers which have a higher functionality than 2 and, therefore, act as crosslinking units. During the polymerisation reaction

not all functional groups are used up. Of the residual reactive groups, those located at the microgel surface are available for further reactions. Reactive microgels can be used as multifunctional crosslinkers for reinforcing purposes. By chemical reactions, new reactive groups may be introduced at the particle surface so that microgels can also be used as components in condensation and addition reactions.

The compact globular structure enables preparation of solutions with high solid content and tolerable viscosity. By reaction of the surface groups with molecules of soluble dyes, new organic pigments with polymer cores may be obtained.



W. Funke

Professor Funke received his doctorate from the University of Stuttgart in 1956. He is an associate of the Research Institute for Pigments and Paints, and has been Professor of Polymer Chemistry at the University of Stuttgart since 1969. Professor Funke's research interests are the structure of crosslinked polymers, especially reactive microgels, the relationships between structure and properties of coatings, and the corrosion protective properties of paint films.

### A study of the effects of thiol modification of alkyd resins on their ease of cure with aminoplasts

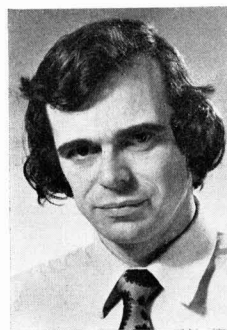
By K. Winterbottom, N. S. Moss and D. J. R. Massey

The thiol group (-SH) is capable of undergoing a variety of facile and efficient reactions. Of relevance to surface coating applications is the possibility of preparing thiol-modified saturated alkyd resins. The thiol groups in such resins should react readily with aminoplasts, forming monosulfide (thioether) crosslinks. Several thiol-modified alkyd resins, including water-soluble products, have been prepared and evaluated in admixture with aminoplasts as potentially fast-curing industrial finishes.

Substantial reductions in curing time and/or temperature are observed compared with control, unmodified alkyd resins blended with the same aminoplasts.

It is thought that this behaviour could usefully contribute to the conservation of energy and the improvement of productivity.

Mr Winterbottom graduated in Textile Chemistry at UMIST in 1960 and has since then worked in the Research Department of



K. Winterbottom

Ciba-Geigy Plastics & Additives Company at Duxford, Cambridge. During this period he has applied his knowledge of textile chemistry to the development of finishing agents for cellulosic fibres and shrinkproofing pre-polymers for wool. More recently he has been concerned with the development of new resins for the surface coating industry.

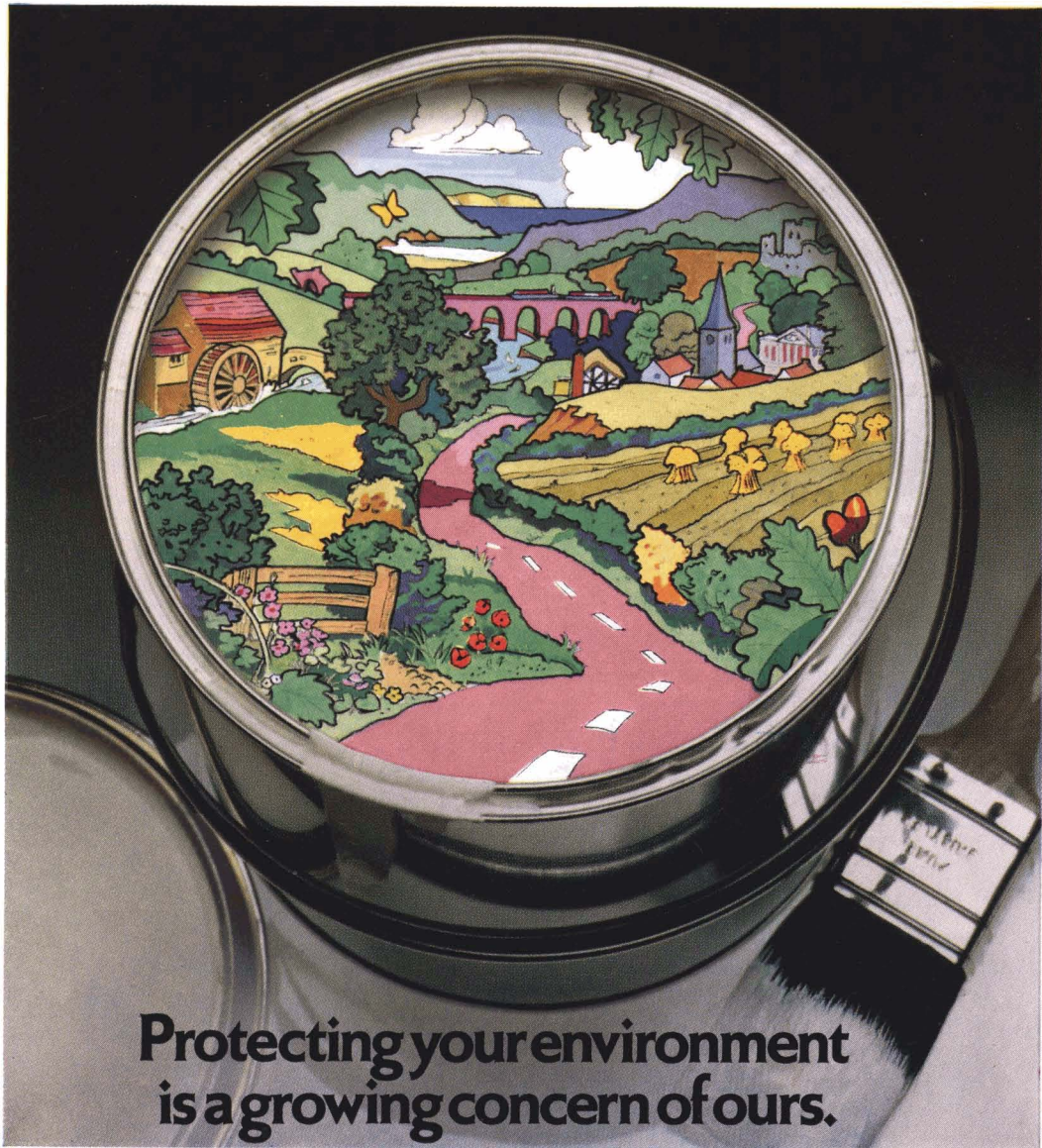


N. S. Moss

Mr N. S. Moss entered the paint industry in 1963. During 1967 he became a Licentiate of the Royal Institute of Chemistry and joined CIBA (A.R.L.) Ltd in the coating resin development group. His present position is that of Laboratory Section Leader responsible for development and customer service work associated with Ciba-Geigy Plastics Division's coatings resins.



D. J. R. Massey



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Mr Massy graduated from Cambridge University in 1952 and has since worked in the chemical industry, mainly in R & D concerned with resins and polymers. His technical interests include vinyl and acrylic polymers, aminoplasts, phenolics and speciality resins and their uses in surface coatings, textile finishing and paper. His present position is that of Section Manager in the Research Department of Ciba-Geigy Plastics & Additives Co.

### Labelling of dangerous substances and preparations in the EEC

By R. W. Kay

The EEC has issued a number of directives on the classification, packaging and labelling of dangerous substances and preparations and this process is continuing. These directives have two main aims, the removal of barriers to trade within the Community and the protection of any user of these products, whether a worker or a member of the public.

In the United Kingdom, regulations to implement the substances and solvents directives were brought into force earlier this year. Another directive, which concerns paints, varnishes, adhesives, printing inks and related products, will be implemented at a later date. It is the intention of the EEC that this series of directives will eventually cover a much wider range of preparations, for example, pesticides and household products.

Mr Kay graduated from Glasgow University in 1951. After fifteen years in industrial research and development, he joined the Chemical Branch of the Factory Inspectorate, working on hygiene problems in industry. For the past three years, he has been concerned with work in the EEC and the Council of Europe on the labelling and use of dangerous substances and preparations.



R. W. Kay

It is expected that a paper will also be given on behalf of the Scandinavian Lacteknikers Forbund (SLF) and further details will be published in a forthcoming issue of the *Journal*.

## PRA 50th Anniversary Dinner



The photograph shown above was taken on the occasion of the fiftieth anniversary Dinner of the Paint Research Association when the President, Mr A. T. S. Rudram, presented to the PRA President Dr F. M. Smith (centre) a congratulatory scroll from the Oil & Colour Chemists' Association to mark the PRA's fiftieth anniversary. Seated is Mr M. J. Methven, Director General of the Confederation of British Industry, who was the principal guest

## 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.

### President Designate



Mr A. McLean, who is Council's appointment as President Designate (see AGM report on page 418)

### Erratum—Commendation Awards

It is regretted that the list published in the August 1976 issue of the *Journal* of all those Members who have received a Commendation Award is incomplete and should include the name of Mr P. B. Hunt, who is an Ordinary Member attached to the Auckland Section. Mr Hunt was one of the earliest recipients of the Award (1969) and our sincere apologies are extended to him for this error.

## 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.

### November

#### Monday 1 November

*Hull Section:* "The fundamentals of UV curing" by Mr R. G. Smith, Arthur Holden & Sons Ltd. To be held at the George Hotel, Land of Green Ginger, Hull, commencing at 6.30 pm.

#### Thursday 4 November

*Newcastle Section:* "The use of microvoids as pigments" by Mr J. Clark, Tioxide International Ltd. To be held at the Royal Turks Head Hotel, Grey Street, Newcastle upon Tyne, commencing at 6.30 pm.

#### Friday 5 November

*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.

#### Tuesday 9 November

*West Riding Section:* Lecture by a speaker from SCC Colours Ltd. To be held at the Griffin Hotel, Board Lane, Leeds, commencing at 7.30 pm.

#### Wednesday 10 November

*Scottish Section—Eastern Branch:* "Industrial solvents" by Mr C. Smith of Carless Solvents Ltd. To be held at 7.30 pm, in the Carlton Hotel, North Bridge, Edinburgh.

#### Thursday 11 November

*Midlands Section—Trent Valley Branch:* "Inflation accounting" by Mr S. Turner, Mebon Ltd. To be held at the Crest Hotel, Pastures Hill, Littleover, Derby at 7.00 pm.

*Thames Valley Section:* Zinc coatings" by Mr F. C. Porter, Zinc Development Association. To be held at the Beaconsfield Crest Motel (White Hart) Aylesbury End, Beaconsfield, Bucks at 6.30 for 7.00 pm.

#### Friday 12 November

*Hull Section:* Disco-Dance to be held at the Cricket Club, Anlaby Road, Hull.

*Manchester Section:* "Possibilities and prospects for metal coatings with polyurethanes" by Dr. Sonntag, Bayer AG. To be held at the Woodcourt Hotel, Sale, Cheshire, commencing at 6.30 pm.

*Midlands Section:* "Paint manufacture" by Mr B. Lucas, Joseph Mason Paints Ltd. To be held at the Birmingham Medical Institute, 36 Harbourne Road, Birmingham.

#### Monday 15 November

*Hull Section:* "Synthetic clays—their properties and uses" by Mr B. J. R. Mayes, Laporte Industries Ltd. *Student lecture.*

#### Tuesday 16 November

*London Section:* "New approaches to the development of specific properties in paint films" by Prof H. P. Schreiber,

Department of Chemical Engineering Ecole Polytechnique, University of Montreal. To be held in the Colbrook Room, Sheraton-Heathrow Hotel, commencing at 11.00 am. and to include luncheon.

#### Wednesday 17 November

*London Section:* "Water thinned anti-corrosive coatings". Joint day meeting with the Institution of Corrosion Science & Technology and in association with Thames Polytechnic, commencing at 10.00 am.

*Ontario Section:* "The ultraviolet screening behaviour of pigments" by Dr R. J. Kennedy of Hilton-Davis, Cincinnati, USA. Venue Skyline Hotel, Toronto.

#### Thursday 18 November

*Scottish Section:* "Car painting" by Dr D. B. Bruce, Goodlass Wall Ltd. Joint meeting with the Society of Dyers and Colourists. To be held at the Beacon's Hotel, 7 Park Terrace, Glasgow G3 at 6.00 pm.

#### Friday 19 November

*Irish Section:* Annual Dinner Dance commencing at 8.30 pm

#### Friday 26 November

*British Section:* "Waste disposal in the surface coatings industries" by Mr H. G. Pullen, Redland Purle Ltd. To be held at the Royal Hotel, Bristol at 7.15 pm.

### December

#### Wednesday 1 December

*Scottish Section—Eastern Branch:* "Put a sparkle into your industrial finishes" by Dr D. R. King, Silberline Ltd, to be held at the Carlton Hotel, North Bridge, Edinburgh, commencing at 7.30 pm.

#### Thursday 2 December

*Newcastle Section:* "Ion beam etching for the study of paint film structure" by Mr J. L. Prosser, Paint Research Association. To be held at the Royal Turks Head Hotel, Grey Street, Newcastle upon Tyne, at 6.30 pm.

*Thames Valley Section:* "Colour and gold decoration on pottery and glass" by Mr O. N. Collier, Johnson Matthey

Ltd (Ladies invited). To be held at the Beaconsfield Crest Motel (White Hart), Aylesbury End, Beaconsfield, Bucks, at 6.30 for 7.00 pm.

#### Monday 6 December

*Hull Section:* "High solid coating systems" by Dr D. Faulkner, Rohm & Haas (UK) Ltd. To be held at the "George Hotel", Land of Green Ginger, Hull, commencing at 6.30 pm.

*Manchester Section:* "Decoration of ceramics" by Mr K. Hopkins, Ben Capper Ltd. To be held at The Woodcourt Hotel, Sale, Cheshire, commencing at 6.30 pm.

#### Thursday 9 December

*Scottish Section:* "Laughter in the Law" by Mr A. Carswell, BL. To be held at the Beacon's Hotel, 7 Park Terrace, Glasgow G3, commencing at 6.00 pm.

#### Friday 10 December

*Irish Section:* "UV curing-principles and practice" by Dr B. E. Hulme, BTP Tioxide Ltd. To be held at the Clarence Hotel, Dublin, commencing at 8.00 pm.

#### Tuesday 14 December

*West Riding Section:* Chairman's Lecture, "Home-made wines, and the effect of alcohol on the body system". Ladies' Evening to be held at the Mansion House Hotel, Roundhay Park, Leeds, commencing at 7.30 pm.

#### Wednesday 15 December

*Ontario Section:* Informal Ladies' Evening with a talk, "Cosmetics" by Mrs H. Hamley, Yardley of London (Canada) Ltd. To be held at the Skyline Hotel, Toronto.

### January

#### Wednesday 19 January

*Ontario Section:* "De-inking of paper" by Dr O. Sepal of Reed Ltd. To be held at the Skyline Hotel, Toronto.

#### Friday 28 January

*Ontario Section:* 1st Annual Dinner Dance of the Section, to be held at "The Old Mill", 21 Old Mill Road, Toronto at 6.00 for 7.00 pm.

## 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.

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## 3. Natural and Synthetic Resins

Natural resins: Gum resin, Dammar resin, Rosin. Synthetic resins: Alkyd resins. Manufacture of alkyd vehicles. Phenolic resins. Vinyl polymers and copolymers. Solvent soluble vinyl resins. Vinyl chloride dispersions. Styrene containing resins. Polyester resins. Urea resins. Mannite resins. Epoxy resins. Epoxy rich media. Polyurethane resins. Acrylic resins. Silicone resins. Coumarone resins. Petroleum resins. Chlorinated rubber. Non-aqueous dispersions.

## 4. Resins as Media for Paints

*Oil-soluble lacquers:* Alkyd resins as media. Cellulose lacquers and media. Solvents for cellulose lacquers. Solvent and driers. Solvents, driers. Some uses of clear finishes. Insulating varnishes, wood finishing by cellulose lacquer. Water-based paints for industrial use.

## 5. Pigments and Extenders

*Dispersion and wetting:* wetting, wetting agents. *Inorganic Pigments:* white pigments: zinc oxide, titanium dioxide, zinc oxide, lithopone, white lead, zinc phosphate, barium sulfate, barium sulphate, barium chromate, barium chromate, china clay, bentonite, state powder, mica, asbestos, black pigments: carbon blacks, vegetable (lamp) black, bone and drop blacks. *Metallic pigments:* aluminium, bronze powders, zinc, oxides of lead, red lead, red oxides, oxides of iron; chemically prepared oxides of iron; yellow hydrated oxides, reds and browns, black oxide. *Chrome pigments:* lead chromate, zinc chromate, zinc tetrachromate, strontium chromate. *Blue pigments:* prussian blue, ultramarine blue, green pigments: Chrome (Bismuth) green. *Flooding pigments:* Lanthanone blue, quinacridone pigments, phthalocyanine blues, phthalocyanine greens, vat colours; *lanthone blue, quinacridone pigments, dioxazine fastness.* Chemical constitution. Purity. Conditions of exposure, measurement of fastness. Optical density or hiding power. Oil absorption. Water-soluble matter. Light fastness. *Pigment hazards:* toxicity, dust.

## 6. Decorative and Structural Paints

*Types of decorative and structural paints. Sealers. Primers:* primers for wood, primers for alkaline and porous surfaces; primers for iron and steel; the mechanism of corrosion, protective mechanism in paint coating, metal cleaning and pretreatment. Blast primers, each of (a) wash primers, zinc silicate primers, zinc phosphate primers, zinc phosphate primers, zinc silicate primers, zinc silicate primers, lead phosphate primers, zinc chromate primers, red oxide/zinc chromate primers, red lead/linseed oil primers, calcium plumbate primers, primers for galvanneal surfaces, *Undercoats, fillers and stoppers:* stoppers, fillers, undercoats. *Finishes:* alkyd-based finishes, 'Non-drip' or thixotropic finishes, semi-gloss and eggshell finishes, oleoresin-

## Glossary—Appendices—Index

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

## 9. Common Defects of Varnishes and Paints

*Varnishes:* bloom, blushing, pinholing and cissing; silkiness; siltiness; blistering; checking; cissing; reworking; dirt collection during drying; fracture; cracking; pinholing; pigment settling; skinning. *Paints:* bleed-through, sagging, curdling and tears; siltiness; skinning. *Pigments, resins, solvents, fillers, other fire risks.*

## 8. Paint Manufacture and Hazards

*Paint millings:* function of paint mills. General principles of paint mills. *Types of mill:* premixers, mixers for pastes, mixers for slurries, paddle mixers, high speed mixers. *Dispersing mills:* Dispersing mills requiring primers. Triple roll mills, single roll mills, colloid mills, the sand mill, sealed sand mill, the 'D'no mill. *Dispersing mills:* direct charges. The ball mill. The stirred mill. *High speed dispersers:* the kady mill. *Thinning mixers:* Straining. *Filtering. Health and fire hazards:* toxicity, aerosol spraying.

## 7. Industrial Stoving and Cold-curing Finishes, Including Marine Finishes

*Industrial finishes:* Table of media used for various purposes, alkyds, alkyd/amino combinations, epoxy and epoxy ester resins, epoxy/pitch combinations epoxy/epoxy resin finishes, polyester finishes, hydroxylated acrylate/epoxy resin finishes, polyurethane water-thinnable finishes, silicone resins, zinc silicate finishes, oil-free polyester finishes. *Stoving Equipment:* convection, infrared radiation, induction, dielectric heating. *UV-curing:* electron beam curing. *Paint application:* padding, spraying, airless spraying, electrostatic spraying, hot spray technique, dipping, 'toe-dip' process, flow coating, curtain coating, roller coating, silk screen method, vacuum and pressure impregnation, electro-deposition, powder coatings, strip coating.

*Marine finishes:* vinyl resin finishes, plastisols and organosol finishes, chlorinated rubber finishes, epoxy ester finishes, vinyl resin finishes, masonry paints. *Emission paints:* nature of emulsions, preparation of polymer emulsions, homopolymers and copolymer emulsions, manufacture of emulsion paints, additives, properties and uses of vinyl emulsion paint, acrylic emulsions.

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An order form is printed on page iii of the issue of the *Journal*.

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