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OCCA AGM Luncheon Lecture

Thursday 26 June 1980

A lecture entitled: "Energy in the world"

will be delivered by Professor Sir Hermann Bondi, KCB FRS at the Piccadilly Hotel. Further information and application forms available from the Association's offices

**JOURNAL OF THE
OIL &
COLOUR
CHEMISTS'
ASSOCIATION**

Esters of polystyrene glycol and rosin in surface coatings
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S. G. Croll

Preservation, priming, putty and paints
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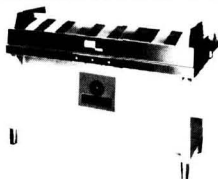
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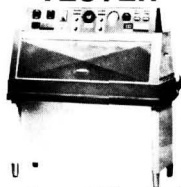
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Statistical Sources for the Paint Industry

The use of statistical information has become an essential part of modern business practice. Figures are required to gauge the demand for a product, to explore new markets, to justify a reallocation of resources, or to back up an investment decision. They can also help a company to monitor its financial efficiency by comparison with its competitors, domestic and foreign. This is especially true in times of a shrinking economy. When stringencies prevail, statistics assume an even greater relevance to industrial decision-making.

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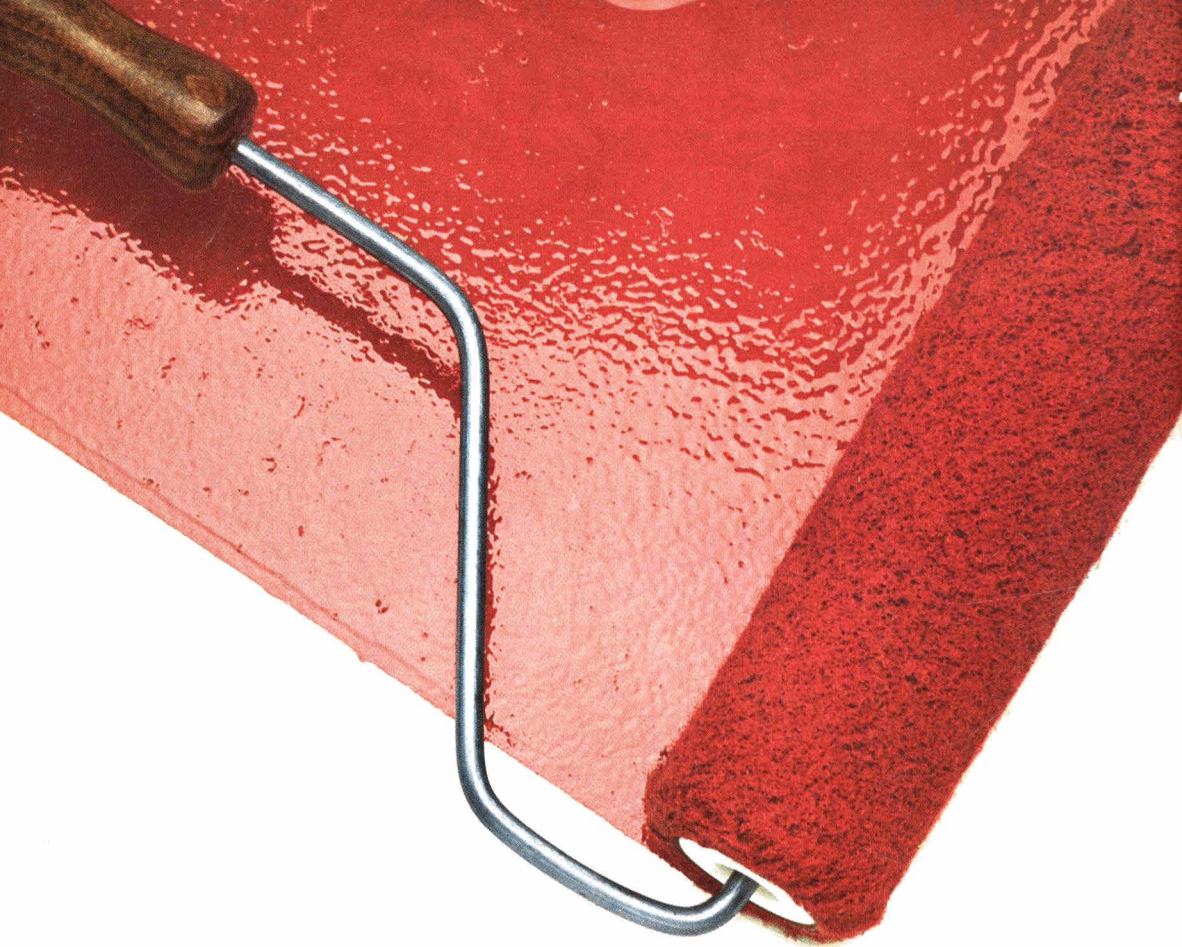
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Contents

Vol. 63 No. 6

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Transactions and Communications

Esters of polystyrene glycol and rosin in surface coatings	225
<i>S. Chandra and S. Pasari</i>	
Effect of solvents on residual strain in clear epoxy coatings	230
<i>S. G. Croll</i>	
Preservation, priming, putty and paints	237
<i>J. J. Froggatt</i>	
The effect of ageing on the ease of decontamination of some paint systems	243
<i>P. Walker and F. Cornish</i>	
<i>OCCA Meetings</i>	254
<i>News</i>	255
<i>OCCA News</i>	258
<i>New members</i>	262
<i>OCCA Diary</i>	262

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Esters of polystyrene glycol and rosin in surface coatings

By S. Chandra and S. Pasari

Department of Oil and Paint Technology, Harcourt Butler Technological Institute, Kanpur - 208 002, India

Summary

Polystyrene glycol was prepared and used in an esterification reaction with rosin. In the preparation of ester gum, a part of glycerol was replaced by polystyrene glycol yielding mixed esters of higher melting points. Oleoresinous varnishes were prepared from these mixed esters using linseed oil. Film properties of these oleoresinous varnishes were compared with those of ester gum varnish and alkyd resin. It was found that a film of mixed ester varnishes dried quicker than those of ester gum

varnish and alkyd. Flexibility and adhesion, scratch hardness, and water, acid, alkali and solvent resistance of the mixed esters were better than those of ester gum. Mixed esters were superior even to alkyds in resistance to alkali and solvent resistance. However, their resistances to water and acid were similar to a linseed alkyd but the scratch hardness was inferior to linseed alkyd.

Keywords

Types and classes of coatings and allied products
clear coating

Properties, characteristics and conditions - primarily associated with
coatings during application

drying rate

materials in general

flexibility
adhesion

dried or cured films

scratch resistance
water resistance
acid resistance
alkali resistance
solvent resistance

Raw materials for coatings
binders (resins, etc.)

polystyrene resin
rosin
rosin adduct

Les esters polystyrène glycol de colophane dans le domaine de revêtements de surface

Résumé

On a préparé le polystyrène glycol et on l'a utilisé dans une réaction d'estérification avec la colophane. Au cours de la préparation de la colophane esterifiée, une partie du glycérol était remplacée par polystyrène glycol, ce qui rend des esters mixtes ayant un point de fusion plus élevé. On a préparé des vernis gras à partir de ces esters et d'huile de lin. Les caractéristiques des films de ces vernis gras ont été comparées avec celles des vernis à base de la colophane esterifiée et aussi à base d'une résine alkyde. On a trouvé que les films des vernis à base des esters

mixtes séchent plus vite que ceux du vernis à base de la colophane esterifiée et ceux de la résine alkyde. La souplesse, l'adhérence, la résistance à la rayure, à l'eau, aux acides, alcalis, et solvants, des esters mixtes étaient supérieures à celles de la colophane esterifiée. A l'égard de la résistance aux alcalis et aux solvants, les esters mixtes étaient supérieures même aux alkydes. Toutefois, leur résistance à l'eau et aux acides étaient semblables à celles d'une alkyde à l'huile de lin, et la résistance à la rayure était inférieure à celle d'une alkyde à l'huile de lin.

Polystyrolglykolester des Kolophoniums in Anstrichstoffen

Zusammenfassung

Polystyrolglykol wurde hergestellt und zur Veresterung von Kolophonium verwendet. Bei der Herstellung des Kolophoniumesters wurde ein Teil des Glycerins durch Polystyrolglykol ersetzt und höher schmelzende gemischte Ester wurden erhalten. Leinölfirnisse wurden dann aus diesen Mischestern erzeugt. Die Eigenschaften dieser Firnisse wurden dann mit normalem Glycerin Kolophoniumester verglichen. Man fand dass die Mischesterfirnisse schneller als schnell als die Kolo-

phoniumesterfirnisse und auch schneller als Alkyde trockneten. Biegsamkeit, Haftfähigkeit, Härte der Mischester, sowie Beständigkeit gegen Wasser, Säuren, Alkalien und Lösungsmittel waren auch besser als die des Kolophoniumfirnisses. Die Mischester waren sogar den Alkyden in Bezug auf Alkali- und Lösungsmittelbeständigkeit überlegen. Die Wasser- und Säurebeständigkeit war ähnlich der des Leinölfalkyds, aber ihre Härte war der des Alkyds unterlegen.

Introduction

Refs. 1-14

Rosin is rarely used as such because of its acidic nature and tendency to crystallise in organic solvents. Rosin is

easily oxidised by the absorption of atmospheric oxygen due to the presence of the conjugated centre. These characteristics give rise to undesirable film properties, such as lack of durability, poor water and chemical resistance, etc. Therefore, it is necessary to modify rosin¹ by the reaction of various chemicals, either at the double

bond^{2,3} or at the carboxylic group^{4,5}. Esterification with alcohol and alkalis gives esters of rosin that are extensively used as varnishes and as common paint vehicles in the surface coating industry⁶.

Esterification with glycerol⁴ (a triol) gives the glyceryl ester of rosin, commonly known as ester gum in the surface coating industry. Other esterified products, in which glycerol has been partially or wholly replaced by other polyols such as pentaerythritol⁷ (a tetraol) and ethylene glycol⁵ (a diol), have also been prepared. Furthermore, many polymers having free hydroxyl groups such as phenolics^{8,9}, alkyds¹⁰ and epoxy¹¹ resins have also been used to modify rosin.

In the present work, polystyrene glycol¹² synthesised by the authors and successfully incorporated into alkyds^{13,14} has been esterified with rosin. In the preparation of ester gum, a part of glycerol has been replaced by polystyrene glycol. The products so obtained were named as mixed esters. Oleoresinous varnishes have been prepared from these mixed esters using linseed oil. In order to establish the usefulness of the products, their film properties have been compared with those of ester gum varnishes and also with those of linseed oil alkyd of 50 per cent oil length.

Experimental

Refs. 12, 15, 16

Materials

Oil

Alkali refined linseed oil was used in the preparation of the linseed oil alkyd and oleoresinous varnishes.

Rosin

WW grade rosin was used in the preparation of various esters.

Glycerol

BDH, LR grade was used in all esterification reactions.

Phthalic anhydride

BDH, LR grade was used in the preparation of alkyd resin.

Driers

Lead and cobalt naphthenates were used as driers.

Solvents

Kerosene oil, white spirit and xylene were used as solvents.

Polystyrene glycol

Polystyrene glycol¹² was prepared by free radical polymerisation of styrene, using benzoyl peroxide as initiator, and subsequent hydrolysis of the benzoate end groups. The sample had an hydroxyl value of 39.8.

Ester gum

Ester gum was prepared from rosin and glycerol according to known methods described in the literature¹⁵.

Alkyd resin

Linseed oil alkyd of 50 per cent oil length was prepared by the monoglyceride process¹⁶. Acid value of the resin was 12. Alkyd resin was dissolved in 1:1 xylene and kerosene oil mixture to give a 50 per cent solid content.

Preparation of mixed esters

Three samples containing 16, 28 and 43 per cent polystyrene glycol were prepared. The amount of polystyrene glycol was calculated on the basis of the hydroxyl value of polystyrene glycol. The amount of the ingredients used in various samples is shown in Table 1.

A typical method for the preparation of mixed esters is described as follows: Polystyrene glycol, rosin and glycerol were placed in a three necked flask fitted with a stirrer, thermometer and Dean and Stark water separator. The mixture was heated to 280°C. Heating was continued until the evolution of water ceased and a drop in acid value was observed. Mixed esters so obtained were characterised by their melting points and solubility (shown in Table 1).

Preparation of oleoresinous varnishes

Oleoresinous varnishes were prepared from mixed esters (prepared as above) and ester gum using alkali refined linseed oil. The ratio of ester to oil was 1:1 by weight. Preparation involved heating linseed oil to 250°C followed by the gradual addition of pieces of ester resin under continuous stirring. The temperature was then raised to 300°C and maintained until a clear product of the desired viscosity was obtained.

Characterisation and evaluation

An adequate amount of driers on the basis of the oil content was added to all oleoresinous varnishes and alkyd resin. All samples were thinned with kerosene oil to a brushable consistency. Films were applied onto 6 × 2 inch

Table 1
Composition and characterisation of ester gum,
mixed esters of rosin and linseed alkyd

Sample	Rosin	Parts by weight Glycerol	Polystyrene glycol	Percentage of polystyrene glycol	Acid value	Melting point °C
Ester gum	100	12.0	—	—	9	69
Mixed esters 1	100	11.6	20	16	10	72
Mixed esters 2	100	11.1	40	28	10	75
Mixed esters 3	100	10.4	75	43	11	86
Linseed alkyd	50	18.4	39	—	12	—

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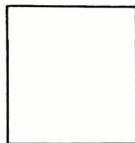
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glass and tin panels, and were evaluated for drying characteristics, flexibility and adhesion, scratch hardness and resistance to water, acid, alkali and solvent.

Results and discussion

Refs. 12, 17, 18

Polystyrene (a commercial sample) and polystyrene glycol were both tested to see if they were compatible with rosin, (i.e., if physical blends of polystyrene or polystyrene glycol and rosin were clear). When the mixtures were heated with linseed oil, clear oleoresinous varnishes were not obtained from these blends because both polystyrene and polystyrene glycol were not compatible with the oil. This suggested that the useful properties of polystyrene could not be incorporated into rosin merely by blending the two. A big advantage of the chemical reaction of polystyrene glycol and rosin was that clear oleoresinous varnishes could be prepared from the mixed esters. In support of this it is worth mentioning that the chemical reaction between polystyrene glycol and linseed fatty acids has also yielded clear products¹⁷.

In fact, the polystyrene glycol¹² used is not exclusively a diol. It also contains a mono-alcohol (polystyrene chains having a hydroxyl group at one end and a phenyl group at the other) and non-reactive polystyrene (chains having phenyl groups at both ends). The mono-alcohol will also take part in the esterification reaction. Therefore, esters obtained were a mixture of diesters, monoesters, and a polystyrene homopolymer.

The mixed esters and their corresponding oleoresinous varnishes were soluble in benzene, toluene, xylene and also in kerosene oil and white spirit. It was interesting to note that polystyrene glycol, being insoluble in kerosene oil and white spirit, when esterified with rosin gave esters which were soluble in these low cost solvents.

The melting point of the mixed esters was determined by capillary method (shown in Table 1). The mixed esters had higher melting points than that of ester gum. The melting point of mixed esters increased with increasing amounts of polystyrene glycol. This is due to the increased molecular weight of the mixed esters.

Films of these mixed esters, ester gum varnish and linseed alkyd were tested for drying characteristics, flexibility and adhesion, scratch hardness and resistance to water, acid, alkalis and solvent. The results are shown in Table 2.

Drying characteristics

Films of all samples, i.e. of linseed alkyd resin and oleoresinous varnishes prepared from ester gum and mixed esters, were air dried. Surface dry, hard dry and tack free times are recorded in Table 2. It was observed that the drying times of mixed ester varnishes were shorter than those of ester gum varnish and alkyd. Furthermore, the drying time decreases with increasing amounts of polystyrene glycol in the mixed esters.

Flexibility and adhesion

Flexibility and adhesion of the dried film was tested on tin panels with a quarter inch mandrel. No detachment of the film from the substrate or visible cracks in the film were observed in any of the samples, showing that all the films had good flexibility and adhesion. Furthermore, it confirmed that polystyrene glycol reacted with rosin and the mixed esters obtained yielded compatible oleoresinous varnishes.

Scratch hardness

Scratch hardness of the dried film was measured on the tin panels with a mechanically operated "Sheen" scratch hardness tester by placing increasing loads on a hardened needle moving over the film. The maximum scratch hardness (in grams) of all samples is shown in Table 2. Mixed esters having polystyrene glycol had better scratch hardness than that of ester gum. Furthermore, scratch hardness increased with increasing amounts of polystyrene glycol in the mixed ester. This is due to the cyclic rings present in polystyrene which impart greater hardness. However, scratch hardness of the mixed ester is inferior to that of linseed alkyd.

Water resistance

All the samples were allowed to air dry in a horizontal

Table 2
Drying characteristics, physical properties and resistance to water, acid, alkali and solvent of linseed alkyd and oleoresinous varnishes prepared from ester gum and mixed esters of rosin

Sample	Drying characteristics			Maximum scratch hardness	Condition of the films immersed in						
	Surface dry	Hard dry	Tack free		H ₂ O	Na ₂ CO ₃	NaOH	HCl	H ₂ SO ₄	HNO ₃	Xylene
	(min)	(hr)	(hr)		(g)	after 4 days	after 4 days	after 4 hrs	after 8 days	after 8 days	after 4 days
Ester gum	30	4	Within 12	1,100	0	2	0	2	3	1	0
Mixed esters 1	20	3	Within 12	1,200	2	3	1	3	4	2	2
Mixed esters 2	15	3	Within 8	1,600	3	3	2	3	4	3	3
Mixed esters 3	10	2	Within 6	1,800	4	4	3	3	4	3	5
Linseed alkyd	60	6	Within 18	3,000	4	0	0	3	4	3	0

5 - Film practically unaffected
4 - Slight loss of gloss
3 - Loss of gloss and change in colour

2 - Film partially cracked
1 - Film cracked and partially removed
0 - Film completely removed

position for 48 hours. The edges of the glass panels were protected with wax before performing this test. Panels were immersed in distilled water at room temperature (about 25°C). The dipped portion of the film was examined for appearance, i.e., loss of gloss, change in colour, and for any other visible damage. It was observed that after 4 days of immersion, the film of mixed esters having 16 per cent polystyrene glycol was partially cracked, and that having 28 per cent polystyrene glycol showed loss of gloss and change in colour. Slight loss of gloss was observed with mixed esters having 43 per cent polystyrene glycol with the linseed alkyd. The film of ester gum varnish was, however, completely removed.

These results clearly indicate that the use of polystyrene glycol during the preparation of esters of rosin, considerably improves the water resistance of the products. Furthermore, the water resistance increases with increasing amounts of polystyrene glycol in the mixed esters. The water resistance of the mixed ester having 43 per cent polystyrene glycol is as good as that of the linseed alkyd.

Acid resistance

For this test the glass panels of all the samples were prepared as described above and were immersed in 2 per cent solutions of each of hydrochloric, sulfuric and nitric acids separately at room temperature. Panels were taken out, washed in fresh running water and were allowed to air dry for an hour and checked at regular intervals of 2 days for appearance, i.e., loss of gloss, change in colour and for any visible sign of disintegration. Table 2 gives a clear picture of acid resistance of all the samples.

After 8 days immersion in hydrochloric acid, linseed alkyd and all the samples of mixed esters showed loss of gloss and a change in colour, whilst the film of ester gum was partially cracked. In sulfuric acid, slight loss of gloss was observed in all the esters except ester gum, which showed loss of gloss and a change in colour. Results of immersion in nitric acid also revealed that mixed esters have better acid resistance than that of ester gum, because, after 4 days of immersion in nitric acid, the film of ester gum was cracked and partially removed. The film of mixed esters having 16 per cent polystyrene glycol was partially cracked, while those having 28 and 43 per cent polystyrene glycol showed loss of gloss and a change in colour. These results established that the mixed esters have better acid resistance than ester gum.

Alkali resistance

Glass panels of all the samples were prepared as described before and were immersed in 2 per cent solutions each of sodium carbonate and sodium hydroxide separately at room temperature. Panels dipped in sodium carbonate solution were taken out at regular intervals of 2 days, washed in fresh running water, dried and the film examined for any visible damage. Films in sodium hydroxide solution were checked at a regular interval of 2 hours. The results are shown in Table 2.

After 4 days immersion in sodium carbonate, the film of ester gum was partially cracked. Loss of gloss and change in colour were observed in mixed esters having 16 and 28 per cent polystyrene glycol. Slight loss of gloss was observed in mixed ester having 43 per cent polystyrene glycol. However, the film of linseed alkyd was completely removed.

Results of immersion in 2 per cent sodium hydroxide solution showed that films of linseed alkyd and ester gum were completely removed in 4 hours. The film of mixed ester having 16 per cent polystyrene glycol was cracked and partially removed, and that having 28 per cent polystyrene glycol was partially cracked. Loss of gloss and change in colour were observed in the mixed ester having 43 per cent polystyrene glycol.

These results show that the mixed esters of rosin have better alkali resistances than those of ester gum and linseed alkyd, and furthermore, alkali resistance increases with increasing amounts of polystyrene glycol in the mixed esters.

Solvent resistance

Panels were dipped in xylene at room temperature. After 2 days, the panels were removed and allowed to stand in a vertical position for 5 minutes and then rubbed with cotton wool soaked in xylene. Table 2 shows the conditions of the films. Films of ester gum and linseed alkyd were completely removed. The film of mixed esters having 16 per cent polystyrene glycol was partially cracked and that having 28 per cent polystyrene glycol showed loss of gloss and change in colour. The film of mixed esters having 43 per cent polystyrene glycol was practically unaffected.

These results indicate that although polystyrene glycol has a linear chain and is soluble in xylene, it does not decrease the solvent resistance of the mixed ester. This might be due to the chemical reaction occurring between rosin and polystyrene glycol forming strong chemical bonding. In fact, the same results were observed with the copolymers¹⁸ of polystyrene glycol and glyceryl phthalate. The copolymers showed better solvent resistance than that of styrenated alkyd.

Conclusion

It can be concluded that polystyrene glycol can successfully replace, partially or wholly, the glycerol used in the preparation of ester gum. These mixed esters may find wide application in surface coatings due to their good physical and chemical properties comparable to those of alkyds.

Acknowledgement

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Effect of solvent on residual strain in clear epoxy coatings

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Summary

Coatings were formed from solutions of solid and liquid epoxy resins, cured with diethylenetriamine. The residual internal strain was measured and found, in all cases, to be considerable. The residual strain in coatings formed from the faster evaporating solvents showed no variation with coating thickness, but exhibited a small dependence on the concentration of the coating solution. These coatings, like lacquers, are considered to solidify

whilst still in a thermoplastic state, with subsequent crosslinking. Coatings cast from a very slow-evaporating solvent had a residual strain which increased greatly with coating thickness, because they solidified owing to the formation of a crosslinked network. Liquid epoxy resin formed coatings with much higher residual strain and poorer adhesion when cast from solution than when applied as a solventless formulation.

Keywords

Types and classes of coatings and allied products

clear coating
catalysed coating
solution coating

Raw materials for coatings binders (resins, etc.)

epoxy resin

paint additives, catalysts, accelerators, inhibitors

amine hardener

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

shrinkage
solvent retention

materials in general

evaporation rate

Miscellaneous terms

strain

L'effet du solvant sur la tension résiduelle en revêtements clairs époxydes

Résumé

Des revêtements ont été préparés à partir des solutions de résines époxydes, liquides ou solides, durcies par diéthylentriamine. La tension résiduelle a été mesurée et trouvée considérable dans tous les cas. La tension résiduelle des revêtements contenant des solvants qui évaporent plus rapidement ne démontre aucune variation en fonction de l'épaisseur du revêtement, mais elle se dépend légèrement de la concentration de la solution de résine. On considère que ces revêtements se solidifient pendant qu'ils sont encore à l'état thermoplastique, et

que la réticulation se produit subséquemment. Les revêtements déposés à partir d'un solvant à une très faible vitesse d'évaporation avaient la tension résiduelle qui augmentait fortement en fonction de l'épaisseur du revêtement, parce que leur solidification était due à la formation d'une structure réticulée. Les résines époxydes liquides donnaient des revêtements dont la tension résiduelle est beaucoup plus élevée et l'adhérence plus faible lorsqu'ils sont déposés à partir de leur solution que dans le cas où ils sont appliqués sous forme d'un produit exempt de solvant.

Der Einfluss von Lösungsmitteln auf die Restspannungen in klaren Epoxidbeschichtungen

Zusammenfassung

Beschichtungen aus mit Diäthylentriamin gehärteten Lösungen von festen und flüssigen Epoxidharzen wurden hergestellt. Die Restspannungen wurden gemessen und waren in allen Fällen beträchtlich. Die Spannungen in Beschichtungen aus schnell verdampfenden Lösungsmitteln waren unabhängig von der Dicke der Beschichtung, zeigten aber eine Abhängigkeit von der Konzentration der Harzlösung. Diese Beschichtungen wurden, wie Lacke, fest solange sie noch thermoplastisch waren und ver-

netzten später. Beschichtungen die aus langsam verdampfenden Lösungsmitteln gebildet wurden, ergaben Restspannungen die mit der Dicke der Beschichtung beträchtlich anwachsen da sie durch Vernetzung fest wurden. Flüssige Epoxidharze ergaben Beschichtungen mit viel höheren Restspannungen und schlechterer Haftfestigkeit wenn sie aus Lösungen aufgetragen wurden, denn wenn sie aus lösungsmittelfreien Formulierungen gebildet wurden.

Introduction

Refs. 1-3

Protective or decorative coatings are frequently applied

from solution by brush, dip or spray. These coatings must shrink because the solvent evaporates. In addition, any chemical changes that occur also tend to reduce the volume of the coating. Because the area of the coating is constrained to remain at its original wet size by its adhe-

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sion to the substrate, any solvent loss or chemical change that occurs after the coating solidifies will produce internal strain (or stress) in the plane of the coating because the coating can no longer flow to accommodate such changes. Internal strain seldom disappears completely even after the normal viscoelastic processes have relaxed the polymeric binder, and it remains to influence the cohesive or adhesive properties of the system.

The final value of strain depends on the drying process occurring between the solidification point of the coating and its final "dry" state.

Studies on solvent cast coatings^{1,2} have shown that the residual internal strain can be very large and may cause failure. Therefore, any reduction in internal strain may extend the service life of a coating and produce economic benefits in the form of savings in raw materials and labour.

Previous work by the author has been concerned with lacquers, which harden by solvent evaporation, and solventless epoxy coatings, which cure chemically³. This paper covers the investigation of the residual strain in solvent cast epoxies, in which both solvent evaporation and chemical cross-linking contribute to the formation of a dry, cured coating. The effects of solvent, coating solution concentration and dried coating thickness are also studied.

Residual strain, rather than stress, is determined because it is a simpler measurement and is a direct measure of the phenomena involved. Stress is the product of strain and modulus (which is itself affected by many factors) and thus may be prone to more experimental uncertainty.

Residual strain due to solvent loss

Ref. 2

In a coating that dries like a lacquer the residual internal strain depends solely on the difference between the volume of solvent present in the film at solidification, and the volume present in the final "dry" film. Before solidification the coating can flow to accommodate the volume change demanded by solvent evaporation; thereafter, further evaporation produces strain in the plane of the coating. The thickness is not constrained and can still contract. In lacquers the solidification point and the final dry state are the same throughout the coating, so they are characterised by a residual strain independent of the coating thickness.

The volume of solvent lost from the coating after solidification, ΔV , is given by:

$$\Delta V = \phi_s V - \phi_r (V - \Delta V) \dots \dots \dots (1)$$

where V = volume of coating at solidification;
 ϕ_s = volume fraction of solvent at solidification;
 ϕ_r = volume fraction of solvent retained in dry film.

Rearranging gives:

$$\frac{\Delta V}{V} = \phi_s - \phi_r \left(1 - \frac{\Delta V}{V}\right) \dots \dots \dots (2)$$

= internal bulk strain, assuming an exact correspondence with the volume of solvent lost

$$= 3\epsilon \dots \dots \dots (3)$$

where ϵ = isotropic linear strain, which is equivalent to the bulk strain.

From equations (2) and (3), internal strain is given by

$$\epsilon = \frac{\phi_s - \phi_r}{3(1 - \phi_r)} \dots \dots \dots (4)$$

The volume fraction of solvent retained in the dry film, ϕ_r , can be measured directly. Measuring the concentration of solvent when the film solidifies cannot be done directly, at present.

Solidification

The lacquer mixture solidifies when it no longer behaves as a viscous liquid. At this point the solution is highly concentrated and the binder molecules retain their positions with respect to one another. Solidification in lacquer systems resembles the glass-rubber transition in solid polymers: above the transition temperature, T_g , large-scale molecular motion can occur; below it, the polymer molecules are "frozen" into a glassy state.

The presence of solvent in a polymer will lower T_g greatly, the depression increasing with solvent concentration. Thus ϕ_s can be identified as the solvent concentration at which the T_g of the coating coincides with the temperature of the test. If T_g is determined as a function of solvent content, ϕ_s can be found. This scheme is complicated by the diffuse nature of the glass transition. Molecular motion does not cease altogether at T_g but at some lower temperature (lower solvent content). It might be expected, then, that:

$$\phi_s = \phi_{23} - \Delta\phi \dots \dots \dots (5)$$

where ϕ_{23} = solvent content that depressed T_g to the test temperature (23°C in this work);
 $\Delta\phi$ = effective width of the transition in terms of solvent content.

The correction (equation 5), together with the equation for the internal strain has been successful in modelling the behaviour of lacquers².

Shrinkage in epoxies

Ref. 4-6

In general, the curing of epoxies results in shrinkage, because the chemical reaction produces a configuration that is more compact than the starting mixture of epoxy and hardener molecules. Thermal shrinkage is also possible if an elevated processing temperature is employed. However, all the coatings produced for this work were maintained at room temperature. The exothermic curing reaction did not produce any significant temperature increase because the coatings were too thin.

Most of the shrinkage takes place while the system is still in a liquid state^{4,5} and so will not manifest itself as internal strain. Nevertheless, values of post-relation shrinkage of 1 to 2 per cent, which will result in a corresponding residual strain, have been reported⁴.

In principle, residual strain (shrinkage) due to the curing reaction should not depend on coating thickness, because it depends solely on a chemical reaction. However, it has

been found for a solventless coating that carbon dioxide and water from air can react with the amine curing agent and block the crosslinking of the epoxy⁶. Thin coatings are affected most because carbon dioxide and water can diffuse furthest into them before the normal crosslinking reaction can take place. The result is a residual stress (or strain) that increases slowly with coating thickness.

In solvent cast epoxy coatings there are two mechanisms whereby the film can harden, solvent evaporation and chemical curing. Separately, neither mechanism should produce an internal strain that would depend to any great extent on coating thickness.

Experimental methods and results

Ref. 3

Ambient conditions were maintained at 23°C (±1°C) and 50 per cent (±2 per cent) R.H. throughout.

Materials

Two epoxy resins were used, Epon[®] 1004 (a solid) and Epon[®] 828 (a liquid); both were cured with diethylene-triamine (DETA). Eleven parts DETA per hundred of Epon 828 and 2.4 parts DETA per hundred of Epon 1004 were used to cure the resins.

Four solvents were used, methyl ethyl ketone (MEK), methyl isobutyl ketone (MIBK), Methyl Cellosolve[®] (MC, chemical name 2-methoxyethanol) and Dowanol[®] TPM (DTPM, chemical name tripropylene glycol monomethyl ether).

Generally coating solutions were applied to the tinplate substrates about one hour after mixing. The exact duration of this initial period did not seem to be crucial, especially because bulk solutions took two or three days to solidify.

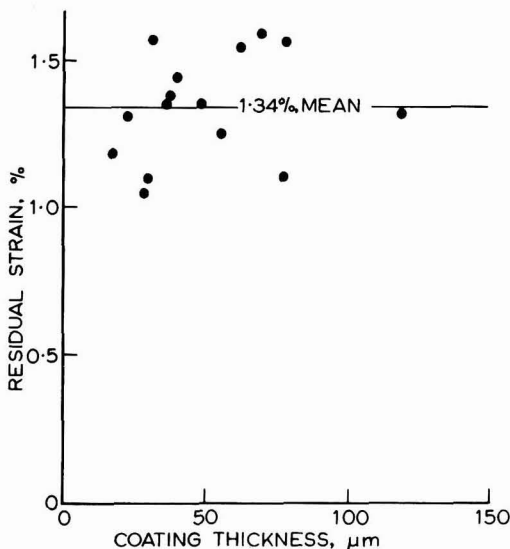


Figure 1. Residual strain (50/50 epoxy/MEK) as a function of cured coating thickness, after 293 days

Residual Strain

Residual strain was measured by releasing a dried coating from a tinplate substrate and monitoring its subsequent shrinkage which was taken to be identical with the internal strain in the coating at that time. Films of varying thickness were drawn down onto a masked area of tinplate. After measuring the lengths of the film once it was dry, it was released by amalgamating the tin with mercury. Drying was monitored by weighing the coating of the substrate. A typical coating weight was 25 mg and differences of 0.1 mg could be measured. Drying took several weeks, depending on the solvent used.

Solid epoxy/MEK

Figure 1 shows the relative shrinkage (residual internal strain) as a function of coating thickness for a 50/50 (by weight) solution of solid epoxy and MEK (cured with the appropriate amount of DETA). These data represent results taken 10 months after application of the coatings.

The graph shows no systematic trend with dried coating thickness.

Mean value of residual strain = 1.34 per cent
Standard deviation = 0.18 per cent

Shrinkage increased with time as can be seen from Figure 2.

Solid epoxy/MIBK

Figures 2 and 3 show similar data for a 50/50 solution in MIBK. Again there is no systematic increase in strain with coating thickness.

Mean value of residual strain = 1.20 per cent
Standard deviation = 0.18 per cent

Solid epoxy/MC

In this case three solutions were made containing 30 per cent, 50 per cent and 70 per cent by weight in MC, with the appropriate amount of DETA added to cure the coatings.

Figure 4 gives the strain after 7 months; none of the results show any trend with film thickness. However, there is a small, but systematic trend with the coating solution concentration (see Table 1).

Table 1
Mean value of residual strain in solid epoxy/MC coatings after 7 months

Concentration per cent w/w	Residual strain per cent (std. dev.)
30	1.81 (0.17)
50	1.67 (0.16)
70	1.38 (0.17)

The change with time is shown in Figure 2. After 10 months the values for the 50 per cent w/w coatings were,

Mean value of residual strain = 1.84 per cent
Standard deviation = 0.16 per cent

It was noted that coatings made from solution in MC could be stripped from the substrates more easily than those from MEK or MIBK solutions.

Solid epoxy/DTPM

Coatings prepared from a 50 per cent w/w solution in DTPM exhibit a behaviour completely different from that of coatings formed from the other solutions. Figure 5 shows that the residual strain increases considerably as

coating thickness increases. Thicker coatings were easily removed from the substrates, whereas thin coatings retained good adhesion. Figure 6 shows the trend of shrinkage with time for a few examples of these coatings.

Liquid epoxy/MC

A 50 per cent w/w solution was made in MC and cured with DETA. Coatings formed from this solution exhibited appreciable residual strain (Figure 7).

Mean value of residual strain = 0.92 per cent
Standard deviation = 0.14 per cent

No trend in ϵ_r can be detected with coating thickness. Some of the thicker coatings peeled from the tinplate substrates spontaneously.

The pot-life of such solutions was about 3 hours as opposed to 40 min. for the solventless formulation³.

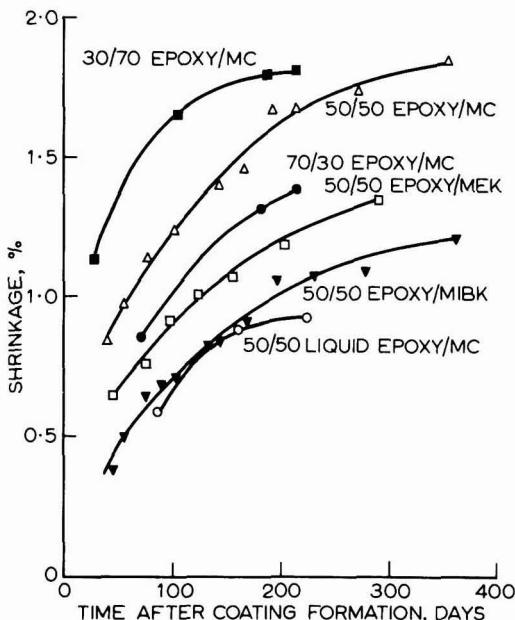


Figure 2. Shrinkage in film length as a function of time after film formation. (Average of all coatings from a given formulation)

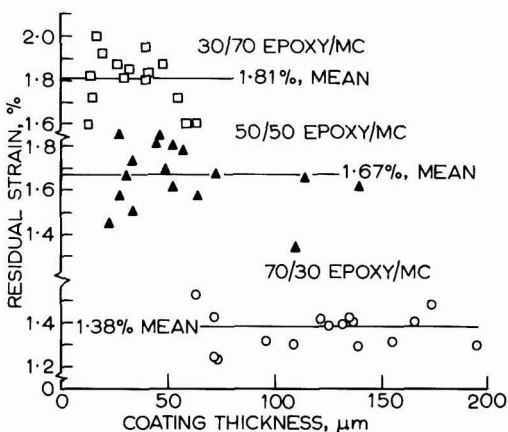


Figure 4. Residual strain (epoxy/MC) as a function of cured coating thickness and solution concentration, after 220 days

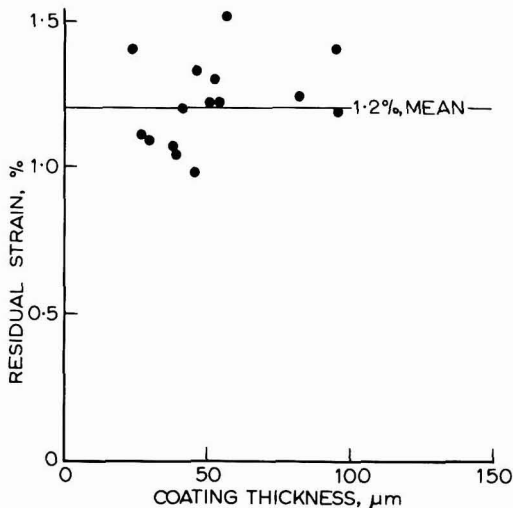


Figure 3. Residual strain (50/50 epoxy/MIBK solution) as a function of cured coating thickness, after 365 days

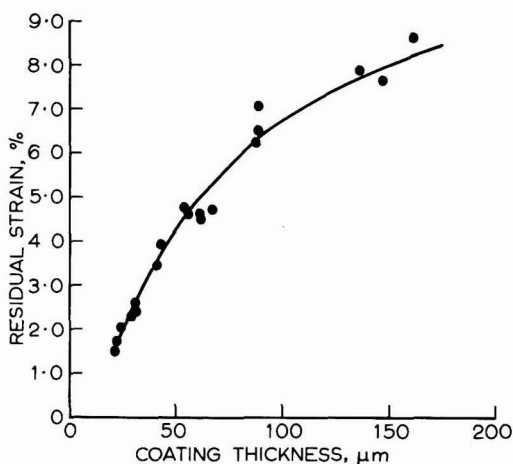


Figure 5. Residual strain (50/50 epoxy/DTPM) as a function of cured coating thickness, after 288 days

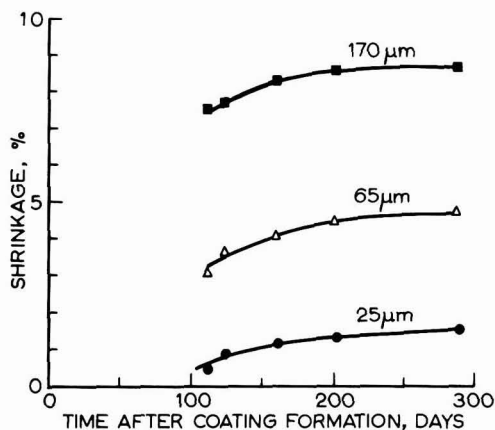


Figure 6. Shrinkage as a function of time after film formation, for various coating thicknesses (50/50 epoxy/DTPM solution)

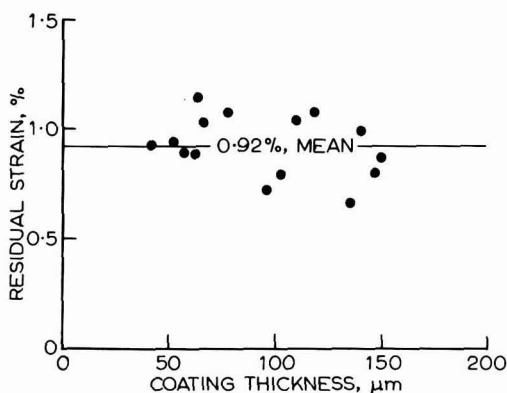


Figure 7. Residual strain (50/50 liquid epoxy/MC) as a function of cured coating thickness, after 226 days

Young's modulus

The Young's moduli of coatings prepared from the different solutions were measured to elucidate the reasons for differences in residual strain between these solutions. Measurements were made on coatings that had been dried for the maximum time indicated in Figure 2. A conventional tensile tester was used at a strain rate of $2 \times 10^{-4} \text{ s}^{-1}$. The modulus was obtained from the initial slope of the load-extension curve. Results are given in Table 2.

Table 2
Various properties of dry crosslinked coatings of solid epoxy

Coating solution	Young's modulus GPa	Softening temp. °C	Retained solvent concentration % v/v
50/50 epoxy/MEK	2.1	64	4.5
50/50 epoxy/MIBK	2.2	52	14.3
30/70 epoxy/MC	2.3	80	0
50/50 epoxy/MC	2.2	70	0
70/30 epoxy/MC	2.1	68	0
50/50 epoxy/DTPM	1.4	43	4.1

Glass transition temperature

Lacquers

Solutions of solid epoxy were made using MEK, MIBK and MC, but no DETA was added. Films containing varying amounts of solvent were formed by drying them at room temperature and above (in an oven). The influence of solvent on T_g is used to explain the residual strain behaviour of lacquers and can be compared to the results obtained for the crosslinked coatings.

Prior to testing, all films were allowed to stand at the controlled room temperature and humidity for several days. Otherwise, it was found that coatings formed at elevated temperatures produced inconsistent results. It is believed that the inconsistency was due to absorption of varying amounts of water (which plasticises epoxies) from the air. No samples were prepared at low temperatures (high solvent content), because there was loss of solvent during the time taken for them to absorb an equilibrium amount of water at room temperature.

Data for T_g were obtained using a Dupont 941 Thermo-mechanical Analyser (TMA). This was used in preference to a Differential Scanning Calorimeter (DSC) (as used in other work) because the peak in specific heat at the glass transition was rather indistinct with this method when significant amounts of solvent were present. At T_g , the coating softens significantly causing the TMA probe to penetrate the coating. The TMA probe was a quartz rod, loaded with a 1 g weight. The machine recorded the derivative of the probe movement, so that the temperature at which the derivative peaked was taken as the softening point (which varies with heating rate). A range of heating rates was employed, 0.5°C/min. to 20°C/min., and the results were extrapolated graphically to zero heating rate to give a value for T_g . For the pure resin, TMA gave T_g as 64.5°C; a similar procedure on the DSC gave 62°C. Since T_g was measured for the purposes of discussing solidification it might be considered that the TMA results are a more direct measure of the phenomenon than DSC measurements of T_g .

T_g is plotted as a function of solvent content in Figure 8. Solvent content was found by redissolving the films in a known amount of cyclohexanone and determining the ratio of the two solvents by gas chromatography (Perkin-Elmer 3920B with Integrator M-1); see ASTM E260 and D3271.

Crosslinked coatings

The softening temperature (T_g) was measured for the solvent-cast, crosslinked coatings on samples that had been used to measure residual strain and were several

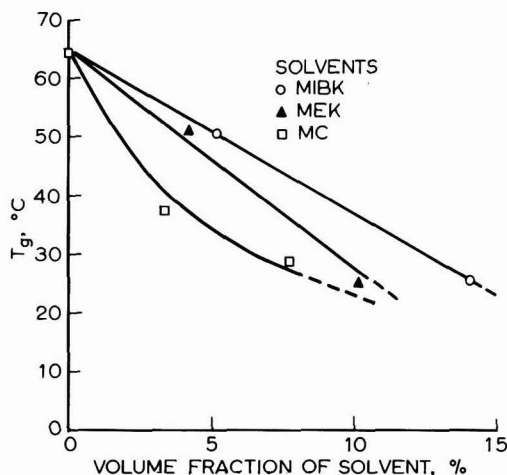


Figure 8. Variation of T_g with solvent content for epoxy lacquers (non-crosslinked)

months old. The purpose was to detect differences between the coatings.

Only one, fast, heating rate was used, $10^\circ\text{C}/\text{min.}$, so that the coatings would not change their crosslink density under prolonged heating. The values of softening point are given in Table 2 for that heating rate.

Residual solvent was detected using the gas chromatograph. Crosslinked films cannot be redissolved, but after standing for 18 hrs in cyclohexanone, samples of the films were swollen and extremely soft. Most residual solvent would be expected to leach out into the cyclohexanone. The quantities of solvent detected are given in Table 2. Hereafter, these values are quoted and used under the assumption that they represent the solvent retained in the cured films. In films deposited from the fast solvents there appeared to be a slight increase in retained solvent with thickness, but the value given would be representative of the average coating thickness employed. The trend was more marked in films deposited from DTPM, where the values given in Table 2 are typical of films $65\ \mu\text{m}$ thick.

Discussion

Refs. 3, 7-10

With the exception of the coatings formed from the solid epoxy in DTPM, there appears to be no variation of residual strain with the dried coating thickness, for a particular formulation. In this respect the coatings seem to behave like lacquers. Variation of internal strain (stress) on coating thickness found in a solventless epoxy coating³ is probably also present in these coatings, although it is a very small effect compared to the strains recorded here and thus would appear as part of the experimental scatter.

Those coatings that contained the highest strain, or were thickest, showed the poorest adhesion, by virtue of their high internal strain energy which may overcome the interfacial work of adhesion⁷.

All coatings showed a gradual, but considerable increase in residual strain over a long period (Figures 2

and 6) after the coating weight had stabilised. This must be due to the crosslinking proceeding to completion. It has been shown that such crosslinking reactions are controlled by viscosity in the coating system⁸, which explains why the shrinkage continues for so long in the solid coatings. This increase in residual strain over an extended period will present an increasing danger to the performance of such coatings.

Coatings with strain independent of thickness

MEK, MIBK and MC all evaporate much faster than DTPM (Table 3). Coatings formed from the fast solvents probably harden because the solvent evaporates, rather than because of a crosslinked network. The fully dried coatings were insoluble and therefore crosslinked, but at the point of solidification they were probably still in a thermoplastic state. Completion of network formation is then reached slowly.

Crosslinking starts immediately after the DETA is added to the resin solution and results in a continuously increasing molecular weight (and thus of T_g and ϕ_p), so that it is impossible to apply equation (4) exactly as for a lacquer. However, extrapolating the curves in Figure 8 to 23°C and using the value for ϕ_p in Table 2 predicts values for ϵ_p , which are at least in the same order as the experimental data (Table 3). The width of the glass-transition ($\Delta\phi$) was not considered here because of the changing nature of the polymer.

The results lend support to the idea that coatings using these fast solvents harden initially as lacquers. The exact amount of residual strain, however, is determined to a great extent by the crosslinking.

It is not possible to perform a similar calculation for the coating that originated as a liquid resin. The results do show that casting from solution dramatically increases the residual strain. For the solventless formulation, strain was 0.01 per cent³ whereas films deposited from MC solution using the same hardener and resin give a strain of 0.92 per cent. If the strain is to be minimised or adhesion improved these films are best formed without solvent.

Solubility parameter data⁹ indicate that MC is the best

Table 3
Evaporation rate of solvents

Solvent	Relative evaporation rate*
Methyl Ethyl Ketone	572
Methyl isobutyl ketone	165
Methyl Cellosolve ^R	47
Dowanol ^R TPM	< 1

*n-butyl acetate = 100

^RRegistered Trademark

Table 4
Predictions of residual strain in solvent cast coatings of solid epoxy using equation (4)

Solvent	ϕ_{23}	ϵ_i	
		Predicted	Observed
MC	0.10	0.033	0.018
MEK	0.11	0.024	0.013
MIBK	0.15	0.003	0.012

solvent for epoxy, followed by MIBK, MEK and DTPM. This ranking does not correlate with the retained solvent concentrations reported in Table 1. For example MC is not retained at all, whereas MIBK seems to be retained in large quantities.

There are differences in residual strain between the three solutions of solid epoxy in MC. Young's modulus and T_g data on the coatings formed from MC also show a small but systematic trend. Coatings formed from the most dilute solution give the highest values, which may indicate a greater degree of crosslinking due to the lower viscosity of the solution. (The 50/50 coatings were older, as shown in Figure 2.) The more the epoxy molecules crosslink before the solution solidifies, the higher T_g becomes. Thus there is more solvent (ϕ_s) in the film at solidification, and more internal strain left when the film is dry and cured.

The coatings formed from the solid epoxy in an MC solution gave a higher value of residual strain than did the coatings produced from solution in the much more volatile MEK. Thus, fast evaporating solvent is not always undesirable from this viewpoint. A similar result has been reported elsewhere¹⁰.

Coatings formed from solution in DTPM

Coatings formed from a solution in DTPM show a residual strain that increases considerably with cured coating thickness. This is because DTPM is an extremely slow evaporating solvent. In this case the coating solidifies by formation of a crosslinked network before the solvent can escape sufficiently to harden the coating like a lacquer.

The solvent content at solidification, ϕ_s , is higher overall in thick coatings because less DTPM has diffused to the surface and evaporated by the time solidification occurs. In thicker coatings, there will be a greater difference between ϕ_s and ϕ_r (the retained solvent) and hence a greater residual strain. This type of behaviour might be regarded as typical of coatings that harden by crosslinking, and will be the subject of a future publication, in which a theory to model the data in Figure 5 will be proposed.

Change in strain with time is only a small proportion of the final value (see Figure 6). This also indicates that more of the crosslinking is carried out before the film hardens.

If a surface coating shows an increase in residual internal stress with thickness, application is best done as a succession of thin coats. Residual strain will be lowered to that of a thin film if sufficient time for solidification by crosslinking is allowed between coats. If another coating is applied whilst the previous coating is still in a thermoplastic state, the resultant internal strain will have a higher value associated with the thicker coating. The solvent in the second coat will be able to redissolve the first coating, and become one with it.

Conclusions

All the solvent cast epoxy coatings showed a significant level of residual strain that would be detrimental to their performance as protective or decorative coatings. In fact, the thickest coatings or those with most strain adhered poorly. In contrast, a solventless epoxy coating, studied

previously showed a negligible residual strain and adhered well.

Except for the films cast from solution in DTPM, none of the formulations exhibited a systematic variation in strain with dried coating thickness. This suggests that the coatings solidify because of solvent evaporation, as with lacquers, and are in a thermoplastic state at solidification. A theory for calculating residual strain in lacquers correctly ranks the formulations according to their internal strain, even though it is inaccurate in terms of the absolute levels of strain. Completion of crosslinking in the films after solidification proceeds very slowly but adds considerably to the internal strain.

The fast evaporating MEK produces a coating with less strain than does the slower evaporating MC, a fact that might not be intuitively obvious.

More dilute coating solutions yield coatings with larger values of internal strain, probably because the molecular weight of the partially crosslinked epoxy is higher at solidification and thus ϕ_s is also higher.

The solidification of coatings cast from solution in DTPM occurs when a crosslinked polymer network is formed, because DTPM evaporates extremely slowly and the crosslinking occurs before the solvent evaporates. At the time of solidification the thicker coatings contain more solvent, which must diffuse out as the coating dries.

Thus the thicker coatings have to lose a proportionally greater volume of solvent after solidification, resulting in a greater internal strain when dry.

In practical situations coatings formed from solution in DTPM would be best applied as several thin coatings, to limit the internal strain.

Acknowledgement

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Preservation, priming, putty and paint*

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Summary

Some of the major factors in the painting of external woodwork, particularly windows, are considered. The principal cause of paint failure and wood decay is the movement at the joints of the lower transome of windows, which allows the ingress of water into the end-grains of the timber.

The properties of linseed oil putty and its manufacture are described, its role in sealing the glass into frames and the importance of its adequate and sufficiently frequent repainting are discussed.

Keywords

Types and classes of coatings and allied products

wood finish
wood stain
wood preservative
house paint
sealant
primer

Supplies and other materials primarily associated with surface preparation before painting

putty

Types and classes of structures or surfaces to be coated

wood

Properties, characteristics and conditions primarily associated with surface preparation before painting

putty

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

water resistance

Préservation, Peinturage, Peintures, et Mastics

Résumé

On considère certains des plus importants facteurs qui entrent en jeu lors du peinturage de menuiserie extérieure et, en particulier les châssis de fenêtres. La cause principale de l'insuccès des systèmes de peintures, et également de la pourriture de bois est le déplacement qui se produit entre les éléments des jointures de la traverse inférieure des fenêtres, ce qui permet à l'eau d'entrer dans le bois de bout.

On décrit à la fois les caractéristiques et le procédé de fabrication du mastic à l'huile de lin, et l'on discute son rôle à l'égard du scellement des vitres dans les croisés et, d'ailleurs, l'importance d'assurer le repeinturage à neuf adéquate et à une fréquence suffisante.

Holzschutz, Grundierung, Glaserkitt und Anstrich

Zusammenfassung

Einige der wichtigsten Faktoren, die beim Anstrich von Holzwerk, insbesondere Fensterrahmen die dem Wetter ausgesetzt sind, eine Rolle spielen werden in Betracht gezogen. Die hauptsächlichste Ursache des Vermoderns von Fensterrahmen ist das Eindringen von Wasser in die Fugen der unteren Querblende des Fensterrahmens.

Die Eigenschaften von Leinölkitt und seine Erzeugung werden beschrieben. Weiterhin werden auch die Rolle des Kitts beim Versiegeln des Glases und die Bedeutung des rechtzeitigen Übermalens diskutiert.

Introduction

Refs. 1, 2

Timber is one of the oldest building materials, whose greatest assets are its lightness, rapidity of construction and its versatility.

The bulk of external joinery in the United Kingdom is produced from Scots Pine (Baltic Redwood) known as *Pinus sylvestris*, whose sapwood is very porous and extremely permeable to moisture. Thus it is very susceptible to decay caused by wood destroying fungi.

Changes in the nature of softwood timber during the

last 30 years or more cause problems with decay never met before in the joinery industry. Before this time, and certainly during the Victorian era, softwood timber was imported from virgin forests where the growth rate was slow; the wood had a dense ring structure and only a small percentage of sapwood. The heartwood was extremely resistant to decay due to the presence of naturally occurring phenolic compounds.

Today the trees are grown quickly under cultivation conditions designed to produce early cropping and consequently contain a much higher proportion of sapwood. In order to reduce or avoid wood decay the Joinery Industry was forced to undertake large scale preservation. Joinery should be produced at a moisture content of less than 20

*An account of a lecture presented to the London Section at a meeting held at the Rubens Hotel on 25 October 1979

per cent because a higher moisture content leads to conditions favourable to decay. Quite apart from decay problems, the high porosity of the timber introduces movements which, in the author's opinion, is the major contributory factor in the failures experienced today in wooden windows.

The variation in moisture content is accompanied by shrinkage and swelling. The maximum swelling occurs in a saturated atmosphere, the wood absorbing 28 per cent moisture. At this point it is said to have reached the "Fibre saturation point".

Further wetting by contact will increase the moisture content above this level until it is said to be "waterlogged", although there is no further dimensional change. In the direction tangential to the annual rings, the movement, with Scots Pine, is about 5 per cent from the dry condition to the fibre saturation point. The movement in the radial direction is somewhat less, probably about 2.4 per cent for a similar change in moisture content.

Although wood preservation has been used for many years, the "new" timber, demands preservative systems which can be applied easily and effectively. The solvent types developed relatively recently contain tributyltin oxide and pentachlorophenol as specified in the Forest Products Research Laboratory's leaflet No. 24.

Large and minor local authorities, and organisations, such as the National Housebuilders Registration Council have insisted that: "Claddings, window frames, casements and sashes, external door frames, window surrounds etc. shall be treated against fungal attack with an approved preservative, except where approved durable timber has been used."

At this point, the author would like to point out that he is specifically concerned with wooden windows and not necessarily with any other part of a building, even if it is constructed from wood.

With regard to the porosity of wood, the author has established that there are wide variations, not only from one delivery of wood to another but also from one cut to another, from the same log. Porosity tests were carried out using the method developed by the Paint Research Association (PRA) employing isopropyl alcohol¹. So great are the differences that the PRA has recorded thirty-fold variations between normal and high porosity timber in the uptake of preservative. This means that in terms of cost (1970 values) a small window measuring 2 x 1 ft would vary from 0.5 p for normal porosity to 15 p for high porosity timber².

In order to improve the water resistance of "new" timber, water repellent types of preservative were produced containing paraffin wax, which due to the high porosity of the timber gave rise to further problems for the building industry.

The author visited a new school where the paint used was said to be "non-drying", the cause of which was directly due to the presence of wax in the preservative used. The primer and undercoat were highly pigmented and appeared to dry and had evidently tolerated the wax present, but when the finishing coat was applied the whole three coat system became a "cheesy mass".

This type of preservative should not be used when a

conventional oleoresinous system is used. It is to be noted that the manufacturer's instructions, usually printed on the paint tins, warn that "All surfaces must be clean, dry and free from wax, grease and dirt".

Priming

Ref. 3

The need for an efficient primer is all important and this cannot be over-stressed. Factory primed window frames are frequently exposed to inclement weather during transport, on the site and more particularly after they have been placed in position in the building.

Priming paints that allow water to penetrate the film, even when the wood has been treated with preservative, promote movement and opening of the joints and there is, therefore, no barrier to prevent water from entering the vulnerable end-grain which will take up water ten times faster than the surface grain.

For brush application white lead primer to BS 2521 is the only one which will give consistently good results. This type of paint has unique properties: high build, twice that of most other paints; excellent filling and sealing properties, particularly on the end-grains; elasticity and good weathering characteristics. The presence of lead soaps undoubtedly contributes to these properties. It has been said that "film thickness equals protection", and this type of paint satisfies this requirement.

White lead primer was in general use as a wood primer 50 years ago. There are an enormous number of houses over 100 years old where the wooden sliding sash windows had undoubtedly been painted originally with white lead paint and are still in good condition.

An alternative primer could be based on a suitable oleoresinous medium and applied by dipping. The medium chosen should have high resistance to water, good filling properties and build - in fact, as many of the features of white lead as can be obtained consistent with the formulation.

Dipping primers assist in "ironing out" the varying porosity of the wood and satisfying the demand at the end-grains. The author has observed this type of primer being applied at joinery works and has seen the resulting frames on site and in finished houses with excellent results. One of these primers was said to contain 20 per cent white lead.

During the last decade there has been an increasing use of water based primers. This type of paint is welcomed by the joinery industry because it is relatively cheap, it is fast drying (an effect which is enhanced by the wood absorbing some of the water), it has little smell and is without fire risk. It could be said that this type of paint helps the economy as it does not involve the use of petroleum solvents.

Water based emulsion primers are said to "breathe"; in fact, they have a much higher permeability than good quality oleoresinous, solvent based primers. The liquid water permeability is considerably higher than their vapour permeability. This implies that when applied to wood, water as a liquid passes more readily through the film than it could pass out as vapour.

Window frames primed with emulsion primers and

stored either on a building site or in position in a building without the protection of further coats of oil based paint would gain more water as the result of rain than could be lost during dry periods³.

The author was asked to investigate a paint and putty failure on a large building site involving 550 houses, where a water based priming paint had been used. In many cases the window frames were waterlogged at the lower transom rail and it was, in fact, possible to remove the whole of the putty from the rebate. The author is convinced that had an efficient oleoresinous primer been used, this failure would not have occurred or, at least, it would not have resulted in a disastrous failure.

Once again, it may be pointed out that under "Instructions for use" on paint tins and also on specifications for the usage of paint, it is laid down that all porous surfaces must be sealed; emulsion primers do not seal. The importance of an efficient primer is shown in Fig. 1, where it is shown that after priming, there is no further protection by paint at the primer-putty and putty-glass interfaces.

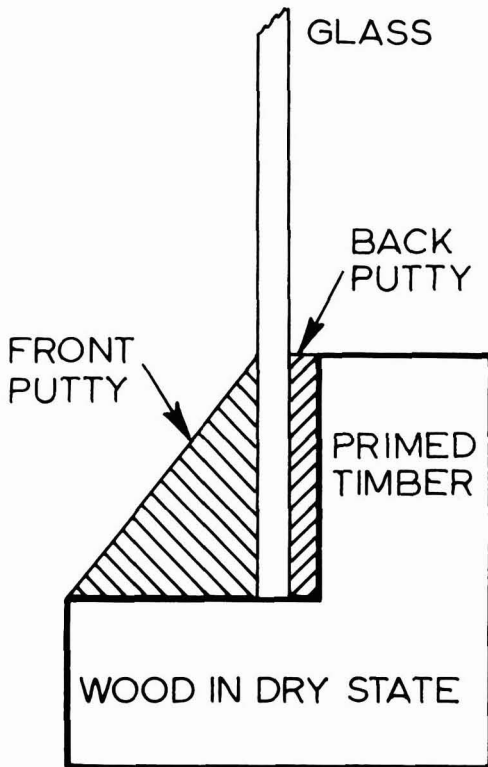


Figure 1.

Putty

Linseed oil putty, BS 544

The word "putty" is derived from the French "potée" (a potful) and its oldest recorded use dates from 1633, the word applied to a stiff paste made from lime and water and used by plasterers to fill the peripheral groove for

leaded lights. The same word is still used for linseed oil putty as known today.

It is usually associated with the glazing of windows, which may be defined as openings to admit light and air. It is interesting to note that in about 650AD St. Wilfred referred to the use of linen in window openings and in about 700AD small leaded lights, now so common in churches, were first imported from Gaul. The method of fixing was to use a mullion grooved to take a leaded light and to secure it with iron stanchions and saddle bars. The end of the 15th century saw Western Europe emerging from the Middle Ages into the Renaissance period, when windows in their present style were first introduced.

In general, the squaring up of window openings by adding transoms and mullions made the casement window possible and this led to the sliding sash window. From the introduction of leaded lights in England, nearly a thousand years were to pass before glass became commonplace. The first recorded use of linseed oil putty was in 1706. Formulation has remained unchanged since that time, except for minor modifications.

There can be few products that have stood the test of time so well. There must be an enormous number of windows in the UK well in excess of a hundred years old with the original putty still in nearly perfect condition. Linseed oil putty consists of approximately 12 per cent raw linseed oil and 88 per cent whiting. The acid value of raw linseed oil is adjusted by means of linseed fatty acids to a value of 2.5 to 4 or sometimes a little higher to facilitate wetting, and a small amount of soft soap is sometimes added for the same reason.

There are two main sources of chalk, whiting or naturally occurring calcium carbonate in England, one from the Thames area and the other from the Humber region. The whiting mainly used in putties comes from the Humber area and has a lower oil absorption than the Thames chalk, although the latter can be used quite successfully. Putty is a partly flocculated material and the degree of flocculation determines the end point. If a mixture of whiting and linseed oil were completely dispersed or flocculated it would be a "goeey" mass incapable of being used by the hands.

"Handleability" together with stability are the most important properties of a putty. BS 544 requires a material which can be stored for three months. When syneresis, or oil separation, is encountered it is difficult and sometimes impossible to reconstitute the putty, particularly in large packs.

Handleability: Putty should adhere to the frame, but not to the hands; it should have "length" which indicates the ease and speed of application; it should be soft enough to form a bed, but resilient enough not to be forced out of the back rebate when the glass is pressed home, as this would result in insufficient putty to form a complete seal.

Linseed oil putty should have good knifing properties and leave a smooth surface for painting. There are two principal methods of glazing, open or face glazing using linseed oil putty as just described. On metal frames it is hand applied or bead glazing is employed using gun mastics or non-setting compounds. Linseed oil putty containing no metallic driers takes 2 or 3 days to acquire an initial set and must be left for a period of time before painting (approx. 14 days) depending on the weather, although

this may be very much extended when painters are not available to do the work.

Attempts have been made to improve the performance of putty by adding polymers, synthetic rubbers etc. which invariably result in a harder working putty. Recently a modified putty has been produced which is easy to work and is claimed to have greater flexibility and can remain unpainted for a considerable period without cracking. If this is acceptable to the glazier it will undoubtedly be an advantage.

Many attempts have been made to define, mechanically or scientifically, the end point or "handleability" of putty, but it seems that only human hands can do this accurately. In order to anticipate changes in temperature, manufacturers produce summer and winter grades of putty. The cost of linseed oil putty is very low, being £2.50 per 5 litres as compared to £10 for the same quantity of paint. On one hand, there is paint which lasts only a few years and is much more expensive than linseed oil putty which, on the other hand, is expected to last for the life of a building, frequently quoted as 50 years and is usually much longer.

It has been observed that "putty usually cracks and falls out" but nothing could be further from the truth. Any failure of this nature is due to movement of the wood caused by open joints coupled with the ingress of water. (See Fig. 1b.) Many windows blown out during the war were replaced and left unpainted for 4 years or more, but the putty maintained a seal against the weather. Anyone who has to replace broken windows will know how hard it is to remove the old putty from the window frames and

**BREAKDOWN
SECOND STAGE**

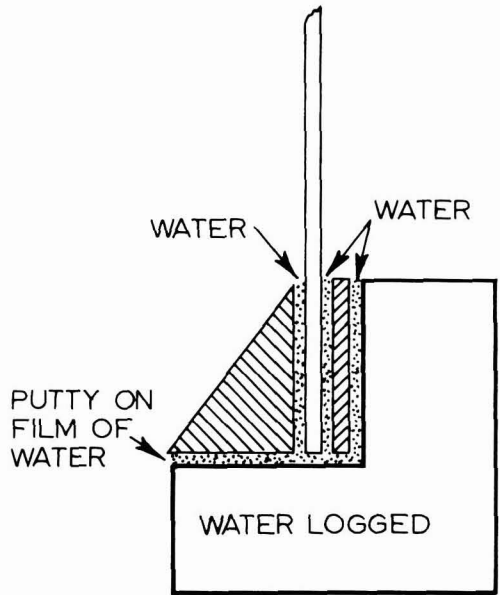


Figure 1c.

**BREAKDOWN
FIRST STAGE**

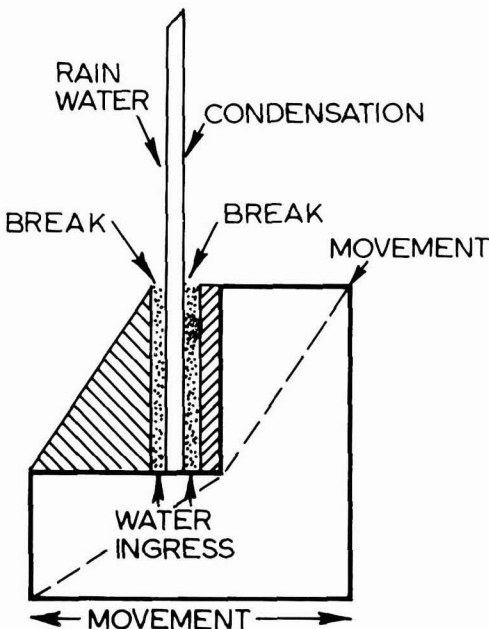


Figure 1b.

how tenaciously the old putty adheres to the rebate. If and when linseed oil putty does fail it is almost exclusively confined to the lower parts of the frames (See Fig. 3a).

Movement of the lower transom joints allowing ingress of water is invariably the principal cause of the joints opening and closing during the rainy seasons. This is fully indicated in Fig. 1. Adulteration is another cause of failure. Paraffin, white spirit, petrol and machine oil are all known to have been added in order to soften putty, particularly in cold weather, poor drying, cracking and wrinkling of the surface being the usual signs that this has been done. Linseed oil putty is often used in garden sheds and greenhouses made of wood. Here, no front putty is used but an efficient seal is still obtained. (Fig. 2.)

Manufacture of putty

In the author's opinion the best method is by edge runner mill. This method develops little heat and, therefore, the end point is easily and quickly determined by hand tests. Other methods, such as trough and vertical mixers, invariably develop heat and the putty has to be left to cool before it can be tested. There is also a danger of over flocculation and if this happens it is difficult, if not impossible, to remix and adjust the putty to produce a satisfactory product. Many attempts have been made to manufacture putty by a continuous process, but in the author's opinion, essentially, it requires batch operation.

Paint and painting

Ref. 4

Wooden windows, and in fact windows generally, have to withstand more wear than any other part of a dwelling.

GREENHOUSE GLAZING

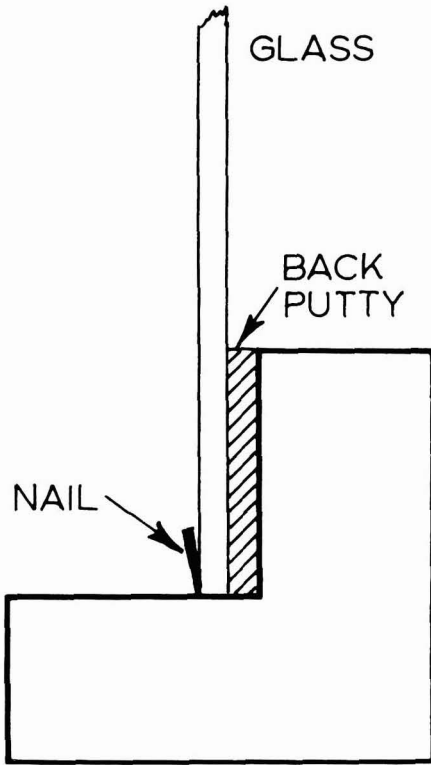


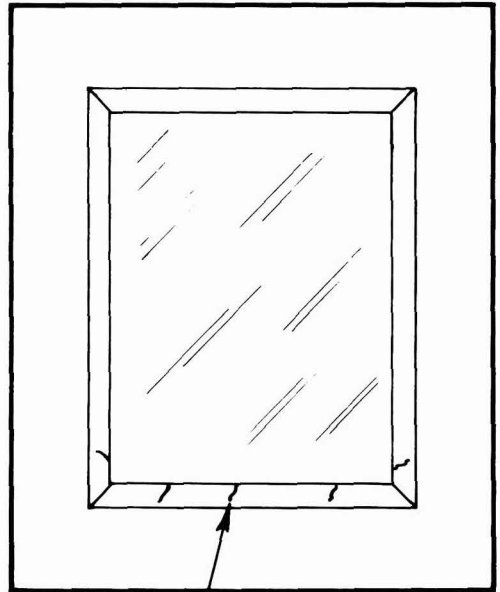
Figure 2.

They are frequently opened and shut and are constantly being cleaned inside and outside. Owing to the dust and dirt that accumulates on the outside of windows, cloths and leathers, used for cleaning, pick up so much dirt that there is a sand papering effect on the painted surface. Internally, if there is no double glazing, the housewife will frequently wipe windows to remove condensation, again causing more wear. Dust which invariably settles on the back putty will be vigorously removed and also the paint.

The author has always held the view that householders should be advised to repaint their wooden windows more frequently than the rest of the building. Another point is to make sure that all end-grains are coated at the top, bottom and sides of the window frames. Paint should be applied up to the sight line and on to the glass, where the putty is thinnest. This ensures a seal at the putty-glass junction (Fig. 4).

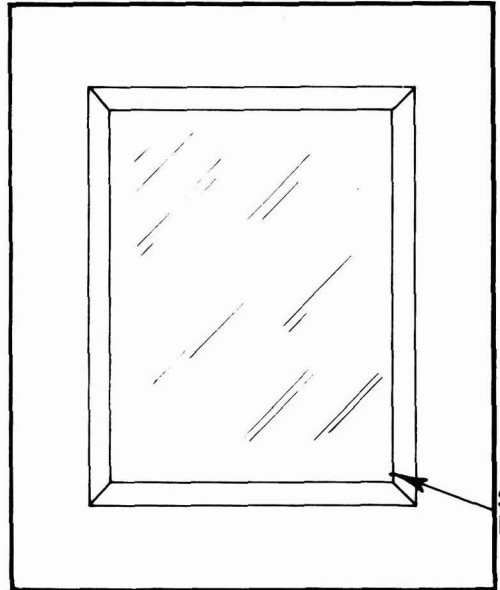
The author considers that good quality, free-flowing paints are better than the structured or gel types. Recently there has been considerable interest by specifying authorities in stain treatments for windows, which is said to be cheaper to maintain than conventional paint. These treatments take three main forms:

1. Lightly pigmented, semi-gloss oleoresinous materials in a variety of colours, pastel and dark, sometimes



PUTTY CRACKING AT LOWER TRANSOME

Figure 3.



PAINT APPLIED UP TO SIGHT LINE AND ONTO GLASS

Figure 4.

2. described as "architectural".
2. Stained or lightly pigmented varnish paints, principally in brown shades.
3. Wax-based coatings in dark colours, usually brown.

Some of these types of material are said to "breathe" or are classed as breathing paints. In the summer of 1976 this type of material was found to be unsatisfactory due to severe splitting of the wood, caused mainly by dehydration of the timber. Conventional paints did not suffer from this defect during the hot summer as they allowed the timber to retain its water content.

However, it has been stated that breathing paints are thermodynamically impossible⁴. It is unlikely that this type of finish would find favour with the general public, if they were free to choose their own colours, and, except where local authorities have specified dark colours on council

owned property, white is universally chosen for window frames. Any housewife will state that a white window will show off any kind of curtain material that may be chosen.

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The effect of ageing on the ease of decontamination of some paint systems

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Summary

The ease of radiological decontamination of a range of paints exposed to a variety of ageing environments has been assessed. Rutile pigmented aliphatic polyurethane and chlorinated rubber based paints have been shown to suffer the least change in ease of decontamination after exposure to water immersion, accelerated and natural weathering.

It is concluded that in general, the ease of decontamination of surface coatings will change considerably with age and that it is not at present possible to assess the long term ease of decontamination by the use of accelerated tests.

Keywords

Types and classes of coatings and allied products

top coat
gloss finish

Raw materials for coatings binders (resins, etc.)

polyurethane
epoxy resin
alkyd resin
acrylate resin
chlorinated rubber
phenolic resin

Processes and methods primarily associated with analysis, measurement or testing

accelerated weathering

service or utility

weathering

Miscellaneous terms

decontamination

L'effet de vieillissement sur la facilité de décontamination de certains systèmes de peinture

Résumé

On a déterminé la facilité de décontamination radiologique d'une gamme de peintures, après leur exposition dans divers environnements vieillissants. On a démontré que les peintures à base de polyuréthane aliphatique ou de caoutchouc chloré et pigmentées avec le dioxyde de titane du type rutile mettent en évidence la plus faible altération à l'égard de leur facilité de décontamination après leur immersion dans l'eau ou après vieillissement accéléré naturel.

On conclut qu'en générale, la facilité de décontamination de revêtements changera, d'une manière importante, en fonction du temps, et qu'à présent il n'est pas possible, au moyens des essais accélérés, de déterminer la facilité de décontamination à la longue.

Der Einfluss des Alters einiger Anstrichsysteme auf die Leichtigkeit der Entstrahlung

Zusammenfassung

Die Leichtigkeit mit der in verschiedenen Umgebungen gealterte Anstrichsysteme von radioaktiven Materialien befreit werden können wurde untersucht. Lacke auf aliphatischer Polyurethan- oder Chlorkautschukbasis, die mit Titandioxid pigmentiert sind, zeigten, nachdem sie Lagerung in Wasser, Schnellwitterung und natürlicher Bewitterung ausgesetzt wurden, die wenigsten

Änderungen in der Leichtigkeit mit der radioaktive Rückstände entfernt werden konnten. Im allgemeinen wird die Schlussfolgerung gezogen dass die Leichtigkeit der Entstrahlung von Beschichtungen sich beträchtlich durch Altern verändert und dass es momentan noch nicht möglich ist mit Hilfe einer Schnellprüfung die Leichtigkeit der Entstrahlung vorherzusagen.

Introduction

Refs. 1-8

The published literature on the decontamination of surface coatings is sparse¹⁻⁵ and, in general, covers *ad hoc* tests on proprietary materials for which no formulation details are available. Papers covering the effect of formulation variables on the ease of decontamination are even fewer^{6,7}, and of these, the paper by Walker⁶ provides reasonably

comprehensive decontamination data on the types of paint systems current at the time of publication. A more recent paper⁸, provides some decontamination data on more modern systems, and includes limited data on the effect of ageing on the ease of decontamination. No other papers are known to the authors which provide data on this important aspect of radiological decontamination.

The present paper attempts to redress the situation to a limited extent, by providing some ageing data on a number

Table 1
Percentage residual contamination
after Stages 1 and 2 of BS 4247,
Part 1, 1967

Test environments	Paint A		Paint B		Paint C		Paint D		Paint E	
	1	2	1	2	1	2	1	2	1	2
Controls (cure 2 weeks at RT)	23.09	22.13	1.03	0.96	16.72	14.01	6.05	1.82	10.32	10.29
BS 3900 Accelerated weathering chalky	2.43	1.52	2.88	1.49	8.21	4.33	2.16	1.62	10.21	5.45
BS 3900 Accelerated weathering non-chalky	7.69	6.92	0.79	0.50	4.48	2.79	1.30	1.09	7.14	4.34
Natural weathering chalky	19.60	15.09	6.08	3.60	17.61	9.69	11.65	8.75	11.81	7.69
Natural weathering non-chalky	17.03	14.72	5.65	3.48	20.99	12.31	5.32	4.16	7.35	4.91
Water immersion	3.67	2.90	0.25	0.20	0.92	0.59	0.09	0.03	0.32	0.24
Laboratory storage 3 years	12.82	14.40	0.13	0.11	1.31	1.06	0.18	0.13	0.22	0.18
Laboratory storage 4.5 years	35.08	32.51	0.03	0.03	8.98	6.66	0.17	0.15	0.56	0.35

of paint systems, aged for short periods in a variety of environments. It is by no means comprehensive, but serves to indicate the nature and complexity of the problem. A subsidiary objective was an attempt to reproduce any natural ageing effects by accelerated exposure methods.

Twelve paints covering the complete spectrum of ease of decontamination have been investigated.

Experimental

Paint Systems and Application

Twelve paints of known composition were applied as a double coat system to 150 × 100 mm mild steel panels and allowed to cure for 16 hours between coats and for 14 days before testing. The sole exception was paint *F* (stoving acrylic) which was stoved at 180°C for 15 minutes between coats and 45 minutes at 180°C for the two coat system. The formulations of the paints are given in Appendix 1.

Paint <i>A</i>	Aromatic cured polyurethane
Paint <i>B</i>	Aliphatic cured polyurethane
Paint <i>C</i>	Amine cured epoxide
Paint <i>D</i>	Polyamide cured epoxide
Paint <i>E</i>	Amine adduct cured epoxide
Paint <i>F</i>	Stoving acrylic
Paint <i>G</i>	Long oil alkyd
Paint <i>H</i>	Tung oil/phenolic oleoresinous
Paint <i>I</i>	Chlorinated rubber
Paint <i>J</i>	Chlorinated rubber
Paint <i>K</i>	Chlorinated rubber
Paint <i>L</i>	Chlorinated rubber

Exposure

The coated panels were exposed to:

- (i) Natural weathering at 45° due South for 2 years (Semi-rural)
- (ii) Accelerated weathering to BS3900 Method F3 (9) for 2000 Hours

- (iii) Immersion in demineralised water for 6 months
- (iv) Laboratory storage for 3 and 4.5 years in vertical racks.

Testing

All decontamination testing was carried out in accordance with BS4247 Part 1: 1967. "The Assessment of Surface Materials for use in Radioactive Areas."

The panels exposed to accelerated and natural weathering were tested under two conditions: on removal from test, half the panel was washed in 5 per cent detergent solution using a soft brush and rinsed in running water to remove surface "chalking"; the other half was untouched. The two surfaces have been designated chalky and non-chalky respectively.

All other panels were tested in the condition in which they were removed from test, except for the water immersion panels which were allowed to dry out for several days before testing.

The results are quoted as percentage residual contamination after each of the two decontamination procedures detailed in BS4247.

Reference to Table 1 will show that the paints tested covered a wide range of decontaminability, ranging from the aromatic isocyanate adduct cured polyurethane (Paint *A*) which retained almost 25 per cent of the original contamination, to four chlorinated rubber paints which retained between 0.04 and 1.0 per cent. The order of merit on the un-aged controls was:

Best	2	3	4	5	6	7	8	9	10	11	Worst
<i>F</i>	<i>K</i>	<i>I</i>	<i>J</i>	<i>L</i>	<i>G</i>	<i>H</i>	<i>B</i>	<i>D</i>	<i>E</i>	<i>C</i>	<i>A</i>

There were large and obvious differences in the amount of residual contamination after exposure to the various ageing environments.

Paint F		Paint G		Paint H		Paint I		Paint J		Paint K		Paint L	
1	2	1	2	1	2	1	2	1	2	1	2	1	2
0.03	0.03	0.80	0.80	0.90	0.84	0.18	0.09	0.95	0.37	0.05	0.04	0.92	0.43
0.91	4.26	2.52	1.51	2.79	1.60	5.57	3.83	17.35	11.29	4.23	2.81	5.21	3.49
0.93	1.92	2.38	1.20	2.94	1.47	3.81	2.51	9.01	6.61	4.69	2.85	3.65	2.76
0.07	9.31	23.04	18.23	12.42	8.06	15.77	13.42	18.72	12.51	15.47	14.77	16.09	11.90
0.87	7.66	17.28	14.77	17.43	13.93	5.67	4.77	9.73	7.26	9.75	8.51	10.11	8.83
0.13	0.07	0.46	0.30	0.96	0.52	1.30	0.85	5.00	3.05	1.77	1.05	2.31	1.16
0.93	0.86	9.87	9.47	2.21	1.92	6.61	6.37	4.78	4.24	0.08	0.07	0.07	0.07
0.49	1.28	5.84	5.84	1.04	0.85	0.37	0.23	7.54	7.54	0.05	0.03	2.73	2.52

An analysis of variance of the first and second decontamination stages is detailed in Appendix 2. Here the Tukey critical differences for the paints showed the order of merit to be:

	Best	2	3	4	5	Worst
First Stage	<i>B</i>	<i>K, D, F</i>	<i>I, E, H, L</i>	<i>G, C</i>	<i>L, J</i>	<i>A</i>
Both Stages	<i>B</i>	<i>D, K, F</i>	<i>I, L, H, E</i>	<i>G, C</i>	<i>J</i>	<i>A</i>

The groupings indicate that statistically the paints within a group are not distinguishable, due to the scatter of results; but the order within a group is simply that of the averages. Although there are minor differences between the "first stage" and "both stages" analysis, the basic order of merit remains the same.

For the ageing environments, the Tukey critical differences showed the order of merit to be:

Best	Water immersion
	Laboratory ageing 3 and 4.5 years, and unaged control
	Accelerated weathering - non-chalky
	Accelerated weathering - chalky
	Natural weathering - non-chalky
Worst	Natural weathering - chalky

There were significant interactions.

Discussion

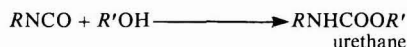
It is clear from Table 1, and the analysis of variance, that there are highly significant differences in the decontaminability of the different paints after the exposure to the various "ageing" environments. Furthermore, there are significant interactions, i.e. the paints do not all behave in the same way in any given environment.

Before discussing individual paints in specific environments, it would be as well to consider the likely curing and ageing processes in the paints, arising from their chemical

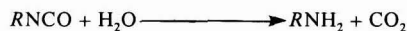
composition. For convenience, these will be discussed in the groups; polyurethanes (Paints *A* and *B*), epoxides (Paints *C*, *D* and *E*), acrylic (Paint *F*), oil containing (Paints *G* and *H*) and chlorinated rubbers (Paints *I*, *J*, *K* and *L*).

Polyurethanes

Paints *A* and *B* are two-pack cold curing polyurethane paints consisting of an isocyanate adduct-cured polyester. Paint *A* is based on an aromatic isocyanate based on toluene diisocyanate and Paint *B* on an aliphatic isocyanate adduct based on hexamethylene diisocyanate. Both types cure by reaction of the isocyanate group with hydroxyl groups:



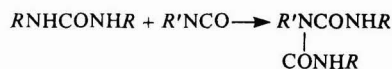
However, this is not the only reaction, and that of the isocyanate with water is important:



The amine formed will react with further isocyanate,



to form a substituted urea. A further reaction between the substituted urea and isocyanate is possible to produce a biuret:



Water extraction of polyurethane paint films has demonstrated the presence of secondary and tertiary amines with a measured pH of >10. On ageing in a UV rich environment (accelerated or natural exterior weathering), photodegradation of the cured film will occur with production of amine, hydroxyl, isocyanate groups, water



x240



x1180

Figure 1. Long oil alkyd paint
(Paint C): 2 years natural
weathering - 25° view



x5900

and carbon dioxide. The photodegradation of the binder will result in the phenomenon known as chalking, in which loose pigment particles appear at the paint surface, exposing the pigment/binder matrix. A major technological difference between aromatic and aliphatic cured polyurethanes is in their resistance to ultraviolet degradation, as indicated by their chalking resistance. Paints based on aromatic isocyanates chalk rapidly and completely whilst aliphatic isocyanate cured paints chalk very slowly, and retain film integrity for many years exterior exposure. The extremely good chalking resistance is attributed to the fact that the binder does not absorb in the 300–400 μm range; ultraviolet radiation is particularly damaging to organics.

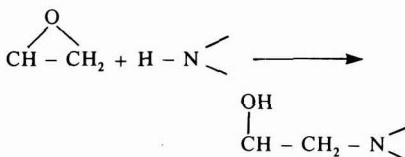
Thus at any stage in its life, a polyurethane paint film may contain unreacted hydroxyl and isocyanate groups, and substituted tertiary and secondary amines; further, the exposed surface may show chalking. This chalking will be more advanced on an aromatic isocyanate cured film than an aliphatic cured film at any given time. Typical chalked surfaces are shown in Figures 1 and 2.

Amine cured epoxides

Refs. 10, 12

Three basic types of amine cured epoxide have been studied.

The first (Paint C) depends on the reaction between epoxide groups and the active hydrogen atoms of primary and secondary amines.



Paint C is based on the aliphatic amine, triethylene

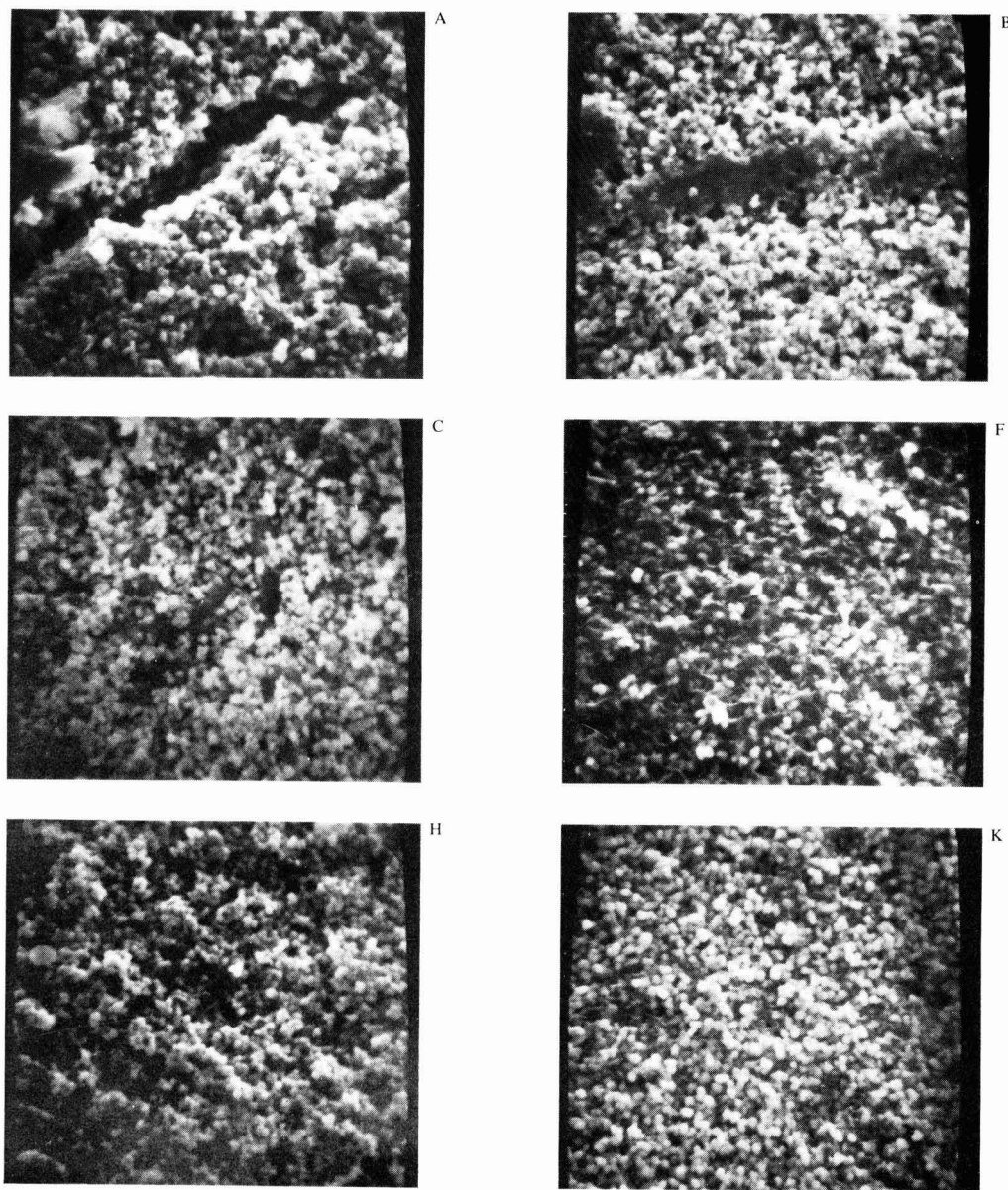


Figure 2. Paint films after 2 years natural weathering – 25° view. Aromatic polyurethane paint (A), aliphatic polyurethane paint (B), amine cured epoxide paint (C), stoving acrylic paint (F), tung oil/phenolic paint (H) and chlorinated rubber paint (K)

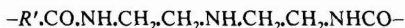
x3740

tetramine. It is normal to use a slightly higher than stoichiometric ratio of amine to epoxide to allow for evaporation from the film. A common defect of this type of paint is known as "amine blush", in which unreacted amine migrates to the surface¹⁰. Amine cured epoxide systems chalk rapidly. An amine cured epoxide film can, therefore, be expected to have free amine groups at the surface, both during the initial cure, and in the early stages

of ageing, and will show pronounced chalking on natural exposure.

Paint E is an amine adduct-cured epoxide and is chemically identical to the amine cured system, though showing a much reduced tendency to "amine blush". It will, therefore, age in the same manner as the amine cured system.

Paint *D* is a polyamide cured epoxide in which the curing agent is produced by reacting an aliphatic polyamine, e.g. diethylene triamine, with "dimer acids" made by limited polymerisation of unsaturated fatty acids, to produce an amide. The polyamide obtained from diethylenetriamine and the dimer acid HOOC-R'-COOH would thus have the following repeat unit in the chain:



where the central NH group would contain the active hydrogen.

Thus, a polyamide cured epoxide may have unreacted amine groups, and will be subject to oxidative attack at residual double bonds. Acid groups are likely to arise on ageing. The film will certainly chalk on exposure to ultraviolet radiation.

Stoving Acrylic

Paint *F* is based on a commercial thermo-setting acrylic resin, the composition of which is unknown. However, it is known that resins of this type consist of the reaction products of "hard" monomers, such as methyl methacrylate and styrene, "soft" monomers, such as ethyl and butyl acrylates, and a "reactive" monomer, such as acrylic acid. As Paint *F* was cured at 180°C before test it is extremely unlikely that it contained any free reactive groups on initial cure. On ageing, free acid groups are likely to be present with a variety of other reactive species. Although fairly chalk resistant, some chalking is likely on exposure to ultra-violet radiation.

Oil Containing Paints

Although grouped under this heading, Paint *G* is based on a long oil alkyd resin and therefore more correctly contains fatty acid esters rather than vegetable oil. Paint *H* contains tung and linseed oil. Both Types contain multiple double bonds in the carbon chain of the fatty acid.

In the cure of such coatings, reaction with atmospheric oxygen at the double bonds forms hydroperoxy groups by removal of a hydrogen atom from the CH_2 group between double bonds. Rearrangement and reaction with further oxygen gives a peroxy radical which may react with more hydrocarbon to form hydroperoxides. Hydroperoxide groups are unstable, and will slowly decompose to give alkoxy and hydroxyl radicals. A consequence of this free radical formation is the production of low molecular weight aldehydes, ketones and acids. A major portion of these volatile and water soluble by-products of the curing mechanism is formic acid, although other acids such as acetic and butyric are also formed. It has been shown that the formation and evolution of formic acid from alkyd resins and oils is a continuous process, and is still taking place after 180 days¹¹.

Alkyds and oil containing paints absorb strongly in the 200-300 millimicron range when exposed to light. The bond energy of the oxygen-oxygen bond in the peroxide linkage is about 167 KJ/mole (compared with 334 KJ/mole for the carbon-carbon bond) and is less than the energy of visible light¹². Thus the peroxide bond breaks on exposure to ultra-violet radiation to form alkoxy radicals. These are extremely reactive and undergo a variety of reactions to form aldehydes, ketones and short chain acids. Alkyd and oil based paint films chalk fairly rapidly. Thus at any time in their life these paints contain a

variety of water soluble reactive species, and may show breakdown of the upper layers of the film due to chalking.

Chlorinated Rubber

Chlorinated rubber paints consist essentially of chlorinated rubber together with a chlorinated plasticiser. Paints *I* and *J* contain a chlorinated-diphenyl, Paints *K* and *L* a chlorinated paraffin. Such coatings are basically inert, and dry by solvent evaporation. Thus there are no chemical reactions involved in their cure, and therefore no by-products. The films remain inert throughout their life as the hydrogen-chlorine bond is stable at room temperature. Elevated temperature e.g. 70-80°C will liberate hydrochloric acid.

Some loss of plasticiser is likely on ageing due to the small but positive vapour pressure and slight water solubility of the plasticiser. On exposure to ultra-violet radiation chlorinated rubber paints chalk freely. Paints *J* and *L* will chalk rather more freely than *I* and *K* as they contain anatase titanium dioxide rather than the rutile form which is much more chalk resistant.

The approximate order of chalking resistance of the paints tested is:

Paint <i>B</i>	Aliphatic polyurethane
Paint <i>F</i>	Stoving acrylic
Paint <i>I</i>	Chlorinated rubber
Paint <i>K</i>	Chlorinated rubber
Paint <i>G</i>	Long oil alkyd
Paint <i>D</i>	Polyamide cured epoxide
Paint <i>H</i>	Tung oil phenolic
Paint <i>E</i>	Adduct cured epoxide
Paint <i>C</i>	Amine cured epoxide
Paint <i>L</i>	Chlorinated rubber
Paint <i>J</i>	Chlorinated rubber
Paint <i>A</i>	Aromatic polyurethane

If the ageing environments are considered in detail it is found that exterior exposure and accelerated weathering both involve exposure to ultraviolet radiation and will induce chalking in the films; water immersion, which is likely to leach out water soluble components, and laboratory ageing which involves only little ultraviolet radiation, but might cause surface contamination by a variety of unknown chemicals; there is then a wide variety of ageing factors involved.

The main processes by which surface coatings may become contaminated have been described by Walker and Haighton¹², and may be summarised as physical adsorption, ion exchange, chelation and permeation; undoubtedly the first three are operative in the control and probably in the laboratory aged samples. It is probable that all the coatings tested, with the possible exception of the four chlorinated rubber paints, possess surface acidic groups, partly by virtue of their composition, and partly due to oxidative degradation and normal ageing processes¹³. At pH values above 2-3 these surface acid groups ionise and function as weak, low capacity ion exchangers, and although weak in comparison with conventional ion exchange resins, they are strong enough to retain considerable amounts of activity¹². Paints *G* and *H* certainly possess surface acid groups which are being constantly regenerated throughout the life of the paint film.

Paint *A* - Aromatic polyurethane

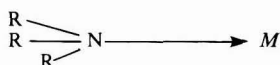
This was the worst paint of the twelve on test. On natural

weathering the residual contamination fell slightly, with little difference between the chalky and non-chalky areas. The films exposed to accelerated weathering showed a marked decrease in retention. The laboratory aged films showed a decrease in retention after 3 years exposure, and a marked increase over the control after 4.5 years.

A hypothesis for the general behaviour of this paint is that nitrogen in the three-valent condition, as in ammonia, organic amines, amides etc., has a lone pair of p electrons which are available for donation, i.e. it acts as a base in the Brönsted (electron donation) sense.



Donation to a suitable acceptor e.g. a metal atom creates a co-ordinate bond between the nitrogen atom and the metal atom (chelation):



This has the effect of attaching the metal atom to the organic structure in which the nitrogen is contained, in this case the isocyanate, or free amine contained in the film, and arising from the reaction of the isocyanate with water. This would explain the high initial retention. Natural weathering would have two main effects; one to leach out water soluble amines or substituted ureas which would have the effect of lowering the residual contamination; the other to induce heavy chalking, thus exposing fresh polymer matrix to the contaminating agent.

The films exposed to accelerated weathering, however, showed markedly less residual contamination than the controls and it is well known that amines oxidise on exposure to air through the formation of amine oxides in which the lone electron pair is donated to oxygen to complete its p electron octet:



The net result of this would be to neutralise some of the lone pairs of the nitrogen atoms which would otherwise be available for chelation of metal atoms. The conditions in the accelerated weathering apparatus (high temperature, ultraviolet radiation and water soak) would encourage both oxidation of the amines and water extraction. Further, the period of exposure was insufficient to induce the degree of chalking found on the natural weathering films.

Obviously, the effect of water immersion would be to extract all water-soluble low molecular weight material; residual contamination would, therefore, be low. The oxidation of amines present may well explain the lower residual contamination of the films exposed in the laboratory for 3.5 years, but the increased retention after 4.5 years is not explicable in these terms.

Paint B – Aliphatic polyurethane

Although also a polyurethane, Paint *B* showed a major difference in the levels of residual contamination, and the least overall change in contamination after all the ageing regimes. After natural weathering, the residual contamination increased. After accelerated weathering there was a

slight increase on the chalky surface and a slight decrease on the non-chalky film. The effect of water immersion and both periods of laboratory ageing was to reduce the residual contamination considerably.

It is interesting to speculate on the difference in residual contamination between Paint *A* and Paint *B*, and it may well be that although aliphatic isocyanates, being stronger bases than aromatic isocyanates, are more likely to chelate metal atoms, they are far more likely to oxidise on ageing, and therefore be unavailable for chelation. The effect of natural weathering is to liberate fresh low molecular weight nitrogen compounds, resulting in an increase in residual contamination, and to induce some slight chalking of the film exposing matrix polymer. Accelerated weathering would have the same effect, but would produce less chalking than the natural weathering. The lower residual contamination of the water immersed films can be explained by the leaching out of active materials which, being aliphatic in nature, are likely to be more water soluble than their aromatic counterparts. The laboratory aged films are likely to show progressive oxidation and, therefore, less residual contamination.

Paint C – Amine cured epoxide

Paint *C* was second only to the aromatic polyurethane paint in the level of initial residual contamination, and showed a slight but positive increase in contamination after Stage 1 decontamination. After natural weathering, and Stage 2, the residual contamination was less. Accelerated weathering resulted in less residual contamination, with a marked difference between the chalky and non-chalky areas of the film. The values after water immersion were low. Interestingly, this paint showed the same pattern as Paint *A* on laboratory ageing; a decrease in residual contamination after 3 years followed by an increase after 4.5 years.

Paint *C* is another film which chalks rapidly and is only slightly superior in this respect to Paint *A*. The same hypothesis for the mechanism of contamination advanced for Paint *A* can also be advanced for this paint. As previously stated, amine cured epoxide paints almost invariably suffer from amine blush, the aliphatic amine being present in quantity as a surface layer. It would tend to be oxidised and leached out in the accelerated weathering test, resulting in a lower retention value, but natural weathering would result in heavy chalking with exposure of matrix material; thus residual contamination might be expected to increase.

Oxidation of the amine surface would explain the initial fall in contamination after 3 years, but not the increase after 4.5 years. It is possible that this increase merely represents a build-up of free amine on the surface which is not entirely oxidised. However, even with this increase, the retention value still remains below that of the control.

Paint D – Polyamide cured epoxide

Paint *D* had the lowest retention value of the three epoxide paints tested, perhaps predictably, since it contained the least free amine. Natural weathering resulted in a slightly lower retention value on the non-chalky film and a higher retention value on the chalky. Accelerated weathering resulted in a lower residual contamination than the control. Water immersion, laboratory ageing for 3 and for 4.5 years resulted in greatly reduced residual contamination levels. The contamination retention of the film is probably due to two mechanisms; chelation due to

nitrogen compounds present in the film and to an ion exchange mechanism involving the surface acid groups arising from the oil residues. Accelerated weathering, although encouraging breakdown of the film and the production of acid groups and nitrogenous materials, also tends to encourage leaching of these components from the film. The act of removing the surface chalk from the film is also likely to remove the active entities from the surface; thus the non-chalky surface shows a lower residual contamination than the chalky. Water immersion obviously results in almost complete removal of the low molecular weight components of the film. The low retention values on laboratory ageing suggest oxidation of free amine.

Paint E – Adduct cured epoxide

Although chemically almost identical to Paint C, the initial level of residual contamination was much lower, due most probably to the absence of "amine blush". In other respects the pattern of behaviour was identical to that of Paint C, except that the laboratory aged films showed much lower retention values than the control. The mechanism governing the contamination must be that advanced for Paint C, modified by the absence of relatively large quantities of surface amine.

Paint F – Stoving acrylic

Paint F, probably due to the high temperature and consequent highly cross-linked nature of the film, showed very low initial retention values, and in this respect was the best of the films under test. Natural exposure considerably increased the retention values, as did the accelerated weathering exposure. Both induce chalking and breakdown of the polymer matrix, resulting in the formation of surface acid groups and thus encouraging contamination by the ion exchange mechanism. The retention values after water immersion were low. The laboratory aged films showed a definite increase in retention values which appeared to be progressive with increasing ageing of the film.

Paints G and H – Long oil alkyd and tung oil/phenolic

Paints G and H showed a similar pattern of behaviour; a marked increase in retention on natural weathering, a lesser increase after accelerated weathering, a lower value on water immersion, an increase after 3 years laboratory ageing followed by a decrease after 4.5 years.

This similarity in pattern is hardly surprising, as both films will produce surface acid groups both on curing and on subsequent ageing. The contamination mechanism is thus likely to be one of ion exchange.

Both films will chalk freely on natural exposure, and to a lesser extent on accelerated weathering.

The initial rise in retention values after 3 years, followed by a fall after 4.5 years is possibly a reflection of the pattern of free radical and acidic products formation in the film, which might be expected to fall over a period of years.

Paints I and J – Chlorinated rubber

Ref. 6

Paints I and J differed only in the type of titanium dioxide used. Paint I contained the relatively chalk resistant rutile

grade, Paint J the freely chalking anatase. On natural weathering both paints showed a marked increase in retention values, greater in the case of Paint J. Accelerated weathering also produced a marked increase in retention, again more pronounced in Paint J. An interesting departure from the behaviour of the paints discussed so far is that water immersion caused a fairly marked increase in residual contamination. The laboratory aged films also showed an increase in residual contamination, although Paint I showed an increase after 3 years, followed by a decrease after 4.5 years. The residual contamination of Paint J increased with increasing age.

The difference in residual contamination between Paints I and J on both natural and accelerated weathering are most probably due to the differences in the chalking rates of the two films. The increase in residual contamination after water immersion may be attributable to loss of plasticiser from the films, as it is known that the ease of contamination of chlorinated rubber based paints increases with decreasing plasticiser content⁶. The same explanation can be advanced for the behaviour of Paint J on laboratory ageing, but would not explain the behaviour of Paint I.

Paints K and L – Chlorinated rubber

Refs. 8, 14–17

Paints K and L differ in the type of titanium dioxide used and differ from Paints I and J in the type of plasticiser (chlorinated paraffin as opposed to chlorinated diphenyl). Both paints showed a marked increase in residual contamination after natural and accelerated ageing, the increase due to natural weathering being very much greater than that due to accelerated weathering. The water immersed films again showed a greater residual contamination. The laboratory aged films of Paint K showed almost no change in residual contamination. Paint L showed an apparent drop from the control level after 3 years, followed by an increase after 4.5 years. In general, paint K was very much better than any of the other chlorinated rubber paints.

The overall picture then, is of the paints showing the greatest initial residual contamination improving on laboratory ageing, and those showing the lowest residual contamination deteriorating, although there are exceptions to this.

Certainly, exposure to natural weathering caused a marked deterioration in the ease of decontamination as indicated by the residual decontamination after Stages 1 and 2 of BS 4247. That this should be so is hardly surprising when the surfaces presented to the contaminant are as porous and friable as those indicated by Figures 1 and 2. A disappointing feature of the present experiment is the lack of correlation between either the accelerated weathering and the natural weathering results on the one hand, and the accelerated weathering and laboratory ageing on the other. This lack of correlation may be due to the different ageing mechanisms attributable to ultraviolet radiation, in which case it is unlikely that correlation could ever be achieved, or to the fact that insufficient work has been carried out to equate the respective exposure periods. The difficulties in obtaining good correlation between accelerated and natural exposure have been stressed by other authors^{14–17}.

There appears to be a reasonably good correlation

Henkel

Dehydtag

Our multi-purpose
paint additive
for
non-aqueous coatings:



Dehydtag

Our TEXAPHOR 963 is already familiar to you as an outstanding anti-settling agent, as anti-flooding and floating agent and also as a wetting and dispersing agent for non-aqueous coatings.

Did you know that TEXAPHOR 963 can also be used as a pre-gelling agent for Bentonite* gellants?

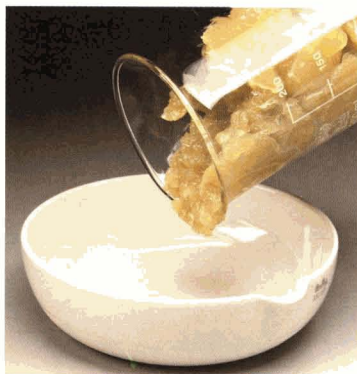
A further interesting field of application for this additive: by contrast with other gels not

containing our product, Bentonite gels with TEXAPHOR 963 have the following advantages:

- softer consistency and thus easier to work into the paint.
- the storage stability of the gel is considerably increased, eliminating crust formation.
- post-addition into the dispersed phase is possible.
- Gels can be produced with TEXAPHOR 963 which are capable of being pumped.
- A synergistic effect occurs between the Bentonites and TEXAPHOR 963.



Bentonite* gel with TEXAPHOR 963



Bentonite* gel without TEXAPHOR 963

Examples using Bentonite and TEXAPHOR 963

- A) Bentonite gel with all-round properties
 10 parts organophilic Bentonite*
 3 parts TEXAPHOR 963
 87 parts white spirit
- B) Bentonite gel capable of being pumped
 8 parts organophilic Bentonite*
 8 parts TEXAPHOR 963
 84 parts white spirit
- C) Bentonite gel with maximum thixotropy
 10 parts organophilic Bentonite*
 10 parts TEXAPHOR 963
 80 parts white spirit

* = e. g. Bentone 34 or 38. Bentone is a registered trademark of NL Industries Inc, New York, USA.

Manufacture of Bentonite gels.

The pre-gelling of Bentonite is dependent essentially on the intensity of the stirring and on the length of time which lies between 15 and 20 minutes. Gels containing aromatic solvents should be stirred for 5 to 10 minutes.

Bentonite is stirred into the solvent. Using aromatic solvents such as Xylene or Toluene, gelling will have already started at this stage. With the use of white spirit this is not the case. TEXAPHOR 963 is then added and the intensive stirring continues.

COUPON '80

Please forward, free-of-charge and without obligation:
 sample(s)
 technical leaflet(s)
 on TEXAPHOR 963

In Great Britain
 TEXAPHOR®963 = PRODUCT 963

From:

.....
 Company

.....
 for the attention of

.....
 Street

.....
 City

.....
 Country

Reply card

Henkel KGaA
 Organische Produkte
 Dehydtag KLF-T
 Werbung
 Postfach 1100

D-4000 Düsseldorf 1
 Germany

In Great Britain
 TEXAPHOR® 963 = PRODUCT 963

Henkel KGaA
 Organic Products Division
 Dehydtag KLF-T
 D-4000 Düsseldorf 1 · P.O.B. 1100
 Tel. (02 11) 79 71 · Telex 08 5817-0 hd d

between the chalking resistance of the films and the overall ease of decontamination:

Order of merit

Chalking	Decontamination
Paint B	Paint B
Paint F	Paint K
Paint I	Paint D
Paint K	Paint F
Paint G	Paint I
Paint D	Paint E
Paint E	Paint H
Paint H	Paint L
Paint C	Paint G
Paint L	Paint C
Paint J	Paint J
Paint A	Paint A

The order of merit for chalking is only approximately correct, as the list was compiled from experience, rather than from direct assessment and measurement at the time. Certainly, the correlation is very good at the best and worst ends of the merit order. It would probably be safer to suggest that rather than the degree of chalking being the cause of increased residual contamination, it is indicative of the relative stability of the film. However, the chalking resistance appears to give a rule-of-thumb assessment.

In making this comparison between the ease of decontamination and chalking resistance it should be noted that the order of merit of decontaminability has been assessed on the results of all the tests, not just those on which chalking was present at the time of the test. With the sole exception of Paint C, the surfaces from which the chalking had been removed were more readily decontaminated than the surfaces on which chalking was present.

The general observations that the two polyurethane paints were at the opposite ends of the decontamination spectrum and that the epoxides showed considerably differences in decontaminability reinforces the views expressed elsewhere⁸ that to refer to coatings by their generic type in the context of decontaminability can be, at best, misleading.

Conclusions

- The ease of decontamination of surface coatings changes considerably with age.
- Exterior exposure results in a major decrease in the ease of decontamination of surface coatings.
- The ease of decontamination of surface coatings after water immersion is considerably improved; chlorinated rubber based coatings are likely to prove an exception.
- Aliphatic polyurethane paints are likely to show the least changes in ease of decontamination.
- At the present time it is not possible to assess the long term ease of decontamination of surface coatings by the use of accelerated tests.

Acknowledgments

The Authors would like to thank B. Ariss for carrying out

the decontamination testing.

[Received 24 January 1980

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Appendix 1

Composition of paints	Parts by weight
<i>Paint A. Aromatic polyurethane</i>	
<i>Base:</i>	
Rutiox HD ⁽¹⁾	200
Daltolac 10 ⁽²⁾	200
10% solution of $\frac{1}{2}$ second butyrate ⁽³⁾	40
Methyl ethyl ketone	200
Cyclohexanone	50
<i>Hardener:</i>	
Suprasec G ⁽²⁾	226
Mixing ratio base/hardener 3/1 by weight.	
<i>Paint B. Aliphatic polyurethane</i>	
<i>Base:</i>	
Rutiox HD	300
Desmophen 650 ⁽⁴⁾	150
10% solution of $\frac{1}{2}$ second cellulose butyrate	50
Methyl isobutyl ketone	114
Butyl acetate	29
Ethylglycol acetate	28
Toluene	28
<i>Hardener:</i>	
Desmodur N 75% ⁽⁴⁾	183
Mixing ratio base/hardener 70/18.3 by weight.	
<i>Paint C. Amine cured epoxide</i>	
<i>Base:</i>	
Rutiox HD	150
Epikote 1001 ⁽⁵⁾	150
Methyl ethyl ketone	30
Methyl isobutyl ketone	30
Cellosolve	15
Toluene	75
<i>Hardener:</i>	
HY 951 ⁽⁶⁾	30

n-butyl alcohol	35	5% calcium naphthenate ⁽⁹⁾	10.0
Toluene	35	White spirit	150
Mixing ratio base/hardener 15/1 by weight.			
<i>Paint D. Polyamide cured epoxide</i>			
<i>Base:</i>	Rutiox HD	100	
	Epikote 1001	100	
	Methyl isobutyl ketone	50	
	Xylene	50	
<i>Hardener:</i>	Versamid 115 ⁽⁷⁾	50	
	Methyl isobutyl ketone	15	
	Cellosolve	20	
	Toluene	15	
Mixing ratio base/hardener 100/44 by weight.			
<i>Paint E. Adduct cured epoxide</i>			
<i>Base:</i>	Rutiox HD	150	
	Epikote 1001	150	
	Methyl ethyl ketone	30	
	Methyl isobutyl ketone	30	
	Cellosolve	15	
	Toluene	75	
<i>Hardener:</i>	EDA Adduct 870 ⁽⁸⁾	100	
	Cellosolve	80	
	n-butyl alcohol	20	
	Toluene	50	
Mixing ratio base/hardener 2/1 by weight.			
<i>Paint F. Thermosetting acrylic</i>			
	Rutiox HD	80	
	Scopacron 100 ⁽⁷⁾	200	
	Cellosolve	30	
	Xylene	12.5	
	n-butyl alcohol	2.5	
<i>Paint G. Long oil alkyd</i>			
	Rutiox HD	200	
	Paralac 10W ⁽²⁾	226	
	6% cobalt naphthenate ⁽⁹⁾	0.8	
	24% lead naphthenate ⁽⁹⁾	2.0	
<i>Paint H. Tung oil/phenolic</i>			
Manufactured by Allweather Evode			
<i>Paint I. Chlorinated rubber</i>			
	Rutiox HD	100	
	Alloprene R20 ⁽²⁾	120	
	Aroclor 1254 ⁽¹⁰⁾	80	
	Xylene	300	
<i>Paint J. Chlorinated rubber</i>			
	Rutiox AE ⁽¹⁾	100	
	Alloprene R20	120	
	Aroclor 1254	80	
	Xylene	300	
<i>Paint K. Chlorinated rubber</i>			
	Rutiox HD	100	
	Alloprene R20	120	
	Cereclor 70 ⁽²⁾	80	
	Xylene	30	
<i>Paint L. Chlorinated rubber</i>			
	Rutiox AE	100	
	Alloprene R20	120	
	Cereclor 700	80	
	Xylene	300	

- (1). British Titanium Products Ltd
- (2). Imperial Chemical Industries Ltd
- (3). J. M. Steel Ltd
- (4). Bayer (UK) Ltd
- (5). Shell Chemicals Ltd
- (6). Ciba Geigy Ltd
- (7). Cray Valley Products
- (8). Anchor Chemicals Ltd
- (9). Nuodex Ltd
- (10). Monsanto Chemicals

Table 2
Mean values of transformed percentage residuals $\log (P/100-P) + 4$, both stages

Treatment	Paint	A	B	C	D	E	F	G	H	I	J	K	L	Treatment mean
Control		3.48	1.98	3.15	2.27	3.06	0.44	1.90	1.91	0.94	1.53	0.65	1.62	1.91
Accelerated weathering (chalky)		2.18	2.18	2.65	2.21	2.73	2.64	2.17	2.21	2.60	3.09	2.44	2.56	2.47
Accelerated weathering (non-chalky)		2.87	1.70	2.39	1.96	2.63	2.23	2.07	2.16	2.40	2.85	2.47	2.45	2.35
Natural weathering (chalky)		2.23	2.57	3.02	2.98	2.90	3.01	3.26	2.94	3.18	3.13	3.24	3.13	3.05
Natural weathering (non-chalky)		3.22	2.55	3.14	2.63	2.71	2.90	3.20	3.20	2.70	2.85	2.96	2.94	2.92
Water immersion		2.47	1.06	1.73	0.47	1.26	0.79	0.48	1.62	1.91	2.47	1.96	2.03	1.60
Laboratory ageing 3 y		3.06	1.05	2.03	1.11	1.26	1.94	3.02	2.29	2.83	2.65	0.84	0.85	1.91
Laboratory ageing 4½ y		3.68	0.45	2.85	1.18	1.55	2.11	2.79	1.93	1.35	2.91	0.73	2.41	2.00
Paint mean		3.02	1.69	2.62	1.85	2.26	2.01	2.49	2.28	2.24	2.69	1.91	2.25	

Appendix 2

Analysis of data

The experimental matrix of results consisted of percentage residual activity, P , values, 5 replicates to each paint/treatment/stage cell. Mean values of P for each cell are given in Table 1. Except for 3 year laboratory ageing on Paint A the Stage 2 residuals are all less than or equal to those after Stage 1, as might be expected. The Stage 1 and Stage 2 matrices were therefore analysed separately.

Unfortunately, individual results for the laboratory ageing treatments (3 and 4.5 year) were lost; only their means were available. Errors on high values of P would be expected to be greater than those on very low values; all values were therefore transformed as $4 \times \log P/(100-P)$, the number 4 being included to avoid negatives. Means of the transforms (or, for laboratory ageing treatments, transforms of the mean percentage residuals) are given in Table 2 for the second Stage. In the analysis the within cells, mean square (error or residual) was derived from those cells in which 5 replicates were available (12 paints, 6 treatments, thus giving $12 \times 6 \times 4 = 288$ degrees of freedom).

Origin	Degrees of freedom	1st Stage Mean Square	2nd Stage Mean Square
Between paints	11	5.75	5.87
Between treatments	7	16.52	15.91
Interaction	77	1.49	1.54
Error (residual)	288	0.026	0.032

Taking the mean error as 0.029, the standard deviation of a single experimental P value is $2.3P(100-P) \sqrt{0.029/100}$, giving

the following errors on P values.

P % residual value	50	40	30	20	10	1	0.1
Standard deviation	9.8	9.4	8.2	6.3	3.5	0.39	0.039

The actual variation of standard error with P is unknown; this table only shows the ascribable errors on the basis of the chosen transform. Nevertheless, the standard error as a percentage of the P value for a single result is about 30 per cent, a high but common value in such experiments, presumably arising largely from the variability of the surfaces tested.

Interaction effects are clearly very significant, that is, the paints do not behave consistently with the treatments. Nevertheless, the individual components of variance ascribable to paints and treatments are also very significant.

A global comparison of paints and treatments was made using a method due to J. W. Tukey (*Statistical Methods in Research and Production*, Davies and Goldsmith, Oliver and Boyd, 4th edition, 1972, page 152) giving the orders of merit quoted in the paper. (Note that the 2 weeks aged at room temperature, labelled control in the tables, are not controls in the statistical sense.)

Although this analysis provides some initial sorting of the results it is not entirely justified, since the experiments were not, and could not have been, performed at one time on a completely random basis and, as noted earlier, individual results for the last two treatments were not available.

Next month's issue

The Honorary Editor has accepted the following papers for publication. They are expected to appear in the July issue of the *Journal*:

Solubility parameter values of hydroxy polymers through their components and chemical group contribution technique by *H. Ahmad*

A comparative investigation of stains for wood by *S. Dombey and B. Slangroom*

An overhanging beam method for measuring internal stress in coatings by *S. G. Croll*

Hazards of static electricity in workclothing by *R. Verschave and R. Firmin*

occa meetings

London

Pollution analysis

An evening meeting of the London Section was held on 20 February 1980, at The Rubens Hotel SW1, where Mr N. A. R. Falla of the Paint Research Association presented a paper entitled "Solving Air Pollution Problems in the manufacture and use of surface Coatings".

Mr Falla reviewed the background to changes in legislation stimulating interest in airborne pollutants. He then went on to describe methods of analysing and identifying either particulate or gaseous pollutants. A description of each technique and the advantages and limitations were detailed.

Ways in which the pollutants could be reduced or eliminated in manufacturing and use of surface coatings were itemised and discussed. Mr Falla finally presented some thoughts on future trends in pollution control.

After an extensive question time a vote of thanks was proposed by Mr G. E. Westwood.

A fuller version of this paper is to be published later in the year.

Manchester

Powder, high-solids and water thinned coatings

Three lectures, summarised below, were presented at the Crest Motel, Bolton on 14 January 1980 by Ad Van Der Werff, Martin Bus and Luke Kwakman of Scado BV, Netherlands. The lectures were presented to 70 members and guests.

(1) Powder coatings by Ad Van Der Werff

After detailing the basic chemistry involved the various cross-linking mechanisms were compared with those of solvent based systems. Details of the advantages and disadvantages of the different cross-linking systems of each type of powder coating were given. Further comparisons of powder type were discussed in relation to performance characteristics both in the laboratory and industry followed by reference to the apparent passive nature of corrosion inhibiting pigments in powder systems.

Further aspects of powder coatings discussed were related to the following:

- Currently successful applications
- Development work at Scado
- Future growth.

(2) High-solid coatings by Luke Kwakman

Mr L. Kwakman started his lecture with a review of the various methods available for producing high-solids or low emission coatings which included radiation cured systems. The attendant properties, both positive and negative, of various systems were discussed along with special reference to cost effectiveness.

Mr Kwakman expanded his main theme, that of low solvent containing systems, by discussing work done in relation to the molecular weight distribution of suitable resins. High molecular weight resulted in paints containing high proportions of solvents whilst low molecular weight resins often gave unsatisfactory film properties.

The various legislative requirements of the major paint using areas were then reviewed by Luke Kwakman, in addition he discussed the complexities involved in supplying additional plant in relation to compliance with current and future legislation.

The virtues of high-solids coatings was balanced between higher initial cost but cost savings exist with respect to the provision of air extraction/fume treatment plant.

(3) Water thinned coatings by Martin Bus

The Lecturer commenced by listing the various types of resins available thus: alkyds, acrylics, oil-free polyesters, epoxy polyesters. He also summarised the differences between emulsion and soluble types suitable for industrial applications.

A trend towards a greater use of emulsion types was claimed, occasioned both by experience and legislation. Reference to the formulation of water-borne systems especially in relation to gloss improvement was made. Martin Bus acknowledged that present industrial paint systems generally performed well and that a change of direction in favour of water-borne systems could not be justified at present on cost or performance alone. He stated that legislation, the need to protect the environment, health and safety at work were all very important factors influencing the rate of change. In conclusion Mr Bus stated that when all the possible alternatives of meeting legislative requirements are considered the cost of the high technology involved in water-borne paints then became commercially viable.

A general conclusion to the three lectures was offered by Luke Kwakman who claimed that the common theme presented had been the three 'E's' - Economy, Energy conservation and Environmental protection in competition with increased costs for solvents and anti-pollution equipment. The excellent lecture evening was complimented by a vote of thanks proposed by George Hurst and acknowledged with enthusiasm.

F. B. W.

Midlands

Greetings - millions of them

The annual Newton Friend lecture of the Midland Section was held on Friday, 21 March 1980 at the Birmingham Chamber of Commerce and Industry, Harborne Road, Birmingham.

Members, guests and their ladies assembled to hear Mr A. Gentles, Managing Director of Webb Ivory Ltd, give a talk entitled "Greetings – Millions of them".

Mr Gentles opened his talk by saying that since Egyptian times greetings had been sent between people, mainly the very rich. In 15th century Britain the greeting in the form of a wood carving was sent between wealthy households. By the 19th century greetings were being sent on wood, cloth, metal and vellum, still mainly by the wealthy. The first recorded Christmas card to be sent was in 1843 by Sir Henry Cole. The card was like a postcard, 1000 were printed and they cost him one shilling each. It was not until 1850 that the exchange of Christmas cards became regular. Today more than 1,000 million of them are sent each year and the idea has spread to all Christian areas of the globe.

The speaker continued his talk by giving some details of how the cards are produced, saying that his company had two machines able to print in five or six colours and black, each capable of producing one million cards per eight hour day. The cards are produced on flat sheets with up to 32 cards per sheet. These are then guillotined to size, folded and packed into boxes or wallets.

Further information on any items mentioned below may be obtained by circling the appropriate Reader Enquiry Service number on the form at the back of the Journal. Enquiries will be forwarded to the organisation concerned.

Albright & Wilson's new phosphate complex

A new £20 million phosphates complex at the Marchon Works in Whitehaven was opened by the Queen on Friday 21 March 1980. With this development Albright & Wilson Limited is now the largest producer of phosphoric acid in the UK.

Reader Enquiry Service No. 31

Bennett Guest

Bennett Guest, the Engineering Group, has concluded an agreement today with the Cole Group to purchase the water chiller and temperature control equipment manufacturing division of Cole Equipment Limited. Both companies have assured that there will be continuity and development of employment and product line.

Reader Enquiry Service No. 32

New ethoxylates plant

Production of ethoxylates is to be significantly increased at ICI on Teesside following a decision to sanction a new 60,000 tonnes a year plant at Wilton. Costing well over £10 million it will be ready during 1982.

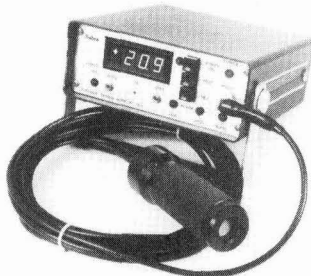
Reader Enquiry Service No. 33

Sabre distributors for Interscan

Sabre Gas Detection Limited of Aldershot have recently been appointed as the sole distributors in the United Kingdom for the Interscan Corporation, one of America's leading manufacturers of gas detection equipment.

The Interscan Toxic Gas Analysers which will now be available through Sabre in the UK, range from personal dosimeters to fixed detection systems. There are analysers available to monitor

carbon monoxide, sulphur dioxide, hydrogen sulphide, nitrogen dioxide, nitric oxide, and chlorine. The use of electrochemical transducers throughout the Interscan range enables the analysers to provide an accuracy of ± 2 per cent FSD.



Sabre's new portable oxygen alarm monitor

Sabre have also recently launched their own new Oxyguard 'OO' Series of Oxygen Alarm Monitors which set a new standard of protection against the hazards of oxygen deficiency and enrichment.

Reader Enquiry Service No. 34

Brenntag (UK) Ltd

Brenntag (UK) Ltd has announced that it is becoming the UK distributor for Olster Asphaltfabriek BV of Holland, who manufacture special tar and pitch products with low water and phenol content for use in epoxy and polyurethane coating systems.

Fluid handling purchase

As part of its plans to increase UK market penetration, and in preparation

occa meetings

Finally, Mr Gentles went on to talk about the charity trading and fund raising aspects of the organisation of which Webb Ivory Ltd is part, saying that no matter what the size of the group, be it a Cub/Scout pack or a major charity, substantial funds can be raised through the sale of greetings cards.

To illustrate his talk the speaker then showed numerous coloured slides.

The meeting was finally brought to a close with a vote of thanks proposed by Mrs C. Chator and warmly endorsed by the audience.

B. E. M.

occa news

for a period of expansion and technical growth, Graco of England Ltd, has agreed with the Steeley Co. Ltd to acquire the Fluid Handling Division of Steeley Industrial Distribution.

The purchase is effective on 1 April 1980. The business will be integrated with Graco of England Ltd.

Reader Enquiry Service No. 36

Coates in USA

Coates Brothers have set up their second North American-based company within a year. In addition to Coates Reprographics Inc. based in Dallas, Pennsylvania, Coates Brothers Inks (USA) Incorporated have now been established at Greenville, South Carolina, to distribute UK-manufactured goods.

Reader Enquiry Service No. 37

PRI announces medal award

The Plastics and Rubber Institute awarded the 1979 Colwyn Medal to Dr Maurice Morton. The medal is awarded for outstanding services to the rubber industry of a scientific, technical or engineering nature.

Dr Maurice Morton was the Director of the Institute of Polymer Science at the University of Akron, Ohio, from 1952 until his retirement in 1978. His scientific publications include more than eighty papers mainly in the areas of emulsion and anionic polymerization, and structure of elastomers.

Reader Enquiry Service No. 38

news

Formation of the Royal Society of Chemistry

On 1 June 1980 with the grant of its Royal Charter, the Royal Society of Chemistry (RSC) will come into being as a result of the unification of the Chemical Society (CS) with the Royal Institute of Chemistry (RIC). Both the CS and RIC at present hold Royal Charters, that of the CS dating from 1848 and that of the RIC from 1885. These will be surrendered and the CS and RIC will cease to function as separate organisations. The Queen has granted her patronage to the new Society.

The RSC will have as its main objectives the advancement of the science of chemistry and its applications, and the maintenance of high standards of competence and integrity among practising chemists (as distinct from pharmacists).

It will speak to Government and all sections of the community on behalf of some 40,000 scientists concerned with chemistry, as the Chemical Society and the Royal Institute of Chemistry have done in the past, but it will do so with a single authoritative voice. The two organisations have existed separately for more than a hundred years and both are thriving. They have always been closely associated and, since 1972, have advanced towards maximum collaboration and a common membership. The establishment of a unified Society is thus the final step in a gradual movement towards complete integration.

Reader Enquiry Service No. 39

new products

Atlacid carmine 4BA

Atlantic Chemical Corporation announces the manufacture of Atlacid carmine 4BA (acid violet 12), it is recommended for use on wool stock, yarn, and piece goods.

Atlacid carmine 4BA is also recommended for nylon, for pastel and medium depth shades where brightness of shade is essential.

Reader Enquiry Service No. 40

Gem plate restorer

The loss of a lithographic plate through poor or ineffective 'gumming-up' prior to storage can now often be avoided following the introduction by Coates of GEM Plate Restorer.

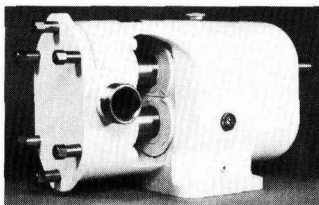
Oxygen attack gives rise to oxidised

areas on the non-image portion of the plate that will accept ink when the plate is removed from store and put onto the press. This danger of dirty printing can be overcome by treatment of the affected areas with GEM Plate Restorer which removes the oxidised layer, and "restores" the plate to its former condition.

Reader Enquiry Service No. 41

New lobe rotor pump

A new range of advanced profile lobe rotor pumps has been introduced by SSP Pumps Ltd of Eastbourne. Called AP pumps, they are self-priming and provide a very gentle, non-shear action, a fully swept pumping chamber and are valveless. They can handle liquid viscosities from one centistoke up to semi-plastic thixotropic liquids; also slurries and suspended solids. In addition they can be run dry and can handle entrained gases and froth without losing prime.



The AP 200 from SSP Pumps Ltd

Key factors that determine efficient pumping performance are the profile of the rotors, their inter-relationship and their relationship within the rotor casing.

Reader Enquiry Service No. 42

A new prepaint treatment

A new no-rinse, non-polluting multi-metal process for liquid prepaint treatment has been launched by Oakite Ltd.

"Okemcoat 2000" is designed for the prepaint coating of metal strips and parts by immersion, flow-on or spray. Two years of testing have shown that the process produces paint adhesion superior to that of iron phosphates and corrosion resistance equal to many zinc phosphates. Costs are also competitive with these traditional materials.

Reader Enquiry Service No. 43

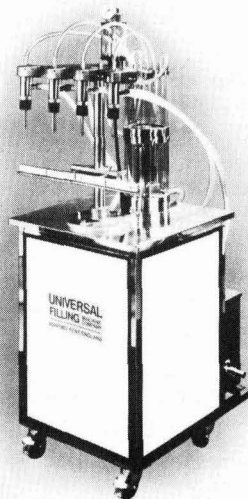
New tiioxide pigments

Tioxide have introduced two new additions to their range of titanium dioxide pigments:

Tioxide R-TC90, a durable chloride grade, setting new standards for gloss in a wide variety of aqueous and non-aqueous media, together with outstanding dispersibility, colour and opacity.

Tioxide R-CR60, the second of the new products is a superdurable grade for use in the effective pigmentation of industrial systems in which long service life is a prerequisite.

Reader Enquiry Service No. 44



Universal's new filling machine

New filling machines

Universal have developed a new range of semi-automatic vacuum operated bottle filling machines for liquids and semi-liquids. The machines can be used to fill glass, tin or semi-rigid plastic containers.

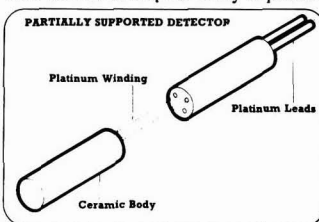
Although a new company, the Directors have been directly involved with the manufacture of these types of machines for many years and have used their experience in designing machines which give ease of use and in particular, a simple design of nozzle which is easily cleaned and can be retubed resulting in a saving in costs.

Reader Enquiry Service No. 45

New temperature sensing device

A new range of platinum-resistance temperature detectors with a resistance at 0°C of 130 ohms ± 0.2 ohm, which give a fundamental interval of 50.7 ohms, is announced by Sensing Devices Ltd of Southport, Merseyside. Known as the D130 series, the new detectors operate from -220°C to +800°C and come in a variety of sizes down to only 1.6 mm diameter.

The detectors consist of very small spirals of platinum wire fed into the bores of a multi-bore high-purity alumina tube. Glass adhesive holds a small section of each turn of each spiral firmly in position



Temperature detector

after firing. Because all three of the materials used have compatible coefficients of expansion, the detectors give consistent readings and do not suffer from mechanical strain even after repeated thermal shocks.

For measurement in liquids and gases, detectors are available with protective sheaths of stainless steel which can also be fitted with light aluminium alloy or plastic connection heads.

Reader Enquiry Service No. 46

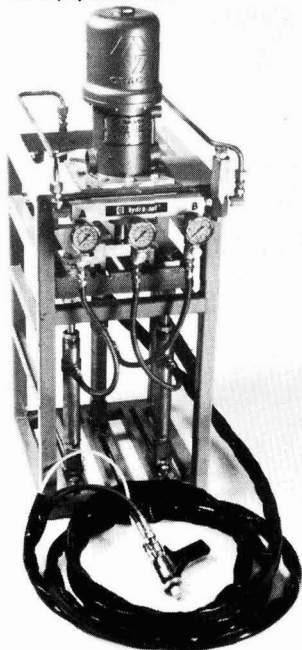
Graco launch

Graco have launched a new quiet-working Bulldog air motor. This motor meets current regulations for pump operating noise. It can be used with all lower pump assemblies that fit Graco's existing Bulldog air motors, giving users a wide choice of fluid pressure and delivery combinations.

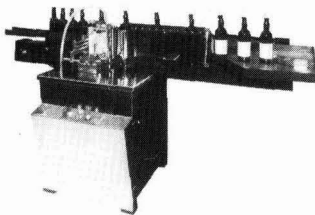
Graco have also launched a new small variable-ratio system for applying plural component materials. Called the Mini Variable-Ratio Hydra-Cat, this versatile system proportions, mixes and applies such plural component materials as paints, coatings, adhesives and silicone rubbers. It handles two- or, in certain cases, three-component materials. For example, it can be used to proportion and mix base material, catalyst and colourant, or a solvent thinner.

Application is by air or airless spray, extrusion or, for potting, by pouring. Made from standard Graco parts, it forms a modular system that offers many delivery, pressure and mix ratio possibilities.

Reader Enquiry Service No. 47



The Hydra-Cat



The MF 100

Small labeller

Morgan Fairest of Sheffield, manufacturers of bottle cleaning, filling, capping and labelling machinery for liquids and powders have extended the development of their smallest automatic labeller, the MF100.

For round containers only, of glass, plastic (PET), metal or board, the labeller will apply spot or wraparound labels from a minimum width of 50 mm up to a maximum width of 457 mm at infinitely variable speeds up to 100 bottles per minute depending upon the label width. The MF100 can also be set up for end labelling rectangular cartons or boxes.

Reader Enquiry Service No. 48

Coating thickness measurements

Vacuum Science Workshop Limited, have announced the availability of their ball-cratering instrument for coating thickness measurements. The technique can be used to determine the thickness of coatings in the range 1-100 μ m and is thus ideal for coatings produced by methods such as electrodeposition, hot-dipping, spraying, vapour deposition and ion plating.

The technique is quick and simple to use. A rotating steel ball, coated with diamond paste, is used to develop a spherical crater in the sample surface. The thickness of the coating is then found by simply measuring the diameter of the crater and the diameter of the exposed substrate. These measurements are made by using an integrally-mounted measuring microscope.

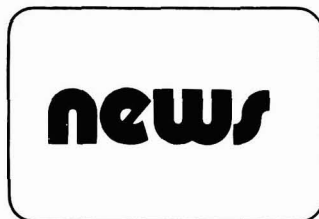
In addition to its use for measuring coating thickness, the technique may also be used to obtain composition-depth profiles when used in conjunction with Auger electron spectroscopy and the Electron probe microanalyser.

Reader Enquiry Service No. 49

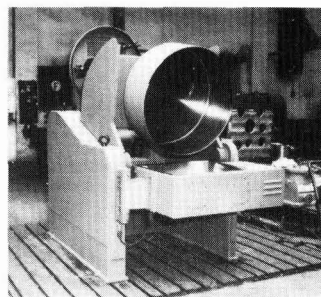
A new mixing machine for the UK

For many years AMK has produced a comprehensive range of advanced mixing, extrusion/kneading and compounding machinery. Now their "R" type mixer range, which has hitherto consisted of three machines, is complemented by the introduction of the 1VR.

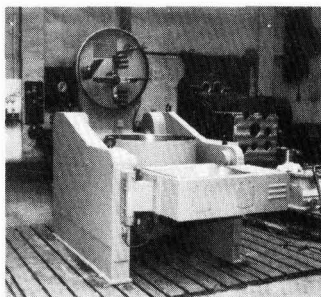
This new equipment embodies the well proven counterflow mixing principle, and the four mixing blades are further supplemented by an additional high speed cutter.



A big market for the "R" range has traditionally been found in the grinding wheel industry, and the development of the 1VR came about because a machine capable of discharging directly into a sieve unit of sufficient height to allow the use of conventional mobile collection vessels was required. This requirement has been met in full by the 1VR with its integral sieve unit which can be swung to one side when not in use, and its tiltable, hydraulically operated mixing trough.



AMK 1VR in discharge position



AMK 1VR with cutting head raised

The high speed cutter is independently driven, and is specially designed to quickly break-down any formation of agglomerates, and this procedure has proved most effective in producing a fast and uniform "wetting out" of difficult powders, and within the grinding wheel industry, for the mixing of abrasive grit of very fine mesh size.

Reader Enquiry Service No. 50

New hardener and polyester resin

Ciba-Geigy are offering a new low risk acid hardener, designated HT 1947GB, as an alternative to conventional anhydride hardeners for use with their T 1946 GB epoxy resin in the manufacture

news

of powder coatings. Using this hardener, high-gloss powder coatings with exceptional adhesion to a wide variety of materials can be produced. They also feature high impact strength and abrasion-resistance properties.

Unlike anhydride hardeners, the need to pregrind is eliminated as this acid functional hardener readily dissolves during the manufacturing process. It has a softening point of approximately 80 degrees centigrade, acid value (KOH/g) of 250 mg, and a flashpoint of 66 degrees centigrade. It is recommended for use in a weight ratio of 27 to 30 parts per 100 of XD 946 epoxy resin.

A new polyester resin specially formulated for use with epoxy resins in a 1:1 ratio for powder coatings has been announced by Ciba-Geigy. A choice of accelerators allows the preparation of either semi- or full-gloss powder coatings with good physical properties, in particular flexibility and overbake stability. It has a softening point of approximately 105°C, an acid value of 53-67 mg KOH/g, a Gardner colour of 5 units and a storage life at 25°C of at least 1 year.

The polyester resin is envisaged for use in powder coating applications by electrostatic spraying, where paint finishes are required with hard finishes of between 1 and 3 thousandths of an inch thick. These epoxy polyester based powder coatings are aimed specifically at manufacturers of products which have traditionally been finished with liquid paints, in particular metal furniture and domestic appliances. The new resin is called XC110.

Reader Enquiry Service No. 51

Inmont automatic mixing machine

Inmont Limited announce the introduction of a 48-place automatic mixing machine, for use by the motor vehicle refinish trade. It is the largest machine

available in the UK, and offers the most comprehensive range of colour bases.

This new model has been developed as a direct result of the variety and complexity of colours being offered by motor manufacturers and the shade changes experienced from painting panels and bodies not simply at separate plants but often in different countries.

Reader Enquiry Service No. 52

Pony adaptation

An ingenious adaptation of a Pony Labelrite semi-automatic labeller has enabled it to operate as a fully automatic unit, increasing the throughput rate two to three times in the wet-glue labelling of small bottles.

It is thought to have potential wherever there is a demand for the economic labelling of quite small cartons or bottles.

Reader Enquiry Service No. 53

literature

Thermal analysis

A new thermal analysis literature list is now available from Perkin-Elmer Limited. The listing includes the titles of 98 technical reports and application studies concerned particularly with pharmaceuticals, plastics, energy research, basic materials research, characterisation and quality control.

Perkin-Elmer have also made available a new brochure on open tubular column chromatography. The brochure is intended to act as a guide to potential users on the various types of open tubular column which are available and the methods of injection used with them. Operation of the new sigma split/splitless injector is described in detail and information is also included on the recent introduction of fused silica and quartz capillary columns.

Reader Enquiry Service No. 54

Thermal oxidiser application profiles

A series of single sheet application profiles, collated into complete sets, have been produced by Hygrotherm Engineering Limited of Manchester to explain in detail the many, varied applications of Hygrotherm Thermal Oxidisers and the

highly successful Hygrotherm/Hirt Burner.

Printed in colour, the complete set comprises individual A4 size sheets each of which cites and describes a specific contract where thermal oxidation facilities were urgently required.

Reader Enquiry Service No. 55

Technical leaflets

Leaflets are available from the Chemicals Division of British Industrial Plastics describing "Beetle" BE691 and BE692, which are recently developed isobutylated amino resins having a much lower odour than most other amino resins currently available.

BE691 is a urea resin and BE692 a melamine resin. In line with market demand, both possess good reactivity. BE692 has excellent low brake properties.

Reader Enquiry Service No. 56

meetings, etc.

Health and safety

The Plastics and Rubber Institute are holding a conference entitled "Health and safety in the plastics and rubber industries". The event is to be held at the University of Warwick from 29 September to 1 October 1980. Registration forms are available from the Plastics and Rubber Institute.

Reader Enquiry Service No. 57

appointments

Mr Alan J. Maltpress has been appointed general manager of Bonded Structures, Plastics Division, Ciba-Geigy Plastics and Additives Company, Cambridge. The appointment became effective on 31 March 1980.

David J. Busby has been appointed Secretary of the Tioxide Group Limited in succession to **John P. Richardson** who has retired.

Mr S. R. Monckton has been appointed Technical Director and **Mr B. Simpson** Deputy Sales Director at Mander-Kidd (United Kingdom) Ltd.

Further information on any items below may be obtained by circling the appropriate *Reader Enquiry Service* number on the form at the back of the *Journal*.

Obituary

Dorothy Lilian Tilleard

For numerous technical people in our industries, and for many years, the name "Miss Tilleard" (or "Tilly" as many knew her) signified first-hand knowledge and expertise in the latest advances in colour measurement and control, from the

earliest days of trichromatic colorimetry to modern computerisation. She joined the Paint Research Association (PRA) in 1930 with an honours degree in physics from East London (now Queen Mary) College. She was one of the very few women who became Fellows of the Institute of Physics in pre-war days and was for 30 years a member of OCCA. She retired in 1979 but continued as a

OCCA news

PRA consultant until her sudden death on 17 March 1980.

As colour measurement grew steadily more precise, she became greatly concerned with relationships between instrumental readings, visual appearance, colour tolerances, specification and subjective assessment by colour matchers. She studied closely colour behaviour and other optical properties of pigments in paint films, and constructed (with N. D. P. Smith) the third electron microscope to be built in the UK, now in the Science Museum.

She was interested in a wide range of physical phenomena and had an almost insatiable thirst for accuracy of information. Her findings and commentaries appeared in many PRA reports, in *JOCCA* and in other journals. She served on the OCCA Lightfastness Committee and on BSI committees covering artificial daylight (for OCCA), photometric integrators, signalling colours, optical tests, colour standards, viscosity measurement, etc. She was active in the Colour Group, and has been Secretary of the Colorimetry Sub-Committee and of the Light, Colour & Vision Group of the National Illumination Committee.

In 1972 she received the MBE for services to science and industry. Hers indeed was a remarkable career, but she will be remembered by many for her kind, gentle and friendly character at least as much as for her scientific knowledge, technical skills and achievements.

S.H.B.

SIR JOHN METHVEN

It is with deep regret that we record the death on 23 April 1980 of Sir John Methven, who delivered the Commemorative Lecture at the Association's Sixtieth Anniversary Celebrations in May 1978.

Manfred Hess

Manfred Hess, CChem, FRIC, FIMF, FTSC died on March 26, aged 81. He had been a member of this Association since 1941. He was born in Pirne near Dresden and after studying chemistry at Dresden, Munich and Würzburg he took over the management of the family business Chemische Fabrik Gustav Hess. Among the specialities manufactured by this company were black enamels and other products for the optical, photographic and electrical industries. After the advent of the Nazi Regime he left Germany, settled in this country and became head of industrial research at Mander Brothers Ltd of Wolverhampton. After nearly 25 years he retired from this position and moved to London where he practised as a consultant to the surface coatings industries until his death. His book "Paint Film Defects: Their Causes and Cure" has appeared in three English editions as well as in French, German and Spanish and is widely regarded as a standard reference work.

Manfred Hess had many other interests. In particular he had a passion for growing orchids which he did to perfection and he was a Fellow of the Royal Horticultural Society.

My own acquaintance with Manfred Hess began in 1956 when he lectured at an OCCA Symposium on the I-SCC Colour Aptitude Test. The acquaintance deepened into friendship and I am proud of the privilege of having known him.

He was a strong supporter of OCCA and attended many section meetings and most OCCA conferences. He also travelled a great deal and was frequently seen at technical conferences, meetings and exhibitions in continental Europe and the USA. He had a wide circle of friends in the paint industry, both in this country and overseas. He was greatly liked by everybody who knew him. A true



gentleman whose many friends will have a deep feeling of loss.

H. R. H.

Hull Section

Ladies' Evening

The Sixth Ordinary Meeting of the Section was held at the George Hotel, Land of Green Ginger, Hull, on Monday 3 March 1980. This was the annual 'Ladies Evening' when Mr J. Wittle, Manager of the 'Seconds Department' of Hornsca Pottery, gave an illustrated talk entitled, 'The Making of Hornsea Pottery'.

Essentially the lecturer talked around a comprehensive series of slides which illustrated not only the making of pottery but also the wide range of facilities and entertainments that the Company offer to the customer at the Hull and Lancaster sites.

The talk was excellently presented in a witty and humorous style which stimulated many questions from the audience.

A free buffet was provided. There was also a small selection of pottery for sale, (seconds of course).

The Hull Section Chairman, Mr Roy Brooks, gave the vote of thanks.

F.D.R.

REPORT OF COUNCIL MEETING

A meeting of Council took place at 2.00 p.m. on Wednesday 2 April 1980 at the Great Northern Hotel, London N1 9AN.

The President (Dr F. M. Smith) was in the Chair and there were 23 members present; Mr D. S. Newton was also present by invitation. The President extended the best wishes of Council to Mr S. R. Finn on his recovery from his recent illness and Mr Finn thanked Council for the Honorary Membership bestowed upon him.

Reports were received on the following:

(a) The final arrangements, including the agenda, for the Annual General Meeting on 26 June and the Luncheon Lecture which will precede it. The lecturer, Professor Sir Hermann Bondi, KCB, FRS had chosen as the

title of his lecture *Energy in the World*.

(b) The Savoy Dinner Dance which would take place on 11 April.

(c) The number of members whose 1980 subscription had not been received and it was agreed by Council, in accordance with Article 15, that any member whose subscription was six months in arrears would cease to be a member at the end of June.

(d) The annual accounts for 1979 and the estimates for 1980 which had been presented to the Finance Committee at its meeting in March.

(e) The 1980 exhibition, where 124 organisations from 14 countries would be showing and a discussion took place on the arrangements for future exhibitions.

(f) The Call for Papers for the conference which will take place at Bath 17-20 June 1981 under the title *Alternative technologies in coatings*.

(g) Representation on BSI committees, where various changes had been notified.

(h) The Professional Grade Committee meeting, where one Associate had been transferred to Fellowship and two candidates admitted as Associates; the Committee was considering the present regulations and expect to report some slight modifications to the Council at its next meeting.

(i) Papers submitted for publication in the *Journal* and the completion of the first monograph on *Marine finishes* by Dr T. Banfield which had appeared in the February and March

OCCA news

issues and which would be available as a separate booklet.

(j) Membership subscription rates for

1981, as recommended by the Finance Committee at the previous Council Meeting, and which were duly confirmed.

It was unanimously agreed to confer upon Mr J. D. W. Davidson, a Commendation Award for his outstanding work on behalf of the Association and the Scottish Section.

Reports were received from the various Section Committees and it was stated that Chairmen's Insignia of Office were being prepared for the Nigerian and Zim-

abwe Branches of the General Overseas Section.

A vote of thanks was recorded to members retiring from Council. The President mentioned that appreciation had been expressed for the floral tributes sent by the Association on the deaths of two distinguished members, Miss D. L. Tilleard, MBE and Mr Manfred Hess.

There being no other business, the President thanked members for their attendance and declared the meeting closed at 4.02 p.m.

Association Dinner and Dance 1980

The Association's biennial Dinner and Dance was held this year on Friday 11 April at the Savoy Hotel, London, when approximately 240 members and guests were received by the President Dr F. M. Smith and Mrs Smith. The Guest of Honour was Professor R. O. C. Norman (President of the Royal Institute of Chemistry) and the other Association guests were:

Mr L. Robson (Master, Worshipful Company of Painter-Stainers) and Baroness Robson.

Dr J. K. Skelly (President, Society of Dyers & Colourists) and Mrs Skelly.

Mr P. G. L. Vivian (President, Institute of Metal Finishing) and Mrs. Vivian.

Professor G. C. Wood (President, Institute of Corrosion, Science & Technology) and Mrs Wood.

Mr L. H. Silver (President, Paintmakers Association of GB Ltd. and Past President OCCA 1973-75) and Mrs Silver.

Mr D. J. McInnes (President, Society of British Printing Ink Manufacturers) and Mrs McInnes.

Mr J. R. Green (Chairman, British Resin Manufacturers' Association) and Mrs Green.

Mr M. A. Kerr (Chairman, British Colour Makers' Association) and Mrs Kerr.

Mr H. B. Meyer (Chairman, British Adhesives Manufacturers' Association) and Mrs Meyer.

Dr S. Wernick, OBE (Hon. Secretary General, Institute of Metal Finishing).

Mr M. Leveté (Director, Paintmakers Association of GB Ltd) and Mrs Leveté.

Dr G. de W. Anderson (Managing Director, Paint Research Association) and Mrs Anderson.

Mr E. Sangster (Director, National Federation of Painting and Decorating Contractors) and Mrs Sangster.

After dinner the President first proposed the Loyal Toast and then welcomed the Association's guests and the ladies in a charming and witty speech.

Professor R. O. C. Norman replied on behalf of the guests. His speech was informative and entertaining, covering such diverse topics as education, its relationship with industry and the greater

public awareness of the value of manufacturing industry.

The evening continued with dancing until 1 am to music from Ted Hetherington and his Modern Orchestra, as well as a highly original carabet act from John Wade.



Left to right Dr F. M. Smith (President), Mrs Smith and Professor R.O.C. Norman FRS President, Royal Institute of Chemistry (Guest of Honour)

OCCA CONFERENCE 1981



Alternative technologies in coatings

CALL FOR PAPERS

The next OCCA Biennial Conference will be held at the Beaufort Hotel, Bath from 17 – 20 June 1981 with the theme "Alternative technologies in coatings".

The future holds both opportunity and challenge for alternative technologies and topics covered by the Conference should include, EEC regulations covering the introduction of new chemicals, alternative means of obtaining opacity, the impact of microprocessors and computers on processing and application methods, high solids coatings, aqueous systems, radiation curing and powder coatings.

A departure from the usual conference format will be the inclusion of a "Discourse" session with the sub-title "Alternatives to coatings", where the use of techniques such as cathodic protection and substitution of coated items by plastics could be discussed.

The Hon. Research & Development Officer now invites offers of papers for presentation at this Conference. Anyone wishing to submit a paper for consideration should notify his intention as soon as possible to: **The Director & Secretary, Oil & Colour Chemists' Association, Priory House, 967 Harrow Road, Wembley, Middlesex HA0 2SF, England (Tel: 01-908 1086; Telex: 922670 OCCA G).**

OCCA news

OCCA Conference

The venue and format

The ancient city of Bath has been chosen as the venue for the Association's next biennial Conference, which will be held from 17-20 June 1981, with headquarters at the Beaufort Hotel.

The theme for the Conference will be "Alternative technologies in coatings" and the technical programme will consist of four sessions, one of which will take the form of a discourse.

A social programme will be arranged for delegates' ladies which will enable them to visit local places of interest.

A call for papers to be presented at the Conference is published elsewhere in this issue, and details of papers accepted will be published later in the year.

Reader Enquiry Service No. 70



Pulteney Bridge Bath

new members

Ordinary Members

BUCK, NEVILLE LESLIE, PO Box 1620, Durban, 4000, Republic of South Africa *(Natal)*

CHAPMAN, RICHARD GEORGE, 11 Lincoln Close, Runcorn, Cheshire *(Manchester)*

LO, VERNON, BSc, MSc, 1031 Quebec Street, New Westminster, BC, Canada V3M 1K6 *(Ontario)*

MAYNARD, NIGEL PAUL, NZCS (Chem), BSc, 68 Kauri Road, Whenuapai, PO Box 1434, Auckland, New Zealand *(Auckland)*

ROSS, PETER JAMES, BSc, Raadter Str, 149 Haarzopf, 4300 Essen I, W. Germany *(Gen. Overseas)*

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

SCOTT, NEVILLE, BSc, DipEd, 22 Oakridge Road, Spital, Wirral, Merseyside L62 2AS *(Manchester)*

YOUNG, RALPH GRANVILLE, Federated Paints Ltd, Blairlinn Industrial Estate, Cumbernauld, Scotland *(Scottish)*

Associate Members

BREWER, MARK GREGORY, F4/17 Patterson Street, Sandringham, Auckland, New Zealand *(Auckland)*

GOW, MICHAEL GASCOIGNE, 1 Manaia Place, Mairangi Bay, Auckland, New Zealand *(Auckland)*

POWELL, WILLIAM ERIC, PO Box 9338, Newmarket, Auckland, New Zealand *(Auckland)*

Registered Students

ASHTAPUTRE, YASHWANT WAMAN, 131/2 Siddeshwar Lane, Baramati Dist; Poona, Maharashtra, India *(Gen. Overseas)*

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

OCCA Golf Trophy being organised by the London Section will be held at the Canons Brook Golf Club in Harlow, Essex. Intending participants should contact Mr. B. F. Gilliam, 25 Regency Close, Chigwell, Essex IG7 5NY.

Wednesday 18 June

'The maintenance and protection against corrosion of North Sea Structures' by M. Small of Berger

(UK) Ltd. Joint meeting with the Institute of Petroleum, London Branch, at 61 New Cavendish Street, London W1 commencing at 6.00 p.m.

Thursday 26 June

Association Annual General Meeting, at the Piccadilly Hotel, London N1, commencing at 3.00 p.m., following a luncheon and a lecture by Prof. Sir Herman Bondi on "Energy in the world".

OCCA diary

June

Wednesday 11 June

The 1980 Golf Tournament for the

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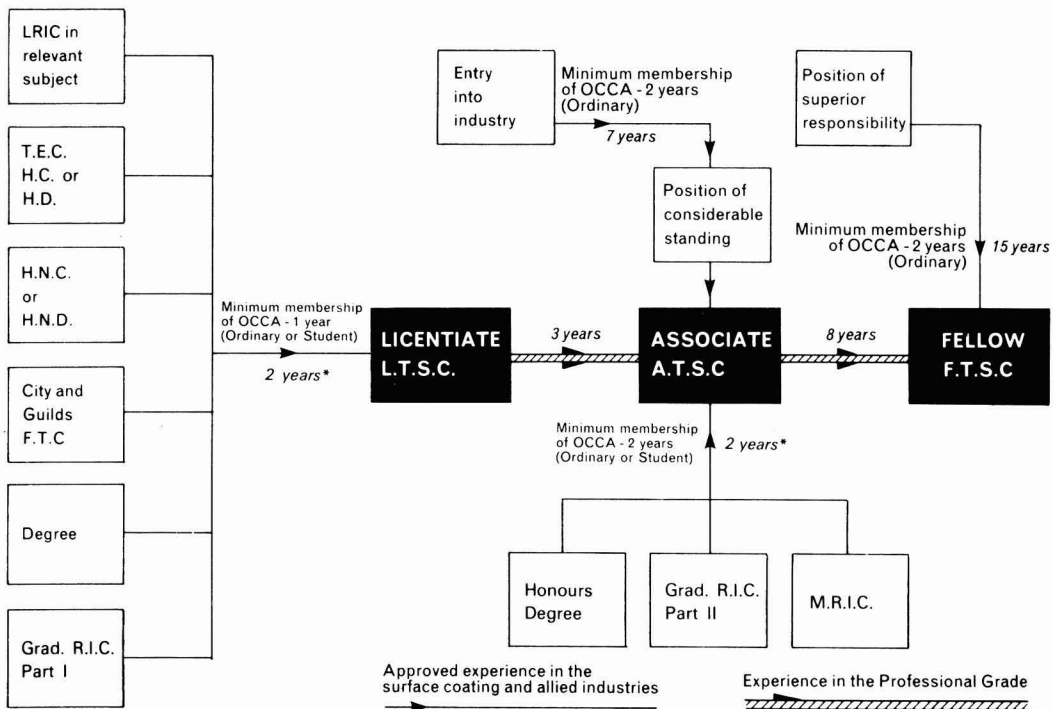
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Optional Professional Grade for Ordinary Members

The innovation of the Professional Grade has proved to be most successful, as evidenced by the impressive list of names in the December issue of the *Journal*. For the convenience of potential applicants, a chart indicating different routes to the various grades is shown below.

Routes to the Professional Grades



*Not necessarily after qualification – see regulations.

Note: At present there is no restriction on Students up to 21; between 21 and 25 a certificate from the employer or college confirming the course being taken is required.

Regulations for admission to the Professional Grade – Amended December 1979

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

A. Licentiate

1. Shall be an Ordinary Member of the Association and have been an Ordinary Member or Student of the Association for not less than one year.
2. Shall have attained the age of 22.
3. (a) Shall be a Licentiate of the Royal Institute of Chemistry in Coatings Technology or another relevant subject, such as advanced analytical chemistry, colour chemistry or polymer science.
- OR (b) Shall have passed the Higher Certificate or Higher Diploma of the Technician Education Council in coatings technology or other relevant subjects.

- OR (c) Shall have passed Higher National Certificate or Higher National Diploma in a relevant subject.
- OR (d) Shall hold the Full Technological Certificate of the City and Guilds of London Institute in a relevant subject.
- OR (e) Shall be a graduate in a relevant subject.
- OR (f) Shall have passed Part I of the examination for the Graduateship of the Royal Institute of Chemistry or Council of Physics.
- OR (g) Shall have passed such other qualifications as approved by the professional Grade Committee from time to time.

4. Shall have attained approved experience in the science or technology of coatings. It is not expected that sufficient experience would be gained in a period of less than two years in the industry. Approved experience may be gained before, during or after the qualifications in paragraph (3) above have been attained.
5. Shall be required to satisfy the Professional Grade Committee, or some other body approved by the Professional Grade Committee in a *viva voce* examination and submit a dissertation on a topic previously approved by the Professional Grade Committee.
6. Shall normally be sponsored by three Ordinary Members of the Association in the professional grade (either

Associate or Fellow) at least one of whom must be a Fellow.

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

B. Associate, being already a Licentiate

1. Shall, since his election to the Licentiate, have practised the science or technology of coatings for not less than three years.
2. Shall provide evidence acceptable to the Professional Grade Committee of his superior professional skill and maturity.
3. Shall have published work which, in the opinion of the professional Grade Committee, is of a sufficiently high standard OR may be required to submit a thesis or dissertation on a topic previously approved by the professional Grade Committee OR shall hold the City & Guilds of London Institute Insignia Award.
4. MAY be required to satisfy the Professional Grade Committee or some other body as approved by the Professional Grade Committee in a *viva voce* examination.
5. Shall normally be sponsored by three Ordinary Members of the Association in the professional grade (either Associate or Fellow) at least one of whom must be a Fellow.
6. Shall have paid the fee stipulated by Council and have paid the current subscription payable by an Ordinary Member.

C. Associate, not already a Licentiate

EITHER

1. Shall be not less than 24 years of age.
2. Shall be an Ordinary Member of the Association and have been an Ordinary member or Student of the Association for not less than two years.
3. Shall hold the Graduateship of the Royal Institute of Chemistry or Council of Physics or a University or Council of National Academic Awards degree recognised by the Royal Institute of Chemistry or Institute of Physics as giving full exemption from the Graduateship examination.
4. Shall have attained approved experience in the science or technology of coatings. It is not expected that sufficient experience

would be gained in a period of less than two years in the industry. Approved experience may be gained before, during or after the qualifications in paragraph (3) above have been attained.

5. Shall normally be required to satisfy the Professional Grade Committee or some other body approved by the professional Grade Committee in a *viva voce* examination.
6. Shall normally be sponsored by three Ordinary Members of the Association in the professional grade (either Associate or Fellow) at least one of whom must be a Fellow.
7. Shall have paid the fee stipulated by Council and have paid the current subscription payable by an Ordinary Member.

OR

8. Shall be not less than 30 years of age.
9. Shall be an Ordinary Member of the Association and have been an Ordinary Member of the Association for not less than two years.
10. Shall have been engaged in practising the science or technology of coatings for not less than seven years and shall have attained a position of considerable standing in the industry.
11. Shall normally be required to satisfy the Professional Grade Committee in *viva voce* examination of his professional competence.
12. Shall normally be sponsored by three Ordinary Members of the Association in the professional grade (either Associate or Fellow) at least one of whom must be a Fellow.
13. Shall have paid the fee stipulated by the Council and have paid the current subscription payable by an Ordinary Member.

D. Fellow

Note: This is the senior award of the professional grade and signifies that the holder has made outstanding contributions to the science or technology of coatings or has reached a position of eminence in the industry through the practice thereof. The Professional Grade Committee will require substantial evidence of professional maturity in the science or technology of coatings although commercial experience will be taken into account in assessing the merits of candidates.

1. Shall be not less than 33 years of age.
2. Shall have been an Ordinary member of the Association for not less than two years.

3. Shall be engaged in a position of superior responsibility in the coatings industry.
4. EITHER (a) shall have been an Associate of the professional grade for at least eight years;
OR (b) shall have not less than fifteen years' experience of the science or technology of coatings in a position of superior responsibility.
5. Shall submit, with his application, an account of his experience, with due reference to scientific and technological interests, achievements and publications.
6. Shall normally be sponsored by three Ordinary Members of the Association in the professional grade, all of whom must be Fellows.
7. Shall have paid the fee stipulated by the Council and have paid the current subscription payable by an Ordinary Member.

The fees payable with applications are as follows:

Fellow-£10.00	Associate-£6.00
Licentiate-£3.00	
(Plus VAT at standard rate)	

Application

Completed application forms should be returned, together with the appropriate remittance, to the Director & Secretary at the Association's offices (except in the case of those Members attached to the Cape, Natal, Transvaal, Wellington, Auckland and Ontario Sections, who should address their forms to their Section Hon. Secretaries).

The Committee wishes it to be known that members rejoining the Association after a period in other industries may include length of service as an Ordinary Member before their resignation as part of the qualifying periods for entry into the Grade.

Students wishing to apply for entry into the Professional Grade must first make application in writing for upgrading to Ordinary Membership, giving the reasons for their eligibility for such regrading. Applications, together with the appropriate remittance, should be addressed as for application for admission to the Professional Grade.

Potential applicants are recommended to give the fullest possible details of their appointments, including the number and type of staff under their control, and indicating to whom the applicant is responsible, as this aids the committee considerably in its deliberations.

CLASSIFIED ADVERTISEMENTS

Classified Advertisements are charged at the rate of £5.00 per single column cm. Advertisements for Situations Wanted are charged at £1.50 per line. A box number is charged at £1.00. They should be sent to D. N. Buddles, Assistant Editor, Oil & Colour Chemists' Association, Priory House, 967 Harrow Road, Wembley, Middlesex HA0 2SF. JOCCA is published EVERY month and Classified Advertisements can be accepted up to at least the 12th, and in exceptional circumstances the 20th of the month preceding publication. Advertisers who wish to arrange for an extension of the copy deadline should contact the Assistant Editor, D. N. Buddles, at the address given above (telephone 01-908 1086, telex 922670 OCCA G).

SITUATIONS VACANT

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Mr. J. D. Reavy,
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Association Ties

The Association single motif tie is once more available for members from the Association's office and can be obtained with either a navy blue or a maroon background.

Orders (prepayment only at £3.50 each inclusive of VAT and postage by surface mail) should state the background colour required and give the name and address of the member concerned.

CLASSIFIED ADVERTISEMENTS



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Dr. N. R. Whitehouse,
Head of Consultancy Department,
Paint Research Association,
Waldersgrave Road,
Teddington,
Middlesex
TW11 8LD

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Write to **PO Box 4661, Salisbury, Zimbabwe.**

OCCA AGM Luncheon Lecture

Thursday 26 June 1980

A lecture entitled: **"Energy in the world"**

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CLASSIFIED ADVERTISEMENTS

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OCCA Monograph No. 1

Marine Finishes

Reprints are now available of the OCCA Monograph number 1 on Marine Finishes by Dr T. A. Banfield from the Association's offices. These bound copies reprinted from the February and March issues of the *Journal* cost £2 including post and packaging.

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Further information on any of the products advertised in this *Journal* may be obtained by circling the appropriate number on the *Reader Enquiry Service (RES)* form above. The *RES* numbers are given in brackets below. Enquiries will be forwarded to the organisation concerned.

C		I	
Ciba-Geigy (UK) Limited, Pigments (Paint) Division (129)	i	ICI Ltd – Organics Division (141)	viii
D		J	
Dynamit Nobel (123)	Insert	Johns-Manville International (105)	Insert
E		O	
ECC International (115)	v	OBS Machines Ltd (107)	Cover
G		P	
George Godwin	Insert	Paint Research Association (142)	Insert iv
Glen Creston Machinery Ltd (125)	Cover	Q	
H		S	
Hardman E., & Son Co. Ltd (103)	Cover	Sachtleben Chemie GmbH (119)	ix
Henkel KGaA (104)	Insert	Sanyo-Kokusaku Pulp Co. Ltd (109)	ii
Hubble, Leslie, Ltd (120)	Cover	Sub-Tropical Testing Service Inc (111)	iv

JOCCA

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For further information circle items listed below:

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20
21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40
41 42 43 44 45 46 47 48 49 50 51 52 53 54 55 56 57 58 59 60
61 62 63 64 65 66 67 68 69 70 71 72 73 74 75 76 77 78 79 80
81 82 83 84 85 86 87 88 89 90 91 92 93 94 95 96 97 98 99 100
101 102 103 104 105 106 107 108 109 110 111 112 113 114 115 116 117 118 119 120
121 122 123 124 125 126 127 128 129 130 131 132 133 134 135 136 137 138 139 140
141 142 143 144 145 146 147 148 149 150 151 152 153 154 155 156 157 158 159 160
161 162 163 164 165 166 167 168 169 170 171 172 173 174 175 176 177 178 179 180
181 182 183 184 185 186 187 188 189 190 191 192 193 194 195 196 197 198 199 200

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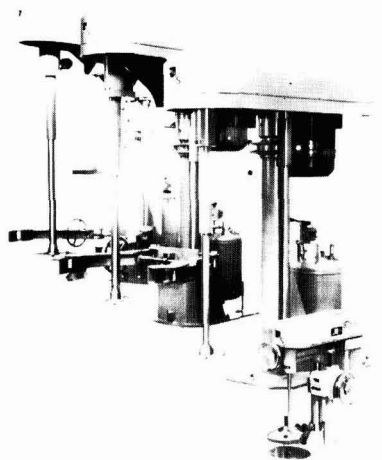
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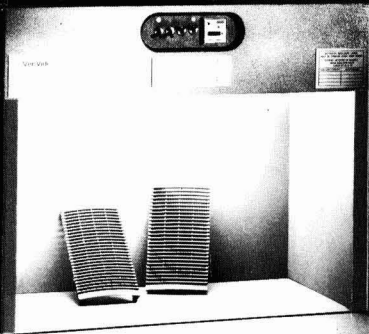


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