

JOURNAL OF THE OIL AND COLOUR CHEMISTS' ASSOCIATION



Vol. 53 No. 1

January 1970

Outline/unit area chromatography: II. Effects due to dye structure

D. W. Poxon

Outline/unit area chromatography: III. The detection and characterisation of residual polar impurities in refined mineral oils

D. W. Poxon

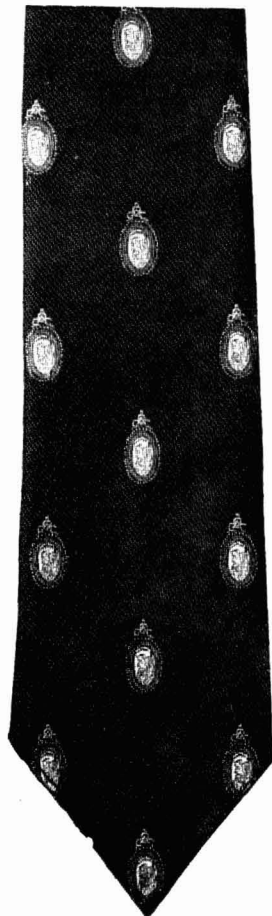
The adhesion of organic surface coatings to cadmium plate

P. Walker

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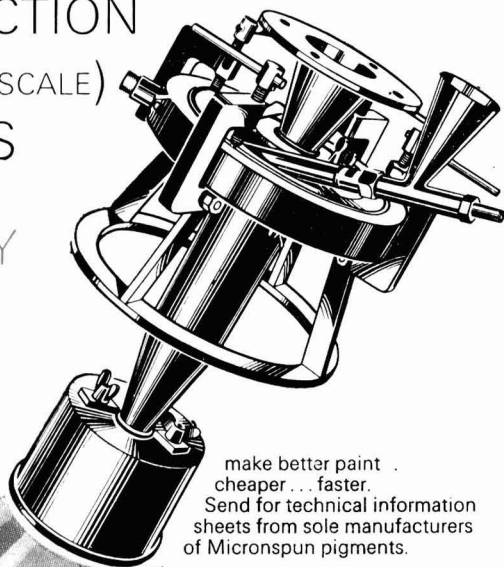
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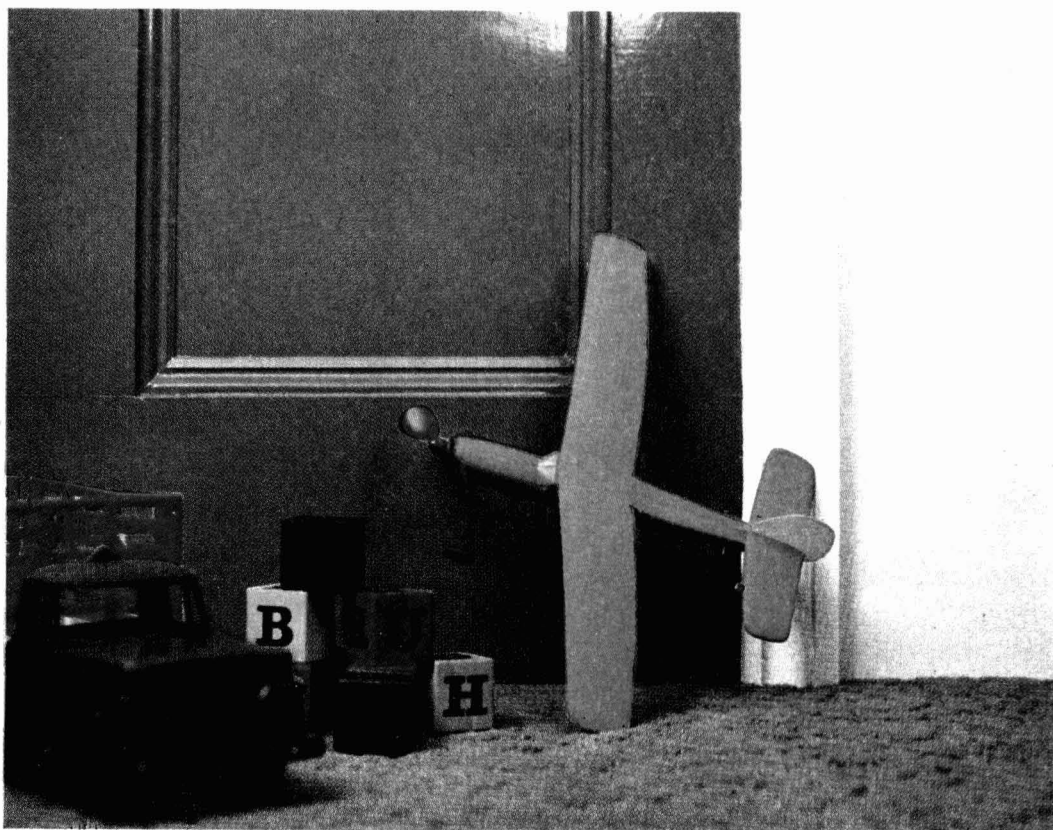
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Transactions and Communications

Outline/area chromatography: II. Effects due to dye structure

By D. W. Poxon

Department of Chemistry, University of Technology, Loughborough, Leicestershire

Summary

A mixture of oleic and palmitic acids, the carrier liquid, gives a single outline/area index peak at 17.6°C, when chromatographed with petroleum spirit on paper impregnated with the dye C.I. 11110, Disperse Red I, and other peaks appear at fixed temperatures characteristic of the polar group when compounds having other polar groups are dissolved in the above mixture.

The effect of using other dyes having structures related to that of the above dye has been investigated for similar mixtures of compounds. It has been shown that the distribution of the hydroxyl group, and to some extent other groups on the dye molecule, affects both the shape of the basic index-temperature curve given by the carrier liquid and the response of the outline/area index to the presence of other compounds.

A tentative theoretical explanation of these effects, involving ionisation of the dye and based on phase changes in interfacial complexes, is proposed.

Key words

Raw materials used in manufacture or synthesis of ingredients for coatings

lauric acid
oleic acid
palmitic acid

Processes or methods associated with analysis, measurement and testing

chromatography

L'influence de la structure des colorants sur l'indice profil/aire unitaire en chromatographie sur papier

Résumé

Un mélange des acides oléique et palmitique, en tant que liquide-porteur, ne donne qu'une maximale indice profil/aire unitaire qu'à 17.6 C, qu'une lors de la chromatographie sur papier imprégné du colorant C.I. 11110, Disperse Red I, à partir d'un distillat pétrolique. D'autres maxima se révèlent aux températures définies qui se dépendent du groupement polaire, dans le cas où des composés ayant de différents groupements polaires sont dissous dans le mélange mentionné ci-dessus.

On a investigé l'effet d'autres colorants, ayant des structures semblables à celle du colorant ci-dessus. Sur de pareils mélanges de composés. On a démontré que la répartition du groupement hydroxylique et également en quelque degré d'autres groupements de la molécule colorante ont une influence sur la forme et à la fois sur la courbe de base de l'indice-température mise en évidence par le liquid-porteur, et en même temps sur la réponse de l'indice profil/aire unitaire à la présence d'autres composés.

On propose une explication tentative au point de vue théorique de ces effets, où il s'agit de l'ionisation du colorant, et basées sur les changements en phases de complexes interfaciaux.

Umriss/Einheit Areachromatographie: Auswirkungen der Farbstoffstruktur

Zusammenfassung

Eine Mischung von Olein- und Palmitinsäuren, die Trägerflüssigkeit, ergibt eine einzelne Umriss/Area Indexspitze bei 17,6°C, wenn mit Petroleumspirit auf mit Farbstoff C.I. 11110 imprägniertem Papier chromatographiert. Andere Spitzen werden bei bestimmten Temperaturen, die für polare Gruppen charakteristisch sind, sichtbar, wenn Verbindungen, welche andere polare Gruppen enthalten, in obiger Mischung aufgelöst werden.

Bei ähnlichen Mischungen von Verbindungen wurde geprüft, wie sich der Einsatz anderer Farbstoffe, deren Struktur dem vorstehend erwähnten Farbstoff ähnelt, auswirkt. Es wurde aufgezeigt, dass die Verteilung der Hydroxylgruppen und in gewissem Masse anderer Gruppen am Farbstoffmolekül, sowohl den Verlauf der basischen Index-Temperaturkurve, die von der Trägerflüssigkeit angezeigt wird, als auch die Empfindlichkeit des Umriss/Area Indexes gegenüber anwesenden anderen Verbindungen beeinflusst.

Versuchsweise wird eine theoretische Erklärung dieser Wirkungen unter Einbeziehung von Ionisierung des Farbstoffes und auf Phasenänderungen in Grenzflächenkomplexen beruhend in Vorschlag gebracht.

Эффекты контурно-единичной поверхностной хроматографии вызванные структурой красителя

Резюме

Смесь олеиновой и пальмитиновой кислот, в качестве несущей жидкости, дает единичный контурно-поверхностный показатель пика при 17,6°C, при хроматографировании бензином на бумаге пропитанной дисперсионным красным красителем марки C.I. 11110,1 и другие пики появляются при определенных температурных характеристиках полярной группы, когда соединения имеющие другие полярные группы растворяются в вышеупомянутой смеси.

Рассматривается эффект применения других красителей со структурами родственными структуре вышеупомянутой краски, для сходных смесей соединений. Показано что распределение гидроксильной группы, и до известной степени и других групп, на молекуле красителя, влияет как на форму основной температурно-указательной кривой несущей жидкости, так и на реагирование контурно-поверхностного показателя на присутствие других соединений.

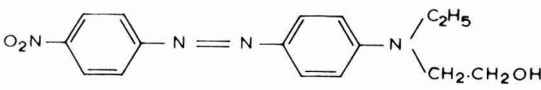
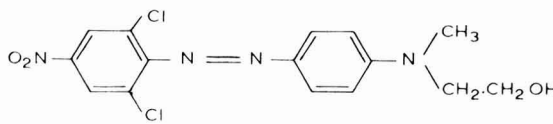
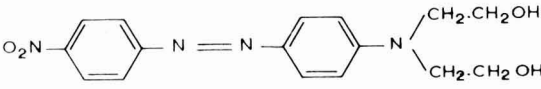
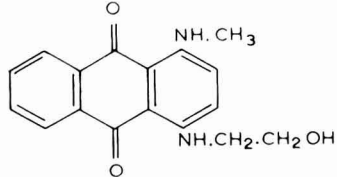
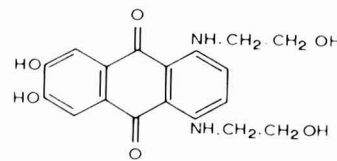
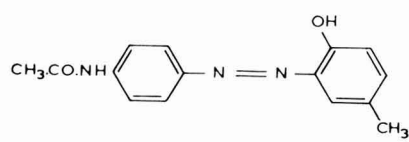
Предлагается предварительное теоретическое объяснение этих эффектов, учитывающее ионизацию краски и основанное на фазовых превращениях в поверхностных разделах комплекса.

Introduction

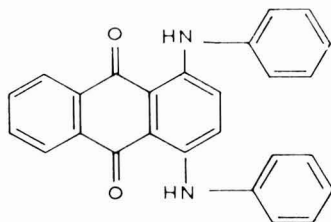
It has been found that on paper dyed with the dye C.I. 11110, Disperse Red 1 (see Table 1) the shape of a chromatographic band as measured by length of outline per unit area is sensitive to temperature, concentration, and type of polar groups present in the material being chromatographed, the eluant being petroleum spirit (boiling range 40-60°C).¹

The above dye gives only one definite peak value of this outline/unit area index at 17.6°C in the temperature range 15.0 to 19.5°C for a mixture of oleic acid containing sufficient palmitic acid to give a setting point of 18°C (the carrier liquid), but when other compounds are present in small concentration further peaks are produced at temperatures related to specific polar groups and at concentrations in the carrier liquid specific to the particular compounds.

Table 1
Dyes

Formula and name	Colour Index No.
1.  Dispersol Fast Scarlet, B.	11110 Disperse Red. 1
2.  Dispersol Fast Orange Brown R.N. 150	11100 Disperse Orange 5
3.  Dispersol Fast Red R. 300	11130 Disperse Red 19
4.  Duranol Brilliant Blue BN 300	61505 Disperse Blue 3
5.  Duranol Blue Green B. 300	62500 Disperse Blue 7
6.  Disperse Fast Yellow G	11855 Disperse Yellow 3

7.



Waxoline Green, G.

61565
Solvent Green 3

Since these phenomena have resulted in the development of a system of chromatography which may be used for complicated mixtures of high molecular weight compounds without the necessity for the separation of individual compounds and which gives information of the types and, to a certain extent, of the number, of compounds present², it was thought desirable to investigate the influence of the structure of the dye.

Four extra dyes were, therefore, used in which the same basic feature of $-\text{CH}_2-\text{CH}_2-\text{OH}$ chains attached to nitrogen atoms was retained, but other structural details of the dye molecule were different.

The actual variations in structure may be seen in Table 1, in which compound 1 is the original dye, 2 and 3 are closely related to it in structure, but 4 and 5 are basically different, being anthraquinoid instead of azo-dyes.

Experimental

Except for the fact that the sectors of the paper were painted in pairs with saturated solutions in ethanol of the four dyes 2 to 5, one to each pair, the experimental details were as previously stated^{1,2}.

The following combinations were tried for each dye:

- carrier liquid alone,
- carrier liquid containing 1 per cent of phloroglucinol,
- carrier liquid containing 1 per cent of lauric acid.

All development was with petroleum spirit (boiling range 40-60°C).

Results and discussion

The results are shown in Figs. 1, 2 and 3, in which the temperatures at which the original dye (no. 1 Table 1) would give index values for the relevant polar groups are indicated by vertical lines, C indicating the position of the carrier peak.

The four curves in Fig. 1 (carrier only) for dyes 2 to 5 show considerable similarity to each other but major differences from the original dye, 1, which would only give a peak at position C.

Dye 5 gives a very much flattened curve, which cannot be due solely to the presence of the two ethanolic hydroxyl groups, since the curve for dye 3, which also has two such groups, is not flattened.

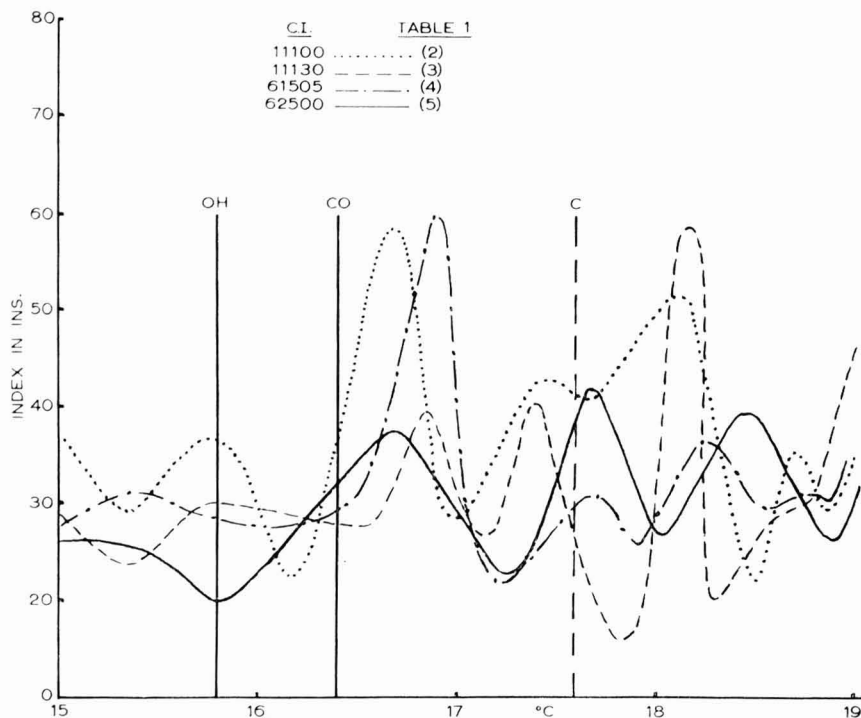
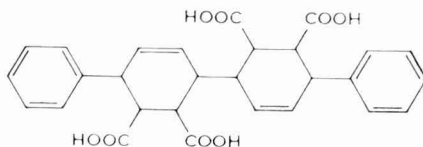


Fig. 1. Carrier only

The reduction in peak height would appear to be due to the hydroxyl groups, probably both ethanolic and phenolic, being widely distributed over the molecule. Compounds of this type,

e.g.



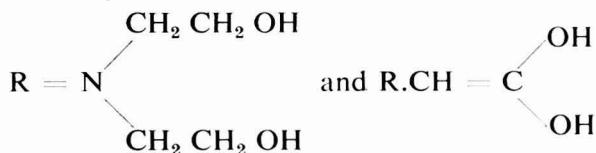
present in the mixture being chromatographed have been found to give similarly flattened curves with the original dye³.

In the region 16.5-17.5°C there is a steady shift of the peak position towards higher temperatures as the dye molecule becomes more complex, i.e. from dye 2 to dye 5, although this is somewhat obscured in the case of the last dye because of the flattened curve.

The region above 17.6°C, i.e. at higher temperatures than the position of the carrier peak given by the original dye, is less definite with the new dyes, although there appears to be some differentiation between the azo and anthraquinoid dyes at about 18°C. The most striking effect is the large peak given by dye 3 at this temperature.

The high peak given by the carrier liquid with dye 3 at 18.1°C is of interest, since many acids also give a peak at this temperature with the original dye when added to the carrier liquid in very small quantities.

The similarity between the structures



the enolic form of a carboxylic acid, is self evident.

If it is objected that the latter would only be present in very small amount, it should be noted first, that all acids do not give this peak and secondly, that an investigation of concentration effects using dodecanol showed that the critical concentration for peak formation could be of the order of 10^{-6} molar.

The introduction of phloroglucinol (Fig. 2) into the carrier liquid-dye system causes a considerable disruption of the pattern shown in Fig. 1.

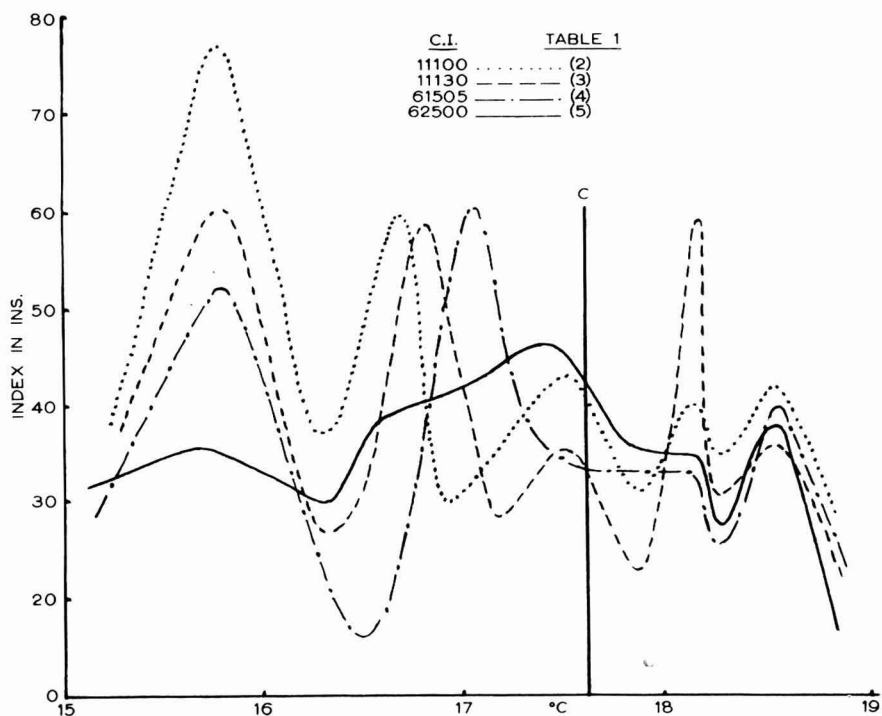


Fig. 2. 1 per cent phloroglucinol in carrier

The curves for the different dyes are now no longer so similar.

The most striking effect is seen at 15.8°C, where the original dye would show a peak index value of hydroxyl. The original peaks of Fig. 1 are either partially or completely suppressed.

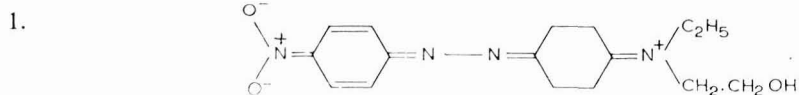
This suggests some form of competition for the oil/water interface between the hydroxyl groups of the phloroglucinol and those of the dye in each case.

A tentative explanation of the sharp changes in outline/unit area index at a certain specific temperature is that a phase change is occurring in an interfacial complex formed between the dye and the other compounds present at the relevant temperature.

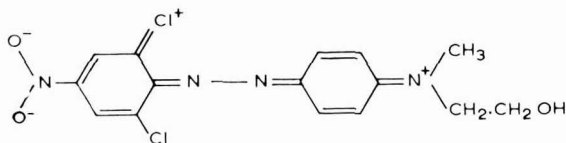
Such interfacial complexes are most easily formed when one of the components is both ionised and strongly adsorbed at the interface.

The original dye (1), and the other dyes given in Table 1, may all be written in the ionised forms (see Table 2), whereas a dye such as Dispersol Fast Yellow G. (C.I. Disperse 3, 11855) Table 1, dye 6, which cannot easily be written in such a stable ionised form, does not show any index temperature relationship.

Table 2
Dyes giving ionic structures

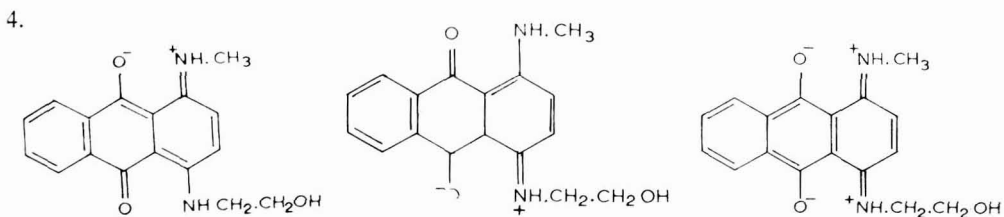


2. Similar to above, and:



for each chlorine atom

3. Similar to No. 1



5. Similar to No. 4

Similarly, in the case of Waxoline Green (C.I. Solvent Green, 3, 61565), Table 1, dye 7, the weakly water soluble groups are "screened" by the phenyl groups and strong adsorption at the oil-water interface is unlikely. In fact, this dye is completely insoluble in water (unlike the peak-producing dyes which are appreciably soluble) and shows no index-temperature relationship.

The fact that the dyes, other than 1 and 3, give extra charged forms of the molecule could account for the more complicated curves. In the case of dye 3 the presence of two hydroxyl groups would also cause differences, as previously indicated.

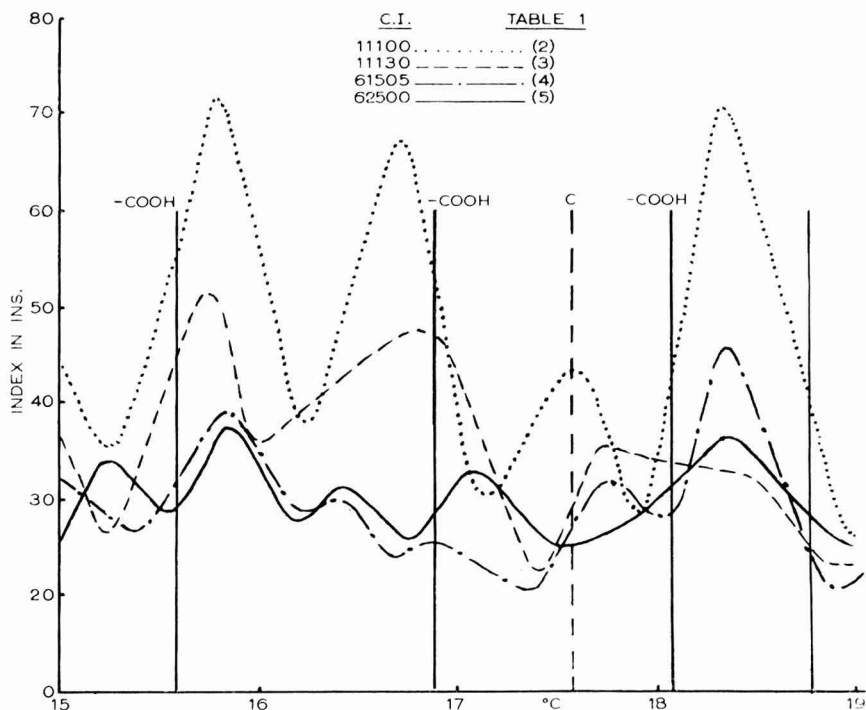


Fig. 3. 1 per cent lauric acid in carrier

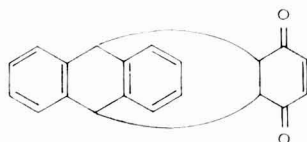
According to this theory, the elimination of the original peak at 15.8°C by phloroglucinol indicates that this complex is no longer being formed, presumably because the dye has been excluded from the interface except, perhaps, in the case of dye 2, which still shows a remnant of the original peak. Similarly, the displacement along the temperature axis of certain other peaks, *e.g.* in the region 16 to 17.5°C , could be explained on the basis of the formation of different types of interfacial complex having phase changes at temperatures different from those given by the complexes formed with the carrier liquid above.

On this hypothesis, the formation of entirely new peaks, such as occur with the original dye (1) on the introduction of further compounds into the carrier liquid, would indicate the formation of interfacial complexes not originally present with the carrier alone. Certain peaks, for example that due to dye 2 at 16.6°C and to dye 3 at 18.1°C , are not changed by phloroglucinol and it would, therefore, seem that the original complexes are not affected by its introduction and the temperature of the phase change therefore remains the same.

Although phloroglucinol exists in two tautomeric forms, hydroxyl and carbonyl, and a peak for each form is given by dye 1, no definite change in the index-temperature curve of any of the other dyes occurs in the region immediately around 16.4°C , the temperature at which dye 1 shows the carbonyl peak.

Presumably, the carbonyl group is too weakly adsorbed at the oil-water interface to compete with the more strongly adsorbed dyes or does not give a suitable complex with them.

The easy suppression of the effect of the carbonyl group is borne out by the fact that compounds of the type



present in the material being chromatographed do not give a carbonyl peak with dye 1 at 16.4°C, although a peak appears at 15.8°C when the carbonyl groups are reduced to hydroxyl and the peak at 16.4°C is given by benzoquinone itself³.

The effects caused by the addition of lauric acid to the carrier liquid, except for dye 2, indicate that there is a general lowering of the index at 15.6 and at 16.9°C, where dye 1 would give peaks due to the carboxyl group.

This is less marked for the azo-dyes, (as compared to the anthroquinoid) and least for dye 2, which is the closest in structure to dye 1.

The picture is clearer in the 18.1 to 18.8°C region in which dye 1 gives marked acid peaks.

Where there was originally a major peak (dye 3) this has been suppressed (*cf.* phloroglucinol). Where there was only a minor peak (dye 2) this has been enhanced and where there was no peak (dyes 4 and 5) peaks have been produced (*cf.* dye 1), although only one peak covering the whole temperature range is produced, and not two as is indicated by the vertical lines showing the peak positions for dye 1.

This is in line with the interfacial complex-transition theory already advanced to explain the effect of phloroglucinol.

A dye would, therefore, seem to require two attributes in order to produce the peak effects; a polar group strongly adsorbed at the oil-water interface

R

(provided by the $\text{—N—CH}_2\text{—CH}_2\text{—OH}$) and the ability to ionise. That is, it must satisfy the conditions put forward by Schulman and Cockbain⁴ as necessary for the formation of stable interfacial complexes, to give condensed films in the presence of oil soluble and water soluble compounds.

The oil soluble compounds are provided by the materials being chromatographed. Some of these may also be water soluble, but in any case the peak-producing dyes are themselves partially water soluble.

Any complication of the dye molecule over and above the simple structure of dye 1, such as the ability to give extra charged species, appears to lead to a more complicated temperature-index relationship, because of the probability of the formation of extra interfacial complexes.

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Outline/unit area chromatography: III. The detection and characterisation of residual polar impurities in refined mineral oils

By **D. W. Poxon**

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Summary

Four samples of solvent refined base oils and one sample of unrefined oil have been examined by outline/unit area chromatography.

This is a method of paper chromatography making use of alterations in the shape of the chromatographic band in the presence of certain surface active dyes, caused by the presence of polar groups in the material being chromatographed, and using a completely standardised development technique which does not require any separation of the polar compounds.

It has been found possible to detect and identify the type of polar compounds present in the oil samples, and to show differences in degree of the purity between the various samples.

Key words

Oils
mineral oil

Processes and methods primarily associated with analysis measurement or testing chromatography

La détection et caractérisation des impuretés polaires résiduelles en huiles minérales

Résumé

On a dosé par chromatographie sur papier, en termes de l'indice profil/aire unitaire, quatre échantillons d'huiles raffinées par solvant, et un échantillon d'huile brute.

Cette méthode de chromatographie où l'on se sert des changements de la forme de la bande chromatographique en présence de certains solvants tensio-actifs provoqués par des groupements polaires qui se présentent dans l'échantillon pour dosage. On utilise une technique de développement tout à fait normalisé que n'exige aucune séparation des composés polaires.

On a trouvé la possibilité de mettre en évidence et d'identifier le type de composés polaires qui se présentent dans les échantillons d'huile et de démontrer les différences dans le niveau de pureté de divers échantillons.

Nachweis und Charakterisierung von Resten Polarer Verunreinigungen in raffinierten Mineralölen

Zusammenfassung

Vier Prüfmuster von mit Lösungsmitteln raffinierten basischen Ölen, sowie eins von unraffiniertem Öl wurden mittels Umriss/Einheit Aereachromatographie geprüft.

Bei dieser Methode der Papierchromatographie macht man von Veränderungen der Form des chromatographischen Bandes in Gegenwart gewisser oberflächenaktiver Farbstoffe Gebrauch. Diese werden durch die Anwesenheit in dem zu prüfenden Material von polaren Gruppen verursacht. Es wird eine völlig genormte Entwicklungstechnik verwandt, bei welcher keinerlei Trennung der polaren Verbindungen nötig ist.

Man fand, dass es möglich war, den Typ der in den Ölmustern enthaltenen Verbindungen zu entdecken und zu identifizieren, und Unterschiede im Reinheitsgrad zwischen den verschiedenen Prüfmustern aufzuzeigen.

Обнаружение и характеристика остаточных полярных примесей в очищенных минеральных маслах

Резюме

Анализируются четыре образца сырых растворено-очищенных масел и один образец неочищенного масла, методом контурно-единичной поверхностной хроматографии. Метод заключается в хроматографировании на бумаге используя изменения формы хроматографической полосы, в присутствии некоторых известных поверхностно-активных красителей, вызванные присутствием полярных групп в хроматографированном материале, применяя полностью стандартизованную технику, при которой не нужна сепарация полярных соединений.

Найдено что возможно обнаружить и опознать тип полярных соединений присутствующих в образцах масла и определить различия в степени чистоты между отдельными образцами.

Introduction

The solvent refining of lubricating oils has as its purpose the removal of polar compounds in order to improve the viscosity characteristics of the oil.

The amount of polar material left in the oil after the refining should be very small and it is, consequently, difficult to detect by normal methods of analysis.

Outline/unit area chromatography is capable of detecting polar compounds at very low concentrations (of the order of 10^{-6} molar) especially when they are present in a non-polar mixture. Each polar group present gives a marked increase in the outline/unit area index (total perimeter of the chromatographic band divided by the area of the band) at a specific temperature for the particular polar group concerned and at a specific concentration when chromatographed in a solvent composed of a mixture of oleic and palmitic acids. (Table I).

Table I
Peak temperatures for various polar groups

Group	Temperature C
COOH ..	15.6 16.9 18.1 18.8
CO ..	16.4
OH ..	15.8 (alcohols, enols, phenols)
Peroxides ..	15.0 — 15.3
Carrier ..	17.6

Thus, three-dimensional diagrams may be constructed with temperature, concentration, and index as the axes, which are specific for any particular mixture of polar compounds.

This has been done for four, solvent refined, base oils containing no additives and one oil sample which has not been subjected to solvent refining.

Experimental

The main details of the method have been described elsewhere^{1,2}. The same system of 11cm filter papers (No. 1 grade), dyed with a mixture of Dispersol Fast Scarlet B (CI Disperse Red 1, 11110) and Waxoline Green G (CI Solvent Green 3, 61565), and cut into eight sectors with development tabs between each section, was used. The chromatographic liquid was petroleum spirit (b.r. 40-60°C), and rigid temperature control was maintained during chromatography.

The samples were dissolved in a mixture of oleic and palmitic acid (the carrier liquid), having a setting point of 18°C.

The range of oil concentrations used was 1 to 8 per cent at 1 per cent intervals, these solutions being applied to the base of each sector of the paper as a continuous band approximately 1mm wide.

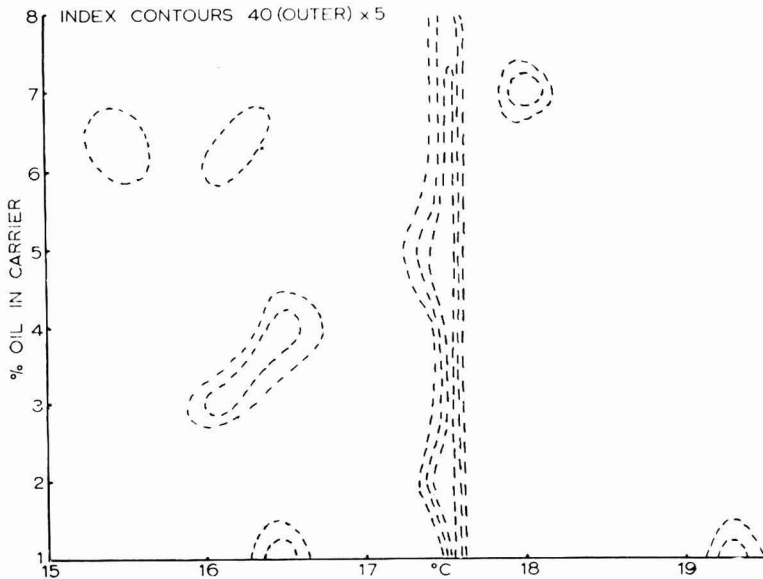


Fig. 1. Lubricating oil, solvent refined. High viscosity index

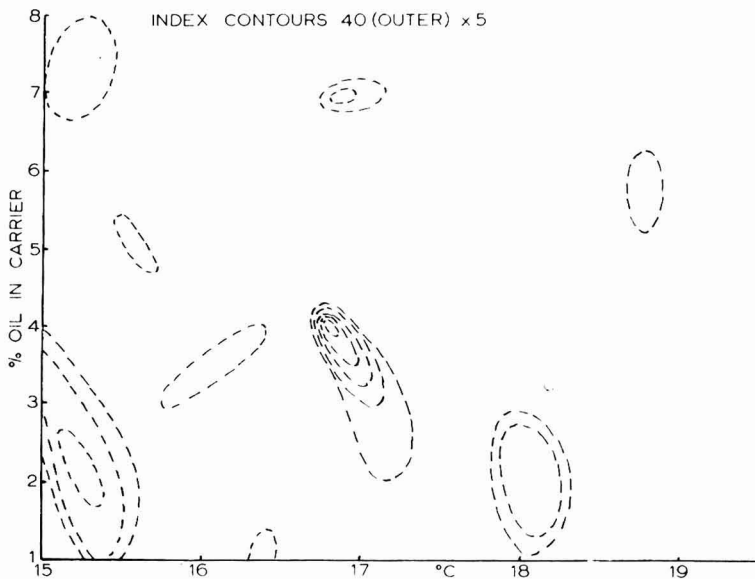


Fig. 2. Lubricating oil, solvent refined. High viscosity index

When the chromatography, which only required a few minutes, was complete, the outline and area of each band was pencilled in, and the necessary measurements and calculations made. Figs. 1 to 5 were then constructed, and these show the results from each oil. Individual points (not shown on the diagrams) were obtained every 0.2 to 0.3°C for each 1 per cent concentration interval.

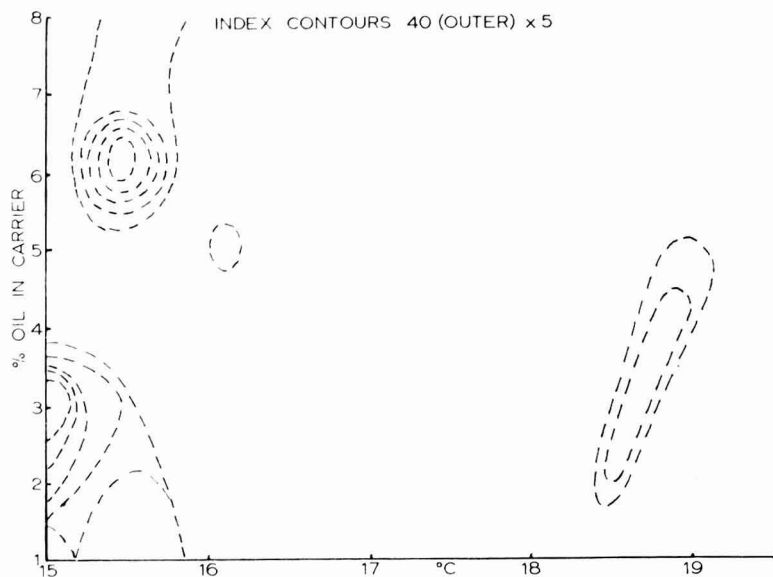


Fig. 3. Lubricating oil, solvent refined. Medium viscosity index

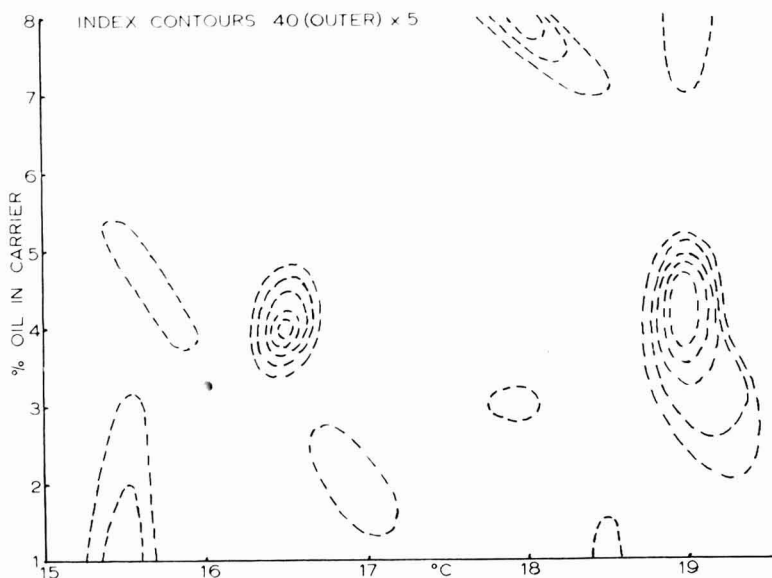


Fig. 4. Lubricating oil, solvent refined. Low viscosity index

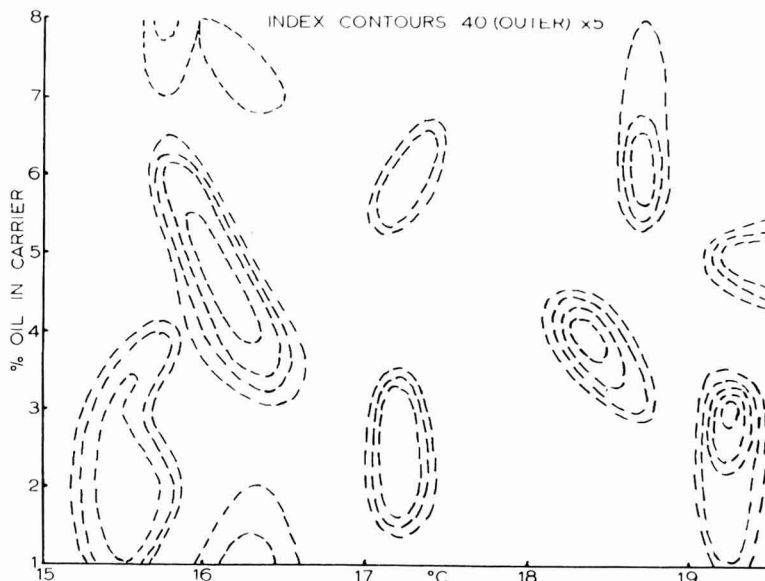


Fig. 5. Lubricating oil. Unrefined residual stock

Results and Discussion

Except for the unrefined sample (Fig. 5), none of the samples contain either a large number or a large quantity of polar compounds.

Figs. 1 and 2 are the diagrams for two samples of oil having a high viscosity index. Ideally, solvent refining should remove all polar material, and for oil no. 1 this is almost the case. The single peak across the diagram at 17.6°C which would be given by the oleic-palmitic acid carrier liquid alone, and which is usually removed or disrupted by the presence of other polar compounds, is still present and scarcely distorted. The compounds present appear to be chiefly ketonic (16.4°C).

Oil no. 2 is not so pure as the above, as is indicated by the disappearance of the carrier peak and the more numerous and higher peaks at other temperatures. Acids and peroxides are the chief impurities here (see Table I).

Fig. 3 shows the results for a medium viscosity index oil. Peroxides and acids are the chief impurities in this case (possibly peroxy-acids) with a trace of ketone.

Sample no. 4 is a low viscosity index oil, the higher and larger peak areas indicating more impurity of a polar type. Again the compounds are ketones and acids.

Finally, the unpurified sample shows a marked increase in the number and quantity of impurities. Ketones and acids again predominate, but there is also evidence of hydroxyl compounds (15.8°C). The method indicates the degree of efficiency of the solvent refining in each case and clearly illustrates the differences in purity of the five samples of lubricating oil.

Conclusions

The outline/unit area index method of chromatography is ideally suited to the detection of polar compounds, at low concentrations, when these are dissolved in a non-polar bulk material, as is the case with polar impurities in lubricating oils.

In the present case the concentrations are such as to be undetectable by orthodox spectroscopic, and most other, methods. In addition, information as to the nature of polar groups present is provided.

Acknowledgments

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The adhesion of organic surface coatings to cadmium plate

By P. Walker

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Summary

The adhesion of a range of organic coating systems to passivated and non-passivated cadmium plate has been measured by the "torque spanner" technique over a period of five years' natural exposure. It has been shown that surface coatings containing vegetable oils or oil residues have poor initial adhesion and lose adhesion rapidly on ageing. This is attributed to attack on the substrate by decomposition products arising from the oils. Chemically cured systems, on the other hand, show good adhesion retention, and the adhesion of an amine cured epoxide and a blocked polyurethane showed no change over the entire period of exposure. The use of a two-pack etch primer may minimise the loss of adhesion but will not prevent it. Chromate passivation of the cadmium surface does nothing to reduce the loss of adhesion and may accelerate it.

Key words

Types and classes of surface
cadmium plated steel

Types and classes of coating
alkyd coating
epoxy ester coating
epoxy finish
urethane finish

Prime pigments and dyes
red iron oxide pigment

Chemically active pigments
zinc chromate

Binders (resins etc)

amide cured epoxy resin
amine cured epoxy resin
chlorinated rubber

L'adhérence de revêtements organiques aux plaques de cadmium

Résumé

L'adhérence d'une gamme de systèmes de revêtements organiques sur plaques de cadmium passivé ou non a-t-elle été déterminée par la technique de "clé de torsion" pendant une période de cinq années d'exposition aux intempéries. On a démontré que des revêtements contenant d'huiles végétales ou leurs restes possèdent une adhérence inférieure au début et aussi ils perdent rapidement d'adhérence lors du vieillissement. D'autre part, les systèmes durcis par ouïl des agents chimiques révèlent de bonne rétention d'adhérence, et d'ailleurs l'adhérence d'une époxyde durcie par une amine ou d'un polyuréthane bloqué ne démontrent pas de changement pendant la période totale de l'exposition. L'utilisation d'un "etch-primer" à deux emballages peut minimiser la perte d'adhérence, quoiqu'elle ne puisse pas l'empêcher. La passivation de la surface du cadmium au moyens de chromate n'est pas effective pour réduire la perte d'adhérence, et en outre peut-elle l'accélérer.

Die Haftung organischer Anstrichmittel auf Cadmierung

Zusammenfassung

Die natürliche Bewitterung von fünfjähriger Dauer diente zur Messung mittels der "Torsionsschraubenschlüssel"—Technik der Haftung einer Reihe organischer Anstrichsysteme auf passiviertem und nicht passiviertem, kadmierten Blech. Dabei zeigte sich, dass Anstrichmittel, die pflanzliche öle oder Ölreste anhalten, von Anfang an schlecht haften

und beim Altern sehr schnell ihre Haftung verlieren. Dies wird darauf zurückgeführt, dass Zersetzungsprodukte der Öle die Substrate angreifen. Dagegen zeigen chemisch gehärtete Systeme gute und bleibende Haftfestigkeit, und die Haftung eines mit Amin vernetzten Epoxides und eines blockierten Polyurethanes blieb während der gesamten Bewitterungsperiode unverändert. Die Anwendung eines Zweikomponenten-Washprimers kann zwar den Haftungsverlust verringern, aber nicht verhüten. Chromatpassivierung der Kadmiumoberfläche hilft in keiner Weise und kann sogar den Verlust der Haftung beschleunigen.

Адгезия органических поверхностных покрытий на пластинах кадмия

Резюме

Измерялось сцепление ряда органических поверхностных покрытий на пассивированных и непассивированных плитах кадмия, с использованием техники применения «ключа крутящего момента», в течение пятилетнего периода естественного обнажения. Показано что поверхностные покрытия содержащие растительные масла, или масляные остатки, обладают слабой начальной адгезией и быстро теряют эту адгезию при старении. Это приписывается коррозии субстрата продуктами разложения масел. Химически осушенные системы, с другой стороны, хорошо сохраняют сцепление, а сцепление эпоксида высушенного аминами и блокированного полиуретана не обнаружило никаких изменений за весь период обнажения. Применение двойной травильной грунтовки может снизить потерю в адгезии, но не предотвратит ее. Хроматное пассивирование кадмиевой поверхности не снижает потерю в адгезии и даже может ее ускорить.

Introduction

In a recent paper¹ the author described work carried out to assess the potential difficulties in using organic coatings in proximity to cadmium plated steel, and to determine to what extent coatings could be used to prevent organic vapour attack on cadmium. A further part of the paper described preliminary work on the adhesion of organic coatings to cadmium plate and the effect of exterior exposure on this property. The results quoted were for an exposure period of 15 months. That exposure series has now been terminated after five years, and is reported in detail in the present paper.

It is perhaps relevant to restate the reasons for painting cadmium and to outline previous recorded experience of organic coatings on this substrate. Although cadmium plating alone is regarded as an effective method of protecting steel against atmospheric attack, it relies for its efficiency on a "sacrificial" mechanism and is liable to be consumed during its protective life. Further, cadmium metal is particularly prone to attack by organic vapours which may be emitted from wood, paints, adhesives etc. in the vicinity. Because of these defects, and because many manufactured goods which are painted overall contain cadmium plated components, and for aesthetic reasons it is undesirable to leave these unpainted, it may be necessary to apply paint over cadmium plate.

It is generally recognised that cadmium is a difficult metal to paint^{2, 3, 4} in that it is not easy to obtain a good initial adhesion and the adhesion is liable to deteriorate on ageing. In this respect, it is considered to present more of a problem than zinc⁵. It has been suggested^{2,5} that a chromate passivation process will materially improve both initial adhesion, and long term retention of adhesion on ageing. Evidence has already been presented which suggests that these statements are not generally valid.

This present paper demonstrates that the conclusions drawn by the authors from relatively short term exposure trials were generally sound, but that points of detail require some modification.

Choice of paint systems and substrate

A total of ten paint systems was exposed, covering most of the room temperature curing resin systems of commercial significance for the coating of metals. In addition, a one-pack blocked polyurethane stoving paint was included as a control. Thus, the ten paint systems included four drying by an oxidative mechanism, four curing by chemical reaction, one thermosetting system and one drying by solvent evaporation. All the coatings selected were exposed as part of a two or three coat system based on the same resin. The exceptions to this were the four chemically cured systems, which had a common primer. The panels chosen were cadmium plated to DTD 904A and cadmium plated and passivated to the same specification. All panels were plated at the same time.

The complete systems exposed are shown below:

- System 1 Red oxide epoxide ester primer to CS 8005
white epoxide ester undercoat to CS 8006
white epoxide ester finish to CS 8007
- System 2 Red oxide styrenated epoxide ester primer to CS 8072A
white styrenated epoxide ester undercoat to CS 8072A
white styrenated epoxide ester finish to CS 8072A
- System 3 Red oxide/zinc chromate primer to DEF 1035A
white undercoat to DEF 1052A
white finish to DEF 1052A
- System 4 Red oxide/zinc chromate short oil alkyd primer
white undercoat on short oil alkyd
white finish on short oil alkyd
- System 5 Red oxide/zinc chromate polyamide cured epoxide primer
two coats white aromatic isocyanate cured polyurethane finish
- System 6 Black one-pack blocked polyurethane finish to CS 8017
white one-pack blocked polyurethane finish to CS 8017
- System 7 Red oxide/zinc chromate polyamide cured epoxide primer
white amine cured epoxide finish
- System 8 Red oxide/zinc chromate polyamide cured epoxide primer
white amine adduct cured epoxide finish
- System 9 Red oxide/zinc chromate polyamide cured epoxide primer
white polyamide cured epoxide finish
- System 10 Proprietary chlorinated rubber primer
cream proprietary chlorinated rubber finish

The formulations of the non-specification paints are given in Appendix I.

Method of test

All panels were painted by spray application to the dry film weight recommended in the specification, or at such a wet film thickness as to give a 1-1.5 mil coat. Panels were allowed to cure at room temperature and humidity (60°F, 40 per cent

RH) for 14 days before test. The panels for the complete programme were exposed in December 1963 at Aldermaston, Berkshire, at an angle of 45° facing due south. The method of assessing the adhesion of the coatings was the "torque spanner" test⁶. In this test, cylindrical steel specimens are bonded to the test panel after gently abrading the test face with a nylon pad. The adhesive used in the present tests was a two-pack epoxide system, "Araldite MY 753" and "Hardener HY 951" at a ratio of 10:1 by weight. The mixture was allowed to age for 30 minutes before use. After allowing the adhesive to cure for 24-36 hours, the test specimens were twisted off using a recording torque spanner coupled to a supporting table through a universal joint. The failing load for each specimen was recorded, together with the site of failure and an estimate of the percentage area of coating removed: this was obtained by a careful examination of both the panel and the cylindrical specimen. Tests were carried out in triplicate.

To carry out the adhesion test, the panels were removed after fixed periods of time and brought into the laboratory to recover under standard conditions. The adhesion measured was, therefore, the actual retained adhesion and not subject to variations caused by weather conditions⁷. On completion of the test, the damaged areas were made good with a high quality quick drying primer and the panels replaced in the exposure racks.

Results

Reference to Table 1 will show that; none of the oxidation cured coatings had good adhesion characteristics on non-passivated cadmium, in general the original adhesion values were low for the type of coating used, and all the coatings sheared cleanly from the substrate. The short oil epoxide ester system, although having an adhesion value more or less equivalent to that which would be expected on other metals, also failed by loss of adhesion from the metal. On other metals, such as mild steel and aluminium, most failures would occur in the cohesion of one or more coats of the system.

The chemically cured systems gave uniformly good adhesion values initially and none of the coatings failed at the maximum shear value which could be applied with the 120lb in torque spanner used. The chlorinated rubber paint showed cohesive failure at an acceptable level.

On exterior exposure, the epoxide ester and styrenated epoxide ester systems showed rapid loss of adhesion, followed by complete failure by flaking after two to three years' exposure. The two alkyd paint systems behaved quite differently, with the long oil alkyd showing only a slight loss of adhesion after five years. The short oil alkyd system showed a progressive loss over the same period, dropping to approximately half the original value. As a group, the oxidation cured systems behaved badly.

The chemically cured systems were again uniformly good and showed no detectable change in adhesion, failing in cohesion of the finishing coat at relatively high values. This cohesive failure was in the surface layers of the paints rather than a deep seated failure, and indicates that the paints had chalked. The values for the one-pack blocked polyurethane stoving paint were excellent.

The chlorinated rubber paint exhibited variable behaviour, with a tendency to show adhesion failures from the substrate. The site of failure in the final

determination was cohesive at a value approximately 100 psi below that of the original, so it is possible that some loss of adhesion occurred but was masked by the low cohesive strength of the system.

In general then, the behaviour of the various paint systems followed closely the pattern which emerged from the work on corrosion of cadmium by vapours from organic coatings¹. This work showed that the oxidation cured systems showed marked attack, with the chemically cured and chlorinated rubber systems showing very little. Thus, the coatings can be divided into reactive and non-reactive types. The reactive types show loss of adhesion, the non-reactive types do not.

Table 2 shows the adhesion values recorded on the passivated cadmium panels. Comparison of the original values with those in Table 1 shows that there is no significant difference and, for all practical purposes, the two sets of values are identical. Thus, there is no gain in adhesion resulting from the passivation process.

On exterior exposure, the adhesion of all the oxidation cured systems fell rapidly and dramatically, with the styrenated epoxy ester failing after only one year's exposure. The long oil alkyd system failed after eighteen months and the short oil alkyd after two years. The short oil epoxide ester paint system gave the longest life in this group, failing between two and a half and three years. In the case of the non-passivated panels, all these systems lasted a minimum of two and a half to three years, and the short oil alkyd system was still adherent after five years' exposure.

Of the chemically cured systems, two, namely the aromatic isocyanate cured polyurethane and the polyamide cured epoxide, showed pronounced loss of adhesion on ageing. The same systems on non-passivated cadmium did not show loss of adhesion. The three remaining chemically cured systems failed in cohesion, at values very close to those measured on the non-passivated panels. The chlorinated rubber paint system also failed in cohesion.

Thus, rather than improving the adhesion and retention of adhesion on ageing, passivation appeared to result in a marked tendency for the adhesion to be lost more rapidly.

Effect of etch primers on the retention of adhesion

In an attempt to insulate the surface coatings from the substrate and thus avoid the loss of adhesion of some systems apparent on both the passivated and non-passivated cadmium, a series of one- and two-pack etch primers were investigated. The primers chosen were: an unmodified WPI type specified in CS2626F (now DEF 1408), two proprietary twin-pack primers stated to be modified with phenolic resin, and two single-pack types of unknown composition. All the etch primers were applied by spray to the dry film weight recommended in the specification or by the manufacturer, and allowed to dry for 24 hours before application of the complete system.

Comparison of the original adhesion values in Table 3 with those obtained on the passivated and non-passivated cadmium panels shows that in almost all cases the adhesion of the oxidation cured materials has been greatly improved by the use of the DEF 1408 etch primer. In all cases, except that of the short

oil epoxide ester, the adhesion values recorded were appreciably higher and the failures were in cohesion of the finish. The values for the short oil epoxide ester were lower, but again the failure was cohesive. The chemically cured systems fell into distinct groups, the one-pack blocked polyurethane and the adduct and polyamide cured epoxides, which gave identical results to those obtained from the non-etch primed panels, and the two-pack polyurethane and amine cured epoxide which failed in adhesion from the etch primer. This defect did not occur on the non etch primed panels. The chlorinated rubber paint values were very similar on all the panels. There did not appear to be any difference in the passivated and non-passivated panels.

On exterior exposure, considerable differences were apparent. None of the oxidation cured systems failed during the five year exposure period when applied over the etch primer, although the adhesion of the short oil epoxide ester system on the passivated panel was very low. This represented a distinct improvement over that of the same paints on the non etch primed panels, where only one panel out of the eight was still intact after five years.

The two-pack polyurethane paint had better adhesion on the etch primed panel than on the passivated panel, but inferior adhesion to that on the non-passivated panel. The one-pack blocked polyurethane and the amine cured epoxide systems were definitely inferior on the etch primed panels.

Both the adduct cured and the polyamide cured epoxide systems failed in cohesion of the finish at approximately the same values on the etch primed panels as on the passivated and non-passivated panels. The chlorinated rubber paint appeared to be stronger on the non-passivated panels than on the passivated and etch primed panels but this is more a function of the efficiency of removal of the chalk layer than a real indication of difference in performance.

Table 4 shows the values recorded on panels primed with a proprietary two-pack etch primer stated to be modified with a phenolic resin. The values and observations on the sites of failure are very similar to those recorded on the specification etch primer except that the amine cured epoxide did not show any sign of adhesion failure from the etch primer in the early stages of exposure. Almost identical results were recorded on a second proprietary two-pack etch primer which has, therefore, not been reported in detail.

Two proprietary single-pack etch primers of the pre-reacted type, containing phosphoric acid, were also examined and the results obtained are shown in Tables 5 and 6. In general, the values and sites of failure for primer 1 are similar to those obtained from the specification and proprietary two-pack etch primers in that all the oxidation cured systems showed a loss of adhesion on ageing while the chemically cured systems did not; all the sites of failure recorded after five years' exposure were in cohesion of the finish.

Proprietary one-pack etch primer 2, however, was very much inferior to 1 and three of the paint systems failed in service after three years on the passivated panels, and of these, one failed on the non-passivated surface also. Further, the two-pack polyurethane and the amine cured epoxide also showed pronounced adhesion failure on the passivated panels and the former also showed this failure on the non-passivated panels. This primer gave rise to a performance which was inferior to that of the paints applied to the non-passivated cadmium without benefit of an etch primer.

Change of adhesion on exterior exposure on

Paint system	Original		6 months		1 year		Adhesion site
	Adhesion psi	Site of failure	Adhesion psi	Site of failure	Adhesion psi	Site of failure	
Short oil epoxide ester ..	3970	100% A/S	3550	100% A/S	3200	100% A/S	X
Styrenated epoxide ester	2710	100% A/S	1920	100% A/S	1470	100% A/S	X
Long oil alkyd	2310	100% A/S	2400	100% A/S	2280	100% A/S	X
Short oil alkyd	2210	100% A/S	1620	100% A/S	1880	100% A/S	X
Two-pack polyurethane ..	4800+	No failure	4400+	No failure	4800+	No failure	0
Single-pack blocked polyurethane	4800+	No failure	4800+	No failure	4800+	No failure	c
Amine cured epoxide ..	4800+	No failure	4800+	No failure	4400+	No failure	c
Adduct cured epoxide ..	4800+	No failure	4800+	No failure	4400+	No failure	c
Polyamide cured epoxide	4800+	No failure	4800+	No failure	4400+	No failure	X
Chlorinated rubber ..	3160	100% C/F	3000	100% C/F	2880	100% A/S	X

A/S — Adhesion from site

Non-passivated

½ years	2 years		3 years		4 years		5 years	
Site of failure	Adhesion psi	Site of failure	Adhesion psi	Site of failure	Adhesion psi	Site of failure	Adhesion psi	Site of failure
100% A/S	1400	100% A/S	System failed		—	—	—	—
100% A/S	700	100% A/S	System failed		—	—	—	—
100% A/S	1900	100% A/S	1980	100% A/S	2160	100% A/S	System failed	
100% A/S	1600	100% A/S	2080	100% A/S	1400	100% A/S	1200	100% A/S
0% C/F	4800	100% C/F	4800	100% C/F	4000	100% C/F	4200	100% C/F
to failure	4800+	No failure	4800	40% C/F	4280	100% C/F	4920	60% C/F
to failure	4800+	No failure	4080	100% C/F	4600	No failure	4440	100% C/F
to failure	4600	100% C/F	4760	100% C/F	4760	100% C/F	3880	100% C/F
100% A/S	4800+	No failure	4660	100% C/F	4340	100% C/F	3960	100% C/F
100% A/S	3360	100% A/S	3540	100% A/S	3860	50% A/S 50% C/F	2160	100% C/F

F — Cohesional failure in finish

Table
Change of adhesion on exterior exposure on cadmium

Paint system	Original		6 months		1 year		1½ year
	Adhesion psi	Site of failure	Adhesion psi	Site of failure	Adhesion psi	Site of failure	Adhesion psi
Short oil epoxide ester ..	3800	100% A/S	2920	100% A/S	2000	100% A/S	1800 10
Styrenated epoxide ester	2620	100% A/S	930	100% A/S	System failed		—
Long oil alkyd	2090	100% A/S	470	100% A/S	200	100% A/S	System fai
Short oil alkyd	1970	100% A/S	1230	100% A/S	200	100% A/S	200 10
Two-pack polyurethane	4800+	No failure	4320	20% A/S	3350	100% A/S	2200 10
Single-pack blocked polyurethane	4800+	No failure	4800+	No failure	4800+	No failure	4600 N
Amine cured epoxide ..	4800+	No failure	4800+	No failure	4400+	No failure	4800+ N
Adduct cured epoxide ..	4800+	No failure	4800+	No failure	4400+	No failure	4800+ N
Polyamide cured epoxide	4800+	No failure	4800+	No failure	4500+	No failure	4800+ N
Chlorinated rubber ..	3300	100% C/F	3350	100% C/F	3400	100% A/S	2400 10

A/S — Adhesion from substrate/F

Table 2
 (b) Passivated

Years	1½ years	2 years		3 years		4 years		5 years	
Adhesion site	Site of failure	Adhesion psi	Site of failure	Adhesion psi	Site of failure	Adhesion psi	Site of failure	Adhesion psi	Site of failure
100	100% A/S	920	100% A/S	System failed		—	—	—	—
—	—	—	—	—	—	—	—	—	—
System	failed	System failed		—	—	—	—	—	—
100	100% A/S	System failed		—	—	—	—	—	—
100	100% A/S	2540	100% A/S	3100	100% A/S	2480	100% A/S	1520	100% A/S
100	No failure	Not tested		4760	100% C/F	4760	10% C/F	4960	40% C/F
100	No failure	4500 +	No failure	4640	100% C/F	4720	10% C/F	4640	50% C/F
100	No failure	4360	30% C/F	4840	100% C/F	4800	10% C/F	3620	100% C/F
100	No failure	4660	100% C/F	4360	100% C/F	4160	100% C/F	2240	50% A/S 50% C/F
100	100% A/S	3120	100% A/S	2880	100% A/S	3540	40% A/S 60% C/F	1860	100% C/F

— C/F — Cohesional failure in finish

Change of adhesion on exterior exposure on cadmium

Paint system	Substrate	Original		1 year	
		Adhesion psi	Site of failure	Adhesion psi	Site of failure
Short oil epoxide ester ..	Passivated	3110	100% C/F	3400	*
Short oil epoxide ester ..	Non-passivated	3230	100% C/F	4640	40% A/S
Styrenated epoxide ester ..	Passivated	3530	100% C/F	4500	100% C/F
Styrenated epoxide ester ..	Non-passivated	3610	100% C/F	4400	10% A/EP 90% C/F
Long oil alkyd	Passivated	3720	100% C/F	4200	100% C/F
Long oil alkyd	Non-passivated	3750	100% C/F	4360	100% C/F
Short oil alkyd	Passivated	3820	100% C/F	3540	100% C/F
Short oil alkyd	Non-passivated	3950	100% C/F	3580	60% C/F
Two-pack polyurethane ..	Passivated	4550	30% A/EP	4680	30% A/EP
Two-pack polyurethane ..	Non-passivated	4600	30% A/EP	4500	20% A/EP
One-pack blocked polyurethane	Passivated	4800+	No failure	4700+	No failure
One-pack blocked polyurethane	Non-passivated	4800+	No failure	4700+	No failure
Amine cured epoxide	Passivated	3090	20% A/EP	2900	30% A/S
Amine cured epoxide	Non-passivated	3230	30% A/EP	3400	20% A/EP
Adduct cured epoxide	Passivated	4800+	No failure	4800+	No failure
Adduct cured epoxide	Non-passivated	4800+	No failure	4800+	No failure
Polyamide cured epoxide ..	Passivated	4800+	No failure	4800+	No failure
Polyamide cured epoxide ..	Non-passivated	4760+	No failure	4800+	No failure
Chlorinated rubber	Passivated	3400	100% C/F	3760	100% C/F
Chlorinated rubber	Non-passivated	3600	100% C/F	3920	100% C/F

* — Passivation pulled away from the cadmium
C/F — Cohesional failure in finish
A/EP — Adhesional failure from etch primer

(Two-pack etch primer to DEF 1408)

2 years		3 years		4 years		5 years	
Adhesion psi	Site of failure	Adhesion psi	Site of failure	Adhesion psi	Site of failure	Adhesion psi	Site of failure
3160	*	2680	100% A/S	1900	100% A/S	400	100% A/S
4400	100% A/S	3460	100% A/S	3080	100% A/S	2000	100% A/S
4160	100% C/F	4100	100% C/F	3600	5% A/S 95% C/F	1640	70% A/S 30% C/F
4040	10% A/S 90% C/F	4320	50% A/S 50% A/S	2960	10% A/S 90% C/F	2280	60% A/S
4180	100% C/F 90% C/F	3800	100% C/F	3680	60% A/S 40% C/F	3480	100% C/F
4020	10% C/EP 90% C/F	3920	100% C/F	4120	60% A/S 40% C/F	3200	100% C/F
4640	100% C/F	4700	100% C/F	4340	30% A/S 70% C/F	3400	100% C/F
4200	10% C/EP 90% C/F	4000	100% C/F	4800	60% A/S 40% C/EP	2880	80% A/S
4040	100% C/F	4800	100% C/F	3520	100% A/S	3560	100% A/S
4760	100% C/F	4300	100% A/S	3480	100% A/S	3240	100% A/S
4800+	No failure	4800	10% C/F	4200	10% C/F	4040	40% A/S 60% C/F
4760+	No failure	4880	10% C/F	3920	10% A/S 10% C/F	3400	10% A/S 90% C/F
3640	100% A/S	3980	100% A/S	3980	100% A/S	3860	100% A/S
4060	100% C/F	4240	100% C/F	4760	100% A/S	4100	100% C/EP
4720	50% C/F	4700	100% A/S	4260	100% C/F	3540	100% C/F
4660	50% C/F	4180	30% A/S	3980	100% C/F	3710	100% C/F
4000	20% C/F	4040	30% C/F	4600	100% C/F	4000	100% C/F
4520	10% A/S 30% C/F	4640	30% C/F	4500	100% C/F	4100	100% C/F
3640	100% C/F	3880	100% C/F	4200	100% C/F	1600	100% C/F
3840	100% C/F	3540	100% C/F	4600	100% C/F	1800	100% C/F

A/S — Adhesion failure from substrate

C/EP — Cohesional failure in etch primer

Table 4
Change of adhesion on exterior exposure on cadmium

Paint system	Substrate	Original		1 year	
		Adhesion psi	Site of failure	Adhesion psi	Site of failure
Short oil epoxide ester	Passivated	3900	100% C/F	4060	30% C/F
Short oil epoxide ester	Non-passivated	4030	100% C/F	4000	30% C/F
Styrenated epoxide ester	Passivated	3950	100% C/F	4000	100% A/S
Styrenated epoxide ester	Non-passivated	4100	60% C/F	3800	30% A/S
Long oil alkyd	Passivated	4210	70% C/F	4320	60% C/F
Long oil alkyd	Non-passivated	4160	100% C/F	4480	60% C/F
Short oil alkyd	Passivated	4150	50% C/EP	4480	30% C/F
Short oil alkyd	Non-passivated	4250	70% C/EP	4440	30% C/F
Two-pack polyurethane	Passivated	4650	20% C/F	4760+	No failure
Two-pack polyurethane	Non-passivated	4750	20% C/F	4800+	No failure
One-pack blocked polyurethane	Passivated	4800+	No failure	4800+	No failure
One-pack blocked polyurethane	Non-passivated	4800+	No failure	4800+	No failure
Amine cured epoxide	Passivated	4600+	No failure	4800+	No failure
Amine cured epoxide	Non-passivated	4720+	No failure	4800+	No failure
Adduct cured epoxide	Passivated	4800+	No failure	4800+	No failure
Adduct cured epoxide	Non-passivated	4700+	No failure	4800+	No failure
Polyamide cured epoxide	Passivated	4700+	No failure	4720	20% C/F
Polyamide cured epoxide	Non-passivated	4800+	No failure	4480	60% C/F
Chlorinated rubber	Passivated	3420	100% C/F	1280	60% A/EP
Chlorinated rubber	Non-passivated	3510	80% C/F	1240	60% A/EP

A/S — Adhesion from substrate
 C/F — Cohesional failure in finish
 C/EP — Cohesional failure in etch primer

Table 4
 Proprietary two-pack etch primer—phenolic modified

2 years		3 years		4 years		5 years	
Adhesion psi	Site of failure	Adhesion psi	Site of failure	Adhesion psi	Site of failure	Adhesion psi	Site of failure
3900	60% A/S	3800	100% A/S	2840	100% A/S	1980	100% A/S
4500	60% A/S	3720	100% A/S	3060	100% A/S	2480	100% A/S
4660	100% A/S	4800	100% C/F	2000	100% A/S	840	100% A/S
3620	60% A/S	3520	60% A/S	2400	100% A/S	1320	100% A/S
3680	10% C/EP 60% A/S	2840	60% C/EP	2840	60% A/S 40% C/EP	1400	100% A/S
4040	20% C/EP 30% A/S	3660	100% A/S	3360	100% A/S	1200	100% A/S
3580	60% C/F	3740	100% A/S	4040	90% A/S	2920	100% A/S
4240	60% C/F	4720	30% C/P	5160	100% A/S	3060	100% A/S
4800+	No failure	4700	100% C/F	3760	100% A/S	3520	100% A/S
4800+	No failure	4500	60% C/F	4140	40% A/S 60% C/F	2900	100% A/S
4800+	No failure	4720	30% C/F	4560	No failure	4600	60% C/F
4800+	No failure	4760	20% C/F	4860	10% C/F	4800	100% C/F
3800	60% A/S	3840	60% A/S	3800	60% A/S	3060	60% A/S 40% C/F
4650	40% C/F	4760	30% C/P	4240	30% A/S	4120	20% A/S 40% C/EP 50% C/F
4660	30% C/F	4300	100% C/F	4340	100% C/F	3810	100% C/F
4530	100% C/F	4140	60% C/F	4560	100% C/F	3580	100% C/F
4280	60% C/F	4200	100% C/F	4200	100% C/F	4240	100% C/F
4640	100% C/F	4680	100% C/F	4320	100% C/F	3960	100% C/F
2200	60% A/EP	2260	100% A/S	2800	50% A/EP 50% C/F	1520	100% C/F
2380	100% A/EP	2760	100% A/S	4080	100% C/F	1910	100% C/F

A/EP — Adhesion from etch primer

C/P — Cohesional failure in primer

Table
Change of adhesion on exterior exposure on cadmium

Paint system	Substrate	Original		1 year		Reference
		Adhesion psi	Site of failure	Adhesion psi	Site of failure	
Short oil epoxide.. ..	Passivated	4400	100% A/EP	4700	60% A/S	480
Short oil epoxide.. ..	Non-passivated	3820	100% A/EP	4500	100% A/S	496
Styrenated epoxide ester ..	Passivated	3330	100% A/EP	2920	100% A/S	260
Styrenated epoxide ester ..	Non-passivated	3410	100% A/EP	2980	100% A/S	244
Long oil alkyd	Passivated	3630	100% C/F	4280	100% A/S	322
Long oil alkyd	Non-passivated	3700	90% C/F	3840	100% A/S	318
Short oil alkyd	Passivated	3090	30% C/EP	3560	100% C/EP	300
Short oil alkyd	Non-passivated	3200	20% C/EP	3640	100% C/EP	322
Two-pack polyurethane ..	Passivated	4800+	No failure	4760+	No failure	440
Two-pack polyurethane ..	Non-passivated	4760+	No failure	4460+	No failure	480
Single-pack blocked polyurethane	Passivated	4800+	No failure	4760+	No failure	460
Single-pack blocked polyurethane	Non-passivated	4800+	No failure	4680+	No failure	480
Amine cured epoxide	Passivated	4410	60% A/EP	4760	100% C/F	460
Amine cured epoxide	Non-passivated	4460	60% A/EP	4340	10% C/F	470
Adduct cured epoxide	Passivated	4200	60% A/EP	3880	10% C/F	470
Adduct cured epoxide	Non-passivated	4120	80% A/EP	3880	10% C/F	480
Polyamide cured epoxide ..	Passivated	4800+	No failure	4800+	No failure	480
Polyamide cured epoxide ..	Non-passivated	4800+	No failure	4800+	No failure	480
Chlorinated rubber	Passivated	1620	80% A/EP	1480	100% A/EP	130
Chlorinated rubber	Non-passivated	1600	50% A/EP	1480	100% A/EP	190

A/S — Adhesion from substrate
C/F — Cohesional failure in finish

Table
 proprietary one-pack etch primer (1)

2 years		3 years		4 years		5 years	
Adhesion psi	Site of failure	Adhesion psi	Site of failure	Adhesion psi	Site of failure	Adhesion psi	Site of failure
4800	100% A/S	2080	100% A/S	2000	100% A/S	2160	100% A/S
4960	100% A/S	3480	100% A/S	3740	100% A/S	1480	100% A/S
2600	100% A/S	3100	100% A/S	2800	100% A/S	1300	100% A/S
2440	100% A/S	2960	100% A/S	2680	100% A/S	2320	80% A/S
3220	100% A/S	3360	100% A/S	3200	100% A/S	1920	100% A/S
3180	100% A/S	3220	90% A/S	3320	100% A/S	1040	100% A/S
3000	30% A/S	3180	60% A/S	2960	100% A/S	1700	100% A/S
3240	20% A/S	3600	80% A/S	400	100% A/S	920	100% A/S
4460+	30% A/S	4500	60% A/S	4340	10% A/S 90% C/F	2480	100% C/F
4800+	No failure	4800	50% C/F	3800	20% A/S 80% C/F	2520	100% C/F
4600+	No failure	4760	40% A/S	4000	20% C/F	3500	30% C/F
4800+	No failure	4600	60% A/S	4140	10% C/F	3640	40% C/F
4640	30% A/S	4600	60% A/S	4100	40% A/S	3400	100% C/F
4760	No failure	4800	100% C/F	4800+	No failure	3520	80% C/F
4720	30% C/F	4760	100% C/F	4240	100% C/F	3500	100% C/F
4800	50% C/F	4640	100% C/F	4560	100% C/F	3680	100% C/F
4840	10% A/S 60% C/F	4440	30% A/S	4200	50% A/S 50% C/F	4140	100% C/F
4820	10% A/S 30% C/F	4360	20% C/F	5240	100% A/S	4340	100% C/F
1380	100% A/EP	1760	50% A/EP	2960	100% A/EP	1700	100% C/F
1940	100% A/EP	1900	100% A/EP	2800	60% A/EP 40% C/F	1820	100% C/F

C/EP — Cohesional failure in etch primer

A/EP — Adhesion from etch primer

Change of adhesion on exterior exposure on copper

Paint system	Substrate	Original		1 year	
		Adhesion psi	Site of failure	Adhesion psi	Site of failure
Short oil epoxide	Passivated	3800	100% A/S	3200	100% A/S
Short oil epoxide	Non-passivated	3930	60% A/S	3710	100% A/S
Styrenated epoxide ester . .	Passivated	3930	50% C/F	900	100% A/S
Styrenated epoxide ester . .	Non-passivated	3980	80% C/F	2640	100% A/S
Long oil alkyd	Passivated	3560	100% C/F	3580	100% A/S
Long oil alkyd	Non-passivated	3630	100% C/F	3920	100% A/S
Short oil alkyd	Passivated	3930	100% C/F	3660	60% A/S
Short oil alkyd	Non-passivated	4020	80% C/F	4240	60% A/S
Two-pack polyurethane . . .	Passivated	4650+	No failure	4200+	No failure
Two-pack polyurethane . . .	Non-passivated	4720+	No failure	4300+	No failure
One-pack blocked polyurethane	Passivated	4800+	No failure	4800+	No failure
One-pack blocked polyurethane	Non-passivated	4800+	No failure	4800+	No failure
Amine cured epoxide	Passivated	4800+	No failure	3700	20% A/S
Amine cured epoxide	Non-passivated	4720+	No failure	4200	10% A/S
Adduct cured epoxide	Passivated	4600	20% A/S	4490	30% A/S
Adduct cured epoxide	Non-passivated	4800+	No failure	4800+	No failure
Polyamide cured epoxide . . .	Passivated	4800+	No failure	4760	No failure
Polyamide cured epoxide . . .	Non-passivated	4800+	No failure	4720	No failure
Chlorinated rubber	Passivated	2900	60% A/EP	1900	100% A/S
Chlorinated rubber	Non-passivated	2710	80% A/EP	1800	100% A/S

A/S — Adhesion failure from substrate
 C/F — Cohesional failure in finish

To
admiralty one-pack etch primer (2)

of re	2 years		3 years		4 years		5 years	
	Adhesion psi	Site of failure	Adhesion psi	Site of failure	Adhesion psi	Site of failure	Adhesion psi	Site of failure
A/S	1000	100% A/S	System Failed		—	—	—	—
A/S	880	100% A/S	System Failed		—	—	—	—
A/S	500	100% A/S	System Failed		—	—	—	—
A/S	2110	100% A/S	2200	100% A/S	2200	100% A/S	1900	60% A/S
A/S	5200	100% A/S	System Failed		—	—	—	—
A/S	1860	100% A/S	3100	100% A/S	3240	100% A/S	1100	100% A/S
A/S	3200	100% A/S	2960	100% A/S	2740	100% A/S	2040	100% A/S
A/S	4440	30% A/S 50% C/EP	4060	100% A/S	4880	100% A/S	3240	100% A/S
ure	4080	10% C/EP	3800	100% A/S	3720	100% A/S	3680	100% A/S
ure	3920	20% C/EP	4000	60% A/S	3840	50% A/S 50% A/S	3380	100% A/S
ure	4800+	No failure	4800+	No failure	4860+	No failure	5000	100% C/F
ure	4800+	No failure	4600+	No failure	4920+	No failure	5000	100% C/F
A/S	3420	10% A/S	3180	60% A/S	2840	100% A/S	2400	100% A/S
A/S	2880	30% A/S 10% C/EP	4240	100% A/S	4200	100% A/S	4000	100% C/EP
A/S	4080	60% A/S	4140	100% A/S	4130	60% C/F	3600	100% C/F
ure	3770	30% A/S	4060	100% C/F	4160	100% C/F	3630	80% C/F
ure	4220	60% C/F	4800	100% C/F	4560	10% C/F	4000	100% C/F
ure	4500	60% C/F	4760	100% C/F	4400	100% C/F	4240	100% C/F
A/S	1280	60% A/S	1260	100% A/S	1440	100% A/S	1200	100% A/S
A/S	1240	60% A/S	1760	100% A/S	1760	100% A/S	1480	10% A/S 90% A/S

ustr
sh A/EP — Adhesion from etch primer
C/EP — Cohesional failure in etch primer

Discussion

The exposure programme has shown that surface coatings which cure by an oxidative mechanism are likely to have low initial adhesion to cadmium plate, and this initially poor adhesion will deteriorate on external exposure to such an extent that complete loss of adhesion will occur. Contrary to published data, a chromate passivation of the cadmium surface does nothing to improve this poor initial adhesion, and fails to prevent loss of adhesion on exposure. In fact, from the evidence presented in this paper, there is a strong indication that the passivation treatment is positively harmful to the long term retention of adhesion, causing early failure of most of the oxidation cured systems.

It is not difficult to see why coatings of this type should lose adhesion on cadmium, as aliphatic acids and aldehydes are produced within the film during the cure and subsequent life of the coating. It is well documented that cadmium plate is attacked by both organic acids⁸ and aldehydes⁹. These materials diffuse through the film and react with the substrate to form the appropriate cadmium salt, which would appear to be of low cohesive strength. Further, cadmium formate, one of the principal products formed, is very soluble in water and the presence of a water soluble layer at the paint-metal interface must have an adverse effect on the adhesion of the coating system. In this respect then, coatings which cure by atmospheric oxidation must be regarded as reactive towards cadmium. This reactivity leads to loss of adhesion, a parallel behaviour with that of zinc.

The chemically cured systems all had good adhesion to both passivated and non-passivated cadmium plate, when tested initially, and the amine and amine adduct cured epoxides maintained this good adhesion over the complete period of exposure. The two-pack polyurethane and the polyamide cured epoxide, however, showed some loss of adhesion on ageing on the passivated cadmium surface, a defect absent on the non-passivated cadmium panels. The stoved one-pack polyurethane paint showed no loss of adhesion on either substrate. This coating, together with the amine and amine adduct cured epoxide coatings, may be regarded as completely unreactive towards cadmium.

The chlorinated rubber coating behaved in a rather erratic manner; when high breaking load values were recorded, adhesion failure from the substrate was observed, and when the failing loads were low, cohesive failure in the finish occurred, suggesting that there was a fine balance between cohesion and adhesion. It did not appear to show a progressive deterioration of adhesion on ageing.

Attempts to improve the initial adhesion and the long term retention of adhesion on exposure by the use of etch primers as insulating layers met with mixed success. Certainly, all the primers used improved the initial adhesion of the oxidation cured systems, but did not entirely prevent the loss of adhesion on ageing of these systems. There was, however, a considerable improvement arising from the use of the two-pack etch primers and none of the systems failed completely over the five year exposure period. The two single-pack etch primers showed considerable variation, and while the better of the two was almost as good as the two-pack etch primers, the other was very much inferior. This suggests strongly that it would be better to use two-pack etch primers should this approach be considered.

Conclusions

It has been abundantly demonstrated that coating systems which contain natural vegetable oils or oil residues are unsuitable for use on cadmium, showing low initial adhesion and rapid and complete loss of adhesion on exposure. A passivated cadmium surface is of no value in preventing this loss and may well accelerate the rate of loss. The loss may be minimised by the use of a two-pack etch primer, but where long term protection is important, a surface coating of the chemically cured type should be used. The preference would be for an amine or amine adduct cured epoxide or a single-pack blocked polyurethane.

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Appendix

Formulations of non-specification paints used

<i>Red oxide/zinc chromate short oil alkyd primer</i>						pbw
Red oxide	42
Zinc chromate	8
Mica	7
Short oil linseed alkyd 35% oil length					..	30
6% Cobalt naphthenate		0.12
24% Lead naphthenate	0.28
Methyl ethyl ketoxime	0.05
Xylene	20
<i>White short oil alkyd undercoat</i>						pbw
Rutile titanium dioxide	23
Barytes	28
Micronised talc	8
Short oil linseed alkyd 35% oil length					..	33
6% Cobalt naphthenate		0.12
24% Lead naphthenate	0.28
Methyl ethyl ketoxime	0.05
Xylene	20

<i>White short oil alkyd finish</i>							pbw
Rutile titanium dioxide	100
Short oil linseed alkyd 35% oil length	250
6% Cobalt naphthenate	1.0
24% Lead naphthenate	2.6
Methyl ethyl ketoxime	0.3
Xylene	190
<i>Red oxide/zinc chromate epoxide primer</i>							pbw
Red oxide	88
Zinc chromate	20
Asbestine	12
Epikote 1007	60
Methyl isobutyl ketone	30
Xylene	30
Versamid 115 ²	50
Methyl isobutyl ketone	18
Cellosolve	24
Toluene	8
<i>White aromatic cured two-pack polyurethane finish</i>							pbw
Rutile titanium dioxide	200
Daltolac 1200 ³	200
10% Solution of $\frac{1}{2}$ second butyrate ⁴ in methyl ethyl ketone	40
Methyl isobutyl ketone	200
Cyclohexanone	50
Suprasec 4175 ³	226
<i>White amine cured epoxide finish</i>							pbw
Rutile titanium dioxide	50
Epikote 1007	50
Methyl ethyl ketone	10
Methyl isobutyl ketone	10
Cellosolve	5
Toluene	25
Hardener HY 951 ⁵	3
<i>n</i> -Butyl alcohol	3.5
Toluene	3.5
<i>White amine adduct cured epoxide finish</i>							pbw
Rutile titanium dioxide	333
Epikote 1007	333
Methyl isobutyl ketone	165
Xylene	167
EDA Adduct 870 ⁶	100
Cellosolve	80
<i>n</i> -Butyl alcohol	20
Toluene	50

<i>White polyamide cured epoxide finish</i>						pbw
	Rutile titanium dioxide	200
•	Epikote 1007	200
	Methyl isobutyl ketone		100
	Xylene	100
	Versamid 115	120
	Methyl isobutyl ketone		54
	Cellosolve	72
	Toluene	24

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- | | | |
|-------------------------|------------------------------|--------------------------------------|
| 1. Shell Chemicals Ltd. | 2. Cray Valley Products Ltd. | 3. Imperial Chemical Industries Ltd. |
| 4. Bayer (UK) Ltd. | 5. Ciba Ltd. | 6. Anchor Chemical Co. Ltd. |

Recent developments in thermally resistant polymers

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Summary

A brief discussion of thermal stability and its evaluation as related to organic polymers is presented. The chemistry and thermal stability of the various high temperature polymers available commercially, or on a limited development scale, have been reviewed, showing the substantial progress that has recently occurred in the field of thermally resistant polymers.

Keywords

Binders-resins etc.
copolymer resin
polymer

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

Développements récents dans le domaine des polymères résistant à la chaleur

Résumé

On discute brièvement la stabilité à la chaleur des polymères organiques et d'ailleurs son évaluation. On a passé en revue la chimie et la stabilité à la chaleur de quelques polymères, destinés à l'usage aux hautes températures, qui sont disponibles actuellement en commerce ou en état de développement. Cette revue démontre le progrès important qui a été mis en évidence tout récemment dans le domaine des polymères résistant à la chaleur.

Neuere Entwicklungen auf dem Gebiete Hitzebeständiger Polymere

Zusammenfassung

Es wird eine kurze Besprechung der Wärmestabilität und deren Bewertung in Bezug auf organische Polymere vorgelegt. Die Chemie und Wärmebeständigkeit der verschiedenen im Handel befindlichen oder in beschränkten Mengen versuchsweise erhältlichen, für hohe Temperaturen bestimmten Polymere wird überblickt. Dabei zeigt sich, dass unlängst auf diesem Gebiete erhebliche Fortschritte gemacht wurden.

Современное развитие теплостойких полимеров

Резюме

Дается краткое обсуждение термической устойчивости и ее оценка по отношению к органическим полимерам. Анализируется химия и тепловая устойчивость различных высокотемпературных полимеров, как коммерчески доступных так и достижимых в ограниченном масштабе, с указанием значительного прогресса достигнутого за последнее время в области теплостойких полимеров.

Introduction

Within the last ten years, great effort has been devoted to the development of thermally stable plastics. As modern technology expands at an ever increasing rate, there is growing need for new materials offering superior performance over those currently available. Conventional plastics have severely limited utility at elevated temperatures. Above 200°C, most conventional plastics fail to perform, either because of softening and loss of mechanical strength, or



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because of overall degradation. The need for more thermally stable materials in the aerospace industry is well known. Advanced design concepts have increased the urgency of this need, and pushed the temperature requirements into even higher regions. Present organic materials research is aimed at providing polymers for extended use in the 200-400°C range.

Thermal stability

It must be noted that the actual concept of thermal stability is quite indefinite, different authors frequently use the term in different senses. Thermal stability as used in this paper will be taken to mean the ability of a polymer to maintain unchanged its useful properties and structure at high temperatures. In a definite sense, thermal stability corresponds to the melting point or softening point of the polymer. In the absence of a softening point, as with the thermosetting polymers, the ultimate thermal stability of a material depends on the strength of the bonds between atoms in the polymeric molecule and the readiness with which the molecules undergo various reactions, primarily those involving degradation. The prolonged action of a high temperature on polymers causes the breakdown, both by the direct action of temperature producing degradation and cross-linking, and the rapidly occurring chemical reactions with oxygen, water and other chemical reagents. All these changes take place rapidly at high temperature and, therefore, the susceptibility of a polymer to undergo them is very important in assessing its practical use.

Heating a polymer to a high temperature can cause the occurrence of two processes, degradation and cross-linking. Degradation is due to the rupture of chain molecules with a progressive decrease in molecular weight of the polymer and the formation of low molecular weight volatiles. Cross-linking involves the formation of bonds between polymer chains, and is accompanied by an increase in molecular weight, which to some extent may tend to improve the physical and mechanical properties of the polymer as well as its thermal stability.

The formation of a large number of cross-linking bonds between separate molecules with the build-up of a rigid network converts the polymer into a hard, brittle, infusible and insoluble product. Most often both these processes take place in polymers at the same time, especially as the primary stage in both processes involves the formation of free radicals. For some time degradation and cross-linking in a polymer can, in effect, balance one another, so that it appears that no change has occurred in the polymer. As the process continues, one of the two reactions begins to predominate, resulting in either rapid softening or a sudden breakdown in the material.

Resistance to oxidation is a most important characteristic of a polymer, since almost all polymers undergo oxidation by air, especially at high temperatures. Reactions caused by the action of atmospheric oxygen on polymers result either in degradation or cross-linking, or both simultaneously, which impairs their physical properties. For example, polyamides, on storage in air at room temperature, gradually oxidise and form peroxides; heating accelerates this process considerably and results in oxidative degradation. The main cause of the appearance of brittleness and loss of strength in a polyamide on being heated in air is oxidative degradation accompanied by hydrolysis.

Polymers containing benzene or heterocyclics of aromatic character show much greater resistance to oxidation than polymers containing aliphatic hydrogen. The hydrogen atoms in aromatic systems are very firmly bound, and are not easily abstracted by free radicals. Thus, it would seem obvious to prepare polymers composed entirely of aromatic rings to obtain the ultimate in thermally stable organic materials. Such polymers, composed entirely of benzene rings, (e.g. poly-*p*-phenylene), have been prepared, and begin to oxidise only at high temperatures (400-600°C); however, their main disadvantage is in processing. These materials are insoluble and intractable. Therefore, a compromise between the ultimate in thermal stability and ease of processing must be reached for these materials to be of commercial use.

Evaluation of thermal stability

An established method of measuring thermal stability of polymers is thermogravimetric analysis (TGA). This method continuously records the weight loss of a sample with increasing temperature in either air or an inert atmosphere. The exposure times usually are short (temperature increases 5-10° per minute) and, therefore, only major changes are recorded. The characteristic inflection temperature in TGA curves indicate the onset of catastrophic thermal degradation. Following the determination of the primary region of thermal instability, isothermal ageing at lower temperatures provides a means for determining the maximum service temperature.

Significant property changes can occur in polymers without a change in weight, for example crystallisation, softening, melting, cross-linking. These changes can be detected with the various calorimetric instruments currently available, e.g. differential thermal analysis (DTA) or differential scanning calorimetry (DSC).

The true thermal stability of a polymer application is most meaningfully defined by the temperature and time over which an end product retains its useful properties, however, the gathering of such data is tedious and time consuming. Manufacturers are finding it increasingly more difficult to supply the requisite data immediately, because the time between the conception of a new high temperature polymer and its commercialisation has been shortened considerably. In addition, the necessary feedback of results in the field is slow. Thus, future development and modifications of new and existing polymers must be directed with somewhat fragmentary evidence, and the user is left with the problem of translating available data into meaningful terms for a given application.

High temperature polymers

A brief summary of the high temperature polymers that have recently received sizeable research and development effort will serve to illustrate the progress that has been made in the development of materials with higher heat resistance. A number of these polymers are commercially available. Others are still in the early stages of development and data are still fragmentary.

The first high temperature polymers were, of course, the fluorocarbons¹ which have been thoroughly covered elsewhere.

Silicones

One very important factor in the improvement and reliability of electrical equipment today is the better insulation materials which have been made available in recent years. Of the many new and improved materials available, silicones offer one of the broadest lines, and their versatility puts them into a wide variety of applications. To cover the subject completely the list would have to include fluids for liquid dielectrics and lubricants, silicone resins for the manufacture of electrical insulation, protective finishes and, more recently, transfer moulding compounds for encapsulating electronic devices. Silicone elastomers are used extensively as insulation for wire and cable, for motor and generator insulation and as moulding compounds to encase electrical equipment completely.

A new product which offers opportunities for the development of new products in a wide variety of industries is a 100 per cent solids, dry, silicone binder resin. This product is formulated with mineral fillers and, when moulded by conventional methods, will produce parts to precision tolerances. Actually, there is a whole class of binder resins available. Some are particularly suitable for use in the fabrication of precision moulded ceramic parts, which may be used at temperatures up to 1,100°C, or higher depending on the choice of filler. Still other resins may be formulated with mineral fillers, asbestos, silica, quartz, metal oxides to provide materials which are useful in the manufacture of electrical components, such as bushings, insulators, connectors and terminal boards. The use of asbestos with these resins suggests their use as a binder for brake linings and clutch facings, where integrity of the finished product at very high temperatures is a basic requirement.

Out of this same technology has come a new type of transfer moulding compound which has higher flow than previous compounds, providing for complete filling of the moulds. Such compounds are non-burning and will not deform during soldering operations. The operating range for devices encapsulated with these materials is from 55°C to above 370°C. These compounds are formulated with fused silica and short glass fibres, and, when properly cured, develop physical properties which protect dielectric devices against shock and vibration, and provide for long operating life. Because they bond well to leads and other components in the device, they provide better moisture protection under humid conditions. In addition, they have low dielectric loss characteristics at high operating temperature and frequencies. These properties make the silicone moulding compounds particularly suitable for encapsulation of semiconductor devices, such as transistors, integrated circuits, rectifiers, resistors and capacitors.

A significant development in the area of silicone elastomers, is a new dimethyl vinyl silicone rubber compound with outstanding physical properties. These physical properties are shown in the following list. It should be noted that the tear strength of this compound is outstanding. In addition, it possesses a high modulus for such a high tear stock. With its low knit time it lends itself readily to extrusion and calendering processes. When properly cured, these new

dimethyl silicone rubber compounds will meet the high strength requirements of many industry and military specifications.

Tensile strength, psi	1300-1500
Elongation, %	500-700
Durometer, Shore A	50
Tear, die B	185-200

The room temperature vulcanising rubbers have traditionally been versatile products developing a high degree of interest for a broad range of applications within almost every industry. Of the most recent developments, two new one-pack silicone rubber compounds have all of the good handling characteristics of earlier one-pack products (RTV), but, in addition, show exceptionally high physical properties, with tensile strengths of 850 psi and elongations to 800 per cent. In addition, the tear strength of 130 pounds per inch gives these compounds a degree of toughness not available previously.

These RTV products are thixotropic, non-sagging, paste compounds. They require no curing agent addition and cure on contact with atmospheric moisture. They show peel strengths greater than 120 pounds per inch, and this, coupled with the 130 pounds per inch tear strength, makes them particularly useful in aerospace applications to produce high strength metal-to-metal bonds over a wide temperature range. The temperature range of the high temperature version of these new one-package compounds is -65 to 315°C . A third one-pack RTV has been designed specifically for high temperature use, where extreme low volatile content is required. Such a product is a red, thixotropic, compound and is ideal for aero-space and similar applications, where out-gassing is of critical importance. Its temperature capability range is -55 to 315°C .

*Polyphenylene oxide*³

General Electric introduced a modified polyphenylene oxide, Noryl, in 1966 as a thermoplastic molding compound. The polymer has good mechanical and electrical properties up to the heat deflection temperature 130°C when stabilised. Unstabilised material is suitable for service for extended periods at 105°C . Processing is on conventional extrusion and injection moulding equipment capable of operating in the range of 220 - 290°C . The polymer offers a potential challenge to chemically resistant fluorinated materials, and to thermosetting plastics in the lower temperature range.

Applications for this material are connectors, computer modules, coil forms, bus-bar insulation, and switch gear bushings. General Electric recently introduced Noryl SE-100, a new flame retardant variety for appliance, business machine, aircraft and automobile applications.

*Polymethylenediphenyloxide*⁴

Developed by Westinghouse Electric Company in 1962 and called Doryl, the polymethylenediphenyl oxides are described as a thermosetting polymer prepared from diphenyl oxide with CH_2 bridges. The prepolymer is soluble in toluene but cures (200°C for two hours) to a tough, insoluble material which has maintained bond strength for over 3,000 hours at 250°C . In laminate form, the

material provides excellent dielectric properties, low water absorption and excellent mechanical strength after extended periods in the 300°C range. Doryl is used primarily as a high temperature varnish in the aircraft industry.

*Polyaryl ethers*³⁰

Uniroyal Chemicals recently introduced a thermoplastic molding resin, Arylon, on a development scale. This polymer is reported to offer good impact strength, ease of processing and good hydrolytic stability. The heat distortion temperature is 150°C. Applications for Arylon resin include exterior and interior automotive parts, range handles, coil forms, valve bodies, stems and bonnets, terminal connectors and blocks, power tool housings and industrial safety and athletic helmets.

*Polybutadiene—glycol*³²

TRW, Inc., and Commonwealth Oil have formed the Hystl Development Corporation to make and sell a new high performance thermosetting resin called Hystl (*Hydrocarbon resin from Space Technology Laboratory*). The material is a 1,2-polybutadiene prepolymer with terminal primary hydroxyl groups (G series), vinyl groups (B series) or carboxyl groups (C series). The functional end groups are capable of further reaction before the final vinyl type cure.

The Hystl resin receiving the most interest is the polybutadiene glycol (the hydroxy terminated, G series prepolymer) which is reacted with a small amount of toluene diisocyanate to form a urethane extended polybutadiene block polymer. This polymer has a high molecular weight, and is soluble and rubbery. The final cure with peroxides yields a hard, tough material, characterised as a cross-linked ladder structure stemming from condensed cyclohexane groups.

One of the significant advantages of Hystl carboxy and hydroxy terminated resins is in the ease of increasing the molecular weight of the resin by reacting it with the various polyfunctional epoxides and isocyanates. This allows the resin to be advanced in molecular weight to the desired consistency for preparing B-stage materials prior to the final cure.

The resin has been transfer- and compression-moulded, cast, laminated, and, in sheet form, match die moulded and vacuum formed.

Hystl has a heat distortion temperature of 260°C, its glass transition temperature. Hystl exhibits the mechanical and thermal properties of high performance epoxy systems and possesses a shelf life of six months at room temperature. The cured resin is reported to be stable to strong bases, glacial acetic acid, and nitric acid. Water absorption is low, as would be expected from the hydrophobic nature of the hydrocarbon structure. The electrical properties of laminates and moulding compounds show high dielectric strength and a low dissipation factor.

Moulding applications are aimed at the electronic, electrical, automobile and appliance industries. Laminate uses include radomes for aircraft, ground bases and ships, flexible circuits and cable packaging, insulating tapes, printed circuit boards and chemical hoods, ducts and scrubbers.

Polybutadiene—Firestone

In 1968 the Firestone Tyre and Rubber Company introduced a thermosetting polybutadiene resin, FCR-1261, for moulding and laminating applications. This 100 per cent hydrocarbon is reported to perform excellently in compression, transfer, and injection moulding operations with practical cure rates. Fillers, such as silica, alumina and metal powders can be incorporated with the resin up to 70 per cent by weight.

High and low pressure laminates have been developed using glass fabric or mat, nylon, cotton, jute and asbestos fibre or paper. Filament winding operations are under investigation. Five resin solutions are available using hexane, styrene and *t*-butyl styrene as solvents. Laminates show superior electrical properties, high hardness, excellent chemical resistance (insignificant loss of weight or flexural strength after seven days in fuming nitric acid), good thermal stability (months at 205°C), very low water absorption (0.2 per cent) and high heat distortion (> 260°C).

Polyphenylene sulphide

Initially developed by Dow Chemical Company⁵, the polyphenylene sulphides represent another class of stable aromatic polymers. Recently, Phillips Petroleum Company introduced both a linear and a branched polyphenylene sulphide under the trade name Ryton⁶. Typical melting points for these polymers are reported to be in the 270° to 290°C range. They can be cross-linked by heat at 400°C to provide increased toughness and adhesive strength. The polymer has good solvent resistance below 200°C, good wetting action and excellent coating adhesion. This material has been used for fluidised bed coating and shows good impact resistance and extensibility. Service at 288°C has been successful.

Polycarbonates

The polycarbonates⁷ were developed in Germany by Farbenfabriken Bayer AG; the polycarbonate resins manufactured in the United States by General Electric (Lexan) and by Mobay Chemical (Merlon) have been on the market for about 12 years. These polymers are prepared by reacting bisphenol-A with either phosgene or a dialkyl carbonate. The standard resins have excellent mechanical and electrical properties, which are maintained over a wide temperature range. The temperature maximum for extended service is about 105°C. These resins are available in transparent, as well as in pigmented form in all colours. Processing of these resins is readily carried out in usual equipment over a temperature range of the melt of from 220°C to 320°C, depending on the molecular weight.

More recently, glass fibre reinforced polycarbonate resins manufactured by General Electric, Fiberfil, and Liquid Nitrogen Processing, have produced other advantages in stability, creep resistance, and slightly higher operating temperatures, as well as increased physical properties, especially the modulus of elasticity. Other modifications of the unreinforced base resins have led to the production of several new flame retardant resins by General Electric. The most flame retardant of these resins, Lexan NB-155, has a deflection temperature some 30°C higher than ordinary Lexan polycarbonate resins. Its oxygen index

value is about 43 per cent. It combines a great number of good qualities in a single material.

The polycarbonates have found wide application in many fields. The applications are far too many to enumerate. It should be mentioned that these materials are also available in film and sheet form for packaging, insulation and vacuum forming applications.

Polysulphone

The polysulphones were introduced commercially by Union Carbide Corporation in 1965⁸. This heat resistant thermoplastic is prepared from the sodium salt of bisphenol-A and *p,p'*-dichlorodiphenylsulphone, and has good electrical properties, and resistance to acids and bases. The material, available in both transparent and opaque forms, has good thermal and oxidative stability for extended periods at 150°C, and a heat deflection temperature of 173°C. Processing is on standard equipment for injection, extrusion and blow moulding at 350-400°C. Applications for this polymer include automotive parts, housings for power tools, computer parts, appliance housings, electrical switches, circuit breakers and wire and cable insulation.

In 1967, Minnesota Mining and Manufacturing Company introduced a high temperature thermoplastic polysulphone⁷ on a limited development scale. It is composed of diphenyl and phenyl groups connected by oxygen and sulphone linkages. The thermal and oxidative stabilities of these polymers are good, and should rival those of the polyimides. The resin retains good thermal, hydrolytic, electrical and mechanical properties to 260°C. It is available as a moulding resin (Polymer 360) and as a film (Polymer 380). Processing can be accomplished on conventional extrusion, compression, transfer and injection moulding equipment. The first substantial application is in the connector field. The properties of the material suggest it for moulding electrical components, coil forms, switch parts, wire wraps and circuit boards. Polymer 360 has been extruded as wire insulation and cable jacketing.

*Polycarboranesiloxane*¹⁰

The polycarboranesiloxanes, developed by Olin Mathieson, and bearing the trademark Dexsil, are thermally resistant to temperatures of 260°C and retain elastomeric properties after short exposure to temperatures as high as 525°C. The development of this new family of polymers is in its infancy, with predicted use as a component in rubber products used for sealing, heat shields, adhesives, protective coatings, and electrical conductor inserts. These polymers can be extruded, compounded and moulded.

*Poly-p-xylylene*¹¹

Parylene is the generic name of a family of polymers developed by Union Carbide in 1961. The polymers are made by a unique vacuum deposition process, which allows pinhole-free films to be deposited on substrates at room temperature in thicknesses ranging from a few hundred angstroms to several mils.

The basic member of the family is parylene N, poly-*p*-xylylene, and has outstanding electrical characteristics. Parylene C, poly (chloro-*p*-xylylene),

exhibits exceptional barrier properties. Both parylenes are inert to almost all acids and bases and are insoluble in organic solvents below 170°C.

In oxygen-free environments, both parylenes maintain long-term (ten years) useful mechanical properties at temperatures in excess of 225°C. For brief periods, both parylene N and C may be used at higher temperatures, figures exceeding 350°C and 265°C, respectively, being quoted. Both polymers may be used for short periods in air at about 200°C and extrapolation of present data indicates that parylene C should maintain useful electrical properties in air for ten years at temperatures greater than 125°C.

Parylene C is being used as a conformal coating for electronic assemblies. This material's favourable combination of barrier and electrical properties has enabled it to surpass the military specifications set forth for circuit board coatings in MIL-I-46058B. Parylene N is rated as a primary dielectric, and is being used in a line of capacitors designed for continuous operation at more than 125°C. These capacitors have a better frequency response and a lower dissipation factor than, and are 20 per cent the size of, an equivalently rated polystyrene capacitor.

The parylenes have also been successfully evaluated as semiconductor passivating films, as hydrophobic surfaces to increase the heat transfer efficiency of a steam condenser, and for particulate encapsulation of reactive materials. Unsupported films as thin as 300 Å are being used in optical and radiation applications. Other uses include corrosion retardation and cryogenic applications.

*Polybenzimidazole*¹²

The polybenzimidazoles (PBI) were first synthesised in the late 1950s, and exhibit excellent thermal and oxidative stability. The thermal breakdown by TGA is as high as 600°C in nitrogen, and oxidative resistance is about 300°C in air; good physical properties are retained after 1,000 hours at 315°C.

The polymer is produced by reacting an aromatic tetramine with an aromatic diacid to form a relatively low molecular weight prepolymer. The material is infusible after curing, and must be converted into useful forms prior to the final cure. Final processing, during which high molecular weight, linear, thermoplastic molecules are developed, occurs at elevated temperatures. Difficult processing has restricted civilian use of this polymer.

The polybenzimidazoles have had limited commercial development by Narmco Materials Division of Whittaker Corporation, for use as high temperature adhesives and laminating resins for the aerospace industry.

Two polybenzimidazole prepolymers are offered by Narmco Materials Division; these are designated Narmco 2801 and Narmco 2803. These prepolymers are formed through the condensation of 3,3'-diaminobenzidine with various isophthalic acid derivatives. Narmco 2801 is a condensation product with tetramines and diphenyl isophthalate. Narmco 2803 is the condensation product of the tetramine and isophthalamide. On heating to a temperature of 370°C, the prepolymers are converted into polybenzimidazole. In the prepolymer state, the polybenzimidazoles are both soluble and fusible and will set and stick to surfaces well.

Narmco 2801 is particularly effective as an adhesive or a binder for a variety of materials and should be useful in the manufacture of high temperature resistant friction materials and abrasives. This polymer is stable in nuclear applications and possesses good electrical properties. Foams, which are tough and lightweight, can be produced from these prepolymers. Narmco 2803 is particularly suitable for use as an unfilled, unreinforced plastic.

Imidite 1850 is a PBI laminating prepreg for aerospace and supersonic transport applications. Specific applications are similar to those described for Narmco 1832 in the polyimide section.

Imidite 850 is a PBI based adhesive formulation supported by a water resistant fibreglass reinforced carrier. Imidite 850 is one of the best commercial adhesives for use at elevated temperatures; maintaining short time (5-15 min) bearing capabilities to 540°C, and on long time exposure (4,000h at 185°C) shows no decrease in strength at 185°C. Applications for Imidite 850 are similar to those described for Narmco's polyimide adhesive Miltbond 840.

Polybenzimidazole fibres are being developed by Celanese. DuPont and the Air Force Materials Laboratory have also shown interest in PBI polymers.

*Aromatic polyamides*¹³

Some of the most thermally stable polymers so far discovered are the aromatic polyamides. DuPont introduced this polymer in 1961 and production reached full commercial scale in 1967 under the trademark Nomex. Although the composition of the polymer has not been officially disclosed, it is believed to be based on poly-*m*-phenylene-isophthalamide¹³. Originally code named HT-1, Nomex is available as a fibre and as a paper structure. The polymer does not melt, it is rapidly self-extinguishing and it is highly resistant to oxidation. The material has high heat resistance, a heat deflection of 280°C, and outstanding Class 180 electrical properties. Underwriters' laboratories recognise it for continuous use at 220°C and MIL-I-24204 (ships) covers its use in electrical machinery at temperatures up to 220°C. The major use of the paper form is in electrical insulation and in applications where heat and chemically resistant fabrics are needed.

Laminates composed of the paper are commercially available, as are composites of the paper and mica; flexible laminates of the paper and other plastic films; various types of round, square, and rectangular tubing, and coil forms; bushing, wedges, slot insulation; and die cut and formed parts of all types. Nomex fibre, in the form of coated and uncoated tapes, braids, cords, and thread, is used on wrapped magnet wire, tie cord, braided sleeving and pressure sensitive tape.

DuPont supplies paper, in thicknesses of 2 to 30 mils, which may be coated with a polyimide varnish to provide heat-sealing characteristics. This material is also found in speciality end uses, such as insulation in high temperature dry type transformers, pipe wrapping for use at high temperatures, and insulation in motors and generators.

*Polyamide-imide*¹⁴ and *polyester-imide*¹⁵

These polymers have high temperature properties intermediate between the aromatic polyamides and the aromatic polyimides. The polyamide-imides are

produced from an aromatic diamine and an anhydride-acid, which forms a polyamic acid capable of being converted to the polyimide through the action of heat. The polyester-imides are produced from aromatic diamines and dianhydrides containing ester links in the central chain. Trishydroxy-ethyl isocyanurates (THEIC) or similar type compounds, have been used to form the ester linkages. In their production and processing, both polyester-imides and polyamide-imides share many points in common with the aromatic polyimides.

Amoco Chemicals Corporation is manufacturing polyamide-imides, called AI Polymer, for use at 290°C. These polymers are gaining acceptance in various application areas, such as magnet wire enamels, high temperature laminates and adhesives, decorative coatings for high-temperature service, and moulded components. The Amoco polymer is supplied as a powder that is soluble in a number of solvents and cures at about 250°C. DeBeers is marketing an amide-imide paint coating for kitchen utensils, and Sterling Varnish has developed an amide-imide varnish for electrical applications.

Ten years ago Westinghouse Electric Company³⁴ developed a polyamide-imide resin based on benzophenone dianhydride, trade marked Aramidyl. At present, Westinghouse is offering commercially a coated copper foil (Aminin) for use in printed circuitry and flat cable systems. Polyamide-imide based laminates and adhesives for aircraft applications undergoing prolonged exposure to high temperature environments related to supersonic flight conditions, were developed under contract from the Air Force.

Glass laminates showed 30,000 hours of ageing at 250°C without serious impairment of properties. Polyamide-imide glass laminated gear and bearing constructions for use in helicopter gear boxes were developed under Army contracts for use in the UH-1 helicopter. This work also resulted in a long-life, high temperature, adhesive system for stainless steel and/or titanium, with a lap shear bond strength of 1,000 psi at 340°C after 100 hours.

A flat conductor cable system for wiring harnesses, based on a polyamide-imide, was developed four years ago.

Rogers Corporation is offering a polyamide-imide coated copper foil material, (Duroid 8150) which can be printed, etched, and film covered to provide a thin flexible printed circuit or flat cable. Computer current and signal conductors are the primary applications.

The polyester-imides are available commercially from a number of manufacturers. General Electric Company (Imidex E) and Schenectady Chemical Company (Isomid)¹³ are both marketing these polymers for magnet wire enamel. In 1967 Westinghouse introduced a polyester-amide-imide called Enamel Omega for electrical applications. Mobil Chemical Company is also active in the area of high temperature insulation, and has been marketing a polyester-imide since 1965. P. D. George Company offers a polyester-imide wire enamel based on a modified isocyanurate.

*Polyimides*¹⁶

The polyimides have received a substantial amount of development, and are among the most promising of the high temperature polymers. They are available in both linear and cross-linked forms. They provide particularly outstanding

thermal and oxidative stability, and have already found a secure place in the technology as electrical insulation and in mechanical applications, such as bearings in severe thermal and wear environments. The aromatic polyimides are produced essentially in two steps by the reaction of a dianhydride with an aromatic primary diamine. In the first step of the reaction, a polyamic acid is formed, which may be converted subsequently by heat or through the use of suitable catalyst to high molecular weight linear polyimides which are non-fusible and very insoluble.

Numerous manufacturers have shown interest in polyimides since 1961, namely, DuPont, General Electric, Monsanto, TRW Inc., Narmco, American Cyanamid, Quantum Inc., Dixon, US Polymeric, and Lash Laboratories.

DuPont has marketed polyimide based wire enamels and varnishes and coated glass fabrics (Pyre ML) since 1961, polyimide film (Kapton) since 1965, precision parts and abrasives (Vespel) since 1962. Polyimide prepregs (Pyralin) are available for high and low pressure laminates. Polyimide fibers, adhesives and foams are also being developed, as well as electrically conductive films and paints.

The polymer is based on pyromellitic dianhydride and oxydianiline, and has a service life of 20,000 hours at 230°C, 1,000 hours at 315°C and short term use to 400°C.

Kapton film is produced in two forms. Type H is a pure polyimide film, available in thicknesses from $\frac{1}{2}$ to 5 mils. Type F is a heat sealable modification, which is a composite of the base polyimide film coated with Teflon (fluorinated ethylene propylene copolymer) fluorocarbon resin. Type F is available in thicknesses ranging from 1.5 to 6 mils, the film consisting of various polyimide substrates with FEP on one or both surfaces. Currently in development are additional product modifications, such as ultrathin (25 gauge) Type H film and very thin heat-sealable structures.

Polyimides are essentially non-fusible, and are difficult to fabricate by conventional polymer moulding processes. DuPont is employing a sintering technique as one means of fabrication to form its polyimide moulding resin (Polymer SP) into finished parts (trade marked Vespel). Four standard compositions are available as moulded products: an unfilled resin, a 15 per cent graphite-filled compound, a MoS₂-filled material and a composition filled with 44 weight per cent glass fibre.

Pyralin prepregs utilising several varieties of E glass, silica, quartz and S glass are under development for structural, dielectric and printed circuitry applications. Thermal stability of Pyralin is good. Time to 40 per cent strength retention is 35,000 hours at 260°C, 1,500 hours at 315°C and 100 hours at 370°C. Pyralin glass roving prepregs are available for structural and dielectric applications. Conductive polyimide coated glass fabric for heating elements, static bleed off, and microwave absorption, and Pyralin coated Nomex paper are marketed. An N-methylpyrrolidone-hydrocarbon solution of Pyralin is being used for coating, sealing and bonding applications.

American Cyanamid currently has available a polyimide moulding compound that differs from other existing polyimides in that it is a true thermoplastic, rather than a thermoset, material. Processing by conventional injection moulding

equipment, XPI-MC154 Polyimide offers the user a dimensionally stable material, with a heat distortion of 260°C, which is suitable for use in demanding electrical, chemical, and thermal environments. Potential applications include switch parts, gears, electrical connectors, and insulators.

A polyimide adhesive film supported by a glass carrier, FM-34, is also available from American Cyanamid. This adhesive film was formulated for strength retention after long term exposure to temperatures of 205 to 213°C in both metal-to-metal and sandwich constructions. There is no significant change in tensile lap shear strength after 20,000 hours ageing at 205 or 260°C. After 2,000 hours ageing at 315°C, FM-34 adhesive film maintains a shear strength of 1,300 psi.

In 1961 Monsanto introduced a series of Skybond polyimide resins, based on benzophenone dianhydride and aromatic diamines, for use in preparing glass cloth laminates. Monsanto has, also, done work with polyimide resins in wire enamel applications, but is not currently offering products for such applications. Recently Monsanto introduced a new polyimide resin with better thermal resistance characteristics than Skybond. Called RX-383, the resin is a polyimide specifically designed for structural and speciality applications where extended exposure at temperatures up to 370°C is required. The resin solution (85 per cent solids in ethylene glycol) is used as a laminating varnish. Glass laminates prepared with this resin system retain a flexural strength greater than 20,000 psi when tested at 370°C and exposed to this temperature for 100 hours.

Dixon Company recently introduced a new line of polyimide moulded parts called Meldon PI. This material has good retention of physical properties after 100 hours at 370°C, and is available as moulded sheets, rods and tubes.

Quantum, Inc. is a newcomer to the polyimide field, introducing a moulding resin, adhesives, varnishes, and laminating resins called Quantad 159 in 1967. Features of Quantad 159 include thermal stability at 315-370°C for extended periods (1,000 hours), high abrasion resistance, and good peel strength. The primary application for this resin system is in producing circuit board laminates.

Narmco Materials Division of Whittaker Corporation is marketing a high temperature resistant fibreglass reinforced tape containing an adhesive material based on a polyimide resin system, Meltbond 840. This material is suitable for use with aluminium, titanium, beryllium and stainless steels, where resistance to continuous exposure to intermediate temperatures (205-315°C) is required. Applications include the bonding of metals to control surfaces of missiles and aircraft, and the bonding of high temperature radomes, deflectors, radiation covers, re-entry nose cones and turbine blades.

Narmco 1832 is a B staged polyimide laminating prepreg. This material was designed for the manufacture of glass fabric reinforced structural laminates, and laminate faced, honeycomb sandwich details, where resistance to long term exposure at temperatures up to 285°C is required. Applications for this structure include high temperature de-icer ducting, wing closure panels, wing to body fairings, wing and fin tips, radomes and antennae, exhaust deflectors, leading edges and re-entry structural substrates.

Lash Laboratories introduced a modified polyimide, high temperature adhesive for use at 260°C in 1968. The material, called Lashclad ID, was developed for use as a high temperature adhesive for bonding polyimide film or laminates to metals, and as a conformal, protective coating for flexible circuits. The resin is supplied as a 25 per cent solids solution in dimethyl sulphoxide and can be applied by spraying, dipping or brushing to thicknesses of 1 mil.

A new polyimide resin solution (dimethyl formamide solvent), "P13N", is being offered by TRW Systems, Inc., for military and commercial applications on a development scale. The first polyimide form available was an impregnating varnish for glass cloth in a formulation designed for press grade laminates. Composites for vacuum or autoclave moulding, a compression moulding formulation, boron, graphite and silica reinforced laminates and adhesives are being developed.

The principle advantages of the P13N resin lie in its ease of fabrication; namely, prepregs are stable and not subject to hydrolytic or oxidative degradation; they can be processed into low void laminates (less than 2 per cent void) using standard moulding techniques at 315°C and 200 psi for relatively short durations (60 minutes); and no post cure or press cooling is required. The final cure involves an addition type polymerisation rather than a condensation reaction with the elimination of volatile side products. Thermal ageing at 315°C for 1,000 hours has been successful. Laminates retain a 40,000 psi flexural strength after 1,000 hours at 285°C.

Applications for this resin are in supersonic aircraft structures, jet engine components, circuit board, flexible electrical cable insulation and reinforced ablative nozzle inserts.

US Polymeric introduced a polyimide glass prepreg, V-303, for vacuum bag moulding in 1967. The Air Force Material Laboratory has extensively evaluated high temperature laminates for various structural substrates. Recently Avoco's Aerostructures Division fabricated a boron reinforced polyimide structural beam for Boeing's supersonic transport project.

Ferro Corporation has worked with various high temperature resins and is making polyimide prepregs available on a limited scale. American Reinforced Plastics Company, a specialist in the manufacture of prepregs for the aerospace industry, is offering a polyimide material.

Polybenzothiazoles

Abex Corporation has developed commercially a polybenzothiazole (PBT) for use in laminating applications. This resin system, requiring a somewhat involved cure process, offers higher thermal and oxidative stability than the polyimides. The resin is soluble in dimethylacetamide and melts and flows before cure. The cure mechanism involves a zinc oxide cross-linking. Thermal ageing at 315°C for 2,000 hours shows 95 per cent retention of strength.

The future

In the immediate future, several new high temperature resins should be appearing on the market. At least half a dozen new polyimides and polybenzimidazoles

resins are being quietly developed around the USA; some not as quietly as others.

Many new and promising polymer systems are also being explored, for instance the polyoxadiazoles²⁹, poly-thiadiazoles,¹⁷ polytriazoles,¹⁸ polybenzoxazoles¹⁹, polythiazoles²⁰, polyquinoxalines²¹, polypyrazoles²², pyrrolones²³, polyhydrazides²⁴, polyphenylenes²⁵, polyperfluorotriazines²⁶, polytetrazopyrenes²⁷, bisbenzimidazobenzophenanthroline-dione (BBB)²⁸. It is evident from the above that an enormous variety of organic polymers have been synthesised in the quest for new materials with superior high temperature performance. As these new materials become commercially available, it seems unavoidable that they will bring an improvement in the processing and reliability, as well as a reduction in cost.

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Next month's issue

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

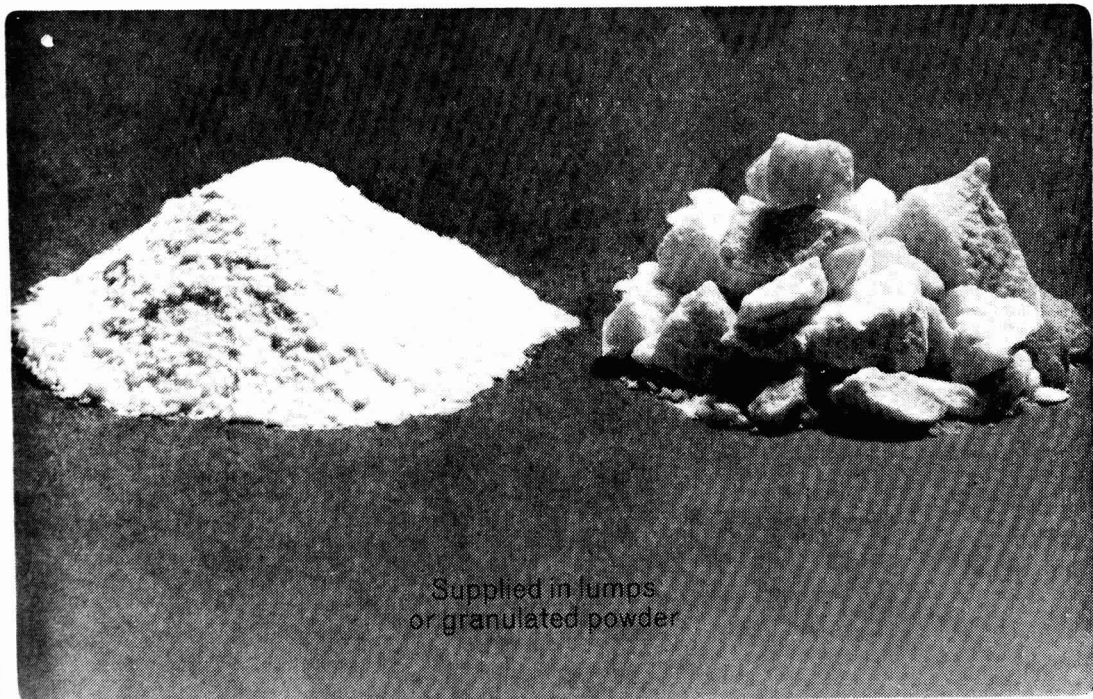
"An international flow cup," by A. N. McKelvie.

"Antifouling paints based on organotin compounds. Part II Spectrographic determination of microgram amounts of bis(tri-n-butyl tin) oxide in aqueous solutions," by L. Chromy, W. Mlodzianowska, K. Uhacz and R. Warchol.

"Towards automation," by H. R. Touchin.

"Coatings based upon thermosetting resins containing reactive liquid polypropylene sulphide as plasticiser," by J. L. Smith, G. T. Williams and R. D. Singer.

Announcement



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introduction to paint technology



second edition with additional chapter

The sales of this Association publication now exceed 13,000, and because of continuing high sales of the second edition, and the need for a work of reference to be constantly abreast of the latest developments in its field, the Council of the Oil and Colour Chemists' Association has authorised the addition of an eleventh chapter to the 'Introduction to Paint Technology.' Entitled 'Recent Developments,' the Chapter incorporates up-to-date information on the latest advances in the technology of the paint and allied industries.

This addition will help the 'Introduction to Paint Technology' to maintain its position as an invaluable aid to young entrants into the industries and to marketing and other personnel requiring a basic knowledge of the principles and techniques of surface coating production and application.

new chapter

The new chapter is subdivided to three sections

resins and media

Including polyester, epoxy, polyurethane resins and developments in water based paints, vinylics, etc.

application techniques

Including electrodeposition, powder coatings, strip-coating, aerosol spraying.

instrumental testing and control methods

Including colour measurement, viscometers, brushability, hardness, film thickness, weatherometers, and use of computers.

The book contains 204 pages including 11 line diagrams, 8 photographs of common paint defects, and comprehensive index. Copies can be obtained from the Oil and Colour Chemists' Association, Wax Chandlers' Hall, Gresham Street, London E.C.2, price 20s. (post free).

Reviews

OUTLINE OF INDUSTRIAL ORGANIC CHEMISTRY

BY A. RIECHE. London: Butterworth 1968. English translation of third German edition. Pp. xix+572. Price £4 18s 0d.

This text-book, which was first published in German in 1955, followed by a second, enlarged, edition in 1961, was based on lectures given by Professor A. Rieche at Jena University. The volume under review is the English translation of the third edition, published in 1966.

In a brief introduction (seven pages) the author discusses the fundamental considerations relating to the planning and design of a large scale process. The main part of the book then deals with the historical development of the most important branches (excepting perfumes and essential oils) of the organic chemical industry, its processes, chemical principles and economic aspects.

The subject matter is divided into three main groups: *A* The chemistry of coal and crude oil and the production of chemical raw materials and intermediates; *B* The chemical processing of raw materials produced in agriculture and forestry; and *C* The manufacture of commodities by chemical processing of natural and synthetic raw materials. These groups are further subdivided into sections dealing with specific aspects including coal, natural gas, petroleum, and synthesis based on carbon monoxide, paraffins, olefins, acetylene and aromatic intermediates (under group A); wood, sugar, fermentation, fats, oils and tanning (under group B); and synthetic fibres, lacquers, plastics, elastomers, resins, textile auxiliaries and washing raw materials, pharmaceutical products, synthetic organic dyestuffs and explosives (under group C).

The book is written mainly from a chemical standpoint, the emphasis being on materials rather than the equipment. The various unit processes (e.g. distillation, filtration, centrifuging, evaporation and drying), control devices (e.g. for the measurement of gas flow, temperature and pressure) and equipment (e.g. pumps, filters and grinders), together with manufacturing processes, are discussed and illustrated by 152 line drawings at appropriate places throughout the book, in connection with a specific chemical process. These sections will provide the student reader with a useful introductory survey of chemical engineering principles.

The specialist may feel that the author does not always present an entirely balanced account of a particular process. For example, only one method is given for the manufacture of bodied linseed oil. However, the author has summarised successfully a vast amount of material covering many technologies and the text appears substantially to be accurate. Only a few trivial errors were noted.

Throughout the text and at the end of each chapter numerous literature references are given. Understandably these have a bias to German sources, since the book was primarily intended for the German student. There is a good index and a comprehensive contents list. Also, the book is well produced and reasonably priced.

The publication of this translation provides an extremely useful and very readable source of information for the student (and possibly the younger qualified man entering industry) which would otherwise be inaccessible to those who do not read German.

R. N. FAULKNER

THE HISTORY OF PVC

By MORRIS KAUFMAN. London: Maclaren and Sons Ltd., 1969. Pp. 208. Price £4 2s.

The author, in his preface, states "This book is the outcome of a conviction that teaching aspiring rubber and plastics technologists the bare bones of polymer technology provides a lifeless two-dimensional study—it throws no light on the reasons for the technology or the mainspring of its progress." This most certainly is not so of Morris Kaufman's "History of pvc." It is alive, interesting, and entirely readable. There are, of course, many books dealing with the history of scientific subjects, usually confined to the first few pages of the first chapter or the preface, but the reviewer has not come across one before where the historical and technical aspects continue side by side throughout the book.

In the first chapter Kaufman gives considerable detail to the early days of pvc. This chapter is refreshing and informative, and must prove of exceptional interest to the non-academic chemist; it will certainly give him a new insight into polyvinyl chloride chemistry. Chapter 2, on the industrial origins of polyvinyl chloride, deals with the appearance of industrial interest, followed by the various early methods and patents involved. Chapter 3, on the preparation of the monomer, gives a clear picture of the economic and technical difficulties associated with early production. This is followed naturally by a chapter on polymerisation, covering the research and reasoning behind photopolymerisation, and thermal, solution, dispersion, suspension, and emulsion polymerisation, finishing with a short resume of progress in the United Kingdom and the USA.

Chapter 5, on modification of vinyl chloride polymers includes work on copolymerisation. Chapters 6 and 7, on plasticisation and stabilisation and also on processing, will provide interesting and informative reading for anyone engaged in this work. The final short chapter, on the early applications of pvc, tidily completes the volume. The standard of reference in this book is very good. There is also a comprehensive index with which one cannot find fault.

The book is an excellent example of balance between historical and technical viewpoints. The author has perhaps laboured the historical angle, but after all it is the history of pvc. It loses nothing from this treatment, and leaves one with the feeling that more "dry as dust" technical publications could well do with a few more interest-making historical facts included. Kaufman's treatment of this volume is both systematic and interesting.

There is no ambiguity in the terminology; the clear type is excellent as is the whole presentation. The author is to be congratulated on the obviously painstaking care and observance of detail which have been embodied in this volume.

G. ANDERSON

Information Received

(In case of difficulty regarding addresses, members and subscribers to the JOURNAL should apply for details to Director & Secretary of the Association at the address shown on the front cover.)

Blundell-Permoglaze Ltd. has recently introduced a new high-bake acrylic finish, specially formulated for high performance on domestic equipment. The new product, designated the 003 range, is based on acrylamide acrylic, and is designed for stoving at 400° F in IR ovens. Full gloss, exceptional hardness, chip and scratch resistance, and hot fat and detergent resistance are claimed for the finish.

“Zinc dust in paints” is the title of the latest in the series of bibliographies produced by the **Zinc and Lead Development Associations**. This is an extended and revised edition of the previous volume, and contains some 850 references to zinc dust paints, covering the period 1931-68.

The introduction of *Adiprene CM*, a new type of millable urethane elastomer, has recently been announced by **Du Pont Company (UK) Ltd.** The product, which is polyether based, is claimed to offer improved processing, lower costs, and good resistance to tear, radiation, chemicals, weathering and ozone. It is said to be vulcanisable with both sulphur and peroxide systems, and to have a hardness range of 45° to 90° Shore A.

A new brochure, entitled “What is carbon black?,” intended for the technician using carbon black, has recently been issued by **Degussa**. The various types of carbon black are described, together with their manufacture and uses, and copies are available from Degussa on request.

Kronos Titanium Pigments Limited has recently announced the availability of the *Titanox-Kronos strength comparator*, as described by Mr F. B. Stieg in his paper to the recent Symposium on water-borne coatings organised by the Manchester Section.

The comparator, which is a slide rule which compares the relative opacity of TiO_2 pigments in a given formulation when used in conjunction with a reflectance photometer, is available from Kronos on request.

“The painting of joinery timber which has been pre-treated against rot” is the title of an information leaflet recently published by the **Paintmakers Association of Great Britain Limited**. Copies are available from the PA.

It has recently been announced that the **Construction Industry Research and Information Association** is to change slightly its field of operations.

Its main functions will still be to promote and manage research for the benefit of its members, and, arising from this work, to provide them with the appropriate and complementary technical intelligence service. However, it is no longer required to proceed with the work of providing a nation-wide advisory service, as the Ministry of Public Building and Works has decided that the **Building Research Station** is to experiment with the running of such a service, to test its commercial viability. CIRIA will continue to depend on subscriptions from members, and to receive a grant from the Ministry of Technology.

BP Chemicals (UK) Limited has recently published a fully illustrated 32 page booklet describing its operations and facilities. Copies are available free of charge from BP's Public Relations and Advertising Dept.

Cerric Cellfast is the name of a new range of inks introduced by **Pinchin Johnson Paints**, designed for use in automatic screen printing, with forced drying.

It is claimed that *Cellfast* provides an ultra-thin film which dries much faster than conventional screen inks, and to have outstanding covering power and light fastness. A range of 27 colours, plus black, white, and opaque white, is available.

The two latest booklets issued by **Vinyl Products Ltd.** in its series of "Technical service reports" deal with "Emulsion paint test methods (C1)" and "Vinyl acetate-acrylate copolymer emulsion paints (C2)".

A new high sensitivity oxygen meter, based on the electron capture detector cell, has been announced by **Analytical Instruments Ltd.** The instrument, which is mains or battery operated, is a portable gas chromatograph with electron capture detector, and, by use of various accessories, can be used to measure the amount of oxygen in most gases, oxygen permeability of films, and oxygen dissolved in liquids.

Koch-Light Laboratories Ltd. has recently brought up to date and re-issued its "Catalogue and price list." Copies are available free of charge from Koch-Light.

It has recently been announced that **Victor Wolf Limited** has concluded a licence agreement with **Kemrez Chemicals**, of Australia. Kemrez are to have exclusive rights to manufacture and sell Wolf's *Wolfamid* polyamides.

Letraset Limited has issued a booklet giving full details of its range of products for marking and decoration of thermosetting and thermoplastic articles. Three systems are available, for in-mould or separate heat transfer operation.

Samuel Banner & Co. Ltd. has now moved its head office and administration to new offices at its Liverpool works.

The Walker Chemical Company Limited has recently issued a data sheet on *Walker paraformaldehyde 87 Flake*, a solid polymer containing 87 per cent formaldehyde and 13 per cent water. The product is said to be ideally suited for the manufacture of p/f, c/f, u/f, and m/f resins, particularly where low water content is required.

Surface Coatings journal is to sponsor a second "International Conference on Plastic Powder Coatings," following the success of the first conference. The venue will be the Criterion-in-Piccadilly, London, from 10-12 February 1970. Additional features will be a workshop session on the 11 February, and an information service by companies manufacturing powder coatings.

Copies of the programme and registration form are available from the conference organisers, **Business Conferences and Exhibitions Ltd.**

A new range of strippers for all types of finishes and substrates has recently been announced by **Croda Polymers Ltd.**

The range includes water and solvent-based types, alkaline and acidic baths for hot and cold dipping, a grade in powder form, additives for traditional caustic baths, and a brush-on grade for conventional paints.

Sample amounts of *SP-815 Heat Stable Dispersant*, a new product to provide high dispersibility of high pigment solids, are now available, announce **Calgon Corporation**. *SP-815* is an amber-coloured, slightly viscous, aqueous solution of an organic dispersant, claimed to be effective over a wide range of pH, and to exhibit excellent hydrolytic and thermal stability.

Four-ounce samples, and technical literature, are obtainable from the Chemical Specialties Division of Calgon.

Badische Anilin- und Soda-Fabrik AG has recently added new products to two of its range of colours.

Luconyl Yellow 3R is a reddish-shade yellow, added to the *Luconyl* range of colour pastes for aqueous paints. Outstanding fastness properties are claimed, even in extensive reduction.

Fanal Blue GNC Supra is an addition to the *Fanal* pigment range for printing inks, having the same colour as, but slightly greener and cleaner shade than, *Fanal Blue B Supra*.

Section Proceedings

Hull

Reactivity of titanium dioxide in vinyl chloride/vinyl acetate copolymer surface coatings

The first meeting of the current session was held at the College of Technology, Hull, on Monday 6 October. The section Chairman, Mr E. A. Brown, introduced Mr F. D. Robinson, who gave a lecture entitled "Reactivity of titanium dioxide in pigmented vinyl chloride/vinyl acetate copolymer surface coatings."

Mr Robinson remarked that, although surface coated titanium dioxide was normally regarded as inert when present in paints, there were certain areas in which reactivity could be detected. One such area was in combination with pvc/pvac co-polymer solutions, and the reactivity in such systems had been followed by measuring the increase in viscosity which occurred on storage for a period of up to six months. In extreme cases this took the form of gelation and could even occur during the actual preparation of the paint.

Three factors were essential to the development of reactivity. These were: the presence of at least a minor proportion of acid in the co-polymer, inorganic treatment of the pigment with silica or alumina, in the presence, or absence, of zinc oxide, and the presence of water. Additional organic treatment of the pigment might exaggerate the effect as with amines, or reduce it as with polyol pigment treatment. Other materials, such as maleic acid and, particularly, citric acid, when added to the paint in minor amounts were effective in reducing the reactivity of the system, and Mr Robinson considered that such materials behaved as chain terminators.

Commercially available polymers, prepared by solution polymerisation, had been employed in this work, but one polymer, incorporating 1 per cent of maleic acid in the backbone, prepared by emulsion polymerisation, had also been examined. This gave total stability at comparable pigment and solids content in the presence of added water.

In concluding his paper, the lecturer stressed that, with careful choice of pigment and resin, stable systems could be obtained. The lecture was extremely well illustrated with slides and it is hoped that it will be possible to publish it in due course.

During the discussion period which followed, Mr Robinson answered questions from Messrs. Gibson, Gilroy, Finn, Smith and Mell. The vote of thanks was proposed by Mr L. W. Wynn. Twenty-five members and seven visitors were present.

J.A.H.

Operational research

The second meeting of the Hull Section was held at the College of Technology, Hull, on Monday 3 November, when Mr S. J. Morrison gave an interesting lecture entitled "Operational research: a service to management."

Mr Morrison referred briefly to the various services available to management, ranging from accountancy and work study to such sophisticated techniques as automation and cybernetics.

Management roots extended back to such large scale projects as the Chinese wall and the Egyptian pyramids, and to large scale organisations like the church and the army in ancient times. With the growth of large productive enterprises in the 18th and 19th centuries, for example mining, brewing, and textiles, management was faced with problems arising from technological change, new industrial systems and the market economy. Landmarks in the contribution of scientific and mathematical thought and inventions were then given.

Operational research originated in 1936 and the years immediately following, with the development of radar and fighter direction. Similar techniques were extended by the Allies during the war to weapons evaluation, convoy tactics, and military organisation, and in the civil sector, after the war, by large basic industries to such problems as production scheduling, stock control, forecasting, and queues.

The OR process was likened to that of work study, the steps taken in the solution of a problem being:

- define the problem
- collect information or data
- construct a (mathematical) model
- find a solution
- test the solution to see if it was currently valid
- set up a control to see if the environment changed
- implement

Feed-back in the control of a process, and the formulation of a mathematical model, were briefly discussed. The primary purpose of the model was to stage a demonstration rather than to solve the original problem. A compromise had to be made between a crude model of poor similitude to real life, giving non-valid and non-useful response, and one of high similitude which took a long time to build or was unworkable.

In the second part of his lecture, Mr Morrison illustrated the use of models and OR techniques with reference to statistical analysis, prediction by regression analysis, stock control, linear programming, simulation and network analysis.

During the long discussion which followed, the lecturer answered questions from Messrs. Brown, Lythgoe, Finn, Mell, Reed and Gilroy. A vote of thanks was proposed by Mr P. J. Gay. The meeting was attended by 18 members and 3 visitors.

J.A.H.

Irish

Paint formulation and performance

The first meeting of the 1969-70 session of the Irish Section was held in the Imperial Hotel, Cork, on 26 September. The subject was "Paint formulation and performance" and the lecturer was Mr A. H. Hipwood from the Chemical Inspectorate, Woolwich. The Chairman of the Irish Section, Mr S. McWade, in opening the meeting, paid tribute to Messrs. Harringtons Goodlass Ltd., Cork, and in particular to Dr Long for the hospitality given to the members during their visit to Cork. He also thanked Messrs. Ford Ltd. for allowing the members to visit their factory that afternoon (see photograph). In introducing Mr Hipwood, Mr McWade wished him and Mrs Hipwood a "Cead Mile Failte" to Ireland. He also pointed out that Mr Hipwood was no stranger to Ireland or to the Irish members, as he had lectured at a seminar at the Institute for Industrial Research and Standards last year.

Mr Hipwood, before beginning his lecture, paid tribute to the Irish hospitality as shown by the members. He then commenced by saying that paint formulation and performance was a vast subject, and in the interest of brevity he would be restricted to a review of the outdoor weathering performance of glossy decorative air-drying alkyd systems. Also, to preserve the confidence established between the manufacturers and the Inspectorate, reference would not be made to details of composition or to proprietary names of any raw materials.

The Chemical Inspectorate was responsible for a very wide range of organic coatings supplied for the decoration and protection of buildings, structures, aircraft,



Members of the Irish Section photographed with Mr Hipwood (front row, second from right) during their visit to the Ford Motor Company

and other diverse types of equipment used by the Services. It had been found that for most requirements a knowledge of the composition of the paint was essential for sound interpretation of quality. Most Service specifications were therefore of the performance/composition type, in which essential composition requirements, such as type of medium, were specified, and a number of performance tests included to ensure that the paint possessed the standard of performance within the general class specified by composition.

Definition of natural weathering

The exposure site, Glascoed, South Wales, was described together with the instruments used to record climatic data. The reasons for its selection and the limitations of "one site" exposure tests were discussed. The assessment of film performance to natural weather was also described, together with the automatic data processing machine used for recording and retrieval of weathering results.

Surface behaviour of alkyd films

With the introduction of alkyd resins, a considerable improvement was achieved over the older oil based and oleo-resinous paints with respect to gloss retention, colour retention, chalking and erosion resistance, and this type of medium was currently most popular for air-drying glossy coatings where decoration was of prime importance. During weathering, all films exhibited surface deterioration, but the rate and degree of degradation varied with each type of paint formulation.

Pigmentation: At Chemical Inspectorate it had been found convenient to divide the range of colours described in B.S.381C and B.S.2660 into "colour groups" of film performance. The general trend of surface behaviour for each group was given.

Group A comprised strong blue colours based on Prussian, phthalocyanine, and indanthrene pigments. Colours in this group exhibited "bronzing" on exposure to natural weathering. It was appreciated that particle size control by pigment manufacturers was important for retarding this defect, but even so, such failures were observed with the so-called "non-bronzing" types of pigment.

Group B comprised strong green colours based on combinations of Prussian blue with light-fast chromes, or phthalocyanine with arylamide yellows. Organic combinations, although more expensive, were in general superior to inorganic varieties with respect to colour retention, but considerable "blueing" or "yellowing" was a frequent failure and was evident within three years of natural exposure.

Group C comprised white, pastel colours, and shades which contained a high proportion of white pigment. In this context it should be observed that even very dark greys contained titanium dioxide as the major pigment constituent. This group of colours was the most attractive for decorative purposes but the films in general were first to reveal loss of gloss, chalking and colour change for any given alkyd medium during exposure to natural weathering. For maximum resistance to weathering, it was essential to observe the recommendations made by the suppliers of the rutile titanium dioxide.

Group D comprised strong browns, red oxide and other "earth colours." Early loss of gloss was a common feature with colours of this group, and in these respects the group was inferior to Group C. When mixtures of pigments were used for the production of colours like the "warm browns," colour retention on exposure could be most disappointing.

Group E comprised strong reds and violets. Light-fast pure organic pigment dyestuffs were superior for colour retention to inorganic pigment combinations containing molybdate chromes or cadmium reds. It should be appreciated, however, that dilution of organic pigments with rutile titanium dioxide, which was a common practice with manufacturers experiencing difficulty in meeting contrast ratio requirements of specifications, resulted in much earlier fading failures. A fair degree of colour retention was evident in this group with the high quality pigmentations.

Group F comprised the various blacks. The highest standards of gloss retention and resistance to other surface failures was always evident with black for any given medium. This was due to absorption of ultra-violet light by the pigments.

Group G comprised aluminium-type paints, a class which Mr Hipwood did not discuss further.

Group H comprised the strong yellows, based on light-fast chromes, arylamide and benzidine pigments. The gloss retention of colours in this group was in general second only to black, and a high standard of performance could be expected from the light-fast chromes even in industrial areas. The organic colours could be extremely disappointing, as a high concentration must be used for acceptable opacity and dilution with titanium dioxide was often necessary for colour matching purposes. Both operations reduce the standard of gloss retention and colour retention on exposure to weathering. The benzidine pigments were more durable when used alone than when reduced with titanium.

Types of alkyds. It was impossible to relate film performance directly to a given range of constants of an alkyd resin and considerable variations of performance have been observed even with resins of the same constants supplied from different manufacturers. Certain trends, however, could be observed.

Linseed, soya and tobacco seed oils were most common in paints supplied to specifications for decorative purposes, and whilst it was appreciated that the linoleic-rich types were to be preferred for non-yellowing purposes, all types were equally acceptable for natural weathering performance. Tung oil modifications, however, exerted a degrading influence with respect to surface deterioration by loss of gloss chalking and colour retention. Some evidence was also available which indicated that with frequent washing, say, once every three months, the recovery of gloss by removal of chalk was marginally less with the linoleic types of oil.

For specification work, medium oil length alkyds of 55 per cent to 65 per cent were preferred for spraying air-drying systems such as those used in vehicle finishing. These paints were, in general, superior for resistance to surface deterioration than the longer oil length 65 per cent to 72 per cent types developed for the decoration of buildings where application by brush was a main requirement. Oiling out (addition of oil to an already cooked alkyd) of an alkyd resin for improvement of application properties likewise had a degrading effect with respect to gloss retention and chalk resistance.

The natural weathering performance of many urethane modified alkyds, in comparison with more conventional types, had so far proved disappointing when considering gloss retention and chalk resistance. However, these modifications might upgrade the resistance of the film to cleansing fluids, etc.

A significant improvement had been observed with the use of co-polymerised silicone alkyd resins with a silicone content in the region of 30 per cent. These resins were commercially unattractive because of their high price, but practical field trials were at present in operation to establish the degree of improvement over conventional alkyd media when used for vehicle re-finishing. Recently introduced silicone modified polyurethane alkyds were only marginally better than urethane alkyds for gloss retention and chalk resistance, although other advantages had been claimed in respect of heat and water resistance.

Presence of extenders in top coat formulations. Improvements in the mechanical properties of paint films had been claimed with addition of extenders, but their incorporation into top coat formulations reduced the standard of gloss retention and chalk resistance.

Pigment/binder ratio. An increase in this ratio produced an inferior performance with respect to gloss retention and chalk resistance. Many high opacity white paints were, therefore, extremely vulnerable on exterior exposure, and severe chalking failures had been observed after weathering periods of only one year at Glascoed.

The influence of off-white undercoats in conjunction with bright red finishes. Pale undercoats had been proposed by manufacturers in order to enhance the appearance of films based on red dyestuff colours of low opacity (55 per cent contrast ratio). Natural weathering tests indicated that, in such systems, the rate of deterioration of the red finishing coat could be accelerated, presumably owing to inadequate absorption of energy by the top coat, the energy being reflected back by the "white" undercoat.

Mechanical properties of alkyd films

Checking and cracking failures were rare with conventional alkyd paint systems on steel, exposed in five year weathering tests. When failures occurred, they were usually worse with the washed areas of the exposed film. It had also been observed that there was a higher incidence of checking failures with strong red finishes. Tall oil alkyds and styrenated alkyds were slightly suspect with regard to checking failures occurring between three or four years of exposure. Cracking failures were always more advanced on wooden substrates.

Inter-coat behaviour of alkyd paint systems

Blister failures. It was an unfortunate fact that with the introduction of glossy alkyd based paints in replacement for the oleo-resinous types, the incidence of blistering had increased. Microwater type blisters (containing liquid and occurring between coats) were frequent causes of failure with alkyd systems under conditions of high humidity and wetness. Under drier conditions, most films made a sufficient recovery so that there was subsistence of the blisters. In certain cases, however, the pressures produced in the process of blistering were sufficient to extend the film of the top coat to beyond its elastic limit, and in such cases blisters, which might be of 1mm diameter or even greater, occurred and permanently marred the appearance of the film.

Flaking failure between coats may occur from sites of such blisters. Investigations carried out by Chemical Inspectorate several years ago indicated that magnesium carbonate as a main extender in undercoat formulations aggravated this phenomenon. There was, however, no clear evidence to suggest that the presence of other carbonate extenders or the double carbonate of dolomite extender in undercoats contributed to the incidence of blistering.

Flaking. Although under damp conditions alkyd films lost adhesion, their recovery was such that flaking failures were rare in three to four years of natural weathering tests. Flaking failures had, however, been observed with alkyd systems in which the type of undercoat used contributed to failure. Undercoats containing a high proportion of oil presented an excellent nutrient for supporting mould growth, and mould spores entering through burst microscopic blisters colonised in the undercoat layer with resultant flaking of the top coat from this layer. Exceptionally hard undercoats were equally undesirable and severe inter-coat adhesion failures had been noted in such systems.

Corrosion of metal substrates

With proper metal preparation and the adoption of the correct anticorrosive primer, corrosion failures with multi-coat alkyd systems were rare in five year natural weathering tests at Glascoed. It had been observed, however, that systems exhibiting heavy brush-marks act as water traps with resultant brush-line corrosion occurring during the latter stages of exposure. It was also observed that there were limitations in corrosion testing at Glascoed, owing to the rural nature of the site.

The lecture concluded with a series of slides illustrating the points made during the lecture and the natural weathering performance of several types of paint exposed in extremes of geographical environment. These included alkyds, epoxies, polyurethane, chlorinated rubber and emulsion based systems. In discussing these types of paint, Mr Hipwood suggested that it could be concluded that whilst the degree of chalking of the paint film varied with each type of formulation and with climatic exposure, the order of severity of exposure sites was not constant for all types of media, e.g., chlorinated rubber was severely degraded in a hot dry climate, whilst, in contrast, this climate had least effect on a two-pack polyurethane (aromatic isocyanate) film. In damper climates, the order of resistance of these two films was reversed. He also suggested that the best that could be achieved from accelerated tests was prediction of performance in one type of climate only.

He also said that B.S.3900, Part F3 cycle was reliable for the prediction of chalking and discoloration failures occurring on exposure in climates similar to that of the UK and Ireland, especially with the paints of colour group "C." This cycle, however, had limitations in the prediction of colour changes occurring with certain other pigments, and it was also weak for inducing checking and corrosion failures with systems exhibiting the defects on exposure outdoors.

Mr McWade, in opening the meeting for discussion, remarked on the endless scope for discussion which he felt had been opened by Mr Hipwood's lecture. He congratulated Mr Hipwood on his lecture and said that he was amazed to learn that Phoenix, Arizona, was a more severe site than Florida. Mr Hipwood then described the details of the exposure site at Arizona and contrasted that method with that of B.S.3900, Part F3.

When Mr Coleman asked for comments on urethane alkyd, urethane oil and two-pack (aliphatic) decorative enamels, he was told that the two-pack material was best. He was also informed that the silicone modified alkyds were better than the one-pack urethanes.

Mr O'Callaghan asked if a red lead/white lead formulation was a good primer. Mr Hipwood said that it was preferred to acrylic emulsion types for application

to wood. He also explained that red lead in oil was preferred for steel; this was tolerant to poor surface preparation, whereas the epoxy/red lead based paints were not.

Mr Sharpe was told that there was no advantage in adding optical whitening agents to the cheaper type titanium pigments as far as chalking resistance was concerned.

Others taking part in the discussion were Messrs. Sommerville, Moorish and Hanlon.

In proposing a vote of thanks, Mr A. Coleman said that Mr Hipwood's lecture was most interesting and comprehensive, and in a very short time he had covered the more important aspects of paint formulation and performance. Another important aspect of the lecture was the way in which it stimulated questions from the audience. Mr Coleman concluded by wishing Mr and Mrs Hipwood "Bon voyage." The lecture was attended by 24 members and seven visitors.

R.B.S.

London

Some aspects of the protection of exterior joinery

A meeting of London Section was held on 23 October 1969 at University College, London WC1, with Mr J. E. Pooley in the chair. Mr R. Dooper, of Verfinstitut TNO, presented the European Liaison Lecture, with the above title.

Mr Dooper said that exterior wood needed protection, which had traditionally been given by paint, but now preservative treatment was being used to an increasing extent. The shrinkage and swelling of wood as a result of changes in the moisture content was the major problem giving rise to cracking and peeling of the paint. In 1961, the specification on quality requirements for timber specified a water content not in excess of 17 per cent, but this could not always be met. Last year, a second edition called for a maximum of 14 per cent moisture at the surface and 18 per cent at the interior for first grade timber, and for a maximum of 21 per cent in second grade timber.

If the timber was dried artificially to a given level, then this level of water content must be maintained on the building site. One coat of primer was insufficient, and two coats were required. However, even after application of the full paint system, a complete seal was not obtained, because of the permeability of the paints to water vapour. Under normal conditions, the humidity was higher on the inside than on the outside, and, consequently, accumulation of water in the wood could be avoided if the permeability of the inside coats was less than that of the exterior.

Mr Dooper showed slides of a "blister hut" which illustrated clearly the effects of variation in the water vapour permeability of the films. The temperature gradient through the wood was also of importance, and a theoretical treatment had been applied. This indicated that the differences in water vapour transport were much greater for window frames (8cm) than for thin panels (1cm). The transport was also related to the season of the year. Even if one used thick films that were almost completely impermeable, e.g., chlorinated rubber or epoxy paints, temperature gradients within the wood could cause the moisture content on the cold side to exceed the safe level of 20 per cent, and would allow fungal growth to occur.

By the use of high standards for design and construction, and the choice of suitable paint systems, it should be possible to avoid wood decay. However, even then some decay occurred, possibly as a result of mechanical damage, or through the joints. It was, therefore, considered necessary to use a preservative treatment on the wood. This must not have an adverse effect on the paint, e.g., softening or staining.

A joint committee made up of representatives of the Foundation for Paint Application and the Centre for Wood Research had been set up some years ago, and was proceeding with an extensive programme of work. The main conclusion of this

committee was that the uptake of preservative by a series of test panels could vary considerably, depending on the amount of sapwood. Redwood showed the highest absorption and hence the greatest number of paint failures, while Swedish spruce was best. The moisture content of the wood was not of importance. Where there was excessive absorption, all pentachlorophenol based preservatives caused trouble. The use of tributyl tin oxide showed to advantage, provided that it contained no water-repellent material. As far as painting was concerned, zinc naphthenate caused less trouble than pentachlorophenol, but more than T.B.T.O. The main cause of failure with copper naphthenate was bleeding. If this was limited to the primer, then the system was satisfactory. With water soluble preservatives, there was swelling of the wood and the development of irregularities on the surface, which meant that filling operations had to be done more carefully.

The conclusion was that, of the three main factors involved, the wood, the preservative and the paint, the wood was the most important variable.

The paper was followed by an extensive discussion, after which a vote of thanks to the speaker was proposed by Mr G. L. Holbrow.

V.T.C.

Manchester

Particle size reduction in ball milling and sand grinding

A section meeting was held on Friday 14 November 1969 at the White Swan Hotel, Bolton, under the chairmanship of Mr W. F. McDonnell. The lecturer, Dr W. Carr, of Geigy (UK) Ltd., presented a lecture entitled "Particle size reduction in ball milling and sand grinding" to an audience of 136, another record attendance for a lecture in Bolton.

Dr Carr commenced by outlining the conditions and procedure for obtaining particle size distribution curves for pigments dispersed in a paint vehicle. Use was made of the ICI-Joyce LoebL disc centrifuge, taking a sample of pigment dispersion diluted to 0.4 per cent pigment with a solvent selected for freedom from attack on the "Perspex" rotor. The buffered line start technique was used to prevent streaming, and the amount of the various size fractions was determined by optical density measurements tailored to the type of pigment: this itself was selected to be non-bleeding in the solvents present in the paint system and during centrifugation.

The lecturer then presented a large number of size distribution curves for these pigments, demonstrating how the parameter varied as a function of duration of dispersion in ball milling and in sand grinding. He showed that for the mill base conditions chosen, sand grinding produced the finer size distribution, that both methods reached limiting values with longer dispersion times, but that neither dispersion method, with any pigment tested, produced ultimate dispersion. The latter conclusion was reached by converting the size distribution of the final dispersion into a pigment surface area and comparing this with the nitrogen surface area measured on the pigment powder. Dr Carr suggested that, since tinting strength increased as the proportion of finer particles was increased, much increased dispersion times might be an economic proposition with expensive pigments.

After a lively discussion period, the vote of thanks was proposed by Mr S. Duckworth.

D.A.P.

Midlands

Symposium—Management and training in the surface coatings industry

The above one-day Symposium was held by the Midlands Section on Thursday 23 October, with the Chairman, Mr D. J. Morris, presiding.

The morning session, on principles and techniques of management, contained

papers by Mr R. E. B. Perrigo, Director of the Small Business Centre at the University of Aston, entitled "The problems of management in the smaller business"; Mr P. K. Digby, General Manager, Management Economics Division, Production Engineering Research Association, on the "Application of advanced management techniques," and Dr G. DeW. Anderson, Director of the Paint Research Association on "Managing the research and development function."

The afternoon session was devoted to training in the surface coating and allied industries and papers were presented by Mr D. J. Worthy, Executive Officer, Chemical Industries Association Ltd., on "The development of training in the chemical industry," and by Mr C. G. Lebeter, Group Training Manager, Carrs Paints Ltd., on "The application of the Industrial Training Act to the surface coating and allied industries".

Mr Perrigo's paper stressed the importance of the small business to the economy as a whole and pointed out that such companies formed a large proportion of the sub-contractors to our big national industries, which, without the small business, would grind to a halt in a very short time. The advantage of the small companies lay in their capacity for flexibility and good internal communication, providing a climate in which initiative could thrive at all levels. The small businesses were particularly suited to the provisions of specialist products for which there was relatively small overall market, and in this field they were well able to compete favourably for quality, price and delivery with much larger companies.

The main problem encountered by the small company, however, was in the day to day pressure of top management, which often found itself unable to delegate sufficiently the day to day decision making to allow time for adequate long term planning. Such companies too often suffered from an imbalance in their resources between sales and production, or finance, from lack of control information and the lack of specialist support staff, such as cost accountants and production planners. Production knowledge was, unfortunately, often the only criterion in the engagement of staff for the smaller company. Training, together with the use of detailed job descriptions of the type encouraged by the Chemical Industry Training Board, could do much to correct these deficiencies, but for the smaller company, short courses were most suitable, as no one man could be spared for more than a short period at a time.

Mr Digby dealt in his paper with general management techniques and pointed out that these techniques must be chosen with due regard to the policy of the company and the external constraints prevailing in its business operations; for example, company policy in the larger company was formed by a team of managers, and this team should not be top heavy in technical experience. Delegated decision-making should be practised within the organisational structure as far below the policy making level as possible, and should be within carefully defined limits. Training in delegation, together with an appreciation of problems of others engaged in different departments, was necessary in order to create better understanding and co-operation. Management based on observation in the smaller company must give way to management based on recorded facts in the larger company.

Dr Anderson, in his paper on management of the research and development function, gave interesting examples of the application of modern management techniques in operation at the Paint Research Station, and it is hoped that this, and some of the other papers, will be published in the *Journal* in due course.

In the afternoon session on training, Mr Worthy traced the development of company training from the early materialistic or altruistic viewpoint to the modern aim of improving productivity and profitability. The purpose of the company training officer was to guide management in training opportunities and it was the responsibility of the management in each section to decide what training should be undertaken.

Management training and development was the most important of all forms of training, and it was necessary for managers to ensure the thorough training of their

subordinates and successors. It was essential to know and understand the company development and objectives and to identify job requirements. The primary function of the Industry Training Board was to act as an adviser on training rather than a dispenser of money.

Mr Lebeter, in his paper, described the methods of company training used within the Carr's Paints Group. He recommended the appointment of a training officer, preferably from outside the company, who could analyse the company's needs and assets as well as its training priorities.

In the laboratory, internal training courses were run taking advantage of the assistance of raw material suppliers lecturing in their own specialist fields. For junior assistants, a six month internal training course was run, weeding out the unsuitable entrants in various stages. For management training, external courses were more suitable, and management games were often used to good effect for sales staff. On the production side, equipment had been carefully designed to reduce menial routine tasks to a minimum.

A.E.C.

Newcastle

Statistical techniques in the optimisation of paint formulation—by computer

The first meeting of the current session was held in the Swallow Hotel, Newcastle, on Thursday 2 October 1969. Mr D. W. Brooker, of Shell Research Limited, presented a paper entitled "Statistical techniques in the optimisation of paint formulation—by computer."

Mr Brooker discussed two techniques for the investigation of the variables influencing the performance of an epoxy/polyamide based paint which had been used in the Shell laboratories. The Simplex technique, which is a simple non-mathematical approach to optimisation of paint formulation, was discussed first. Mr Brooker went on to say that the Simplex technique did have particular limitations in that it was a slow method for tests which took a long time to carry out, such as salt spray, and was more suited to work involving the assessment of only one index of performance.

A more sophisticated method was also employed in this work, namely, a Rotatable Central Composite design (RCC). This provided a considerable volume of information together with an estimation of experimental error, and the results obtained were suitable for analysis by computer.

Mr Brooker concluded by saying that the RCC design technique was successful in predicting an optimum area of formulation. With the formulations tested, for example, the level of polyamide and the PVC were significantly different to those values which had previously been accepted as approaching the optimum. The testing of the predicted formulation did, however, confirm the high level of performance.

After question time, in which Messrs. James, Tate, Dowling, Farrow and Davison took part, the meeting was closed with a vote of thanks to Mr Brooker by the Chairman, Mr Tate.

A.L.



Technical Exhibition

Lord Sherfield to open Exhibition

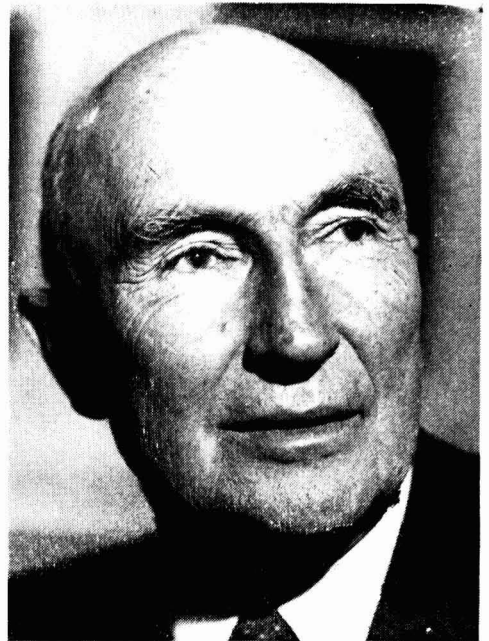
The Exhibition Luncheon will be held at 12.45hr on Monday 27 April 1970, at the Savoy Hotel, London WC2.

Lord Sherfield, GCB, GCMG, President of the Parliamentary and Scientific Committee, has accepted the Exhibition Committee's invitation to be the Guest of Honour at the Luncheon and to reply to the Address of Welcome given by the President, Mr A. S. Fraser. Lord Sherfield, a former Ambassador to the United States and Chairman of the United Kingdom Atomic Energy Authority from 1960 to 1964, is also Chairman of the Industrial and Commercial Finance Corporation and Chairman of the governing body of Imperial College for Science and Technology.

Principal officers of other scientific bodies, industrial research associations and organisations representing the interests of suppliers and consumers have been invited by the Committee to attend.

At 15.00hr on the same day, the Opening Ceremony will be performed by Lord Sherfield in the Empire Hall at Olympia.

A further application has been accepted by the Exhibition Committee for the Exhibition. The list of exhibitors



Lord Sherfield, GCB, GCMG

is to be amended as follows:

Delete Joyce Loebel and Co. Ltd.

Add Winn and Coales Ltd.

The number of companies which have never shown at previous OCCA Exhibitions has now increased to 11, whilst 23 did not show at the 1969 Exhibition.

There will be 107 stands, including exhibitors from 11 overseas countries—Belgium, Denmark, Finland, France, Germany, Holland, Italy, Norway, Sweden, Switzerland and United States of America.

Copies of the "Official Guide," which is planned for publication at the beginning of February, will be distributed free of charge to chemists and technologists in Europe and to all members of the Association, wherever resident. Anyone wishing to have a copy should

apply to the Association's offices. Copies will also be available free of charge to visitors at the entrance to the Exhibition, together with a folder for technical literature and a personal identification badge.

Information leaflets in six languages will be available prior to the Exhibition, and will be sent to paint manufacturing companies in Europe and the United Kingdom. Anyone requiring copies to send to colleagues overseas should apply to the Association's offices.

Scottish Symposium and Association AGM

Dispersion in theory and practice

The programme for the Symposium "Dispersion in theory and practice," to be organised by the Scottish Section at East Kilbride, near Glasgow, in conjunction with the Association's Annual General Meeting, has now been arranged.

Careful selection of lecturers and subjects has been made, so that the Symposium committee's stated object, that of bringing together research workers carrying out fundamental studies and technologists and managers concerned with dispersion as an industrial operation in a commercial context, can best be achieved.

A panel of speakers of recognised authority has been assembled to cover the various aspects of the subject. After each lecture, about thirty minutes has been allocated for discussion, and at the close of the Symposium, an overall summary will be made, and a longer and more general discussion will take place.

The detailed programme is given below.

Thursday 21 May

- 10.30 Opening address.
11.00 "Some aspects of pigment dispersion, with particular emphasis on carbon black," by Mr W. M. Hess and M. D. Garret, of Columbian Carbon Co. Ltd.

- 12.30 Lunch.
14.00 "Dispersion in aqueous media" by Dr W. Carr, of Geigy (UK) Ltd.
15.30 "The influence of non-aqueous media on dispersions," by Mr K. Pond, of Lorilleux & Bolton Ltd.
17.30 Annual General Meeting of the Association.

Friday 22 May

- 09.30 "Some aspects of dispersion in relation to titanium dioxide," by Dr R. Amberg, of Kronos Titanium Pigments Ltd.
11.00 "The influence of interfacial activity in paint films on their properties," by Dr W. Funke, of the Forschungsinstitut für Pigmente und Lacke EV.
12.30 Lunch.
14.15 "Dispersion machinery" by Dr J. B. Slinger of ICI Ltd.
15.45 Summing up by Dr S. H. Bell, of the Paint Research Association, followed by a general discussion.

All lectures will be held in the Ballerup Hall, in the Civic Centre, East Kilbride. Lunch will be served to all registrants in the Bruce Hotel, East Kilbride. Morning and afternoon coffee will also be supplied.

Association AGM and Dinner

The Association's Annual General Meeting will be held in the Ballerup Hall at 17.30 on Thursday 21 May, and will be followed (19.30 for 20.00) by a dinner at the Bruce Hotel. All registrants, whether members of the Association or not, will be welcome at the dinner, the charge for which will be £2 10s inclusive of wines. Dress will be informal.

Accommodation

All bedrooms at the Bruce Hotel have been reserved for the Symposium for the nights of Wednesday 20 and Thursday 21 May, and the charge has been agreed at 55s per night for bed and breakfast. Participants wishing to extend their stay over the weekend may do so at the same rate, if the reservation is made on the Symposium reservation form.

Arrangements have also been made to provide additional accommodation in other hotels of comparable standard within easy reach of the Ballerup Hall, should bookings exceed the capacity of the Bruce Hotel. Payment in all cases will be direct to the hotel in question.

Transport

East Kilbride is within easy reach of Glasgow, local trains departing at regular intervals from Glasgow Central Station, which is close to the terminus for airport coaches in St. Enoch Square, and thus convenient for both air and rail travellers. If there is sufficient demand, the Bruce Hotel will provide transport direct from Glasgow Abbotsinch Airport.

Exhibition

A small exhibition of the latest developments in industrial dispersion equipment will be held in the foyer of the Ballerup Hall during the Symposium.

Social Programme

A programme of outside activities has also been arranged, primarily for the benefit of the families of delegates. All-day tours, to the Loch Lomond area on the Thursday, and to Edinburgh on the Friday, will take place, and a conducted tour of the East Kilbride fashion goods factory of Lerosé is to be organised. Numbers on some excursions may have to be limited, and applications should be made as early as possible to ensure a place.

All delegates and their guests will have the use of the excellent local golf course, and it will be possible for those interested to visit the National Engineering Laboratory at East Kilbride.

Fees

Fees for the Symposium, which will include a set of short summaries of all the papers to be presented, will be as follows:

Members	£5
Student Members	£1 10s
Non-members	£7 10s

Application forms are being dispatched to all UK, Irish and General Overseas Members, and any other interested person should contact the Symposium Secretary, Mr I. R. McCallum, P. W. Hall Ltd., Woodilee Industrial Estate, Kirkintilloch, Glasgow.

Association Dinner Dance 1970

Council has approved the arrangements for the Association's Biennial Dinner Dance, to be held at the Savoy Hotel, London WC2. This will be held on Friday 29 May with the reception at 7.00 p.m. prior to dinner at 7.30.

After dinner there will, on this occasion, be three short speeches, so that dancing to the Jerome Orchestra may begin as soon as possible, and this will continue

until 1 a.m. There will be an interval at 11.30 p.m., for refreshments, and a cabaret has been arranged to take place at this time also.

Applications for tickets (at £4 4s 0d each) are being circulated to Members early in January, and non-members wishing to attend should apply to the Director and Secretary, at the Association's offices.

Association Conference, 4-8 May 1971

~~Surface~~ properties and appearance

Council has decided that the theme of the 1971 Association Conference shall be "Surface Properties and Appearance." It is intended to hold four technical sessions, each of three papers, on the three mornings of the Conference and one afternoon. The other two afternoons will be devoted to workshop sessions and the Annual General Meeting of the Association. It is also intended, on this occasion, to hold a session on "The Management of Innovation," to run parallel with the afternoon technical session.

Submission of papers for the technical sessions

An invitation is now extended to any person, whether or not a member of the Association, who feels that a report of his work in this field may be suitable to be one of the 12 technical papers, to submit a synopsis (of approximately 250-500 words) to the Honorary Research and Development Officer of the Association (Mr A. R. H. Tawn, 34 Crest View Drive, Petts Wood, Kent) **not later than Friday 3 April 1970**. A few authors have already been invited to submit papers, but the remainder will be chosen from those synopses received. Papers selected for presentation at the Conference will be required in final draft

by 1 September 1970. It will be appreciated that, on this occasion, the Association's Conference is being held earlier than usual, i.e. 4-8 May 1971, and it is necessary to prepare full pre-prints to be sent to all those who have registered at least one month before the conference.

It will be recalled that it is the custom at the Association's Conferences for the authors merely to outline their papers, highlighting points of interest, and for a general discussion of the paper to follow. The author will not be expected to deliver the paper *in toto*, since the object of sending out the pre-prints in advance is that delegates may read these thoroughly before the Conference.

Venue

The venue for the Conference will be the Palace Hotel, Torquay, which has been the venue for three previous Association Conferences, in 1957, 1961, and 1965.

Full details concerning the registration fees, and a form of application, will be sent to all Members of the Association before the end of this year; non-members wishing to receive these details, when available, should apply in writing to the Director and Secretary at the Association's offices.

Manchester Section

Annual Dinner Dance

The Section Annual Dinner Dance was held on Friday 24 October, in the Peacock Suite at the Piccadilly Hotel, Manchester, in the presence of the Chairman, Mr W. F. McDonnell, and Mrs McDonnell. Some 400 members, guests and ladies were present but, regrettably, the President, Mr A. S. Fraser, and Mrs Fraser were unavoidably absent. The principal guest was Mr W. J. Green, Managing Director of Walpamur Limited, with Mrs Green, and the other

guests were Section Chairmen: Mr D. J. Morris (Midlands) and Mrs Morris, Dr L. J. Watkinson (West Riding) and Mrs Watkinson, Mr K. R. W. Chitty (Thames Valley) and Mrs Chitty and Mr J. R. Taylor (Bristol) together with Mr R. H. Hamblin (Director and Secretary).

After a meal which was a tribute both to the Hon. Social Secretary's epicurean sensibility and the ability of the hotel's catering staff, Mr Green rose to propose the toast to the Manchester Section.



At the Manchester Dinner Dance (left to right) Mrs McDonnell, Mr W. F. McDonnell (Chairman, Manchester), Mrs Green, Mr W. J. Green (Managing Director, Walpamur Limited)

After stating that he was more used to listening to after dinner speeches than delivering them, the speaker continued to make a well constructed commentary on some aspects of present day social and political behaviour. Only in the case of one anecdote, concerned with reliable, presentable packaging, was it necessary to refer to its origin, a dinner at which the speaker had been a guest.

Mr McDonnell followed in response and in proposing the toast to the guests, a duality of role which he claimed to be unique, but which this reporter must deny¹. During a delivery which was a happy blend of the witty and the serious, the Chairman referred to the highly

successful symposium held in Manchester in September and thanked the Committee members who were responsible for its organisation. He also referred to the role of OCCA in a changing world and, while regretting a certain lack of interest among the younger technical men in the industry, pledged his and the Section's full co-operation in correcting this state of affairs. In thanking the ladies for their presence, Mr McDonnell referred to a young lady of Kent who may have been a member of the London Section.

Dancing then commenced, to a band whose output might have been measured in megawatts, and who added considerably to the pleasure of the evening by their methods of awarding spot prizes.

Reference

1. *JOCCA* 1968 51, 95.

Midlands Section

Trent Valley Branch

The venue for Mr F. K. Daniel's lecture on "The operating and design principles of high-speed dispersers," has now been

arranged. The meeting will be held in the Main Lecture Theatre T1, Applied Science Building, Nottingham University, on 23 April, at 6.30 p.m.

City and Guilds of London Institute examination results

Congratulations are extended to the following Members of the Association upon their success in the recent City and Guilds of London examinations. The class of membership and the section to which the member is attached are shown in italics.

Paint Technician's Certificate Part I

N. Harding (*Student-Manchester*)
M. Smith (*Student-West Riding*)
A. L. Spencer (*Associate-Manchester*)

Paint Technician's Certificate Part II

M. Baker (*Student-West Riding*)
T. A. Chessman (*Student-London*)
R. Cobb (*Student-London*)
M. J. Conroy (*Student-Midland*)
P. R. Davis (*Student-Thames Valley*)
A. J. Durdey (*Ordinary-Newcastle*)
N. R. Eatough (*Ordinary-Manchester*)
V. Handforth (*Student-Manchester*)
K. Henderson (*Ordinary-Manchester*)
E. A. Hobday (*Associate-Newcastle*)
D. Howell (*Student-London*)
J. S. Lyne (*Student-Midland*)
L. Morpeth (*Student-Manchester*)
W. Postma (*Associate-Newcastle*)
P. M. Proudley (*Ordinary-West Riding*)
I. Riddell (*Student-Newcastle*)
P. J. H. Watson (*Ordinary-Newcastle*)
B. D. Yellop (*Student-London*)

Paintmaker's Certificate

A. W. T. McNaughton (*Student-Scottish*)
A. Robertson (*Student-Scottish*)

News of Members

Mr D. J. Loader, an Associate Member attached to the London Section, has been appointed as Technical Sales and Service Consultant by Blundell-Permoglaze Ltd., Decorative Division. Mr Loader, who was previously the company's technical adviser for the South, will now operate on a national basis.

Dr F. H. Kroch, an Ordinary Member attached to the Manchester Section, has been elected to the Council of the Chemical Industries Association.

Mr R. H. Sudekum, an Ordinary Member attached to the London Section, has been appointed as Manager of the new product group recently introduced by the 3M Company Limited to handle its encapsulated products. Mr Sudekum will be responsible for both technical and sales aspects of the products, which involve the capturing of fragrances of various substances, e.g. cosmetics, by encapsulation, and coating on to paper.

IUPAC Report

A report on the work of the International Union of Pure and Applied Chemistry, from 1957 to 1967, written by Mr W. Klemm, the President of IUPAC from 1965 to 1967, has recently been published.

Copies of the 20-page booklet are available on request from the Executive Secretary of The Royal Society.

Eighth International Symposium on Gas Chromatography

The eighth International Symposium on Gas Chromatography, organised by the Gas Chromatography Discussion Group of the Institute of Petroleum in co-operation with the Institute of Chemistry of Ireland, will be held in Dublin, on the premises of the Royal Dublin Society, from 28 September to 2 October 1970. An exhibition of chromatographic equipment will be held in conjunction with the symposium.

The full technical programme will be available early in 1970, and a social programme will be arranged.

Attendance at the symposium will be limited to 600. Fuller details are available from the Executive Society of the Organising Committee, at the Institute of Petroleum.

Register of Members

The following elections to membership have been approved by Council. The Sections to which the new members are attached are given in *Italics*.

Ordinary Members

- ANDREW, JAMES, 5 Blairlogie St., Ruchazie, Glasgow E3, Scotland. (*Scottish*)
 CHRISTENSEN, KNUD, DrTechn.HC, Sadolin & Holmblad Ltd., Holmbladsgade 70, DK—2300 Copenhagen, S. Denmark. (*Overseas*)
 GRAHAM, GEORGE, 15 Valleyfield St., Glasgow N1, Scotland. (*Scottish*)
 MEYER, URS THEODOR, 16 The Holdings, Hatfield, Herts. (*London*)
 MILNE, DAVID GRAEME, PhD, BSc, British Titan Products Co. Ltd., Billingham, Teesside. (*Newcastle*)
 PAN, QUONII KUN-YU, Rue General Severiano, 40 Apto. 408, Botafogo, Rio De Janeiro, Brazil. (*Overseas*)
 PANG, CHAN TING, 200 Thomson Road, Singapore 11, Rep. of Singapore. (*Overseas*)
 SCHIERBAUM, HELMUT, Hughs' Tower, 224 The Hides, Harlow, Essex. (*London*)
 TAI, CHEY-MING, No. 22 Chung Shan N. Road, Sec. 3 Taipei, Rep. of China. (*Overseas*)
 WHITE, DAVID JAMES, BScChem., 71 Newbury Lane, Silsoe, Bedford. (*London*)

Associate Members

- TRAYNOR, JOSEPH JACKSON, 114 Cuikew Terrace, Penicuik, Midlothian, Scotland. (*Scottish*)

Student Members

- HILLIS, PETER LEWIS, 2315 Dumbarton Road, Yoker, Glasgow W4, Scotland. (*Scottish*)
 HILL, HUGH DANIEL, Flat 20/3 33 Petershill Drive, Balornock, Glasgow N1, Scotland. (*Scottish*)
 KENNEDY, SAMUEL, 40 Southdeen Avenue, Drumchapel, Glasgow W5, Scotland. (*Scottish*)
 LAIRD, DAVID ALEXANDER, 17 Leyden Gardens, Glasgow NW, Scotland. (*Scottish*)
 MCGUIRE, DAVID STEWART, 20 Glenpark Avenue, Thornliebank, Glasgow, Scotland. (*Scottish*)
 SMITH, DAVID MARTIN, 3 Arundel Drive, Bishopbriggs, Glasgow, Scotland. (*Scottish*)
 YOUNG, CRAIG, 28 Melrose Avenue, Rutherglen, Glasgow, Scotland. (*Scottish*)

Forthcoming Events

Details are given of meetings in the United Kingdom up to the end of the month following publication, and in South Africa and the Commonwealth up to the end of the second month.

Monday 5 January

Hull Section. "Paint Production Today" by Mr R. Ward of Torrance & Sons Ltd., to be held at the Bullock Lecture Theatre, Hull College of Technology, at 7.00 p.m.

Wednesday 7 January

London Section. Joint Meeting with The Colour Group (Great Britain) and the London Region, Society of Dyers and Colourists. "Instrumental shade passing" by Mr K. McLaren of ICI Ltd., and "Visual and instrumental colour tolerances in plastics" by Mr R. Best and Miss S. R. Williams of BIP Chemicals Ltd., to be held at the Physics Building, Imperial College, Prince Consort Road, South Kensington, London SW7, at 6.00 p.m.

Thursday 8 January

Newcastle Section. "The Prevention of Bacterial Corrosion of Mild Steel with Paint Films" by Mr A. V. Robinson of Camrex Ltd., to be held at the Royal Turks Head Hotel, Grey Street, Newcastle upon Tyne, at 6.30 p.m.

Friday 9 January

Manchester Section. "Management in R. & D." by Mr A. W. Pearson of the Manchester Business School, to be held at the Manchester Literary and Philosophical Society, Manchester 1, at 6.30 p.m.

Tuesday 13 January

West Riding Section. "The Overpainting of Preservative Treated Wood" by R. W. Watson of Hickson & Welch (Holdings) Ltd., to be held at the Griffin Hotel, Boar Lane, Leeds 1, at 7.30 p.m.

Wednesday 14 January

Manchester Section—Student Group. "Metal Organic Compounds in Surface Coatings," by Mr W. K. H. Lakin of Hardman & Holden Ltd., to be held at the Manchester Literary and Philosophical Society, Manchester 1, at 4.30 p.m.

Thames Valley Section. "Thermosetting Acrylic Resins—Recent Applications" by Mr J. R. Taylor of BP Chemicals (UK) Ltd., to be held at the Manor Hotel, Datchet, Bucks, at 7.00 p.m.

Thursday 15 January

Scottish Section. "Management by Objectives in Research and Development" by Mr E. W. Huggins, of Urwick Technology Management Ltd., to be held at the St. Enoch Hotel, Glasgow, at 6.00 p.m.

Friday 16 January

Midlands Section. Winston Restaurant Dinner Lecture. "The Significance of Particle Size, Shape and Packing in Emulsion Paints" by Dr C. Bondy of Revertex Ltd., to be held at the Winston Restaurant, Birmingham, at 6.30 p.m.

Scottish Section. Annual Dinner Dance, to be held in the Kintyre Suite, Central Hotel, Glasgow, at 6.30 p.m.

Saturday 17 January

Scottish Section—Student Group. "Fire Prevention and Factory Safety" by a speaker to be arranged, to be held at the St. Enoch Hotel, Glasgow, at 10.00 a.m.

Thursday 22 January

Midlands Section—Trent Valley Branch. "Oil-Free Alkyds" by Mr A. G. North of Cray Valley Products Ltd., to be held at the British Rail School of Transport, London Road, Derby, at 6.30 p.m.

Friday 23 January

Irish Section. "Industrial Effluent" by Dr Dickinson of the Institute of Industrial Research and Standards, to be held at the Clarence Hotel, Wellington Quay, Dublin, at 8.00 p.m.

Tuesday 27 January

Irish Section. Annual Dinner Dance, to be held at the South County Hotel, at 8.00 p.m.

Wednesday 28 January

Scottish Section—Eastern Branch. Brains Trust—"Problems of Paint Making and Applications" by Mr E. A. Bullions, Mr C. G. Cochrane and Mr H. Muirhead, to be held at the Carlton Hotel, North Bridge, Edinburgh 1, at 7.30 p.m.

Friday 30 January

Bristol Section. "Colloids in Emulsion Paints" by Mr A. Reveley of British Celanese Ltd., to be held at the Royal Hotel, Bristol, at 7.15 p.m.

Monday 2 February

Hull Section. "Theory of emulsion polymerisation" by Mr D. Kinsler of Dunlop Ltd., to be held at the Bullock Lecture Theatre at Hull College of Technology, at 7.00 p.m.

Thursday 5 February

Newcastle Section. "Functional coatings for the packaging industries" by J. Howarth-Williams of Swale Chemicals Ltd., to be held at the Royal Turks Head Hotel, Grey Street, Newcastle, at 6.30 p.m.

Friday 6 February

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

Tuesday 10 February

London Section—Southern Branch. "Recent developments in polyurethanes" by a speaker from Bayer Chemicals Ltd., to be held at the Pendragon Hotel, Clarence Parade, Southsea, at 7.00 p.m.

West Riding Section. Forum on "Primers for metal" with three speakers: Mr H. F. Clay of Cromford Colour Company Ltd. discussing zinc chromate, Mr H. G. Walker of Associated Lead Ltd. discussing red lead and calcium plumbate, a speaker from Imperial Smelting Co. Ltd. discussing zinc phosphate. To be held at Griffin Hotel, Boar Lane, Leeds 1, at 7.30 p.m.

Wednesday 11 February

Newcastle Section—Student Group. "Zinc pigmented silicate coatings" by Mr T. Harbottle of Durham Chemicals Ltd., to be held in Lecture Theatre A406, Rutherford College of Technology, Newcastle, at 3.00 p.m.

Thursday 12 February

London Section. "New resin systems for use in coil-coating application" by L. A. Tysall of Shell Research Ltd., to be held at the New Engineering Block, University College, London WC1, at 6.30 p.m.

Scottish Section. "Any questions," Dr D. Atherton—Craig Hubbuck Ltd., Mr J. W. Davidson—Federated Paints Ltd., Mr E. W. Huggins—Urwick Technology Management Ltd., Mr C. Stephenson—Thomas Hinshelwood & Co. Ltd., to be held at St. Enoch Hotel, Glasgow, at 6.00 p.m.

Friday 13 February

Manchester Section. "Does the paint industry deserve a research association?" by Dr G. De Winter Anderson of the Paint Research Station, to be held at Manchester Literary and Philosophical Society, 36 George Street, Manchester 1, at 6.30 p.m.

Saturday 14 February

Scottish Section—Student Group. Annual General Meeting followed by a lecture on the manufacture of golf balls, to be held at the St. Enoch Hotel, Glasgow, at 10.00 a.m.

Thursday 19 February

Thames Valley Section. "Polishes" by Mr E. M. Clough of S. C. Johnson Ltd., to be held at the Manor Hotel, Datchet, Bucks, at 7.00 p.m.

Friday 20 February

Irish Section. "An examination of aqueous interior semi-gloss paints and the development of an aqueous semi-gloss vehicle" by Mr R. Ficken of Lening Chemicals Ltd., to be held at the Clarence Hotel, Wellington Quay, Dublin, at 8.00 p.m.

Midland Section. "Corrosion problems in highway research," by Dr R. R. Bishop of the Road Research Laboratories, to be held at Chamber of

Commerce House, 75 Harbourne Road, Birmingham 15, at 6.30 p.m.

Newcastle Section. "Ladies Night," at the County Hotel, Newcastle.

Wednesday 25 February

Scottish Section—Eastern Branch. "Ink and paper relationship" by a speaker from the Printing Ink Research Association, to be held in the Carlton Hotel, North Bridge, Edinburgh 1, at 7.30 p.m.

Friday 27 February

Bristol Section. "Lithographic printing inks" by Mr A. Stone of Redcliffe Inks Ltd., to be held at the Royal Hotel, Bristol, at 7.15 p.m.

Oil and Colour Chemists' Association

President : S. R. FINN, B.Sc., F.R.I.C.

The Oil and Colour Chemists' Association was formed in 1918, to cover paint, printing inks, pigments, varnishes, drying and essential oils, resins, lacquers, soaps, linoleum and treated fabrics, and the plant, apparatus and raw materials useful in their manufacture. In 1924 it absorbed the Paint and Varnish Society. The stated purpose of the Association is to promote by discussion and scientific investigation the technology of the industries concerned with the above-mentioned products, and to afford members opportunity for the interchange of ideas. This is achieved by the regular holding of ordinary meetings at which papers are presented, and the organisation of annual technical exhibitions, biennial conferences, educational activities and practical co-operative experimental work. Details of these activities are given in the *Journal of the Oil and Colour Chemists' Association*, which is published monthly, and whose pages are open to receive communications and other pronouncements on scientific and technical matters affecting the members of the Association and the industries concerned. The Association's meetings also afford opportunities for members to meet informally and socially.

There are Sections of OCCA in Auckland, Bristol, Hull, Ireland, London, Manchester, the Midlands (with a Trent Valley Branch), Newcastle upon Tyne, Scotland (with an Eastern Branch), South Africa (with Branches in the Cape, Transvaal and Natal), Thames Valley, Wellington, and the West Riding, and these are responsible for the conduct of their own local affairs. There is also a General Overseas Section. There is also a close alliance between the Association, the Federation of Societies for Paint Technology in the United States, and the Fédération d'Associations des Techniciens de l'Industrie des Peintures, Vernis, Emaux et Encres d'Imprimerie de l'Europe Continentale (FATIPEC). The Association also maintains cordial relations with the Scandinavian Federation of Paint and Varnish Technicians (SLF).

The five Sections previously maintained by the Association in Australia formed (1.1.68) the Oil and Colour Chemists' Association Australia, having the same aims and activities as, and working in close liaison with, the parent body.

Ordinary Membership is granted to scientifically trained persons, and Associate Membership to others interested in the industries covered. Student Membership, which is intended primarily for students, is open without restriction to persons under the age of 21 and to those up to 25 who are following a course of technical study. The annual subscription in each case is five guineas, except for Student Members whose subscription is one guinea. An entrance fee of 10s. is payable by all members. Applications for membership are invited from suitably qualified persons who are engaged or otherwise interested in the industries noted above. Applications, which should be supported by two members of the Association (one of whom must be an Ordinary Member), should be forwarded to the Director & Secretary at the address given below. Application forms and full details of membership may be obtained from the offices of the Association.

PUBLICATIONS

Journal of the Oil and Colour Chemists' Association. Published monthly. Subscription rate to non-members in UK and abroad, £7 10s. p.a. post free ; payable in advance.

An Introduction to Paint Technology (Second Edition with additional chapter). Pp. 187, illustrated, with index, £1 (including postage).

Paint Technology Manuals

Part 1 : "Non-convertible Coatings," Second Edition, Pp. 343, 36s.

Part 2 : "Solvents, Oils, Resins and Driers," Second Edition, Pp. 268, 36s.

Part 3 : "Convertible Coatings," Pp. 318, 35s.

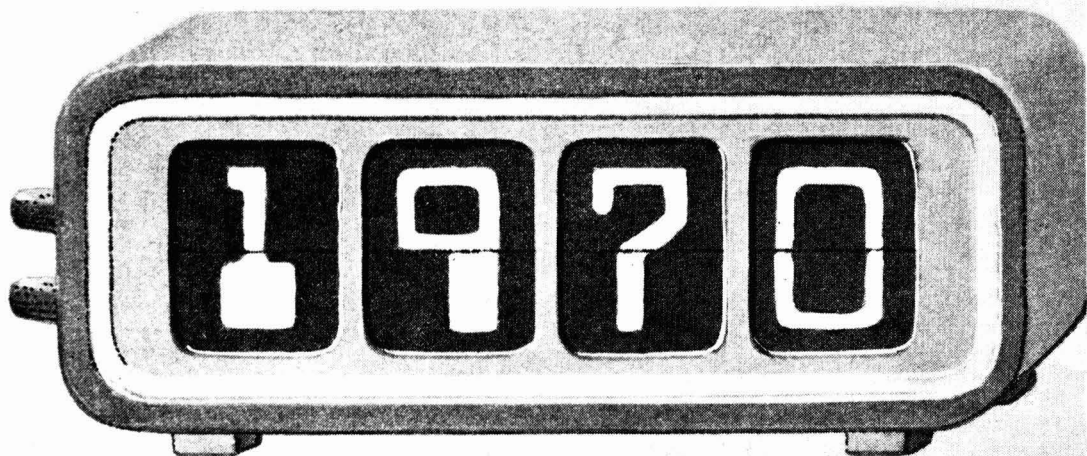
Part 4 : "The Application of Surface Coatings," Pp. 345, 35s.

Part 5 : "The Testing of Paints," Pp. 196, 35s.

Part 6 : "Pigments, Dyestuffs and Lakes," Pp. 340, 35s.

Director & Secretary : R. H. Hamblin, M.A., F.C.I.S., F.C.C.S., Wax Chandlers' Hall, Gresham Street, London, E.C.2.

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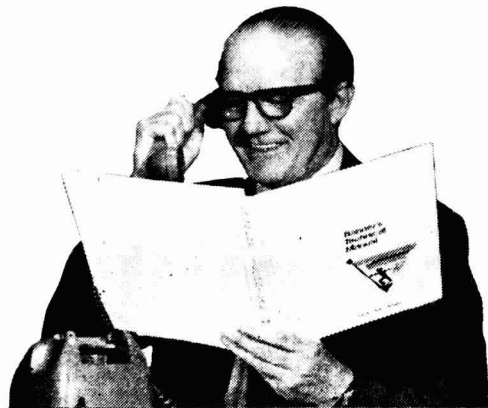
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Oil & Colour Chemists' Association

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Association Dinner Dance 1970

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Oil and Colour Chemists'
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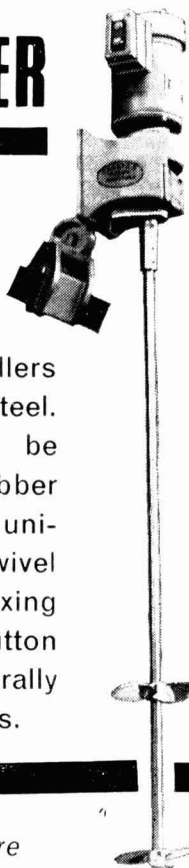
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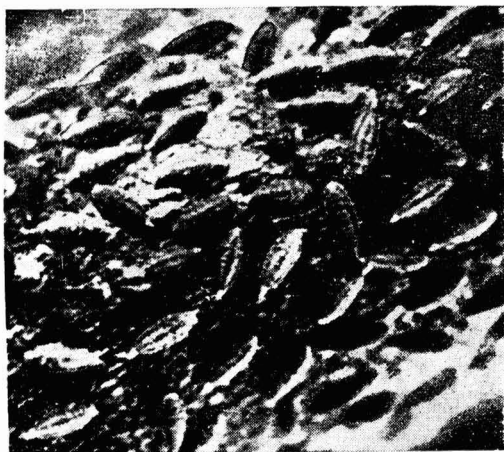
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