





J O C C A / Vol. 60 No. 1

JOCCAB 60(1) 1-42 (1977)

January 1977

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OIL&COLOUR CHEMISTS ASSOCIATION

OURNAL OF THE IL & OLOUR HEMISTS' SSOCIATION

Molecular structure and film properties of alkyd resins

G. Walz

Recent aspects on the preparation and evaluation of some polyesteramides for surface coatings. Part I: Parameters affecting the formation of various dihydroxy-diethylamide derivatives of fatty acids *A. M. Naser, M. A. El-Azmirly and A. Z. Gomaa*

Surface films produced by electrostatic powder deposition A. W. Bright

Colour, structure and electrons—a student review C. H. J. Wells



See page 40 for details

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The Association's twenty-ninth Technical Exhibition will be held at Alexandra Palace, London N22, from 22 to 25 March 1977. Further details regarding the arrangements for OCCA-29 and the many facilities offered at Alexandra Palace appear on pages 38 and 39 of this issue of the *Journal*.

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

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

"Official Guide"

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

This unique publication will be circulated to all members of the Association, both at home and overseas, and as far as possible to all consuming companies in Great Britain. Copies will also be sent to those applying as a result of the Association's wide-spread publicity. The advantages of an advertisement in the *Official Guide* need hardly be stressed, since it will contain details of each Exhibitor's stand and thus constitute a summary of those recent technical developments which will be shown; consequently, it will be read widely and kept for reference.

For details of advertising in this and other Association publications, write to the Assistant Editor at the address on the Contents page of this issue.

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The optional Professional Grade for Ordinary Members of OCCA was instituted by Council in 1971. Since that time, over 400 candidates have been successful in their applications to join the Professional Grade and a full list was published on page 459 of the December 1976 issue of the *Journal*. The various routes to the three Grades (see above) in diagrammatic form and the full regulations for admission are published in the July 1976 issue.

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

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Journal of the Oil and Colour **Chemists'** Association

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FTSC FELLOW (OF THE OIL AND COLOUR CHEMISTS' ASSOCIATION) IN THE TECHNOLOGY OF SURFACE COATINGS

ATSC Associate (of the Oil and Colour Chemists' Association) in the Technology of Surface Coatings

LTSC LICENTIATE (OF THE OIL AND COLOUR CHEMISTS' ASSOCIATION) IN THE TECHNOLOGY OF SURFACE COATINGS

Full details of the regulations for admission to the Professional Grade were last published in the July 1976 issue of the *Journal* (*JOCCA*, 1976, **59**, 264). They may also be obtained from the Association's offices by sending a stamped self-addressed envelope marked "P. G. leaflet" in the top left-hand corner. The address of the Association's headquarters, and the telephone and telex numbers are printed above.



OIL & COLOUR CHEMISTS' ASSOCIATION

CURRENT PUBLICATIONS

Introduction to Paint Technology

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

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

Paint Technology Manuals: Part Seven "Works Practice"

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

Price: £3.00

Paint Technology Manuals: Part Three "Convertible Coatings"

Part Three of the Association's series of Paint Technology Manuals has been reprinted as a second edition in 1972 and a limited number of copies are still available. Price: £2.80

Ultraviolet Polymerisation

A volume, bound in limp covers, comprising papers presented at a symposium of the Newcastle Section of the Association in 1975 and subsequently published in the *Journal*.

Price: £5.00

Conference Preprints

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

The 1973 Conference was held at Eastbourne with the theme "Towards 2000" and the bound set of preprints for this Conference includes 16 papers all dealing with various aspects of the surface coatings industry towards the year 2000.

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

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

The Association's 1977 Conference will be held once again at Eastbourne; further details are given on page 40 of this issue.

History of the Association

As part of its 50th Anniversary celebrations the Association published in 1968 "A Fascinating Story—the History of OCCA 1918-1968". This is an illustrated book comprising 89 pages and bound in hard covers. Price: £1.50

Journal of the Oil and Colour Chemists' Association

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

Price: £25.00 (\$50.00) per year

Back issues

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

Price: £2.50 (\$5.00) each

Consolidated Indexes

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

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

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

Price: Consolidated Indexes for 1966-1975-£5.00 (The three indexes for 1918-1965-£1.50 each.)

For order form see page ix

Transactions and Communications Molecular structure and film properties of alkyd resins*

By G. Walz

Hoechst AG, Werk Albert, Anwendungstechnische Abteilung, 6212 Wiesbaden

Summary

An air-drying alkyd resin, of medium oil length and with a broad molecular weight distribution specified by a ratio of $M_w/M_n \simeq 54$, has been separated into 13 fractions by preparative gel-permeation chromatography.

The original resin and fractions with $M_{\rm m}/M_{\rm n} \simeq 1.5$ have been characterised by chemical composition, hydroxyl value, acid value and viscosity. Equilibrium studies of these fractions at elevated temperatures, together with the analytical data mentioned

Keywords

Raw materials (binders, resins etc.) alkyd resin

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

gel permeation chromatography

manufacture or synthesis fractionation above, have proved the existence of large ring structures. After the molecular structure had been determined, the drying time and film properties of the resin and its fractions were measured.

Alkyd resins with a molecular weight distribution varying between $M_w/M_n \simeq 1.5$ and $M_w/M_n \simeq 54$ have been prepared by mixing suitable fractions. Some of these mixtures show properties superior to those of the original material.

Properties, characteristics and conditions primarily associated with:

materials in general molecular weight

coatings during application drying rate

La structure moléculaire et les caractéristiques des films de résines alkydes

Résumé

Au moyen de la chromatographie par perméation de gel, on a séparé en 13 fractions, une résine alkyde de moyenne longueur en huile et d'une répartition large de poids moléculaire qui est caracterisée par un rapport $M_w/M_n \simeq 54$.

La résine intégrale et des fractions d'un rapport $M_w/M_n\simeq 1,5$ ont été caracterisées par la composition chimique, l'indice d'hydroxyle, l'indice d'acide et la viscosité. Les études sur l'équilibre de ces fractions aux températures élevées, ainsi que les données analytiques mentionnées ci-dessus, ont confirmé la présence des structures à

Molecularstruktur und Filmeigenschaften

Zusammenfassung

Durch präparative Gel-Permeationschromatografie wurde ein lufttrocknendes, mittelfettes Alkydharz mit einer breiten Molekulargewichtsverteilung Verhältnis gekennzeichnet durch ein $M_w/M_n \simeq 54$ in 13 Fraktionen getrennt.

Das ursprüngliche Harz und Fraktionen mit $M_w/M_n\simeq 1,5$ wurden bezüglich ihrer chemischen Zusammensetzung, Hydroxylzahl, Säurezahl und Viskosität charakterisiert. Gleichgewichtsuntersuchungen dieser Fraktionen bei erhöhten Temperaturen bewiesen, zusammen mit den oben erwähnten analytischen

Introduction

Alkyd resins are polymers having a very inhomogeneous molecular structure. Apart from the types with a high oil content, they have a broad molecular weight distribution and a high chemical inhomogeneity. Investigations of the molecular structure in relation to the properties of the resins in their technical application have been described in literature¹⁻⁴. The grand cycle. Après la structure moléculaire fut déterminée, la durée des échage et les caractéristiques des films de la résine elle-même et de ses fractions on été également déterminées.

Les résines alkydes ayant des répartitions de poids moléculaire qui se varient entre $M_w/M_n\simeq 1,5$ et $M_w/M_n\simeq 54$ ont été préparées par l'admixtion des fractions appropriées. Certains de ces mélanges démontrent des caractéristiques supérieures à celles de la résine initiale.

Ergebnissen, das Bestehen grosser Ringstrukturen. Nach Bestimmung der Molekularstruktur wurden Trockenzeit und Filmeigenschaften des Harzes und seiner Fraktionen gemessen.

Es wurden Alkydharze mit einer Molekulargewichtsverteilung variierend zwischen $M_w/M_n\simeq 1,5\,$ und $M_w/M_n\simeq 54\,$ durch Mischung geeigneter Fraktionen hergestellt. Einige dieser Mischungen zeigen Eigenschaften, welche denen des Originalharzes überlegen sind.

method generally employed is the separation of the resins into fractions by fractional extraction with solvents of different polarity, or by fractional precipitation with mixtures of solvents and non-solvents, followed by a detailed examination of the fractions.

The broad molecular weight distribution overlaps ranges of different chemical constitution, so that poorly defined

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

fractions are obtained, which do not allow precise conclusions to be drawn.

Fractionation of an alkyd resin was performed using a preparative gel-permeation chromatograph, which is described in more detail in the appendix: this method avoids the disadvantages mentioned above.

Fractionation

A commercially available, medium oil length, air-drying alkyd resin of the type suitable for car refinishing lacquers was fractionated. Analysis gave the following composition:

Phthalic anhydride	26.8%
Pentaerythritol	21.0%
Fatty acids	39.8%
Benzoic acid	8.6%
p-tert. butyl benzoic acid	10.3%
Non-saponifiable components	3.0%
Hydroxyl number	52
Acid number	8.3
Viscosity (50% in mineral spirits	
130/175 xylene 3:1)	370 cP

(Total percentage exceeds 100 because the water of esterification has not been deducted.)

Gas chromatography gave the fatty acid composition as:

Palmitic	15.8%
Stearic	5.0%
Oleic	23.6%
Linoleic (9, 12 cis-cis isomer)	48.0%
Linolenic	6.2%
Linoleic not identified (9, 11 cis-trans and	
10,12 cis-trans isomers)	1.4%

In the analytical gel-permeation chromatograph (see Appendix) an initial gel-permeation chromatogram was prepared; the result is shown in Fig. 1, in which both the elution volume $V_{\rm E}$ and the molecular weight of the polystyrene



Fig. 1. Gel permeation chromatogram of the original alkyd resin

standard (*ex* Waters Associates, USA) are plotted along the abscissa, and the ordinate gives the difference in refractive index $\Delta \eta$ between the eluated solution and the pure solvent as a measure of concentration in arbitrary units. The molecular weight, calculated on the basis of the polystyrene standard of Fig 1, shows a number average M_n equal to 4700 and a weight average M_w equal to 254 000, giving a ratio of M_w/M_n equal to 54 for the whole range of the molecular weight distribution. To determine whether this broad molecular weight distribution is the result of an association by hydrogen bonding or polar forces, the alkyd resin was acetylated and methylated under mild conditions: the gel-permeation chromatogram obtained from the product did not differ significantly from that of the original resin.

Thirteen fractions were obtained by preparative fractionation of about one kilogram of resin in a gel-permeation chromatograph. After concentration of the fractions in a rotational evaporator, gel-permeation chromatograms were taken in an analytical apparatus in order to estimate the quality of the separation. The gel-permeation chromatograms of fractions 1-10 are shown in Fig. 2. Fractions 11-13 consist only of starting products and oligomer components so that these were disregarded.



The mean M_n and M_w values, calculated on the basis of the polystyrene standard, are given in Table 1.

It is evident from the M_w/M_n values that the separation is very good and uniform: changes in composition of the fractions cannot have taken place during the separation and concentration procedure as all operations were performed at ambient temperature or at only a slightly increased temperature.

	Table 1	
M_w and M_n	values of the fractions and	l the original resin

Resin or fraction	Weight per cent	Mw	Mn	M_w/M_r
Original resin	100	254 000	4 700	54
F1	2.7	1 180 000	1 000 000	1.2
F2	4.8	580 000	390 000	1.5
F3	5.4	294 000	203 000	1.4
F4	6.5	226 000	105 000	2.2
F5	7.5	148 000	49 000	3.0
F6	8.9	39 000	27 000	1.5
F7	12.3	21 000	13 000	1.6
F8	16.9	10 000	5 800	1.7
F9	17.9	4 900	2 650	1.9
F10	11.6	2 720	1 660	1.6
F11	4.5			
F12	0.8			
F13	0.2			

All the molecular weights given in Table I were obtained by calibration of the gel-permeation chromatograph with the polystyrene standard. Therefore, the true molecular weights may vary on either side of those given in Table I. For this reason the molecular weights were determined by methods which would give absolute values. The lower number averages (M_n up to about 25 000) were measured in a vapour pressure osmometer (Hitachi-Perkin Elmer model 115). The fractions with higher molecular weight would have been examined in a membrane osmometer for the determination of M_n and in a light scattering photometer for M_w but this equipment unfortunately was not available. The results of measurements by vapour pressure osmometry are given in Table 2.

 Table 2

 Comparison of the M_n values from vapour pressure osmometry and gel permeation chromatography (GPC)

Resin or M fraction	In as measured by vapour pressure osmometry	Mn from GPC measurement with polystyrene standard calibration
Original resin	3 400 + 200	4 700
F10	1000 ± 50	1 660
F9	1540 + 100	2 650
F8	2030 + 150	5 800
F7	9000 ± 1500	13 000
F6	16000 ± 2500	27 000
F5	25000 ± 4000	49 000

Measurements in the vapour pressure osmometer show lower values than those calculated from the GPC calibration with polystyrene.

Chemical composition of the fractions

The fractions obtained were saponified and analysed according to a method recommended by DIN 53 183 "Test of simple alkyd resins for paints". The results are shown in Figs 3 and 4. The weight average molecular weight of fractions (from Table 1) is plotted as the abscissa on a logarithmic scale, and the percentages by weight of the individual components are plotted on the ordinate.



Fig. 3. Chemical composition of the fractions





Fig. 3 shows that the phthalic anhydride content (PA content) of fraction 10 (F10) is 15.5 per cent, which is very low. It increases linearly up to F6 at 34 per cent and then remains constant to F1. This constancy of the phthalic acid content from a certain molecular weight has also been confirmed quantitatively using resins prepared with radio-actively marked phthalic anhydride (C¹⁴) by gel-permeation chromatographic separation and subsequent radiometric determination of the phthalic anhydride content in the fractions obtained.

The fatty acid concentration follows an opposite course to that for phthalic anydride concentration. F10 has a high fatty acid content of 52 per cent. The fatty acid content then decreases continuously until F6 and then it also remains constant to F1 with 32 per cent of fatty acid.

The composition of the fatty acids in each fraction is constant within the experimental error, which is proved by the result of the gas chromatographic analysis of the fatty acids, as shown in Table 3. No evidence could be found to indicate fatty acid polymerisation.

Contrary to the behaviour of the fatty acids, the two other chain stoppers, benzoic acid and p-tert. butyl benzoic acid, are built into the resin in a wholly different way (Fig. 4). These two components are uniformly esterified into the alkyd resin, and this reaction is independent of the molecular weight. Only with F9 and F10 is a slight enrichment observed.

The polyol pentaerythritol slightly increases in concentration from F10 (20 per cent) to F6 (22.2 per cent) and then remains constant to F1. The fact that the chemical composition from F6 to F1 is constant is an important matter for a further consideration. This extraodinarily different chemical composition of the low molecular weight fractions is one of the causes for the observed broad molecular weight distribution.

Ring formation

A statement of the molecular weights of a resin and its fractions and also of the chemical composition of the resin does not completely define its structure. In a linear polymer, the amount

	1	able 3			
Gas	chromatographic	analysis	of the	fatty	acids

Fatty acid	Amount of acid present in each fraction (per cent)									
	F1	F2	F3	F4	F5	F6	F7	F8	F9	F10
Palmitic	15.0	15.5	15.2	13.5	15.8	16.3	14.3	15.0	15.5	15.1
Stearic	4.7	4.9	4.9	4.3	4.7	4.5	4.9	4.4	4.4	4.6
Oleic	25.9	25.7	25.8	28.3	26.6	25.8	26.5	26.1	26.2	26.3
Linoleic (9,12 <i>cis-cis</i>) Linolenic and linoleic aci	48.3	48.1	48.0	47.9	47.3	47.2	48.2	48.0	47.6	47.8
(9,11 and 10,12 cis-tran	<i>is</i>) 6.1	5.8	6.1	6.0	5.6	6.2	6.1	6.5	6.3	6.2

of short-chain or long-chain branching is of great importance. This question of the extent of branching is very difficult to decide with linear polymers and at present there is no method known for determining the structure of resins having a very heterogeneous structure.

During the determination of acid value (AV) and hydroxy number (OHN) as well as stability of the fractions in a completely closed autoclave at a higher temperature, effects have been found which allow some conclusions to be drawn about the geometric structure of the alkyd resin providing certain additional assumptions are made.

The AV and OHN of the starting resin and its fractions are shown in Figs. 5 and 6, where the weight average molecular weight of the fractions is plotted against acid value and hydroxyl number. From F10 to F8 the true weight averages have been inserted, the remaining values of M_w being taken from Table 1. The true weight averages for F10 to F8 have been calculated approximately from the vapour pressure osmometer measurements which are still within the range of molecular weights, where accurate values can be obtained for this purpose; the osmometrically determined number averages from Table 2 were multiplied by the M_w/M_n values from Table 1.



Fig. 5. Acid value of the fractions

The acid value of 180 in F13 is extremely high; as can be seen from Fig. 5, the value decreases rapidly for F12 and F11, and passes through a minimum between F10 and F9 with an acid number of 4.7; it subsequently increases rapidly up to a value of 10.9 for F1. The variation in hydroxyl number (Fig. 6) shows a similar course.



Fig. 6. Hydroxyl number of the fractions

For a linear polyester consisting, for example of equimolar quantities of a dicarboxylic acid and a diol, such a change in the acid value and hydroxyl number with molecular weight would be inexplicable. The acid value and hydroxyl number must decrease with increasing molecular weight and tend towards zero. Measurement of the number of functional terminal groups in such polymers is, therefore, a method for determining the value of M_n .

A simple consideration shows that with these branched chain polyesters, the acid value and the hydroxyl number tend

towards a constant (usually positive) value with increasing molecular weight, providing the chemical composition of the fractions remains constant. From F6 to F1 the chemical composition is constant. Therefore, at high molecular weights, the hydroxyl number and the acid value should also be constant within this range. As this evidently is not the case, three possible explanations will be discussed.

The only component which can form a free carboxylic group if it is present as an ester is the phthalic acid. An increase in acid value over the range of fractions 1 to 6 is possible, if the concentration of phthalic acid increases with the molecular weight in this range. For this to be so, the phthalic anhydride content must increase from 34.0 to about 35.2 per cent. Chemical analysis of the phthalic anhydride content gave a result of 34.0 ± 0.5 per cent and the independent, more exact measurement using radioactively marked phthalic acid, showed a constancy within this range of experimental error. The observed increase in acid value cannot, therefore, be explained by the assumption of increased phthalic acid concentration.

The fatty acids present can polymerise and by building these polymers into the alkyd resin, free carboxylic groups may be formed. On the assumption that only dimer fatty acid polymers are formed, the fatty acid concentration must increase over the range F6 to F1 from 32 to 36.2 per cent, that is about 11 per cent of the starting fatty acid should polymerise. This figure is also outside the limits of experimental error, so that the assumption cannot be correct. An oil polymerisation of the required extent would change the percentage composition of the fatty acid in the high molecular fractions so that the conjugated fatty acids should be found preferentially therein as polymers. There is no indication that this is so from the information in Table 3.

The only explanation how, in spite of unchanged chemical composition, the acid value and the hydroxyl number can increase with molecular weight is by the formation of ring compounds. This statement refers strictly to the range of the constant chemical composition, that is from F1 to F6. Ring formation by esterification must give a ring consisting of a certain number of phthalic acid and pentaerythritol molecules, together with chain stoppers in which a carboxyl and hydroxyl group are consumed without changing the chemical composition. The following model is possible and is in accordance with analytical data.

As the acid value increases with increasing molecular weight in the low molecular weight fractions (that is from F6 upwards), more ring compounds must be present than in the higher molecular weight fractions.

A carboxyl and a hydroxyl group are consumed with every ring formed. Thus, the acid value will always increase or decrease to the same extent as the hydroxyl number changes, and this can be seen to be true (at a good approximation) for the fractions F6 to F1.

If a critical molecular weight is not reached, ring formation between phthalic anhydride and pentaerythritol molecules can no longer occur for steric reasons, and the acid value and hydroxyl number will then increase again, which is evident for the range between F9 and F10. A strict requirement for this consideration is that chemical composition remains constant. This condition is not fulfilled from F10 to F7, so the statement can be only approximately true in this region, and it cannot be concluded, therefore, that the very low acid value and hydroxyl number for F9 mean that it must contain the greatest number of rings. The minimum in Fig. 5 gives an indication of the smallest rings which can be formed from phthalic anydride and pentaerythritol. The molecular weight M_w of the minimum, determined from Fig. 5, is between 1700 and 2900. A ring of two molecules of phthalic anhydride and two molecules of pentaerythritol and chain stoppers, corresponding to F9 according to its quantity and composition, has a molecular weight of about 1600. A corresponding ring from three molecules of phthalic anhydride and three molecules of pentaerythritol has a molecular weight of about 1600. A corresponding ring from three molecules of phthalic anhydride and three molecules of pentaerythritol has a molecular weight of about 2400. Both rings were studied with molecular models ("Dreiding") and there appear to be no steric reasons which would prevent the existence of such rings. Thus, the smallest rings formed are considered to have the structure suggested above, and it is assumed that larger rings still are possible.

During preparation of the alkyd resin, a point is reached in the course of condensation when for the first time either 2, 3 or 4 molecules of phthalic anydride react with 2, 3 or 4 molecules of pentaerythritol together with the corresponding chain stoppers. A certain percentage of these molecules may form rings, whilst the others remain linear. The rings have less functional groups than a linear chain of comparable size. Consequently, there is less chance that they will reach a high molecular weight.

Provided that the stated hypothesis is correct, then if the fractions are given the opportunity to adjust their equilibrium in a closed system at an increased temperature, they should show a certain behaviour. The high molecular weight fractions, which ought to have less rings, should be able to form further ring structures. In a closed system, this means a decrease in the molecular weight. On the other hand, those fractions which cannot form rings on account of their size or which already contain almost the maximum possible number of rings should remain stable. In order to illustrate this, the high molecular weight fractions F3 and F4 as well as the low molecular weight fraction F9 were used as examples. After separation in the gel-permeation chromatograph and subsequent concentration, the samples were prepared as solutions in specially cleaned and dried xylene and were adjusted with the same solvent to give a 22 per cent solution in every case. A trans-esterification catalyst was added to the solutions and 250 ml samples were then treated in an autoclave for 6 hours at 220°C and about 10 atmospheres pressure. Great attention was paid to the tightness of the autoclave seal to ensure that no components could escape from the vessel.

Figs. 7 and 8 show the results of the treatment. The fractions F4 and F5 are decomposed to a great extent to polymers having nearly the same molecular weight, whilst F9 remains practically stable.



Fig. 7. High molecular weight fractions before and after treatment in an autoclave



Fig. 8. Low molecular weight fractions before and after treatment in an autoclave

Properties for technical applications

The fractions and the starting resin were mixed with 2.5 per cent of a drier solution (calculated on the basis of solid resin), with composition:

- 7.2% Cobalt octoate (6% metal content)
- 22.0% Lead octoate (24% metal content)
- 6.2% Zirconium octoate (6% metal content)
- 64.6% mineral spirits 130/175.

Subsequently, all the clear lacquers were adjusted to an efflux time of 90 seconds in the 4 mm D1N cup with a solvent mixture of mineral spirits 130/175 and xylene (3:1). The solids content of these clear lacquers is given in Fig. 9.



Fig. 9. Solids content of alkyd fractions in xylene at a constant viscosity of 370 cP and temperature of 23°C

Drying of clear lacquers

Glass plates were coated with the solutions to give a dry film thickness of about 40 μ m and the times for each coating to reach a tack-free and a through-dried condition at ambient temperature were measured (Fig. 10). The curves are almost parallel. F10 is not tack free even after one week; F9 and F10 are not through-dry after one week. The drying times



Fig. 10. Drying time of different alkyd fractions (including driers)

decrease with increasing molecular weight and decreasing fatty acid content. F7 reaches a tack-free state in only one quarter of the time and a through-dry state in only half the time required by the starting resin, although the chemical composition and the viscosity of F7 are nearly the same as those of the starting resin. In Fig. 10 the curves for F6 to F3 show a constant change in spite of the rapidly increasing molecular weight and there is a distinct increase at F2 and F1. This increase is attributed to solvent retention: during the drying of the very high molecular weight fractions, the solvent is entrapped in the coating.

Storage stability of clear lacquers

With other samples of the clear lacquers (including driers) the storage stability was determined in closed 50 ml glass vessels maintained at 70°C until the onset of gelation.

Fig. 11 shows that the storage stability of fractions F1 and F2 is less than one day, whilst the starting resin gels after 8 days. F6 to F10 are not gelled even after 90 days, at which time the experiment was stopped. The steep increase of the storage stability between F5 and F6 cannot be explained. It is interesting that F7 has excellent drying characteristics and vet possesses more than ten times greater storage stability than the starting resin.



Fig. 11. Storage stability of the fractions (including driers) at 70°C

Mechanical properties of clear lacquers

The mechanical properties of linear polymers improve with increasing molecular weight. However, very hard and solventresistant films (resistant, for example, to petrol) are obtained from fractions F7 to F1 after drying at ambient temperature; the films which were prepared showed no flexibility in the Erichsen cupping test and very bad results from crosshatching experiments. This behaviour is assumed to be a consequence of the special geometric arrangement of these fractions, with an accumulation of rings causing a rigid structure.

The dried, clear lacquer films of the starting resin show an Erichsen cupping of greater than 8 mm because fractions F1 to F7 (which by themselves give much too rigid a structure

during crosslinking due to their high ring content) are plasticised by the remaining fractions, which contain few rings but a large quantity of fatty acid and are consequently less rigid.

White lacquers

The optimum of drying time, storage stability and required film properties should be found in the range of fractions F6 to F8 and for the above mentioned reasons not with pure fractions but with mixtures. For this reason, fraction F7, a mixture of F6, F7 and F8 having an essentially narrower molecular weight distribution than the starting resin, and the starting resin itself were each pigmented in a conventional manner in a bead mill. Driers were added and the solutions adjusted to a spraying viscosity of 20 DIN seconds in a 4 mm beaker: all three resins had approximately the same solids content at this viscosity. Fig. 12 shows the gel-permeation chromatogram of the three resins. In the case of the starting resin, the formulation of a white lacquer suitable for spraying is shown in Table 4.



Fig. 12. Gel permeation chromatogram of F7, a mixture of F6, F7, F8 and the original resin

Table 4

Formulation of a white lacquer suitable for spray application

- 26.0g Alkyd resin
- 22.0g Titanium dioxide
- 2.5g Drier solution
- 0.7g Silicone oil (15% in xylene) 0.2g "Aerosil"
- 0.5g Antiskinning agent

29.1g Solvent: mineral spirits 130/175 and xylene (3:1 by volume) This solution has a viscosity of about 110 seconds in the 4mm DIN cup and is adjusted for spraying with mineral spirits 130/175 and xylene (3:1) to 20 seconds in the 4mm DIN cup.

In order to confirm the results obtained with clear lacquers. the fractions F6, F7 and F8 were examined as white lacquers containing driers in a further series. All lacquers were applied to metal sheets by spraying. The results are given in Table 5.

Film properties of fractions and mixtures of fractions with driers in white lacquer formulations								
Resin base	Film thickness (µm)	lm Erichsen Penduli kness cupping accordi m) after		lum hardness Cross- ing to König hatch te after after		ross- Resistance to ch test gasoline after after:		Gloss after 20 days (60° Lange)
		20 days (mm)	2 days	20 days	20 days*	1 day*	20 days*	
Starting resin Mixture (F6 + F7	50	8.2	70	136	2.5	3.5	0	96
+ F8)	50	8.2	113	152	3	0.5	0	96
F7	55	0.5	129	167	5	0.5	0	97
F6	50	1.5	160	183	5	0	0	No gloss
F7	55	0.5	129	167	5	0.5	0	97
F8	55	9.3	56	109	2.5	5	0	105

Table 5

*0 equals best result; 5 equals worst result.

The results are as expected: by addition of F8 to the resin mixture of F6, F7 and F8, the elastic properties of the hardened film, such as Erichsen cupping and cross-hatch tests, approximate to the values of the starting resin, whilst all other properties remain essentially unimpaired or are improved. The values for F6, F7 and F8 in Table 5 also confirm the values already obtained with clear lacquers. Additionally, it is to be noted that F6 and all higher molecular weight fractions have no gloss.

Conclusions

Alkyd resins based on o-phthalic acid and pentaerythritol may contain large ring structures. Fractions containing a significant proportion of these rings show excellent drying properties, but the films prepared from them have no elastic properties because the molecular structure is too rigid. The elastic properties of such films are improved by plasticisation with low molecular weight fractions rich in fatty acids or containing a small number of rings.

Alkyd resins of the same chemical composition, the same viscosity at the same solids content, but with a narrower molecular weight distribution than ordinary alkyd resins, have excellent storage stability. They give very fast drying, hard and solvent resistant films.

The other film properties remain unchanged as long as the ring content is not too high and the amount of plasticising components is not too low.

[Received 24 April 1976

Appendix

Preparative gel-permeation chromatograph

System

Constructed from elements of "Waters Assocs, Framingham, Massachusetts" with an automatic sample injection system and an automatic sample collector.

Columns

Four "Styragel" columns from "Waters Assocs". Length Im (3 feet); outer diameter 2.5cm (1 inch); exclusions limits (a) $0.1\mu m \times 1$, (b) $1.0\mu m \times 1$, (c) $10.0\mu m \times 2$. Flow: 10ml per minute.

Solvent

Xylene purified with Woelm's Alumina adsorbent W 200. Sample size 3g resin in 20ml xylene.

Analytical gel-permeation chromatograph

System

Gel-permeation chromatograph ALC 100 from "Waters Assocs" with an automatic sample injection system.

Columns

Eight "Styragel" columns from "Waters Assocs". Length 1m (3 feet); outer diameter 1cm (3 inch); exclusion limits (a) 6nm×1, (b) $20nm \times 1$, (c) $100nm \times 1$, (d) $1\mu m \times 1$, (e) $3\mu m \times 2$, (f) $10\mu m \times 2$.

Flow

0.66ml per minute.

Solvent

Tetrahydrofurane

Sample size

2mg in 2ml tetrahydrofurane.

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Recent aspects on the preparation and evaluation of some polyesteramides for surface coatings. Part I: Parameters affecting the formation of various dihydroxy-diethylamide derivatives of fatty acids

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Summary

Hydroxyethylamide derivatives of fatty acids are used as starting materials in the formulation of the recently developed "polyesteramide" resins. Parameters affecting the formation of such compounds from oils and diethanolamine—such as the type and con-

Keywords

Raw materials:

binders-oils

castor oil

dehydrated castor oil linseed oil soya bean oil

binders-resins, etc. polyesteramide resin

Used in manufacture or synthesis of ingredients for coatings rice germ oil centration of catalyst used and the reaction temperature—are investigated. The course of the reaction is followed by a thin layer chromatographic technique and by a turbidity technique.

Processes and methods primarily associated with analysis, measurement or testing

thin layer chromatography

Les aspects récents de la préparation et de l'évaluation de certains polyesteramides destinés aux revêtements de surface. 1^{ère} Partie: Les paramètres portant sur la formation de divers dérivés dihydroxy-diéthylamides des acides gras

Résumé

On a utilisé les dérivés hydroxyéthylamides des acides gras en tant que matières premières dans la préparation des résines polesteramides qui ont été récemment mises au point. On a étudié les paramètres, tels que le type et la concentration du catalysateur utilisé et d'ailleurs la température de réaction, qui exercent une influence sur la formation de ces composés à partir des huiles et du diéthanolamine. On remarque le progrès de la réaction au moyen des techniques de la chromatographie en couche mince et de l'opacimétrie.

Neuere Betrachtungen über die Bereitung und Bewertung einiger Polyesteramide für die Lackherstellung

Teil I: Parameter, welche die Bildung verschiedener Dihydroxi-diäthylamidderivate von Fettsäuren beeinflussen

Zusammenfassung:

Hydroxyäthylamidderivate von Fettsäuren werden als Ausgangsmaterial für Vorschriften für die enlängst entwickelten "Polyesteramid" Harze benützt. Parameter, welche die Bildung solcher Verbindungen aus Ölen und Diäthanolamin beeinflussen,-wie

Introduction

Refs. 1-3

Research work described for the reaction of oils with diethanolamine (DEA) is scant and the subject has not been exdie Art und Konzentration des benutzten Katalysators und der Reaktionstemperaturen-werden untersucht. Der Reaktionsverlauf wird durch eine Dünnschicht-Chromatografie und eine Trübungstechnik verfolgt.

tensively studied. Theoretically, oils react with DEA with the formation of diethanolamide derivatives of the corresponding fatty acids, acording to the following series of equations:



where R, R' and R'' represent the various fatty acids present. The net reaction can be simply illustrated as follows:

CH,OCOR CHOCOR CH,OCOR	C,H,OH 3HN C,H,OH	3RCO-N	СН,ОН + СНОН СН,ОН
Glyceride	DEA	Bis-hydroxyethyl fatty amide (HEFA)	Glycerol

The aminolysis of oils is generally catalysed by the presence of bases. The hydroxyethylamide derivatives of linseed and soya bean oils were prepared by heating one mole of oil, six moles DEA and 0.07 mole sodium methoxide as a catalyst. The reaction temperature was 115°C and the reaction was completed in about 35 minutes. The course of the reaction was followed by a thin layer chromatographic technique¹⁻³. After completion of this reaction, the hydroxyethylamide derivatives were extracted from the liberated glycerol by ether; this was followed by successive washing of the ether solution with 15 per cent sodium chloride solution and drying over anhydrous sodium sulfate.

The mixed HEFAs so prepared were considered to be dihydric alcohols suitable for the preparation of polyesteramide resins with the polybasic acid or anhydride. Part II of this paper will deal with the preparation and evaluation of various types of polyesteramide resins.

Experimental

Refs. 4, 1, 5-11

All reagents used throughout the work were of the purest grades available, except where mentioned.

Preparation of bis(2-hydroxyethyl) fatty acid amides (HEFA)

A mixture of freshly distilled DEA (14.5 ml; 0.15 mole) and the catalyst (0.0014 mole) was placed in a 4-necked, 250ml flask equipped with an efficient stirrer, inert gas inlet, thermometer and dropping funnel. The mixture was heated to the specified temperaure, then the oil (0.05 mole) was added dropwise over a period of 15–20 minutes. The reaction temperature was kept constant and samples of the reaction product were withdrawn periodically with a syphoning device in order to study the progress of the reaction. This was carried out by using thin layer chromatography and testing the solubility of the product in absolute methanol. The oils used in the investigation were linseed (LO), castor (CO), dehydrated castor (DCO), rice germ (RGO), soya bean (SO) and coconut oils. The catalysts employed for the aminolysis were zinc oxide, lead monoxide, barium hydroxide, calcium hydroxide, lithium hydroxide, sodium hydroxide and sodium methoxide. The reaction temperatures investigated were 115°, 150°, 180°, 200° and 220°C.

Thin layer chromatographic technique

Testing of the progress of the reaction, to determine whether it was complete or whether it should be terminated, was carried out by thin layer chromatography according to a method described in the laboratory handbook⁴. The method involved the application of a minute spot of a solution of the sample in acetone on to a glass microscope slide which had previously been coated with silica gel G and dried. The solvent system used for developing was a mixture of ether, hexane and acetic acid (70:30:1 by volume). Iodine vapour was used to detect the spots. The chromatogram, which showed the progress of the reaction at the indicated times, was compared with the oil as the reference material (top spot). The disappearance of the top spot indicated the completion of amide formation¹.

Turbidity technique

The progress of the reaction was also measured by testing the solubility of the product in twice its own volume of absolute methanol at room temperature⁵.

Product evaluation

The colour⁶, viscosity⁷, acid value⁸, iodine value⁹, saponification value¹⁰ and hydroxyl value¹¹ determinations were carried out according to standard methods, described in the literature references given.

Results and discussion

Refs. 1, 12

Polyesteramide resins were introduced in 1966 and are one of a number of recently developed resins for protective surface coatings. They combine the useful properties of polyester and polyamide resins and are prepared in two stages: the first step¹ involves preparation of the fatty acid derivatives of secondary dihydroxyamine by reaction of either the oil itself or its methyl esters with excess DEA in the presence of sodium methoxide catalyst at 115°C; the second step involves preparation of polyesteramide resins in a manner similar to those described for alkyd resins¹³. No explanation is given for the use of excess amine in the aminolysis step.

This part of the paper deals with the preparation of various fatty acid amides and a study of the various parameters which affect their formation. The oils used in the investigation were analysed and their characteristics are given in Table 1.

Tab	le	1		
Characteristics	of	the	oils	used

Type of oil	Acid value (mg KOH/g)	Saponifi- cation value (mg KOH/g)	Iodine value (cg I ₂ /g)	Specific gravity	Hydroxyl value (mg KOH/g)	Colour value
Soyabean	0.5	192	135	0.92	0.00	10
Linseed	2.0	190	180	0.93	0.00	11
Castor	2.0	180	87	0.96	166.0	8
Dehydrated castor	4.0	191	150	0.94	8.0	5
Coconut	2.0	251	8	0.91	0.00	3
Rice germ	40.0	181	107	0.91	0.00	13

Diethanolamine (DEA) molar ratio

Gast *et al*¹ prepared dihydroxydiethyl linseed fatty acid amide(HELA) by reaction of the oil, catalyst and excess DEA. They did not explain the reason for the presence of such a large excess of DEA (6 moles per mole of oil). For this reason, the study of the DEA ratio is included amongst the aims of this work. Formulations of linseed oil with various stoichometric amounts of DEA (3, 4, 5 and 6 moles per one mole oil) were prepared at constant temperature (115°C) for 90 minutes and using sodium methoxide or lead monoxide as catalyst (0.07 mole). In both cases, no amide products could be detected, either by thin layer chromatography and or by testing for solubility in absolute methanol. These findings contradict the data given by Gast *et al*¹ for the formation of amide derivatives.

Further experiments were conducted under similar experimental conditions, except that the temperature was kept at 200°C. The results are given in Table 2. It should be noted that lead monoxide was used as the catalyst.

Table 2 Amide derivative formation at various amine concentrations after 60 minutes at 200°C

Molar ratio DEA:LO	HELA formation (TLC)	Solubility in methanol
6:1	Positive	Soluble
5:1	Positive	Soluble
4:1	Positive	Soluble
3:1	Positive	Soluble

The main conclusion to be drawn from the data in Table 2 is temperature that effects formation of the amide derivative. There is also an obvious relation between the solubility of the product in absolute methanol and detection of the amide using TLC. Thus, all detectable (by TLC) amide products show solubility in methanol.

Effect of temperature

As has been pointed out, the main factor affecting the formation of HELA is the reaction temperature, not the excess molar ratio of secondary hydroxy amine. For this reason, experiments were performed using 3 moles only of DEA and one mole of LO in the presence of 0.07 moles lead monoxide at various reaction temperatures. The results are given in Table 3.

	Table 3
Effect of temperature on	the formation of HELA

Temperature (°C)	Time of HELA formation (TLC) (minutes)	Time at which the product shows solubility in methanol (minutes)
115	negative	negative
150	60	40
180	50	30
200	15	10
220	10	5

It can be concluded from the data in Table 3 that increasing the reaction temperature leads to a decrease in the reaction time (as measured by TLC), which is in agreement with the previous observation. Analogous behaviour was also noticed regarding the solubility of the product in absolute methanol but, in general, such products exhibit solubility in methanol at an earlier time than that measured by TLC.

Type of catalyst

The work was extended to include catalysts other than lead monoxide (litharge) and the results are given in Table 4.

	Table 4	
Effect of vari	cus catalysts upon	HELA formation*

						1	lime in	n m	inutes					
Temperature N (°C) <i>TL</i>	NaC	н	Ca(C	H);	Zn	0	NaOC	CH3	PbC)	Ba(O	H)2	LiO	н
	TLC	S	TLC	S	TLC	S	TLC	S	TLC	S	TLC	s	TLC	S
115	n	n	n	n	n	n	n	n	n	n	n	n	n	n
150	120	80	170	80	70	40	100	70	60	40	n	n	150	75
180	80	15	100	30	30	14	30	15	50	20	100	50	60	20
200	60	5	80	20	15	10	25	5	15	10	90	15	30	10
220	50	5	70	17	5	5	20	5	10	5	60	12	20	5

n indicates negative results, *TLC* represents the time at which HELA is detected by *TLC*, and *S* represents the time at which the product shows solubility in absolute methanol.

The conclusions drawn from the data given in Table 4 are:

There are almost no amide derivatives formed at 115°C.

Increasing the reaction temperature leads to a decrease in the time of HELA formation as detected by TLC. A similar behaviour is observed for the time at which the products show solubility in methanol.

The time at which the products show solubility in methanol is always shorter than that observed for HELA formation as petected by TLC.

The various catalysts used for aminolysis can be classified as follows:

Highly reactive catalysts: zinc oxide and lead monoxide

Moderately reactive catalysts: sodium methoxide, sodium hydroxide and lithium hydroxide

Weakly reactive catalysts: calcium and barium hydroxides.

The HELA formed under different experimental conditions were subjected to various characterisation tests and the results obtained are given in Table 5.

 Table 5

 Characteristics of HELA formed under various conditions

			_					
Catalant	150°C		180°C		200°C		220°C	
Catalyst	C*	V*	С	V	С	V	С	V
Sodium hydroxide	14	н	17	к	15	А	18	н
Calcium hydroxide	14	D	15	F	14	н	17	G
Zinc oxide	13	н	14	G	14	A	14	G
Sodium methoxide	17	J	18	H	18	K	18	F
Lead monoxide	14	K	15	K	18	G	18	H
Barium hydroxide	n	n	16	н	16	H	14	H
Lithium hydroxide	n	n	18	Н	18	Н	18	Н

*C = the colour as measured by Hellige colour comparator, V = Viscosity by Gardner scale,

n = indicates that no products are formed under such conditions.

It is clearly seen from Table 5 that temperature has a marked effect on both the colour and viscosity of the HELA formed. Moreover, zinc oxide and lead oxide are found to be

the most reactive catalysts for aminolysis, the former being preferable because it produces lighter-coloured amides.

Catalyst concentration

Following the selection of zinc oxide as the most suitable catalyst for amminolysis of linseed oil, attention was directed towards the effect of catalyst concentration upon the formation HELA. Experiments were conducted using three moles of DEA and one mole of LO at 200°C using different amounts of zinc oxide as catalyst. The results obtained are shown in Table 6.

	Table 6	
Effect of zinc oxide	concentration upon	HELA formation

Zinc oxide (mole)	Time of HELA formation (mea- sured by (TLC) (minutes)	*Time for the appearance of methanol-soluble products (minutes)	Yield (%)
0.0028	5	5	80.9
0.0014	15	5	96.6
0.0007	30	5	89.0
0.00035	50	5	80.9

From the data in Table 6, it can be concluded that increasing the catalyst concentration leads to a decrease in the time necessary for the formation of HELA. However, using higher concentrations of catalyst leads to some difficulties separating the amide from the ether solution—which is not encountered when using lower catalyst concentrations. For this reason, the most suitable catalyst concentration is considered to be 0.0014 mole or 0.1613 per cent (by weight) of the total charge.

Order of addition of the reactants

In order to show the influence of the order of addition of the reactants, trials were performed on the preparation of HELA by charging all the ingredients, followed by the usual heating and testing schedules at the pre-determined optimum experimental conditions.

The most important finding of this investigation is that the order of addition has no significant effect on the formation of HELA. A comparison between the general characteristics of the HELA products obtained by the two-step and one-step methods shows that they have similar properties.

The above data are illustrated in Figs. 1–3, representing the general features for the parameters affecting the formation of HELA.



Fig. 1. Effect of temperature on the time of appearance of alcoholsoluble products



Fig. 2. Formation of HELA (thin layer chromatography) using different catalysts



Fig. 3. The effect of zinc oxide catalysts on the formation of HELA

Elemental analysis of the HELA prepared showed the following: C 71.60%; H 10.86%; N 3.43%. The compound $(C_{22}H_{42}NO_3)$ would require: C 71.90%; H 11.24%; N 3.81%.

After determination of the most suitable experimental conditions for the formation HELA, it was thought desirable to extend the work to other oils. The characteristics of the various bis(hydroxyethyl) fatty acid amides were determined and these are shown in Table 7.

			Table 7				
Characteristics	of	various	bis(hydroxy	ethyl)	fatty	acid	amides

	Time formation	for the n of HEA			Viald
Oil used	TLC (minutes)	S (minutes)	Colour	Viscosity	(%)
Rice germ	40	10	18	Q	65 -
Coconut	100	5	18	B	86
Castor	105	10	18	L	82
Dehydrated castor	90	15	18	Z	67
Soya bean	30	5	18	Н	64

The data given in Table 7 clearly indicate the dependence of the time needed for the formation of the hydroxyethyl amide derivatives upon the type of the fatty acids present. The unsaturated oils have been shown to be the most reactive and hence shorter times are required.

Conclusions

Bis(hydroxyethyl) amide derivatives of various fatty acids are prepared in high percentage yield by reacting the oil (one mole), DEA (three moles) and zinc oxide (0.0014 mole) at 200°C. Lead oxide catalyst behaves in a manner similar to zinc oxide, except that the product shows a darker colour.

The time of formation of such amide derivatives depends upon the temperature, catalyst concentration and the type of fatty acid unsaturation present. Saturated oils require longer times than those containing unsaturated acids.

During the course of aminolysis, the solubility of the HELA in twice its own volume of absolute methanol can be used as a criterion for judging the time of completion of the reaction, since it represents about 60 to 70 per cent of the actual time to completion as detected by TLC. The method is recommended as a rough means for following the progress of the reaction.

[Received 23 June 1976

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Surface films produced by electrostatic powder deposition*

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Summary

The paper is intended to provide an introductory survey of the present state of the art.

Basic phenomena associated with electrostatic powder coating are reviewed and the principal applications of the process to film production are described.

Over the last eight years, fundamental research has begun to yield data which are being used to establish design criteria for polymer film production. It is found that powder charging mechan-

Keywords

Types and classes of coatings and allied products

powder coating

Equipment primarily associated with application of coatings and allied products

electrostatic spray equipment

Processes and methods primarily associated with application of coatings and allied products

electrostatic coating

Revêtements de surface produits par la déposition électrostatique de poudres

Résumé

Le but de cet article est de fournir un aperçu introductif à l'etat actuel du métier.

On passe en revue les phénomènes de base à l'égard des peintures en poudre destinées à être déposées électrostatiquement, et l'on décrit les applications principales de ce procédé à la production des films.

Au cours de ces dernières huit années, la recherche fondamentale se met à rendre les données que l'on peut utiliser en établissant les critères dans le domaine du dessein de la production des films polymères. On trouve que les mécanismes utilisés pour effectuer la isms are closely related to the adhesion properties and to the distribution of the unfused powder layer, and these relationships are discussed.

In addition to an investigation of the conventional type of corona gun currently used in industry, some new developments are described. The properties of a novel gun, in which triboelectric processes are used to charge the powder, are described. Electrostatic processes for the deposition of vitreous enamels and metals are discussed.

Properties, characteristics and conditions primarily associated with materials in general

adhesion

Miscellaneous terms

tribbelectric effect

déposition des poudres ont un rapport extrêmement étroit avec les caractéristiques d'adhérence et avec la répartition de la couche de poudre non fondu, et l'on discute ces rapports.

Ainsi qu'une investigation du type normal de pistolet à couronne que l'industrie utilise actuellement, on décrit certains développements récents. Egalement, on décrit les caractéristiques d'un nouveau pistolet, où l'on fait appel aux processus tribo-électrostatiques pour effectuer la déposition du poudre. On discute les processus, électrostatiques pour effectuer la déposition des émaux vitrifiés et des métaux.

Filme von durch elektrostatsichen Pulver-Niederschlag erzeugten Beschichtungen

Zusammenfassung:

Diese Abhandlung soll eine einführende Übersicht über den gegenwärtigen Stand der Technik geben.

Grundsätzliche mit elektrostatischer Pulverbeschichtung verbundene Phänomene werden besprochen, und die hauptsächlichen Anwendungen des Verfahrens zur Erzeugung von Filmen beschrieben. Im Laufe der letzten acht Jahre hat fundamentale Forschung begonnen, Resultate zur Festlegung von Entwurfskriteria für die Erzeugung von Polymerfilmen zu ergeben. Es wird gefunden, dass Mechanismen der Pulverladung eng mit den Haftungseigenschaften

ntroduction

Electrostatic painting has been widely used for over 30 years. It is essentially a deposition method and the paint film produced has properties similar to those obtained by other methods.

Electrostatic powder coating, developed as a polymer coating process in the 1960s, is increasingly used as an alternative und der Verteilung der ungeschmolzenen Pulverschicht in Beziehung stehen; diese Beziehungen werden besprochen.

Ausser einer Untersuchung der konventionellen, augenblicklich von der Industrie benutzten Type der Corona Pistole werden einige Neuentwicklungen beschrieben, insbesondere die Eigenschaften einer neuartigen Pistole, in welcher triboelektrische Prozesse zur Ladung des Pulvers benutzt werden. Besprochen werden elektrostatische Verfahren für den Niederschlag von Feueremaillen und Metallen.

to painting. However, the properties of polymer films produced by powder coating differ from those produced by wet paint processes. The nature of the deposition processes has a considerable influence on the final finish.

The purpose of this paper is threefold: (i) to review the basic phenomena associated with powder coating; (ii) to describe the main applications of powder coating to film production; and (iii) to describe some of the ways in which

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

recent research has provided information which may facilitate control of the quality of polymer coatings using electrostatic powder coating.

Essentially electrostatic powder coating, unlike electrostatic painting, involves a number of independent phenomena which can be optimised to give better control of the final coating. Electrostatic powder coating is currently used to produce polymer, vitreous enamel, metal and ferrite films.

Basic processes

The basic arrangement in a typical powder coating system is shown in Fig. 1. The powdered coating material is fed to the gun and charged in the nozzle region. The charged powder then moves towards the earthed object under the influence



Fig. 1. Typical arrangement for powder coating

of the electric field between gun and object. The powder is held by electrostatic forces to the surface of the substrate. The article is subsequently heated in an oven, where the unfused powder layer melts to give a continuous film. It is found, perhaps unexpectedly, that the nature of the unfused powder layer has a profound effect on the properties and physical nature of the fused layer.

The most important mechanisms involved are shown in Fig. 2 and are as follows:

- 1. Powder charging
- 2. Powder transport
- 3. Adhesion mechanisms
- 4. Back ionisation
- 5. Self-limitation.



This paper is concerned only with those mechanisms which have a direct influence on film formation. A number of review papers has been published giving detailed treatment of the

mechanisms and these may be consulted if additional information is required. To understand the process of film formation it is necessary to examine the mechanisms 1, 4 and 5 in some detail.

Powder charging

Corona charging

In most commercial powder guns, the powder is charged by a corona discharge mechanism. In the gun nozzle, the powder stream is exposed to an ion cloud in the region of the charging electrode (Fig. 3). The charge q acquired in time t is determined by the Pauthenier² relation:



Fig. 3. The electric field between gun and object

where *a* is the particle radius, $E_{\rm C}$ the electric field in the charging region and *J* the ion current density. *J* is a function of the gun/electrode geometry, the applied voltage and distance between gun and object. Experiments^{3,4} show that the ion stream from a corona point flows in a sharply defined region. With point-plane geometry the ion current I_i arriving on the earthed plane is fairly uniformly distributed over a circular area of radius *R*. Thus:

$$I_{\rm i} \simeq J \pi R^2 \qquad (2)$$

The value of I_i can be experimentally determined. Fig. 4 shows the voltage/current relationship for different spacings between gun and object. The voltage regulation curve for the high tension power unit is also plotted. From the intersections $(A_1, A_2$ etc.) of these curves, I_i can be found for a given situation.



Fig. 4. Gun voltage versus current for different spacings between gun and object

It was at one time thought that the gun voltage V and spacing *d* were the factors which primarily influenced powder velocity and charging, but it has now been found⁶ that I_i has a considerable influence on the quality of the coating as well.

Recently, it has become possible to carry out experiments on triboelectric powder guns⁴ in which the powder is charged by the triboelectric (rubbing) process. There is no ion current in such guns, so that it has been possible to compare the quality of coatings with and without ion current.

Triboelectric charging

Any two dissimilar materials can acquire a charge following impact, whether violent or not. The charge q acquired will depend on the difference in work function between the two materials, and on the resistivity of the polymer powder and of the surface which it contacts. Williams⁵ has found that the relation is of the form:

The terms ϕ_P and ϕ_S are the work functions of particle and surface, *t* the contact time and τ is a characteristic time depending on powder resistivity. If it is arranged for the powder stream to a contact a suitable surface (for example, the tube on which the nozzle is mounted), a stream of charged powder can be produced. One obvious advantage is that no high voltage power supply is required.

Research at Southampton University sponsored by the National Research Development Council (NRDC) is in progress on a "tribo-gun" in which powder charging is carried out by impact on a rotor coated with a polymer chosen for each specific powder. This gun has proved useful in tests to study the influence of ion current on film properties.

Charge/mass ratio

The most useful parameter to characterise powder charging is the charge/mass ratio q/m (measured in C kg⁻¹). This is a measure of the ratio of electrostatic to gravitational forces and can also be used to calculate particle velocities.

For conventional corona guns, a typical range for q/m is 10^{-3} to 3×10^{-4} C kg⁻¹. With the Southampton/NRDC tribo-gun the range for epoxy powders is 2 to 3×10^{-4} C kg⁻¹ and up to 10^{-3} C kg⁻¹ for nylon. The two systems, therefore, give q/m values in similar ranges. The ion current in a typical corona hand gun may be 20 to 50 μ A (being zero with tribo-guns). In both types, the powder current $I_P = q/m \times$ (powder mass flow) and is about 2 to 5 μ A for typical guns.

Back ionisation

Until recently, the self-limiting mechanism which determined the coating thickness of unfused powder polymer films was thought to be due to repulsion by the charged layer. In 1974 Cross and Bassett⁷ showed that this was not correct. It is found that during deposition, an electrical discharge occurs in the powder layer. This will occur when the product $J\rho_P > E_C$ (ρ_P is powder layer resistivity, E_C the critical onset field \simeq 6×10^6 V m⁻¹). This discharge leads to two important secondary processes:

(a) a stream of counter ions, opposite in sign to those from the gun flow to the gun nozzle;

(b) physical agitation occurs in the powder layer and particles are ejected from it (sometimes violently in a craterforming manner). A detailed description of the mechanism has been published⁷. The process is complex and further work co-sponsored by a major Science Research Council (SRC) multi-company research project is in progress at Southampton, using laser anemometry and other techniques. It is sufficient here to establish that back ionisation, which depends essentially on J, is the most important process which determines coating quality in electrostatic powder coating.

Fig. 5 shows the variation of mass of deposited powder, gun current and light emission as a function of time. Fig. 6 shows that q/m for powder approaching the layer during coating decreases with time, due to the discharging effect of counter-ions from the layer.



Fig. 5. Mass of deposited powder, gun current and light emission plotted as a function of time for a tribo-gun



Fig. 6. Charge/mass ratio for the tribo-gun as a function of time

Cross and Bassett have made a series of experiments which establish that the degree of physical disruption of the powder surface increases as the intensity of local discharges increases. As J is increased, there is an increasing tendency for local pinpoint discharges to occur. High-speed photography has shown that these can result in micro-eruptions of the powder layer, which leave behind the familiar "moon craters". When the powder layer is fused in an oven, the final surface finish tends to retain local irregularities in the deposited film.

Adhesion

The adhesion of electrostatically deposited powders has been studied at Southampton for a number of years, mainly by Cross^{6,7}, supported by the SRC. A brief discussion of the mechanisms is justified because it is found that, in general, techniques which can give an improvement in surface finish can lead to a reduction in adhesion force for the unfused powder layer.

The electrostatic pressure holding the deposited layer to the surface is given by:

where E_e is the electric field in the layer. The acceleration required to remove the layer is given by:

where ρ_P is powder density. The electric field in the layer is found to be approximately 6×10^6 V m⁻¹ and ϵ_r approximately 2 for most polymers, so *f* is approximately 3600 m sec^{-a}. In practice it is found that the adhesion force for powders deposited by conventional electrostatic guns is about 10 times greater than this. The adhesion of powders deposited by tribo-guns is found to be in general agreement with the predicted values based on electrostatic forces.

Ion current, particle shape and smoothness have a considerable effect on adhesion, whereas resistivity has a complex influence which cannot at present be accounted for theoretically⁹. Some typical values obtained by Cross⁹ for various powders are given in Table 1. These were obtained with

 Table 1

 Some results of powder spraying obtained by Cross⁷

Material	Powder resistivity (Ωm)	Radius (µm)	Measured adhesion $(\times 10^{-6} \text{ N})$	Calculated electrostatic adhesion force $(\times 10^{-6} \text{ N})$	
Perspex spheres (smooth)	>1015	37	7.0	1.8	
Epoxy	1015	20	1.4	0.37	
Nylon	1013	17	0.5	0.23	
PVCl Glass	5×10^{12}	50	0.9	0.6	
spheres (smooth)	6×10^{11}	30	3.4	0.36	

positive polarity spraying with a conventional charging system. Perhaps the most important result obtained by Cross is shown in Fig. 6. This shows the way in which the mass of deposited powder varies with removal force on a centrifuge.

The unfused mass was deposited by three methods:

- 1. Tribo-gun (Southampton/NRDC type)
- 2. Corona gun (positive and negative polarity)
- 3. Tribo-gun, followed by corona spray.

It will be noted that the adhesion of tribo-deposited layers is appreciably less than for other systems. With conventional corona application, positive polarity gives greater adhesion. The best results, however, are obtained with an initial tribocoating followed by a treatment with a low current corona discharge. With positive polarity and the latter two-stage treatment it was difficult to remove as much as 50 per cent of the layer.

These investigations are continuing, but they show how the ion current, which is important for adhesion and perhaps dielectrics to finish, plays a vital role in powder coating. Tribogun coatings are vulnerable because of the relatively low adhesion forces, but they give the best finish. It is not yet known how polarity affects finish. Experiments are in progress to determine the effect on finish of the two-stage treatment described above.

Comparison of corona and tribo-gun for surface finish

Using the Southampton tribo-gun⁵ and a commercial 90 kV powder gun, a number of experiments has been made to compare performance.

Much has been published about the performance of corona guns. With the tribo-gun, it is found that coating thicknesses tend to be greater, being similar to those obtained with electrostatic fluidised beds. The limiting coating thickness⁵ depends considerably on powder charge density ρ_{SP} in the powder jet. Thus:

where *n* is the number of powder monolayers, *q* the particle charge, v_a the air velocity, and *l* the coating thickness.

However, the main difference observed was the appreciably high gloss and smooth coatings obtained with the tribo-gun. This is clearly seen with visual observation of specimens. Talysurf traces for epoxy coatings obtained by both methods are shown in Fig. 7. Both traces show the characteristic



Fig. 7. Talysurf traces for the tribo-gun and corona gun

undulating trace observed with polymer films, similar to those reported by Rankin¹⁰, but the tribo films had a peak-topeak amplitude which was considerably less than the 10μ m value observed with corona guns.

The electric field in the tribo-deposited layer can be readily estimated and for epoxy powders this is about 2×10^6 V m₃⁻¹ (which is lower than the breakdown value). This estimate is confirmed by comparison with adhesion force F_a which should be in the ratio:

It would seem that the electrostatic forces in the case of the tribo-gun provide uniform deposition with uniform compression to give good packing. There is no back ionisation and hence no disruption of the unfused layer or disturbance of the good packing.

It is interesting to note that tests with a commercial gun in which corona charging took place inside the barrel also tended to produce smooth coatings. This would seem to confirm the view that suppression or reduction of ion current can be used to produce smoother coatings, provided that the consequent decrease in adhesion is acceptable.

Enhanced powder velocity due to ion induced motion (around 1 m sec⁻¹) can lead to local regions of increased thickness, but also localised enhanced penetration.

The space charge due to the powder cloud from any gun is responsible for the coating performance inside screened areas and both types of gun are on equal footing here. Typical results are summarised in the Table 2 for both types of gun for throughputs of about 2 g sec⁻¹ with epoxy powder.

 Table 2

 Characteristics of the tribo-gun and corona gun

Characteristic (Se	Tribo-gun outhampton-NRD type)	Corona gun C
Power unit	None	40 to 120 kV
Ion current	0	10 to 100 µA
Powder current	0.6 µA	2 µA
Layer adhesion (acceleration required for removal)	200G	2000G
Coating quality	Very smooth	Smooth
Back ionisation	None	Yes
Self limiting process	By repulsion	By ejection and back ionisation
Powder q m ⁻¹	$3 \times 10^{-4} \mathrm{C/kg}$	10 ⁻³ C kg ⁻¹
Limiting coating thickness	100 to 150 µm	50 to 100 µm

New types of coating

It is easy to take the view that electrostatic processes can be used only for the production of polymer coatings (wet or dry), but polymers having a resistivity in the range >10¹¹ Ω m are now deposited by conventional electrostatic means. For $\rho = 10^{11} \Omega$ m it is possible to obtain satisfactory films provided that precautions are taken. At least two new powder materials are currently being applied by a modified electrostatic technique. These are briefly described below.

Vitreous enamel

The Wolfson Applied Electrostatics Unit at Southampton has developed and patented an encapsulation technique in collaboration with an industrial firm. By treatment with a hydrophobic chemical, the resistivity of vitreous enamel powders can be raised from $10^9-10^{10} \Omega m$ to $>10^{13} \Omega m$. Vitreous enamel can then be applied using conventional electrostatic powder guns. The unfused coating behaviour is similar to that obtained with pôlymers.

The advantages of such a film are low material cost, excellent anti-corrosion properties and good high temperature performance, plus low material costs. The main disadvantage is that very high fusion temperatures are required.

Aluminium coating of sheet steel

The British Steel Corporation at Shotton has developed a fullscale plant in which aluminium powder is deposited electrostatically on to steel sheet in a continuous process. The problem of powder layer adhesion is solved by using a liquid pretreatment of the steel, followed by cold rolling and heat treatment.

The film has a diffusion bond with the substrate. In this way sheet steel having the anti-corrosive properties of aluminium can be obtained. Any subsequent paint treatment is required only for decorative purposes.

Optimum coating conditions

It is possible to plot on one graph the various parameters which are important in determining the final finish (Fig. 4). The corona current from gun to object is plotted against gun voltage for various spacings. The powder current is given by $I_P = (dm/dt)(q/m)$ and an example is given for a powder throughput of 5 g sec⁻¹. To produce this current by corona charging, an ion current of about 25 μ A is required. The maximum values of V_g and I_g for the gun are determined by the powder unit regulation curve (shown shaded). A back ionisation current density $>5 \times 10^{-3}$ A⁻² m causes moon cratering and this gives a maximum I_g of about 120 μ A for a 20 \times 20 cm object.

It will be seen that the optimum operating conditions are contained in a relatively restricted region ABCD for a spacing of 10 cm. With a tribo-gun there would obviously be more of these restrictions.

Conclusions

Electrostatic powdet coating provides an extremely powerful method for producing polymer films. As scientific understanding of the basic processes improves, it should be possible, by improving the deposition systems, to produce films having superior appearance, and to do so with greater reproducibility than is possible at present.

[Received 24 April 1976

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Next month's issue

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

Pigmentation of water-soluble alkyd/melamine resin combinations by N. A. Ghanem F. F. Abd El-Mohsen and S. El-Zayyat

The influence of metal surface structure on the adhesion of a coating of a thermosetting resin by E. W. Garnish

The packaging of hazardous goods by C. Swinbank

The painting of timber under adverse weather conditions by J. Boxall

The role of new technologies in the packing industry by A. J. Newbould

Relative permittivities of some aniline- and toluidine-substituted anthraquinones (Short Communication) by G. F. Lewin and S. J. Chapman

Student Review Colour, structure and electrons

By C. H. J. Wells

School of Chemical and Physical Sciences, Kingston Polytechnic, Kingston upon Thames, Surrey

Couleur, structure et électrons-un aperçu pour stagiares

Farbe, Struktur und Elektronen-Eine Studentenbesprechung

The rainbow is a dramatic display in nature of the range of colours in the visible spectrum—violet, indigo, blue, green, yellow, orange and red. This phenomenon of the dispersion of sunlight into its component colours was reported by Newton in 1672 when he observed that sunlight shining through a glass prism also gave rise to the visible spectrum. Since then the factors which give rise to colour in organic compounds have been the subject of extensive study, and the relationship between colour, structure and the electronic processes occurring in organic compounds is now well understood.

When visible radiation is absorbed by a molecular system, the energy uptake results in the promotion of an electron from a molecular orbital of lower energy to one of higher energy. It may be considered that molecular orbitals are formed by a linear combination of the atomic orbitals of atoms within the molecular structure. The combination of two atomic orbitals gives rise to two molecular orbitals-one a bonding orbital and the other an anti-bonding orbital-and the electrons forming the σ or π bond in the molecular structure occupy the corresponding bonding orbital. There is a third type of molecular orbital to be considered in the case of organic compounds containing heteroatoms. In this situation, those electrons associated with the heteroatom which are not involved in the bonding system of the molecule occupy non-bonding molecular orbitals (n-orbitals). This is illustrated for formaldehyde in Fig. 1 where there are two electrons in the non-bonding $2p_y$ orbital on the oxygen atom.



Fig. 1. Non-bonding orbital in formaldehyde

The relative energies of the different types of molecular orbital in organic molecules is shown in Fig. 2. The energy of a non-bonding orbital is normally greater than that of a σ or π orbital, but less than that of the anti-bonding orbitals. However, there are instances of molecules having a high degree of conjugation where the energy of the π orbital is greater than that of the *n* orbital.

The possible types of electronic transition between orbitals in organic compounds are represented by the vertical arrowed lines in Fig. 2. Electronic transitions occur upon absorption of electromagnetic radiation of energy equal to the energy difference between the orbital from which the electron originates and the orbital into which the electron is promoted,



Fig. 2. Relative energies of molecular orbitals and possible transitions between orbitals

and are seen as absorption bands in the ultraviolet/visible absorption spectrum of a compound. For example, the ultraviolet absorption spectrum of formaldehyde exhibits three bands (Fig. 3) and these have been assigned to the different types of electronic transition as shown in Fig. 3. The $n \rightarrow \pi^*$ transition which gives rise to the absorption band at 280nm can be depicted in terms of the electron configuration



Fig. 3. Absorption spectrum of formaldehyde

of the molecule before and after the transition in the following way:

[inner electrons] $\sigma^2 \pi^2 n^2$		[inner electrons] $\sigma^2 \pi^2 n^1 (\pi^*)^1$
	transition	
Ground state, S ₀		Excited state, S_1

Here, the ground state molecule S_0 is converted to an energetically excited state molecule S_1 upon absorption of the energy required to promote the $n \rightarrow \pi^*$ transition. This transition and the other two transitions; $n \rightarrow \sigma^*$ and $\pi \rightarrow \pi^*$, which give rise to the absorption bands of Fig. 3 are represented by the vertical arrowed lines in Fig. 4.



Fig. 4. Electronic states of formaldehyde and electronic transitions between states

A band in an ultraviolet/visible absorption spectrum is indicative of an electronic transition, and since the band spreads over a range of wavelengths, the corresponding electronic transition must occur over a range of energies. This is a consequence of the fact that each electronic state $(S_0, S_1, S_2, ...)$ has an associated set of vibrational energy levels. These levels are quantised and each has a vibrational quantum number, V. As shown in Fig. 5a, there can be a number of transitions originating from the lowest vibrational level (V=0) of an S₀ state to the various vibrational levels of the S₁ state, and these transitions will require slightly different energies. If a high resolution spectrometer is used to record the absorption spectrum then the individual transitions may sometimes be observed in the form of vibrational fine structure on the absorption band (Fig. 5b). Normally, however, bands in the absorption do not exhibit fine structure and only the band envelope (dotted line of Fig. 5b) is observed. Obviously, vibrational energy levels play an important role in determining the colour of a compound since the existence of these levels causes the absorption of the compound to be spread over a range of wavelengths.





Fig. 5a. Electronic-vibrational transitions, and Fig. 5b. Absorption band showing vibrational fine structure



Fig. 6. Relationship between colour and absorption in the visible region

Compounds are coloured when one or more of the absorption bands fall within the wavelength range of visible radiation (Fig. 6). For organic compounds, such bands usually correspond to $n \rightarrow \pi^*$ or $\pi \rightarrow \pi^*$ transitions. The other possible electronic transitions, $\sigma \rightarrow \sigma^*$ and $n \rightarrow \sigma^*$, normally require greater energy than $n \rightarrow \pi^*$ or $\pi \rightarrow \pi^*$ transitions, so that the corresponding absorption bands occur at shorter wavelengths, usually outside the visible region.

The energy range of the visible region is from 4.96 $\times 10^{-19}$ J (equivalent to 400nm) to 2.48 $\times 10^{-19}$ J (800nm), and for an organic compound to be coloured the energy difference between a *n* and a π^* orbital or between a π and a π^* orbital must fall within this range. Compounds in which this occurs generally possess one or more of the following structural features:

- (i) an extended conjugated system of double bonds,
- (ii) a conjugated system of multiple bonds, which involves atoms having non-bonding electrons,
- (iii) a multiple bonded system, involving non-bonding electrons and charges.

The relationship between colour and the extent of double bond conjugation is demonstrated in Fig. 7 for a series of



Fig. 7. Band envelope of longest wavelength absorption band of some aromatic hydrocarbons

hydrocarbons—benzene, naphthalene, anthracene, naphthacene and pentacene. The Figure shows the position of the longest wavelength absorption band for each of the compounds, and it can be seen that as the extent of conjugation increases so the band moves to longer wavelengths, and that in the case of naphthacene and pentacene the band has moved into the visible region. Naphthacene and pentacene are coloured yellow and blue respectively, whilst benzene, naphthalene and anthracene are colourless. The bands in Fig. 7 each represent $a \rightarrow \pi^*$ transition, and thus the effect of increasing conjugation is to decrease the energy for the $\pi \rightarrow \pi^*$ transition. Examples of other coloured compounds exhibiting extended conjugation and which have $\pi \rightarrow \pi^*$ absorption bands in the visible region are given in Fig. 8.



Fig. 8. Some examples of compounds which are coloured due to the influence of extended conjugation

Absorption bands arising from $n \rightarrow \pi^*$ transitions frequently occur in the visible region for compounds which have the structural features (ii) and (iii) listed above. Some examples of coloured compounds with these features are shown in Fig. 9.



Fig. 9. Some examples of compounds which are coloured due to the influence of extended conjugation and non-bonding electrons

In conclusion, colour in an organic compound normally arises because the energy of an electronic transition of the $n \rightarrow \pi^*$ or $\pi \rightarrow \pi^*$ type falls within the range corresponding to visible radiation and the compound has the ability, in consequence, to absorb such radiation. The actual colour of the compound is dependent upon the wavelength range of the visible radiation absorbed, which in turn is dependent upon the structure of the compound.

[Received 24 July 1976

Review

Treatise on Coatings. Vol. II Characterisation of Coatings; Physical Techniques Pt. II

By R. P. Myers and J. S. Long (Eds)

Basle and New York; Marcel Dekker Inc., 1976 Pp. xvi \pm 661. Price Sw. Fr. 222

The present volume completes the section on Physical Techniques in Myers and Long's "Treatise on Coatings". It comprises ten chapters by different authors, with consequent differences in approach and emphasis. The most notable difference, readily apparent, is in the amount of space and detail given to the application of the techniques in the field of surface coatings. In Chapter 1, for example, "Dielectric Phenomena", after a systematic treatment of the theory, Shiniki Negami devotes one page to a rather cursory review of applications (Is not the identity of the α transition erroneous? Surely not glassy-to-liquid?). K. A. Boni devotes two pages to "Gel Permeation Chromatography" (the treatment of the subject is particularly readable). K. M. Oesterle, in a comprehensive review of "Viscometry of Coating Materials", gives seventeen pages. Most of the other authors are also reasonably generous in this direction, namely D. M.

Gans in "Interfacial Energetics", M. Levy in "Nuclear Magnetic Resonance" (excellent on identification and structure of polymers), R. Davies on "Particle Sizing" (principally non-microscopic methods), L. H. Princen on "Scanning Electron Microscopy in Coatings Research" (surface structures of films and fractures), J. L. Gardon and J. P. Teas in "Solubility Parameters" (the tables of solubility parameters of media and pigments are particularly useful here), G. S. Park in "Transport in Polymer Films" (a comprehensive account of the sorption and permeation of gases, vapours and liquids in polymers, with an interesting account of the effect of pigmentation), R. W. Scott in "X-ray analysis; Diffraction and Emission". Reference to applications has been emphasised because most readers, like the reviewer, will consult such a book to see what problems can be solved by the techniques described.

What would have been particularly valuable would be a chapter by an independent authority reviewing critically the various methods and where they overlap (as many do), and giving some guidance as to the precise applications and limitations. Admittedly, this is not usual in compendia of this nature, but it would have been a very useful addition for the hard pressed technologist. Otherwise, the volume takes its place as an excellent work of reference.

J. L. PROSSER

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Chromate based pigments—epidemiological studies

A statement issued by the British Colour Makers' Association

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In August 1975, the British Colour Makers' Association issued a statement, "Lead chromate based pigments—toxicological considerations", in which reference was made to epidemiological studies being carried out in the USA and Europe. The results of the epidemiological study commissioned by the Dry Colour Manufacturers' Association in the USA are now available. They have been issued as a final report of Equitable Environmental Health Inc., with the following conclusion:

One is left with the fact that there is an excess of respiratory cancer in the workers studied; that the excess generally occurs in older long-term workers; that the excess excluding men with mixed exposure is based on only three deaths; and that the relative amount of the excess cannot be determined precisely. The findings are consistent with the hypothesis that lead chromate is a respiratory carcinogen, but do not justify any other conclusion.

Extensive studies are now being undertaken in Europe and the United Kingdom with large populations of lead, zinc and other chromate workers. The evidence provided by those studies may clarify the situation.

Thus, the United States study does not enable the announcement of an unequivocal statement with regard to lead chromate.

The US study covered 548 workers, compared with the UK study which will cover approximately 1100 workers, in one plant going as far back as 1933, and the continental study which will cover approximately 2000 workers.

The problems of collating medical data are greater in some countries than in others, which will result in some delay in the completion of the total study. It is expected that the Cancer Research Institute, London, and the German Cancer Research Institute will complete their evaluations during 1977.

The American Conference of Governmental Industrial Hygienists (ACGIH) recommends threshold limit values (TLVs) for chemical substances and physical agents. In its 1976 booklet, chromates of lead and zinc have been included under "Notice of intended changes—Appendix A, Carcinogens". These compounds have been categorised as "industrial substances suspect of carcinogenic potential for man" and have been assigned a TLV of 0.1 mg m⁻³ as CrO₃.

Similarly, in the Threshold Limit Values List 1976, under Item B of Appendix III "Carcinogenous work materials", the Senate Commission of the "German Research Association for Checking Work Materials that Put Health in Danger" has taken the precaution of indicating that for lead chromate a carcinogenic potential may be assumed. Until further data is available in this respect, the present figure of the maximum concentration at the working place for chromic acid in the form of CrO_3 will be maintained at 0.1 mg m⁻³.

It is most desirable to limit contact with chromate based pigments as far as possible. If operating practices are such that the TLV quoted cannot be maintained, protective equipment (dust masks, etc.) should be used as supporting measures.

The TLV for chromates is established (by the ACGIH) as the time weighted average concentration for a normal 8-hour work-day or 40-hour work-week, to which nearly all workers may be repeatedly exposed, day after day, without adverse effect. In the chromate pigment user industries, in contrast to the pigment manufacturing industry, there is little likelihood of continuous eight-hour exposure, but constant vigilance is, nevertheless, essential.

Purchasers requiring further information are requested to contact their own pigment suppliers.

Section Proceedings

Hull

The fundamentals of UV curing

The second ordinary meeting of the Section was held at the "George Hotel", Land of Green Ginger, Hull on Monday 1 November 1976. Sixteen members and visitors assembled to hear a lecture delivered by Mr R. C. Smith of A. Holden & Co. Ltd, entitled "The fundamentals of UV curing".

Mr Smith began by comparing the thermal and UV processes for curing films. The main attributes of UV curing were considered to be (a) its energy efficiency (b) no solvent emission (c) small production units and less maintenance, and (d) suitability for curing films on heat sensitive substrates.

The basic photochemistry involved was fully discussed, together with the pertinent reactions of carbonyl groups. The various ingredients in a typical UV-curable formulation were detailed and information was given on the requirements on properties of photoinitiators of various chemical types, oligomeric media and diluents. Photochemical ionic polymerisation was also mentioned in the discussion.

The UV process was used in the following application areas: (a) printing and overprint varnishing of paper, card, tin-plate and metal foil; (b) metal decoration of beer cans, etc; (c) wood finishing; and (d) vinyl floor tiles. No pigmented systems (except in inks) were being used commercially. Limited pigmentation of lacquers was possible, but the level of cure obtained became dependent on the amount of transmitted UV radiation, a factor which in turn was dictated by the absorption coefficient of the pigment used.

This lecture was well received and provoked much thought and discussion. The meeting was brought to a close with a vote of thanks from Mr A. R. Vanspall, which produced a cordial response from the audience.

D.M.W.

London

New approaches to the development of properties in paint films

On Tuesday 16 November, the Section held a lunch-time meeting at the Sheraton Heathrow Hotel, Colnbrook, Middlesex, when 28 Members from London and Thames Valley Sections and guests heard Professor Henry Schreiber present a lecture entitled "New approaches to the development of properties in paint films". Professor Schreiber (Head of the Chemical Engineering Department, Ecole Polytechnique, University of Montreal and Visiting Professor at McGill University) in opening his lecture discussed the factors which affected and directed development work within the coatings industries.

A number of areas of expertise were detailed which Professor Schreiber considered it necessary for the successful development chemist to understand—these were the fundamentals of a highly sophisticated and technical industry. A knowledge of synthetic organic chemistry, of the physical characterisation of polymers, of colloid and interfacial science and mechanical engineering were some examples. In practice, however, a problem was usually solved by reformulating or developing a new product, which did not give the particular problem. This method avoided the approach of evaluating and examining the fundamental aspects of the problem.

Although some basic concepts were used to predict the properties of solutions and coatings, their value was often restricted. Solubility parameter was quoted as an example. Accurate results were obtained only for ideal solutions—which practical formulations were not! It did not work very well for polar solvents and so could not be used for water-borne Professor Schreiber then indicated some alternatives to solubility parameter, which could be used to predict the properties of solutions. He concentrated on the concept of surface tension, which was both time and temperature dependent.

He showed how it was possible to determine a value, critical surface tension γ_c , for the surface tension of the solid polymer from measurement of the contact angle of liquids of known surface tension.

The temperature dependence of surface tension was determined and measured with a thermogradient bar and Professor Schreiber described how γ_e could be calculated from measurement of droplet areas of liquids of known surface tension at different temperatures on the bar.

Some of the work carried out at the Ecole Polytechnique was described in detail. During the course of the work, as well as indicating the properties of particular solutions, the method also confirmed previously determined parameters, such as glass transition temperatures etc. In particular, Professor Schreiber described how the solubilities of different plasticisers in a terpolymer resin varied with temperature from the measurement of γ_c values and how this could be used to predict the response of the plasticised films to such physical phenomena as water absorption.

It is hoped that a fuller account of the work will be published in the *Journal*.

A.J.N.

Water-thinned anticorrosive paints

A one-day meeting of the Section was held on Wednesday 17 November in conjunction with the London Branch of the Institution of Corrosion Science and Technology at Thames Polytechnic, S.E.18. Four lectures were presented under the title of "Water-thinned anti-corrosive paints".

The morning session was chaired by Dr C. Booker (Chairman of London Branch, I. Corr, T.), and after an introduction by Dr R. Grzeskowiak of Thames Polytechnic, Mr G. Keith (Rohm & Haas Ltd) presented a lecture entitled "Acrylic emulsions in anticorrosive paints".

With a series of slides, Mr Keith indicated the ways in which emulsions could be used to replace conventional alkyd paints for outside maintenance and decorative painting. He concentrated on maintenance painting and showed how, on both exterior and accelerated weathering, emulsion primers and systems gave as good, and sometimes a better result than solvent-borne alkyds, epoxies, polyurethanes and vinyls.

The results of various field trials in a number of different industrial atmospheres were shown and discussed. In conclusion, Mr Keith said that although acceptable results had been obtained with acrylic emulsions there were still some areas which needed to be improved before they were universally used.

Mr B. Gilliam of Cowan Colours talked about "Anticorrosive pigments in aqueous primers". He first indicated that if all the toxicity data were taken into account none of the pigments currently considered for their anticorrosive properties could be used, as they were all poisonous to some animals or fishes.

Mr Gilliam then reviewed in some depth a number of different anticorrosive pigments for water-borne coatings which were currently considered acceptable from the human toxicity view-point.

The results of an independent evaluation of six anticorrosive pigments in various resin systems were discussed and it was shown that a particular resin required a particular anticorrosive pigment for optimum substrate protection. In general, however, strontium chromate gave the best humidity results and basic lead silico-chromate the best salt-spray results. These results also showed the importance of the correct choice of pigment for a particular atmospheric environment.

In his lecture on "Water-thinned paints for structural steelwork" Mr J. Bravery of Berger Paints firstly stated that there was very little interest in general in water-borne coatings, but that in specific areas, such as the painting of double-skinned ships' hulls, they had been used to eliminate fire risk and solvent fume exposure.

The limitations of water-borne coatings both from formulation and testing practicalities and of the final coating's performance were discussed. The importance of correct surface preparation was stressed and type of surface was shown to be more variable than the coating on exterior exposure.

Mr F. Richardson (Thomas Swann & Co.) and Mr M. Shulz (Akzo Chemie) presented a joint paper entitled "Waterthinned epoxy coatings". Mr Richardson described the development work involved in producing a fully water dispersible epoxy system which was viscosity stable and had acceptable resistance properties. At present the most suitable systems still had slower curing rates and shorter pot lives than solvent-borne materials.

The lecture also described how a water-borne epoxy/pitch coating could be formulated with the same flexibility, cost and chemical resistance as solvent-borne coatings.

It was thought that the short pot life of water-borne epoxies would be overcome in the future, but in all probability the poor cure under adverse conditions, such as high humidity and low temperatures, would have to be suffered.

Mr Shulz in his part of the presentation showed slides of the uses of water-borne epoxy coatings in various industrial environments in Europe and Scandinavia.

The day symposium was concluded by the Chairman of the afternoon session, Mr J. Tooke-Kirby, Chairman of the London Section, with a vote of thanks to both the lecturers and the audience for their contributions and participation.

Manchester

Moisture curing polyurethane one-pack coatings with the emphasis on corrosion protection

A meeting was held on Friday 12 November 1976 at the Woodcourt Hotel, Brooklands Road, Sale, for a talk to be given on "Moisture-curing polyurethane one-pack coatings with emphasis on corrosion protection" and a total of 27 Member and visitors attended this interesting lecture at a fog-bound venue.

The speaker, Dr M. Sonntag (Bayer AG), on a flying visit, described the full range of coatings from clear coatings, via zinc dust primers, micaceous iron oxide intermediate coats to aliphatic isocyanate based polyurethane polymer top-coats. Single-pack pitch coatings were also discussed; the subject of moisture removal from the various components remained important. The lecturer claimed no problems with intercoat adhesion.

The partial replacement of zinc dust with zinc oxide appeared a surprising reversal of previous technical trends. Two years' Florida and 5/6 years' natural weathering experience was claimed for the one-pack coatings.

After a lively question time, based on both moisture pre-removal and post-exclusion, Mr W. McCallum offered a mixed metaphorical Germanic vote of thanks, remarking that in Manchester it never rained on a Sunday, but was always foggy on a Friday.

F.B.W.

ALN

Newcastle

Use of microvoids as pigments

The second meeting of the 1976/77 session was held on 4 November 1976 when 28 Members and guests assembled to hear Mr J. Clark of Tioxide International Ltd present a paper entitled "The use of microvoids as pigments". After reviewing the use of air as an opacifying agent in bubble coatings, Mr Clark outlined the process for the manufacture of vesiculated polymer beads and demonstrated the properties of paints prepared from them. Although only suitable for matt applications, vesiculated polymer beads produced paints with a greater resistance to staining than conventional paints of the same degree of pigmentation. Good flow properties could be obtained if the paints were correctly formulated and viscosity stability and freeze-thaw resistance had been shown to be satisfactory. This potentially exciting new development lead to a question and answer session involving a number of Members of the audience before the meeting was closed.

T.H.

Transvaal

Coal tar enamels

A lecture was given to a meeting of the Transvaal Section on 26 August 1976 entitled "Coal tar enamels—their manufacture and preparation" by Mr R. E. Rouse.

Mr Rouse started his lecture with a résumé of the formution of the different coals from peat to anthracite, containing roughly 50 to 94 per cent carbon and about 65 to 15 per cent volatiles at 900°C on a dry, ash-free basis. Coking of low ash coals at 1000°C to produce metallurgical coke, yielded the crude tars plus liquids and gases, whilst the crude tars in turn yielded the well-known refined tars, pitches and creosotes.

As surface coatings, the tars and pitches, whilst having excellent adhesion and water resistance, suffered from the disadvantages of "alligatoring" and a temperature dependence which could result in a definite flow with small increases in temperature or a very marked embrittlement with small temperature decreases. Modifications with other binders only partially alleviated the problems.

The methods for reducing the temperature susceptibility which had been proved best by experience, was that of high temperature digestion of 15 to 25 per cent of suitable powdered bituminous coals in soft pitches or anthracene oils to produce so-called "modulates". The softening point of the coal tar was increased, the brittle point decreased, the "solution" became stable and the enamel no longer showed "alligatoring". To enhance mechanical strength, up to 35 per cent of fillers, such as talcs, might be incorporated.

These enamels were usually applied to clean, preferably grit-blasted steel as molten compounds at temperatures above 180°C, often in conjunction with paper or other wrappings, for example to pipelines.

R.B.

West Riding

Water-based stoving finishes

A meeting of the Section was held at the Griffin Hotel, Leeds on Tuesday 12 October 1976. Mr G. R. Brown of Harlow Chemical Co. Ltd, gave a lecture on the subject of water-based stoving finishes.

By way of introduction, the lecturer said that the achievements so far in this field fell short of the aims, although there had been a few areas of success. There were three main categories of aqueous polymer systems: aqueous dispersions, solubilised polymers and water-reducible systems. Mr Brown discussed the characteristics of each type with particular reference to particle size, molecular weight, viscosity and solids content and compared them with solvent-based polymer systems. He then made some comments concerning the formulation characteristics of each type. Finally, there was reference to the types of functional groups used to achieve crosslinking in these systems and an illustration of some of the possibilities available from a particular commercial product. In response to questions, Mr Brown emphasised that there was no universal product to satisfy all requirements and there was a need for close co-operation between resin supplier and user to design a product for a particular end use. He agreed that water pollution from solid waste was a problem which should not be ignored and could be expensive to overcome. At the present time, it was also true that water-based systems tended to be more expensive than solvent-based systems.

A vote of thanks was proposed by Mr K. Smith.

R.A.C.C.

South African Division Sixth National Symposium

Non-conventional coatings

The sixth South African National Symposium was held at the Holiday Inn, Port Elizabeth on the 8 and 9 October 1976. The theme of the symposium was "Non-conventional coatings". Sixteen papers were presented, and 170 delegates attended, including a number from overseas: this represented the highest attendance ever achieved at an OCCA South African Symposium.

The Symposium was opened by Mr K. M. Dienst, Chairman of the South African Paint Manufacturers' Association, who referred to the close collaboration between the SAPMA and the South African Division of OCCA in many matters of mutual interest and in particular in the field of education.

The keynote address was given by Dr T. L. Webb, Director of the National Building Research Institute of the Council of Scientific & Industrial Research. Dr Webb felt that some indication of the Symposium's importance and potential benefits could be obtained from the fact that in 1974 no less than 112 million litres of coating material was manufactured in South Africa. He then went on to discuss ways and means of reducing health hazards and pollution, energy conservation and the economics of coatings, including labour costs involved in application. He concluded with some speculation on possible future developments in specialised coatings.

The First Technical Session consisted of two papers:

"The performance of some non-conventional anti-corrosive primers for steel" by Professor D. E. A. Williams-Wynn*.

This paper described the work carried out on the evaluation of several pigments which were being promoted as replacements for the more toxic anticorrosive pigments used in primers. Performance was evaluated by salt-spray tests and natural exposure in Durban.

"Adhesion failure of organic coatings" by Professor W. J. McGill*.

This paper dealt with the adhesion to solid surfaces of organic coatings applied in the fluid state. The basic science of adhesion was covered and the principle of wetting and adhesion were discussed.

The Second Technical Session consisted of two papers:

"Toxicity and the paint industry" by Mr E. A. Duligal.

This paper reviewed current knowledge of the toxic hazards which might arise in the application of coatings. The proposed National Standard covering the toxicity of coatings was explained.

"Future developments in industrial coatings with particular reference to ecological and energy requirements" by Mr A. G. North. This paper was delivered by Mr M. C. Horton.

A wide range of industrial coatings, including powder coatings, radiation cured and water based, high solids and non-aqueous dispersions were compared for performance, environmental pollution and energy requirements.

The Third Technical Session consisted of two papers:

"Developments in electrodeposited coatings" by Dr R. L. Nicolay*.

The process of electropainting was fully described with specific reference to the situation in South Africa. The problems which could arise were discussed and possible solutions were suggested.

"The application of methylated amino resins in nonconventional coatings" by Mr R. McD. Barrett*.

In non-conventional coatings, such as powders, waterborne and high solid systems, methylated amino resins had been found to be more suitable than conventional butylated resins. The advantage of hexamethoxy methyl melamine (HMMM) and its use as a co-monomer to introduce functionability into polymer chains was fully described.

The Fourth Technical Session consisted of two papers:

"Some new epoxy coating systems" by Mr W. A. Williams.

Various types of curable epoxy coatings were discussed with special reference to solvent-free coatings, powder coatings and water-dispersible coatings. A discussion of the effect on film properties and overall performance of the newer curing agents and diluents and the improvements which could be expected from new resins currently under development in solvent-free systems was included.

"Testing methods developed for the evaluation of epoxy powder coatings" by Mr C. H. J. Klaren*.

Production control methods particularly applicable to powder coating manufacture were discussed, together with details of a study made to correlate the thermal behaviour of powder coating systems with their ultimate film properties.

The Fifth Technical Session consisted of three papers:

"Prefinished metals" by Mr P. P. Williams and Mr P. L. Kimber. This paper was presented by Mr P. L. Kimber.

The major features of a modern coil coating line were described. Cleaning, pretreatment and coating were discussed

^{*}The papers marked with an asterisk in this report have been accepted by the Hon. Editor for publication in the *Journal*. It is expected that they will be appearing in issues printed during the summer this year.

1977 (1) SECTION PROCEEDINGS



The photograph above was taken at the South African Division's Symposium Dinner and shows, from left to right: Mr E. Wright, Chairman of the Transvaal Section, Mr D. J. House, Chairman of the Natal Section, Dr H. Marais, guest speaker, Mr D. J. Pienaar, Symposium Chairman and S. A. Division Vice-President, Mrs Marais, and Mr G. Warman, Chairman of the Cape Section

in detail and the various coating systems which could be applied were detailed. This lecture was supplemented by a film.

"High performance sealants" by Mr I. Nel.

This paper reviewed the various modern sealants used in the construction and transport industries—namely those based on polysulfide, polyurethane and silicone elastomers. Applications and possible future developments were discussed.

"Synthetic resin seamless floor coverings and their application" by Mr R. E. Cromarty.

Seamless floorings based on epoxy, polyurethane and polyester resins were described and compared. The necessity for good substrate preparation was stressed and various methods detailed. This lecture was also supplemented by a film.

The sixth Technical Session consisted of two lectures:

"Processing automotive surface coatings" by Mr R. Meyer.

This paper dealt with production processing and subsequent field performance. Factors determining in-plant process controls with regard to phosphating, dip priming, primer surfacer and enamel top coats were discussed. Sealer and surfacer application, enamel processes and repair techniques were described. Differences between alkyd and acrylic top coats, including metallics, were analysed.

"Future trends in automotive finishes" by Mr L. L. Hagemann.

The present system used for the primer, primer surfacer and topcoat were reviewed. At present about 85 per cent of the South African assembly plant paint usage was solvent borne and spray applied. It was explained that the ideal paint system for the future had to be non-polluting, should cure at low temperatures, reduce the number of processes and improve corrosion protection. For the future, water-borne, high solids and powder coatings were the main contenders, with waterborne types emerging as the current favourite.

The seventh Technical Session consisted of two lectures:

"Can coatings" by Mr P. G. Nicolay.

The various current techniques of applying organic coatings in can manufacture were reviewed. In surveying the roles played by the various types of internal coatings to prevent reaction between contents and the metal of the container, the conditions peculiar to each of the categories of foodstuffs and beverages were discussed. Possible future developments in can coatings, including the use of UV curing, were covered.

"Liquid polybutadiene resins for surface coatings" by Dr P. Aukett and Dr A. R. Luxton*. The paper was presented by Mr G. C. Curme.

This paper reviewed the various types of liquid polybutadiene resins now commercially available. It was explained that the major influences on surface coating properties were due to the micro-structure and the molecular weight. The chemistry of n-butyl lithium catalysed polymers was described and properties and applications of this type of resin were discussed.

The eighth Technical Session consisted of one lecture and the open discussion on all papers:

"The importance of specialised coatings and their use in the nuclear power industry" by Mr M. A. Brett. This paper was presented by Mr C. Brett.

It was explained that with the advent of nuclear power construction in South Africa, a new challenge was presented to the surface coatings industry to produce suitable coating systems for use in nuclear power stations. The paper dealt with the manufacture, surface preparation and application of specialised coatings for use in nuclear power construction plants.

"Open discussion".

A panel of all the speakers at the symposium was formed and written questions were dealt with as well as questions from the floor. The discussion was brisk and this form of questioning proved most successful.

A summation of papers was presented by Mr E. P. Wright, Chairman of the Transvaal Section. The Symposium was closed by the Symposium Chairman, Mr D. J. Pienaar, South African Division Vice-President. A vote of thanks was expressed to the staff of the Conference Division of the Council of Scientific and Industrial Research, with whom the Conference was organised and arranged. The technical sessions were followed by a Symposium Dinner.

The sixth National Symposium was the first South African symposium since the inauguration of the South African Division. Apart from papers presented great benefit was obtained from very free discussion held outside the conference room. Many old friendships were renewed and new friends made.

P.A.J. G.

Information Received

Baxenden go Scandinavian

Baxenden Chemical Company Ltd, the Lancashire based manufacturer of urethane chemicals and rigid polyurethane foam, has set up a wholly owned subsidiary, Baxenden Scandinavia ApS, based in Sorø, Denmark. Baxenden has been supplying urethane foam systems to Scandinavia for many years through its licensee, Chemitan A/S.

Baxenden has acquired the assets of Chemitan, and is now manufacturing in Denmark for all its Scandinavian customers. The Danish company has its own technical service laboratories, and these will be expanded to offer the same high degree of customer service with which the UK company is associated.

In the UK Baxenden supplies urethane chemicals, formulated systems and elastomers to a wide variety of markets, including construction, transport, furniture, surface coatings and industrial insulation. The company plans to use the Danish base as a springboard to these markets across the whole of Scandinavia.

Baxenden Scandinavia ApS will also distribute and service the machinery supplied by the UK subsidiary company, Isofoam Ltd.

Joint distribution for ICI propylene glycols

ICI Petrochemicals Division has appointed two official distributors in the United Kingdom for small lot unit orders of its mono- and dipropylene glycols. The distributors are Durham Chemical Distributors Ltd and Brenntag (UK) Ltd. A pharmaceutical grade of monopropylene glycol is available for cosmetics, food and essences and pharmaceutical applications.

New service for US paint and colour specifications and standards

London Information Ltd, based in Ascot, Berkshire, has signed an exclusive agreement with Global Engineering of California to distribute the US government, military, industrial, and technical Society and Association specifications and standards.

Global has the largest military/industrial technical specifications and standards library of its kind in the world with hundreds of thousands of specifications, standards, handbooks and technical reports in stock. London Information has an index of all the information which is stored in America and on-line access direct to the Global Engineering data-bank in California.

UV coatings for paper

A new company, Paper Lacquers Ltd, has been formed for the purpose of marketing a range of ultraviolet-cured coatings specifically for application to paper and cardboard. The coatings will be manufactured by Pearl Paints Ltd, of South Wales, under licence from Detmolder Lackfabrik Niessen & Söhngen of Detmold, West Germany.

Paper Lacquers Ltd will be responsible for all technical servicing and full laboratory facilities have already been established in South Wales for the preparation and application of samples. Detmolder Lackfabrik has been engaged in the manufacture and supply of these lacquers for a number of years and has gained considerable 36



A new fully automatic filling machine for the paint industry developed by Bexuda Limited

experience which will be available to UK customers.

UK agents for Air Products and Chemicals Incorporated

Alfa Chemicals Ltd, 9a High Street, Staines, Middlesex, has announced its appointment with effect from the beginning of this year as the sales representatives of the Chemical Additives Division of Air Products and Chemicals Inc., USA. The Air Products "Dabco" range of urethane catalysts, the "LK" series of non-silicone polyurethane foam surfacants, the "PEP" promotors for polyesters and the "VS 103" vinyl foam stabilisers will be available.

New products

Centrifugal dry discharge filter from M & M-Schenk Filters back in production

The British built Schenk ZHF centrifugal dry discharge filter is again in full production at the new enlarged premises of the Mason & Morton Group in Hemel Hempstead after removing from their previous smaller premises in Watford and Harrow Wealdstone. The filter consists of a vertical pressure vessel in which horizontal filter elements are mounted on a central hollow shaft. These horizontal elements are fitted with filter media which vary according to the product. Solids build up on the filter elements in the form of a cake which is removed by spinning the filter stack mechanically or hydraulically. The cake can also be treated before spinning for discharge.

New instrument for particle size analysis

Coulter Electronics Ltd has announced the Coulter Counter Model TA II with Population Count Accessory as a result of increasing demand from industry for instrumentation to carry out particle size analysis quickly and accurately. The measuring range of the new instrument is between 0.6 and 800 µm and the time taken for analysis can be reduced to as little as 90 seconds. Results are displayed visually on an integral oscilloscope and can be plotted on an XY recorder or presented digitally on a built-in strip printer in the Population Count Accessory. The new machine individbally counts and sizes up to 5000 particles per second, and particle size distributions are reproducible to within ± 1 per cent over a wide range of differing materials.

New tinting system from Berger

Berger Paints, UK paints division of the Berger group, has launched a completely new paint tinting system, "Colorizer 420", based on technology developed jointly by Berger and its parent company Hoechst.

Colorizer 420 is to replace the existing Colorizer system, which already has over half the tinting sector of the market. There are 420 colours in the range, covering the whole colour spectrum and including the deep, bright shades which have not so far been available from shop-tinted paint.



The Schenk ZHF centrifugal dry discharge filter from M & M-Schenk Filters Ltd

The products have also been completely reformulated and now give parity of performance in all respects with premium brand ready-mixed paints.

Colorizer 420 is built around 12 colourants and five bases. The colourants, based on Hoechst "Hostatints", were developed by the Berger Paints Laboratories in Hengrove in conjunction with Hoechst pigment experts in Frankfurt.

Solar radiometer calibration

Desert Sunshine Exposure Tests Inc, based in Arizona, USA, one of the world's largest independent outdoor weathering and solar device testing facilities, has recently announced a new service, the calibration of solar radiometers (pyranometers and pyrheliometers). DSET is the only independent commercial source in the United States for solar instrument calibration.

Calibrations are performed in accordance with World Meteorological Organization procedures whenever applicable and include 0° horizontal or any other angle from the horizontal. Tilt angle errors, cosine response, azimuth response, and linearity of response are also determined. DSET's primary standard is an Eppley HF Absolute Cavity Radiometer, which can be operated either in the Pancred or Angstrom mode. DSET's secondary working standard is an isolated Eppley Model PSP precision pyranometer, fully characterized by the HF Cavity Radiometer.

Conferences, courses etc.

Conservation: pigments and dyes

A one-day symposium with the title "Studies in conservation: colouring agents, pigments and dyes" is being organised by the University of Edinburgh Extra-Mural Department, in conjunction with the UK Group of the International Institute for the Conservation of Historic and Artistic Works. The symposium will take place on Friday 15 April 1977 in Edinburgh. It is designed to bring together workers, researchers and historians concerned with historic and artistic materials and to promote discussion on the problems of conservation of this type of material.

Particle size analysis

A Conference organised by the Particle Size Analysis Group of the Analytical Division of the Chemical Society will be held at the University of Salford, Manchester, from 13 to 15 September 1977. It is envisaged that all aspects of particle characterisation will be covered. Those wishing to attend the Conference or to present a paper should write to Dr D. Dollimore, Reader in Physical Chemistry, University of Salford, M5 4WT.

Pigment school

The Wolfson Organic Powders Research Unit in the Department of Colour Chemistry and Dyeing. The University of Leeds, is to hold a residential course on modern pigment technology at the University from 6 to 9 September 1977. This will be the third such course in the past ten years devoted to the research, manufacture and use of



A twin-head modular semi-automatic filling machine for foaming and non-foaming liquids for the food, paint and chemical industries, recently introduced by Neumo Limited, Newhaven, Sussex

organic and inorganic pigments in the coloration industries. Full details are obtainable from Director of Special Courses, Department of Adult Education and Extramural Studies, The University, Leeds LS2 9JT.

Literature

Carboxylic acid ester

Dynamit Nobel Chemikalien has extended its range of information sheets for the works, laboratory and lecture-hall with its new brochure on ortho-carboxylic ester. The possibilities of use and chemical mechanism of orthoformic and orthoacetic acid ester are presented and give the reader a detailed review of the materials, which goes well beyond the scope of just an information sheet. The booklet comprises 45 pages with an index, and can be obtained free of charge on request.

Cellulose ester lacquers

R. H. Chandler Ltd has published the twenty-sixth part in its series of bibliographies in paint technology "Recent developments in cellulose ester lacquers 1965–1975" by Mrs G. Chinnick. The work revises the literature (consisting of mostly patents), provides paint production statistics and gives individual abstracts of 132 patents.

Electronic spectra of anthraquinones

An English translation (185 pages) of "The electronic spectra of anthraquinones" is now available in a spiral bound form, from Dr R. W. A. Oliver, 71 Harboro Road, Sale, Cheshire, England. The Russian text was produced originally by Fain in Moscow in 1970. It consists of an introduction and tables containing data on 2830 absorption spectra of anthraquinones, 1315 substituted forms and of anthraquinone itself in a variety of different solvents.

Polyurethane resin index

A new publication has been issued by Synthetic Resins Ltd, Liverpool, listing the company's range of polyurethane resins for surface coating, sealing, adhesive, flooring and impregnating applications. The index, printed in three colours, tabulates the physical and chemical properties of oil based, modified alkyd, moisture curing, pre-polymer, elastomeric and water based resins.



A very small part of the specifications and standards library in America which is now on-line to London Information Ltd (see page opposite)



New Exhibitors

The Exhibition Committee is pleased to report a number of additions to the first list of Exhibitors published in the December 1976 issue of the *Journal*, and these organisations are mentioned below.

Any company still wishing to apply for Exhibition space should contact the Director & Secretary immediately, since there is only a very limited amount of stand space left in the hall.

Venue

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

Dates and times

The 1977 Exhibition will take place at Alexandra Palace, London, N.22 on the following dates and times:

Tuesday	22	March		09.30 -	17.30	hrs.
Wednesday	23	March	G.	09.30 -	17.30	hrs.
Thursday	24	March		09.30 -	17.30	hrs.
Friday	25	March		09.30 -	16.00	hrs.

Theme of the Exhibition

The Committee emphasises that whilst it naturally encourages the showing of new products it does not stipulate that new products have to be shown by exhibitors each year and it attaches equal importance to the advantage to personnel at all levels of meeting and discussing their common technical problems in an informal atmosphere.

Refreshments on stands

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

Facilities at Alexandra Palace and travel arrangements

Visitors to OCCA-28 last year were impressed by the facilities available, which included two restaurants, two bars, a cafeteria and an exhibitors' bar. Other facilities include this year ample free car

OCCA-29 Exhibition

22-25 March 1977 at Alexandra Palace, London

attracts support from 15 countries

★ Belgium ★ Denmark ★ East Germany ★ Holland ★ Hungary ★ ★ Italy ★ Norway ★ Poland ★ Romania ★ Spain ★ Sweden ★ ★ Switzerland ★ UK ★ USA ★ West Germany ★

The continuous dialogue between suppliers and manufacturers

parking space, which is of considerable benefit especially to those using the M1 motorway, which links with the North Circular Road.

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



A view of part of the Edinburgh Room at Alexandra Palace, which will be open between 11.30 and 14.30 hrs on the four days of the Exhibition and where tables may be reserved

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

"Official Guide"

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

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

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

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

For the last two exhibitions, it was decided that a small charge of £2.00 (US \$5.00) should be made both for the "Official Guide" and for season admission tickets to the exhibition. This policy undoubtedly deterred casual visitors who might otherwise be attracted to exhibitions for which no admission charge was made and who gathered quantities of technical literature from the stands. The innovation was welcomed by many exhibitors and in no way acted as a deterrent to visitors to this exhibition. A similar charge will be made for the "Official Guide" to OCCA-29.

Information in foreign languages

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

News of Exhibitors at OCCA-29

Chemolimpex

The Hungarian trading company for chemicals, *Chemolimpex*, will again be showing its export range of products for the surface coatings industry, including resins, driers, solvents and catalysts. A hydroxyl-function synthetic resin for polyurethane resins and xanthan resin/emulsifier will be on display.

Degussa

The Pigments Division of Degussa, Frankfurt am Main, has developed a new furnace black which is marketed under the designation Special Black 10 and is recommended for toning and pigmenting primers and finishes. Special Black 10 helps greatly in eliminating flooding and floating problems with grey finishes in paint systems with differing binders, for example in alkyd resin, acrylate resin and polyurethane finishes. Very good results can be achieved even with low pigment volume concentrations.

Special Black 10 is readily dispersible and can be processed rationally. In many cases, only little dispersion energy is required: that is, stirring in of the carbon black with with a high-speed agitator or dissolver suffices. When using machines, such as the pearl mill, ball mill or three-roller mill, it is possible to reduce considerably the milling time usually required for carbon black or the number of milling passes. The use of wetting agents is generally unnecessary.

Degussa will be showing on Stand 18 at the 1977 Exhibition.

ICI Limited

Erratum: It was incorrectly reported in the December 1976 issue of the Journal that ICI Mond Division would be exhibiting additives and chemical intermediates at the 1977 Exhibition. The company will, in fact, be exhibiting the additive "Thixomen", a new thixotropic agent for chlorinated rubber based paints and other systems, and the raw materials "Alloprene" chlorinated rubber and "Cereclor" chlorinated paraffin for the manufacture of paints and allied products.

A new single system process for separating oil and water, with distinct advantages over other currently available systems, has been introduced by the *Pollution Control Systems* organisation of *ICI*.

The new process is available internationally, along with the company's other effluent treatment products, knowledge and services.

The separation system known as "Flofoil", was developed within *ICI* and results from the company's wide range of technologies. In one simple process, it can reduce concentrations of oil in water by better than 99 per cent, treating high volumes of effluent at low running costs.

The heart of the system is the coalescing media which have special surface properties: the oil droplets are captured, and as they migrate through the media, they coalesce into larger droplets; their release on the other side is controlled so that they do not break up, as often happens with other systems, but rapidly rise to the surface to form a continuous layer which is easily removed. The media are very easily installed, and the process can be used either alone, as an ancillary to upgrade an existing oil separation system, or in conjunction with other *Pollution Control Systems* products and processes, like "Flocor" plastics media or flotation.

Synres

For the research department of *Synres*, the emphasis remains on the development of binders giving less pollution problems than the present paint and varnish systems. *Synres* will show the important progress which has already been made, particularly with regard to water dilutable wood coatings, decorative paints and coil coatings.

A polyester resin for high solids stoving enamels will be demonstrated, as well as a new grade of resin-modified phenolic resin for the printing ink industry, which completes the *Alsynol RL* range so successfully introduced to the heatset web offset sector.

The stand will also feature *TROY Chemical's* range of preservatives, fungicides and other products.

UCB s.a.

In addition to its range of resins for the surface coatings industry, UCB hopes to display on its stand a particular application of one of its latices normally used for dispersion paints. This consists of a mixture of a latex with sand, fillers and appropriate additives, which is used as a glue (resinous mortar) for fixing ceramic tiles instead of using cement mortar.



A Chemcol mixer, which will be shown on the John Godrich stand

Additional Exhibitors at OCCA-29

Since the printing of the first list of Exhibitors at OCCA-29 in the December 1976 issue of the *Journal*, the following organisations have applied for stand space or will be represented in conjunction with another exhibitor:

G. J. Erlich Limited will be returning to the Exhibition in 1977 and will show on stand 61. The company has moved premises since it last exhibited and is now at Erli House, Tillington Place, Petworth, West Sussex, (Tel: 0798 42805. Telex 877287).

Industrial Colours Ltd, Parsons Green Lane, London SW6, will be exhibiting its range of aqueous fluorescent pastes and other pigment preparations.

Schwerdtel GmbH will be exhibiting in conjunction with *KWR Chemicals Ltd* on Stand 36.

Shear Colour Ltd, Chilton Industrial Estate, Sudbury, Suffolk, the sole UK distributor for *Sun Chemical Corporation*, *Pigments Division*, will again be represented on the *Sun* stand.

Sweco (Europe) SA, Belgium, will be exhibiting on the *Herbert Smith & Co* stand and will show a separator which, in addition to being used for the sieving of dry coatings, is also recommended for the separation of solids from liquids and for specific dewatering situations and scalping applications with relatively coarse screen meshes. Many parts and accessories are available. Warwick Chemical Ltd, Willow Lane, Mitcham, Surrey, exhibited at OCCA-28 last year and the then newly formed company illustrated many of the application areas in the surface coatings field which it serves and also gave an insight into its activities in the chemical and textile sector.

This year greater emphasis is being laid on the company's commitment to the surface coatings industry and a number of newer products will be shown for the first time. These will include *Muichanol* 42, which is a phenolic modified rosin ester specially developed to meet the latest requirements of the heatset, offset process, as well as other types in the *Michanol* range of resins for ink making.

The company will also be illustrating the latest additions to the *Surkyd* and *Mitchalac* ranges, including alkyds for industrial paints and specialised coatings.

Werner & Pfeiderer (UK) will be exhibiting in conjunction with Herbert Smith & Co Ltd and will show a range of twinscrew compounding machines for the production of powder coatings. These versatile units are available in three sizes for production rates of either (approximately) 20, 240 or 750 kg per hour. The machines continuously melt and mix a preblend of all ingredients (resins, pigments, fillers, hardeners and other additives, including preworked powder) and discharge the compound in the form of a viscous liquid on to a cooling band for subsequent crushing and grinding.

ASSOCIATION CONFERENCE 1977



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

In the November 1976 issue of the Journal (pp 420–425) full details were given of the lectures which are to be presented at the Association's Conference together with biographies of the lecturers.

Copies of the Conference brochure and registration form were enclosed in all copies of the December 1976 issue of the Journal sent to Members attached to the General Overseas Section and non-member subscribers to the Journal. The brochures are being sent to all Members attached to the UK and Irish Sections with Section circulars. Any non-member who has not yet received a copy of the brochure and wishes to register for this important Conference should apply for the necessary form to the Association's offices. Non-members wishing to avail themselves of the preferential Conference fee for Members may do so, by sending in a membership application form at the same time as they submit their Conference registration form and the fee enclosed should cover both the Membership entrance fee, 1977 subscription and the Conference registration fee. Membership application forms can be obtained from the Association's offices.

The registration fees for the Eastbourne Conference will be £40.00 (plus Value Added Tax at the standard rate) for Members, £60.00 (plus VAT) for nonmembers and £15.00 (plus VAT) for wives. The Association Conference has been organised on this occasion to include a weekend and it will assemble on the evening of Thursday 16 June 1977 and disperse on the morning of Sunday 19 June. The headquarters of the Conference will be the Grand Hotel, Eastbourne, Sussex, England, where the lecture sessions will be held on Friday 17 and Saturday 18 June. The Association's AGM will take place on 18 June at the Grand Hotel.

Preprints of the papers will be prepared in the coming months and will be circulated to those who have registered for the Conference several weeks before the event, so that lecturers will be able to give a brief introduction to the topic and a longer period will then be available for discussion purposes. This has been the practice at revious Association Conferences and has proved of immense value to those attending. Of the 14 papers being presented to the Conference, two from the United State, are designated as being presented to behalf of the Association's sister society, the Federation of Societies for Coatings Technology. There are two contributions from Germany and one from Holland. In addition, it is hoped that a paper from a Scandinavian source will be presented on behalf of the Scandinavian Federation of Paint & Varnish Technologists.

A full social programme has been arranged for the benefit of those attending the Conference, including delegates' wives, and this includes golf and table tennis tournaments, and coach parties to places of interest and beauty spots in the locality.

A theatre party and a Reception for Overseas Visitors will be arranged for the evening of 16 June, a Civic Reception for the evening of 17 June, and the Association's Dinner Dance for the evening of 18 June.

SLF Convention 1976



The photograph above was taken on the occasion of the 1976 SLF Convention held in Helsinki and shows the four Presidents of the international alliance. From left to right: W. Dunn (FSCT), L. O. Portin (SLF), A. T. S. Rudram (OCCA), Dr L. J. Kovacs (FATIPEC)

News of Members

The following Ordinary Members have been appointed as Officers and Council of the British Colour Makers' Association for the year 1976-77:

> Chairman Mr G. K. Burrell (London—Associate member)

Vice-Chairman Mr H. Slack (Manchester)

Honorary Treasurer Mr F. B. Mortimer-Ford (London) Mr J. Bradley (Manchester)

Mr W. B. Cork (Hull)

Mr P. Perkin (Manchester)

Mr H. B. Smith, an Ordinary Member attached to the London Section and an Associate in the Professional Grade, has been appointed to the Board of Odhams (Watford) Limited as Technical Director.

Mr Smith studied chemistry at the Northampton Polytechnic, EC1 and has been employed by the company for some 25 years.

Hull Section Dinner and Dance



The photograph taken at the 1976 Dinner and Dance of the Hull Section shows, standing from Inc photograph taken at the 1976 Dinner and Dance of the Hull Section shows, standing from left to right: Mr A. R. Van Spall (Social Secretary, Hull Section), Mr S. R. Finn (Honorary Editor), Mr R. Brooks (Vice-Chairman, Hull Section), Mr T. W. Wilkinson (Chairman, Hull Section), Mr J. Mitchell (Chairman, Manchester Section), Mr K. V. Hodgson (Chairman, Newcastle Section), Mr R. P. Bartrum (Chairman, West Riding Section), Mr J. Wenham (Hon. Secretary, Hull Section), Mr E. Armstrong (Immediate Past-Chairman, Hull Section, and Toastmaster). Seated from left to right: Mrs Van Spall, Mrs Brooks, Mrs Wilkinson, Mrs Mitchell, Mrs Bartrum, Mrs Wenham and Mrs Armstrong

Annual Dinner and Dance

The Section's annual Dinner and Dance was held at the Willerby Manor Hotel, Willerby, near Hull on Friday 1 October 1976. As in the past, the function was well attended by members and guests from far and wide. After an enjoyable meal, the Chairman, Mr T. W. Wilkinson, welcomed the top table guests and also explained the differences between the choice of plants available, as is customary, for the ladies' gifts. The toast was then proposed to the ladies and guests.

The reply on behalf of the ladies and guests was made by Mr J. E. Mitchell, Chairman of the Manchester Section. In conclusion, he asked all present to join him in a toast to the Hull Section. Dancing continued until the early hours of the morning

OCCA Sixtieth Anniversary

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

Midlands Section

Trent Valley Branch

Works visit

Twenty-two members and friends spent a very interesting evening on 17 November touring the factory of J. W. Thornton Ltd, of Belper, manufacturer of sweets and chocolates. New and intricate methods of manufacture were explained, and the traditional old-fashioned hand-made chocolates were also shown. Visitors were allowed to sample the wares, and after tea and biscuits in the canteen, when management and staff were thanked by Mr J. A. Burns, they also visited the sweet shop. The Branch's next visit is on 24 February to Mandora Soft Drinks Ltd, at Mansfield.

Register of Members

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

Ordinary Members

- ACLE DE CACERES, JOSE ARTAZA 2, 3 C-Lejona, Vizcaya, Spain.
- Амвику, John, 125 Jeffcoat Drive, Rexdale, Ontario M9W 3B9, Capada
- Canada. (Ontario) ASBURY, GEORGE, 12 Pritchett Road, Birmingham B31 3NL.
- (Midlands) BELL, ERIC JAMES, 1 Doral Way, Carshalton, Surrey SM5 3ST.
- (London) BLAKE, ROGER MALCOLM, 18 Queens Road, Tamboerskloof, Cape Town, South Africa. (Cape) BROWN, GEORGE DIXON, Lawter Chemicals (Canada) Ltd, 29 Iron
- Street, Rexdale, Ontario M9W 5E3, Canada. (Ontario) CROWLEY, LORNE ANTHONY, Ciba-Geigy (NZ) Ltd, PO Box 2103,
- Wellington, New Zealand. (Wellington) EBINGER, ADOLF, 158 Rosemount Avenue, Weston, Ontario,
- Canada. (Ontario) GASCOYNE, JOHN, LRIC, 37 Grayshott Close, Birmingham.
- (Midlands) HARTMAN, NICHOLAS PETER, BSc, 29 Gatteridge Street, Banbury, Oxfordshire OX16 8DJ. (London)
- HITCHINS, MICHAEL WARREN, W. Graham Hitchins, Box 13,
- HITCHINS, MICHAEL WARKEN, W. Granaut Hitchins, Do Lee, Renwick, Marlborough, New Zealand. (Wellington) REID, JAMES CURRIE, BSC, PhD, Furniture Industry Research Association, Maxwell Road, Stevenage, Herts. (London) RIORDAN, RORY, BA, 7 Ben Viljoen Street, Summerstrand, Port (Corrol)
- Elizabeth, South Africa. (Cape)

SAXBY, ALAN CYRIL, 15 Morley Hill, Stanford-le-Hope, Essex.

- (London) SHED, TIAN CHUAN, BSc, 55 Jalan 21/31, S.E.A. Park, Petaling Jaya, Selangor, Malaysia. (General Overseas)
- STOKES, CHRISTOPHER JAMES, R. J. Stokes & Co. Ltd, Little London
- STOKES, CHRISTOPHER JAMES, N. J. SIOKES & Co. Lid, Ethic Echnolin Road, Sheffield S8 OUH. (West Riding)
 VERVOORT, ADRIAN, Marine Oil Refiners of Africa Ltd, Dido Valley, Simonstown 7995, South Africa. (Cape)
 WALDRIDGE, DERKE JOHN, MA, BSC, ICI Ltd, Paints Division, Wexham Road, Slough, Berks. (Thames Valley)
- WATKINS, LESLIE, 368 Newlove Drive, Bolton, Ontario, Canada. (Ontario)

Registered Student

GRAHAM, SEAN KEITH, 207 City Road, Sheffield S2 5HG. (West Riding)

Associate Members

- BUKIN, DOUGLAS, 50 Clares Green Road, Spencers Wood, Berks RG2 IDY (London)
- BYRNE, MICHAEL, 19 Old Coach Road, Johnsonville, Wellington, New Zealand. (Wellington)
- FEIKES, KLAUS KARL, 636 Petrolia Road, Downsview, Ontario M3J 2W3, Canada. (Ontario)
- HOLLIS, ROBERT PHILLIP, 41 Thistledown Road, Shard End, (Midlands) Birmingham.
- JACKSON, KENNETH FISHER, K & K Greeff Industrial Chemicals Ltd, Westburn Road, Cambuslang, Glasgow. (Scottish) (Scottish)
- KERR, DAVID, 48 Gainsborough Drive, Durban North, Natal, South Africa South Africa.
- RANSON, STEPHEN GEORGE, Buckley & Young Ltd, PO Box 30148, Lower Hutt, New Zealand. (Wellington) (Wellington)

Forthcoming Events

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

January

Thursday 6 January

Newcastle Section: "New coatings based on aromatic acids" by a speaker from Amoco Chemicals, to be held at the Royal Turks Head Hotel, Grey Street, Newcastle upon Tyne, commencing at 6.30 p.m.

Monday 10 January

Hull Section: "The optimum use of organic pigments" by Mr D. M. Varley, CIBA-GEIGY (UK) Ltd. To be held at the George Hotel, Land of Green Ginger, Hull, commencing at 6.30 p.m.

Tuesday 11 January

West Riding Section: "Extenders in emulsion paints" by Mr D. J. Huxtable, English Clays Lovering Pochin & Co Ltd, to be held at the Griffin Hotel, Boar Lane, Leeds, commencing at 7.30 p.m.

Wednesday 12 January

Manchester Section: "Micro-emulsions" Manchester Section: Micro-emuisions by Mr G. H. Falkin, Perstorp AB, to be held at the Manchester Literary and Philosophical Society, George Street, Manchester, commencing at 4.30 p.m.

Thursday 13 January

London Section: "Micro-emulsions" by Mr K. H. Falklin, Perstorp AB, Sweden. Evening meetings at the Princess Alice, Romford Road, London E7, commencing at 7.00 p.m.

Midlands Section-Trent Valley Branch: "Bulk handling of powders and pigments" by Dr N. Harnby, University of Bradford, to be held at the Crest Hotel, Pastures Hill, Littleover, Derby, commencing at 7.00 p.m.

Friday 14 January

Manchester Section: "The use of micro-voids as pigments" by Mr J. Clark, BTP Tioxide Ltd, to be held at the Manchester Literary and Philosophical Society, George Street, Manchester, commencing at 6.30 p.m.

Scottish Section: Annual Dinner Dance at the Albany Hotel, Glasgow.

Wednesday 19 January

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

Thursday 20 January

Scottish Section: "Pigment dispersions" by Mr R. S. Monk, Kenroy Dispersions Ltd, to be held at the Beacon's Hotel, 7 Park Terrace, Glasgow G3, commencing at 6.00 p.m.

Friday 21 January

Irish Section: Ladies' Evening with "Antiques" lecture by Mrs A. Dalton, RIDpl, AIDP, Andrian Interiors, to be held at the Clarence Hotel, Dublin commencing at 8.00 p.m.

Midlands Section: Dinner Lecture: "High solids systems" by Dr Uerdingen, Bayer AG, to be held at Birmingham Chamber of Commerce and Industry, Harbourne Road, Edgbaston B16, at 6.30 for 7.00 p.m.

Thursday 27 January

Thames Valley: "Pigments for the printing industry" by Mr A. J. Green, Horace Cory & Co. Ltd, to be held at the Beaconsfield Crest Motel (White Hart), Aylesbury End, Beaconsfield, Bucks, at 6.30 for 7.00 p.m.

Friday 28 January Bristol Section: "Smoke emission from polymer and paint films" by Mr K. A. Safe, Vinyl Products Ltd, to be held at the Royal Hotel, Bristol, commencing at 7.15 p.m.

Friday 28 January

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

February

Thursday 3 February

Newcastle Section: "Some techniques in the management of research" by Mr F. Westwick, Department of Management Studies, Sunderland Polytechnic, to be held at the Royal Turks Head Hotel, Grey Street, Newcastle upon Tyne, commencing at 6.30 pm.

Friday 4 February

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

Monday 7 February

Hull Section: "The alternative technologies for industrial finishes" by Mr J. Rackham, BTP Tioxide Ltd, to be held at the "George Hotel", Land of Green Ginger, Hull, commencing at 6.30 pm.

Tuesday 8 February

West Riding Section: "Silicone resins in the surface coatings industry" by a speaker from Dow Corning Ltd.

Wednesday 9 February

London Section: "Printing on textiles". Joint day meeting with the Society of Dyers and Colourists and in association with Thames Polytechnic, commencing at 10.00 am.

Scottish Section: "Aspects of packaging" by Mr A. D. Lotte, Metal Box Company Limited. Joint meeting with the Eastern Branch at the Carlton Hotel, Edinburgh, commencing at 7.30 pm.

Friday 11 February

Manchester Section: "Organic versus in-organic coatings" by Mr J. R. Lyon, Goodlass Wall, to be held at The Woodcourt Hotel, Sale, Cheshire, commencing at 6.30 pm.

Thursday 17 February

Midlands Section: "The Health and Safety at Work Act" by Mr M. Kelly of the University of Aston in Birmingham. Joint meeting with Trent Valley Branch to be held at the Birmingham Medical Institute, 36 Harbourne Road, Birmingham.

Friday 18 February

Irish Section: Lecture by a speaker from Bayer AG to be arranged.

Newcastle Section: Ladies' Night at the Five Bridges Hotel, Gateshead.

Thursday 24 February

Thames Valley Section: "Masonry paints" by Mr P. Whiteley, Building Research Establishment, to be held at the Beaconsfield Crest Motel (White Hart), Aylesbury End, Beaconsfield, Bucks, at 6.30 for 7.00 pm.

Friday 25 February

Bristol Section: "Paper in relation to printing processes" by Mr A. T. Franklin of Reed Paper and Board (UK) Ltd, to be held at the Royal Hotel, Bristol, at 7.15 pm.

March

Wednesday 23 March

Ontario Section: "Organic pigments for trade sales paints" by Mr R. E. Edelman, Du Pont USA. To be held in the Starlight Room, Skyline Hotel, Toronto.

Newcastle Section

Second UV Symposium—September 1977

A second symposium entitled "UV polymerisation and the surface coatings industry", sponsored by the Newcastle Section of the Association, is to be held at the University of Durham on 14 and 15 September 1977. Delegates staying at the University will assemble on evening of 13 September and disperse on evening of 15 September. Papers are to be presented by industry covering the practical aspects of the rapidly developing technique of UV curing. Topics will include photoinitiated polymerisation, UV curing equipment, resins, photoinitiators and photosensitive monomers. Further details may be obtained from Mr H. Fuller, Tioxide International Ltd, Carlton Weathering Station, Yarm Back Lane, Stockton on Tees, Cleveland TS21 1AX.

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4. Resins as Media for Paints

Oleoresinous media: Alkyd resins as media. Cellulose lacquers and media. Solvents for cellulose lacquers. Manufacture of lacquers. Other solvent-soluble, film-forming high polymers. Spirit variables. Solvent and dirers: solvents, driers. Some uses of clear finishes: insulating variables, wood finishing by cellulose lacquer. Water-based paints for industrial use.

5. Pigments and Extenders

5. Pigments and Extenders Dispersion and wetting: wetting, wetting agents, *Inorganic Pigments*: white pigments: titanium dioxide, antimony oxide, zinc oxide, lithopone, white lead, zinc phosphate, Extenders: barytes, blanc fixe, whiting, precipitated chalk, silica, choina clay bentonite, state powder, mica, asbestime, talc. Black, pigments: carbon blacks, vegetable (lamp) black, bone and drop blacks. Metallic pigments: aluminum, bronze powders, zinc. Oxides of lead; red leaf instance, the state state oxide of the instance of the state oxide of the state of the state of the state oxide of the powders, zinc. Coxides of lead; red leaf instance, the state oxide of inori-yellow, bydrated oxides, reds and browns, black oxide. Chrome pigments: lead chromes, zinc chromes, zinc tetroxychromate, stronium chromate. Blue pigments: prussian blue, ultramarine blue, Green pigments: chrome (Brumswick) green. Flooding and floating, Cadmium pigments. Nickel titanate. Organic pigments: aclasification of organic pigments; sigment dyestuffs, toners, lakes, Chemical classification: zao pigments, alkid ibue or reflex blue, phthalocyanine pigments; sitaning power or tinting strength. Opacity or hiding power. Oil absorption Water-soluble matter. Light-fastness, *Chemical constitution*. Purity: Conditions of exposure, measurement of lightfastness, *Pigment lazards*: toxicity, dust.

6. Decorative and Structural Paints

Types of decorative and structural paints. Sealers. Primers: primers for wood, primers for alkaline and porous surfaces, primers for iron and steel, the mechanism of corrosion, protective mechanism of paint coatings, metal cleaning and pretreatment, blast primers, etch (or wash) primers, zinc-rich primers, zinc silicate primers, zinc phosphate primers, red coid/aline chrome primers, red oxid/aline chrome primers, red coid/aline chrome primers, red oxid/aline linseed oil primers, calcium plumbate primers, primers for galvanised surfaces.

Undercoats, fillers and stoppers: stoppers, fillers, undercoats, Finishes: alkyd-based finishes, 'Non-drip' or thixotropic finishes, semi-gloss and eggshell finishes, oleoresin-ous finishes, epoxy-resin finishes, epoxy-resin finishes, epoxy-resin finishes, maxoury paints. Emulsion paints: nature of emulsions, preparation of polymer emulsions, homopolymers and copolymers, manufacture of emulsion paints, additives, properties and uses of vinyl emulsion paints, acrylic emulsions

7. Industrial Stoving and Cold-curing Finishes, including Marine Finishes

Industrial finishes. Table of media used for various purposes, alkyds, alkyd/amino combinations, epoxy and epoxy ester resins, epoxy/pitch combinations epoxy/ acrylamide acrylic resins, hydroxylated acrylic/amino resins, hydroxylated acrylic, isocyanate resins, thermoplastic acrylic resins, cellulose finishes, polyurethane finishes, polyester finishes, non-aqueous dispersion finishes, phonolic resin finishes, water-thinmable finishes, solicone resins, zinc silicate finishes, oil-free polyester finishes, solicone resins, infrared radiation, induction, dielectric heating, U.V. curing, electron beam curing, *Paint application*; padding, spraying, airless spraying, electrostic spraying, hot spray technique, dipping, 'toto-dip' process, flow coating, curtain coating, roller coating, silk screen method, vacuum and pressure impregnation, electro-deposition, powder coatings, strip coating, aerosol spraying.

8. Paint Manufacture and Hazards

Paint milling: function of paint mills, General principles of paint mills, *Tipes of mill*: premixers, mixers for spates, mixers for slovers, paddle mixers, high speed mixers. Dispersing mills, Dispersing mills, mills requiring premixers, *Triple* roll mills, single roll mills, colloid mills, the sand mill, sealed sand mill, the Dyno' mill. Dispersing mills, direct charge: the ball mill, the attrictor mill, high speed dispersers, the kady mill, Thiming mixers. Straining, Filling, *Health and fire hagards*; toxicity, pigments, resins, solvents, fire, other fire risks.

9. Common Defects of Varnishes and Paints

Varnishes: bloom, blushing, pinholing and cissing, silking, Paints: blistering, checking, cissing, crawling, dirt collection during drying, flotation, livering, pinholing, pigment sedimentation, rivelling, sagging, curtaining and tears, silking, skinning.

10. Paint Testing

Liquid paint: viscosity, brushability, colour measurement, colour matching, lightfast-ness, opacity, drying time, *The diried film*; adhesion and elasticity, hardness, gloss, film thickness, wet and dry, fineness of grind, water resistance, humidity resistance, salt spray resistance, durability, weatherometers.

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