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January 1978



OCCA CONFERENCE 1979

OURNAL OF THE IL & OLOUR HEMISTS' SSOCIATION

A new zinc phosphating process designed for automatic control of composition N. M. Ness and B. A. Cooke

The determination of the formation times of films cast from polymer dispersions I. Anzur, U. Osredkar and I. Vizovišek

Anomalous swelling in reactive polymers

J. Sickfeld

Recent aspects on the preparation and evaluation of some polyesteramides for surfacing coatings. Part II: Preparation and evaluation of amide containing resins *A. M. Naser and A. Z. Gomma*

FIRST LIST OF EXHIBITORS OVER 100 ORGANISATIONS REPRESENTED See page 33



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Increased DPP capacity, Stanlow, UK.

Opening of Polymers Research Centre, Amsterdam. New VeoVa plant, Moerdijk, Netherlands. New DPP plant, Pernis, Netherlands. New Versatic acid capacity, Pernis, Netherlands.

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The Association celebrates the 60th Anniversary of its foundation in May 1978, and Council has arranged two functions to commemorate this important event:

Thursday 11 May 1978

A Commemorative Lecture and Dinner will take place at the Painters' Hall, Little Trinity Lane, London EC4 when John Methven, the Director-General of the CBI, will give the Foundation Lecture on "The place of business in our society". The lecture will be followed by the presentation of a special silver medal to John Methven by the Master of The Worshipful Company of Painter-Stainers, Sir Ralph Perring, Bt. The charge for tickets, which includes the reception, wines with the Dinner, and port or brandy, will be £12.00 each, plus VAT.

Friday 12 May 1978

The Biennial Dinner and Dance will take place at the Savoy Hotel, London WC2, when principal officers of other societies will be invited to attend as guests of the Association. The reception will take place in the River Room at 7.00 p.m. and Dinner will commence at 7.30 p.m. in the Lancaster Room. Arrangements for a cabaret have been made and dancing to the Jay Langham Orchestra will continue until 1.00 a.m. The charge for a single ticket will be f15.00, plus VAT. 1

It is expected that there will be a heavy demand for tickets for these functions, and persons wishing to receive application forms should write to the Director & Secretary at the address below. Early application is advised.

OIL AND COLOUR CHEMISTS' ASSOCIATION

Priory House, 967 Harrow Rd., Wembley HA0 2SF

JANUARY

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Full details of the regulations for admission to the Professional Grade were last published in the April 1977 issue of the *Journal* (*JOCCA*, 1977, **60**, 157). 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. The full list of members in the Professional Grade is published each year in the December issue of the *Journal*.

Transactions and Communications-

A new zinc phosphating process designed for automatic control of composition*

By N. M. Ness and B. A. Cooke

Imperial Chemical Industries Ltd, Paints Division, Wexham Road, Slough.

Summary

It is generally accepted that automatic control of solution composition of a spray zinc phosphating process can provide useful gains in coating quality and consistency. Hitherto, the complexity and expense of the equipment dictated by the complicated process chemistry has militated against wider adoption of automatic

Keywords

Types and classes of structures or surfaces to be coated steel

control. A recent study of the chemistry and measurements of a parameter "chemical efficiency" have shown that given careful formulation of the accelerator, automatic control by sensing only conductivity is perfectly feasible.

Processes and methods primarily associated with:

surface preparation before coating

pretreatment surface preparation zinc phosphate treatment

application of coatings and allied products

spray application

Un nouveau procédé de traitement au phosphate de zinc pour permettre le contrôle automatique de composition de la solution

Résumé

On admet en général que le contrôle automatique de composition de la solution de phosphate de zinc utilisée dans un procédé de phosphatation par pistolage peut assurer des bénéfices utiles à l'égard des frais, de la qualité, de la constance. Jusqu'a present, la complexité et les prix du matériel exigé par la chimie complexe du procédé s'opposent à l'utilisation plus importante des systèmes de contrôle automatique. Une étude récente sur la chemie et d'ailleurs les mesures d'un paramètre, "l'efficacité chimique", ont démontré que le contrôle automatique basé uniquement sur le dosage de la conductivité au moyen d'un système sensible est entièrement practicable, pourvu que la composition de l'accélérateur soit soigneusement étudiée.

Ein neues Zinkphosphatierungsverfahren zur Verwendung für die automatische Kontrolle der Zusammensetzung

Zusammenfassung

Es wird allgemein anerkannt, dass automatische Kontrolle der Zusammensetzung einer Lösung von im Phosphatierungsprozess verwandten Zinkphosphal-Spritzlösung von grossem Nutzen für die Qualität und Gleichmässigkeit der zu erzeugenden Schicht sein kann. Bisher wirkten sich die Kompliziertheit, sowie die Kosten für die Ausrüstung, welche die erforderlichen umständlichen

Introduction

Ref. 1

It is now generally accepted that the most satisfactory types of spray zinc phosphate process for use prior to painting, and especially prior to electro-painting, are those which yield fine complete coatings of relatively low terminal coating weight, say in the region of 2gm⁻². Several of the recent advances in the field of pre-paint treatment of steel, especially those adopted in the motor industry, have had as an objective the provision of an intrinsically fine, compact zinc phosphate coating. Whilst these processes have undoubtedly been successful, it remains the case that the result produced in a given plant often varies from time to time, perhaps from hour to hour or even more frequently. Obviously, such chemischen Prozesse diktierten, gegen die Einführung automatischer Kontrolle aus. Eine kürzliche Untersuchung des Chemismus und der Messungen eines Parameters "chemischer Nutzeffekt" erwies, dass bei sorgsamer Rezeptierung des Beschleunigers automatische Kontrolle lediglich durch Wahrnehmung der Leitfähigkeit durchaus durchführbar ist.

fluctuations can arise as a result of variations external to the phosphating stage (quality of steel, efficiency of cleaning, rinsing, etc), but the sheer complexity of the chemical processes occurring during zinc phosphating renders this stage especially prone to cause variations in coating efficiency. Some of the reasons underlying changes in bath composition, which could cause fluctuations in quality, are discussed in a previous paper¹ which dealt with the continuous changes in composition predictable from the differing reactivities of the various constituents of the bath. Shorter term fluctuations can be of even greater significance; they might be caused by line stoppages of short to medium duration or by the corrective measures associated therewith, as well as a host of possible inadvertent factors.

Many of these problems, and especially the short term

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fluctuations, can be countered by the adoption of automatic composition control. However, the established methods of automatic bath control available for zinc phosphating stages are relatively complex and expensive and have, consequently, made little progress in this country. The authors' objective was, therefore, to develop a spray zinc phosphate process giving intrinsically fine coatings of low terminal weight which is amenable to composition control by the sensing of a single bath parameter using simple, robust and inexpensive electrochemical and electronic equipment. Electrolytic conductivity was considered especially commendable in this regard.

Selection of accelerator

Refs. 2, 3

The volatility of nitrous acid and its consequent fluctuation in concentration, independent of other factors in the bath composition, excludes conventional nitrite-accelerated processes from consideration if automatic control is to be based on the sensing of only a single parameter. A further difficulty with such processes lies in the fact that the additive supplying the nitrite, generally sodium nitrite, performs a dual function: it is alkaline in relation to the bath and, therefore, reduces its free acid content. Such a reduction is, in fact, necessary owing to the excess acidity of the acidic zinc phosphate concentrate added and conventional processes have been arrived at by experience, so that the dual functions balance approximately under average conditions. It is impossible, however, to contrive that they do so under all possible line conditions. A separation of the two functions, acceleration and neutralisation, is therefore required to meet the objective.

This can be achieved by means of an involatile accelerator present in excess in the bath. The best known accelerator of this kind is chlorate, which has long been known to produce intrinsically fine coatings and many successful processes have been based on this fact. The disadvantage found in operating chlorate-accelerated spray processes at a realtively low temperature (around 50 $^\circ C)$ is that, although chlorate satisfactorily depolarises (and thus accelerates) the reaction at the metal surface, it does not rapidly oxidise ferrous iron arising from the dissolution of the steel. The bath, therefore, accumulates ferrous ions and this has been held responsible for variability in bath performance². Chlorate-accelerated solutions may also contain nitrate: this serves partly to increase the reactivity of the chlorate³ and is also a valuable constituent in formulations to maintain the cation/anion balance. The increase in reactivity due to nitrate is not sufficient, however, to ensure rapid oxidation of dissolved ferrous iron.

It was considered that the chlorate or chlorate-nitrate accelerator could be best used as the "primary" oxidant, *ie* depolariser of the surface coating reaction, and that the build-up of ferrous iron could be controlled by dosing a carefully regulated amount of a rapid-acting oxidant—the "secondary" oxidant—in an amount just sufficient to react with the ferrous iron more or less immediately. Then, if the correct balance could be achieved, the secondary oxidant would not attain, nor need to attain, a significant concentration in the solution, so that it would not interfere with the action of the primary oxidant.

The argument underlying the design of such a process will be developed in the next section, starting with a consideration of the stoichiometry of the fundamental processes expected to occur. It will be assumed that conditions of steady composition have been reached either naturally or by the use of a suitable bath make-up formulation.

Chemistry of the zinc phosphating process

It is assumed that tertiary zinc phosphate tetrahydrate, *ie* hopeite, is the sole constituent of the coating and the sequence of processes which commences with anodic dissolution of iron from a steel substrate is considered. It is convenient to consider a four-electron process, *ie* dissolution of 2 gram-atoms of Fe.

The anodic process is:

$$2 \text{ Fe} - 4 e \rightarrow 2 \text{ Fe}^{2+} \dots (a)$$

Spray-applied zinc phosphating processes are fairly strongly cathodically depolarised in order that they should act at moderate temperature (below 60° C) and within short contact times (0.5 - 3 min). Hydrogen evolution can then be ignored in writing the depolarised cathodic reaction as:

$$2 O + 2 H_2O + 4 e \rightarrow 4 OH^- \dots (b)$$

Here O represents active oxygen supplied by the accelerator. The OH^- liberated can now participate in several processes. One is reaction with free phosphoric acid:

$$H_3PO_4 + OH^- \rightarrow H_2PO_4^- + H_2O \dots (c)$$

an essentially wasteful reaction, as far as coating formation is concerned. The overall reaction of coating formation, involving primary phosphate $(H_2PO_4^-)$ and zinc ions, can be written

$$3 \operatorname{Zn}^{2+} + 2 \operatorname{H}_2\operatorname{PO}_4^- + 4 \operatorname{OH}^- \rightarrow \operatorname{Zn}_3(\operatorname{PO}_4)_2 \cdot 4 \operatorname{H}_2\operatorname{O} \cdot \cdot (d)$$

The "chemical efficiency", x, of the coating process might be defined, on this basis, as a 100 per cent efficient process, one g-mol of hopeite will result from the dissolution of 2 g-atoms of Fe, *ie*

$$x = \frac{2 \times 56 W}{458 F} = 0.245 W/F \dots \dots (1)$$

where W and F are coating weight and iron loss in consistent units such as gm^{-2} .

A third possible destination of OH⁻ arises in the further reaction of the liberated ferrous ions. They undergo oxidation

$$2 \text{ Fe}^{2+} + 0 + 2 \text{ H}^+ \rightarrow 2 \text{ Fe}^{3+} + \text{H}_2\text{O} \dots$$
 (e)

followed by precipitation as ferric phosphate which is highly insoluble at the pH of standard spray phosphating baths, viz 2.6 to 3.0.

$$2 \operatorname{Fe}^{3+} + 2 \operatorname{H}_2 \operatorname{PO}_4^- + 4 \operatorname{OH}^- \rightarrow 2 \operatorname{FePO}_4 + 4 \operatorname{H}_2 \operatorname{O}_{\cdot} (f)$$

As (e) is necessarily followed by (f), they can be combined as

$$2 \operatorname{Fe}^{2+} + O + 2 \operatorname{H}_2 \operatorname{PO}_4^- + 2 \operatorname{OH}^- \rightarrow 2 \operatorname{FePO}_4 + 3 \operatorname{H}_2 O \dots (g)$$

The overall process (g), which affects all Fe^{2+} not remaining in solution, thus requires half as much oxidant as the primary process (b) or one-third of the total required to sustain the reaction sequence. If the oxidant functions are split, the required dosage of secondary oxidant will thus be about one-third of that required in a conventional process in which a single agent accomplishes both functions.

Process (g) also requires 2 g-mol of OH- to run to completion. This can be supplied either from process (b), in which case processes (c) and (d) will suffer from lack of OH-, or by an external supply of alkali. An indication of which of these possibilities predominates in practice is afforded by the value of x. Unable to find much guidance from the literature except for an indication that x can vary widely⁴, and considering that such an important point could not be left to theoretical speculation, x was measured not only in the laboratory, but also by extensive computer-aided monitoring of commercial plant operations. The conclusion was that, whilst a wide range of values could be produced under freak laboratory conditions, this parameter attained a steady value under continuous non-aberrant plant conditions. This observation makes it possible to proceed with the design of the process because a constant value of x implies a constant relationship between the ferrous iron dissolved and the coating formed on the substrate; hence it is feasible to feed the secondary oxidant at a rate proportional to that of the zinc phosphate concentrate.

To proceed from this picture of the underlying processes (a) to (g) to estimating the composition of the bath under steady conditions and the formulations of concentrate, toner and secondary oxidant, the most helpful consideration is that of conservation in the spray system. Let the constituents, whether of bath, concentrate, toner, coating or sludge be labelled numerically with values *i* and the various streams entering and leaving the system (concentrate and toner feed, work throughput, sludge produced, *etc*) be labelled *j*. Then, if f_j is the (algebraic) flow rate of stream *j* and $c_{i,j}$ the concentration of species *i* therein, conservation of every species that remains unchanged requires that in the steady state

Equation (2) can be used to generate a family of simultaneous equations, one for each unchanged species plus further equations describing changes (like that from chlorate to chloride) whose relationship to the coating process can be derived from the stoichiometry of the processes (a) to (g). An example of one of these equations is afforded by the zinc balance of the system. Zinc is present in the concentrate feed, the coating produced, the liquid loss stream (carry-over) and the sludge produced. If for zinc i = 1 and for the streams mentioned j = 1, 2, 3 and 4 respectively, equation (2) becomes

$$f_1c_{1,1} + f_2c_{1,2} + f_3c_{1,3} + f_4c_{1,4} = 0$$
(3)

If for the bath j = 0, since the carry-over loss is, in fact, a loss of bath liquid

$$c_{i,3} = c_{i,0} \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots (4)$$

for each *i*. This provides data for the ultimate solution of the equation for the composition of the bath.

By setting up the complete family of equations derived from (2) together with the relations covering electroneutrality of bath, concentrate and toner, and remembering (4), it is possible to arrive at the required estimate of steady bath composition as well as formulations of the chemicals to be supplied. In addition, estimates of chemical consumption and of sludge produced can be made. A crucial quantity is the liquid loss rate f_3 . The best method of finding this is probably by mass balance calculations. The authors reported previously¹ that its value differs widely from plant to plant; it can, of course, also be affected by the shape of work passing through the plant.

Extensive laboratory utilisation trials confirmed the general validity of the argument proposed and enabled refinement in respect of some of the numerical values required in the simultaneous equations. This work confirmed the theoretical expectation that such a process, properly designed, is amenable to automatic replenishment by sensing only the conductivity of the bath provided precautions are taken to deal with temperature fluctuations and probe fouling in the sensing system. The control system thus consists of a special conductivity sensing arrangement actuating feed pumps supplying zinc phosphate concentrate and a highly specific toner which accomplishes both the required degree of neutralisation and ensures the virtual absence of ferrous iron from the bath. Routine manual checks are required no more often than once per 8-hour shift and consist of a simple titration for free and total acidity.

Conclusion

Automatic composition control of a zinc phosphating process involving the sensing of only a single parameter, electrolytic conductivity, is possible if a suitable choice of accelerator system is made. Line experience has shown that the system described gives a coating weight normally within ± 10 per cent of a mean value, although the mean value itself can, by suitable choice of formulations, be set at a point within the range 1-5 to 3-0 gm⁻²

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The determination of the formation times of films cast from polymer dispersions

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Summary

An attempt has been made to determine, on films prepared from polymeric dispersions, the time needed after casting for the films to be fully formed i.e. to mature to their final properties.

Methods, such as measuring the tensile strength, the elution of water soluble matter, several methods of measuring the glass transi-

Keywords

Raw materials for coatings binders (resins, etc.)

emulsion resin

Properties, characteristics and conditions primarily associated with materials in general

glass transition point

tion temperature and measurement of the proton relaxation time by nuclear magnetic resonance (NMR) were used.

It was found that the measurements of water absorption and water soluble material elution give the same estimates of the film forming time, whilst the tensile strength reaches stability sooner; other methods gave no useful indication of the time for film formation.

Processes and methods primarily associated with analysis, measurement or testing

> absorption water absorption nuclear magnetic resonance spectroscopy

manufacturing or synthesis leaching

La détermination des temps de formation des films de dispersions polymères

Résumé

On a tenté de déterminer, dans le cas des films en provenance des dispersions polymères, le temps qui se passe entre la déposition et la formation totale du film, c'est-à-dire le moment ou les propriétés ultimes ont été atteintes.

On a utilisé des méthodes telles que la détermination de résistance à la traction, l'élution de matière soluble à l'eau, les divers méthodes de mesure de la température de transition vitreuse

la mesure au moyen de la résonance nucléaire magnétique du temps de relaxation de proton.

On a trouvé que les mesures de l'absorption d'eau et de la teneur en matière soluble à l'eau donnent de pareilles indications du temps de formation de film, tandis que la résistance à la traction devient constante en avance de ce temps, et les autres méthodes ne donnent pas d'indication utile à l'égard du temps de formation de film.

Bestimmung der Bildungsdauer von aus Polymerdispersionen gegossenen Filmen

Zusammenfassung

Es wurde versucht an aus polymeren Dispersionen bestehenden Filmen zu bestimmen, in welcher Zeit sie nach Aufgiessen völlig gebildet sind, d.h. sie bis zu ihren entgültigen Eigenschaften gereift sind.

Methoden, wie das Messen der Zugfestigkeit, die Elution wasserlösicher Bestandteile, verschidene Messmethoden der Glasübergangstemperatur, und Messung der Protonenrelaxation-

Introduction

Refs. 1-12

The formation times (or maturing) of films cast from polymeric dispersions, in contrast to the formation of films prepared from polymeric solutions, is a complex process which takes place in three stages and is influenced by the actions of several physical forces. In the first stage, water starts to evaporate and the concentration of water soluble materials, as well as of polymeric particles, increases. In the second stage, which is the most important, the remainder of the water evaporates and the polymeric particles coalesce under the influence of surface tension or capillary forces and because of self adhesion. All three of these influences can be complementary^{1-6,12}. At this stage the surfactants leave the surface of the polymeric particles. In the third stage the szeit durch kernmagnetische Resonanz (KMR) wurden benutzt.

Es wurde festgestellt, dass die Messungen von Wasserabsorption und der Elution wasserlöslicher Stoffe zu den gleichen Bestimmungen der Filmbildungsdauer führen, wohingegen die der Zugfestigkeit ihren Endpunkt früher erreicht; andere Methoden ergaben keine nützliche Information hinsichtlich der Filmbildungsdauer.

interdiffusion of the ends of polymer chains starts, and the polymeric film reaches its final state.

The term "film forming time" as used in this paper has been chosen arbitrarily to indicate the time required after casting for the films to achieve the final values of film properties.

The film forming process is influenced by many factors. It is important to know the time required for the process to be concluded. The process can be followed by several methods⁷⁻¹¹. In the present case an attempt was made to use methods for the measurement of properties which are usually employed for the characterisation of polymers, such as tensile strength, water absorption, elution of water soluble materials, the glass transition temperature and the proton relaxation time. It was supposed that the polymeric film was formed when no further change in the properties measured was observed.

Experimental

Preparation of emulsions and films

Experimental work was carried out on a copolymer emulsion of butyl acrylate/acrylonitrile/acrylic acid in the weight proportion 64/34/2. The emulsion was prepared by the delayed addition technique, using potassium persulfate as the initiator; an anionic surfactant was used. The particle size of the emulsion was 0.25μ .

The films were prepared by pouring the neutralised emulsion on to framed glass plates. After 24 hours, the films were removed from the glass plates and dried at 25° C in a freehanging position. The thickness of the dried films was 300-350 μ .

Measurements

The proton relaxation time was measured at 4, 10, 30, 45, 63 and 90 days after the films were cast. The tensile strength,

water absorption, elution of water soluble materials and the glass transition temperature (T_g) were measured also after ageing for 120 and 150 days.

The tensile strength was measured with a Franck dynamometer; the water absorption and elution of water soluble materials were measured by differential weighing of the sample before and after immersion in water; the T_g was measured with a Perkin-Elmer differential scanning calorimeter (DSC) and the proton relaxation time (T_2) with a Bruker spectrometer. In addition, the T_g was measured by the method of differential thermal analysis (DTA), by torsional vibration and by dilatometry on samples after 4 days' and 1 year's ageing.

Results

Tensile strength

Fig. 1 shows the tensile strength versus the time of ageing. A change from 60 kp/cm² to 180 kp/cm² is seen after a short period of ageing. After approximately 10 days almost the ultimate value of the tensile strength of the film is reached.



Water absorption and elution of water soluble materials

Fig. 2 shows the amount of water absorbed versus the immersion time for films after different ageing periods. The films aged for 4 and 10 days absorb water very quickly and reach maxima at 24 hours of immersion. Due to elution of surfactants, the water absorption after 24 hours immersion decreases and becomes constant. Films after ageing times of longer than 10 days absorb water without showing maxima. After 7 days of immersion the absorption of water is still increasing.

Fig. 3 shows the percentage of water absorption versus film ageing time for two different immersion times. It can be seen that film formation is a long process and that the influence of ageing time on absorption of water disappears only after 90 days.

Fig. 4 shows the percentage of elution of water soluble materials from the films versus ageing time. The elution is large for films aged for less than 30 days and very small for films with more than 90-100 days ageing.

Glass transition temperature

In Table 1 the values of T_q for films after 4 to 150 days' ageing, as measured with DSC, are shown. The differences

in T_g values are small and probably in the order of the experimental error. Comparing the values for films aged for 4 and 150 days a tendency of increasing T_g with time can be seen.

film ageing time (days)	T_g (°C)
4	9.0
10	9.5
21	11.0
30	10.5
45	11.0
60	13.0
91	11.0
120	12.0
150	11.0

By measuring the T_g by other methods on samples after 4 days' and 1 year's ageing, it was established that the methods of DTA and torsional vibration gave increases in T_g of 6°C for the films with the longer ageing time. By using dilatometry, differences of 0.3° C only were found for the two samples. These unexpectedly small differences are probably



due to changes in the films caused by the high vacuum used with this method.

Proton relaxation time

Measurements are shown in Table 2. T_{2a} , T_{2b} , and T_{2c} correspond to relaxation times for protons of non-equivalent places; however, not for protons of water. Measurements of T_2 values for water protons have not produced any signal, indicating that the concentration of water protons was too low for the instruments to detect even in cases of shorter forming times. This is in agreement with the gravimetric determination of weight loss of film samples versus ageing time. The measurements of water loss indicate that between the third and 13th day after the film has been cast the water loss amounts to only 1 per cent of film weight. After this times the weight of samples became constant.

Table 2

Relaxation time and relative concentration of protons of different T₂

tratio	concentr	Relative of	T_{2c}	T_{2b}	T_{2a}	Films ageing
/ T ₂	$T_{2b} /$	T2a /	(usec)	(µsec)	(µsec)	time (days)
/ _	30 /	70 /		29	13	4
1 -	32 /	68 /		45	20	10
1 4	33 /	67 /	80	22	14	20
1 1	23 /	75 /	130	25	12	30
/ 1	22 /	77 /	160	22	10	45
<i>i</i> 1	19 /	80 /	175	30	12	60
/	14 /	85 /	180	25	10	90

Changes of concentration for the three different non-water protons with values T_{2a} , T_{2b} and T_{2c} suggest some structural changes of the film. The measurements, however, are not conclusive.

Conclusions

The experiments on polymeric films cast from dispersions give indications of the relative usefulness of various methods for determination of film forming time.

It appears that properties measured by two simple methods, water absorption and water soluble materials elution measurements, indicate saturation after the same ageing period.

Tensile strength value, however, becomes constant after approximately 10 days, when apparently the process of film formation has not been completed.

Measurements of T_g and proton relaxation time gave little information on time changes in the film.

In this paper, the results of measurements for a butyl acrylate/acrylonitrile/acrylic acid copolymer system only are presented. Measurements on polymeric system of butylacrylate and ethylacrylate with acrylic acid and styrene indicate the same behaviour, but the time needed for film formation is shorter.

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Anomalous swelling in reactive polymers*

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Summary

The swelling behaviour of normally cured epoxy resin films for the swelling in strongly polar solvents does not obey the second Fick's Law. The "anomaly" of gravimetric weight change curves can be explained by comparing length change and thickness change curves: in the initial stage swelling only results in a one dimensional thickness change and compressive stresses are built up in the surface layers, because swelling in the area of the film is prevented by the unattacked hard core of the sheet, until the forces involved in

Keywords

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

Miscellaneous terms strain

Le gonflement anormal de polymères réactifs

Résumé

Le gonflement des films de résines époxides durcies normalement et immergées en solvants fortement polaires, n'obéit pas à la deuxième loi de Fick. On peut expliquer l'anomalie, dans le cas des courbes de changement de longueur auprès de celles de changement d'épaisseur. Au début le gonflement ne provoque qu'un changement d'épaisseur mono-dimensional et les forces compressives s'accroîtrant dans les parties superficielles, parceque tout gonflement y est empéché par le noyau dur et inaltéré de la couche,

Anormale Quellung in Reaktionsharten

Zusammenfassung

Das Quellverhalten normal vernetzter Epoxidharzfilme folgt bei der Quellung in stark polaren Quellmedien nicht dem 2. Fick schen Gesetz. Durch Vergleich der Längen- und Dickenänderungskurven kann die Quellanomalie bei Gewichtsänderungskurven in folgender Weise erklärt werden: Im Anfangsstadium bewirkt die Quellung nur eine eindimensionale Dickenänderung, und es werden Druckspannungen in Oberflächenschichten aufgebaut, weil eine Flächenquellung des Films durch den noch ungequollenen harten Filmkern

Introduction

Refs. 1-8

The present work originated from the investigation of the behaviour of reactive coatings under the influence of organic media. It has been found from a great number of swelling-tests^{1,2} that the \sqrt{t} -relation of diffusion:

$$\frac{c}{c_s} = \frac{4}{\sqrt{\pi}} \cdot \frac{\sqrt{D \cdot t}}{s}$$

c = average concentration of swelling-medium at time t

 $c_{s} = \text{concentration of saturation}$

D = diffusion coefficient

the swelling process reach the limit of the tensile strength of the inner core. At that moment the inner core is disrupted and expansion of the area starts. In plasticised or undercured films with a glass transition temperature in the region of room temperature or below, practically no compressive stresses can be built up. That is why these films behave relatively "normally", *ie.* conforming to the \sqrt{t} relation of diffusion.

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

glass transition point

dried or cured films

solvent sensitivity stress cracking

jusqu'à ce que les forces impliquées dans le processus de gonflement atteignent à la limite de la résistance à la traction du noyau. A ce moment le nayau est rompu et l'expansion de la superficie commence. Dans le cas des films plastifiés ou incomplétement durcis ayant la température de transition vitreuse dans la région de la température ambiente ou même au-dessous, pratiquement aucune force compressive ne peut s'accroître. C'est la raison pour laquelle ces films se comportent assez normalement, c'est-à-dire conformément au rapport √/ de diffusion.

solange verhindert wird, bis die Druckspannungen die Grenze der Zerreissfestigkeit des harten Filmkerns überschreiten. Erst zu diesem Zeitpunkt reisst der innere Kern, und die Flächenvergrosserung setzt spontan ein. Bei weichgemachten oder untervernetzten Filmen nit einer Glastemperatur im Bereich der Raumtemperatur oder darunter können sich praktisch keine Druckspannungen aufbauen. Daher verhalten sich diese Filme relativ "normal", d.h. dem $\sqrt{I-Geset2}$ der Diffusion entsprechend.

[approximate solution of the second Fick's Law for samples in the form of plates or foils of definite thickness, s] is realised only for the weight change during the initial stage of swelling in *non-polar n*-paraffins. The diffusion coefficients were in the region of 10^{-6} to 10^{-7} mm² min⁻¹ according to the mole volume of the swelling medium (Fig. 1).

In comparison with this *normal* behaviour, an *anomalous* swelling was observed with *strongly polar* media, *e.g.* ketones, esters, primary alcohols (Figs. 2 and 3): it was not possible to measure the weight change from the very beginning of the swelling test. Some kind of "induction period" ranging from a few minutes to nearly one hour or more was observed and it is believed that during this period a normal diffusion takes place with a very small diffusion coefficient, possibly in the

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Fig. 1. Swelling of polyurethane resin films (350 μm) as a function of the chain length of *n*-paraffins

same range as with the diffusion of *n*-paraffins mentioned above, but probably confused by solution reactions, on account of which the net sum of weight change is not measurable. After this period and during the course of the swelling reaction as a consequence of structural changes, diffusion coefficients, formally calculated from the swelling curve using the equation mentioned above, change abruptly before the maximum concentration c_s is reached. The maximum values of D were found to be 3 or 4 powers to ten higher than those observed with non-polar media.

An "anomalous" behaviour has been characterised by Crank and Park³ as "non-Fickian-behaviour". It has also been reported for a number of hard or glassy polymers, provided that the swelling tests were carried out below the glass-transition temperature, T_g , eg. for cellulose nitrate⁴ and acetate⁵, for polystyrene^{6,7}, even for rubber vulcanisates⁸. An explanation was given for this phenomenon as due to the formation of "internal stresses" produced in the course of swelling. The present investigation is a contribution to the discussion of the reasons for these stresses in the case of epoxy resin films.

Experimental

Free films 500μ m thick were prepared from commercially available pigmented epoxy resin coating materials (Table 1). All swelling tests were carried out at a temperature of 23° C. The weight change was measured gravimetrically in the usual way. The change in length of the films was observed micro-



Fig. 3. Swelling of epoxy resin film B (500 μ m) in ketones and esters as a function of the constitution of the media

scopically by inserting the samples in narrow glass cells to avoid erroneous values due to curvature of the samples during the course of swelling. Change in thickness was recorded as a function of swelling time by means of the thickness measuring device of a thermo-mechanical analysing equipment (TMA).

 Table 1

 Characteristics of epoxy resins examined in the tests

Epoxy resin	vehicle	curing agent	epoxy resin/ curing agent ratio ¹	pigment/ vehicle ratio ²
A	liquid resin + reactive thinner	modified polyamine	1:0.57	0.87:1
В	liquid resin	aliphat. amines + polyamino- amides	1:0.53	1.23:1
С	liquid resins + special tar	aromatic poly- amines	1:0.43	0.83:1
D	liquid resins	modified polyamine	1:0.4	0.14:1

¹referring to pure resins and curing agents ²referring to cured resin films

Results

Ref. 2

Referring to Fig. 4, showing the swelling of a normally crosslinked epoxy resin in two different ketones, it is naturally obvious that weight change and thickness change start almost simultaneously. The maximum in the weight change curves is due to the solution of soluble portions of the polymer (portion of the vehicle not fixed by cross linking) by the aggressive polar media². This is also demonstrated by the second weak maximum in the thickness change curves. Prior to this second maximum followed by a minimum, *ie. shrinkage* of thickness.



Fig. 4. Weight and thickness change of normally cured epoxy resin B (500 μ m)

The length change curve (Figs. 5 and 6) makes it obvious that length change is retarded at first and starts rather rapidly after the first thickness swelling peak has ceased.

Before discussing the behaviour of the coating as a function of stresses caused by swelling, it is necessary to consider the glass transition of the polymers. Normally crosslinked epoxy resin films are characterised by a glass transition value above room temperature (Table 2). If the resins are cured with an excess of curing agent the excess curing agent acts



Fig. 5. Weight, thickness and length change of normally cured epoxy resin B (500 μ m) in acetone



Fig. 6. Weight, thickness and length change of normally cured epoxy resin A (500 $\mu m)$ in acetone

as a plasticiser and the glass transition region is reduced almost to the level of room temperature (Table 2). In a swelling test, the plasticising efficiency of a surplus of curing agent in the polymer provokes a strong decrease in the interval between thickness change and length change (Fig. 7), and the thickness change maxima shown in the curves of Figs. 4-6 are degenerated (Fig. 7) or only weakly shown (Fig. 8).

 Table 2

 Glass transition temperature T_g (°C) of epoxy resin under investigation

investigution								
Sample	resin/curing agent equivalent ²	detern DTA ¹	nined by TMA ¹					
	equivalent	DIM						
epoxy resin	1:1	43	42					
A	1:1.5	20	26					
	1:0.5		$2(6, 8)^3$					
epoxy resin B	1:1	_	37					
epoxy resin	1:1		40					
C	1:1.5		28					
	1:0.5		$4(8, 23)^3$					
epoxy resin D	1:1		59					

¹heating rate DTA: 10 K min⁻¹, TMA: 5 K min⁻¹

 $^{2}1:1 = normally cured$

1:0.5 = cured with a deficit of 50% of curing agent 1:1.5 = cured with a surplus of 50% of curing agent ³curing time 5, 14 and 21 days respectively



Fig. 7. Weight, thickness and length change of epoxy resin A (500 μ m) cured and plasticised with a surplus of 50 % curing agent, in acetone



Fig. 8. Weight, thickness and length change with epoxy resin C (500 μ m) cured and plasticised with a surplus of 50% curing agent, in acetone

If the film has remained soft by undercuring, the thickness change curve can show nearly a "normal" behaviour (Fig. 9). In this case, the T_g -value was 8°C. According to the author's



Fig. 9. Thickness change of epoxy resin films A (500 μ m) cured with different ratios of curing agent, in acetone

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experience, an aftercuring reaction is observed in such undercured films which still contain a number of free reactive epoxy groups. Depending on the specific reactivity of the respective resin-hardener-combination and the steric conditions, this reaction can start more or less abruptly several days or even several weeks after the application of the mixed resin and it is manifested by a sudden rise in the mechanical hardness and the glass transition temperature² (Table 2). After the aftercuring reaction has started, the thickness change curve shows again the well marked peak shown by the normally cured resins (Fig. 10).



Fig. 10. Thickness change of epoxy resin film A (500 μm), normally cured and undercured, in acetone

Discussion

Refs. 2, 9, 10

Comparing the behaviour of normally cured hard epoxy resin films with those of plasticised or undercured soft films, the swelling anomalies especially demonstrated by the first well marked peak of the thickness change curves of *normally* cured films can be explained as follows:

In the course of swelling a solvent concentration gradient is formed in the film: In the case of normally cured epoxy resins, the inner core of the film remains almost unswollen in the glassy state ($T_g > 30^{\circ}$ C), whereas the outer layers are completely swollen and soft*. It has been shown by comparison of thickness and length change curves that the swelling of normally cured films in the initial stage results only in a one dimensional volume change in a direction perpendicular to the surface. As a consequence, in the originally isotropic polymer, compressive stresses are caused in the surface layers which are sometimes manifested in structural irregularities on the surface of the film (Fig. 11a to f). Even in case of complete softening of the extreme surface layers and consequent stress relaxation in these regions, there will always remain a transition region in deeper layers where compressive stresses are being maintained. These compressive stresses of intermediate layers result in tensile stresses exerted on the inner core of the film, increasing with the progress of swelling until reaching the limit of tensile strength when the core is disrupted and cracks and scores are formed. At that moment tensile stress and compressive stress are relaxed instantaneously, the whole

^{*}The T_g -value of such completely swollen zones has been determined to $\leq -25^{\circ}$ C.



(g)

Fig. 11. Surface of epoxy resin film D (400 μ m) at different stages of swelling in acetone

(h)

(a) unswollen	(e) swollen for 4.25 hours
(b) swollen for 2 hours	(f) swollen for 4.5 hours
(c) swollen for 3 hours	(g) swollen for 5.5 hours
(d) swollen for 4 hours	(h) swollen for 16 hours

film expands spontaneously, and assuming the volume to be constant temporarily, the thickness must be reduced as indicated by the peak demonstrated in the thickness change curve.

The fracture of the inner core of normally cured films can be demonstrated by swelling tests with unpigmented epoxy resin films: it was proved that the beginning of length change was connected with the appearance of partly isolated cracks approximately $150-200\mu$ below the surface of the film (Fig. 12*a*) quickly increasing in density to form a network of cracks at two levels (Fig. 12*b*). Such "swelling cracks" in the inner core have already been reported by Hermans⁹ for the swelling of cellulose fibres in water and by Wapler¹⁰ for the swelling of plexiglas in methylmethacrylate.

In contrast to this behaviour of normally cured epoxy resin films, it is nearly impossible for the softer inner core of plasticised or of undercured films to build up such stresses. Stresses caused in the swollen surface layers also result at once in a measurable expansion of the inner layer, which accounts for the small interval between thickness change and length change in the course of swelling.



Fig. 12. Swelling cracks in an unpigmented epoxy resin film produced in the course of acetone swelling on the left: swollen for 1 hour on the right: swollen for 1.3 hours

In the early stages of swelling the sum of tensile stress and of compressive stress results in a compression of the surface layers. The effect of such a compression was examined by fixing the epoxy resin film in the form of a coating on to a rigid substrate and so making area change during swelling impossible without loss of adhesion. In this way diffusion coefficients were reduced to about one-tenth of those values measured with free films². That means that the diffusion coefficients of samples, whose surface layers are compressed as a result of the swelling reaction to the initial stages, must be lower than those of a sample free from stresses. After relaxation of stresses an increase of diffusion coefficients should, therefore, be the normal case, which is superimposed upon, in the present case, an intensified diffusion as a consequence of the capillarity of the cracks and scores formed after the breaking of molecular links of the network.

As soon as the film has been completely swollen, the stresses will have resulted in the formation of inner deficiencies, and the film should behave completely differently on repetition of the swelling test after de-swelling, because the effect of, for example, swelling cracks in crosslinked polymers, should be irreversible. Indeed, the second thickness change curve of an epoxy resin film pretreated by swelling and de-swelling (Fig. 13) no longer shows a peak resulting from the one dimensional thickness swelling; the curve is fairly continuous, *ie.* conforming to the \sqrt{t} -relation of diffusion. Because of the irreversible changes in structure of the inner core of the film, internal stresses can no longer be formed, and swelling results in a *three*-dimensional volume change.



Fig. 13. Thickness change of epoxy resin film A (500 μ m) as a function of pretreatment

Conclusion

It has been shown by a comparison of length change and the thickness change curves recorded as a function of swelling time of normally cured epoxy resin films in strongly polar solvents that the "anomaly" in the gravimetric weight change curves can be explained as follows: in the initial stage, swelling results only in a (one dimensional) thickness change. in the course of which compressive stresses are built up in the surface or in the transition layers respectively. Swelling in the area of the film is prevented by the unattacked hard centre of the sheet until the forces involved in the swelling process reach the limit of the tensile strength of the inner core. At that moment the inner core is disrupted and expansion of the area takes place spontaneously. Practically no compressive stresses can be built up in plasticised or undercured films whose glass transition is in the region of room temperature or below. That is why these films behave relatively "normally", ie. conforming to the \sqrt{t} -relation of diffusion. The same is true for a normally cured epoxy resin film, the core of which has been irreversibly changed by preswelling.

It is believed that the results are of some importance from a practical point of view : anomalous swelling behaviour is equivalent to instability of a film formed from a given reactive resin towards the action of the swelling solvent present.

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Recent aspects on the preparation and evaluation of some polyesteramides for surface coatings. Part II: Preparation and evaluation of amide containing resins

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Summary

Various hydroxy-ethyl fatty acid amides were prepared and used as the dihydric alcohol in a resin formed by condensation with dibasic acid anhydride. Two main types of resins were prepared;

Keywords

Raw materials binders (resins, etc.)

polyesteramide

Processes and methods primarily associated with

drying or curing of coatings

drying air drying namely polyesteramide and alkyd/polyesteramide. These resins were evaluated as vehicles and the variations in film performance in relation to the amide linkages were studied.

Properties, characteristics and conditions primarily associated with

dried or cured films water resistance solvent resistance acid resistance

alkali resistance

Aspects récents de la préparation et de l'évaluation de certaines polyesteramides en revêtements de surface. 2ème. Partie: La préparation et l'évaluation des résines amides

Résumé

Diverses amides des dérivés hydroxy-éthyle des acides gras ont été préparés et utilisés en tant que l'alcool dihydrique à partir duquel on obtient une résine par condensation avec l'anhydride d'un acide dibasique. On a prépare deux types principaux de résines, à savoir; polyesteramide et alkyde/polyesteramide. On a évalué la variation des caractéristiques du film en fonction des liaisons amides.

Neuere Betrachtungen über die Herstellung von Präparaten sowie die Bestimmung einiger Polyesteramide für Anstrichmittel. Teil 11. Herstellung von Präparaten von und Bewertung von Amid enthaltenden Harzen.

Zusammenfassung

Es wurden eine Anzahl von Hydroxy-Äthylfettsäureamiden präpariert und als zweiwertige Alkohole bei der Bildung von Harz durch Kondensation mit zweibasischem Säureanhydrid benutzt. Es wurden zwei Haupttyps von Harzen hergestellt, und

Introduction

Refs. 1-8, 10

Alkyd resins show satisfactory film performance and mildew resistance. This is attributed to the presence of the easyhydrolysable ester linkages present. However, the inclusion of imide or imino linkages in the alkyd backbone would be expected to lead to the formation of resins of improved film durability over the conventional alkyds.

Modified alkyds of this type have not previously been studied extensively and the literature is scanty. Varnishes prepared from vegetable oils and containing imide linkages have been reported. Their course of preparation involves the use of amino alcohols¹⁻⁴. The term "polyesteramide" was adopted for such resins. zwar Polyesteramide. Diese Harze wurden als Bindemittel bewertet, und die Unterschiede in ihrem Verhalten als Filme hinsichtlich ihrer Amidbindungen beurteilt.

Gast *et al*⁵ in 1966 reported the preparation of polyesteramide resins by refluxing linseed diethanolamide in xylene with dibasic acids or anhydrides. Azelaic, maleic, fumaric, phthalic, terephthalic, itaconic, brassylic, and dimer acids were the dibasic acids studied in this work.

The diethanolamide derivatives of various drying and nondrying vegetable oils were discussed in Part 1 of this paper. Their method of formation depends on the reaction of the fatty oil with an equivalent amount of diethanolamine at 200° C and in presence of zinc oxide as a catalyst⁶.

The condensation of dihydroxy-diethyl linseed amide (HELA) with a dibasic acid (*eg o*-phthalic acid) can be explained by the following equation, in which R represents linseed fatty chain:



However, there is a possibility of acyl group migration from the nitrogen atom to the oxygen atom. Such a side reaction could yield a polyesteramide polymer containing both the ester and amide linkages of the following structure:



There is also a tendency for acidolysis of the polyesteramide polymer under the influence of the dibasic acid employed. Displacement of the fatty acyl radical during the acidolysis leads to the formation of a trifunctional molecule capable of forming cross-linked structures. Thus acidolysis may increase the probability of gelation.

Two years later, Gast *et al.* extended their work to include various low-acid value polymers⁷. Their study was mainly concerned with the formation of resins having various excess hydroxyl groups using the solvent technique. Linseed and soybean diethanolamides were the diols used in combination with 11 different dicarboxylic acids.

More recently in 1969, they were able to prepare new polymeric coating materials by base-catalysed reaction of the hydroxy-terminated polyesteramide of soybean or linseed oils with diisocyanates⁸.

Recently, attention has been directed towards improving the polyesteramides with diisocyanate in a manner similar to that used for alkyd modification⁸⁻¹⁰. This modification greatly improves the drying time, alkali, acid, solvent and impact resistances. A pigmented paint based on this type of modified-polyesteramide showed no sign of checking, cracking, erosion, discolouration or loss of gloss one year after application.

These types of resins are viscous and the structure of the dibasic acid or anhydride used affects to a large extent their viscosities and drying characteristics.

The presence of driers or a drier system is essential for the air-drying of this type of resin. The polyesteramides of linseed fatty acids show significantly better alkali and solvent resistances when compared with normal alkyds.

Increasing the amount of HELA, and thus increasing the excess hydroxyl content, lowers the molecular weight of the

finished product. It also has a marked effect on the final properties. As a matter of fact, as the excess hydroxyl increases, the drying characteristics improve whilst the hardness decreases. As with alkyds, low acid value products are made possible by the presence of excess hydroxyl groups.

This paper deals mainly with the formation of various resins starting with diethanolamide derivatives of different fatty acids. Formulations were calculated on the basis of the average functionality method using the concept of the alkyd constant.

Experimental

Materials

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

Methods and technique

Preparation of polyesteramide resins (General) (Resins I-IV)

The hydroxy-ethyl fatty amide (HEFA) was placed in a 4necked 250 ml flask equipped with an efficient stirrer, an inert gas inlet, a thermometer and a syphoning device for withdrawing samples without interrupting the reaction. Then phthalic anhydride (PA) was charged and the mixture was heated to 220°C until an acid value below 10 mg KOH/gm was reached.

The weight ratio of HEFA/PA depends on the desired excess hydroxyl content. Table 1 illustrates the charge weights corresponding to 0, 10, 20 and 30 per cent excess hydroxyl resins.

 Table 1

 Charge weights of polyesteramide resins

Resin	Excess hydroxyl	HELA	PA
No.	%	gm	gm
1	0	73.6	29.6
II	10	80.8	29.6
111	20	88.2	29.6
IV	30	95.6	29.6

Preparation of alkyd/polyesteramide resins (Resins VI-IX)

The alkyd/polyesteramide resin was prepared in a manner similar to that described in the previous preparation using the same apparatus. In this case, the oil (1 mole) was aminolysed with DEA (2 moles) at 220°C and in presence of ZnO (0.0012 mole) as a catalyst.

The temperature was maintained constant until the product showed complete solubility in twice its volume of absolute methanol. The reaction mixture was then allowed to cool to about 100°C and PA (2 moles) was added and the temperature was raised to 220°C and kept constant until the desired resin specification was reached.

Table 2 illustrates the charge weights for various alkyd/ polyesteramide formulations.

 Table 2

 Charge weights of alkyd/polyesteramide resins

 Everes bydroxyd

Resin No.	Excess hydroxyl %	LO gm	PA gm	DEA gm
VI	0	96.0	27.6	19.2
VII	10	86.1	29.6	21.2
VIII	20	70.0	29.6	23.2
IX	30	46.9	29.6	25.0

Preparation of 30 per cent excess hydroxyl alkyd (Resin V)

In a similar manner, as described previously, 30 per cent excess hydroxyl alkyd resin (based on linseed oil) was made. The process involved heating a mixture of linseed oil (106.4 gm), glycerol (38.0 gm) and PbO (0.1 gm) as a catalyst at 220°C till monoglyceride formation was obtained. The reaction mixture was allowed to cool to 100° C, followed by an addition of PA (88.8 gm). The temperature was raised to 220°C and maintained constant until a low acid value product was obtained.

It should be mentioned that a petroleum fraction $(147-203^{\circ}C)$ was added to the resin prepared in order to obtain 50 per cent solids.

Methods of analysis and evaluation

Refs. 11-26

The turbidity method¹¹, acid value¹², iodine value¹³, colour¹⁴, viscosity¹⁵, non-volatile content¹⁶, drying time¹⁷, flexibility¹⁸, and adhesion¹⁹, resistance towards water²⁰, acid²¹, alkali²², solvents²³, scratch²⁴, gloss²⁵, and hardness²⁶ determinations were carried out according to standard methods described in the literature references given.

Results

Ref. 5

Following the successful preparation of the ethanolamide derivatives of various fatty acids in high percentage yield, attention was directed towards their utilisation in resin formation. Such resins, of course, contain both ester and amide linkages and therefore would be expected to show outstanding film performances over the conventional alkyds. Previous publications were concerned only with the preparation of polyesteramide by the solvent process⁵. It was thought of interest to extend the work to include polyesteramide preparation by a fusion method.

Various polyesteramide resins were formulated covering a wide range of resin compositions based on the hydroxy ethyl fatty acid amide (HEFA). These resins correspond to 0, 10, 20 and 30 per cent excess hydroxyl contents.

The time-temperature programme for the resin preparation is shown in Figure 1. The resin constants were computed from charge weights and are given in Table 3. An alkyd formulation having 30 per cent excess hydroxyl groups (short oil length) was introduced amongst the formulations for the sake of comparison.

The course of esterification of the various resins is shown graphically in Figure 2. It is clearly seen from these figures that the rate of esterification depends upon the per cent of hydroxyl content. In fact, the 30 per cent excess resins containing linseed oil fatty acids can be reacted to a low acid value without danger of gelation. These observations are in agreement with the theoretical expectations.





Table 3 Polvesteramide resin constants

Resin No.	Excess —OH	W	Ε	eo	e _A	e _B	F	mo
I	0% HELA PA	36.76 14.80	184 74	0.199 0.200	0.20	0.199		0.10 0.10
п	10% HELA PA	40.40 14.80	184 74	0.220 0.200	0.20	0.220		0.11 0.10
ш	20% HELA PA	44.12 14.80	184 74	0.239 0.200	0.20	0.239		0.20 0.10
IV	30% HELA PA	47.79 14.80	184 74	0.259 0.200	0.20	0.259		0.13 0.10

Where: W is the weight taken; E is the equivalent weight; e_o is the total equivalent at start of the reaction; e_A is the number of acid equivalents; e_B is the number of hydroxyl equivalents; F is the functionality; and m_o is the total moles at start of the reaction.



Fig. 2. The course of preparation of polyesteramide resin using raw linseed oil and alkyd resin

The characteristics of the prepared resins were determined and are given in Table 4.
Table 4 Polyesteramide resin characteristics

Resin No.	Time of formation, hr	AV mg KOH/gm	IV cg I ₂ /gm		Viscosity, Gardner
I	3.50	12.3	121.0	>18	А
п	4.25	9.7	115.0	>18	A
ш	3.00	8.0	101.0	>18	Α
IV	3.25	6.0	80.0	>18	A
V(alkyd)	2.00	16.4	83.0	18	F

It can be seen from the above data that all the resins are characterised by dark colours. This is attributed to the presence of the nitrogenous base during the reaction, which increases the yellowing tendency. Moreover, as the per cent hydroxyl content increases, the possibility of obtaining low acid value products increases.

Another set of experiments was conducted using hydroxyethyl boiled linseed oil fatty acid amide instead of the corresponding derivatives of raw linseed oils (Figure 3). Similar results were obtained, except that low iodine-value resins were produced. This is expected on account of the polymerisation of the oil.



Fig. 3. The course of preparation of polyesteramide using boiled linseed oil and alkyd resin

After the preparation of the various polyesteramides, attention was directed towards the preparation of a similar set of resins from the oil, secondary hydroxyamine and the anhydride in one step and in the same reaction assembly. The following equation illustrates their mode of formation:

CH2OCOR	C ₂ H ₄ OH	CH2OCOR	C ₂ H ₄ OH
CHOCOR + 2 HN	< →	CHOH + 2 RCON	
CH2OCOR	C ₂ H ₄ OH	CH₂OH	C ₂ H ₄ OH
Triglyceride	DEA	Monoglyceride	HEFA

Both monoglyceride and HEFA contain two hydroxyl groups capable of condensing with polybasic acids. The condensation of these dihydric derivatives leads to polyester in case of the monoglyceride (alkyd) and to polyesteramide in case of HEFA, hence the combined term "alkyd/polyesteramide" was adopted for such resins.

The most important finding from these data is the elimination of the tedious and time consuming step encountered during the extraction of the hydroxyamide derivatives from the liberated glycerol⁶. A case which is not valid for polyesteramide resins.

Similarly, various alkyd/polyesteramides (having various excess —OH groups) were formulated according to the principles applied to polyesteramides in order to obtain resins having similar constants. The course of esterification of these resins is graphically shown in Figure 4. It can be clearly seen from the figure that increasing the percent hydroxyl content leads to a decrease in the esterification time and, consequently, the 30 per cent excess hydroxyl resin esterifies in a shorter time than others.



Fig. 4. The course of preparation of alkyd/polyesteramide resin and alkyd resin

The characteristics of the various alkyd/polyesteramides were determined and are given in Table 5.

Table 5 Alkyd/polyesteramide resin characteristics

Resin No.	Time of formation, hr	AV mg KOH/gm	$IV \\ cg l_2/gm$		Viscosity, Gardner
VI	3.50	16.0	122.1	>18	A
VII	4.00	13.8	116.0	>18	A
VIII	3.50	11.6	103.6	>18	A
IX	1.25	10.0	85.5	>18	A

Similar observations (to that of polyesteramide) were also noticed.

Resin and film properties

After the resins have been reacted, they were thinned with the suitable solvent to 50 per cent solid content and filtered. The following driers were added to the resin solution:

Pb as naphthenate 0.05 per cent Co as naphthenate 0.05 per cent

Mn as naphthenate 0.02 per cent.

The drying characteristics of the varnishes were measured and are given in Table 6.

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Drying characteristics

Resin No.	Drying performances						
	Air drying (hr)		Stoving at 110°C for 3 hr	Stoving at 150°C for 2 hr		Stoving at 165°C for 2 hr	
1	within	18	Tacky	Hard	dry	Hard	dry
П	,,	,,	,,	,,	"	"	,,
111	.,,	.,		.,	,,	"	.,
IV	.,	,,	"	.,	.,	,,	••
V(alkyd)	,,	24	Hard dry	,,	,,	,,	,,
VI		12	S.T.	V.S	Т.	,,	,,
VII	.,	,,	,,	,,		"	,,
VIII	,,	,,	,,	,,		,,	.,
IX		,,	.,	,,		.,	.,

Where: S.T.	= slight	thickness and	V.S.T.	 very slight 	thickness.
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The film evaluation of the resins was conducted according to well-known methods. The resistance data against water, acid, alkali, and solvent are given in Table 7.

Table 7

Water, acid, alkali, and solvent resistance of the dried films

	Resistance against					
Resin No.	Water	Alkali	Acid	Solvent		
I	Ex	Ex	Ex	Ex		
н	Ex	Ex	Ex	Ex		
Ш	Ex	Ex	Ex	Ex		
IV	Ex	Ex	Ex	Ex		
V(alkyd)	F	Ex	Ex	Ex		
VI	F	Ex	Ex	Ex		
VII	F	Ex	Ex	Ex		
VIII	Ex	Ex	Ex	Ex		
IX	Ex	Ex	Ex	Ex		

Where: Ex = excellent (almost no film defects); and F = fair (considerable film defects).

The general conclusions drawn from the data given in Tables 6 and 7 are:

- (a) Decreasing the acid-amide linkage content in the resin leads to an improvement in the air drying characteristics.
- (b) The water, acid, and alkali resistances are greatly improved as the per cent excess hydroxyl content increases. On the other hand, the drying characteristics are considerably improved by decreasing the per cent of acidamide linkages.

Comparative study between the 30 per cent excess hydroxyl polyesteramide, alkyd/polyesteramide and alkyd resin

Table 8 shows a comparison of the three different types of resins, each having 30 per cent excess hydroxyl content with the same fatty acids.

Table 8 Comparison of the various resins

Property	Polyesteramide resin	Alkyd/ polyesteramide resin	Alkyd resin
Hard dry time (air)	18 hr.	12 hr.	24 hr.
Hard dry time (stov.) "for one hour"	150°C	182°C	105°C
Acid resistance	Ex	Ex	Ex
Alkali resistance	Ex	Ex	Ex
Water resistance	Ex	Ex	F
Solvent resistance	Ex	Ex	Ex
Colour, Gardner	> 18	>18	18
Viscosity, Gardner	А	A	F
Acid value, mg KOH/gm	7.6	10.0	16.4
Iodine value, cg I ₂ /gm	80	85.5	83
Time of cooking, hr.	3	1.25	2.0
Adhesion, Grid Test	Class 1	Class 1	Class 1
Gloss	100	100	100
Flexibility	Flex.	Flex.	Flex.

The data given in Table 8 is the average value of three measurements. The following generalisations were noticed:

- (1) When air dried, the alkyd/polyesteramides reach the hard dry state in the shortest time compared with polyesteramide and alkyd resins. On the other hand, the alkyd resin requires the lowest baking temperature when compared with other resins at constant schedule.
- (2) All three types of resin possess the same resistance to water, alkali, acid and solvent. The water resistance of alkyds was considered in this generalisation.
- (3) The amides containing resins have darker colour than the conventional alkyds. Their colours would be expected to improve when using the solvent technique for their preparations.

Extensive study on film properties of the resins

In order to examine the extent to which the film breaks down and to show how far the films will withstand various chemicals and physical effects, baked and air-dried films were subjected to water, acid and alkali resistance tests until complete film failure occurred. The data obtained are shown in Table 9.

The gloss measurements and the scratch resistance of the three types of resins are given in Table 10.

In conclusion the above data were extended by rating the three 30 per cent excess hydroxyl resins for maximum film performance. "A" means the most outstanding resin with optimum performance property. "B" is an intermediate, and "C" the least. The data are given in Table 11.

Table 9 Film performances of various resins

Resistant against	Time	Polyesteramide		Alkyd/ polyesteramide		Alkyd	
		Air	Stov.	Air	Stov.	Air	Stov.
Water	1 day	Ex	Ex	Ex	Ex	Р	Р
	2 day	F	Ex	F	F	F	Р
	3 day	F	Ex	F	F	F	F
Alkali	6 hr.	Р	Ex	Ex	Ex	Ex	Ex
	12 hr.	F	Ex	F	Ex	Ex	Ex
	2 day	-	F		Ex	Ex	Ex
	3 day				Ex	Ex	Ex
	4 day				Р	Ex	Ex
	5 day			1000	F	F	Ex
Acid	1 day	Ex	Ex	Р	Ex	Ex	Ex
	2 day	Р	Ex	Р	Ex	Ex	Ex
	3 day	F	Ex	F	Ex	Ex	Ex
	4 day	F	Ex	F	Ex	Ex	Ex
	5 day	F	Ex	F	Ex	Ex	Ex
	8 day	F	Ex	F	F	Ex	Ex

Where: Ex = excellent; un-affected films,

P = partially attacked films, and F = complete film failure.

 Table 10
 Gloss and scratch resistance measurements

Resin	Film	Gloss	Scratch, Kg	Flexibility
Polyesteramide	air	100	<1	Satisfactory
	stoved	100	< 1	,,
Alkyd/	air	100	>1	,,
polyesteramide	stoved	100	>1	,,
Alkyd	air	100	>1	,,
	stoved	100	>1	,,

Table 11

Comparison of	of the film	properties o	f the three	prepared resins	
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Item	Polyesteramide resin		Alkyd/ polyesteramide resin		Alkyd resin		
	air	stov.	air	stov.	air	stov.	
Drying time	В	В	A	С	С	Α	
Water resistance	A	A	A	A	В	В	
Alkali resistance	В	С	С	В	Α	A	
Acid resistance	В	Α	С	В	A	A	
Solvent resistance	A	Α	A	Α	Α	Α	
Scratch resistance	В	в	A	Α	Α	Α	
Hardness	В	Α	A	Α	В	В	
Gloss	100	100	100	100	100	100	
Adhesion	А	Α	Α	Α	Α	Α	
Flexibility	Α	A	Α	Α	Α	Α	

It can be concluded from the above data, and taking the stoved films as a final stage of film curing, that:

- The three types of resins have the same degree of gloss, adhesion, and solvent resistances and flexibility.
- (2) The polyesteramide resins are superior with regard to the water resistance, whilst the alkyd resins are superior for acid and alkali resistance.
- (3) The alkyd/polyesteramides show the highest degree of hardness and scratch resistance.

[Received 9 March 1977

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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:

Antifouling paints based on organotin compounds. Leaching of organotin toxins from paint films by L. Chromy and K. Uhacz 2-Furfuraldehyde (furfural) formation from local by-products and its utilisation in the field of surface coatings by S. M. Saad, A. M. Naser, M. T. El-Zimaity and H. F. Abdel-Meged

Identification of shellac by R. Prasad and S. C. Sengupta

A new simple method of determining the oxirane oxygen content of vegetable oils by B. M. Badran

Subscriptions for 1978 and binding of 1977 volume

1978 Members' subscriptions

Members are reminded that 1978 Membership subscriptions to the Association are payable on 1 January 1978. Forms were despatched to all members in October or November, depending upon address.

The Commissioners of Inland Revenue have approved of the Association for the purpose of the 1970 Income and Corporation Taxes Act Section 192, so that a Member subject to United Kingdom income tax is entitled to a deduction from the amount of his emoluments assessable to income tax under Schedule E for the whole of the annual subscription to the Association, provided the subscription is defrayed out of the emoluments of his office or employment and that the interests covered by the objects of the Association are relevant to such office or employment.

1978 library subscriptions

The Journal subscription rate to non-Members, including libraries, for 1978 will be £25.00 (\$60) post free by surface

Correspondence ____

Recent developments in antifoulings

Sir,

I would not want to prolong unduly the correspondence on the subject of Dr Christie's paper "Recent advances in antifouling", but I have to declare an interest.

The work referred to in the quotation "Test plates attached to a ships underwater hull conclusively demonstrated the correlation between progressive surface smoothing and decreased drag as measured in a hydrodynamic flume", refers to work which I carried out in the I.R.H.M.C. laboratories in 1975–76, work which was in fact written up after my resignation, for the purpose of publication, and with assurances to that effect.

The object of the correspondence was to give Dr Christie the opportunity to acknowledge the source of his information.

Section Proceedings-

London

Conservation

The second evening technical meeting of the session was held at the Rubens Hotel, SWI on Thursday 20 October, 1977 when Mr H. Barker, Keeper of Conservation and Technical Services of the British Museum presented a paper entitled "Corrosion and Conservation" to over 40 members and guests.

Based on the work carried out by the Conservation Departments of the British Museum, Mr Barker illustrated the various techniques used to restore and preserve antiquities.

The particular method used by the restorer depended on the materials from which the object was made, the state of the article and the type of preservation that was to be carried out. mail, home and abroad. Inividual copies can be purchased for $\pounds 2.50$. Remittance should be sent with order to the Association's offices.

Binding of Journal

Members will be pleased to know that J. S. Wilson & Son, 14a Union Road, Cambridge CB2 1HE, will undertake the binding of back volumes of the Association's *Journal* sent in by individual Members at a cost of £6.00 (including postage and packing) per volume.

Members wishing to avail themselves of this facility should send the parts, securely wrapped, direct to J. S. Wilson & Son Ltd, enclosing a remittance of £6.00 (including postage and packing) per volume, and ensuring that notes bearing their names and addresses are enclosed with the parcels. It is particularly important that packets are sufficiently wrapped to negate the possibility of damage in the post.

He has chosen not to do so. In fact there is evidence that he did not read the letter. I did not criticise Dr Christie for attacking the exaggerated claims of the competition for some of their products. I found it "welcome and stimulating" and shared his scepticism.

Neither can he claim lack of space, since under "new biocides" he was able to review the use of T.M.T. and D.D.T. (1945!), and to suggest that I should have gone all the way to Eastbourne to learn about T.M.T. and D.D.T., is a joke, the full humour of which I cannot unfortunately share with your readers.

Yours faithfully, A. MILNE

Camrex (Holdings) Ltd, Research & Development Division, Camrex House, P.O. Box 34, Sunderland.

1 December, 1977

Metal objects corroded in different ways, the metal being converted into simple and complex salts in all instances. The choice available was to strip off the corrosion products back to the original metal, to halt corrosion and stabilise the surface, or convert the salts back to the original metal. Chemical methods were invariably used to do this. Alkaline Rochelle salt or alkaline glycerine was used to strip copper salts off bronzes; the reaction between silver oxide and basic cupric chloride (green spotting on bronzes) to silver chloride was used to halt green spotting corrosion; ion exchange resins were used to convert lead salts back to the metal on lead objects.

Very often removal of corrosion products left the remaining metal in a fragile state. Methods such as fixing in epoxy resins or reinforcing with glass fibre and methacrylate resins and subsequently colouring to match the original were used.

Another facet of the conserver's job was demonstrated with

Very often, simple techniques were used to preserve objects. Venetian glass was hygroscopic and the easiest way of preserving it was to store and display it at relative humidities of 35 per cent and less.

Waterlogged wood presented more problems however. Prolonged immersion led to replacement of the cell contents by water. Simple drying out caused shrinkage of the wood. Various methods were available, but the British Museum used polyethylene glycol waxes, slowly diffusing the wax into the wood, immersed in an aqueous solution of the wax, by raising the temperature of the bath from 0° C to 60° C over a six month period.

Polyethylene waxes were also used to preserve stonework and these were better than paraffin waxes previously used, since the waxy surface of the latter retained dirt and led to further corrosion. Polyethylene waxes were water soluble and cleaning was easier.

Successful conservation depended not only on the skill, dexterity and patience of the preserver, but also on the ability of experienced staff to look more than just superficially at the object and accurately determine its composition.

After a question time and discussion period a vote of thanks to the speaker was proposed by Mr A. J. Newbould, which was warmly endorsed by the audience.

A.J.N.

Manchester

Silicone protective coatings

The first meeting of the new season was held at the Woodcourt Hotel, Sale, on Monday 10 October. 50 members and guests attended to hear a paper by Mr J. G. Price of Dow Corning Ltd entitled "A review of silicone protective coatings".

Mr Price discussed the chemistry, properties and basic processes for the manufacture of silicones and reviewed their applications in paint.

Particular reference was made to recent developments in coil coating of prefinished buildings panels, high performance maintenance paints, heat resistant finishes, and release coatings for bakeware. Numerous slides were used to illustrate the points and an extremely lively discussion period followed; Mr G. Topham gave the vote of thanks.

Filtration

Friday 11 November 1977, Manchester Literary and Philosophical Society, George Street, was the date and venue for a lecture entitled "Theory versus practice in vacuum pressure and centrifugal filtration". The presenter was Dr A. Rushton of the University of Manchester Institute of Science and Technology.

Thirty-three members and their guests heard Dr Rushton describe fully filtration principles, flow rates, filter cloths etc.

in respect to Newtonian liquids. The lecturer ably developed his chosen subject to include practical applications, such as the variable chamber principle, rotary vacuum filters (UMIST speciality) and made reference to the work of Shirato (Japan) in respect to paint filtration. The resultant discussion period was concluded by a vote of thanks proposed by Mr. Hurst and acknowledged in the usual enthusiastic manner.

Epoxide resins

"Epoxide resins for anti-corrosive coatings" was the title of the lecture given to the Student Section by Mr A. G. McKay of Ciba Geigy Plastics and Additives Company. Date and venue was 17 November, Manchester Literary and Philosophical Society. 51 members and guests were present to hear the lecturer describe fully the chemistry of epoxide resins and associated curing agents.

Further subjects discussed included amine curing at room temperatures and the relationship between adhesion and corrosion inhibiting properties.

The lecture was illustrated with a series of slides and concluded by a vote of thanks from Mr S. Horridge which was enthusiastically received.

F.B.W.

Newcastle

Sensible methods of test

The first meeting of the 1977/78 session was held on Thursday 6 October when some 50 members and visitors assembled at St. Mary's College, Durham to receive a paper from Mr F. D. Timmins of British Rail entitled "Corrosion control and sensible methods of test—at last!" In his own unique manner, Mr Timmins pointed out where we had all made mistakes and demonstrated through slides, panels and examples the direction in which we ought to proceed. The speaker held the audience's attention without the aid of notes for over an hour and was provocative, informative and amusing. Following a lively question period the Chairman, Mr F. Hellens, proposed a vote of thanks to the speaker, which was enthusiastically received by the members.

This was the first meeting to be held at St. Mary's College and the lecture was followed by a buffet meal, which together with private bar facilities, allowed members and visitors to continue in conversation until 10.00 p.m.

T.H.

West Riding

Wood preservatives

A meeting of the West Riding Section was held on Tuesday 1 November 1977, at the Mansion Hotel, Leeds. The speaker was Dr Gavin Hall of the Timber Research and Development Association.

Wood is basically cellulosic with 28-30 per cent lignins and fair proportions of hemicelluloses and other constituents. Solvent extractable materials are those which give different types of timber their characteristic appearance, feel etc. The outer 10-12 years growth, sapwood, is the only living part of a tree. The inner part, heartwood, is "dead" and largely structural in function.

1978 (1) SECTION PROCEEDINGS

Natural durability is of considerable significance and different species vary in this respect. Moisture content is also critical and, in general, the moisture content of timber must be above about 22 per cent for decay to proceed. Insect damage can also occur eg. woodworm, deathwatch beetle, but in the UK this is less of a problem than fungal attack.

Preservatives are designed to render the timber poisonous to these organisms. Ideally they should have high durability and low mammalian toxicity. Those in common use can be put into three categories.

- Creosote and derivatives. Dwindling supply, but still have a traditional place.
- (2) "Synthetic creosotes". Mixtures of chemicals, such as pentachlorphenol, copper naphthenate, tributyl tin oxide dissolved in distillates or white spirit.
- (3) Water based, of which two types are commonly encountered in the UK.
 - (a) Active agent is copper chrome arsenate which complexes with the timber over a period of days and has a degree of fixation.
 - (b) A boron treatment applied in country of origin to "green" timber. This is not fixed chemically and can leach out under adverse conditions.

Most preservatives give only a shell of protection around the outside of the treated article. The method of application is critical and brush or spray is of very little value, except for the eradication of insect attack, since the depth of penetration is too low. It is more efficient to immerse in the treating solution for up to a maximum of 1 hour. Immersion is really the minimum acceptable treatment. Double vacuum processing is more effective, but requires specially designed equipment. The timber is loaded into the plant which is then evacuated, flooded with preservative, drained and the vacuum applied again before the timber is removed and dried. These normally

use solvent borne systems. Water based systems are used in processes which utilise pressure as well as vacuum and these are, in general, the most effective.

In relation to a paint finishing process, preservation treatment can be advantageous since it prevents growth of organisms under the paint film. Incompatibility problems are not particularly important. Copper naphthenate systems have been suspected of retarding drying and creosote will bleed through a paint film. Under certain conditions penthachlorphenol based products can create yellowing problems but this is not widespread and the cause has not been established.

Following some lively questions and answers a vote of thanks was proposed by Mr Keith Smith.

R.A.C.C.

Hull

De-foaming agents

The second ordinary meeting of the 1977-78 session was held at the 'George Hotel', Land of Green Ginger, Hull on Monday 7 November 1977. Mr T. W. Wilkinson, the Section Chairman, introduced Mr R. W. Harrison of Diamond-Shamrock Chemicals (UK) Ltd, who gave a lecture entitled "De-foaming agents : The theory and application with regard to the paint industry".

Using visual aids, Mr Harrison discussed the mechanism of foam formation and the action of de-foaming agents. The two principal methods for evaluating the efficiency of defoamer in emulsion paint systems were considered in detail.

Mr Harrison's short lecture was followed by a discussion on the subject. The meeting, which was attended by some 15 members and visitors, was brought to a close with a vote of thanks from Mr R. Brooks.

D.M.W.

Information Received

New plastics factory

After a detailed technical and economic analysis, orders for 95 per cent of the requirements for a new plastics factory being set up in Saudi Arabia have been placed with British manufacturers, including GKN Windsor Ltd, RHR Ltd, Leesona Plastics Machinery. Consulting engineers for the project are the Yarsley Polymer Engineering Centre, and the new 1,000 sq.m factory is scheduled to go into production in March 1978.

New polymerisation unit

Cole Polymers Ltd have ordered a complete polymerisation unit from Leonard Smith (Engineers) Ltd, which includes a 600 gallon nominal capacity stirred reactor, condenser, prefeed tank, heaters, coolers, and a complete temperature control loop.

UK agency

One of Europe's leading manufacturers of powder coatings for industrial finishing, Helic, a subsidiary of CdF Chimie, are seeking an agent for their products in the UK. The company was a pioneer in the development of thermosetting powders and their Tritorga range is widely used in 37 countries. The need for a UK agent has arisen because of the increase in the interest being shown in powder coatings by British industry.

Change of address

The commercial departments of Burrell Colours Ltd and its parent company, Burrell & Co. Ltd, are moving to a new modern office building, Burrell House, 44 Broadway, Stratford, London E15.

Kratos acquisition

Continuing their programme of planned international expansion, Kratos Ltd has acquired the Schoeffel Instrument Corporation of New Jersey, USA, which specialises in instruments for biochemical and analytical chemistry. The company also has plants near Hamburg, Germany.

New products

New vibrating screen separator

Triton Enginering Co. Ltd have available a new vibrating screen separator which is designed to remove a high degree of unwanted solids from crude chemical liquids used in industrial processes. This results in an easy to handle material which can be stacked or bagged for transportation and re-use, and leaves a comparatively clean liquid readily processed, disposed of, or re-cycled.

New coating thickness gauge

Surfatest have available the Elektrotest coating thickness gauge, a highly accurate instrument for measuring coating thickness on both ferrous and non-ferrous substrates. Measurements are taken with a single contact constant pressure probe, and thicknesses up to 10 mm on ferrous and 40 mm on nonferrous metals can be measured.

Photon counter for spectroscopy

The new Spex DPC2 photon counting system from Glen Creston Instruments Ltd has been designed for use in Ramen spectrometers, spectrofluorometers and other spectroscopic instruments where low light levels are being measured. The unit incorporates not only a photon counter but also a DC electrometer and a high voltage supply for a photomultiplier tube.

Pressure leaf filters

The new range of Magnum large-area pressure leaf filters is available from Amafilter (UK) Ltd. The compact automated filter is designed to occupy a small floor

INFORMATION RECEIVED JOCCA

area, and was originally built for brine filtration in chlorine alkali electrolysis. The filter also has applications wherever large filtration areas of between 200 and 400 sq. meters are required at normal working pressures of 4 bar.

Safety autospout for chemical products

W. Langley & Co. Ltd have available the new Autospout P179, a safe self-sealing pouring attachment for containers of petroleum products and chemical solutions. It is particularly useful insituations where inflammable or toxic liquids have to be handled frequently and prevents accidental spillage of hazardous substances.

New Atlas Fade-ometer

Westlairds Ltd, representatives for the Atlas Electric Devices Company, USA, have available the new Atlas C.135 Fade-ometer which is claimed to represent a major advance in light fastness testing of paints, inks and other colour critical products. The C.135 uses a xenon lamp as light source and special light filter systems to provide a constant spectral distribution closely approximating to that of natural daylight behind window glass. This enables products under test to receive special amounts of irradiance under accurately controlled conditions of temperature and humidity.

Notes and News-Manchester Section

Annual Dinner Dance

The Annual Dinner Dance and Ladies' Night was held on Friday 21 October 1977 at the Piccadilly Hotel, Manchester.

430 members and guests attended and the section was pleased to welcome as their chief guest Mr M. E. D. Jarrett, Chairman, Texales Division, Wallpaper Manufacturers, a member and former Chairman of the Section. Other guests were The Presi-



The new Atlas C.135 Fade-ometer

Literature courses etc.

New reference catalogue

Electrochemical Publications Ltd have available a new catalogue containing a com-

dent, Mr A. McLean and Mrs McLean and the Chairmen of Hull, Scottish and Irish sections with their respective partners. Also present was the Director & Secretary.

Those present, who had previously heard Mr Jarrett speak, were not surprised at his very entertaining discourse in proposing the toast to the Manchester Section; this was followed by Mr Mitchell's reply, with the formal events being completed by a presentation to Mrs Mitchell.

Varied dance routines, combined with other enioyable activities, continued until the bewitching hour of 2 a.m.

F.B.W.



Top Table guests at the Manchester Section Dinner (*left to right*): Mrs Jarrett, Mr J. E. Mitchell, Mrs McLean, Mr A. McLean (President), Mrs Mitchell, and Dr M. E. D. Jarrett

prehensive list of all reference books available in the English language in the general field of 'Paint'.

Magnesium alcoholates

Dynamit Nobel have published a new leaflet giving information on specifications, possible uses and safe handling of magnesium alcoholates, particularly magnesium methylate and magnesium ethylate, which are used in the polymerisation of unsaturated compounds, in condensation reactions, in transesterification reactions, for example in the preparation of esters of acrylic and methacrylic acids.

PPC year book

The 1978 edition of the 'Polymers Paint and Colour Year Book' has now been published and contains up-to-date information on suppliers, manufacturers and processors for the Surface Coating Industry.

Safety in laboratories

Ciba-Geigy Ltd have republished their booklet 'Safety in laboratories' which deals with many aspects of laboratory safety, applying rules and procedures which have been developed over many years in Ciba-Geigy's own laboratories. The booklet is widely used in many schools, colleges and industrial laboratories as a guide to safety.

London Section

Ladies' night



Photograph: D. E. Eddowes

Mr A. McLean (President) proposing the toast to the London Section, being watched by Mr D. Bayliss

The London Section Ladies' Night was held at the Piccadilly Hotel, W1 on Friday 28 October 1977 when over 160 members and guests enjoyed the annual dinner and dance.

The principal guest of the evening, Mr A. McLean, President of the Association, proposed the toast to the Section; the response was given by Mr D. Bayliss, Chairman of London Section.

Amongst the guests present were the Chairman of the Thames Valley Section, Mr M. H. Prigmore and the Director & Secretary.



Allocation of space

The Exhibition Committee of the Oil & Colour Chemists' Association announces that the allocation of space for OCCA-30 (the thirtieth annual technical exhibition of raw materials, plant and equipment for the paint, varnish, printing ink, colour, adhe-sives and allied industries) has now taken place.

It is particularly pleasing to note the return of many exhibitors from previous years, as well as new exhibitors, thus showing the strength of the support for this annual focal point for the surface coating industries. At present, there will be participation by organisations in the following 15 countries.

Belgium, Canada, France, East Germany, West Germany, Holland, Hungary, Italy, Japan, Poland, Spain, Sweden, Switzerland, the UK and the USA.

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

Dates and times

The 1978 Exhibition will take place at Alexandra Palace, London, N.22 on the following dates and times:

Tuesday	18 April	$\sim \infty$	09.30 - 17.30 hrs.
Wednesday	19 April	$\sim r$	09.30 - 17.30 hrs.
Thursday	20 April	a a	09.30 - 17.30 hrs.
Friday	21 April		09.30 - 16.00 hrs.

First list of Exhibitors

The following organisations from the UK and 14 overseas countries will be represented; product classifications are given in parentheses.

- A. B. Metall -Sweden (manufacturing equipment) ACNA—Italy (resins, pigments, chemical
- intermediates)
- A. M. F. International Ltd) (manufacturing equipment)
- Allied Colloids Ltd (resins, additives, chemicals)
- Allied Polymer Group (manufacturing equipment)
- Amoco Chemicals Switzerland (resins, chemical intermediates) Bachofen, Willy—Switzerland (manufact-uring equipment)
- Baird & Tatlock Ltd (laboratory apparatus)

OCCA-30 Exhibition

18-21 April 1978 at Alexandra Palace, London

★Belgium ★ Canada ★ France ★ East Germanv★ \star West Germany \star Holland \star Hungary \star Italy \star *Japan*Poland*Spain*Sweden*Switzerland* * UK * USA *

The continuous dialogue between suppliers and manufacturers

- Barford Chemicals (resins)
- British Industrial Plastics Ltd (resins) Campbell, Rex & Co. Ltd (resins, pigments, additives)
- Canadian Worsdall Chemical Co. Ltd Canada (resins, pigments, additives)
- Capricorn Chemicals (pigments)
- Chemetron Corporation-USA (resins, pig-
- ments, additives)
- Chemolimpex Hungary (resins, additives) Chemviron Belgium (additives, extenders)
- Ciech-Import & Export of Chemicals Ltd Poland (pigments, chemical intermediates) CIRP
- -France (manufacturing equipment Cole Chemicals Ltd (resins, extenders, addi-
- lives) Contraves Industrial Products Ltd (laboratory apparatus)
- Croxton & Garry Ltd (additives)
- Curwen, M.D. Ltd (additives, driers)
- D.H. Industries Ltd (manufacturing equipment)
- Daltrade Ltd (pigments, chemical intermediates)
- Diffusion Systems Ltd (laboratory apparatus)
- Dow Chemical Co.-Switzerland (resins, additives)
- Draiswerke GmbH-Germany (manufacturing equipment
- Dresser Europe S.A. (manufacturing equipment)
- Durham Raw Materials Ltd (additives) Eiger Engineering (manufacturing equipment)
- Elcometer Ltd (laboratory apparatus)
- Emser Werke A.G. Switzerland (resins) Erlich, G.J. Ltd (manufacturing equipment)
- Erichsen GmbH Germany (laboratory ap-
- paratus) Ferchim Engineering S.A. - Belgium (manu-
- facturing equipment) Ferranti (laboratory equipment)
- Fischer Instrumentation (GB) Ltd (labora-
- tory apparatus) Fluoridienne S.A.
- Belgium (pigments) Gallenkamp, A. & Co. Ltd (laboratory apparatus)
- Ge-Halin Germany (manufacturing equipment)
- Glen Creston Ltd (manufacturing equipment) Godrich, John (manufacturing equipment,
- laboratory apparatus) Goodyear, Compagnie Francaise-France (resins)
- Grilon (UK) Ltd (resins)
- Haeffner, H. & Co. Ltd (pigments)
- Hercules Powder Co. Ltd (resins, pigments, additives)
- Hooker Chemicals & Plastics Corp.-USA (resins, additives)
- Hopkin & Williams Ltd (laboratory appara-(us)
- Industrial Dispersions Ltd (pigments, additives)

Instrumental Colour Systems (laboratory apparatus)

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- Jenag Equipment Ltd (manufacturing equipment)
- Kirklees Chemical Co. (resins)
- K & K Greeff Industrial Chemicals Ltd (resins, pigments, additives)
- KWR Chemicals Ltd (manufacturing equipment)
- Langer, Georg M. (pigments)
- Laporte Industries Ltd (pigments, chemical intermediates)
- Loebl, Joyce Ltd (laboratory apparatus)
- Lucas Mejer (additives)
- Ludwig Schwerdtel-Germany (manufacturing equipment) Macbeth Color & Photometry Division of
- Kollmorgen (UK) Ltd (laboratory appara-(us)
- Marchant Brothers Ltd (manufacturing equipment)
- Mastermix Engineering Co. Ltd (manufacturing equipment)
- Meca-Inox-France (manufacturing equipment)
- Melbourne Chemicals (additives)
- Micro Products Co. (pigments)
- Microscal Ltd (laboratory apparatus)
- Millroom Accessories & Chemicals Ltd (manufacturing equipment) Molenti (UK) Ltd (manufacturing equip-
- ment)
- Montedison Group-Italy (resins, pigments, chemical intermediates)
- Nagema, Veb Kombinat-East Germany (manufacturing equipment)
- Necof B.V.-Holland (resins)
- Netzsch-Germany (manufacturing equipment)
- Neville Cindu Chemie-Holland (resins)
- Nuodex Ltd (additives) Oliver & Batlle Spain (manufacturing
- equipment) Paintmakers Association of Great Britain
- Ltd (technical education)
- Pamasol Willi Mader-Switzerland (manufacturing equipment)
- Pilamec Ltd (manufacturing equipment) Polymers, Paint & Colour Journal (tech-
- nical journal, services) Portcullis Press Ltd
- PQ International—USA (additives) Pye Unicam Ltd (laboratory apparatus)
- Q-Panel Co-USA (laboratory apparatus)
- Radiant Colour (pigments)
- Research Equipment (London) Ltd (labora-
- tory apparatus) R.K. Print-Coat (laboratory apparatus)
- Roban Engineering Ltd (manufacturing equipment)
- Sanyo Kokusaku Pulp Co. Ltd-Japan (additives, resins)
- Silberline Ltd (pigments) Sussmeyer, Ateliers-Belgium (manufacturing equipment)

Swada (London) Ltd (pigments)

Ten Horn Pigments (pigments)

- Tiszamenti Vegyimuvek, Szolnok—Hungary
- (pigments) Torrance & Sons Ltd (manufacturing equipment)
- Victor Blagden & Co. Ltd (resins, pigments,
- additives) Vollrath, Paul—Germany (manufacturing equipment)
- Vree, J. De-Holland (manufacturing equipment)
- Ward Blenkinsop & Co. Ltd (additives)
- Westerlins Maskinfabrik-Germany (manu-
- facturing equipment) Wheatland Journals Ltd (technical journal,
- services) Worsdall Chemical Co. Ltd (resins, additives chemical intermediates)

Services

OCCA Information Centre Interpreters Mark Allan Travel Agency Red Cross Society National Westminster Bank—mobile unit G.P.O. self-service unit

The Exhibition provides an excellent annual opportunity for the technical personnel in the supplying industries to meet their counterparts in the manufacturing industries and to discuss their common technical problems. The advantage both to exhibitors and visitors of meeting in an informal atmosphere needs hardly be stressed, since the cost to exhibitors sending representatives to all the countries from which the visitors are drawn might well be prohibitive, particularly to smaller companies.

Refreshments on stands

Exhibitors were allowed for the first time in 1977 to serve alcoholic refreshments on their stands and this innovation will be continued at OCCA-30. Many exhibitors expressed their appreciation of this as it allowed their personnel to remain on the stands with visitors for the whole period of the Exhibition.

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.

Official Guide

This unique publication contains descriptions of all companies and their exhibits at the Exhibition and is issued to each Member of the Association at home and abroad together with season admission tickets. As in 1977, several Sections will be organising coach parties to visit the Exhibition and any Members interested should contact their local Section Hon. Secretary. (Full Section Committee lists for 1978 are published in this issue of the Journal.) It is also hoped that several parties will be organised from overseas to visit the Exhibition. As in pre-vious years the Official Guide and season admission tickets will be available several weeks in advance of the Exhibition (prepayment only) from the Association's offices but they will also be available for purchase at the entrance to the Exhibition Hall. A charge is made for both the Official Guide and the season admission tickets to the Exhibition. The policy was introduced several

years ago to deter casual visitors who otherwise collected large quantities of technical literature from exhibitors' stands; the policy has been welcomed by exhibitors and has in no way acted as a deterrent to bona fide visitors to the Exhibition.

Facilities at Alexandra Palace

The Association has full use of the facilities at Alexandra Palace during the period of the Exhibition which include restaurants, two bars, a cafeteria and an exhibitors' bar. Another facility which is of considerable benefit to those travelling to the Exhibition by car is the ample free car parking space within the Palace grounds.



Take the Piccadilly Line to Heathrow Airport. It's the only way to fly.

Heathrow Central Terminal Now Open

The extension of the Piccadilly Underground Line to Heathrow Central Terminal was opened by Her Majesty the Queen on 16 December.

The first stage of the project was completed with the opening of the Hatton Cross station on the outskirts of the airport in July 1975. Work on the Heathrow Central Terminal was begun in April 1971 and has cost about £27 million. This development makes Heathrow the world's first main airport to be directly linked into the underground system of a major city. All three main passenger terminals are connected to the station by moving walkways and underground passages. Trains will be running from the airport at four minute intervals during peak hours, and the Piccadilly Line has been equipped with new trains to cope with the increased passenger traffic. The new trains are similar in appearance to the latest "silver" tube trains, but have a different size and layout which give more space for passengers with luggage. This makes the new enlarged Piccadilly Line one of the most reliable and comfortable forms of transport in London.

The advantage to overseas visitors travelling to OCCA-30 is enormous, as it now greatly simplifies the journey, both to hotels in central London where they may be staying, and to the Exhibition. Visitors arriving at Heathrow Airport will now be able to board a Piccadilly Line train at the airport building itself which will take them directly to Turnpike Lane Station, from where the Association will be running a free bus shuttle service to and from the Exhibition. The journey from central London to Turnpike Lane station takes approximately 18 minutes.

All OCCA Exhibitions have been held in London, which affords excellent travel and hotel facilities for visitors from both overseas and the United Kingdom. In pursuance of the theme of the Exhibition —The continuous dialogue—it is felt that Exhibitions will like to know that the Exhibition Committee has also reserved Alexandra Palace for April 1979 and will hold its annual Exhibitions there subsequently in April each year.

It will also be appreciated that for exhibitors and visitors staying in London, the capital city offers the finest variety of entertainments for the evenings after the Exhibition.

News of Exhibitors Stand 44

Pye Unicam Ltd

Colour Measuring System

A new versatile colour measuring system will be shown by Pye Unicam Ltd on Stand 44. Based on the SP8-100 UV/visible spectrophometer, it provides accurate and reproducible results without the high cost of a fully computerised controlled instrument. The spectrophotometer is also capable of making normal laboratory measurements. Several systems are available differing in their data handling, with the most sophisticated being an online programmable calculator version, for which an adaptable software package has been developed. This system enables materials to be studied in transmission, or by diffuse or total reflectance with a choice of standard illuminants, observation angles and reporting units.



The new colour measuring system based on the SP8-100 UV/visible spectrophotometer

A large integrating spheroid located within the cell compartment of the SP8-I00conforms fully with CIE requirements for colour measurement. Its geometry ensures that there is no direct optical path between any patch of specularly reflected light and the photomultiplier. This feature, which is believed to be unique, contributes considerably to averaging, and hence accuracy.

Stand 15 Pilamec Ltd

New Paint Mill design

With ten years of experience in the application of vibrational energy to the sizereduction of solids, *Pilamee Ltd* have adapted their medium-sized mill to a paint function. The two mill chambers contain a freely-moving helix and zircon beads, the circular motion of the chambers inducing a helical motion of the beads. No glands are required in the mill and the energy input to a product can be varied by an external adjustment. A pump and tanks complete the production unit. fully used in the most diverse fields of application for both bath and continuous mixing, on Stand 2 of the OCCA 30 Exhibition. The unique kneading action of the *Simpson Mullers* means that solids can be mixed with the very smallest amounts of solid or liquid admixtures to the highest degree of homogeneity.

The working principle, which combines mechanical action and pressure, is quite simple with two multers providing an intensive kneading action against the base of the chamber, causing both a sliding or rolling effect, which turns the particles, and a sideways motion. All sizes of *Simpson Multers* have relatively small space requirements and are easy to incorporate into existing installations. *Simpson Multers* are particularly recommended in industries where complete homogeneity of components is of crucial importance.



The new Megacol No. 1 paint mill from Pilamec Ltd

The overall dimensions of the Megacol No.1, as this new unit is called, are approximately 71 cm \times 68 cm \times 36 cm and the mill itself is under 120 kilos. Where a changeover of colour is required, the chambers can be replaced conveniently, so reducing cleaning-down time. Added versatility is offered by the fact that the mill can be switched to dry powder grinding and other functions by simply replacing the chambers with those containing steel or alumina balls.

Pilamec Ltd will be demonstrating this new mill on Stand 15.

Stand 2 Glen Creston Ltd

Simpson Mullers

Glen Creston Ltd will be showing the Simpson Mullers, which have been success-

Stand 19 Mastermix Engineering Co. Ltd

The Masternix Engineering Co. Ltd will be drawing attention to the new 1 litre Masternil on Stand 19 at OCCA-30. The new mill is a laboratory size horizontal bead mill complementary to the range of production Masternills.

Also on show will be the new Mark 5 Underdriven P.M.D. in sizes from 1350 to 6750 litres (working capacity). The slow speed drive under the vessel driving a completely new type of agitator not only increases the mixing/dispersing efficiency, but also makes room on top of the vessel for much improved loading facilities, particularly where any form of automation is involved. In addition to these new models, the complete range of *Mastermill* mills will be on show, including the many refinements which are constantly being introduced.

Stand 1 Ward Blenkinsop & Co. Ltd

Formation of new subsidiary

As part of their current expansion programme, Ward Blenkinsop & Co. Ltd, who will be exhibiting on Stand 1 at OCCA-30, have announced the formation of a new subsidiary, Ward Blenkinsop Trading Ltd. The new Company will market the small range of chemicals which have for some years been marketed by the parent company as an extension of their wide range of pharmaceutical chemicals, intermediates and industrial additives, manufactured at their Widnes, Merseyside plant. Ward Blenkinsop Trading Ltd will operate from the group head office at Wembley.

Additives for industry

Ward Blenkinsop & Co. Ltd have manufactured pharmaceutical and fine chemicals on Merseyside for thirty-eight years, and during this period have diversified into the industrial additives field to such an extent that they now account for approximately 20 per cent of the Company's total sales.

The nature of their business involves the successful operation of a wide variety of organic reaction routes and techniques, with applications in the adhesives, cosmetics, electroplating, fibres, surface coatings, reprographic, printing ink, resin, plastics and starch industries.

The additives range includes Aduvex ultraviolet light absorbers, catalysts, fungicides, antioxidants, sunscreen agents and cationic reagents.

Last year at OCCA-29 the Quantacure range of UV initiators was introduced, and the Company is now regarded as one of the leaders in this specialised field. A wide range of initiators and combination products will be shown, and intensive technology in the Company's research and development laboratories ensures that the product range is continuously updated and improved.

News of Members

Mr J. D. Easton, BSc, PhD, FRIC, FTSC, an Ordinary Member now attached to the General Overseas Section and formerly attached to the Ontario Section, has taken up the appointment as Technical Director of The Harshaw Chemical Company, at their plant in Louisville, Kentucky, U.S.A.

Mr D. M. Wilson has taken up a senior appointment with Sericol Group Ltd, Broadstairs, Kent, performing long term development in the company's new laboratories. Mr Wilson, since April 1974, has been the Honorary Publications Secretary of the Hull Section.

Midland Section

Annual Dinner Dance

The Midland Section held their Annual Ladies' Night Dinner Dance at the Botanical Gardens, Edgbaston, Birmingham on the 16 September. Amongst the 200 members and and their guests who attended were the President of OCCA, Mr A. McLean and his wife, Mr and Mrs A. Moffatt, and Mr and Mrs B. J. Addenbrooke (President of the Birmingham Paint Varnish and Lacquer Club), Mr and Mrs H. Prigmore, Mr and Mrs J. E. Mitchell, Mr and Mrs J. D. W. Davidson, Mr E. G. Warman of Cape Section and Mr R. H. Hamblin, Director & Secretary of OCCA. Mr R. J. King, Chairman of Midland Section, gave an amusing toast to the ladies, with a reply by the President, Mr A. McLean, resplendent in kilt, in similar vein. An enjoyable evening ended at 1 a.m. with hot soup to help the revellers on their way."

R.K.C.

Jordan Award

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

The rules of the Award are:

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

Top Table guests at the Midland Section Dinner (from left): Mr J. A. Burns, Mrs Burns, Mr R. J. King, Mrs King, Mr A. McLean, Mrs McLean and Mr E. G. Warman

of 35 at the date of application.

2. The final date for submission of applications will on this occasion be 31 December 1978 and it is hoped to present the award at the Stratford-upon-Avon conference in the following June.

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

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

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

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

- BANNER, RICHARD JOHN, 44 Balcarres Street, Morningside, Edinburgh. (Scottish-Eastern Branch)
- BOND, JOHN RUSSELL, PhD, BSC, ARTS (Pigments & Printing Inks), ICI Organics Division, Blackley, Manchester. (Manchester)
- CANOSSA, GIULIO, Montedison SPA, Via Toce 7, 28029 Villadossola (NO), Italy. (General Overseas)
- COLLINS, GORDON MALCOLM, BSc, 22 Glenshee Avenue, Kilburn Park Estate, Neilston, Glasgow. (Scottish)
- COWLEY, ALAN, BSc, ICI Organics Division, Hexagon House, Blackley, Manchester M9 3DA. (Manchester)
- FERGUSON, DONALD ARCHIBALD, Van Leer Australia, PO Box 11, Chester Hill 2162, Australia. (General Overseas)
- HOLMES, PETER NEVILLE, BSc, 19 Thormanby Lawns, Howth, Co. Dublin, Ireland. (Irish)
- KIRKWOOD, THOMAS, 39 Northfield Road, Kilsyth, Scotland. (Scottish)
- O'HARA, KEVIN JOHN, LRIC, 8 Speldhurst Court, Queens Road, Maidstone, Kent. (London)
- PAUL, IVAN KEITH, BSc, Shell Chemicals NZ Ltd., PO Box 1085, Auckland, New Zealand. (Auckland) RICHARDSON, ROBERT KEITH, 4 Dell Farm Road, Ruislip, Middlesex.
- Richardson, Robert Keith, 4 Den Parint Road, Ruisinp, Middesex. (London)
- ROBBE, CASPARUS, Shell Internationale Chemie Mij BV, Postbus 162, The Hague, Holland. (General Overseas)

ROBINS, MALCOLM DOUGLAS WAKEFIELD, MSC, Buckley & Young Ltd, PO Box 5641, Auckland, New Zealand. (Auckland) SHEPHERD, DENNIS JOHN, BSC, E J Arnold & Son Ltd, 17/25 Napier

Place, Ward Park North, Cumbernauld, Glasgow. (*Scottish*) STODDART, IAN OLIPHANT CURRIE, Interbobbin (UK) Ltd, Middle-

ton Mill, Tillcoultry, Clackmannanshire. (Scottish) SUMMERS, GEOFFREY GORDON, BSc, 62 Woodside Court Road,

- Addiscombe, Croydon CR0 6RU. (London) Taylor, Philip James, BSc, ICI NZ Ltd, PO Box 900, Auckland,
- New Zealand. (Auckland) THUKRAL, PREM SAGAR, BSc, PhD, FRIC, FPRI, BP Research
- Centre, Chertsey Road, Sunbury-on-Thames, Middlesex. (London)

Associate Members

DUNN, JOHN, 16 Stanneylands Drive, Wilmslow, Cheshire.

(Manchester)

Registered Students

GRIGGS, PHILIP WILLIAM, 22 Hillfoot Road, Romford, Essex

- (London) HEWETT, ALAN DAVID, Berger Chemicals, Portland Road, Newcastle-upon-Tyne. (Newcastle)
- ROYSTON, IAN, I Sydenham Terrace, Sunderland, Tyne and Wear. (Newcastle)
- YOUNG, STEPHEN, Berger Chemicals, Portland Road, Newcastleupon-Tyne. (Newcastle)



- **OCCA CONFERENCE**
- STRATFORD HILTON HOTEL
- 20-23 JUNE 1979



Shakespeare's birthplace

Photograph by Herald Photographic Studio, Stratford-upon-Avon



The challenge to coatings in a changing world

It is envisaged that the papers to be presented will be concerned not only with the challenge of new materials and the substitution of traditional ones, new methods of construction and the problems of application. but also with the effects of new legislation (both national and international) and the sociological, climatic and leisure aspects of life in the last quarter of the twentieth century.

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

Forthcoming Association Conference and Section Symposia

The Association's next biennial Conference will be held at the Stratford Hilton Hotel, Stratford on Avon, Warwickshire from 20 to 23 June 1979. The title that has been chosen is "The Challenge to Coatings in a changing world". The attention of members is drawn to the call for papers notice on page 37 of this issue.

Midlands Section Symposium

The Midlands Section is organising a one

Forthcoming Events-

ern technology and legislation on paint manufacture" to be held at the Metropole Hotel National Exhibition Centre. Birmingham on Wednesday 8 March.

day symposium entitled "The effects of mod-

Full details of the Symposium may be obtained from Mr R. J. Chater (Hon. Publications Secretary, Midlands Section), Blundell-Permoglaze Ltd, Tyseley, Birmingham B11 2BD.

Thames Valley Symposium Call for papers

The Thames Valley Section will be holding a one day symposium on "Wood and its protective treatment" on Thursday 12 October at the Princes Risborough labora-tory of the Building Research Establishment. Papers for presentation at this symposium are now being invited and anyone interested in presenting a paper should contact the Section's Hon. Programmes Officer, Mr G. V. Hill, 60 Heath Road, Holtspur, Beaconsfield, Buckinghamshire.

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 following publication.

1978

January

Tuesday 3 January

West Riding Section: "Flexible liquid epoxy resin systems" by Mr A. G. McKay and Mr P. T. Brown of Ciba-Geigy Plastics Division at The Mansion Hotel, Roundhay Park, Leeds 8, commencing at 7.30 p.m.

Thursday 5 January

"The Central Newcastle Section: Newcastle Section: "The Central Electricity Generating Board require-ments for surface coatings" by Mr D. A. Bayless, Scientific Services Dept., CEGB, at St. Mary's College, University of Durham, Elvet Hill Road, Durbam.

Monday 9 January

Hull Section: "Prediction of performance: fact or fiction", by Mr D. M. Bishop, The Railway Technical Centre, Derby, at the George Hotel, Land of Green Ginger, Hull at 6.30 p.m.

Friday 13 January

Manchester Section: "Modern paint manufacturing techniques" by Mr B. Lucas, Joseph Mason Ltd, to be held at the Manchester Literary & Philosophical Society, Manchester, commencing at 6 400 pm 6.30 p.m.

Scottish Section: Ladies' Annual Evening Dance in Albany Hotel, -Dinner Glasgow.

Wednesday 18 January

Scottish Section-Eastern branch: "Printinks for paper-present and future developments" by Mr G. H. Hutchinson at the King Malcolm Hotel, Dunfermline at 7.15 p.m.

Ontario Section: "Anticorrosive func-tional pigments" by Dr P. Marr, Reed Ltd, at the Skyline Hotel, Toronto, at 6.00 p.m.

Thursday 19 January

London Section: "Finishing of exterior timber" by Dr E. R. Miller, at the Princess Alice, Romford Road, E7 commencing at 7.00 p.m.

Scottish Section: Speaker from Sigma Coatings Ltd.

Friday 20 January

Irish Section: Ladies' Evening. 'Interior designs" by Mrs A. Dalton, RIDipl, AIDP, of Andrian Interiors, at the Clarence Hotel, Dublin 2 at 8.00 p.m. Midlands Section: "Export/import of technology" by Mr A. G. North, Cray Valley Products Ltd, at the Birmingham Chamber of Industry and Commerce.

Thursday 26 January

Thames Valley Section: "Chemical colouring of metals" by Mr G. Pollock of Osro Ltd, at the Beaconsfield Crest Motel (White Hart), Aylesbury End, Beacons-field, Bucks at 6.30 for 7.00 p.m. Manchester Section: Student lecture.

Friday 27 January

Bristol Section: "Abatement of paint stoving effluent" by Mr N. A. R. Falla of The Paint Research Association, at the Royal Hotel, Bristol at 7.15 p.m.

February

Thursday 2 February

Newcastle Section: "Water, water every-where..." by Mr G. W. Rothwell, Building Research Establishment, at St. Mary's College, University of Durham, Elvet Hill Road, Durham.

Monday 6 February

Hull Section: "Exterior wood finishes" by Mr P. Whitely, Building Research Estab-lishment, Garston, at the George Hotel, Land of Green Ginger, Hull at 6.30 p.m.

Tuesday 7 February

West Riding Section: "Fundamentals of modern emulsion paint formulation" by Mr J. Clark, BTP Tioxide Ltd, at the Mansion Hotel, Roundhay Park, Leeds 8 at 7.30 p.m.

Thursday 9 February

Midlands Section-Trent Valley Branch: "Recent developments in decorative paints" by Mr J. Briddle, Cray Valley Products, at the Crest Hotel, Pastures Hill, Littleover, Derby at 7.00p.m.

Scottish Section: "Microscopic studies of gloss of surface coatings" by Dr 'S. G.

Lawrence, Ciba-Geigy (UK) Ltd, at the Bellahouston Hotel, Glasgow at 6.00 p.m.

Friday 10 February

Manchester Section: "Purchasing strategy in the chemical industry with reference to paint" by Mr D. F. Brocklehurst, Berger Group Supplies, at the Manchester Lit-erary & Philosophical Society, Manchester at 6.30 p.m.

Scottish Section-Eastern Branch: Burns Supper to be held at the Commodore Hotel, Marine Drive, Edinburgh.

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

Thursday 16 February

London Section: "Evaluation of the corrosion performance of organic coatings" hv Prof. W. Funke, Furschungsintitut für Pigments und Lacke, Stuttgart. Held in association with the Thames Polytechnic, Woolwich, at the Polytechnic, commencing at 12 noon.

Friday 17 Febuary

Irish Section: "The influence of emulsions on paint properties" by Mr D. Wallace, Vinyl Products Ltd, at the Clarence Hotel, Dublin 2 at 8.00 p.m.

Newcastle Section: Ladies' night. Five Bridges Hotel, Gateshead.

Thursday 23 February

Manchester Section: Student film evening, to be held at the Manchester Literary & Philosophical Society, Manchester, at 4.30 p.m.

Midlands Section: "Surface defects in surface coatings and their remedy" by Mr H. Vltavsky, Byk Mallinkroft, in the Cal-thorpe Suite, County Ground Edgbaston, Birmingham at 6.30 p,m.

Thames Valley Section: "Cathodic protec-There's rule's betrown callocate of bocce-tion" by Mr J. H. Morgan of Morgan, Berkeley & Co. Ltd, at the Beaconsfield Crest Motel (White Hart), Aylesbury End, Beaconsfield, Bucks at 6.30 for 7.00 p.m.

Friday 24 February

Bristol Section: "Painting aircraft" by Mr A. R. Peppitt of British Aircraft Corpora-tion Ltd, at the Royal Hotel, Bristol at 7.15 p.m.

JANUARY



1. A free bus shuttle service will operate between Alexandra Palace and Turnpike Lane station on the Piccadilly Line (Underground), which is denoted by the thick coloured line. Destinations of trains may be marked as "Cockfosters" or "Arnos Grove" or "Wood Green",

2. Those travelling by road will find free car parking facilities at Alexandra Palace.

3. Visitors arriving at West London Air Terminal may board the Piccadilly Line trains at Gloucester Road station.

4. The Piccadilly Line has now been extended to the Heathrow Central Terminal at the airport, and visitors can now board a train at the airport which will take them directly to Turnpike Lane station, or to hotels in central London at which they are staying.

5. The map also shows the position of the mainline stations in relation to the Piccadilly Line.

CURRENT PUBLICATIONS



The Introduction to Paint Technology, of which 18,000 copies have already been sold, forms an excellent introduction to the whole field of surface coatings and related technologies. The fourth edition contains the important addition of a glossary of the terms used in the book together with explanations of their derivations, as well as the completely revised and updated text.

Price £5.00 (Registered Students of the Association £2.50) Prepayment only. OIL AND COLOUR CHEMISTS' ASSOCIATION Priory House, 937 Harrow Rd., Wembley HA02SF Special Reduction! TEN-YEAR CONSOLIDATED INDEX

of Transactions and Communications

1966-1975 Volumes 49-58

The remaining copies of the Consolidated Index are offered at a specially reduced rate for a limited period only.

The special rate will be:

£2.00 to non-Members; £1.00 to Members

AVAILABLE (prepayment only) from:

OIL AND COLOUR CHEMISTS' ASSOCIATION

Priory House, 967 Harrow Rd., Wembley HA0 2SF

Newcastle Section Symposium 1975



polymerisation The bound copies of the twelve papers presented covering many aspects of this rapidly expanding field are available. Price: £5.00



Paint Technology Manuals Works Practice

This publication, which is of great use both to the practical man within the industry and the student entering the industry, is concerned with the practical aspects of making paints. As very little has been published on this subject, a fairly broad coverage is attempted including factory layout and organisation, paint and media manufacturing processes, legal aspects and safety precautions. **Price: £3.00**

Convertible Coatings

The second edition of Convertible Coatings, published in 1972, is invaluable to those dealing with this aspect of the industry, and a few copies are still available.

Price: £7.50 Published by Chapman & Hall Ltd on behalf of the Association.

Biennial Conference PREPRINTS

The Association organises an international Conference every two years and preprints of the papers are prepared for delegates. A strictly limited number of the following are available to those who wish to have the complete bound sets of papers.

1977 Eastbourne The conservation of energy, materials and other resources in the surface coatings industries. Fourteen papers presented. **Price: £5.00**

1975 Scarborough *Performance of surface coatings does reality match theory?* Seventeen papers presented. **Price: £5.00**

1973 Eastbourne Towards 2000. Sixteen papers presented. Price: £5.00

JANUARY

To: The Director and Secretary,	BOOK ORDER FORM		Å
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ix

CLASSIFIED ADVERTISEMENTS

Classified Advertisements are charged at the rate of £3.00 per cm. Advertisements for Situations Wanted are charged at 80p per line. A box number is charged at 50p. They should be sent to D. M. Sanders, Assistant Editor, Oil & Colour Chemists' Association, Priory House, 967 Harrow Road, Wembley, Middlesex HA0 2SF. JOCCA is published EVERY month and Classified Advertisements can be accepted up to at least the 12th, and in exceptional circumstances the 20th of the month preceding publication. Advertisers who wish to arrange for an extension of the copy deadline should contact the Assistant Editor, D. M. Sanders, at the address given above (telephone 01-908 1086, telex 922670 OCCA Wembley).

APPOINTMENTS VACANT



Please note our name and address for your future requirements.



Speed Electrics, The Wells Road, Nottingham. Tel: 0602-609782

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Wanted!!!

Flame-proofed battery electric forklift trucks in any condition. State type and price. Immediate inspection and settlement

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For Sale

16"×40" Lehmann Triple Roll Mill 15"×30" Vickers Triple Roll Mill

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6"×12" Marchant Laboratory Triple Roll Mill.

One off Heavy Duty 1000 lb Baker Parkins U. Trough mixer twin Naben Blades 100 h.p. electrics. Oil heated jacket.

One off 50 lb Baker Perkins twin Naben blade mixer jacketed.

One off 100 lb Baker Perkins twin Naben blade mixer jacketed.

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experience in organic polymer technology and the ability to communicate, orally and in writing, within a multi-discipline environment are the essential qualifications. Previous formulation experience with paints, inks, hot foil stamping, film converting and laminating will also be a definite advantage.

A salary of c.£5,800 p.a. and a competitive range of benefits are offered by our clients who are situated West of London.

Please write, giving full details of career to date quoting ref: /114, and listing any companies to whom you do not wish your application forwarded to Robin Atkins, Riley Management Selection (Services) Limited, Old Court House, Old Court Place, Kensington, London W8 4PD



30TH TECHNICAL EXHIBITION OF THE **OIL & COLOUR CHEMISTS'ASSOCIATION** Alexandra Palace · London 18-21 APRIL 1978

The world's most important international exhibition of raw materials, plant and equipment used in the Paint, Printing Ink, Colour, Adhesives and Allied Industries.

TUESDAY 18 APRIL	09.30	17.30
WEDNESDAY 19 APRIL	09.30	17.30
THURSDAY 20 APRIL	09.30	17.30
FRIDAY 21 APRIL	09.30	16.00

The Association will operate a free bus service to the Exhibition from (and return) Turnpike Lane Station (Piccadilly Line, London Underground).

Copies of the "Official Guide" including season admission tickets, at £2.00 each (including VAT) are obtainable in advance from the address below or at the entrance to the Exhibition.

Oil & Colour Chemists' Association, Priory House, 967 Harrow Road, Wembley, Middlesex, England, HAO 2SF Telephone: (01) 908 1086 Telex: 922670 (OCCA Wembley)

CLASSIFIED ADVERTISEMENTS

APPOINTMENTS VACANT

D. H. Industries Ltd, leading suppliers of modern equipment to the paint, printing ink and general chemical industries, has the following two vacancies.

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Reporting to the Sales Manager at Barking the person appointed will assume responsibility for the above territory. The range of products includes mixers, resin plants, dispersers, grinders, filters and filling machines.

Ideal background for this appointment will have been gained as a Sales Representative within one or more of the industries mentioned but candidates (male/female) with sound experience in an allied field will be considered.

In addition to a good basic salary (from £3900) an attractive commission scheme is in operation, and a car is provided. We are part of the Steetley Group and other benefits are as you would expect from a company of our size.

Paint/Ink Technologist

Based at Barking, the successful candidate will liaise with customers and representatives and be responsible for demonstrating the Company's range of equipment (mixers, dispersers, grinders, filters and filling machines) within the paint, printing ink and associated industries throughout the U.K.

The ideal background for this appointment will have been gained in a laboratory/works environment within one or more of the industries mentioned and the successful applicant (male or female) will need to demonstrate a mechanical aptitude as well as a sound appreciation of the industrial processes involved.

Commencing salary will depend on experience and be from £3500 p.a. and a Company car will be provided. We are part of the Steetley group and other benefits are as you would expect from a company of our size.

If interested in either vacancy please write giving brief career details to: Mr D. Sullivan, Managing Director, D.H. Industries Ltd, Lyon Industrial Estate, River Road, Barking, Essex.

PAINT TECHNOLOGIST FOR ZAMBIA

Progressive medium sized paint factory (originally British Paints subsidiary) manufacturing range of decorative and industrial finishes requires experienced paint technologist with H.N.C., City and Guilds Certificate or similar qualifications. Minimum 5 years' experience with knowledge of formulations. Excellent prospects for the right man, with good salary, generous leave allowances, 25% gratuity of total salary on completion of 2 year contract, free housing, car and passages. Contract renewable. $33\frac{1}{2}$ % of monthly salary may be remitted to home country. Excellent climate and facilities for sport and wild life viewing.

Apply with full details to:

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