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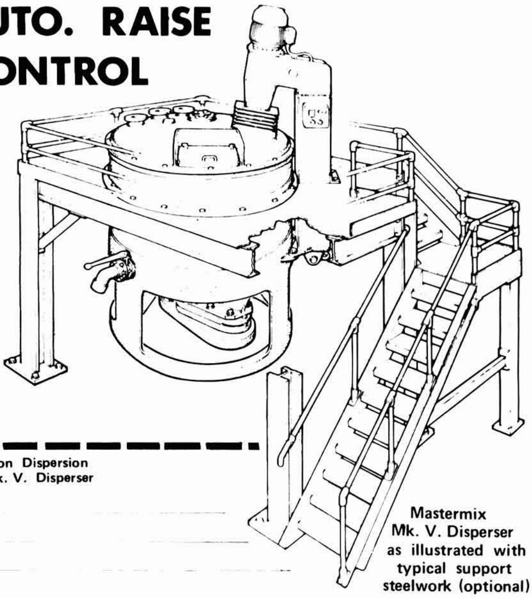

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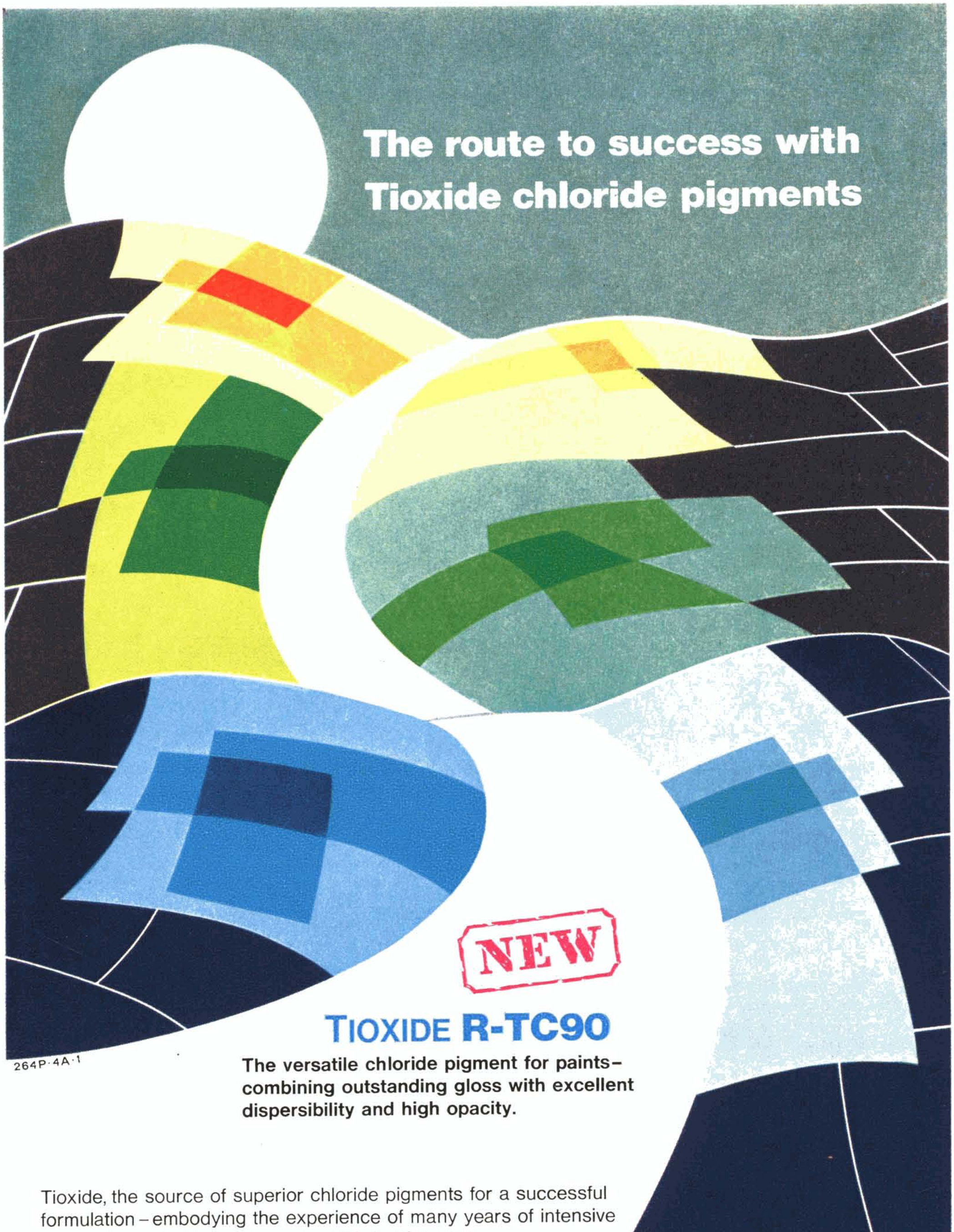

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Transactions and Communications

Synthesis of modified phthalocyanine compounds suitable as pigments

By N. A. Ghanem*, A. M. Naser, M. F. Ismail and M. A. Ghafar

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Summary

Highly coloured materials similar to the phthalocyanines were prepared by the stoichiometric replacement of phthalic anhydride and/or mono-nitrophthalic anhydride by pyromellitic dianhydride in such a manner as to prevent the formation of polymeric structures. Evaluation of these compounds indicated their suitability as pigments for coating applications.

Keywords

Raw materials for coatings
prime pigments and dyes
phthalocyanine pigment
phthalocyanine blue

La synthèse des phtalocyanines modifiées et aptes à être utilisées en tant que pigments

Résumé

On a préparé des substances fortement colorées et semblables aux phtalocyanines en remplaçant stœchiométriquement l'anhydride phtalique et/ou l'anhydride mono-nitrophtalique par le dianhydride pyromellique, de telle manière que l'on empêche la formation de structures polymères. L'appréciation de ces composés a indiqué leur aptitude à être utilisés en tant que pigments pour peintures.

Synthese von sich als Pigmente eignenden, modifizierten Phthalozyaninverbindungen

Zusammenfassung

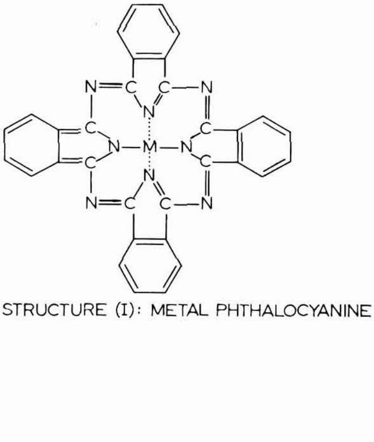
Sehr farbige, den Phthalozyaninen ähnliche Produkte wurden durch stöchiometrische Verdrängung von Phthalsäureanhydrid und/oder Mono-Nitrophthalsäureanhydrid durch pyromellitsaures Dianhydrid hergestellt und zwar so, dass die Bildung polymerischer Strukturen verhindert wird. Diese Verbindungen wurden als für Beschichtungsmittel geeignete Pigmente befunden.

Introduction

Refs. 1-6

Macrocyclic compounds containing four pyrrole units are of commercial importance in the manufacture of coloured pigments. The naturally occurring haemoglobin and chlorophyll and the phthalocyanines all belong to this class of compound[1].

Metal phthalocyanine (I) was prepared as early as 1907 in which the metal may be one of a variety of transition or main group metallic ions[2]. There is no general method of preparation suitable for all types of metal phthalocyanines, but all involve the interaction of a metal or metal salt with *o*-dicyano derivatives of an aromatic compound or its equivalent[3].



STRUCTURE (I): METAL PHTHALOCYANINE

The phthalocyanine pigments possess a number of physical and chemical characteristics which are ideal for many coating applications.

The purpose of this work is to combine the remarkable properties of the phthalocyanine molecule and the tetrafunctionality of the pyromellitic dianydride. Thus, attention was directed towards the partial replacement of the bifunctional phthalic anhydride by pyromellitic dianhydride in such a manner as to prevent the formation of polymeric structures and to minimise the alkali sensitivity. Such a treatment would be expected to produce higher molecular weight macrocyclic compounds, resembling the phthalocyanines in their structures. The literature is scant regarding the use of pyromellitic dianhydride in the formation of phthalocyanines, but polymeric copper phthalocynine and base-soluble dye have been prepared through the incorporation of pyromellitic dianhydride[4-6].

Experimental

Refs. 7, 8

Preparation of copper phthalocyanine[7] (Pigment VIII)

It is prepared according to the method described in the Paint Technology Manual by the urea fusion technique.

Preparation of modified copper phthalocyanine (Pigment I)

A mixture of phthalic anhydride (6 moles), pyromellitic dianhydride (1 mole), cupric chloride (2 moles) and urea (excess) were fused together in presence of few mg of ammonium molybdate as catalyst. The fusion was continued until a highly coloured mass developed. The reaction mass was then allowed to cool, pulverised and washed several times with dilute HCl, distilled water, ethanol and ether and dried at 105°C for 3 hours.

Preparation of modified nitro-phthalocyanine (Pigments II-VII)

The same technique adopted for the preparation of modified copper phthalocyanine was also used for their nitro derivatives. Various nitro compounds were prepared by introducing mononitro-phthalic anhydride (3- or 4-isomer) into the reaction mixture in such a manner as to produce various nitro derivatives. The final product obtained by using nitrophthalic anhydride alone is modified copper nitrophthalocyanine.

The crude pigments were purified using the "acid pasting" technique, followed by the usual washing system. The dried mass was then ground to a suitable degree of fineness. Further purification was carried out by sublimation under vacuum when required for analysis.

Controlled oxidation of the pigment[8]

The method used for the controlled oxidation of the pigments involves oxidation with potassium dichromate in an acidic medium under controlled experimental conditions.

Evaluation of the compounds prepared as pigments were conducted according to standard methods.

Results, discussion and conclusions

Refs. 4-6, 8

The formation of copper phthalocyanine by the urea fusion method offers an easy and simple means for its preparation. It was thought of interest to determine if replacing part of the phthalic anhydride by the tetra-functional pyromellitic dianhydride would lead to structures similar to the phthalocyanines, but of higher molecular weights. A similar approach has been described in the literature which involves the use of pyromellitic dianhydride[4-6]. These products were, however, substantially affected by alkalis and this was attributed to the presence of hydrophilic groups in the molecular network.

For this reason, it was felt of interest to extend the urea fusion method to the preparation of similar compounds using pyromellitic dianhydride and phthalic anhydride under such conditions as to prevent alkali dissolution and polymer formation.

It was found possible to prepare a brilliant bluish-green compound by heating a mixture of one mole of pyromellitic dianhydride and six moles phthalic anhydride with two moles cupric chloride and excess urea in the presence of ammonium molybdate as a catalyst at 200°C until a highly coloured mass was formed. After washing, purification, drying and grinding, the product showed a brilliant greenish-blue colour. The absence of any chlorine in the product was confirmed qualitatively and the possibility of chlorination during the formation was eliminated. Chlorination has been described as a means for producing phthalocyanine green. The insolubility of the product in dimethylformamide eliminates the possibility of polymer formation, since polymers of pyromellitic anhydride would contain peripheral hydroxy groups and would be soluble.

Sublimation under vacuum resulted in the formation of a small amount of a white impurity, soluble in alkali and having no definite melting point. Copper phthalocyanine blue was also noticed in traces.

The product obtained is analogous to the phthalocyanine and shares the following aspects in common:

(a) Their method of preparation is the same; starting from 1:2-dicarboxylic acid anhydride and 1:2-, 4:5-tetracarboxylic dianhydride respectively.

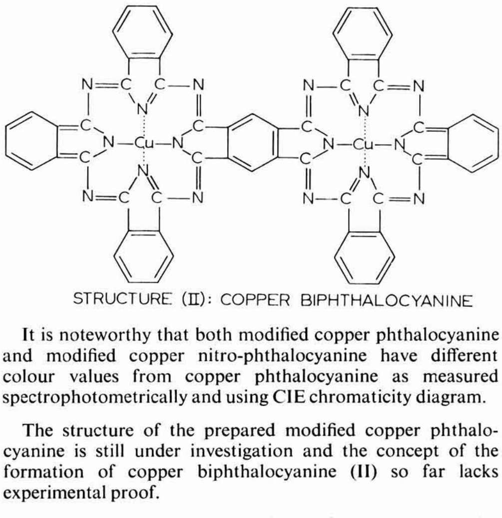
(b) They are stable towards the action of light and heat and are insoluble in most organic solvents. Their mass-tone colours showed metallic lustre.

(c) Their acidified solutions in sulphuric acid are considerably affected by various oxidising agents. Also, both copper phthalocyanine and modified copper phthalocyanine can undergo controlled oxidation to the corresponding amide[8].

(d) They are capable of forming various metallic derivatives.

(e) UV and IR spectra showed the characteristic peaks present in the porphyrazine ring.

(f) End group titration of modified copper phthalocyanines using a potentiometric technique clearly indicated the absence of free carboxylic groups in the peripheral benzene rings, thus eliminating the possibility of polymer formation[5].



STRUCTURE (II): COPPER BIPHTHALOCYANINE

It is noteworthy that both modified copper phthalocyanine and modified copper nitro-phthalocyanine have different colour values from copper phthalocyanine as measured spectrophotometrically and using CIE chromaticity diagram.

The structure of the prepared modified copper phthalocyanine is still under investigation and the concept of the formation of copper biphthalocyanine (II) so far lacks experimental proof.

Following the preparation of modified copper phthalocyanine, attention was directed towards the preparation of their nitro derivatives. A series of nitro compounds was prepared starting with phthalic anhydride and/or 3- and 4-nitrophthalic anhydride. These compounds are given the reference numbers shown in Table 1 and are used throughout the rest of the text. Copper phthalocyanine (VIII) is included also for the sake of comparison.

It should be mentioned that compounds II—VI include more than one isomer.

The compounds prepared were tested for the following pigment properties:

Acid-pasting, oil absorption, specific gravity, (Sp.g.), critical pigment volume concentation (CPVC), bulking value (BV) and hiding power

Table 1
Composition of pigments

Pigment No.	Composition of acids employed (moles) Phthalic anhydride	 Nitro-phthalic anhydride	 Pyromellitic dianhydride
I	6	–	1
II	5	1	1
III	4	2	1
IV	3	3	1
V	2	4	1
VI	1	5	1
VII	0	6	1
VIII	4	0	0

The acid pasting treatment is a well recognised technique for improving the properties of the phthalocyanine pigments, especially with respect to crystal growth and tinting strength. Applying this technique to the pigments prepared indicated its suitability, since more than 90 per cent of the original pigment was recovered. The physical characteristics of the prepared pigments are given in Table 2.

Table 2
Pigment characteristics

Pigment No.	Oil abs. g/100 g.	Sp.gr.	CPVC	BV	Mass-tone colour	Tint-tone colour
I	37.1	1.387	64.3	8.7	Greenish blue	Light green
II	34.3	1.428	65.4	8.4	„	„
III	31.6	1.448	67.1	8.3	„	„
IV	29.7	1.465	68.0	8.2	Violet	„
V	26.9	1,484	69.9	8.1	„	„
VI	24.1	1.509	71.8	8.0	„	„
VII	22.3	1.548	72.9	7.8	„	„
VIII	41.8	1.357	62.0	8.9	Blue	Light blue

It is worthy of mention that all the pigments studied showed moderate hiding power. The main conclusion drawn from these studies clearly illustrates the effect of the nitro group on pigment properties. Thus, increasing the nitro group content leads to a decrease in oil absorption and bulking values. On the other hand, it leads to an increase in the specific gravity.

Tinting strength, gloss and spot test measurements

The tinting strength was measured relative to zinc oxide and was found to give high values. These observations are in agreement with the theoretical consideration, since most organic pigments are characterised by low hiding power values and are of remarkable tictorial strength. Such pigments also showed no effect on the gloss of their dried films in linseed oil.

The results obtained from the spot test studies showed that all pigments behave similarly to the phthalocyanines.

Bleed, permanence to light and heat and chemical resistances

The bleeding test was conducted in various organic solvents. Alkali resistance was conducted in 2% NaOH, saturated $Ca(OH)_2$ and 5% Na_2CO_3 solutions, whilst the acid resistance was carried out in 5% H_2SO_4 solution. Both permanence to light and heat tests were conducted on actual coating films using boiled linseed oil as a vehicle. These films were tested after seven days exposure to direct sunlight (December) and after 60 minutes at 105°C, 45 minutes at 120°C and 30 minutes at 135°C, 15 minutes at 150°C and 10 minutes at 175°C. Results of these studies indicated a high degree of stability towards alkalis, acids, solvents, light and heat. These remarkable and outstanding properties should make them excellent pigments for various coating applications.

[Received 7 March 1978

References

1. Roberts, J. D. and Caserio, M. C., Basic Principles of Organic Chemistry, *W. A. Benjamin, Inc.*, N.Y., 1964.
2. Moder, F. H. and Thomas, A. L., Phthalocyanine Compounds, *Reinhold Publishing Corp.*, N.Y., 1963.
3. Dahlen, M. A., *Ind. Eng. Chem.*, 1939, **31,** 839.
4. Marvel, C. S. and Rassweiler, J. H., *J.Am.Chem.Soc.*, 1958, **80,** 1197.
5. Drinkard, W. C. and Bailer, J. C., *J.Am.Chem.Soc.*, 1959, **81,** 4795.
6. British Patent 801, 488 (1958); C.A. 7622g, 1958.
7. Paint Technology Manuals, Part VI, *OCCA*, 1966.
8. Islam, A. M., et al, *JOCCA*, 1974, **57,** 134.

J. Oil Col. Chem. Assoc. **61**, 372-374

Recent aspects of some coloured urea/dibasic acid fusion adducts

By A. M. Naser, M. M. Naoum, A. A. Salman and A. Taha

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Summary

The formation of various coloured products by the fusion of urea with different dibasic acids or anhydrides in the presence of inorganic salts has been studied. These acids include both aromatic and aliphatic types. The colour of the compound prepared was measured spectrophotometrically. These compounds showed promising properties when evaluated as pigments in various coating applications.

Keywords

Raw materials for coatings
prime pigments and dyes
phthalocyanine pigment

Raw materials used in manufacture or synthesis of ingredients for coatings
dibasic acid

Aspects récents de certains produits d'addition colorés résultant de la condensation de l'urée et un acide dibasique

Résumé

On a étudié la formation de divers produits colorés résultant de la condensation de l'urée et divers acides dibasiques en présence des sels inorganiques. Les acides comprennent des types et aromatique et aliphatique. La couleur de chaque composé préparé a été dosée spectrophotométriquement. Ces composés ont démontré des caractéristiques intéressantes au cours des essais destinés à les apprécier en tant que pigments pour diverses peintures.

Neue Betrachtungen einiger farbiger Harnstoff/zweibasische Säure Verschmelzungsaddukte

Zusammenfassung

Es wurde die Bildung verschiedener farbiger Produkte durch Verschmelzung von Harnstoff mit verschiedenen zweibasischen Säuren oder Anhydriden in Gegenwart von anorganischen Salzen untersucht. Diese Säuren schliessen sowohl aromatische als auch aliphatische Typs ein. Die Farbe der hergestellten Verbindung wurde spektrophotometrisch gemessen. Auf ihre Eignung als Pigmente in verschiedenen für Anstrichzwecke geeigneten Bindemitteln geprüft, zeigten diese Verbindungen ein günstiges Verhalten.

Introduction

Refs. 1, 2

The fusion of urea with organic acids is frequently used for the preparation of their corresponding ammonium or amide derivatives. However, fusion with a dibasic acid or anhydride leads to the formation of a wide variety of products depending on the type of acid present and the experimental conditions employed. For example, fusion with phthalic anhydride leads to the formation of the well-known phthalocyanine[1], whilst reaction with substituted malonic esters results in the formation of barbituric acids[2].

This paper extends the application of the urea fusion technique to a wide variety of dibasic materials with the aim of forming coloured products suitable as pigments.

Experimental

Refs. 3, 4

The preparation of copper phthalocyanine (I), copper tetranitrophthalocyanine (IIa,b), copper tetra-aminophthalocyanine (IIIa,b) and copper tetra-chlorophthalocyanine (IV) were based on the method described in the Paint Technology Manual[3] starting with phthalimide, nitro-phthalimide, aminophthalimide and chloro-phthalimide respectively (3- and 4-isomers).

On the other hand, copper phthalocyanine (V) and modified nitro-phthalocyanine (VI) were prepared by the incorporation of pyromellitic dianhydride in the fusion mixture[4].

The purification of the phthalocyanines and modified phthalocyanines was carried out by acid pasting and vacuum sublimation.

The urea fusion products with aliphatic acids (maleic, succinic, adipic, glutaric and oxalic acids, VII-XI) were also prepared in a similar manner to that described previously[4]. The purification of these compounds was performed by successive washings with 5% HCl, 1% NaOH, distilled water, ethanol and ether and drying at 105°C for three hours.

Evaluation of the compounds prepared to assess their suitability as pigments was conducted according to standard methods.

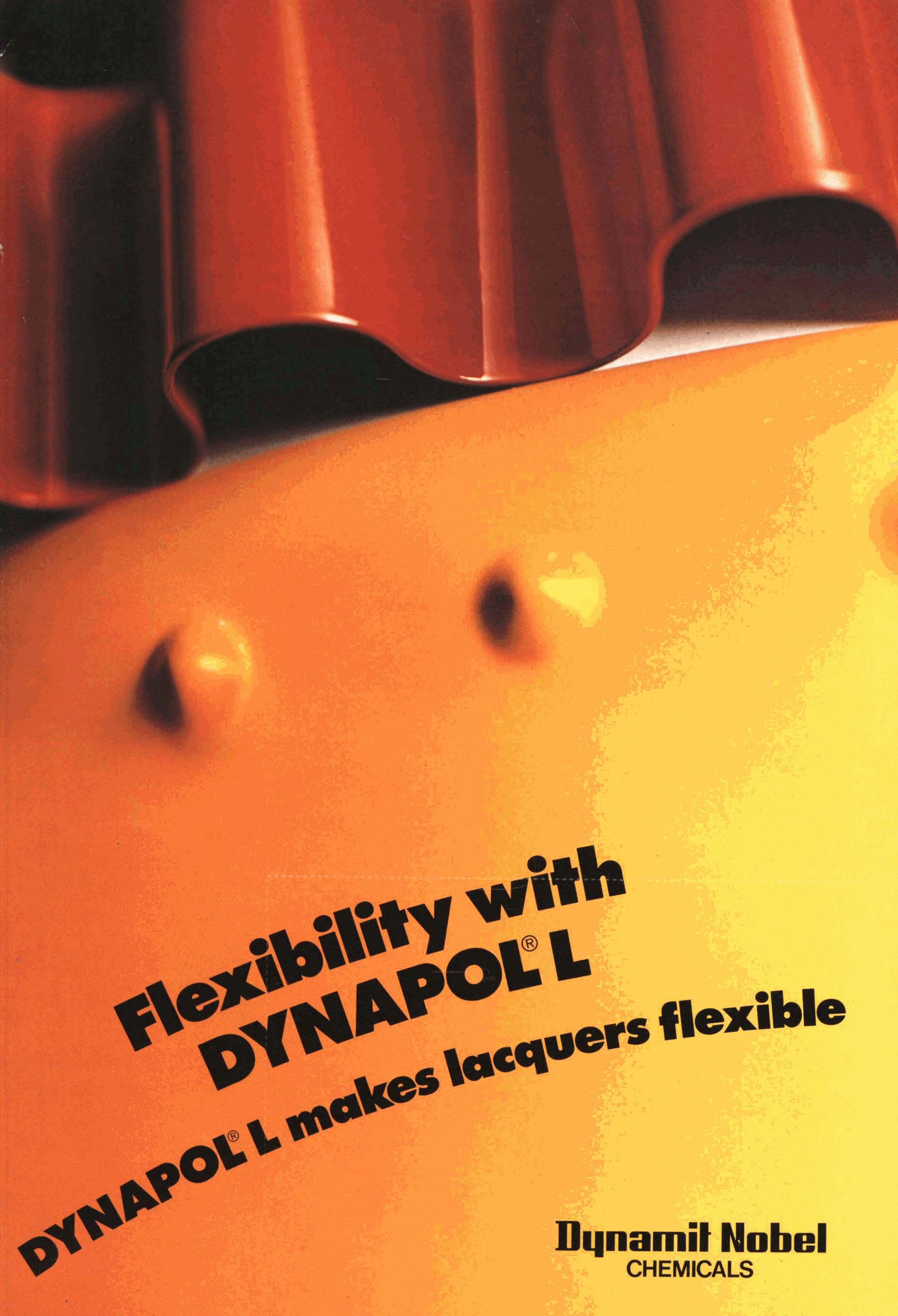
Results, discussion and conclusions

Ref. 5

Coloured compounds were prepared by the fusion of urea with dibasic materials in the presence of cupric chloride at 200-220°C. The end product depends upon the acid employed. Table 1 gives a list of these compounds.

It is worth mentioning that purification of aliphatic type compounds by the acid pasting technique leads to a substantial loss of the material, only less than 5 per cent of the original compound being recovered.

The structure of the phthalocyanines is well established whilst those of the rest are uncertain and lack experimental proof[5]. This aspect is still under investigation.



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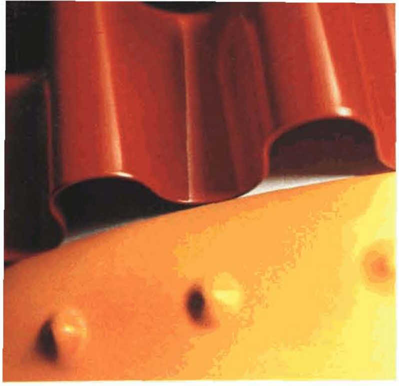

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Table 1

List of compounds prepared

Number	Suggested name
I	Copper phthalocyanine
IIa	Copper tetra-nitrophthalocyanine (3-isomer)
IIb	Copper tetra-nitrophthalocyanine (4-isomer)
IIIa	Copper tetra-aminophthalocyanine (3-isomer)
IIIb	Copper tetra-aminophthalocyanine (4-isomer)
IV	Copper tetra-chlorophthalocyanine (4-isomer)
V	Modified copper phthalocyanine
VI	Modified copper nitro-phthalocyanine
VII	Copper urea/maleic fusion adduct
VIII	Copper urea/succinic fusion adduct
IX	Copper urea/adipic fusion adduct
X	Copper urea/glutaric fusion adduct
XI	Copper urea/oxalic fusion adduct

Although the aliphatic type of compounds showed a considerable degree of stability towards heat and most organic solvents, their capability of producing metallic derivatives and their mode of formation make them similar to the phthalocyanines, but elemental analyses and alkali instability eliminate the possibility of the formation of macrocyclic molecules of the porphyrazine series (tetra-aza-porphin), especially in the case of urea/oxalic fusion adduct. Such a molecule is difficult to obtain under such conditions.

The characteristics of the compounds prepared are shown in Table 2.

Rating of the alkali and acid resistances was described as follows: *Ex* means excellent; almost unaffected, *G* means good; slightly affected, *P* means poor; considerably affected and *F* means failure; almost completely affected.

All pigments showed metallic lustre in the crude form and exhibited excellent bleed resistance. Pigments of an aromatic nature were found to be blue or green in colour, whilst those of aliphatic nature showed dark brown or black colours. Both aromatic and aliphatic types showed remarkable differences regarding their stability towards alkalis, the latter being easily affected. In addition, they behave differently in the spot test.

The phthalocyanine and nitrophthalocyanines are blue in colour whilst the amino- and chloro-phthalocyanines showed different green shades. Modified phthalocyanines are intermediate between them.

Following the preparation of the various coloured pigments, it was considered of interest to measure their specified colours using a reflection technique and to seek a relation between the chemical constitution of the pigment and its colour (Figure 1 is the CIE chromaticity diagram). Colour measurements data are given in Table 3.

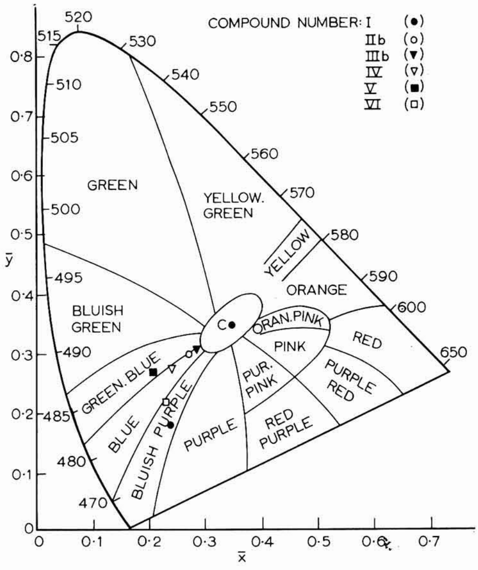

Fig. 1. The CIE chromaticity diagram.

Table 2

Characteristics of the pigments prepared

No.	Spot test a	Spot test b	Spot test c	Mass-tone colour	Tint-tone colour	Resistance against 1% NaOH	Resistance against 5% HCl
I	Green	Blue	Blue	D. violet	Blue	*Ex*	*Ex*
IIa	Blue/green	Blue	Green	D. violet	Blue	*Ex*	*Ex*
IIb	Blue/green	Blue	Blue	D. violet	Blue	*Ex*	*Ex*
IIIa	Brown	Brown	Brown	D. brown	Brown	*Ex*	*Ex*
IIIb	Green	Green	Green	D. yellow	Yellow/green	*Ex*	*Ex*
IV	Brown	Blue	Blue	D. blue	Blue	*Ex*	*Ex*
V	Light/green	Blue/green	Blue	Green/blue	Light green	*Ex*	*Ex*
VI	Light green	Blue	Blue	Blue	Blue	*Ex*	*Ex*
VII	Brown	Brown	Brown	D. brown	Light brown	*F*	*G*
VIII	Black	D. brown	Black	Grey	Grey	*F*	*G*
IX	Brown	Brown	Brown	Brown	Brown	*F*	*G*
IX	Brown	Brown	Brown	Brown	Brown	*F*	*G*
X	Brown	Brown	Brown	Brown	Brown	*F*	*G*
XI	Brown	Brown	Brown	Brown	Brown	*F*	*G*

where: a = conc. sulphuric acid, b = dilute sulphuric acid solution, c = ammonium hydroxide neutralised solution,
D = dark.

Table 3

Colour Measurements Data

No.	x	y	z	λnm	Per cent purity
I	23.64	18.25	58.10	470	53.6
IIa	23.46	24.42	52.12	480	44.9
IIb	27.21	29.99	42.80	484	27.6
IIIa	31.96	31.56	36.48	477	14.2
IIIb	29.37	31.60	39.16	485	20.4
IV	24.49	27.98	47.98	482	37.6
V	20.87	27.19	51.94	485	34.6
VI	24.15	21.93	53.92	475	46.8
VII	30.80	31.17	38.03	000	00.0
VIII	30.33	30.69	38.98	000	00.0
IX	35.60	36.80	29.40	000	00.0
X	35.80	35.60	28.40	000	00.0
XI	35.90	35.90	28.40	000	00.0

The following generalisations were drawn from the above studies:

(1) The presence of substituted groups in the benzene ring at either the 3- or 4-positions does not significantly affect the colour value.

(2) 3-aminophthalocyanine (IIIa) behaves differently and shows a dark brown appearance. This is attributed to the presence of the strong activating group which makes the molecule susceptible to oxidation with the possibility of partial formation of dark compounds.

(3) In general, the presence of amino, nitro and chloro groups in the pigment molecule results in a considerable change in the colour characteristics. The degree of effectiveness is in the following order:

$$\text{Amino} > \text{Nitro} \simeq \text{Chloro}$$

As a matter of fact, they cause a shift in the colour towards the green as indicated from the CIE chromaticity diagram. The dominant wavelength was found to be unaffected upon dilution with zinc oxide.

(4) Modified copper phthalocyanine showed more greenish shade than that of phthalocyanine. On the other hand, modified nitro-phthalocyanine has an intermediate shade between the other types.

(5) The colour of the alphatic types of pigments lie in the central zone of the chromaticity diagram indicating their blackish appearance.

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References

1. Moser, F. M., and Thomas, A. L., Phthalocyanine Compounds, *Reinhold Publishing Corporation*, N.Y., 1960.
2. Roberts, J. D., and Caserio, M. C., Basic Principles of Organic Chemistry, *Benjamin Inc.*, N.Y. 1964.
3. Paint Technology Manuals, Part VI, *OCCA* 1966.
4. Abd El-Gafar, M. A., M.Sc. *Thesis*, Al-Azhar University, 1975.
5. Ghanem, N. A., Naser, A. M., Ismail, M. F., Ghafar, M. A., *J.O.C.C.A.*, 1978, **61**, 0000.

J. Oil Col. Chem. Assoc. 1978, **61**, 375-382

Cellulose based resins for surface coatings

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Summary

Cellulose esters of dibasic acids such as adipic, maleic and phthalic acids, and cellulose half-esters of phthalic acid in which one carboxyl group is reacted with cellulose and the other with glycol or glycerol were prepared and their film properties studied. Rosin modified cellulose glycol phthalate and cellulose glycerol phthalate were also prepared and studied for film properties. Films of cellulose esters of dibasic acids showed excellent physical and chemical resistance, whereas those modified with rosin showed inferior properties. On comparing the film properties of these derivatives with those of nitro-cellulose, it was observed that films of cellulose maleinates and phthalates compare favourably with the nitro-cellulose films.

Keywords

Raw materials for coatings
binders (resins, etc.)
cellulose ester
cellulosic resin
polyester resin

Raw materials used in manufacture or synthesis of ingredients for coatings
dibasic acid

Processes and methods primarily associated with analysis, measurement or testing
mandrel test

Properties, characteristics and conditions primarily associated with coatings during operation
drying time

dried or cured films
scratch resistance
water resistance
solvent resistance
chemical resistance

Résines à base de cellulose destinées à être utilisées en peintures

Résumé

Les esters cellulosiques des acides dibasiques, tels que les acides adipique, maléique ou phtalique et les semi-esters de l'acide phtalique où un groupement carbonyle est fait réagi avec cellulose et l'autre avec glycol ou glycérine et ensuite les caractéristiques de leurs films ont été étudiées. Le phtalate de cellulose et de glycol et le phtalate de cellulose et de glycérol, tous les deux étant modifiés par colophane ont été préparés et les caractéristiques de leurs films ont été étudiées. Les films des esters cellulosiques d'acides dibasiques ont démontré une excellente résistance et chimique et physique, tandis que ceux modifiés par colophane ont mis en évidence les caractéristiques inférieures. En faisant la comparaison des caractéristiques des films de ces dérivés auprès de ceux rendus par nitrocellulose, on a observé que les films des maléinates et des phtalates de cellulose se comparent très favorablement auprès des films nitrocellulosiques.

Lackharze auf Zellulosebasis

Zusammenfassung

Präparate von Zelluloseestern zweibasischer Säuren, wie Adipin-, Malein- und Phthalsäure, und Zellulosehalbestern der Phthalsäure, in welchen die eine Karboxylgruppe mit Zellulose, die andere mit Glykol oder Glyzerin in Reaktion gebracht worden waren, wurden hergestellt, und deren Filmeigenschaften geprüft. Ebeuso wurden harzmodifizierte Zelluloseglykolphthalate und Zelluloseglyzerinphthalate präpariert und auf die Eigenschaften ihrer Filme hin untersucht. Filme von Zelluloseestern zweibasischer Säuren zeigten ausgezeichnete physikalische und chemische Beständigkeit, die mit Harz modifizierten zeigten dagegen ungünstiges Verhalten. Beim Vergleich der Filmeigenschaften dieser Derivate mit denen von Nitrozellulose wurde beobachtet, dass sich Filme von Zellulosemaleinaten und -Phthalaten mit denen von Nitrozellulose messen konnten.

Introduction

Refs. 1-4

Unlike the many polymeric compounds that are produced by the polymerisation of relatively simple low molecular weight components, the cellulose derivatives owe their polymeric nature to that of the basic component cellulose itself. Cellulose is composed of a variable but extremely large number of anhydro-glucose units linked chemically in a hemiacetal configuration. Since each unit has three hydroxyl groups, the cellulose can assume the role of alcohol in conventional esterification and etherification reactions[1].

The chemical structure of cellulose can be changed in three ways[2]:

(i) by preparing derivatives, *e.g.*, esters, ethers, *etc.*

(ii) by preparing crosslinked cellulose.

(iii) by preparing a copolymer or branched cellulose.

Of the above, the first method of modifying the chemical structure of cellulose as esters or ethers is, of course, the oldest and most widely used. The crosslinking of cellulose is also well known and an established technique for the treatment of cellulose to impart crease resistance, shrink resistance and a number of other properties. The cellulose chain becomes part of a three-dimensional network; the material becomes harder and more rigid and loses most of the properties which are desirable for the coatings. The copolymers or branched cellulose, on the other hand, present a means of modifying the cellulose molecule by the creation of branches which impart to the cellulose certain desirable properties without destroying the properties of natural cellulose. This accounts for the great interest in cellulose copolymers based on branching techniques[3,4].

The present work is based on the third method in which cellulose is modified with dibasic acids of which one or both of the carboxylic groups are esterified with cellulose. Here, again, three types of products are possible:

(a) those in which both carboxylic groups are combined with cellulose,

(b) those with one carboxylic group attached with cellulose and the other in free acid form, and

(c) those with one carboxylic group attached with cellulose and the other to another molecule.

Derivatives of the first type are not soluble in common solvents and do not lend themselves to useful application unless there is a controlled degree of substitution. Those of the second type are also not of much use because of the free carboxylic group. This third type of derivative, however, offers unique properties and can become soluble in most of the common solvents. Therefore, in the present work cellulose has been modified as esters of dibasic acids and half esters of phthalic acid further modified with glycol, glycerol and rosin. Rosin has been incorporated in order to reduce the cost of the product and to make it soluble in hydrocarbon solvents.

Experimental

Materials

Cellulose

Waste cotton linters of short fibre length were collected from local cotton processing industry and were used as the basic material after degradation.

Sulphuric acid

BDH, LR grade sulphuric acid was used as catalyst for the preparation of cellulose acetate and as a chain breaker for cellulose degradation.

Sodium hydroxide

Sarabhai M. Chemicals, LR grade was used for the purification of raw cotton.

Phthalic anhydride

BDH, LR grade was used for the preparation of cellulose phthalate.

Maleic anhydride

BDH, LR grade was used for the preparation of cellulose maleinate.

Adipic acid

BDH, LR grade was used for the preparation of cellulose adipate.

Ethylene glycol

BDH, LR grade was used for the esterification of cellulose phthalate and for the preparation of glycol ester of rosin.

Glycerol

BDH, LR grade was used for the preparation of cellulose glyceryl phthalate and estergum.

Rosin

Best grade (N) was used for the modification of cellulose glycol phthalate and cellulose glyceryl phthalate and also for the preparation of esters of rosin.

Pentaerythritol

BDH, LR grade was used for the preparation of penta-estergum.

Pyridine

Commercial grade, was redistilled at 115-116°C.

Magnesium perchlorate

BDH, LR grade was used as catalyst.

Nitro cellulose

A sample of commercial ½ sec. nitrocellulose was obtained from a local firm.

Solvents

All solvents used were of BDH, LR grade for the purpose of dissolving and thinning the cellulose derivatives. Mineral turpentine used for rosin based cellulose derivatives was of commercial grade.

Degradation of cellulose

Cellulose was depolymerised with sulphuric acid. In a typical experiment, 300 g cotton linters were taken in a three litre beaker. To this, 100 ml of concentrated sulphuric acid diluted with 2 litres of water was added whilst stirring. The material was heated to 60°C and kept at this temperature for 2 hours with stirring. During this time the cotton linters shortened in length. A further heating of the product gave a powder form of cellulose.

The product was washed with hot water in order to remove any trace of acid. Finally it was purified as described below.

Purification of cellulose

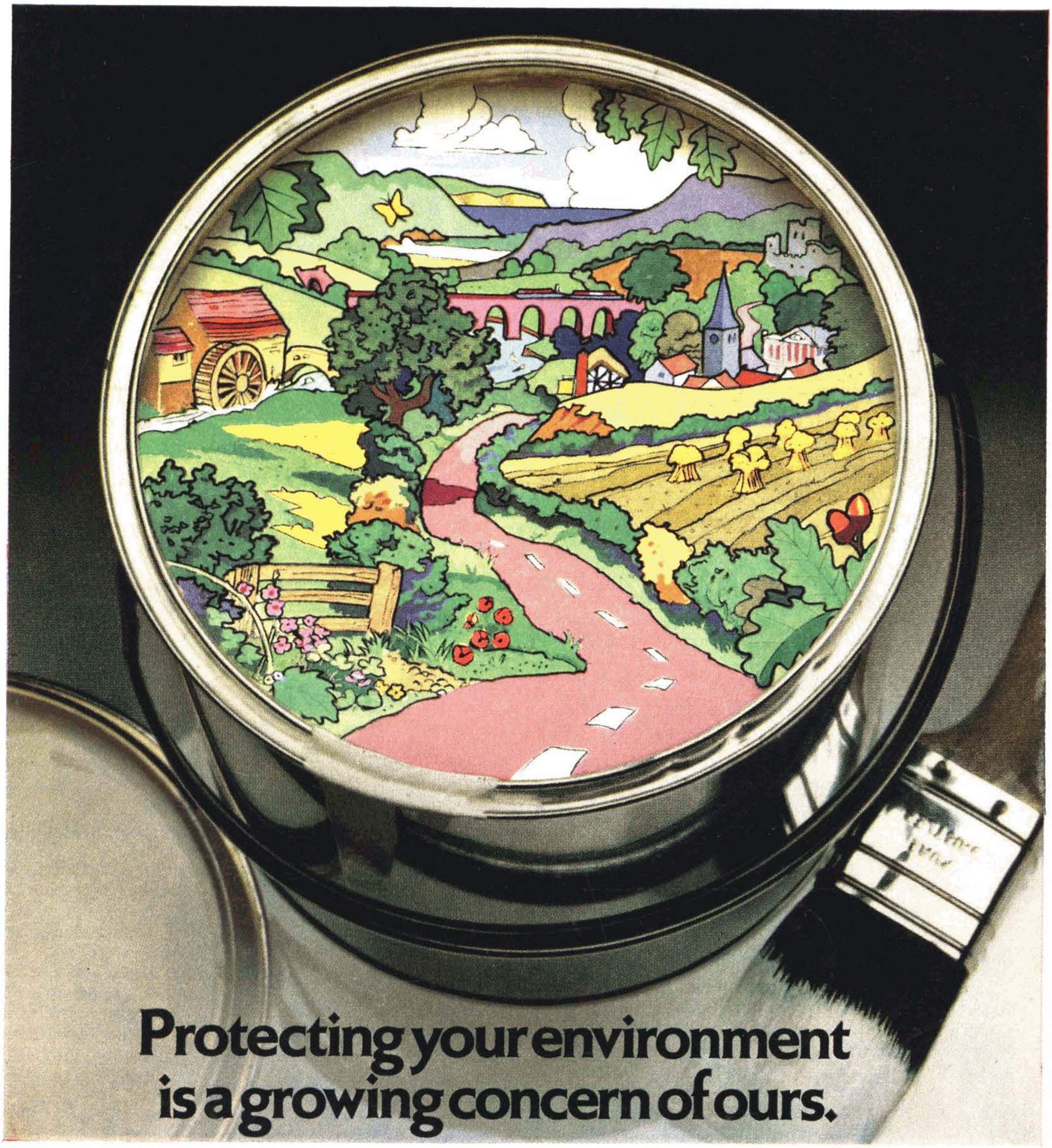
Degraded cellulose was purified by taking the cellulose powder as obtained above into a beaker to which 10% sodium hydroxide solution was added. The material was heated on a water bath at a temperature of 98°C for 8 hours with continuous stirring. The whole mass was dropped into large volume of water, washed well, and dried at 60°C for 4 hours.

Preparation of cellulose esters

The various cellulose esters were prepared in the following manner:

Cellulose adipate

40 g cellulose and 85 g adipic acid were heated in a three necked flask fitted with stirrer and condenser in the presence of 65 g pyridine, 8 g magnesium perchlorate and 5 g chloroacetic


Protecting your environment
is a growing concern of ours.

anhydride at a temperature of 130°C for 10 hours. After this time the material was dissolved into a homogeneous mass of dark brown colour. Pyridine was removed as a salt by the addition of 70 g concentrated hydrochloric acid. The precipitate was washed free of acid and finally dried at 60°C for 4 hours.

The yield of the product was 120 g. The true hydroxyl value of the sample was found to be 111.5 from which the degree of substitution was determined with the help of Fig. 1. The degree of substitution of the sample was found to be 2.66. The sample was dissolved in the following combination of solvents to 50 per cent solid content.

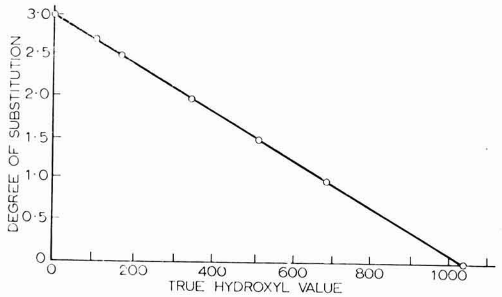


Fig. 1. Relationship between true hydroxyl value and degree of substitution.

Amyl acetate	20 parts by wt.
Benzene	30 parts by wt.
Toluene	25 parts by wt.
Butyl alcohol	45 parts by wt.

The solution of cellulose adipate was brown in colour and of fair clarity.

Cellulose maleinate

35 g degraded and purified cotton linters were heated with 45 g pyridine, 80 g of maleic anhydride, 3 g of magnesium perchlorate and 4 g chloroacetic anhydride at 100°C for 10 hours with continuous stirring. After this time the whole material was converted into a viscous homogeneous solution. The product was then reacted with 45 g of hydrochloric acid so as to remove the pyridine salt. The remaining portion was washed well, dried at 60°C for 4 hours. The degree of substitution of the sample was found to be 2.45 from its true hydroxyl value of 185.3 with the help of Fig. 1. The product was dissolved in the following combination of solvents to 50 per cent solid content.

Butyl acetate	15 parts by wt.
Acetone	15 parts by wt.
Benzene	30 parts by wt.
Butyl alcohol	40 parts by wt.

The solution of cellulose maleinate was of light yellow colour and excellent clarity.

Cellulose phthalate

25 g of degraded and purified cotton linters and 80 g of phthalic anhydride were taken in a three-necked flask with 100 g of pyridine, 3 g of magnesium perchlorate and 3 g chloroacetic anhydride. The whole material was kept with continuous stirring for 12 hours at 120°C. During this time, the material formed a clear viscous solution, which was precipitated in a large volume of water containing 85 g of hydrochloric acid. The product was then washed with hot distilled water until free from any residual acid. Finally the product was dried at 60°C for 4 hours and the degree of substitution determined and found to be 2.60 from its true hydroxyl value of 134.6 by means of Fig. 1. The sample was dissolved in the following combination of solvents to 50 per cent solid content.

Amyl acetate	15 parts by wt.
Acetone	15 parts by wt.
Xylene	20 parts by wt.
Butyl alcohol	10 parts by wt.
Benzene	10 parts by wt.

The solution of cellulose phthalate was red in colour and had very good clarity.

Cellulose acetate

50 g acetic acid and 75 g acetic anhydride were charged into a three-necked flask containing 50 g cellulose. The material was heated to 50°C for 1 hour with continuous stirring. Again 40 g acetic acid, 70 g acetic anhydride and 1 g sulphuric acid were added to the material taking precautions not to increase the temperature above 60°C in order to minimise the degradation of the cellulose. The whole material had formed a clear viscous solution within 1 hour.

The above solution was then kept for hydrolysis to which 80 g water had been added and the temperature of the solution was maintained between 60 to 70°C for 4 hours. The product was first tested for acetone solubility by taking a few ml of the sample, flooded in water, filtered, washed and dried by pressing between filter papers. The dried sample was then dissolved in acetone to about 50 per cent solids which gave a clear solution.

Finally, the material was poured into a large volume of water acidified with acetic acid. The precipitate was washed free of acid and stabilised by boiling in water (containing 0.001 per cent H_2SO_4). Thereafter the material was dried at 60°C for 4 hours. The yield of cellulose acetate was 128 g, which was dissolved in acetone to a 50 per cent solid content.

Preparation of cellulose half ester and other derivatives

Cellulose phthalate half ester

Cellulose phthalate half ester was prepared by taking 25 g degraded and purified cotton linters, 60 g phthalic anhydride and 45 g pyridine into a 3-necked flask. The material was heated in the presence of 5 g magnesium perchlorate at 110°C for 8 hours. During this period the whole mass became a clear viscous solution. The pyridine was removed as a salt by addition of 45 g concentrated hydrochloric acid. The material was then washed and dried at 60°C for 3 hours. This product was used for the preparation of other cellulose derivatives.

Cellulose glycol phthalate

Cellulose phthalate half ester as prepared above was further reacted with 40 g ethylene glycol at a temperature of 150°C for 3 hours, yielding a clear viscous mass. Cellulose glycol phthalate was dissolved in the following combination of solvents to give 50 per cent solid content.

Butyl acetate	10 parts by wt.
Acetone	20 parts by wt.
Benzene	20 parts by wt.
Toluene	25 parts by wt.
Butanol	25 parts by wt.

The varnish of cellulose glycol phthalate in the above solvent mixture was of yellow colour and good clarity.

Cellulose glyceryl phthalate

Cellulose phthalate half ester was prepared in the same manner as described above and was reacted with 40 g of glycerol at 150°C for 4 hours, giving a clear, transparent, viscous material and thinned with the following combination of solvents to 50 per cent solid content.

Butyl acetate	10 parts by wt.
Benzene	20 parts by wt.
Toluene	25 parts by wt.
Acetone	20 parts by wt.
Butanol	25 parts by wt.

The solution of cellulose glycerol phthalate in the above solvents was yellowish red in colour and had good clarity.

Cellulose glyceryl phthalate modified with rosin

Cellulose glyceryl phthalate was prepared as described above. It was then heated with 65 g of rosin at 150°C for 3 hours. The material was cooled and thinned with the following combination of solvents to 50 per cent solid content.

Mineral turpentine	15 parts by wt.
Benzene	35 parts by wt.
Toluene/xylene (50/50)	25 parts by wt.
Acetone	15 parts by wt.
Ethyl acetate	10 parts by wt.

The varnish of cellulose glyceryl phthalate modified with rosin in the above solvents was reddish in colour and had good clarity.

Cellulose glycol phthalate modified with rosin

Cellulose phthalate half ester was prepared as mentioned earlier. It was then reacted with 40 g glycol at a temperature of 150°C for 3 hours. It was then heated with equal parts of rosin at 150°C for 2 hours. The solution was cooled and thinned with the following combination of solvents to 50 per cent solid content.

Mineral turpentine	15 parts by wt.
Benzene	30 parts by wt.
Xylene	20 parts by wt.
Toluene	15 parts by wt.
Acetone	10 parts by wt.
Ethyl acetate	10 parts by wt.

The solution of cellulose glycol phthalate modified with rosin in the above solvents was of brown colour and had fair clarity.

Cellulose phthalate modified with ester gum

Cellulose phthalate half ester, prepared as mentioned earlier, was reacted with ester gum prepared separately with an excess of glycerol (30 per cent on the weight of rosin). The final product was then dissolved in the following combination of solvents to 50 per cent solid content.

Xylene	60 partsby wt.
Acetone	15 parts by wt.
Butanol	20 parts by wt.
Ethyl acetate	5 parts by wt.

The solution of cellulose phthalate modified with ester gum was of brownish red in colour having good clarity.

Cellulose phthalate modified with pentaester gum

30 g of pentaester gum prepared separately and 50 g of the cellulose phthalate half ester were heated together at 150°C for 3 hours. Finally the material became viscous and clear which was thinned with the following solvents to 50 per cent solid content.

Xylene	35 parts by wt.
Toluene	20 parts by wt.
Acetone	10 parts by wt.
Butanol	25 parts by wt.
Ethyl acetate	10 parts by wt.

The above product was light brown in colour and had good clarity.

Determination of degree of substitution

Ref. 5

In order to determine the degree of substitution in the modified cellulose samples, free hydroxyl groups in the cellulose were determined by the method used by Bryant *et al*[5] by acetylating the samples with mixture of acetic anhydride and pyridine followed by titration with standard alkali. The drop in hydroxyl value of cellulose after modification indicated the degree of substitution.

Since the free carboxylic group present in the cellulose esters prepared would also combine with alkali, it was

Table 1

Particulars of cellulose derivatives

Sample No.	Cellulose derivatives prepared	Colour, Lovibond, 50% solution 1 cm cell	Clarity	Viscosity of 50% solution by Ford cup No. 4 at 30°C (in seconds)
A	Cellulose adipate	22.3 Y+11.9 R	Slightly hazy	115.0
B	Cellulose maleinate	0.4 Y+0.2 R	Clear	98.0
C	Cellulose phthalate	4.3 Y+2.2 R	Clear	108.0
D	Cellulose glycol phthalate	2.5 Y+0.5 R	Clear	128.7
E	Cellulose glyceryl phthalate	6.8 Y+1.6 R	Clear	97.5
F	Cellulose glyceryl phthalate modified with rosin	18.0 Y+2.8 R	Clear	123.7
G	Cellulose glycol phthalate modified with rosin	15.2 Y+2.3 R	Slightly hazy	82.4
H	Cellulose phthalate modified with ester gum	18.0 Y+2.6 R	Clear	92.0
I	Cellulose phthalate modified with pentaester gum	17.6 Y+1.2 R	Clear	93.0
J	Cellulose acetate	0.7 Y+0.4 R	Clear	112.0
K	Cellulose nitrate	0.9 Y+0.5 R	Clear	136.2

necessary to subtract the acid value of these samples from the observed hydroxyl value to obtain true hydroxyl value.

The true hydroxyl value of six standard samples of known degree of substitution were first determined and their relationship was plotted as shown in Fig. 1. The degree of substitution of the samples prepared was obtained from Fig. 1 after determining the true hydroxyl value as described above.

Results and discussions

All the samples prepared as above were studied for colour, clarity and viscosity. These results are given in Table 1. The colour of 50 per cent solution of all derivatives as determined by Lovibond tintometer using 1 cm cell showed that cellulose esters, namely, cellulose maleinate, cellulose phthalate and cellulose acetate were light in colour, except for the cellulose adipate. The samples containing rosin had large units of yellow and red colours. Solutions of almost all the samples were clear.

The samples were also evaluated for drying characteristics, flexibility, scratch hardness and resistance to water, acids, alkali and solvents.

Drying characteristics

The varnishes of cellulose derivatives were applied on glass and tin panels and baked under the following baking schedules:

(a) 100°C for 40 minutes, 50 min., and 60 min.
(b) 120°C for 30 minutes, 40 min., and 50 min.
(c) 140°C for 20 minutes, 30 min., and 40 min.
(d) 150°C for 20 minutes, 25 min., and 30 min.
(e) 160°C for 15 minutes, 20 min., and 25 min.

From the above baking schedules, it was found that the film of all cellulose derivatives dry at 100°C in 50 minutes, at 120°C in 40 minutes, at 140°C in 30 minutes, and at 150°C in 25 minutes without showing any appreciable tackiness or change in colour. Darkening of the films was observed when baked at 160°C for 15 minutes or longer. This could be due to heat degradation and charring of the cotton linters. Therefore, the maximum temperature for baking was taken as 150°C, although a slight change in colour was observed.

Flexibility

Two per cent by weight of dibutyl phthalate plasticiser was thoroughly mixed with all the samples before performing this test. Flexibility of the dried film was tested on tin panels with ¼″ mandrel. Samples baked at 100°C, 120°C and 140°C showed good flexibility (Table 2). Cellulose-alcohol-phthalates and their rosin derivatives baked at 150°C also passed the flexibility test, but other samples baked at 150°C failed. The possible explanation for this behaviour is that at higher baking temperature some cleavage of cellulose chain could have occurred leading to embrittlement of the film and loss in flexibility in some of the samples.

Scratch Hardness

Table 2 also includes the results of scratch hardness test. The scratch hardness (at a load of 1 kg) of cellulose derivatives showed good results when baked at 120°C, 140°C and 150°C. Samples of cellulose glycol phthalate, cellulose glycerol phthalate and cellulose phthalate modified with ester gum baked at 150°C failed the scratch hardness test, possibly due to the degradation of cellulose chain at higher baking temperatures. Samples baked at 100°C did not show good scratch hardness, except those of cellulose phthalate, cellulose maleinate, and cellulose phthalate modified with pentaester gum which passed the scratch hardness test. The failure in scratch hardness at lower baking temperature for most samples could have been due to improper drying which would leave a softer film after drying.

It is interesting to note that the cellulose derivatives containing rosin showed satisfactory results with respect to scratch hardness because the large number of cyclic rings present in these cellulose derivatives contribute towards the hardness.

Alkali resistance

Table 3 shows the alkali resistance of various cellulose derivatives baked at different baking schedules. Two solutions of 10 and 20 per cent sodium hydroxide were taken. Panels were immersed in the solutions for 8 days. It was observed after 8 days' test that the samples of cellulose maleinate, cellulose phthalate, cellulose glycol phthalate, and cellulose glyceryl phthalate had good resistance to alkali when baked at 120°C for 40 minutes and 140°C for 30 minutes. Some of

Table 2
Mechanical properties of cellulose derivatives

Sample No.	Flexibility test (¼″ Mandrel) with 2% Dibutyl Phthalate, when baked at				Scratch hardness test (at 1 kg load) when baked at			
	100°C for 50 min.	120°C for 40 min.	140°C for 30 min.	150°C for 25 min.	100°C for 50 min.	120°C for 40 min.	140°C for 30 min.	150°C for 25 min.
A	P	P	P	F	F	P	P	P
B	P	P	P	F	P	P	P	P
C	P	P	P	F	P	P	P	P
D	P	P	P	P	F	P	P	F
E	P	P	P	P	F	P	P	F
F	P	P	P	P	F	P	P	P
G	P	P	P	P	F	P	P	P
H	P	P	P	F	F	P	P	F
I	P	P	P	F	P	P	P	P
J	P	F	F	F	F	P	P	P
K	P	P	F	F	P	P	P	P

P = Passed; F = Failed.

Table 3
Alkali resistance of cellulose derivatives at 33°C for 8 days

Sample No.	Resistance to 10% NaOH solution when baked at				Resistance to 20% NaOH solution when baked at			
	100°C for 50 min.	120°C for 40 min.	140°C for 30 min.	150°C for 25 min.	100°C for 50 min.	120°C for 40 min.	140°C for 30 min.	150°C for 25 min.
A	1	2	2	1	0	0	0	0
B	5	5	5	5	5	5	5	4
C	5	5	5	5	1	3	1	0
D	4	5	5	5	2	5	5	4
E	4	5	5	4	2	5	5	4
F	2	2	4	3	0	1	3	1
G	1	2	3	2	0	1	1	1
H	1	2	3	3	0	1	1	0
I	3	3	3	2	1	3	3	1
J	1	2	0	0	0	0	0	0
K	5	5	5	5	1	3	3	3

5 = unaffected
4 = slight loss in gloss
3 = loss in gloss
2 = Film partially cracked
1 = Film cracked and partially removed
0 = Film removed

these samples behaved better than cellulose nitrate in this respect. Above and below these baking schedules the cellulose derivatives showed poor resistance, especially the cellulose esters modified with rosin, ester gum, and pentaester gum.

Most of the samples baked at 100°C for 50 minutes and 150°C for 25 minutes had poor alkali resistance. This could again be due to improper drying of the film at 100°C and degradation of the cellulose at 150°C. The cellulose maleinate, cellulose glycol phthalate and cellulose glyceryl phthalates had excellent alkali resistance. These samples were not affected even by a 20 per cent solution of the sodium hydroxide. Cellulose phthalate also had good alkali resistance. Cellulose phthalate modified with ester gum and pentaester gum or cellulose glycol and glyceryl phthalates modified with rosin had inferior alkali resistance in comparison to cellulose maleinate and cellulose phthalate when immersed in 20 per cent sodium hydroxide solution, although these products showed satisfactory resistance to 10 per cent sodium hydroxide solution, particularly cellulose phthalate modified with pentaester gum and cellulose glyceryl phthalate modified with rosin.

Acid resistance

Table 4 gives a clear picture of the acid resistance of all the samples. Acid resistance of cellulose phthalate and cellulose maleinate was as good as that of cellulose nitrate, but the cellulose adipate showed poor resistance to acids.

The cellulose derivatives modified with rosin showed poor resistance to hydrochloric, nitric and sulphuric acids and the films were completely or partially removed by them.

Almost all the cellulose derivatives showed better results when baked at 120°C for 40 minutes or 140°C for 30 minutes. Baking schedule of 100°C for 50 minutes for most of the samples resulted in poor resistance to acids, whilst the higher baking temperature gave slightly improved results. This could be due to the fact, as already mentioned, that at 100°C for 50 minutes the films do not seem to be sufficiently hard and tough.

Water resistance

Water resistance of various cellulose derivatives are sum-

Table 4
Resistance of film to 20% acid solutions at 33°C after 8 days

Sample No.	Resistance of film when baked at 100°C for 50 min. to			Resistance of film when baked at 120°C for 40 min. to			Resistance of film when baked at 140°C for 30 min. to			Resistance of film when baked at 150°C for 25 min. to		
	HCl	H_2SO_4	HNO_3	HCl	H_2SO_4	HNO_3	HCl	H_2SO_4	HNO_3	HCl	H_2SO_4	HNO_3
A	0	0	0	0	1	1	0	1	1	0	0	0
B	2	2	5	5	5	5	5	5	5	5	5	4
C	2	0	5	5	3	5	5	3	5	2	2	5
D	0	0	1	1	1	1	3	1	1	1	1	1
E	1	1	1	3	3	1	3	3	1	1	1	1
F	0	0	0	1	1	1	1	1	1	0	0	1
G	0	0	0	1	1	0	1	0	1	0	0	1
H	0	0	0	1	0	0	1	1	0	1	0	0
I	1	0	0	3	1	1	3	1	1	1	1	0
J	0	0	0	0	0	0	0	0	0	0	0	0
K	5	5	0	5	5	1	5	4	3	5	2	1

5 = Unaffected
4 = Slight loss in gloss
3 = Loss in gloss and colour change
2 = Film partially cracked
1 = Film cracked and partially removed
0 = Film removed

marised in Table 5. From the results obtained it can be concluded that the water resistance at 30°C of all the samples was good, even of those modified with rosin. However, when immersed in boiling water for 24 hours, the films of cellulose glycol phthalate and glyceryl phthalate were cracked and partially removed. Though cellulose maleinate and phthalate showed excellent water resistance, slight loss in gloss was observed when immersed in boiling water and thus these samples behaved similarly to cellulose nitrate. The films of other cellulose derivatives were partially or completely removed by boiling water.

Solvent resistance

The resistance of cellulose derivatives to toluene/xylene (50/50), white spirit and petrol have been summarised in Table 6. From the results obtained it has been found that the films of cellulose adipate and cellulose esters modified with rosin were completely removed by the solvents. This could be due to the presence of a linear chain in case of the adipate and monobasic nature of rosin, in case of rosin based samples, where no crosslinking could take place. However, the dibasic acid esters of cellulose were not affected by these solvents due to their crosslinked nature and compactness, except that loss in gloss was observed in the films. Thus these products are comparable with cellulose nitrate in this respect.

Conclusions

From the results obtained, it can be concluded that amongst all the cellulose derivatives prepared, the cellulose maleinate and cellulose phthalate showed excellent physical and chemical properties. The colour retention on baking was also better as compared to other cellulose derivatives. The cellulose derivatives modified with rosin, had poor film properties. However, an improvement in results was observed in the case of pentaester gum modified cellulose phthalate.

Better physical and chemical properties were obtained when the films were baked at 120°C for 40 minutes or at 140°C for

Table 5
Resistance of film to water

	Resistance to water at 30°C for 8 days, when baked at				Resistance to boiling water for 24 hours, when baked at			
Sample No.	100°C for 50 min.	120°C for 40 min.	140°C for 30 min.	150°C for 25 min.	100°C for 50 min.	120°C for 40 min.	140°C for 30 min.	150°C for 25 min.
A	2	3	3	3	3	3	3	3
B	5	5	5	5	3	4	4	5
C	5	5	5	5	3	4	5	5
D	3	4	4	3	1	1	1	1
E	5	5	5	5	1	1	1	1
F	3	4	3	3	0	0	0	0
G	3	3	3	3	0	0	0	0
H	3	4	4	4	0	1	1	1
I	4	5	5	4	1	1	1	1
J	5	4	4	3	0	0	0	0
K	5	5	5	5	4	4	5	5

5 = Unaffected
4 = Slight loss in gloss
3 = Loss in gloss
2 = Film partially cracked
1 = Film cracked and partially removed
0 = Film removed

Table 6
Resistance of the film to solvents at 30°C for 5 days

	Resistance of film baked at 100°C for 50 min. to			Resistance of film baked at 120°C for 40 min. to			Resistance of film baked at 140°C for 30 min. to			Resistance of film baked at 150°C for 25 min. to		
Sample No.	Toluene/ Xylene	White spirit	Petrol	Toluene/ Xylene	White spirit	Petrol	Toluene/ Xylene	White spirit	Petrol	Toluene/ Xylene	White spirit	Petrol
A	0	1	0	0	1	0	1	1	0	1	1	0
B	3	5	3	4	5	4	4	5	4	4	5	4
C	3	5	3	4	5	4	4	5	4	3	5	4
D	3	3	0	0	4	1	1	4	1	1	4	1
E	1	4	0	1	4	1	1	4	1	1	4	1
F	0	1	0	0	1	0	0	1	0	0	1	0
G	0	0	0	0	0	0	0	1	0	0	1	0
H	0	1	0	1	1	0	1	1	1	1	1	1
I	1	1	0	1	1	1	1	1	1	1	1	1
J	1	1	1	1	3	1	3	3	1	3	3	1
K	5	5	3	5	5	5	5	5	5	5	5	5

5 = Unaffected
4 = Slight loss in gloss
3 = Loss in gloss
2 = Swelling of the film
1 = Film partially removed
0 = Film completely dissolved

30 minutes. Above and below this range of baking temperatures, the film properties varied quite significantly from sample to sample.

Hence, it can be concluded that cellulose maleinate and cellulose phthalate have nearly the same properties as those of the versatile cellulose nitrate with an added advantage of low inflammability.

On this ground it can be suggested that cellulose maleinate and cellulose phthalate could be used for refrigerator and domestic appliance finishes. On blending with epoxy resins or silicone resins, it can safely be recommended for washing machines. Because of the colour retention properties of cellulose maleinate and cellulose phthalate, a white finish based on these resins can also be suggested for refrigerators, electric fans and other articles where the brilliant whiteness is necessary for sales appeal. Cellulose esters modified with rosin and rosin esters can be made compatible with conventional film formers such as alkyds, phenolics and epoxy esters and the combinations could be used in various surface coating formulations.

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References

1. Sweeting, O. J., 'The Science and Technology of Polymer Films', *Wiley Interscience Publishers*, N.Y.; Vol. 2, 1972, p. 414.
2. Mark H. F., and Gaylord, N. G., 'Encyclopaedia of Polymer Science and Technology', *Interscience Publishers*, N.Y., Vol. 3, 1965, p. 242.
3. Spurlin, H. M., and Graffin, M. W., 'Cellulose and cellulose derivatives', *Interscience Publishers*, N.Y.; Part-I, 1954, p. 147 and Part-III, 1955, pp. 169, 223, 504.
4. Malm, C. J., and Fordyce, C. R., *Ind. Eng. Chem.*, 1940, **32**, 405.
5. Bryant, W. M. D., Mitchell, J., and Smith, D. M., *J. Am. Chem. Soc.*, 1940, **62**, 608.

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[illegible]

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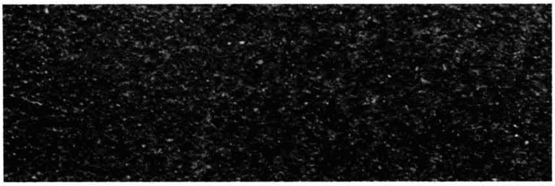

Bare panel weathered 6 months – rough de-rusting before primer

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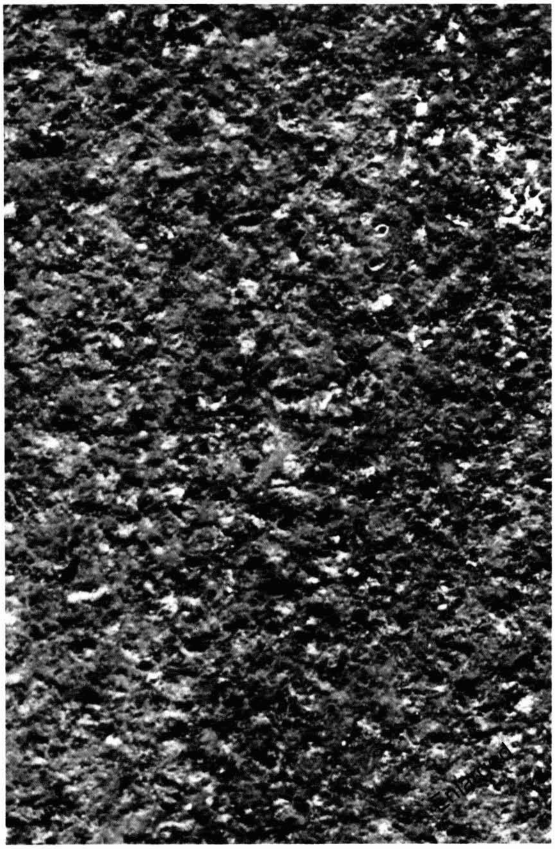

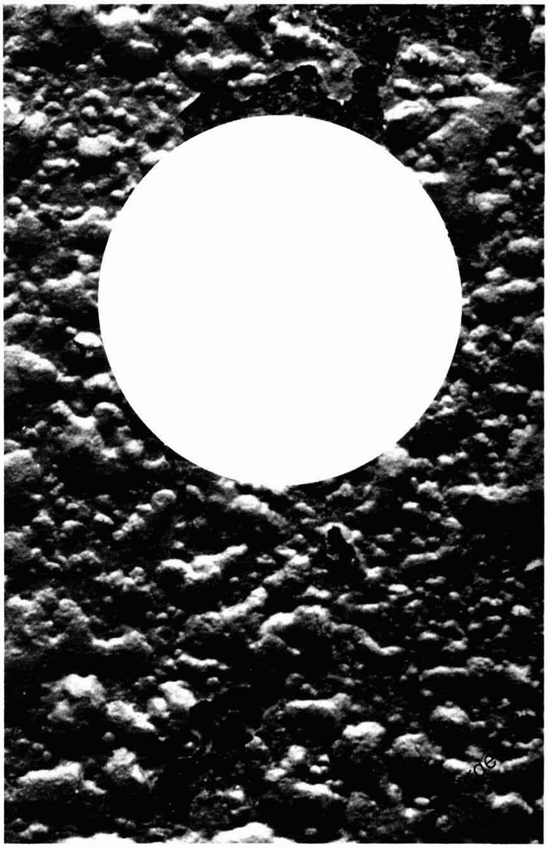

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1.00 ppw	BENTONE® 34 / TEXAPHOR® 277 gel, 10 % in xylene
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Ship's trials of oleoresinous antifouling paints. Part I: Formulations with high and medium toxicant contents

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Summary

Ship's trials were performed using oleoresinous antifouling paints of the soluble matrix type. The toxicant is cuprous oxide reinforced in some of the samples with mercurous arsenate and zinc oxide. Whiting was employed as extender on account of its previously established influence on the antifouling performance in raft tests.

The binder was formulated with Rosin WW plasticised with a phenolic varnish. Two binders of different solubilities were obtained.

Three vessels in service under different conditions (a destroyer, a cruiser and a tug boat) were used. Patches of 3.5 × 3.5 metres were applied on each hull. Observations of the ship's bottoms were made 12 and 15 months later.

In all the cases the fouling settlement varied from none to very rare. There are practically no differences in the bioactivity between the paints at the maximum and minimum toxicant levels.

Keywords

Types and classes of coatings and allied products

- antifouling coating
- ship bottom paint

Raw materials

prime pigments and dyes

- copper oxide

binders (resins, etc.)

- rosin
- phenolic

biologically active ingredients

- copper salt
- mercurous arsenate

extender pigments

- calcium carbonate
- whiting

Types and classes of structures or surfaces to be coated

- steel

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

- sea water immersion test

Les essais, effectués sur navires, des peintures "anti-fouling" à base de vernis gras. 1ère Partie: Peintures à haute et à moyenne teneur en agents toxiques

Résumé

On a effectué des essais sur navires avec les peintures "anti-fouling" à base de vernis gras et dont la matrice était soluble. L'agent toxique était l'oxyde cuivreux, augmenté en certains cas par l'arséniate mercureux et l'oxyde de zinc. A cause de son influence sur le rendement des peintures "anti-fouling", qui a été constatée antérieurement au cours des essais sur radeaux, on a utilisé le blanc d'Espagne en tant que matière de charge.

Le liant était composé de colophane WW plastifiée par un vernis phénolique. On a obtenu deux liants ayant de différentes solubilités.

Pour les essais on s'est servi de trois bâtiments, dont les conditions de service étaient différentes (un destroyer, un croiseur et un remorqueur). A chaque coque la peinture a été appliquée sur des zones de 3,5 × 3,5 metres. On a noté la condition des coques après 12 et 15 mois. Dans tous les cas la croissance des organismes nuisibles se varie entre nul et très peu. Dans la pratique, il n'existe pas de différence entre la bio-activité des peintures à teneur maximale et a teneur minimale en agent toxique.

Schiffsversuche mit Antifoulingfarben auf Ölbasis. Teil I: Rezepturen mit hohen und mittleren Giftgehalten

Zusammenfassung

An Schiffsböden wurden Versuche mit Antifoulingfarben auf Ölbasis vom löslichen Matrixtyp durchgeführt. In manchen der Probeu war das Giftmittel Kupferoxidul verstärkt durch Merkuroarsenat und Zinkoxid. Als Extender wurde Schlemmkreide benutzt, weil diese in früheren Raftversuchen ihre Wirkung hinsichtlich des Antifoulingverhaltens erwiesen hatte.

Das Bindemittel bestand aus Harz WW, weichgemacht mit Phenolharzlack. Zwei Bindemittel verschiedener Löslichkeit wurden hergestellt.

Drei im Dienst befindliche Schiffe wurden für die Versuche benutzt (ein Zerstörer, ein Kreuzer und ein Schleppboot). Auf jeder Hülle wurden Felder von 3.5 × 3.5 m gestrichen. 12 und 15 Monate später wurden die Schiffsböden geprüft.

In allen Fällen waren die Unterschied im Anwuchs sehr gering: von gar keinem bis zu sehr seltenem Anwuchs. Zwischen den maximalen und minimalen Giftniveaux der Farben waren praktisch keine Unterschiede hinsichtlich der Bioaktivität feststellbar.

Introduction

Refs. 1-11

In order to keep a ship's bottom free from fouling during a predetermined period of time, it is essential to use antifouling paints. No other method has so far been found to be effective from both the technical and economical points of view.

The above mentioned paints prevent the fouling attachment. This property is especially important in the case of those species which degrade the paint film and cause discontinuities in the protective system (sea water comes into contact with the metal producing corrosion), as well as with bulky organisms which oppose resistance to the displacement of the ship and increase the friction of water on the hull.

At a previous stage of this work the different variables involved in the behaviour of antifouling formulations have been studied on experimental rafts[1-10]. The present tests correspond to tests made on a pilot plant scale, as well as to the results obtained from experiments performed on hulls of ships of the Argentine Navy[11]. New processes and operations have been introduced for the purpose of making an exhaustive study of the formulation parameters, method of application and influence of the different operating conditions of the ships.

The mode of action of the paints studied is based on the dissolution of toxicants and binder in sea water. The control of the rate of solubility of Rosin WW is made by using an adequate plasticiser (phenolic varnish). Toxicants usually dissolve faster than the binder, and so the solubility of the matrix determines the useful life of the paint.

Rosin WW contains about 85 per cent of resinic acids (of which the abietic acid is the most important). Free carboxylic groups present give this resin an acidic character and solubility in alkaline solutions. In contact with sea water (pH 8.2) it forms soluble sodium and potassium resinates.

Service tests, whilst justified because the experimental conditions are more adequate than those existing on a raft, do not allow a rapid diagnosis of the behaviour of the paints, since periods of not less than 12 months are necessary.

Such studies also present the disadvantage that the different experimental circumstances vary from one ship to another: operating conditions, velocity and itineraries, exposure in harbours with different fouling conditions, difficult control of the uniformity of application, quality of workmanship, different ambient conditions, etc.

As a secondary objection, in this case the observations of the painted patches can be made only while ships are dry-docked since the use of self-contained diving equipment and underwater photography or television cameras are not available at present.

Variables studied

Ref. 7

The variables studied on a pilot plant scale correspond to two different lines of research:

Variables which involve formulation parameters

(a) *Influence of the type and content of toxicant*

Cuprous oxide was used as main toxicant because of its effective action against most of the fouling organisms and its relatively low price in relation to other toxicants. Mercurous arsenate, which provides a greater protection against Cirripedia and algae, and zinc oxide, were used as reinforcing toxicants in some of the formulations.

(b) *Influence of the extender contents*

Calcium carbonate was used as extender in order to obtain a reduction in the cost of formulation of the antifouling paints. Whiting does not reduce the bioactivity of the formulations[7] and by hydrolysis in sea water it influences the pH of the paint film/sea water interface and allows a better control of fouling.

Owing to its low specific weight, calcium carbonate also gives less sedimentation and improves paint stability.

(c) *Influence of the chemical composition and solubility of the matrix*

In soluble binder paints the toxicant originally exposed at the surface of the film determines the initial leaching rate of the paint. The solubilisation of the matrix allows the establishment of the necessary steady state leaching rate. The solubility of the binder is regulated using an adequate plasticiser which also improves flexibility and adhesion of the film to the substrate.

Two different solubilities of matrix were tested in this work; Rosin/varnish 3/1 and 5/1 were used. The greater film solubility corresponds to the latter ratio.

Variables related to the painting operations

(a) *Number of coats, film thickness and areas of application*

Two coats were applied by roller under normal conditions of dry-dock of Puerto Belgrano and patches with different film thickness were obtained. Because the different hydrodynamical conditions may influence the leaching rate of the paints, the patches were placed on one side of the vessel from midships to stern and on the other side from midships to bow. The paints were applied in reverse order on each side (Figures 1, 2 and 3).

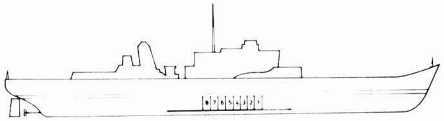

Fig. 1. Arrangement of the painted panels on the starboard side of the destroyer

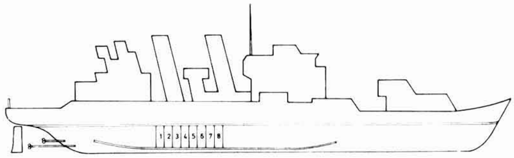

Fig. 2. Arrangement of the painted panels on the starboard side of the cruiser

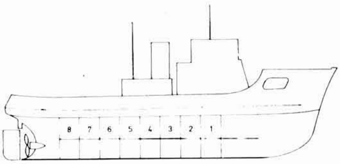

Fig. 3. Arrangement of the painted panels on the starboard side of the tugboat

(b) *Ship's operating conditions*

Three ships with different operating characteristics were selected for the present series of tests:

A *destroyer*, which is at sea for rather long periods throughout the year; for this reason the experimental conditions are markedly different from those of the raft trials. The solubilisation of the toxicant increases and the paint film becomes exhausted more rapidly.

A *cruiser*, for which operating conditions are characterised by shorter periods of sailing and longer periods at port. Thus, the aggressive action of fouling could increase, but the solubilising action due to the water flow over the hull decreases. For that reason the useful life of the paint may be longer.

A *tugboat* which operates for long periods, mainly within the port and at low speed. This would most resemble the conditions of the raft tests.

Antifouling paints formulation

The composition of the paints tested are shown in Table 1.

Paints 1 and 3 contain only cuprous oxide as toxicant. In paints 2 and 4, 10 per cent of the toxic substance was replaced by zinc oxide. In paints 3 and 4 calcium carbonate was used as extender. The binder employed for these formulations has the lower solubility.

In samples 5, 6, 7 and 8 the same formulation criteria were applied, but mercurous arsenate was used as a reinforcing toxicant. In this case samples 7 and 8 contain whiting. The binder solubility for this series is higher than in the above series.

Various additives were used in order to obtain rapid drying of the film and low gloss of the painted surface.

Antifouling paints: preparation and application

Refs. 12-16

All the samples were first prepared on a laboratory scale (1 litre) and once the final formulation was established, the work continued on the pilot plant scale.

For this purpose a discontinuous stirred reactor was designed and built[12] and its characteristic parameters were determined.

The next step was to prepare the different components of the binder (the linseed standoil 60 poises, the phenolic varnish and the solution of Rosin WW in the solvents)[13-16].

Finally, the antifouling paints were prepared in ball mills using porcelain jars of 26 litres capacity.

The painted panels each have an area of about 12 m^2, requiring 6 litres of paint per panel. The antifouling paints were applied over the ordinary anticorrosion system used by the Navy. An average thickness of 80-100 μm was obtained for the different patches.

The two coats were applied at 24 hour intervals and the vessel was launched 24 hours after the application of the last coat.

The tests were started in December, 1975, for the destroyer, in June, 1976, for the tugboat and in August, 1976, for the cruiser.

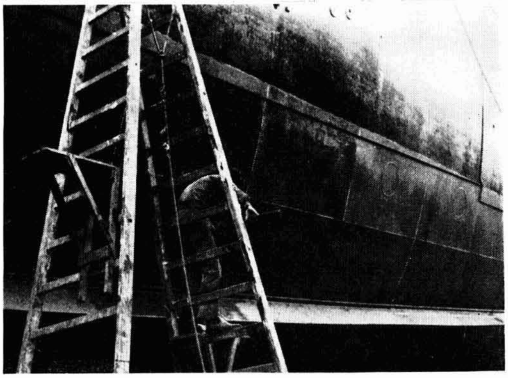

Fig. 4. View of the experimental paints being applied

Results and discussion

Refs. 17-26

The purpose of the research is to develop highly efficient antifouling paints, having a long useful life which will allow the bottom surfaces of either warships or merchant vessels to remain completely free from fouling organisms for long periods.

The quality of the antifouling formulation should be commensurate with the longer drying periods and ensure that premature dry-docking is not necessitated by fouling[17].

The cost of such paints should be as low as possible. The cost is a function of the amount and type of toxicants incorporated in the formulation, but it is also necessary to assure adequate solubilisation.

Table 1

Composition of the antifouling paints tested (per cent in weight)

	Paint							
	1	2	3	4	5	6	7	8
Toxicants and extenders	50.6	50.6	50.6	50.6	54.9	54.7	54.9	54.7
Binder (solids)	22.9	22.9	22.9	22.9	24.1	24.1	24.1	24.1
Additives	3,6	3.6	3.6	3.6	0.7	1.1	0.7	1.1
Solvents	22.9	22.9	22.9	22.9	20.3	20.1	20.3	20.1

Formulations based on cuprous oxide have been developed because the objectives mentioned above can be attained with this toxicant and also because it has a relatively low cost and is manufactured in this country using up-to-date technology giving a high quality product.

The mercurous arsenate which was used in some of the samples has been prepared in our laboratory by a technique which was described in the existing literature references[18]. The use of mercurous arsenate in small quantities gives, as already stated, a significant increase in the bioactivity of these paints, mainly against spores of certain vegetable organisms such as the case of the algae *Enteromorpha intestinalis* or *Ectocarpus* which appear to be particularly resistant to the toxic action of the copper compounds.

Other properties of the paints studied are as follows: short drying time, but consistent with the roller-painting technique used; easy workability during the application; compatibility with the anticorrosive primers usually employed in underwater areas; compability with the environmental conditions existing in the dock during the paint operations; and finally, adequate stability in the container.

The mode of action of paints based on cuprous oxide involves the dissolution of the toxicant[19,20] with formation of a complex copper chloride. The leaching rate is controlled by the diffusion of this substance through the matrix to the water layer adjacent to the paint film.

With paints of the soluble matrix type, such as in the case of the samples tested, it is necessary to obtain a leaching rate not lower than 10 $\mu g/cm^2/day$ for copper, but a higher rate (20 $\mu g/cm^2/day$) is needed to prevent the growth of algae. That leaching rate does not ensure surfaces completely free from bacteria and diatoms which form a slimy film. These species do not impair the operational efficiency of the ships and are largely eliminated during navigation.

The provision of an adequate leaching rate depends, in practice, not only on intrinsic aspects of the paint composition, but also on the conditions of the water flow over the painted surface. The dissolution of copper is maximum at sea and minimum in port, where the ship is stationary.

The greatest part of the toxicant consequently is leached without fulfilling its specific function, since at a speed of over 4 or 5 knots there is practically no attachment. In spite of this it is possible to obtain oleoresinous paints of good quality assuring in practice a good bioactivity for a period of at least two years.

This first series of experiments concerns those formulations with the higher toxicant contents (from 50 to 30 per cent), but in other tests started subsequently, lower toxicant levels are being studied.

The economics of antifouling control involves not only the paint cost per litre, but also other related factors. It is essential for the Navy to keep ships' bottoms clean and free from fouling, both from the operational and tactical points of view. It is very important for merchant ships to make dry-docking less frequent. In either case, an efficient antifouling protection minimises the possibilities of hull corrosion.

For the above reasons, there should be a change in the criteria of the shipowners especially in the case of the construction and painting of new vessels. Within the overall cost involved when a ship is dry-docked, the price of the antifouling paint has little significance if the paint is highly efficient. Most important are the cost of the dry-dock rental, the surface cleaning operations and eventual repairs due to corrosion of steel surfaces, standard of workmanship, problems arising from the ship being taken out of service, etc.

The above considerations are complemented by the fact that a ship's bottom without fouling represents a very important economy in fuel consumption. This largely covers, over a period ranging from 12 to 24 months, the higher cost of the paints.

Considering that the research plan includes experiments on 10 to 12 vessels, at least, and that only after a period of three years will it be possible to obtain final conclusions, only the results obtained to date on the three vessels previously mentioned will be considered in this paper.

Whilst the experiments have had about the same duration in the cases considered (15 months on the destroyer and the tugboat and 12 months on the cruiser) it must be pointed out that they have been started at different seasons and consequently under different fouling conditions.

The most important results relate to the test on the destroyer, carried out between December, 1975 and March, 1977. In this case the paints have been exposed to two fouling periods of high aggressiveness with a duration of four to five months each, one at the beginning and the other at the end of the test. An idea of the conditions of the experimental medium can be obtained from the species encountered, established in a previous study by Bastida[21-23] and shown in Table 2.

Table 2

Most important fouling species registered[21-23] in Puerto Belgrano, 1971/72

Algae	Diatoms Chlorophyceae (*Enteromorpha intestinalis*) Rhodophyceae (*Porphyra* sp.)
Protozoa	
Coelenterata	*Tubularia* sp.
Annelida	*Halosydnella australis* *Syllis prolixa* Serpulidae juv.
Cirripedia	*Balanus amphitrite* *Balanus* sp.
Amphipoda	*Corophinum* sp. *Caprella penantis* *Caprella equilibra*
Brachyura	*Cyrtograpsus angulatas* *Cyrtograpsus altimanus*
Bryozoa	*Bugula neritina* *Bugula* sp.
Tunicata	*Ciona intestinalis* *Botryllus schlosseri*

Both on the cruiser and the tugboat, as the dry-docking was performed during winter, the paints experienced only one period of intense fouling during the summer at Belgrano Harbour. In spite of this, no significant differences in antifouling behaviour have been observed which may be attributed to the different experimental conditions.

The test on the destroyer was considered complete after 15 months.

As the experiments on the cruiser and the tugboat will continue until the regular drydocking of these vessels according to the Navy's dry-docking schedules, there exists the possibility of obtaining highly valuable complementary information.

Fouling settlement values on the scale 0 to 5 are shown in Table 3 (see the key of the table). Considering the whole of the tested patches, only in the case of the paints 5 and 6, with settlement 1-2 on starboard panels, is value 1 exceeded. This value is the maximum experienced in the tests. These same paints, however, show settlement 0 and 0-1, respectively, on the port-side of the vessel.

In all the other cases the fouling registered varies between 0, 0-1 and 1, but settlement always appears on isolated areas, welds, plate joints, etc., no wide-spread paint failure being observed in any case.

It is known that in soluble matrix antifouling paints, whilst the toxicant and matrix solubilise simultaneously, as already stated, the residual skeleton of the matrix as well as products from reaction of the toxicants or the binder components with the sea water remain adherent and they may block areas or points of the film, thus allowing fouling fixation.

Consequently, up-to-date underwater cleaning methods tend to eliminate this inert surface by renewing the activity of the underlying film.

It is important to point out that no case of Cirripedia settlement has been observed, the little existing fouling of some of the patches being limited to protozoa, diatoms, algae and bryozoa with a very poor development.

The results obtained on the tugboat are particularly significant. This ship has been permanently in the experimental area for 15 months, either anchored or sailing, and all the paints applied show settlement 0 (without fouling), with the exception only of sample 2, on the port-side, with settlement 0-1.

When the influence of the different variables considered in the experiments is analysed, the following observations can be made:

(a) *Composition variables*

Similar results have been obtained both in the case of paints with a high toxicant content (50 per cent) and those with a medium content (30 per cent). The use of either mercurous arsenate or zinc oxide as reinforcing toxicants does not seem to be necessary for the test periods mentioned nor for the operating conditions of the ships involved. Only in the case of the destroyer, with paints 5 to 8, port-side patches, was a slightly lesser settlement seen in the paints containing arsenate. It should be possible to corroborate this by the new observations to be made on the cruiser and on the tugboat after 18 to 24 months of immersion.

Regarding the use of calcium carbonate (whiting) to replace up to 40 per cent of the toxic material, it does not seem to produce any decrease in the bioactivity of the paints tested, corroborating the results obtained in previous raft trials.

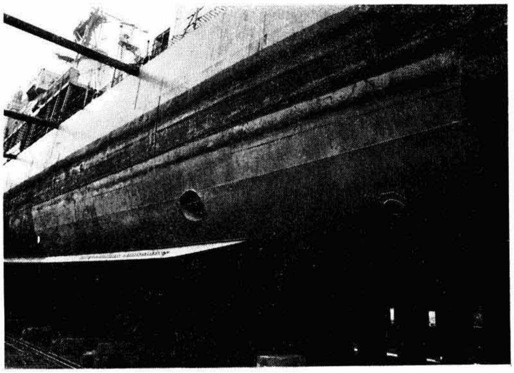

Fig. 5. General view of the patches on the port side of the destroyer after 15 months of immersion

The two solubilities of binder used provided paints equally effective. Further observations will be necessary after longer periods than 12 to 15 months to verify whether this variable influences results in a similar way to that observed in the raft; where paints formulated only with cuprous oxide required binders of less solubility than those in which mercurous arsenate was used as a reinforcing toxicant.

The only paint which shows settlement 0 in all the experiments is sample 8. This formulation contains cuprous oxide reinforced with mercurous arsenate and is the one with the highest amount of whiting (Fig. 6).

(b) *Location of the patches on the ships*

As already stated, the painted panels were located in different order and position on the ships. In many cases that location

Table 3

*Fouling settlement in the different tests**

Paint	Destroyer		Cruiser		Tugboat	
	Port-side	Starboard	Port-side	Starboard	Port-side	Starboard
1	0–1	0–1	0–1	0–1	0	0
2	0	1	0–1	1	0–1	0
3	0–1	0–1	0	0–1	0	0
4	0–1	0–1	0	0–1	0	0
5	0	0–1	0	1–2	0	0
6	0–1	0–1	0–1	1–2	0	0
7	0	0–1	0	1	0	0
8	0	0	0	0	0	0

*Key: (0) without settlement; (1) rare; (2) common; (3) very common; (4) abundant; (5) completely fouled.

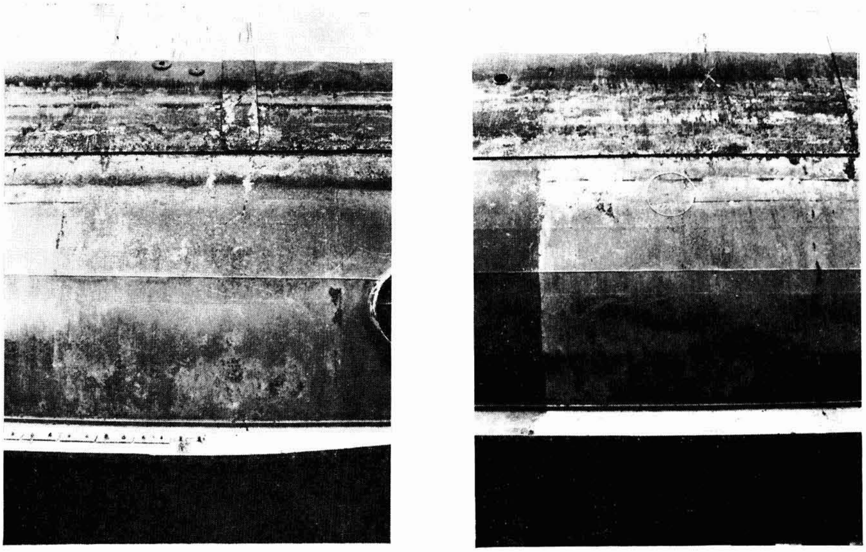

Fig. 6. Detail of the patches corresponding to paints 4 (*left*) and 8 (*right*), with the maximum content of whiting, after 15 months of immersion

depends on the existence of water discharges from scruppers. Paint work is often ruined locally by this cause.

In the destroyer, an area was chosen on the port-side from midships to stern and another on the starboard side from midships to bow, the order of paints being reversed in the latter. No significant difference due to this different order was observed.

On the cruiser the paints were applied on the port-side as well as on the starboard side from midships to stern, again reversing the order of paints in each series. Again, in this case there are no differences in behaviour.

Finally, in the case of the tugboat, because of its shorter length, the patches covered 85 per cent of the ship's underwater area. Paints 1 and 8, which correspond to the two extreme positions, always show settlement 0 both at the bow and at the stern and at the rudder.

On the destroyer and the cruiser, the results obtained on the starboard side for all the panels are slightly lower than those obtained on the port-side. The only difference in the experimental conditions was during the painting of such panels; because of the position of the dock, they are subject to the action of the sun, which could accelerate drying excessively.

It is known that antifouling paints require a short drying time (no longer than 24 to 48 hours) and rapid immersion; longer drying periods or excessive ageing in the air increase the solubility of rosin[24] and could, therefore, reduce the useful life of the paints.

(c) *Ships' operating conditions*

No significant differences in the behaviour of the various formulations were noticed on the ships chosen, whose operating characteristics are markedly different. Settlement is practically non-existent on the tugboat (0-1 on paint 2, and 0 in the other cases) and slightly greater on the destroyer (ranging from 0 to 0-1) and on the cruiser (from 0 to 1-2).

The results may be considered similar, since according to the most recent investigations on the subject[25,26] it has been demonstrated that the bottom's surface roughness introduces an important factor in the behaviour of antifouling paints, as it has a significant effect on the flow conditions of sea water, which, although generally non-turbulent, may develop turbulence zones which may favour solubilisation. Considering that all the cases deal with ships with many years of service, this variable should be well borne in mind when judging the behaviour of these types of paint, as it could have a very great influence on the results. The roughness of the surfaces used was not determined.

(d) *Stability in the container*

The stability in the container of the formulations studied was checked after 6, 12 and 24 months. It proved to be satisfactory, no separation of components or gelation being observed in any of the cases.

Acknowledgments

The authors acknowledge the assistance given and the very efficient coordination carried out by the Servicio Naval de Investigación y Desarrollo (SENID) as well as by the Head and Staff of the General Workshops Division and of the Laboratory of the Naval Base of Puerto Belgrano, which allowed the scheduled experiments and observations to be carried out.

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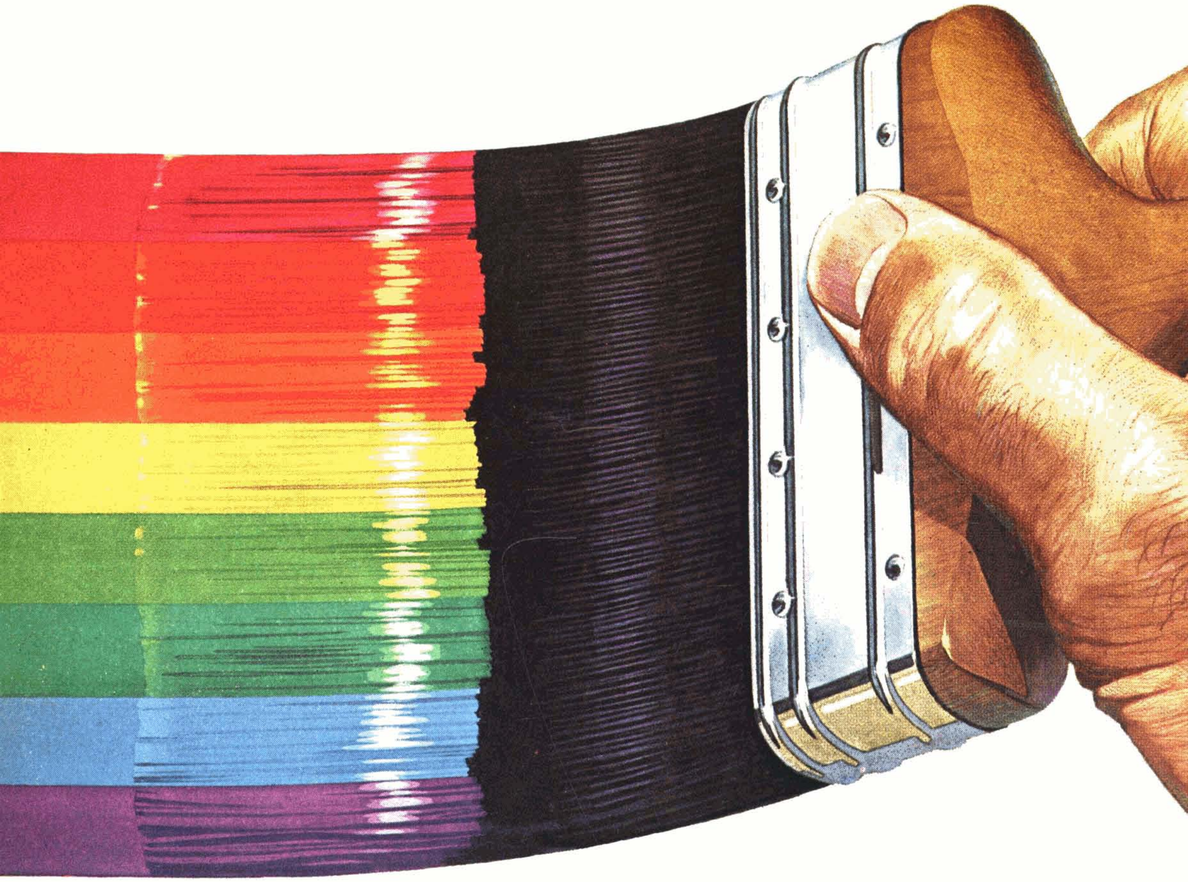
References

1. Rascio, V., and Caprari, J. J., *Industria y Química*, 1968, **26** (3).
2. Rascio, V., and Caprari, J. J., *Peintures, Pigments, Vernis*, 1969, **45**, 182.
3. Rascio, V., Caprari, J. J., and Bastida, R., *Peintures, Pigments, Vernis*, 1969, **45**, 724.
4. Rascio, V., and Caprari, J. J., *Corrosión y Protección*, 1970 **1**, 19



5. Rascio, V., and Caprari, J. J., *Proceedings del 1er. Congreso Nacional de Corrosión y Protección*, Madrid, 1970, p. 415.
6. Rascio, V., and Caprari, J. J., *Rev. Lat. Ing. Quím. y Química Apl.*, 1972, **2**, 117.
7. Rascio, V., and Caprari, J. J., *J.O.C.C.A.*, 1974, **57**, 407.
8. Rascio, V., and Caprari, J. J., *J.O.C.C.A.*, 1977, **60**, 161.
9. Bastida, R. O., Adabbo, H. E., and Rascio, V., *Corrosion Marine-Fouling*, 1976, **1**, 5.
10. Rascio, V., Caprari, J. J., Chiesa, M. J., and Ingeniero, R. D., *Corrosion Marine-Fouling*, 1976, **1**, 15.
11. Convenio SENID-CONICET, Programa ECOMAR I, 1974-77.
12. Benítez, J. C., and Giúdice, C., *CIDEPINT-ANALES*, 1978, p. 157.
13. Petit, J., *Peintures, Pigments, Vernis*, 1946, **22**, 3.
14. Petit, J., *Peintures, Pigments, Vernis*, 1951, **27**, 143.
15. Petit, J., *Peintures, Pigments, Vernis*, 1959, **35**, 555.
16. Rascio, V., *LEMIT*, internal report, unpublished.
17. *BSRA*, Recommended practice for the protection and painting of ships. London, 1972.
18. Rascio, V., Caprari, J. J., Chiesa, M. J., and Iasi, R., *CIDEPINT-ANALES*, 1976, p. 41.
19. De la Court, F. H., and De Vries, H., *J.O.C.C.A.*, 1973, **56**, 388.
20. De la Court, F. H., and De Vries, H., *Progress in Organic Coatings*, 1973, **1**, 375.
21. Bastida, R., Spivak, E., L'Hoste, S. G., and Adabbo, H. E., *LEMIT-ANALES*, 3–1974, p. 97.
22. Bastida, R., L'Hoste, S. G., Spivak, E., and Adabbo, H. E., *LEMIT-ANALES*, 3–1974, p. 167.
23. Bastida, R., and Lichtschein, V., *CIDEPINT-ANALES*, 1978, p. 55.
24. Rascio, V., Bruzzoni, W. O., Bastida, R., and Rozados, E., Protección de Superficies Metálicas. *LEMIT*, La Plata, 1977.
25. Christie, A. O., *J.O.C.C.A.*, 1977, **60**, 438.
26. Kronstein, M., and Denninger, C. R., *Mod. Paint Coat*, Oct. 1976, 29.

Next month's issue

The Honorary Editor has accepted the following papers for publication, and they are expected to appear in the November 1978 issue of the *Journal:*

The theory and practice of pigment dispersion *by W. Carr*

The corrosion and protection of metals in the building and construction industries *by B. G. Callaghan*

Measurement of conductivity, capacity, electrical resistance and permeability of paint films in an aqueous solution *by V. Vetere, E. Rozados and R. Carbonari*

Electrical resistance of electrodeposited rubber per unit coulomb *by S. Guruswamy*

Information Received

Change of name

Drynamels Ltd, the Birmingham coatings company of Tube Investments Ltd, has now changed its name to TI Drynamels Limited. The change is to emphasise that the company has the support of the Tube Investments group, particularly in its plans to maintain and improve its position in the thermoset powder coating field.

UK agency

Manchem Ltd have been appointed distributors bythe European Chemical Division of Compagnie Francaise Goodyear of *Pliolite* paint resins for the UK and Eire.

IP expansion

International Paint—Industrial Coatings, have announced a programme for a major expansion of their east London plant at Silvertown. The company has acquired a a wharf adjacent to their present site which will increase the size of the facilities by 50 per cent.

Increased manufacturing facilities

Atlantic Chemical Manufacturing Corp. of New Jersey has recently completed additions to its manufacturing facilities, including the addition of glass lined reactors and coupling tanks. This expansion is intended to allow Atlantic Chemical to develop its product line of dyes and to manufacture many of the basic intermediates which are used in their production lines.

Oil resistant coatings

Following increased demands for non-polluting coatings, Claremont Polychemical Corp. is now supplying commercial quantities of a new series of water-based lacquers suitable for finishing paper and cardboard. The new products comply with EPA and OSHA anti-pollution regulations, and are available as gloss or dull coatings which are applied in both web-fed and sheet-fed operations using commercially available gravure and flexographic coaters as well as dampening units attached to offset presses.

New marketing arrangements

Nextel coating, the spray-on material from 3M which gives a suede-like finish to a wide range of interior surfaces and consumer products, is now available from a single authorised distributor, G. F. Haynes & Co. of Leicester. Nextel consists of microscopic neoprene granules suspended in a carrying medium, each granule being individually pigmented, The coating can be sprayed on to wood, ferrous or non-ferrous metals, glass, asbestos, card and even paper. The material has high resistance to wear and tear and offers the additional benefit of significantly reducing sound transmission.

New products

Electron beam curing unit

Otto Dür (GB) Ltd has available the new ESH 150 Dür Electron beam curing unit which is claimed to be adaptable to many situations, including painted, varnished or lacquered finishes, laminating substrates, printing, polymerisation etc. The unit uses relatively low acceleration voltages of 150 kV which eliminates the need for heavy screening to cut-out secondary radiation: 5 mm thick lead sheeting is enough to satisfy safety requirements.

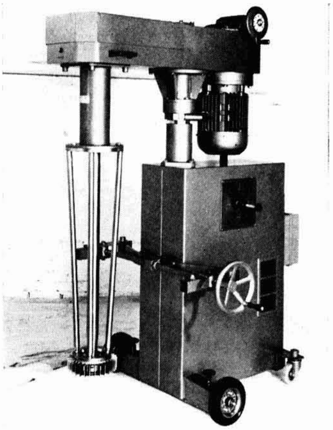
Reciprocating surface tester

Timperley Engineering's reciprocating surface tester, originally developed for the wallpaper industry, where it is standard equipment, can now be supplied with a range of different reciprocating heads to test sample batches of inks, paint and other surface coatings and treatments.

The new reciprocating surface tester from Timperley Engineering, which can be used to test the durability of coatings

Chemcol/Mirap mixing machines

John Godrich has available the new Chemcol/Mirap liquid mixing machines, which were first introduced into the UK at the OCCA Exhibition earlier this year. The machines are mobile and compact, and have efficient mechanisms for raising and lowering. The Mirap machines, which do not have immersed bearing heads, have variable speeds and interchangeable dissolver discs and paddle wheels.



The Chemcol/Mirap liquid mixing machine which was introduced in the UK at the OCCA-30 Exhibition

Electrical connectors for harsh environments

Techmation Ltd have available a wide range of Brantner Sea Con electrical connectors which were first developed for use in underwater and offshore installations. The Brantner connectors can withstand high pressures of up to 10,000 psi and heavy weights, especially useful in field installations where heavy vehicles may be driven frequently over cables.

Dispensing pump

ChemLab Instruments Ltd, agents for Ste.Asti, have announced that a new dispensing pump manufactured uniquely in PTFE and glass covering the range from 0·2 to 10ml. is now available. The new model PSP is available with pre-selection counter and timer unit, and liquid handling parts can be easily dismantled for steam sterilisation.

New ink systems

Claremont Polychemical Corporation, New York, has begun marketing a new low energy water-based crosslinking ink system suitable for gravure and flexographic printing on vinyl coated fabrics, which has good scrub and solvent resistance and has already been used for printing vinyl wallcoverings. Named the Vynaprint GV-25000 series, it is available in a wide range of colours.

Stannicide M

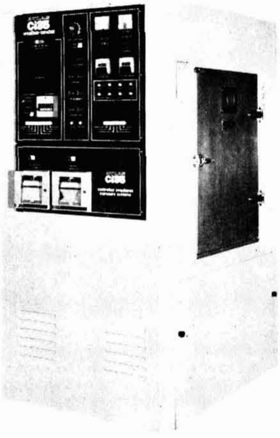
Thomas Swan & Co. Ltd has added a new tributyltin biocide, Stannicide M, to their range of organotin compounds. Its main application is in water based paints, and it is effective against a wide range of organisms, including bacteria, fungi, moulds, algae, molluscs and crustacea. It is readily dispersed in water producing stable emulsions, and is supplied as a 40 per cent solution.

New anti-static coating

A graphite water-borne paint with special anti-static properties is now available from International Paint—Industrial Coatings, which is claimed to have good stability characteristics. The coating was originally developed for coating the backs of television cabinets, and has an electrical resistance of less than 100 ohms between two points 14 inches apart.

Xenon Weather-Ometer

Atlas Electronic Devices Co. of Chicago have added the new model Ci35 to their range of Atlas Weather-Ometers. It is a constant irradiance xenon system for laboratory exposure testing, and incorporates several sophisticated control features which improve test uniformity whilst simplifying operation.



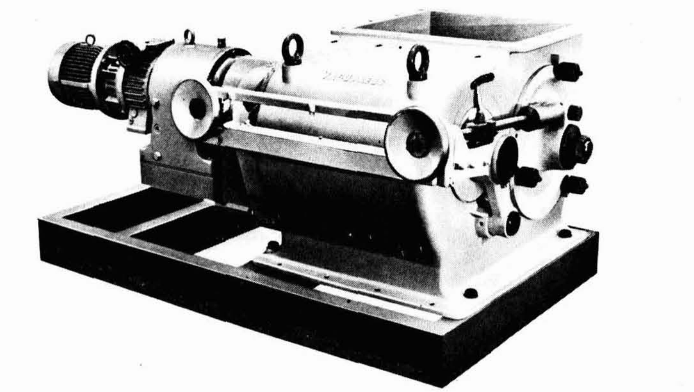
The Model Ci35 Atlas Weather-Ometer constant irradiance xenon system

Digital coating thickness gauge

A new portable digital thickness gauge manufactured by Zorelco Ltd of Ohio, is now available from Wentworth Instruments Ltd. The model 747 is designed to measure any dry non-conductive coating applied to a ferrous or non-ferrous surface, using the eddy current principle.

Weld porosity eliminated

The microscopic porosity inherent in welds can now be eliminated by applying Loctite Superfast 290, thus preventing leakage of liquids and gases, claim the manufacturers, Loctite, (UK) Ltd. Loctite 290 is a liquid sealant which can be applied direct from the bottle. It penetrates the porous surface by capillary action, and hardens without shrinkage.



One of the Vacupress range of densifying machines for de-aerating powders

Space savings

Universal Process and Plant Ltd has available the new Vacupress range of densifying machines, which have applications where finely divided powders need to be de-aerated. The densification is achieved by the combined action of controlling vacuum and pressure, and the machine can be installed directly below milling or grinding machines.

Flow meter accuracy

Two instruments, designed to count and indicate pulses from flow meter proving systems where the flow of liquids or gases are being controlled, are now available from Jiskoot Autocontrol Ltd. The meter prover counter types MC2 and FMC3, which incorporate the latest integrated circuits, work on the principle of two displays, one of the pulse of the flow meter under test, and one of the pulse of the master meter. By comparing the two figures the error of the meter being tested can be accurately determined.

Conferences, courses etc.

BSI—Pigments

Two revised British Standards for paint pigments have been issued by the BSI: *BS 303 Lead chrome pigments for paints*, and *BS 1851 Titanium dioxide pigments for paints*.

All-India Paint Conference

The ninth all-India paint conference will be held on 13-14 January 1979 at the Park Hotel, Calcutta, with the theme 'Paint or perish'.

Solids handling/processing technology

Simon-Solitec has issued a six-page leaflet describing their ranges of plant and machinery for handling solids and processing technology.

Titanates leaflet

Dynamit Nobel AG has issued a revised leaflet entitled "Titanic acid esters, titanchelate—Reactions and applications", which deals with detailed specifications, properties, solubility, hydrolysis-stability etc. It also gives information regarding classes of risks and indications regarding safe handling.

Adhesives and sealants brochure

The Industrial Distribution Division of Loctite (UK) Ltd has published a new brochure which describes the new Loctite Superfast range of adhesives and sealants, and their particular relevance to plant and maintenance engineers.

Adhesion measurement

The American Society for Testing and Materials, ASTM, has published a book entitled "Adhesion measurement of thin films, thick films, and bulk coatings". Edited by K. L. Mittal of IBM Corporation, the book comprises 410 pages and is available price, US $39·25.

Internal can linings

R. H. Chandler Ltd has published a book entitled "Internal can linings" which covers this area of paint technology which is often overlooked. The book reviews recent advances and points out general trends.

Progress in perspective

This publication is a personal review by R. Cutforth of the progress of the Bayer organisation, and the contribution and influence of the chemical industry in our society. The booklet also lists the structure, products and services of Bayer UK Ltd.

Australian Raw Materials Index

OCCA Australia has available the fourth edition (1977) of its Raw Materials Index, a publication which lists suppliers to the paint and allied industries in Australia. Product names, product descriptions and applications are listed, as well as the names and addresses of the suppliers. This book is invaluable to those who have dealings with the Australian industry, and is available price Australian $20.00 plus postage and packing, from the Chairman, OCCAA RMI Sub-Committee, GPO Box 3605, Sydney 2001, Australia.

OCCA-31 Exhibition

Alexandra Palace, London, 3-6 April 1979

The international focal point for the surface coatings industries

Many enquiries received from home and overseas

Arrangements for OCCA-31

The thirty-first Annual Exhibition of raw materials, plant and equipment for the paint, printing ink, colour, adhesive, polymer and allied industries organised by the Association will be held at Alexandra Palace, London N22 from 3–6 April 1979.

Already, support for the Exhibition has been received from many organisations both from the UK and overseas countries, comprising applications for space from organisations new to the exhibition as well as established exhibitors. **The closing date for applications to exhibit is given as 31 October 1978, and during November the first allocation of space will take place to applications registered before that date. Applications for space received after the closing date will be considered, and allocated any space available after the initial allocation has taken place, but organisations wishing to take space at the Exhibition are urged to ensure that their applications are registered in good time so that any preferences they may have as to stand size, shape and position may be carefully considered.**

Theme of the Exhibition

The aim of the Exhibition is the presentation of technical and commercial information relating to advances in the surface coatings industries. The Committee stipulates that a technically trained person who has knowledge of the products displayed be available on the stand throughout the Exhibition.

Whilst the Committee naturally encourages the showing of new products, it does not stipulate that a new product has to be shown each year, since it fully appreciates that this is not possible. Accordingly, the Committee draws attention to the fact that new technical data on existing products is regarded as acceptable subject matter.

Motif for the Exhibition

The motif, designed by Robert Hamblin, emphasises the unique aspect of the OCCA Exhibitions, which annually provide an international focal point for the surface coatings industries. In 1978, exhibitors were drawn from 16 countries and visitors from over 50 countries. The two main colours of the motif, green and yellow, will be incorporated on the facias of the stands and it is intended to organise complementary floral displays in the Exhibition Hall. The use of these colours will create a pleasing contrast as visitors move from corridor to corridor at the Exhibition, as the green colour will be used from north to south and the yellow colour from east to west.

Dates and hours of opening

The Exhibition will be open as follows:

Tuesday 3 April ..	09.30 to 17.30 hrs.
Wednesday 4 April	09.30 to 17.30 hrs.
Thursday 5 April ..	09.30 to 17.30 hrs.
Friday 6 April ..	09.30 to 16.00 hrs.

Invitation to Exhibit

Copies of the Invitation to Exhibit have been despatched to companies and organisations in the UK and overseas which have shown at previous OCCA Exhibitions or have requested information for the first time for the 1979 Exhibition. Completed application forms for stand space must be returned to the Director & Secretary of the Association **not later than 31 October 1978.**

Any organisation wishing to receive details of OCCA-31 should write to the Director & Secretary of the Association for a copy of the Invitation to Exhibit.

The interest shown in the OCCA-31 Exhibition as a direct result of the highly successful OCCA-30 Exhibition held in April this year has been considerable, and already companies have asked for larger stands than they have had in previous years. Additionally, several companies who have not previously shown at OCCA Exhibitions as well as some who were not able to be present at OCCA-30 have contacted the Association requesting that sites be reserved for them.

Visitors to the OCCA-30 Exhibition came from over 50 countries and admissions by season ticket of over 10,000 were recorded. *Left:* **A view of the Association's Information Centre at the OCCA-30 Exhibition.**

The Exhibition Committee was particularly pleased to see the large number of exhibits at OCCA-30 from companies new to the Exhibition, both from the UK and many overseas countries, and this emphasises the continuing importance of this annual Exhibition to the world surface coatings industry and demonstrates its international character. The number of stands at OCCA-30 was larger than at the Exhibition for several years, and the number of companies was also greater. It will be appreciated that an exhibitor may request a stand of the size to suit his requirements, and thus the Committee points out that it is possible for organisations to demonstrate their products in a cost effective manner; however, there is no restriction on the maximum size of stand an exhibitor may request, and exhibitors are now permitted to serve alcoholic refreshments to their visitors on the stands.

This innovation, introduced in 1977, was particularly welcomed by exhibitors as it allowed their personnel to entertain visitors on their stands throughout the period of the Exhibition.

Admissions at OCCA-30

Visitors to the OCCA-30 Exhibition are known to have come from over 50 overseas countries, and admissions by season ticket of over 10,000 were recorded at the entrance during the four days.

All Members of the Association receive a free season admission ticket with their copy of the Official Guide, and organisations exhibiting receive complimentary admission tickets to distribute to their customers. In addition, many trade associations are also sent tickets for distribution to their members. Thus, many visitors to the Exhibition have season admission tickets provided in advance.

However, an analysis* of the tickets purchased at the entrance to the hall, sold mainly to visitors who have come as a result of the Association's widespread publicity rather than having been invited by exhibitors, shows that 34 per cent of these visitors came from overseas, with a significant proportion coming to the Exhibition from outside Europe. Particularly impressive amongst these visitors from overseas was the high proportion of top personnel: 61 per cent of them were managers, directors or owners of companies, with a 55/45 split between technical and commercial people.

With visitors from the United Kingdom, the split between technical and commercial personnel was 64/36; again there was a large proportion of senior management, although there was also a strong representation of section heads/group leaders and chemists.

The west side of Alexandra Palace

These figures indicate the great value to organisations of exhibiting their products and developments at OCCA Exhibitions, which continue to attract each year large numbers of interested visitors from all over the world. The Exhibition provides a forum where exhibitors can discuss their products with the senior executives and technical personnel of many organisations whom they would not otherwise be able to contact, or to whom it would be prohibitively expensive to send representatives.

Facilities at Alexandra Palace

The Association has full use of the facilities at Alexandra Palace during the period of the Exhibition which include a restaurant, bars, a cafeteria and a private 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.

For visitors travelling to the Exhibition on the underground system, the Association will once again be running a free bus shuttle service to and from the Exhibition, this year from Wood Green Station on the Piccadilly Line. The extension of the Piccadilly Underground Line to the Heathrow Central terminal was opened in late 1977, and this now provides a direct link for travellers arriving at the airport to both central London and to Wood Green Station and the bus shuttle service to the Exhibition.

Improvements at Alexandra Palace

In July 1978 work was started on a comprehensive refurbishment of the facilities at Alexandra Palace including the completion of the work to the exterior, the repainting of the West Corridor and the walls of the Great Hall, and the redecoration of the restaurants and bars. The floor of the Great Hall has already been resurfaced, and the remainder of the work is scheduled to be completed by the late summer this year. These improvements will add to the pleasantness of the venue, set in the extensive parklands, and will provide an excellent setting for this international focal point.

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. Interpreters will once again be in attendance at the Information Centre at the Exhibition to assist foreign exhibitors and visitors with their queries.

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 as well as to all visitors to the Exhibition with their admission tickets. It is published several weeks in advance of the dates of the Exhibition to allow visitors the maximum opportunity to plan the itinerary of their visits. In 1978, for the first time, the *Official Guide* contained a chart classifying the products on show into the various types, and it is intended to produce a similar chart for the *Official Guide* to OCCA-31. This chart is invaluable to visitors, especially those who have only a limited amount of time available, as it provides a quick, easy reference of all the companies present who are displaying the products in which they are interested.

Advertising facilities

Advertising space is available in the *Official Guide*, both to companies who will be exhibiting at the Exhibition, and also to other organisations. This important publication is constantly referred to both before the Exhibition and afterwards as a convenient record of the latest developments in the industry. Consequently, organisations wishing to take advertising space in the Guide should book their requirements as soon as possible in order to ensure a prominent position. Details of the advertising rates and media data are available from Mr D. M. Sanders at the Association's address.

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*Sample of 1600 completed tickets taken from those purchased at the entrance to the Exhibition.

OCCA Biennial Conference 1979

Stratford-on-Avon

20–23 JUNE 1979 STRATFORD HILTON HOTEL

The next biennial Conference of the Association will be held at the Stratford-upon-Avon Hilton Hotel, Stratford-upon-Avon, from Wednesday 20 to Saturday 23 June 1979, and the theme for the Conference will be:

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.

As on previous occasions, full preprints of the papers will be published for despatch to delegates in advance of the Conference, since it is the custom at OCCA Conferences for the author to outline briefly his paper, highlighting the points of interest, thus allowing much more time for general discussion with the delegates who will already have been able to familiarise themselves with the work by reading the preprints. It is important, therefore, that any person, whether or not a Member of the Association, who feels that a report of his work could form the basis of a suitable paper, should contact immediately the Honorary Research and Development Officer by writing in the first instance to the Director & Secretary at the Association's address on the Contents page.

Stratford-upon-Avon is a new venue for the OCCA Conference, and it is expected that it will prove popular and interesting to delegates. It is well connected by major roads and motorways, and there is a good service from London by coach. For the ladies attending the Conference, the social programme is hoped to include coach parties to nearby Warwick Castle, Leamington and Coventry Cathedral. There will also be a visit to Oxford and Blenheim Palace. The Royal Shakespeare Theatre is close to the Stratford Hilton Hotel, and delegates will be able to attend performances in the evenings. It is also hoped to arrange a boat-trip along the River Avon. The Association's Annual General Meeting will be held on the Friday afternoon after the last Technical Session, and in the evening the Association will organise a Dinner Dance.

Forms for registration will be despatched to all Members of the Association attached to Sections in the UK, Ireland, and the General Overseas Section early in 1979, and any non-member wishing to receive a registration form should write to the Director & Secretary of the Association who will send details as they become available.

The Royal Shakespeare Theatre on the banks of the River Avon where delegates will be able to attend performances in the evenings

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 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.

Obituary

Charles M. Beavis

Older Members of the Association will be saddened to learn of the death of C. M. Beavis (Peter Beavis), the former Managing Director of Golden Valley Colours Ltd, of Wick, near Bristol, for many years. He was 71 and had been in poor health for some time.

A memorial service was held on Friday 18 August at Bitton Church near Bristol. Peter had lived in the area all his life and it was a tribute to him that, although Golden Valley Colours Ltd had been closed for around 10 years, a good number of the older employees of the company attended the memorial service.

Peter was an engineer especially interested in the introduction of new micronized pigments, and he did much pioneering work in this important field.

W. G. Wade

Obituary

Dr G. Swann

The death occurred recently of Dr Geoffery Swann who had retired from Synthetic Resins Ltd in June 1972 as Development Director. He leaves a wife and two married sons.

After gaining a first class honours degree in Chemistry at St Andrew's University (1934), he obtained his doctorate in terpene chemistry, by which time he was employed as a Scientific Officer with Forest Products Research Laboratories (Timber Research). In 1938 he joined BX Plastics Ltd as Research Officer (Camphor products) and became manager of their Chemical Research Department (Plastics and Polymers) in 1945. In 1949 he joined Beck Koller & Co. (England) Ltd as Research Manager in which he took an interest in OCCA, not only by lecturing, but by encouraging his staff to join. By 1958 he was Research Director and Assistant Managing Director of 'BK' and by 1967, the Deputy Managing Director, a post which he held until the acquisition of 'BK' by Unilever.

He will be remembered by those who worked with and for him, (Margaret Thatcher being amongst the latter) as a man with a sharp wit and yet gentle sense of humour. Above all he was a dedicated family man and a dedicated scientist. His other interests included fly-fishing, gardening and photography.

A. C. J.

News of Members

Mr J. C. Bax, an Ordinary Member attached to the London Section, will present the OCCA nominated paper at the FSCT Annual Meeting to be held in Chicago, 1-3 November 1978, with the title "New concepts in the formulation of gloss latex paints". Mr Bax, of Scott Bader Ltd, has previously presented a paper on this subject to the XIIIth FATIPEC Congress in Nice.

Mr Svend Johnsen, an Ordinary Member attached to the General Overseas Section and an Associate in the Professional Grade, has been appointed research manager of GORI Research Ltd, Denmark.

Mr S. Johnsen

Dr T. A. Banfield, an Ordinary Member attached to the London Section and a Fellow in the Professional Grade, retired from the Berger Group at the end of July after 31 years' service.

He has been a Member of OCCA since 1947, has served on Council, and on the Newcastle Section Committee as Hon. Research Liaison Officer and as Hon. Treasurer. After the takeover of British Paints Ltd by Berger Jenson & Nicholson Ltd, he moved from Newcastle in 1972 to BJN's Group Research Laboratory at Stratford as Deputy Manager of the Group Technical Information Office. He was recently elected Vice-Chairman of London Section.

Although retired, he intends to retain his interest in the paint industry through his OCCA activities, and also hopes to undertake consultancy work on the protective painting of ships (including antifouling compositions) and of structural steel.

Scottish Section

Eastern Branch

The Eastern Branch of the Scottish Section is organising a Printing Ink Symposium to be held at Stirling University on Wednesday 19 January 1979. The symposium is aimed primarily at junior technologists, and will cover the basic aspects of printing ink manufacture, raw materials and processes. Further details may be obtained from Mr M. Nixon, Chairman Eastern Branch, 3E Lairds Hill, Seafar, Cumbernauld, Glasgow G67 1HH. (Tel: 02366 61221)

Newcastle Section

British Titan Cup Golf Competition

The seventeenth annual tournament for the British Titan Cup was held on Saturday 10 June, 1978 at the Catterick Garrison Golf Club. The competition, a Stapleford, was won jointly by Mr M. G. Harvey of Durham Chemicals Ltd and Mr R. G. Carr of Tor Coatings Ltd.

All the contestants enjoyed playing this very picturesque and interesting course.

H.F.

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

Beugger, Johan Jakob, 11 Ngobeni Road, Athlone Park, Amanzimtoti 4125, South Africa. *(Natal)*

Bognar, Joseph, NZ Forest Products Ltd, Private Bag, Auckland, New Zealand. *(Auckland)*

Bruwer, Benjamin Edward Cornelis, BSc, SA Bureau of Standards, Private Bag X 191, Pretoria 0001, South Africa. *(Transvaal)*

Gallagher, Peter, BSc, CChemMRIC, Cabot Carbon Ltd, Stanlow, Ellesmere Port, S. Wirral. *(Manchester)*

Jones, Martin Douglas, Coates Bros (NZ) Ltd, PO Box 12068, Auckland, New Zealand. *(Auckland)*

Laffoley, Reginald Stanley, 26 Hammer Gate, St. Albans, Herts. *(London)*

Muller, Dietmar, 803 San Martinho, Leicester Road, Bedfordview 2008, South Africa. *(Transvaal)*

Shubbar, Hashim Ahmad Mohammad, PO Box 11029, Masarif, Baghdad, Iraq. *(General Overseas)*

Thompson, Samel James, 99 Vorster Avenue, Glenanda, Johannesburg 2091, South Africa. *(Transvaal)*

Wong, Thomas, 60 Stanley Street, London, Ontario N6C 1B1, Canada. *(Ontario)*

Yap, Tee Ping, BSc, 27 Jalan 4/54, Petaling Jaya, Selangor, Malaysia. *(General Overseas)*

Associate Members

Assen, Henri, Eirchem Ltd, Raystown, Ratoath Road, Ashbourne, Co. Meath, Eire. *(Irish)*

Buchling, Dolena Otillia, PO Box 3050, Witbeeck 1729, South Africa. *(Transvaal)*

Campbell, Barry Ian, 308 Glendower Place, Linksfield Road, Glendower, Edenvale, South Africa. *(Transvaal)*

Foggin, Graham David, Mair Industrial Marketing Ltd, PO Box 37-442, Auckland, New Zealand. *(Auckland)*

Spencer, Stephen John, 58 Croft Drive, Moreton, Wirral, Merseyside L46 0QT. *(Manchester)*

Taeuber, Konrad Ernst, Bayer SA (Pty) Ltd, Chemical Division, PO Box 1366, Johannesburg 2000, South Africa. *(Transvaal)*

Van Dyk, Burgert Jacobus, Bayer SA (Pty) Ltd, Chemical Division, PO Box 1366, Johannesburg 2000, South Africa. *(Transvaal)*

Forthcoming Events

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

October

Auckland Section: 'Hot method adhesives and coatings' by Mr D. Reid of Petersons Chemicals. *Details to be announced.*

Monday 2 October

Hull Section: 'The practical problems of painting and maintaining large industrial plant' by D. A. Bayliss of BIE Anti-Corrosion Ltd. Joint meeting with the Institution of Chemical Engineers, at the Humber Bridge Hotel, Barton, South Humberside at 6.45 p.m.

Tuesday 3 October

West Riding Section: 'Instrumental colour control in the paint industry' by Mr R. P. Best of Instrumental Colour Systems Ltd, at the Mansion Hotel, Roundhay Park, Leeds 8 at 7.30 p.m.

Thursday 5 October

Newcastle Section: 'Solvent recovery for the surface coatings industry' by Mr I. Smallwood of Fraser McNaughton Ltd, at St. Mary's College, Elvet Hill Road, Durham at 6.30 p.m.

Friday 6 October

Hull Section: Annual Dinner Dance, to be held at the Willerby Manor Hotel, Willerby, near Hull.

Thursday 12 October

Midlands Section—Trent Valley Branch: 'Coil coatings' by Dr Gilmore of Conway Coatings Ltd, at the Crest Hotel, Pastures Hill, Littleover, Derby at 7.00 p.m.

Scottish Section: 'The practical problems of painting and maintaining large industrial plants' by D. Bayliss of BIE Anti-Corrosion Ltd, at the Albany Hotel, Glasgow, at 6.00 p.m.

Thames Valley Section: 'Coatings for wood'. One day meeting at Building Research Establishment, Princes Risborough. *Details to be announced.*

Friday 13 October

Manchester Section: 'Theory versus practice of dispersion' by Dr W. Carr of Leeds University at the Manchester Literary & Philosophical Society, George Street, Manchester, at 6.30 p.m.

Tuesday 17 October

London Section: Afternoon visit to the Metropolitan Police Foresnic Science Laboratory, Lambert Road, London SE1 at 2.30 p.m.

Wednesday 18 October

London Section: 'Finishes and materials for window frames' by Mr P. Whiteley of Building Research Establishment, at Rubens Hotel, Buckingham Palace Road, SW1 at 7.00 p.m.

Ontario Section: "Resins for flexo inks" by G. Laudato at the Constellation Hotel, Toronto at 6.00 p.m.

Thursday 19 October

Midlands Section: 'Coil coating' by Mr E. Sharpe of Merritts Ltd, at the Calthorpe Suite, County Ground, Birmingham at 6.30 p.m.

Friday 20 October

Irish Section: 'Aqueous based printing inks' by Mr R. Millard of Harlow Chemical Co. Ltd, at the Clarence Hotel, Dublin at 8.00 p.m.

Manchester Section: Annual Dinner Dance at the Piccadilly Hotel, Manchester. *Details to be announced.*

Friday 27 October

Bristol Section: 'The refinishing of motor vehicles' by Mr D. E. Sellen of Berger Paints, at the Royal Hotel, College Green, Bristol at 7.15 p.m.

London Section: Ladies' night, to be held at Selsdon Park Hotel, Sanderstead, Surrey, at 7.00 for 7.30 p.m.

Midlands Section—Trent Valley Branch: Halloween Dance, at the Cross Keys Inn, Turnditch, *details to be announced.*

Scottish Section—Eastern Branch: Skittles match at the Civil Service Club, Marine Drive, at 7.00 for 7.30 p.m.

November

Auckland Section: Talk and inspection of the Water Treatment Plant, Auckland Regional Authority at Titirangi, by Mr D. Payne. *Details to be announced.*

Thursday 2 November

Newcastle Section: "Recent advances in pigment dispersion and dispersion equipment" by Mr R. W. English and Mr J. Perkins, Mastermix Engineering Co. Ltd, at St. Mary's College, Elvet Hill Road, Durham, at 6.30 p.m.

Monday 6 November

Hull Section: "The safe use of solvents and the trend towards development of low aromatic grades" by G. Hicks of Esso Chemicals Ltd, at the George Hotel, Land of Green Ginger, Hull, at 6.30 p.m.

Manchester Section: "Quality control" by Mr Murray of Crown Decorative Products Ltd, at the Woodcourt Hotel, Sale, at 6.30 p.m.

Tuesday 7 November

West Riding Section: "The testing of hazardous chemicals with particular reference to the requirements of the Health and Safety at Work Act" by Mr D. H. Pullinger, Assistant Scientific Director, Bazelton Laboratory Europe Ltd, at the Mansion Hotel, Roundhay Park, Leeds 8, at 7.30 p.m.

Thursday 9 November

Midlands Section—Trent Valley Branch: "Effect of coatings on the quality of drinking water" by Dr N. P. Burman, at the Crest Hotel, Pastures Hill, Littleover, Derby, at 7.00 p.m.

Scottish Section:—"The academic-industrial interface". Joint meeting with ASDC, at Albany Hotel, Glasgow, at 7.30 p.m.

Thames Valley Section: "N.A.Ds. A progress report" by Mr M. Waghorn of ICI Paints Ltd, at the Beaconsfield Crest Motel (White Hart), Aylesbury End, Beaconsfield, Bucks, at 6.30 p.m. for 7.00 p.m.

Wednesday 15 November

Manchester Section: Student Lecture "Additives improve emulsion paints" by Mr J. B. Clarke of Allied Colloids Ltd, at Manchester Literary & Philosophical Society, George Street, Manchester, at 4.30 p.m.

Ontario Section: "A review of current drier technology" by Mr L. Campay, at the Constellation Hotel, Toronto, at 6.00 p.m.

Thursday 16 November

Midlands Section—Student Lecture: "Mineral pigments through the ages" by Mr E. Hare, at the Calthorpe Suite, County Ground, Birmingham, at 6.30 p.m.

Friday 17 November

London Section: "Colour instrumentation". Day meeting in association with the Society of Dyers and Colourists, London Region, at Thames Polytechnic, Woolwich, at 10.00 a.m.

Wednesday 22 November

Scottish Section—Eastern Branch: "Hydrocarbon resins" by A. Malatesta of Esso Chemicals Ltd, at the Murrayfield Hotel, 18 Corstorphine Road, Edinburgh at 7.30 p.m.

Friday 24 November

Bristol Section: Ladies 'Evening "The colouring and decoration of pottery" by Mr A. J. Simkin of Blythe Colours Ltd, at the Royal Hotel, College Green, Bristol, at 7.15 p.m.

Irish Section: Annual Dinner Dance, at the Clarence Hotel, Dublin, at 8.00 p.m.

West Riding Section: Dinner Dance, at the Crown Hotel, Harrogate, *details to be announced.*

December

Wednesday 13 December

Ontario Section: Ladies' Night, *details to be announced.*

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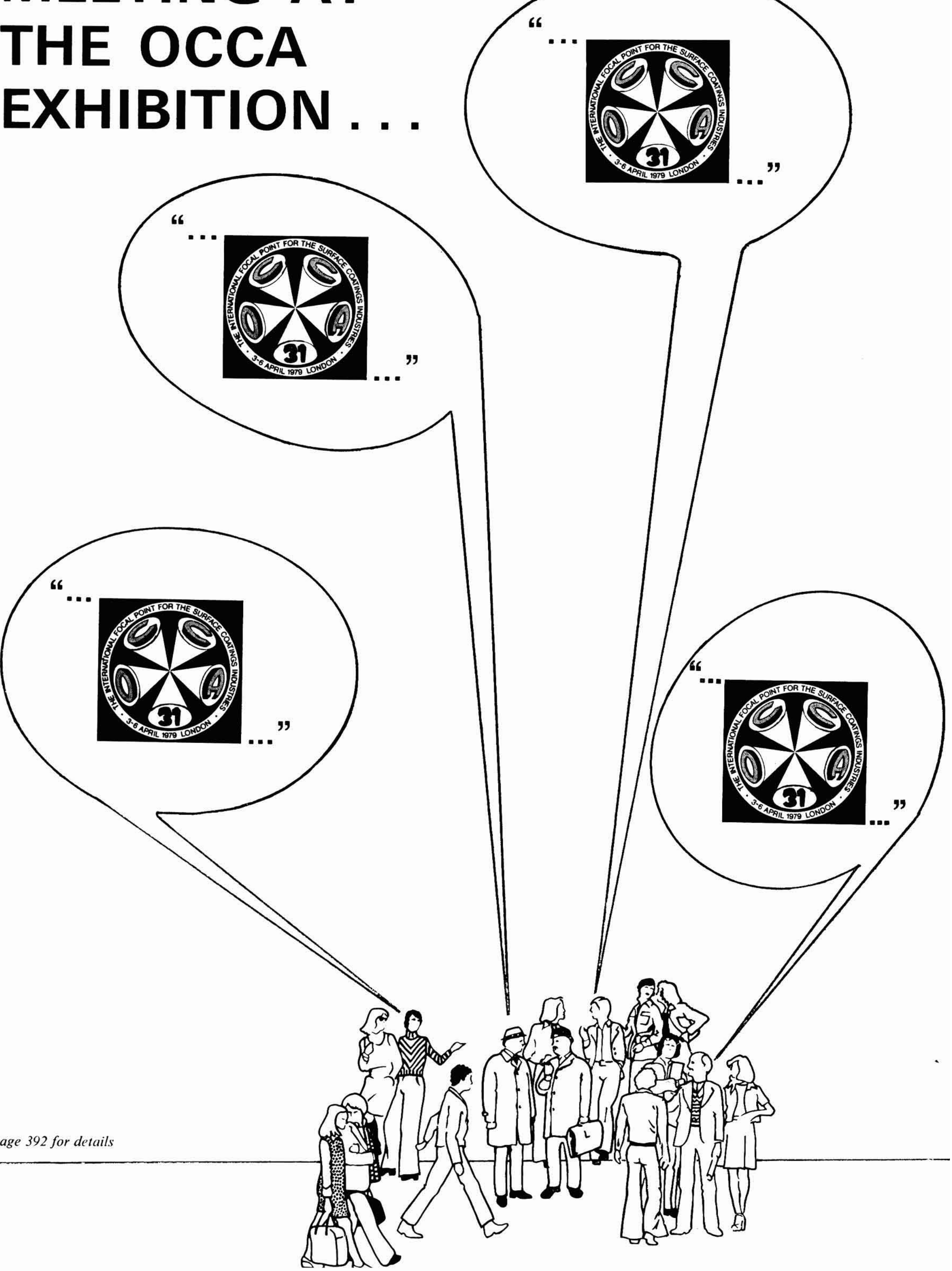
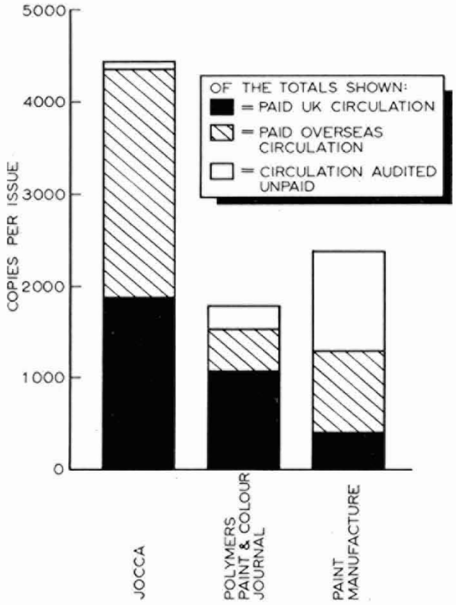


See page 392 for details

Comparison of circulations of U.K. publications to the paint, printing ink and allied industries



(Reference Audit Bureau of Circulations Reviews. Jan-Dec 1977)

For full details of advertising in this, and other Association publications, contact D. M. Sanders, Assistant Editor

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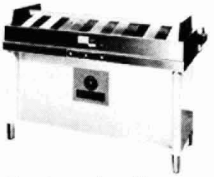

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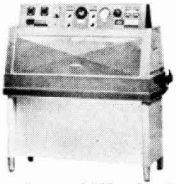

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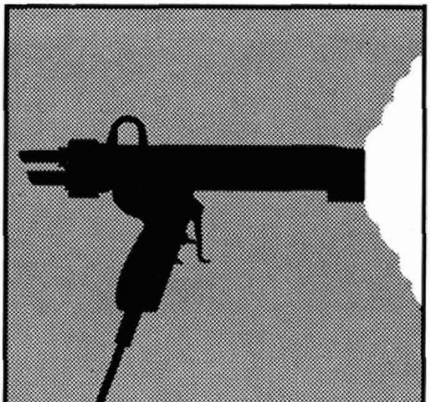

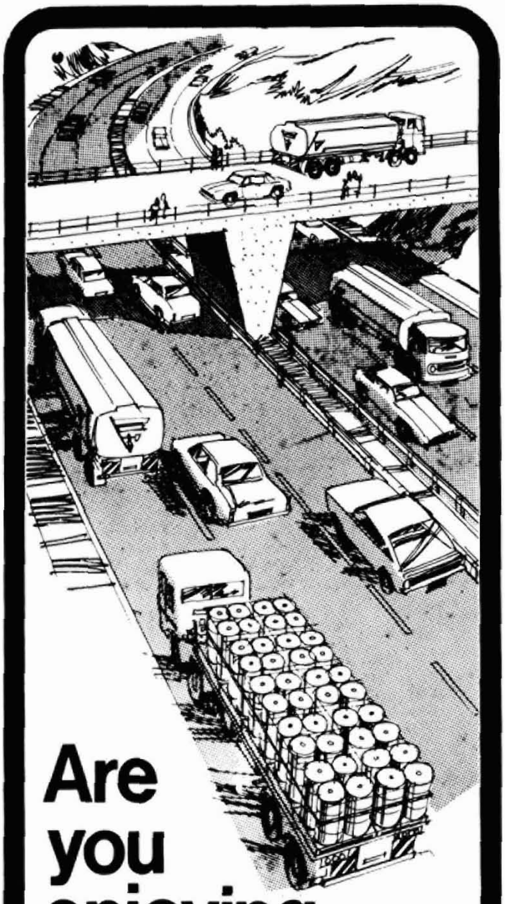

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OIL & COLOUR CHEMISTS' ASSOCIATION—PUBLICATIONS (Order form page iv)

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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.

Special offer

Ultraviolet Polymerisation and the Surface Coatings Industry

The two volumes of the papers presented at the Newcastle Section Symposia on this subject held in 1975 and 1977 are offered at a special price of £10.00 for both volumes purchased together.

Normal price: *Ultraviolet polymerisation (1975)* £5.00; *Ultraviolet polymerisation 2 (1977)* £7.50 (*see below*)

UV2 UV2

The eleven papers in this volume were originally published in the *Journal* earlier in 1978. They are based on lectures given at the Second International Symposium of the Newcastle Section of the Association held at Durham University on 14 and 15 September 1977 with the theme "Ultraviolet polymerisation and the surface coatings industry". Titles and authors are listed below:

Exciplex interactions in photoinitiation of polymerisation by fluorenone amine systems *by A. Ledwith, J. A. Bosley and M. D. Purbrick*

Recent developments in photoinitiators *by G. Berner, R. Kirchmayr and G. Rist*

Present status of ultraviolet curable coatings technology in the United States *by J. Pelgrims*

The design and construction of ultraviolet lamp systems for the curing of coatings and inks *by R. E. Knight*

New developments in ultraviolet curable coatings technology *by C. B. Rybny and J. A. Vona*

Cure behaviour of photopolymer coatings *by R. Holman and H. Rubin*

Photoinitiator problems in clear coatings *by M. De Poortere, A. Ducarme, P. Dufour and Y. Merck*

The UV curing of acrylate materials with high intensity flash *by R. Phillips*

Parameters in UV curable materials which influence cure speed *by A. van Neerbos*

The use of differential scanning calorimetry in photocuring studies *by A. C. Evans, C. Armstrong and R. J. Tolman*

The UV curing behaviour of some photoinitiators and photoactivators *by M. J. Davis, J. Doherty, A. A. Godfrey, P. N. Green, J. R. A. Young and M. A. Parrish*

To obtain this book, complete the order form and send with the necessary remittance to the Association's offices. *Price: £7·50 (US $15) each.*

[See order form on page iv

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