Vol. 61 No. 2 JOCCAB 61(2) 39-62 (1978) February 1978

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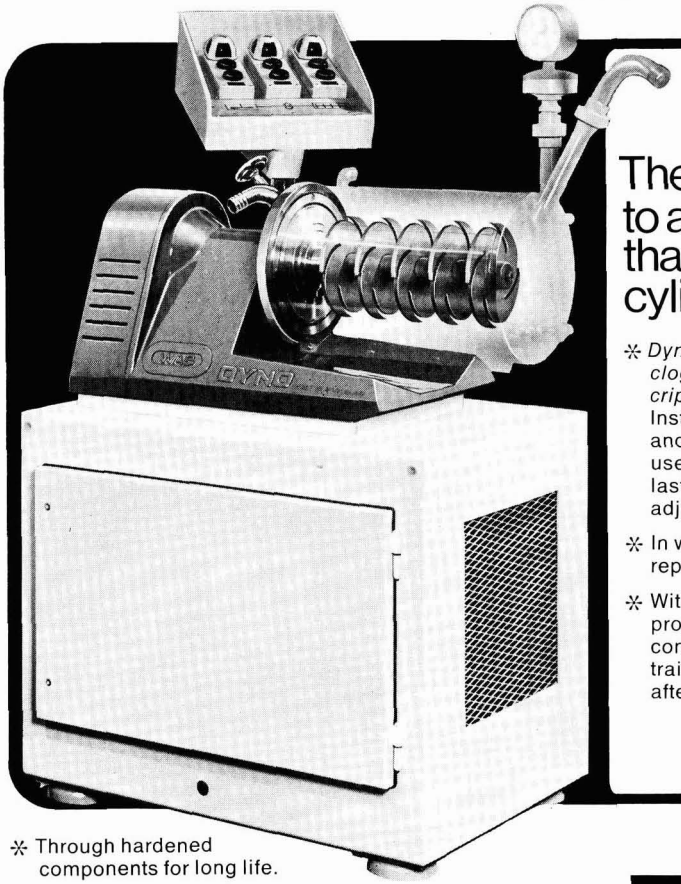

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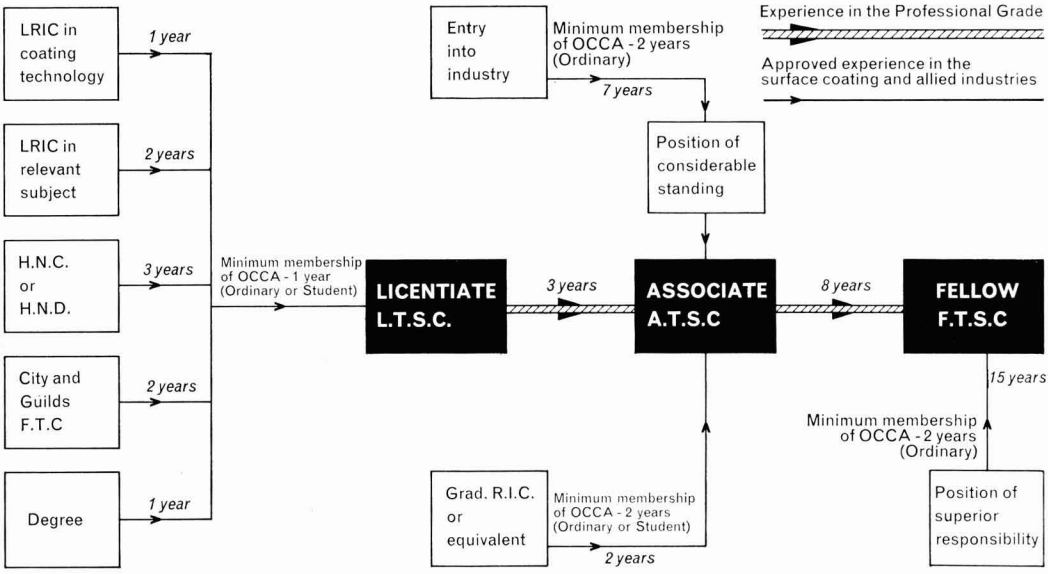
Optional Professional Grade for Ordinary Members

The innovation of the Professional Grade has proved to be most successful, as evidenced by the impressive list of names in the December 1977 issue of the *Journal*. For the convenience of potential applicants, a chart indicating different routes to the various grades is shown below

Routes to the Professional Grades



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

Regulations for admission to the Professional Grade

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

A. Licentiate

1. Shall be an Ordinary Member of the Association and have been an Ordinary Member or Student of the Association for not less than one year.

2. Shall have attained the age of 22.

3. (a) Shall be a Licentiate of the Royal Institute of Chemistry in Coatings Technology (*viz.* Higher National Certificate + Endorsement in coatings technology + 1 year approved experience in the science or technology of coatings after passing the endorsement examination).

OR (b) Shall be a Licentiate of the Royal Institute of Chemistry in another relevant subject such as advanced analytical chemistry, colour chemistry or polymer science, and shall have two years' approved experience of coatings since so qualifying.

OR (c) Shall hold the Full Technological Certificate of the City and Guilds of London Institute in a relevant subject as approved by the Professional Grade Committee and shall have two years' approved experience in the science or technology of coatings since gaining the FTC.

OR (d) Shall have passed Higher National Certificate or Higher National Diploma with three years' approved experience in the science or technology of coatings since qualifying, but two years' approved pre-qualification experience shall be deemed equivalent to the third post-qualification year.

OR (e) Shall be graduate in relevant subject with not less than 1 year's approved experience.

OR (f) Shall have passed such other qualifications as approved by the Professional Grade Committee from time to time.

4. Shall be required to satisfy the Professional Grade Committee, or some other body approved by the Professional Grade Committee in a *viva voce* examination and submit a dissertation on a topic previously approved by the Professional Grade Committee.

5. Shall normally be sponsored by three Ordinary Members of the Association in the professional grade (either Associate or Fellow) at least one of whom must be a Fellow.

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

B. Associate, being already a Licentiate

1. Shall, since his election to the Licentiateship, have practised the science or technology of coatings for not less than three years.

2. Shall provide evidence acceptable to the Professional Grade Committee of his superior professional skill and maturity.

3. Shall hold the City & Guilds of London Institute Insignia Award OR shall submit a thesis or dissertation of comparable level on a topic previously approved by the Professional Grade Committee OR shall have published work which, in the opinion of the Professional Grade Committee, is of comparable merit.

4. MAY be required to satisfy the Professional Grade Committee or some other body as approved by the Professional Grade Committee in a *viva voce* examination.

5. Shall normally be sponsored by three Ordinary Members of the Association in the professional grade (either Associate or Fellow) at least one of whom must be a Fellow.

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

C. Associate, not already a Licentiate

EITHER

1. Shall be not less than 24 years of age.

2. Shall be an Ordinary Member of the Association and have been an Ordinary Member or Student of the Association for not less than two years.

3. Shall hold the Graduateship of the Royal Institute of Chemistry or Council of Physics or a University or Council of National Academic Awards degree recognised by the Royal Institute of Chemistry or Institute of Physics as giving full exemption from the Graduateship examination.

4. Shall have not less than two years' approved post-graduate experience in the science or technology of coatings.

5. Shall normally be required to satisfy the Professional Grade Committee or some other body approved by the Professional Grade Committee, at a *viva voce* examination.

6. Shall normally be sponsored by three Ordinary Members of the Association in the professional grade (either Associate or Fellow) at least one of whom must be a Fellow.

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

OR

8. Shall be not less than 30 years of age.

9. Shall be an Ordinary Member of the Association and have been an Ordinary Member of the Association for not less than two years.

10 Shall have been engaged in practising the science or technology of coatings for not less than seven years and shall have attained a position of considerable standing in the industry.

11 Shall normally be required to satisfy the Professional Grade Committee in *viva voce* examination of his professional competence.

12 Shall normally be sponsored by three Ordinary Members of the Association in the professional grade (either Associate or Fellow) at least one of whom must be a Fellow.

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

D. Fellow

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

1. Shall be not less than 33 years of age.

2. Shall have been an Ordinary Member of the Association for not less than two years.

3. Shall be engaged in a position of superior responsibility in the coatings industry.

4. EITHER (a) shall have been an Associate of the professional grade for at least eight years;

   OR (b) shall have not less than fifteen years' experience of the science or technology of coatings in a position of superior reponsibility.

5. Shall submit, with his application, an account of his experience, with due reference to scientific and technological interests, achievements and publications.

6. Shall normally be sponsored by three Ordinary Members of the Association in the professional grade, all of whom must be Fellows.

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

The fees payable with applications are as fellows:

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

Application

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

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

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

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

It is felt that applicants for admission to the Licentiate grade might wish to have further information on the pattern which it is suggested should be followed for dissertations, the subjects of which have first to be approved by the Professional Grade Committee.

The dissertation should be preceded by a short summary and commence with a brief introduction and some account of the current state of knowledge. Where practicable it should follow the general format of a paper in *JOCCA*.

The dissertation may be a review of a subject, a theoretical treatment, descriptive of practical work or a combination of these. It must indicate that the candidate has a reasonably wide and up-to-date knowledge of his chosen subject and understands the basic scientific principles underlying it, and that he is able to think critically and constructively.

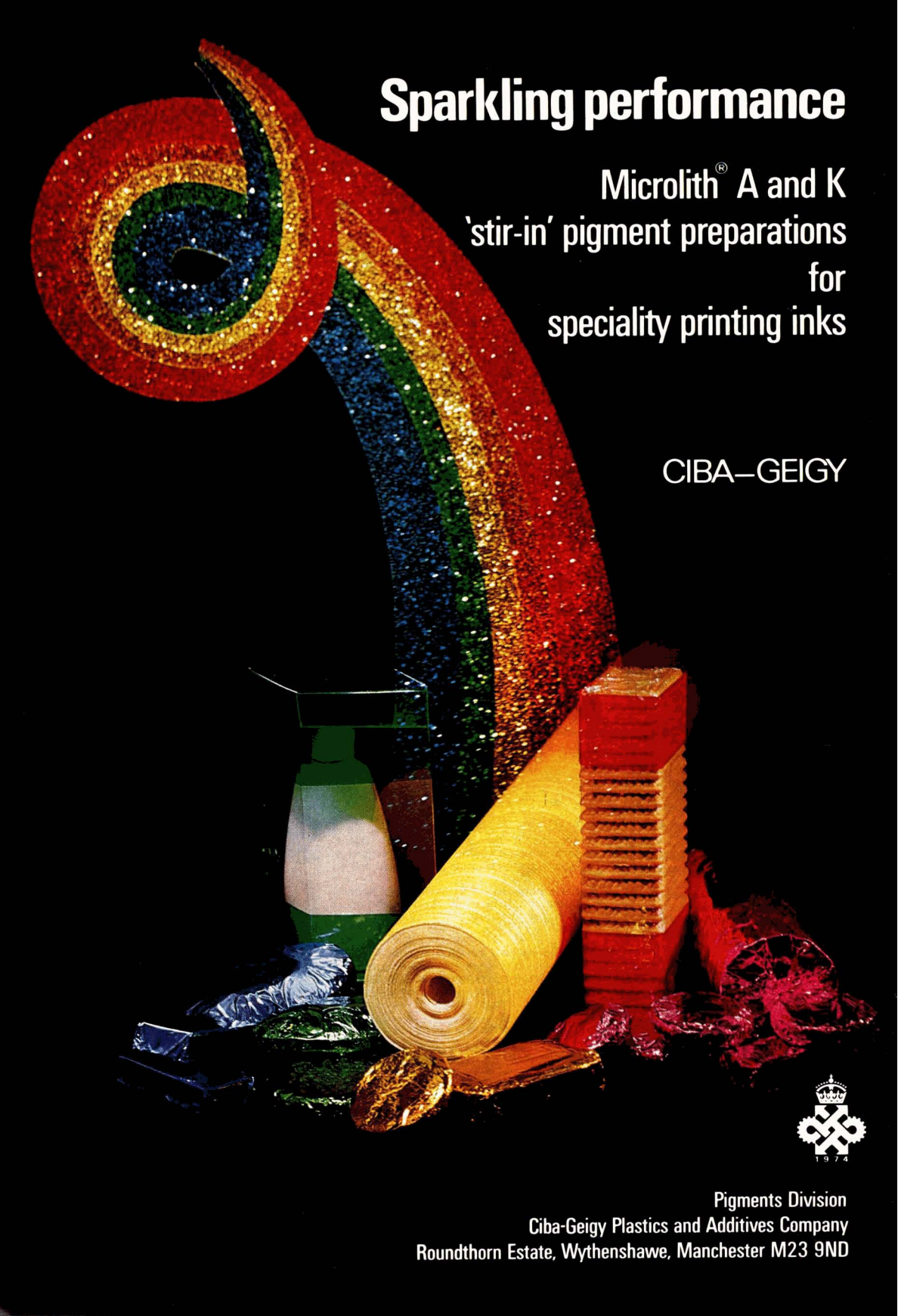
Where practical work is described, some attempts should be made to draw theoretical conclusions or to form some provisional hypothesis, together with an outline of what further work would be required to confirm the views put forward or further to advance the knowledge of the subject.

Where the dissertation is a review or a theoretical treatment, there should be an attempt to contrast and compare any opposing views expressed in earlier works, to examine the views critically, to suggest any compromise interpretation, to account for all the known facts and to outline any further work by which the opposing views could be tested.

Where applicable, references should be given to published work, graphs, diagrams etc. to be appended.

Length: Text should be approximately 5 000 words.

Applicants should refer to the paper by Moss which appeared in the January 1973 issue; the Professional Grade Committee feels that candidates for the Licentiate grade could with advantage use this paper as a model for their dissertations.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Transactions and Communications

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Transactions and Communications

Antifouling paints based on organotin compounds. Leaching of organotin toxins from paint films

By L. Chromy* and K. Uhacz**

*Institute for Chemistry, Silesian University, Katowice, Poland. **Paint Research Institute, Gliwice, Poland

Summary

The results of work on leaching of organotin compounds, bis-(tri-n-butyltin) oxide and tri-n-butyltin acetate, from antifouling paint films is described.

The solubility of these toxins in distilled water and in an aqueous solution of sodium chloride was determined.

Critical leaching rates of toxins were evaluated by making comparisons of the leaching characteristics with the biological properties of the coatings.

Keywords

Types and classes of coatings and allied products

antifouling coating

Properties, characteristics and conditions primarily associated with

dried or cured films

leaching

Raw materials

binders (resins, etc)

vinyl resin

prime pigments and dyes

iron oxide

biologically active ingredients

organotin

tributyl tin oxide

Peintures "antifouling" a base des composés organostanniques. L élution des composés organostanniques a partir des enduits "antifouling"

Résumé

On a mis au point la détermination de la dissolution des composés organostanniques en la solution aqueuse et ensuite en la solution aqueuse de chlorure de sodium.

Par la comparation du degrée d'élution avec les 'antifouling' propriétés on a déterminé la valuer critique d'élution.

Les composés étudies; l'oxyde de bis-/tri-n-stannbutyle; et l'acetate de tri-n-stannbutyle.

Antifoulingfarben auf Basis organischer Zinnverbindungen. Über das Auslaugen von organischen Zinnverbindungen aus Antifouling—Kompositionen

Zusammenfassung

Es wurde das Auslaugen von organischen Zinnverbindungen aus Antifouling-Kompositionen untersucht. Die Löslichkeit des bis-/tri-n-Butylzinn/Oxides und tri-n-Butylzinnacetats in distilliertem Wasser sowie in wässeriger Lösung des Natrium Chlorides wurde bestimmt.

Die kritischen Auslaugenswerte wurden berechnet.

Introduction

The period of effective action of antifouling paints, generally, depends on biological properties of toxins used and on the manner in which they are leached from the coatings.

The relative toxicity of toxins used in antifouling coatings may be correlated with their critical leaching rates. The value of critical leaching rate of toxin is usually expressed as the amount of toxin which has to be leached from a given area of coating during a defined period of time in order to prevent the attachment of sessile organisms.

The determining of critical leaching rate of various toxins is, therefore, important for the proper formulation of effective antifouling compositions. Moreover, the study of the leaching process of toxins from paint films provides useful information regarding the antifouling effect of the coatings.

Leaching of toxins from the paint films in a natural sea environment depends on physico-chemical properties of the toxins and on physico-chemical properties of the coatings.

In this paper, the results of work on the leaching of organotin compounds: bis(tri-n-butyltin) oxide and tri-n-butyltin acetate from antifouling paint films are described. The solubility of these toxins in distilled water and solutions of sodium chloride were determined. Critical leaching rates of toxins were evaluated by comparing leaching characteristics with biological properties of coatings.

Experimental

Refs. 1–2

The determination of microgram amounts of bis-(tri-n-butyltin) oxide, (TBTO) and tri-n-butyltinacetate (TBTA) was performed using methods described in earlier papers.[1,2]

Solubility of organotin compounds in water and solutions of sodium chloride.

Saturated solutions of organotin compounds in distilled water and in solutions of sodium chloride were prepared by stirring about 2g of the compound with 200ml of water or a solution of sodium chloride. The mixture was heated to 60°C and left overnight at room temperature. Saturated solutions of compounds were decanted from the undissolved excess and filtered through hard analytical paper. The concentration of organotin compounds in the solutions was then determined.

Solubility of organotin compounds in water and solutions of sodium chloride is given in Table 1 and in Figure 1.

Table 1
Solubility of organotin compounds

Compound	solubility (times 10^{-6} gl^{-1}) in					
	water	solutions of sodium chloride				artificial sea water
		0.5%	1%	2%	3%	
TBTA	256	9.2	6.0	5.1	4.9	5.0
TBTO	19.5	2.0	1.8	1.45	1.4	1.4

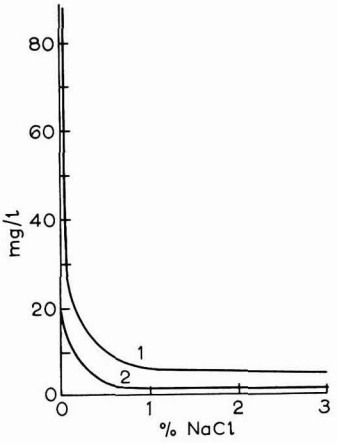

Fig. 1. Solubility of TBTA (1) and TBTO (2) in solutions of sodium chloride

As expected, the solubility of organotin compounds decreases with an increase of sodium chloride concentration in water. Within the range of sodium chloride concentrations in water from 0.5 to 3 per cent, which also covers the concentration of electrolytes in artificial sea water, the solubility of organotin compounds is almost constant.

The corresponding values are as follows:

1–2 $\times$ 10^{-6} gl^{-1} for TBTO, and 5–10 $\times$ 10^{-6} gl^{-1} for TBTA

The above mentioned range of solubility can, therefore, be taken as the solubility of TBTA and TBTO in sea water.

Leaching of organotin compounds from paint coatings in water and solutions of sodium chloride.

Distilled water and 2 per cent solution of sodium chloride were used for leaching the toxins from the paint films.

The above concentration of leaching solutions were chosen for following reasons:

(a) Process of leaching of toxins from paint films into 2 per cent solution of sodium chloride should be similar to that into sea water because the solubility of the organotin compounds is in the same range.

(b) It was expected that the process of leaching using distilled water would be accelerated because of greater solubility of organotin compounds in water than in solution of sodium chloride.

Characteristics of paints and sample preparation

Vinyl paints (insoluble matrix) pigmented with red iron oxide at PVC 10 and 30 containing 15 per cent by weight of toxin on dry paint film weight were used. Paints were applied to both sides of 7.5 $\times$ 2.5 cm glass panels. The dry paint film weight on the panels selected for leaching study was approximately 250 mg which corresponds to a dry paint film thickness about 30–50 micrometers.

Leaching of toxins from the paint films

The glass panels coated with antifouling compositions were immersed in 500 ml of leaching solutions (water and 2 per cent solution of sodium chloride).

The leaching process was performed under static conditions (without mixing) during 150 days.

The leaching solutions were changed every 10 days. In the leaching solutions the concentration of organotin compounds was determined using methods described previously. The concentration of compounds reached in the leaching solutions was always lower than that of the saturated solution.

From the concentration of toxin found in the leaching solutions the amount of toxin, (A), leached from coating during subsequent extractions was calculated. The results of leaching of the toxins are shown in Figures 2 and 3 where the sum of toxin leached (ΣA) was plotted against time of extraction. Figures 4 and 5 show the calculated leaching rates of toxins plotted against time of extraction.

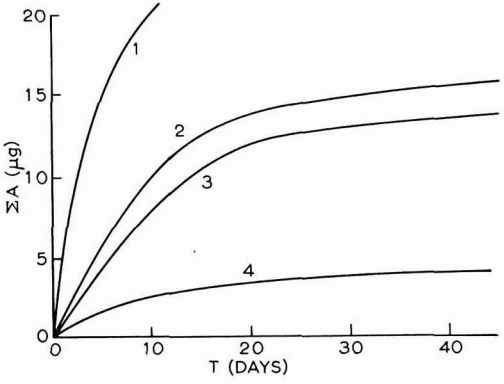

Fig. 2. Leaching of organotin compounds from paint films in distilled water. ΣA μg = f(T) days

(1) TBTA, PVC = 30 (3) TBTO, PVC = 30
(2) TBTA, PVC = 10 (4) TBTO, PVC = 10

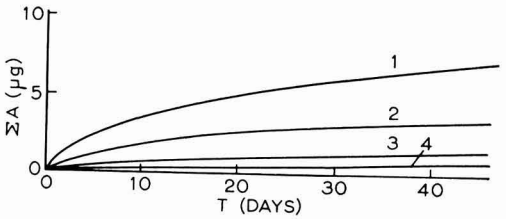

Fig. 3. Leaching of organotin compounds from paint films in 2 per cent solution of sodium chloride. ΣA μg = f (T) days
(1) TBTA, PVC = 30 (3) TBTO, PVC = 30
(2) TBTA, PVC = 10 (4) TBTO, PVC = 10

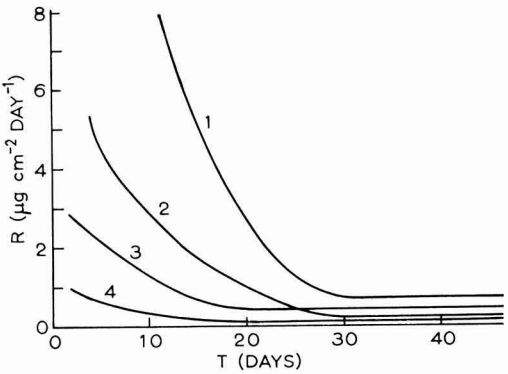

Fig. 4. Leaching of TBTA and TBTO from paint films in 2 per cent solution sodium chloride. R μg cm^{-2} day^{-1} = f (T) days
(1) TBTA, PVC = 30 (3) TBTO, PVC = 30
(2) TBTA, PVC = 10 (4) TBTO, PVC = 10

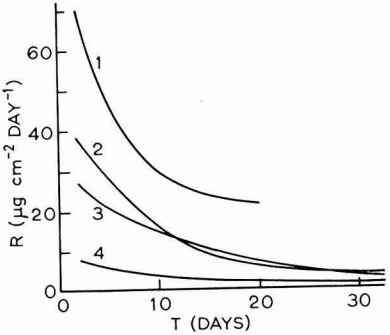

Fig. 5. Leaching of TBTA and TBTO from paint films in distilled water. R μg cm^{-2} day^{-1} = f (T) days
(1) TBTA, PVC = 30 (3) TBTO, PVC = 30
(2) TBTA, PVC = 10 (4) TBTO, PVC = 10

The process of leaching presented in Figures 2–5 may be characterised as follows:

After the initial stage, where a rapid decrease of leaching rate is observed, the function becomes linear, which means that the process of leaching becomes steady. The value of leaching rate and amount of toxin leached out from the coatings depends on the solubility of toxin in the leaching solution and on pigment volume concentration in the paint film.

Performing the process of leaching in sodium chloride solution, it was found that the leaching rate remains constant after an equilibrium rate is reached. During about 100 days run there was observed no significant decrease in leaching rate.

The relatively rapid decrease in leaching rate occurs when the process is performed in distilled water. The leaching rate of toxin falls to zero after the reserve of toxin in the paint film has been removed.

The decrease in leaching rate during the period of extraction is more pronounced for paints of higher PVC.

The constant values of leaching rates of toxins from the paint films in sodium chloride solution are shown in Table 2. The leaching process in distilled water is about 10–20 times more rapid than in solution of 2 per cent sodium chloride. Therefore, this may be considered as an accelerated method for the evaluation of the properties of paints based on organotin compounds and insoluble matrix.

Table 2
Constant values of leaching rates of toxins

Characteristics of paint film		Leaching rate in 2% solution NaCl
PVC	Toxin	R (μg cm^{-2} day^{-1})
30	TBTA	0.7
10	TBTA	0.2
30	TBTO	0.4
10	TBTO	0.1

Biological properties of paint films based on organotin compounds

In order to correlate the antifouling properties with the leaching characteristics of toxin from the paint films, the paints described above were tested in natural marine environments.

Assessment of biological properties were performed in an under-water testing station of the Institute located in Wladyslawowo (Baltic Sea).

The samples for test were prepared by applying the antifouling paints (60–80 micrometers dry film thickness) to sandblasted steel plates 320 × 240mm coated previously with suitable anticorrosive primer Coated panels were immersed about 1·5m below the average sea level for 15 months from May until August of the following year Biological characteristics at the testing station are as follows:

—seasonal sequence of fouling: June to August,

—main fouling organisms: diatoms, algae, barnacles, encrusting, bryczoa, mussels, hydroides,

—average weight of fouling organisms (wet): 17kg m^{-2} year^{-1}

The antifouling properties of tested paints after the period of immersion with respect to the main animal growth are given in Table 3, together with constant values of leaching rates, R, of toxins in 2 per cent solution of sodium choloride.

The antifouling properties were assessed by a numerical scale, 10–1, where 10 means no animal growth present on tested surface, and 1 indicates the surface completely fouled.

Table 3

Antifouling properties of the paints

PVC of the paint	toxin	antifouling properties	leaching rate R ($\mu g\ cm^{-2}\ day^{-1}$)
30	TBTA	9	0.7
10	TBTA	1	0.2
30	TBTO	10	0.4
10	TBTO	2	0.1

The critical leaching rate necessary to prevent fouling is not greater than 0·4$\mu g\ cm^{-2}\ day^{-1}$ for TBTO and 0·7$\mu g\ cm^{-2}\ day^{-1}$ for TBTA, assuming that:

(a) the leaching process of toxins in natural sea environments resembles the process performed in 2 per cent solution of sodium chloride,

(b) after the process has reached the steady state, the value of leaching rate remains constant or does not increase.

Discussion

The antifouling properties of paints based on organotin toxins depend on leaching rate of the toxins from paint films during the period of exposure in natural environments.

The values of critical leaching rate of organotin compounds necessary to prevent fouling are about 10–20 times lower than the generally accepted value for copper compounds (10$\mu g\ cm^{-2}\ day^{-1}$).

[*Received 25 April 1977*

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1. Chromy, L. and Uhacz, K., *J.O.C.C.A.*, 1968, **51**, 494.
2. Chromy, L., Mlodzianowska, W., Uhacz, K. and Warchol, R., *J.O.C.C.A.*, 1970, **53**, 121.

J. Oil Col. Chem. Assoc. 1978, **61**, 43-48

2-Furfuraldehyde (furfural) formation from local by-products and its utilisation in the field of surface coatings

By S. M. Saad, A. M. Naser, M. T. El-Zimaity and H. F. Abdel-Meged

Chemistry Department, Faculty of Science, Al-Azhar University, Nasr City, Cairo, Egypt

Summary

Acid hydrolysis of the Egyptian bagasse pith to produce furfural under mild conditions was carried out. Hydrochloric acid is found to be the most efficient medium for the hydrolysis. A resole-type of phenolic resin containing furfural is prepared in situ after the hydrolysis. Preliminary evaluation of the resin in order to assess its suitability for surface coatings formulations gave promising results.

Keywords

Raw materials for coatings
binders (resins, etc.)
furfural resin
phenolic varnish

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

dried or cured films
gloss
water resistance
acid resistance
alkali resistance
scratch resistance

La préparation de furfurol à partir des sous-produits indigènes, et son utilisation dans le domaine des revêtements

Résumé

On a effectué l'hydrolyse acide, sous des conditions légères, de la poix de bagasse, afin de produire le furfurol. On a trouvé que l'acide chlorhydrique est l'agent de hydrolyse le plus éfficace. Après l'hydrolyse on a préparé *én situ* une résine furfurophénolique du type résole. Une évaluation préliminaire de la résine en vue d'apprécier sa valeur en tant que constituant de revêtements a donné des résultats encourageants.

2-Furfuraldehyd-(Furfural-) Bildung aus an Ort und Stelle gefundenen Nebenprodukten, und dessen Einsatz auf dem Anstrichmittelgebiet.

Zusammenfassung

Saure Hydrolyse von ägyptischem Zuckerrohrmark wurde unter "milden Bedingungen" zwecks Erzeugung von Furfural vorgenommen. Es wurde festgestellt, dass Salzsäure das wirksamste Agens für die Hydrolyse ist. Nach erfolgter Hydrolyse wird ein Phenolharz vom Resol-Typ in situ hergestellt, welches Furfural enthält. Vorläufige Beurteilung des Harzes auf seine Eignung für Verwendung im Anstrichmittelaufbau verspricht günstige Resultate.

Introduction

Refs. 1–7

The importance of 2-Furfuraldehyde (furfural) and its derivatives has increased due to the developments of the chemical industry. The use of furfural has been described for the following applications [1]:

(i) A selective solvent in the purification of butadiene, lubricating oils, wood rosin, tall oil and sulfate turpentines.

(ii) An extraction solvent for acetic acid and glyceride oils.

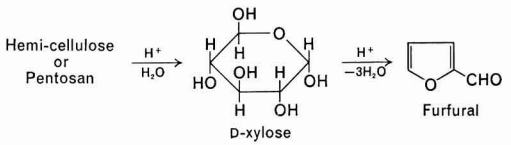
(iii) A solvent for coating materials, dyes, gums, resins, nitrocellulose and cellulose acetate.

(iv) A wetting agent.

(v) In the preparation of dyes, plastics and maleic acid.

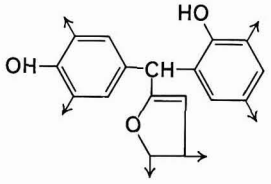
(vi) A key compound for the preparation of α-furan and α-furan derivatives.

It seems that there is as yet no specific industrial method described for the synthesis of furfural and attempts at its synthesis have failed [2]. As a matter of fact, furfural is exclusively produced by the hydrolysis of plant by-products, eg. corn cobs, rice hulls, bagasses etc. These by-products accumulate as wastes in the processing of agricultural products. These pentosan-containing cereals yield furfural upon treatment with acids according to the following equation [3]:



The furfural formation is conducted under a wide variety of experimental conditions ranging from acid impregenation to high temperature and pressure conditions. Its method of production has been described in detail in many text-books and there is no need for repetition here.

Furfural is the only aldehyde, other than formaldehyde, which has gained commercial importance in the preparation of aldehyde resins. It contributes special and useful flow properties to commercial phenolic resins. These resin-forming properties were noticed as early as 1940 by Stenhouse[4] and Fownes[5]. The presence of two double bonds in furfural gives various advantages over formaldehyde with respect to its reactions with phenol. It behaves as a saturated aldehyde until the final stages of curing are reached and then cross-links via the double bonds. Thus, the molecule exhibits a functionality greater than two and two different reactions occur; firstly a condensation reaction at the ortho and para-reactive hydrogens with the elimination of water, and then vinyl-type polymerisation. A typical unit in a cross-linked cured structure may be represented by[6,7]:



Furfural has other features which enhance its use as a replacement for formaldehyde, such as better resin yield, its excellent qualities as a plasticising agent, considerable improvement in the flow properties of the final compositions and high wetting and pentrating properties. Furfural also plays an important role in the production of phenolic varnishes. Its use in combination with formaldehyde improves greatly the electrical and mechanical properties of the resultant phenolic varnish. They also find application as binders or constituents of friction compositions used in automotive brakes and clutches.

This paper deals with the determination of the optimum conditions for the furfural formation from acid hydrolysis of the Egyptian bagasse pith. The preparation *in situ* of a resole type phenol/furfural-formaldehyde condensate is another aspect taken into consideration.

Experimental

Materials

Bagasse pith was kindly provided from Edfo pulp mill, Egypt, A.R.E. Bituminous material was kindly provided by El-Seuz Petroleum Company. It is characterised by a flash point of 288°C and shows 99·99 per cent solubility in carbon tetrachloride.

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

Methods and techniques

1. Treatment and analysis of bagasse pith

The pith was thoroughly mixed, to ensure complete homogenity, followed by screening over 60 mesh screen to separate the fibres which were rejected. The chemical constitution of the pith was determined according to Tappi standards[8].

2. Acid hydrolysis of the pith

A known weight of the raw material was refluxed with fifty times its weight of standard acid in a 250 ml round-bottom flask. Heating was performed in a sand-bath to avoid local over heating, which may result from direct heating and leads to considerable loss of the furfural formed.

The heating time was recorded immediately on reaching boiling. After the pre-determined hydrolysis time, the flasks were removed and allowed to cool. The reaction mixture was filtered, washed with the same standard acid and made up to the required volume.

The acids employed in this study include hydrochloric, sulfuric and phosphoric acids within a concentration range of 1·5N to 5·5N and for various hydrolysis times ranging from 30 minutes to four hours.

3. Furfural estimation

An aliquot portion (25 ml) of the hydrolysable solution was transfered to a 250 ml conical flask. 20 ml of 0.05 N potassium bromate/bromide solution was added and the mixture was allowed to stand for five minutes. A solution of potassium iodide (5 per cent) was then added and the liberated iodine was titrated against standard sodium thiosulfate solution. A blank experiment was conducted under similar conditions[9].

4. Phenol/furfural-formaldehyde condensate

To the acidified solution containing the extracted furfural (0·05 mole), phenol (0·1 mole) was added and the mixture boiled under reflux until the development of a highly coloured product. The reaction mixture was cooled and 30 per cent formaldehyde (0·06 mole) was added, and the temperature was raised to about 80°C and maintained until the formation of a dark resinous product. Water was removed under reduced pressure[10].

5. Preparation of air-drying varnish

95 gm of linseed oil was placed in a 250 ml three-necked flask equipped with an efficient stirrer, thermometer and insert gas inlet, and heated to 250°C with the aid of a heating mantle. Five grams of the phenolic resin was added portionwise in small increments over a period of two hours. Heating was continued under stirring until complete compatibility was achieved. This can be clearly shown by the transparency of a cold drop of the mixture on a glass plate. After the mixture had been cooled to 150°C, xylene was added to obtain 75 per cent solids followed by more thinning with a petroleum fraction (60-80°C) to adjust the solids to 50 per cent. The varnish was filtered and cobalt and lead naphthenate driers were added in amounts of 0.005 per cent and 0·01 per cent respectively.

6. Preparation of modified bituminous coating composition

In a similar manner to that described above a bituminous coating was prepared by the incorporation of the phenolic resin with local asphalt at 200°C. In this case, the mixture was heated for four hours longer under constant stirring. The ratios of phenolic resin/bituminous material were 15 per cent and 25 per cent.

7. Evaluation techniques

The viscosity[11], drying times[12], gloss[13], water[14], acid[15], alkali[16] and corrosion resistances[17] and scratch hardness[18]

determinations were carried out according to standard methods described in the literature references given.

Results

Refs. 18, 19

The main purpose of this work is concerned with the possibility of utilising some local by-products. Amongst these by-products are the bagasse pith obtained during sugar manufacture and the bituminous material obtained in petroleum industry. Both are produced as waste products in local factories.

The bagasse pith was subjected to chemical analysis and the results obtained are given in Table 1.

Table 1
Chemical analyses of the bagasse pith

Alcohol/benzene solubility	7.5%
α-cellulose	53.7%
Lignin	20.2%
Pentosan	27.9%
Ash	6.6%

In order to study the furfural formation by acid hydrolysis of the bagasse pith, three main parameters, namely type of acid, acid strength and hydrolysis time were taken into consideration. It is worth a mention that throughout the work, the standard deviations were estimated to test the reproducibility of the results obtained, which proved to be of small value within the permissible experimental error[18]. For this reason preliminary trials were first conducted to determine the effect of solid-acid ratio on the yield of furfural. From such a study, the ratio of 1 : 50 (by weight) gave the optimum yield of furfural and this was used throughout the investigation. Such a solid-acid ratio was in good agreement with the data of previous work[19].

In another set of experiments different acid concentrations were used and the corresponding furfural yield was estimated after various periods of reflux. The data obtained is shown in Figure 1. From this Figure, it can be clearly seen that increasing the acid normality leads to an increase in the maximum yield of furfural.

The above data was replotted in Figure 2 to show the effect of reflux time on the rate of furfural formation. The main observation from this figure is that the yield of furural increases with increased hydrolysis time. Acid hydrolysis by hydrochloric acid behaves differently from sulfuric or phospheric acids. The yield of furfural increased with increased acid normality to 2·5 N, where the maximum furfural yield of 9·48 per cent was reached after three hours. Higher acid normalities resulted in a decrease in the yield of furfural at longer hydrolysis times. This can be attributed to the side reactions that may occur; an effect which is not encountered when using either sulfuric or phosphoric acids. However, using 3·5 N acid, the maximum yield of furfural (9·15 per cent) was obtained earlier, after two hours hydrolysis, but 10·91 per cent furfural was obtained after one hour's hydrolysis in case of 4·5 N acid. Increasing the acid normality to 5·5 N, the maximum yield of furfural was found after 30 minutes hydrolysis time (11 per cent).

The most important conclusion drawn from Figures 1 and 2 indicates that the highest furfural yield was given by hydrochloric acid followed by sulfuric acid and the lowest value was given by using phosphoric acid. The work of Kinovalov and Sharkov[20] who studied the conversion of xylose to furfural confirms these results.

The work was extended to include a kinetic study in order to show the effect of hydrogen ion activity on the rate of furfural production. For this reason, the initial concentration of both

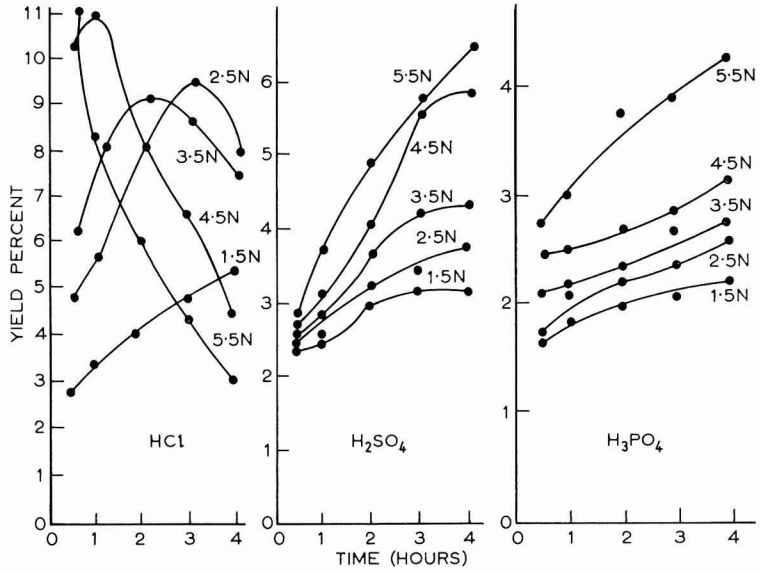


Fig. 1. Effect of hydrolysis time upon the yield of furfural at different acid concentrations

the raw material (pentosan) and of the acid were kept constant. The total amount of furfural obtained from the raw material, by distilling off with 12·1 per cent hydrochloric acid in presence of sodium chloride, was found to be 17·08 per cent. This value was considered to be equivalent to the maximum conversion of pentosan to furfural, and therefore it was referred to as the initial concentration (a). The percentage conversion of pentosan to furfural by refluxing using different acids and at times of conversion (t) was referred as (x).

Table 2
Reaction constants of pentosan conversion

Acid	Normality	Reaction constant (k) $\times 10^{-5}$	Half-life period ($t_{0.5}$) hours
HCl	1.5	1.70	11.3
	2.5	4.02	4.7
	3.5	5.76	3.3
H_2SO_4	1.5	0.68	28.3
	2.5	0.74	26.0
	3.5	1.27	15.3
	4.5	1.98	9.7
	5.5	2.54	7.6
H_3PO_4	1.5	0.30	64.6
	2.5	0.37	51.9
	3.5	0.42	54.8
	4.5	0.44	43.9
	5.5	0.95	20.2

Figure 3 shows a straight line relationship between log a/a-x versus (t), from which the calculated values of the reaction constant (k) and half-life period ($t_{0.5}$) are collected in Table 2.

It can be clearly seen from the data given in Table 2 that, in general, increasing the acid concentration leads to an increase in the rate constant (k). This means that the acid concentration has a pronounced effect on the rate of furfural formation. This generalisation is valid for sulfuric and phosphoric acids within the limits of acid concentaration investigated. However, there is some limitation with regard to hydrochloric acid, where the maximum yield of furfural depends upon both acid concentration and time of conversion due to the interference of side reactions. This becomes noticable at acid concentration higher than 3·5 N and at more than two hours hydrolysis time.

Analogous observations were also drawn from this kinetic data with regard to the values of the reaction rate constant (k). Hydrochloric acid has the highest value, followed by sulfuric acid, whilst phosphoric acid shows the lowest value.

Another set of experiments was performed to determine the degradation effect of the acids on pentosan in the bagasse pith after the hydrolysis. This was carried out by measuring the pentosan percentage in the residue of the pith after the acid hydrolysis. The results obtained are given in Table 3.

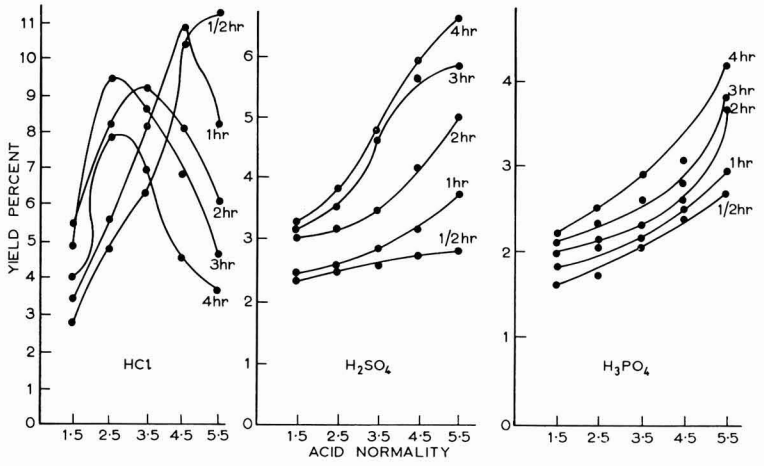


Fig. 2. Effect of acid concentration upon the furfural yield after various hydrolysis times

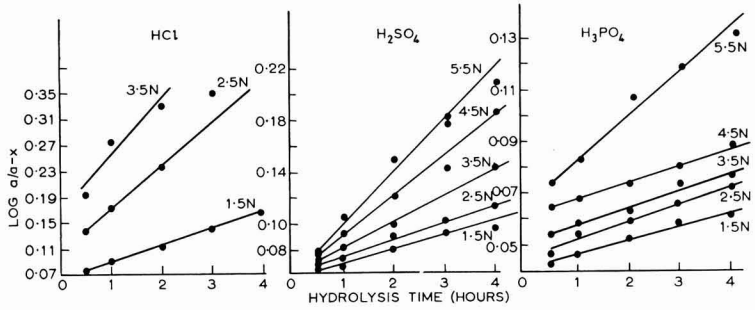


Fig. 3. $Log_{10}(a/a-x)$ vs. time at various acid concentrations

Table 3

Pentosan percentage in pith residue after hydrolysis

Acid	Hydrolysis Time (hours)	Normality 1.5	2.5	3.5	4.5	5.5
HCl	0.5	5.78	2.41	1.52	1.52	0.99
	1	4.04	1.86	1.43	1.43	0.96
	2	2.70	1.48	1.00	1.00	0.83
	3	2.30	1.35	0.90	0.90	0.47
	4	1.79	0.93	0.76	0.76	0.21
H_2SO_4	0.5	9.55	9.42	7.95	7.28	3.38
	1	8.10	7.66	6.92	5.94	2.70
	2	6.07	5.57	4.60	2.95	2.07
	3	6.60	5.46	4.28	2.21	2.01
	4	5.18	4.06	3.28	1.81	1.61
H_3PO_4	0.5	23.41	19.27	16.19	12.72	9.58
	1	18.75	14.23	12.37	9.99	8.70
	2	14.36	11.71	10.66	7.17	6.60
	3	11.31	10.70	8.43	6.89	5.89
	4	10.44	8.98	7.92	6.75	4.98

An opposite feature was seen in this study indicating that the pentosan percentage in the pith residue after hydrolysis was the highest in case of phosphoric acid, whereas it was of the lowest value in case of hydrochloric acid. These results can be easily explained by the high reactivity of hydrochloric acid.

After the determination of the most suitable conditions for maximum furfural formation from the hydrochloric acid hydrolysis of the Egyptian bagasse pith, attention was directed towards its utilisation in resin preparation. It was felt of interest to combine the useful properties imparted by using furfural and the preparation *in situ* of soluble resin. For this reason, a resole type phenolic/aldehyde resin was prepared in which furfural partially replaced formaldehyde.

The prepared phenol/furfural-formaldehyde condensate is characterised by a black oily appearance at room temperature, yielding a product which solidified upon cooling. The resin is soluble in acetone and insoluble in toluene.

Preliminary trials for the utilisation of the resins prepared in the field of surface coatings showed:

(a) Compatibility of the phenolic resin with drying linseed oil

It was conducted by the incorporation of 5 per cent resin with the oil and resulted in the formation of an amber viscous product. The modified oil showed a high degree of miscibility with xylene and petroleum fraction (60-80°C). The relationship between the resin content and viscosity of the varnish is shown from the data given in Table 4.

Table 4

Solid content and viscosity relationship

Solid content (%) in xylene/pet. fraction (1/1)	Viscosity (Ford cup No. 4) seconds
60	110
55	100
50	90
45	65
40	35
35	15
30	10

The data given in Table 4 clearly illustrates the effect of phenolic resin in bodying linseed oil, especially when comparison is made with that of the unmodified oil (15 seconds).

To the 40 per cent solids varnish, cobalt and lead naphthenate driers were added in amounts of 0·005% and 0·10% respectively. The characteristics of the dried films were determined and are given in Table 5.

Table 5

Characteristics of modified linseed oil

Drying time	
air-drying touch dry	8 hours
hard dry	16 hours
stoved at 150°C	30 minutes
Gloss	100%
Water resistance	Excellent
Acid resistance	Excellent
Alkali resistance	Good
Scratch hardness (20μ thick)	over 500 grams load

(b) Modified bituminous compositions

The modifying effect of the phenolic resin was examined with bituminous materials obtained during the processing of petroleum. The modified coating compositions are soluble in xylene and a fine residue remains after complete dissolution. This residue increases with increasing amounts of phenolic resin incorporated, and it is not soluble in aliphatic or aromatic solvents, which may be due to the formation of cross-linked products.

Two different bituminous compositions were formulated corresponding to 6 and 10 per cent phenolic resin respectively. The formulations also contained 34 per cent and 30 per cent bitumin respectively. Films of both compostions were coated over suitable panels and the dried films were subjected to the usual testing procedures. The result obtained are given in Table 6.

Table 6

Characteristics of bituminous coatings

	Type I (6% phenolic)	Type II (10% phenolic)
Viscosity at 25°C for 40% solids in xylene/pet. fraction	15 seconds	50 seconds
Drying time	within 24 hours	within 24 hours
Water resistance	excellent	excellent
Acid resistance	excellent	excellent
Alkali resistance	excellent	excellent
Corrosion resistance	excellent	excellent
Film homogenity	Good	Good

The important conclusion drawn from these preliminary studies shows the possibility of utilising the extracted furfural in various coating compositions. Furfural imparts remarkable and outstanding properties to modifying drying oils and bitumen and further detailed evaluation is under investigation.

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Identification of shellac

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Summary

Jalaric and laccijalaric acids, which have the aldehydic function in them, are two constituent acids of shellac. Taking advantage of their presence in the aqueous mother liquor remaining after acid decomposition of the alkali hydrolysate of the resin, a spot test with fuchsin-sulfurous acid has been developed for the identification of shellac, either as such or in admixture in various lac based formulations.

Keywords

Types and classes of coatings and allied products

shellac

Supplies and other materials primarily associated with analysis, measurement or testing

analytical reagent

Raw materials for coatings

binders (resins, etc.)

natural resin

L'identification de gomme-laque

Résumé

Les acides jalarique et laccijalarique, qui tous les deux renferment le groupement aldéhydique, sont deux des constituants acides de gomme-laque. On a profité de leur présence dans la liqueur-mère aqueuse, subsistant après la décomposition par acide de l'hydrolysat alcalin, pour développer un essai par touches utilisant la fuchsine et l'acide sulfureux et permettant de l'identification de gomme-laque soit seule, soit melangée avec les autres constituants des divers revêtements à base de gomme-laque.

Identifizierung von Shellac

Zusammenfassung

Jalarische und Lac-jalarische Säuren, welche die Aldehydfunktion darin einnehmen, sind die beiden Säurebestandteile des Shellacs. Unter Benutzung ihrer Gegenwart in der wässrigen Mutterlauge, welche nach der Zersetzung des alkalischen Hydrolysates des Harzes übrig bleibt, wurde ein Tüpfeltest mit Fuchsin-Schwefliger Säure zur Identifizierung von Shellac als solchem oder als Bestandteil in verschiedenen Lac enthaltenden Vorschriften entwickelt.

Refs. 1–5

Shellac, the versatile natural resin, possesses a rare combination of many valuable and desirable properties and consequently finds innumerable industrial uses, especially in various adhesives, cements, varnish and lacquer formulations, either as such, in modified form, or in conjunction with other natural or synthetic resins. Its identification has been attempted by various investigators and the methods adopted have been reviewed[1], but none of these tests could serve the purpose with certainty even on pure samples. The test becomes more difficult when shellac is present in an admixture with other natural and/or synthetic resins.

Shellac is always associated with an odoriferous compound, a wax and a mixture of dyes such as erythrolaccin, *iso*-erythrolaccin and desoxyerythrolaccin which are hydroxyanthraquinone derivatives. Shellac when heated, either dry, moist or in aqueous alkaline solution, gives a characteristic honey or musk-like odour. Due to the presence of the dyes, shellac gives a characteristic colour reaction with alkali. It also gives colour reactions with other reagents. However, according to Vollmann[2], these are of very little significance as the colours obtained under the conditions of the tests differed with different grades of shellac. He, therefore, advocated the colour test obtainable with alkali. This colour test, however, may not be so prominent with decolourised shellac and bleached lac or when shellac is present in admixture. Of course, it can be detected in admixture by smell on heating provided it is present in an appreciable amount. Taking various factors into consideration, Bhattacharya[3] recommended the isolation of hot water insoluble zinc salt of shellolic acid, a constituent acid of shellac, from the alkaline hydrolysate of the resin mixture. Vollmann[2], on the other hand, laid stress on the isolation of aleuritic acid from shellac after its separation as sodium aleuritate from the alkaline hydrolysate. The preparation of a zinc salt of shellolic acid or isolation of aleuritic acid as recommended is very laborious and time consuming. Moreover, when the percentage of shellac present is low, it becomes very difficult. It will thus be evident that there is no simple test, apart from smell when heated and colour reaction in presence of alkali, for its identification either alone or in admixture. Attempts have, therefore, been made to develop a rapid, simple and reliable test for its identification.

The functional groups present in shellac are carboxyl, aldehyde and hydroxyl including vicinal hydroxyl, and the probable linkages ester, acylal, acetal and ether. Other resins taken into consideration during this study were rosin, damar, sandarac, gamboge, kauri, pontianac, dragon's blood, benzoin and mastic. Most of these resins also have carboxyl and hydroxyl groups as they have measurable acid and hydroxyl values. Shellac very easily undergoes periodic acid oxidation, which is specific for vicinal hydroxyl groups and also to Tollen's reagent, due to the presence of the aldehyde group giving a beautiful silver mirror. Most of the other resins tested

also responded to these two tests. Hence the identification of shellac by means of its free functional groups is not possible.

On hydrolysis, the constituent acids of shellac are liberated and consist mainly of hydroxy aliphatic and terpenic acids. The aliphatic acids are almost insoluble in water, whilst the terpenic acids are readily soluble and these are present almost in the proportion of 50:50. Of the aliphatics, the main constituent acid is aleuritic (I, ~ 35 per cent) and of the terpenics jalaric (II, ~ 25 per cent). Next in importance are butolic (III, ~ 8 per cent), shellolic (IV)/epishellolic (V) and laccijalaric (VI, ~ 8 per cent) acids. (See Fig. 1).

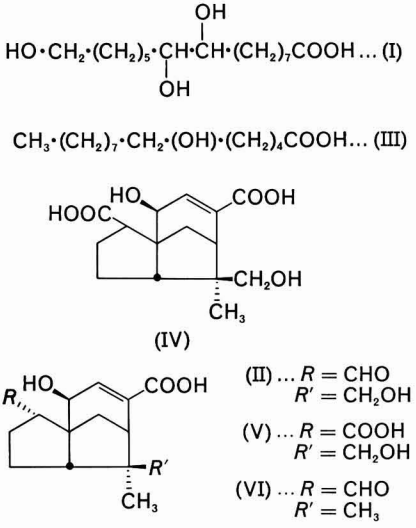

Fig. 1.

After hydrolysis of the shellac, the isolation and identification of aleuritic, butolic or shellolic/epishellolic acids is not feasible as has been mentioned earlier. Of course, the presence of aleuritic acid in the mixture of fatty acids can be identified by subjecting the mixture to von Rudloff's oxidation[4] with identification of the oxidised products as pimelic and azelaic acids by means of thin layer and/or gas liquid chromatography. This is again a time consuming process. Next remains the isolation and/or identification of the highly water soluble jalaric and laccijalaric acids which have aldehydic functions in the molecule. These acids could be very easily detected in the mother liquor, remaining after alkaline hydrolysis and subsequent decomposition with mineral acid, by means of Tollen's reagent, 2:4-dinitrophenyl hydrazine (2:4-DNPH) or fuchsin-sulfurous acid. In a similar way, other resins were also treated and the mother liquors subjected to these tests. The results are shown out in Table 1. It will be evident from the Table that 2:4-DNPH gave in all cases some coloured precipitate; the difference between shellac and other resins being that in case of shellac there was instantaneous formation of yellow precipitate, whilst in other cases orange to brown or simply brown precipitates formed after some time. In case of pure shellac, this test may be adopted, but in admixture it may not be very definite due to blending of colours. The other two tests were found to be positive only with shellac. Both are equally satisfactory and confirmatory even when shellac is present in admixture. Tollen's reagent requires a little time for the formation of silver mirror whilst fuchsin-sulfurous acid gives an immediate indication. On the basis of this observation, a quick, convenient and confirmative spot test[5] has been standardised for the identification of shellac which is as follows:

Table 1

Tests for aldehyde radical in the aqueous solution with different reagents

Resin	Tollen's Reagent	Fuchsin-sulfurous acid	2:4-Dinitrophenyl hydrazine solution
Shellac	Silver mirror is formed	Blue spot develops	Immediately a yellow precipitate is formed
Rosin	No mirror formation	No spot develops	After 30-40 seconds an orange precipitate starts forming, which turns brown
Sandarac	,,	,,	,,
Gamboge	,,	,,	,,
Damar	,,	,,	,,
Kauri	,,	,,	,,
Dragon's Blood	,,	,,	,,
Pontianac	,,	,,	Immediately colour changes to brown and after one minute gradually starts precipitating
Benzoin	,,	,,	After 1–2 minutes colour of the solution changes to brown and gradually starts precipitating
Mastic	,,	,,	,,

(a) *Pure shellac*

Powdered shellac (0.1-0.5 g) is taken in a test tube (20×2.5 cm) and hydrolysed on steam bath with 2-5 ml of 0.5N aqueous sodium or potassium hydroxide solution for one hour. After cooling to room temperature, the hydrolysate is treated with 0.5N aqueous sulfuric acid to make the solution just acidic to congo red paper. The aqueous portion is filtered through Whatman No. 40 filter paper and one drop of the clear filtrate added to a solution of freshly prepared fuchsin-sulfurous acid placed on a spot plate. The specific blue colouration[5] is developed within 0.5 to 2 minutes, indicating the presence of jalaric and laccijalaric acids in the filtrate.

(b) *Shellac in admixture*

(i) When shellac is present to the extent of 10 per cent or more in a physical mixture and 15 per cent or more in a fused one, 2 g of the powdered mixture is hydrolysed with 10 ml of 0.5N alkali for 3-4 hours and proceeded as before.

(ii) When shellac is present in lower percentages no appreciable colour development is observed. A slight modification of the method, however, gives the desired result. Here, 5 g of the sample is hydrolysed with 25 ml of the alkali for 3-4 hours. After acid decomposition of the hydrolysate and filtration, the aqueous filtrate is extracted with ether. Ether is removed by evaporation, and a few drops of water added to the residue. This aqueous solution is then used in the test. By this modified procedure shellac in admixture up to 3-4 per cent could be very easily detected.

In the above tests sulfuric acid has been found preferable to hydrochloric and nitric acids. The amount of acid added should not, under any circumstances, be in excess, because in that case a very weak blue colour then develops instead of a brilliant one. For Tollen's test, nitric acid should be used

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Big things are happening to ICI's phthalocyanine green pigments. We're already world leaders in the green pigment market... and now we're taking a giant step forward with a massive new £6m plant. It's a huge development—designed to double our capacity to an annual total of 3000 tonnes.

Today, the Monastral range of phthalocyanine green pigments is proving particularly valuable to the printing ink industry as a replacement for blue-yellow mixtures.

Monastral Green pigments produce cleaner, brighter shades than any alternative and give excellent all-round fastness. They're completely versatile and cover an entire spectrum of colours from bright yellowish-greens to bright bluish-greens. They also cover many grades to meet the requirements of all pigment users.

In fact, ICI's phthalocyanine greens are now used extensively for all types of inks, paints and plastics. And they're growing in popularity in other areas... especially for the coloration of paper, textile printing compositions, viscose rayon, artist's paints, powder coatings, waterproof canvas, and for soaps and detergents too!

Things really are happening to phthalocyanine green pigments—the biggest thing in greens... from ICI.

ICI Organics Division

Hexagon House, Blackley, Manchester M9 3DA

OD 143

for the decomposition of the hydrolysate. This test, however, has been found successful with pure shellac only.

Conclusions

The spot test has been found applicable to all grades of seedlac and shellac including bleached and garnet lacs. It has further been found that shellac can thus be detected when present in conjuction with various synthetic resins, other than the phenolic and amino resins where aldehyde groups are present.

When testing for shellac in varnishes and lacquers the resin should be precipitated by pouring in water, or in any other non-solvent, and washed repeatedly to remove the last traces, if any, of aldehydes present in the orginal solvent.

Acknowledgments

The authors are grateful to Dr T. P. S. Teotia, Director, for permission to publish the paper.

[*Received 1 March 1977*

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A new simple method of determining the oxirane oxygen content of vegetable oils

By B. M. Badran

Laboratory of Polymers and Pigments, National Research Centre, Dokki, Cairo, Egypt

Summary

The oxirane oxygen content of epoxidised linseed and epoxidised dehydrated castor oil has been determined by the N.M.R. technique. The method is especially suitable for oxirane values higher than 1.5 per cent. The suitable conditions for determination are: 0.1g epoxy oil sample, 0.5ml solvent (deuterated chloroform) and two drops of tetramethylsilane (T.M.S.) .The method is simple, sensitive and reproducible. It depends principally on the accuracy of weighing of the oil sample (to be determined), the volume of solvent ($CDCl_3$) and T.M.S.

Keywords

Raw materials for coatings binders (oils)
epoxidised oil

Processes and methods primarily associated with analysis, measurement or testing
nuclear magnetic resonance spectroscopy

Properties, characteristics and conditions primarily associated with raw materials for coatings and allied products
oxirane content

Une nouvelle méthode pour faciliter la détermination de la teneur en oxygène issu des groupements oxiranes dans huiles végétales

Résumé

Au moyen de la N.M.R. on a déterminé la teneur en oxygène issu des groupements oxiranes d'huile de lin époxydisée et d'huile de ricin déshydratée et époxydisée. La méthode est particuliérement apte dans le cas des valeurs d'oxirane supérieures à 1,5 pour cent. Les conditions convenables pour effectuer les déterminations sont; 0,1 grm. d'huile époxydisée, 0,5 ml, de solvant (chloroforme deuterique) et deux gouttes de tétraméthylesilane (T.M.S.). la méthode est facile, sensible et reproductible, elle dépend en premier lieu de la précision de pesage de l'échantillon d'huile, le volume de solvant ($CDCl_3$) et de T.M.S.

Eine neue, einfache Bestimmungsmethode für den Oxiransauerstoffgehalt pflanzlicher Öle

Zusammenfassung

Mit Hilfe der N.M.R. Technik wurde der Oxirangehalt von epoxidiertem Leinöl und epoxidiertem dehydratisierten Rizinusöl bestimmt. Die Methode ist besonders für Oxiranwerte über 1,5% geeignet. Für die Bestimmung geeignete Bedingungen sind: 0,1g Epoxy Ölmuster, 0,5 Milliliter Lösungsmittel (deteriertes Chloroform) und zwei Tropfen Tetramethylsilan (TMS). Die Methode ist einfach, empfindlich und reproduzierbar. Sie ist hauptsächlich abhängig von der Genauigkeit, mit der das zu bestimmende Ölmuster eingewogen wurde, dem Lösungsmittelvolumen ($CDCl_3$) und T.M.S.

Introduction

Refs. 1–11

Epoxy compounds have assumed commercial importance in recent years with the advent of epoxy resins, epoxy plasticisers, epoxy stabilisers and epoxy insecticides. These have focused attention on analytical methods for the determination of the oxirane oxygen. Many techniques have been employed for the determination of epoxy materials, including thin layer[1,2] and gas liquid[3,4] chromatography, colourimetric[5–7] and the usual volumetric estimation of the hydrogen halide[8–10] techniques.

The chromatographic techniques are not accurate, gas chromatography gives too low results owing to the partial decomposition of the labile epoxy esters on the column[11] and the thin layer chromatography gives high results[2]. The colourimetric methods are simple and reproducible, but diepoxy fatty acids could not, up till now, be detected[6]. In some volumetric methods (the indirect methods) the presence of carboxylic acids may interfere, and thus a correction for the amount of acid present in the sample has to be made. In general, these methods require an appreciably greater time, especially in the preparation of the standard solutions.

The aim of the present work is to determine the oxirane oxygen content of epoxidised vegetable oils by a simple and more precise technique (by N.M.R.). To the best of the author's knowledge, the oxirane oxygen content of vegetable oils has not previously been determined by the N.M.R. technique.

Experimental

Refs. 12–17

Materials

Linseed oil. Egyptian alkali refined linseed oil was used (I.V. 173).

Dehydrated castor oil (D.C.O.). Egyptian castor oil was dehydrated under inert atmosphere (refined and dry CO_2) at 270°C using sodium bisulfate as catalyst[12].

Zeo-Karb 225. (Permutit Co. Ltd). A sulfonated poly(styrene-divinylbenzene). Dark yellow, particle size (−14+52) mesh. Sulfur content 17 per cent, sulfonyl group content 43 per cent.

Hydrogen peroxide. Chemically pure grade. Its strength was determined by the thiosulfate method[13] and was found to be 32 per cent.

Deuterated chloroform ($CDCl_3$). Analytical grade $CDCl_3$ was used.

Solvents and chemicals. All solvents and chemical reagents were of pure grade and were further purified if necessary by the usual techniques.

Techniques

Epoxidation. Linseed oil[14] and D.C.O.[15] were epoxidised with hydrogen peroxide and acetic acid using Zeo-Karb 225 as catalyst.

Oxirane content. The oxirane content was determined volumetrically by titrating the sample dissolved in benzene directly against HBr-acetic acid solution using crystal violet as indicator[16,17].

Results

Ref. 16

The oxirane oxygen content of epoxidised linseed and epoxidised dehydrated castor oils was determined by the N.M.R. technique. The method of determination is simple. The parameters studied were: the weight of oil sample, the amount of solvent and the amount of tetramethylsilane added. It was found that the suitable conditions for the determination of oxirane oxygen content that gave best results are:

0.1g of the epoxidised oil was accurately weighed into the N.M.R. tube, exactly 0.5ml deuterated chloroform ($CDCl_3$) was added, shaken thoroughly until the oil sample completely dissolved, and then two drops of tetramethylsilane were added. The N.M.R. equipment was adjusted according to the following conditions:

Filter bandwidth	4.0cps
R. F. field	0.2mG
Sweep time	250.0 sec.
Sweep width	500.0cps
Sweep offset	0.0 cps
Spectrum amp.	25.0

The peak heights appeared at 7.48ppm (which refer to the epoxide group band) were measured in centimeters, from the base line, as in Figure 1. The relationships between the peak height and the oxirane oxygen content of the epoxidised oils are shown graphically in Figure 2. The values of oxirane oxygen content in the oils were measured according to the Durbetaki's[16] technique. It can be seen, from Figure 2 that the peak height of the epoxy group band increases with an increase of oxirane oxygen content, ie, a linear relationship was obtained. Also, it can be concluded that epoxy oil samples of oxirane values below 1.5 per cent cannot be measured accurately by the N.M.R. technique, and consequently, this method is suitable only for oxirane values higher than 1.5 per cent.

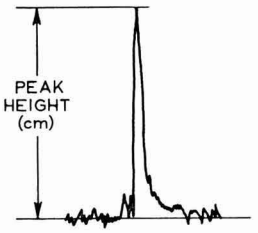

Fig. 1. Peak height measurements

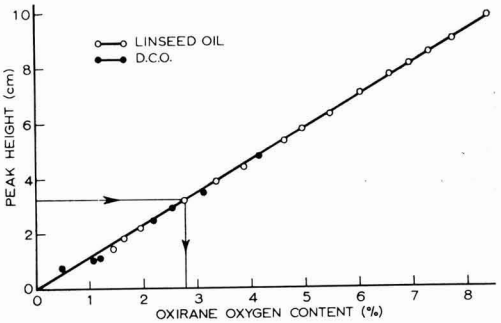

Fig. 2. Oxirane oxygen contents vs. the peak height of the epoxy group band

The relationship shown in Figure 2, can be considered as a standard curve for determining the oxirane oxygen content of epoxidised linseed and dehydrated castor oils. The advantages of this are the simplicity, precision, reproducibility and the saving of time. It is necessary only to determine the peak height from the N.M.R. chart, and then from the standard curve, Figure 2, the oxirane oxygen content in the oil could be determined. It depends principally on the accuracy of weighing of the oil sample and on the amount of solvent and T.M.S. added. Carbon tetrachloride (CCl_4) can also be used as solvent with satisfactory results.

Conclusions

The optimum conditions for determination of oxirane oxygen content of epoxidised linseed oil and epoxidised D.C.O. by the N.M.R. technique have been obtained. The method is suitable for oxirane values higher than 1.5 per cent.

Acknowledgments

The author would like to thank Prof. Dr A. A. Yehia and Assistant Prof. E. M. Abd-El-Bary for their kind encouragement and interest. The help of Dr M. A. El-Souckary is very much appreciated.

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Next month's issue

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

Goethe's contribution to colour physiology *by J. D. Frazee*

The structure of Prussian blue and analogues *by M. F. Dix and A. D. Rae*

Permeability of epoxy coal tar films *by H. Corti, G. Baro, R. Fernandez-Prini and A. G. J. Maroto*

The ultraviolet screening behaviour of pigments *by P. Marvuglio, R. F. Sharrock and R. J. Kennedy*

Review

Solvent-based paint formulation

By E. W. Flick

Noyes Data Corporation 1977

1st Edition. Pp x + 450. Price 28 U.S. Dollars

It would be difficult to imagine a title which conveyed to the prospective reader a more precise key to the contents of this book.

The objectives are quite simple and can, therefore, be judged in equally simple terms. Such an assemblage of paint recipes must have been attempted by countless paint technologists, during periods of enthusiastic application to their craft. It must follow that the great majority of these attempts have aborted due to encroaching pressures of industrial or domestic origin. It is, therefore, timely that such a compilation should have been made available.

The real measure of this work must be of the manner in which it is arranged and indexed. Obviously, this is done according to American classification of paints and varnishes and, therefore, the British reader must indulge in some mental re-classification when applying to his own needs. The reviewer found that Section IV and V were repeatedly used as appendices during the study of the formulations, and as such were invaluable. It is inevitable that there will be a certain lack of standardisation in the presentation of formulations from so many different sources. Each formulator has given the data which he/she feels relevant, but which might well have been supplemented in order to give more common threads throughout the book, for example:—

(1) *Fineness of grind* might well have been quoted in a common measure throughout, as well as in the formulator's preferred units.

(2) *Type of disperser mill* used or intended for use would have been helpful if quoted in many more formulations.

(3) *Common yield* (say 103 gallons) for all liquid formulae throughout the book, would have made for easier comparison by the reader.

The presentation is enhanced by the combination of Subject Index with Contents. The classification of this most important part of the book is impeccably done according to the intended use of the coating.

References with respect to formulations, ingredients and suppliers are all very accessible.

To epitomise a compilation is almost a contradiction in terms. May it suffice to say that the reviewer finds this book useful, and expects it to become increasingly effectual over the years to come.

F. B. REDMAN, A.T.S.C.

Section Proceedings

Ontario

Ink resins

The first Technical Evening of the 1977/78 Season was held on 19 October 1977 at the Skyline Hotel, Toronto.

A record turnout was addressed by Ludwig Horn of Lawter Chemicals, Illinois, whose topic, "Trends in ink resins" embraced a historical development of ink varnish manufacture, before turning to specific novelty varnish and resin systems for emerging infrared and ultraviolet cured films.

Also considered was the preparation of such vehicle systems through the use of novel modified resin types.

Mr Horn gave an excellent presentation which was warmly received; the question period was very lively. Mr L. Campey proposed the vote of thanks.

Paint film properties

On Wednesday 16 November Prof. Henry Schreiber, of the Ecole Polytechnique, University of Montreal, presented a paper on "New approaches to the development of properties in paint films".

Prof. Schreiber detailed the key areas of expertise which were essential to the successful coating development chemist, amongst which were a knowledge of synthetic organic chemistry, physico-chemical polymer and colloid sciences.

The properties of solutions and coatings have been predicted from considerations of such functions as solubility parameter. The speaker indicated an alternative and more useful value, critical surface tension, and in particular the use of a thermogradient bar to determine the temperature dependence of surface tension.

This then allows an accurate calculation of critical surface tension.

The lecture was profusely illustrated and very professionally presented. Professor Schreiber, who was recently awarded the first prize in the Protective Coatings Award of the Chemical Institute of Canada, received questions throughout his presentation. Mr Walter Fibiger, Ontario Section Chairman, presented the vote of thanks at the end of the evening.

A.M.C.

Newcastle

External pipeline coatings

The second meeting of the 1977/78 session was held at St. Mary's College, Durham on 3 November when Mr D. Gray of British Gas Corporation presented a talk entitled "External pipeline coatings".

In 1976 the British Gas Corporation employed some 100,000 people and produced over thirteen thousand million therms of gas, having a sales value of over thirteen hundred million pounds. The gas is transported throughout the country by 130,000 miles of low and medium pressure pipelines and 3000 miles of high pressure pipelines, and protection of

these against corrosion is vital to ensure continuous supply to consumers and to maintain very high safety standards.

Mr Gray discussed the coal tar systems which had been used for many years and described a variety of problems associated with such coatings. Emphasis was placed on recently developed powder coatings which were under investigation by the Gas Corporation and these were discussed together with laboratory test methods and field trials which could be used to establish performance characteristics.

The lecture was followed by a lively discussion period before the Chairman, Mr F. Hellens, proposed a vote of thanks to the speaker, which was warmly received by the members.

For the second month in succession some 50 members and visitors attended St. Mary's College and, after the lecture, partook of the buffet meal with private bar facilities.

T.H.

Information Received

New pigments plant

The new ICI Organics Division phthalocyanine green pigments plant at Grangemouth, Stirlingshire, has started production, and deliveries from the plant have now commenced. This expansion has doubled ICI's manufacturing capacity for phthalocyanine green pigments to a total of nearly 3,000 tonnes a year. This makes ICI probably the world's largest producer of these pigments. The products are marketed principally under the names '*Monastral*' and '*Vynamon*' green pigments and cover a wide range of green shades, with high fastness to solvents, heat, chemical attack, light and weathering.

Research organisation

The Engineering Department of Du Pont (UK) Ltd have announced plans to form an international research organisation devoted to the study of mechanical-chemical processing of materials leading to system design procedures. The organisation will be called the International Mechanical-Chemical Processing Research Institute and is expected to be completed by March 1978. The Institute will place emphasis on relating macroscale performance to micro properties, and major attention will be devoted to fine particle systems and other mechanical processing such as surface chemical engineering, liquid-solids separation, and rheology.

Increased anthraquinone capacity

A new plant being constructed in Northern Germany as a joint venture by Bayer AG and Ciba-Giegy AG is to utilise a new process for the manufacturing of anthraquinone which has both economic and ecological advantages over conventional production methods. The starting material is napthalene, which is readily available both from coal tar and as a derivative of petrochemical processes. The new plant, which is expected to go on stream at the end of 1980, will have a capacity of between 12,000 and 15,000 tonnes per annum and should ensure a sufficient supply of anthraquinone for industry.

Change of name

Berol Kemi AB of Sweden have announced that from October 1977 the trade name of their well known cellulose ether *Modocoll* will change to *Bermocoll*. Individual grade references will also change to show more direct relationships with viscosity characteristics. *Modocoll* was first introduced in 1945 and since then continuous development has ensured it a leading place in the water soluble cellulose derivative field. It has applications for stabilising and thickening water borne paints as a dispersing agent and binding agent and film former.

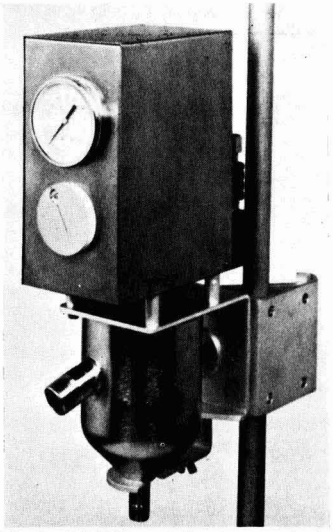
New products

Thickness gauge

Zorelco Ltd, Ohio, have available a new pocket size wet film thickness gauge (wheel type) which is a precision machined, stainless steel instrument for accurately measuring wet film thickness of paints, varnish, epoxy, adhesives, inks and other wet coatings.

New viscosity control unit

Baird & Tatlock (London) Ltd have available the new Brookfield *Viscosel* automatic viscosity control system developed to handle a wide range of fluids, including inks, coatings and lacquers. It accurately senses the viscosity of the fluid (reacting to solid/liquid systems as a whole rather than to the liquid phase only) and regulates the addition of solvent to keep viscosity at a preset value.



The Brookfield Viscosel viscosity control unit

Low-cost waste water treatment system

A new low-cost waste water treatment system which screens out solids down to 20 microns is now available in Europe from Sweco Europe S.A., Belgium. Besides reclaiming an exceptionally high proportion of raw materials, the system also permits the processed water itself to be recycled, as well as helping manufacturers in a variety of industries to meet stringent water pollution control standards. The system consists of two long-established Sweco waste water treatment devices mounted together with flexible tubing, pumps and other accessories on a compact, sturdy skid-frame. The units can handle up to 100 cubic metres an hour and can result in a major saving of raw materials.

New zinc phosphates

Jenolite Division of Duckhams Oils has announced the introduction of two new zinc phosphates which operate at room temperature (as low as 25 C).

Zinfos 200NLT is applied by spray and Zinfos 500NLT by immersion and coating weights of up to 500 mg/sq ft are deposited. The working concentration for the spray grade is 2 per cent by volume and for the immersion grade 5 per cent by volume and the products are intended principally for the phosphating of cold rolled steel.

Freight container paints

International Paint have launched a complete range of coatings for freight containers known as *Nulac*, including five primers and three finishes which are guaranteed to give protection for at least three years when applied in accordance with the appropriate specifications.

New butanediol plant

GAF-Hüls Chemie GmbH, a joint venture of Chemische Werke Hüls AG and GAF Corporation, New York, will shortly be opening a new plant with a capacity of approximately 30,000 tons per annum of 1.4 butanediol which is used for the manufacture of special polyurethanes, rubbers, polyesters and plasticisers.

Courses, Literature, etc.

Expansion of computer search service

The Science Reference Library has announced that the range of files available through their Computer Search Service has been increased to cover all aspects of science and technology. The service has now been extended to operate once again from the Holborn Branch as well as the Bayswater Branch.

Can paints compete?

The Derby Lonsdale College of Higher Education is organising a symposium entitled "Can paints compete?" to be held at the College on 21 March 1978.

New steel protection standards

The British Standards Institution has extensively revised standard CP 2008, which has been reissued as BS 5493 "Protective coating of iron and steel structures against corrosion". The new standard lists various methods of protecting steel structures and gives indications of their likely performances in various applications and environments.

OCCA-30 Exhibition

18-21 April 1978 at Alexandra Palace, London

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

The continuous dialogue between suppliers and manufacturers

New Exhibitors

The Exhibition Committee is pleased to report a number of additions to the first list of Exhibitors published in the January 1978 issue of the *Journal*, and these organisations are mentioned below.

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

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

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

Theme of the Exhibition

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

Dates and times

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

Tuesday	18 April	.. 09.30 - 17.30 hrs.
Wednesday	19 April	.. 09.30 - 17.30 hrs.
Thursday	20 April	.. 09.30 - 17.30 hrs.
Friday	21 April	.. 09.30 - 16.00 hrs.

Since the first list of Exhibitors was published in the January issue of the *Journal*, the Association has been informed that the following companies' products will also be on display:

Blagden & Noakes Chemical Division (*resins, pigments, additives*)

CdF Chimie—France (*resins*)

Chemical Supply Co. Ltd (*resins, pigments, additives*)

Contraves AG—Switzerland (*laboratory apparatus*)

Cordova Chemical Co.—USA (*adhesives, additives*)

Gardner Laboratory (*laboratory apparatus*)

Haake—Germany (*laboratory apparatus*)

Johnson, S. C. & Son Inc. (*pigments*)

Maschinenfabrick Heidenau (*manufacturing equipment*)

Micro Powders Inc.—USA (*pigments*)

MSE Scientific Instruments (*laboratory apparatus*)

Rondec S. P. Ltd (*manufacturing equipment*)

Societe Francaise D'Organo Synthese—France (*resins*)

Teledyne Taber (*laboratory apparatus*)

UPA (*laboratory apparatus*)

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

Refreshments on stands

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

Information in foreign languages

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

Official Guide

This unique publication will contain descriptions of all exhibits and advertising space is available both to exhibitors and those organisations not able to show at the 1978 Exhibition. The "Official Guide" will be published **at the end of February 1978,** so that intending visitors can obtain copies and plan their itineraries.

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

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

As in 1977, several Sections will be organising coach parties to visit the Exhibition and any Members interested should contact their local Section Hon. Secretary. (Full Section Committee lists for 1978 were published in the January 1978 issue of the *Journal*.) For example, the Manchester Section has already organised group travel to the Exhibition by train and has negotiated a fare of just over one-third of the normal day return fare. It is also hoped that several parties will be organised from overseas to visit the Exhibition. As in previous years the Official Guide and season admission tickets will be available several weeks in advance of the Exhibition (prepayment only) from the Association's offices but they will also be available for purchase at the entrance to the Exhibition Hall. A charge is made for both the Official Guide and the season admission tickets to the Exhibition. The policy was introduced several years ago to deter casual visitors who otherwise collected large quantities of technical literature from exhibitors' stands; the policy has been welcomed by exhibitors and has in no way acted as a deterrent to bona fide visitors to the Exhibition.

Facilities at Alexandra Palace

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

The Heathrow Central Terminal of the Piccadilly Line was opened by Her Majesty the Queen on 16 December. The advantage to overseas visitors travelling to OCCA 30 is enormous, as it now greatly simplifies the travelling both to hotels in Central London where they may be staying and to the Exhibition. Visitors arriving at Heathrow Airport will now be able to board a Piccadilly Line train at the Airport building itself which will take them directly to Turnpike Lane Station from where the Association will be running a free bus shuttle service to and from the Exhibition. The travelling time from Central London to Turnpike Lane Station is approximately eighteen minutes.

News of Exhibitors at OCCA-30

Stand 76

Hooker Chemicals & Plastics Corp.

The *Hooker Chemicals & Plastics Corp.* will be exhibiting at OCCA 30 for the first time and will be demonstrating their *Ferrophos* enhancer for zinc-rich paints which can result in significant cost savings. The *Ferrophos* enhancer (Fe_2P) is specifically designed for effective use as a partial substitute for zinc dust in zinc-rich coatings and acts synergistically with zinc dust to provide outstanding corrosion protection. Among the main advantages of *Ferrophos* enhancer is the improved welding performance it provides. During welding, *Ferrophos* pigment greatly reduces zinc smoke, yields very low or no weld porosity, improves continuity and strike and reduces electrode wear in resistance welding. In addition, welding speeds are faster, slag removal is easier and the need for blasting before welding is virtually eliminated.

The use of *Ferrophos* enhancer offers other advantages over conventional zinc-rich coatings, such as increased torch cutting speeds, cleaner cuts and stronger intercoat adhesion, allowing for better top coating. These advantages are possible because *Ferrophos* pigment is electrically conductive, electrochemically active and chemically inert.

Stand 6

Tiszamenti Vegyimuvek

Tiszamenti Vegyimuvek, Szolnok, Hungary will present pigments for the production of paints and lacquers, including zinc chromate, chrome yellows and chromium oxide green, when they exhibit at OCCA 30 for the first time. Chromium oxide green is a pigment with excellent overall properties, especially lightfastness and weathering, whilst zinc chromate is a basic zinc potassium chromate for priming paints and is also of value as a brilliant greenish yellow, either in self shades or in admixture with blue. The lead chromate pigments are produced by *Tiszamenti Vegyimuvek*, under licence from *I.C.I.*

Stand 9

Sanyo-Kokusaku Pulp Co. Ltd

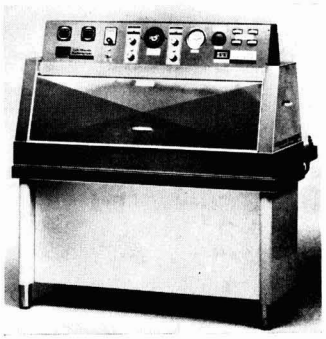
Sanyo-Kokusaku Pulp Co Ltd will be showing their range of *Superchlon* chlorinated polyolefins including chlorinated polypropylene (*CPP*) and chlorinated polyethylene (*CPE*). Especially featured will be the new *Superchlon 500* series of chlorinated polyethylene. *Superchlon CPP* and *CPE* have unique features which have enabled them to be predominant in the Far Eastern market for many years. They are easily soluble in many common solvents, giving solutions of rather low viscosity, resulting in savings in varnish preparation. The plasticised and pigmented dry films have excellent properties including acid and alkali resistance, water permeation resistance and good exterior durability.

Stand 13

The Q-Panel Company

The *Q-Panel Company* of Ohio, U.S.A. will be featuring their ranges of products for research and quality control. The *QCT Condensation Tester* tests finishes for resistance to moisture in the form of rain or dew. Condensation tests are faster than humidity and more representative than salt spray.

The *QUV Accelerated Weathering Tester* tests resistance to both moisture and sunlight. A condensation system simulates rain and dew and eight fluorescent UV lamps simulate the effects of sunlight. This new approach has replaced traditional methods of accelerated weathering in over two-hundred research laboratories and users report excellent results in outdoor performance.



The QUV Accelerated Weathering Tester

Stand 77

Ferranti Ltd

The *Instruments Division* of *Ferranti Ltd* will be demonstrating new viscosity measurement systems, which include the *Ferranti Portable Viscometer*, a coaxial cylinder viscometer which consists of a pair of cylinders, one located co-axially within the other. The outer cylinder is rotationally driven by a small motor and the inner cylinder is free to rotate against a calibrated scale and has a pointer to register the amount of deflection.

Also demonstrated will be the *Ferranti-Shirley Viscometer* system which uses a cone and plate technique to enable the flow behaviour of many types of simple or complex fluids to be examined. Dynamic flow characteristics are automatically plotted as a graph and accurate repeatability of test results are assured.

Stand 11

CIRP

Chimique Industriel et Recherche de Procedes, France, have specialised for more than 25 years in the building of Turnkey chemical units, particularly for synthetic resins production and one of the features on their stand at OCCA 30 will be a one unit model plant.

Stand 54

Compagnie Francaise Goodyear

The *European Chemical Division* of *Goodyear* will be exhibiting a wide range of decorative and protective coatings which can be formulated with the *Pliolite* line of synthetic rubber copolymer resins produced in Le Havre, France, and readily available throughout Europe. The range of *Pliolite* resin based coatings cover exterior and interior masonry finishing systems, both low and high build, concrete curing membranes, waterproofing sealers, road marking, metal primers, intumescent paints, multicolour finishes etc.

Due to their high performance characteristics such as adhesion, water and alkali resistance, chemical inertness, mechanical and heat stability and reliable drying control, *Pliolite* resin based coatings represent a step forward in meeting ever increasing market demands in many applications.

Stand 12

Meca-Inox

Meca-Inox, France, will be demonstrating their ranges of ball valves, gate valves and other kinds of stainless steel apparatus for the chemical industry. The main products are stainless steel fittings and unions and some valves will also be shown.

West Riding Section

Annual Dinner Dance

The West Riding Section held its nineteenth Annual Dinner and Dance at the Crown Hotel, Harrogate on 25 November. In statistical terms, the 264 present constituted a record number for the function, but in social terms it was the same as in past years; a convivial meeting of old friends, representing a wide cross section of members, with their wives, and guests from all OCCA Sections.

The Top Table, Chaired by Mr Michael Cochrane and Mrs Pamela Cochrane, was honoured by the presence of the President with the President's Lady, supported by Chairlady Mrs E. N. Harper (Bristol), Chairmen Mr T. W. Wilkinson (Hull), Mr J. E. Mitchell (Manchester), Mr R. J. King (Birmingham), Mr F. Hellens (Newcastle), Mr J. Davidson (Scotland), with their glamourous wives, and Mr Harper; last but not least Mr R. H. Hamblin, Director & Secretary.

The Toast of welcome to the Guests was given by the Chairman, with the response by the President, admirably adorned in his National dress.

The President's Lady made the draw of ticket numbers, the prize of a "Weekend for Two" being won by Mr S. Pierce, with consolation prizes being won by Dr Tordoff, Mr R. Smith and Mr R. H. Hamblin.

Dancing to Peter Edwards and his Music followed until the early hours, but even so the general opinion was it had ended all too soon.

The Section would like to record its appreciation to Mr Peter Robinson, General Manager of the Crown Hotel and his staff, who in no small way assured the success of the function.

N.J.C.

West Riding Section Annual Dinner Dance. Back row (*left to right*): Mr Harper, Mr F. Hellens, Mr R. H. Hamblin, Mr J. Davidson, Mr M. J. Cochrane, Mr A. McLean, Mr R. J. King, Mrs T. Mitchell and Mr T. Wilkinson. **Front row** (*left to right*): Mrs E. N. Harper, Mrs F. Hellens, Mrs J. Davidson, Mrs P. Cochrane, Mrs A. McLean, Mrs J. Mitchell, Mrs T. W. Wilkinson

Scottish Section

Junior Technologists' Symposium

The Scottish Section held a very successful Junior Technologists' Symposium in Glasgow on 2 December 1977. There were approximately seventy delegates at the meeting who came from paint and associated companies in Scotland and the north of England.

Some of the delegates at the Scottish Section Junior Technicians Symposium

Ontario Section

Ladies' Evening

The Ontario Section held its Annual Ladies' Evening on Wednesday, 14 December at the Skyline Hotel, Toronto.

The topic was "Accessories and colour in fashion", and was conducted by members of the Fashions Bureau of the T. Eaton Company.

The presentation was a professional modelling of the latest in fashions and accessories, with an emphasis on colour coordination.

This was a most successful evening and a warm vote of thanks to the mannequins and organisers was made by W. Fibiger, Section Chairman.

Irish Section

Annual Dinner Dance

The Annual Dance of the Irish Section was held on the evening of Friday 4 November at the Clarence Hotel, Dublin.

A full house of 106, including Members, their wives and guests, sat down to a splendid meal, followed by some lively speeches, introduced by Mr T. Corrie as M.C.

The toast to the Irish Section was proposed by the charming lady Chairman, Miss Pat McGee and the reply was given by the President, Mr A. McLean in a witty and amusing address. Mr K. Callaghan, Chairman Elect of the Irish Section, offered the toast to the Guests and Mr R. Morehead, President of the Irish Paint Manufacturers' Federation, responded.

The principal guests at the Chairman's table included the President and his wife, Mr and Mrs McLean, Mr and Mrs R. Morehead and Mr J. Mitchell, Chairman of the Manchester Section, with his wife.

The evening was rounded off by dancing into the small hours and a good time was had by all.

Irish Section Annual Dinner Dance (*left to right*): Mr J. Mitchell, Mr A. McLean, Miss P. McGee, Mr R. C. Somerville and Mr R. Morehead

Midlands Section

Symposium Programme

The Midlands Section is organising a one-day Symposium entitled "The effects of modern technology and legislation on paint manufacture" to be held at the Metropole Hotel, National Exhibition Centre, Birmingham on Wednesday 8 March.

Programme for the Symposium is:

9.45	Registration
10.15	Presidential address
10.30	"The possible effects of some recent or projected legislative Acts on paint manufacture" by Mr D. J. T. Howe, Paintmakers Association of Great Britain
11.00	Discussion

11.15 Coffee
11.30 "Manufacture of solvent thinned and water thinned paints" by Mr I. Berg, BSc, CChem, MRIC, AIInfSc (Process Development Manager, Berger Paints)
12.00 Discussion
12.15 Lunch
13.45 "Anomalies in dispersing organic pigments" by Dr G. Kirchmer, Lack. Ing., BASF Pigment Applications Department
14.15 Discussion
14.30 "Instrumental colour control" by J. F. Brown, Chief Analyst, Mander-Kidd (U.K.) Ltd
15.00 Discussion
15.15 Tea
15.30 "Powder manufacture" by R. L. Staples, Director, Midland Speciality Powders Ltd
16.00 Discussion
16.15 Summing-up by Chairman

The charge for the Symposium will be £20.00 for members, and £30.00 for non-members, plus VAT, and includes luncheon. Full details of the Symposium may be obtained from Mr R. J. Chater (Hon. Publications Secretary, Midlands Section), Blundell-Permoglaze, Tisley, Birmingham B11 2BD.

Obituary

I. C. R. Bews

1919–1977

Ian Bews, sometime Honorary Secretary and sometime Honorary Editor of the Association, died on 17 November 1977. He was 58 and had been in poor health for several years.

He was born in Birmingham on 7 June 1919 and was educated at King Edward's School, leaving in 1938 to join Docker Brothers in the laboratory. He was soon commissioned in the Territorial Army, and was mobilised on the outbreak of war, serving throughout and reaching the rank of Major. On demobilisation he rejoined Docker Bros. but was released in 1946 to attend Birmingham College of Technology full-time, where he took his B.Sc. (London External) in 1947. He remained with Docker Bros. until 1954, and then joined British Titan Products Co. Ltd as a Sales Representative. In 1970 he was appointed Home Sales Manager, and in 1973 Market Information Manager; but it was at the end of that year that ill-health forced him to retire.

A keen supporter of OCCA, Bews had been serving as Honorary Publications Secretary of the Midlands Section for two years when he was asked to accept the position of Honorary Editor (1959-62), following this with the equally onerous position of Honorary Secretary (1963-69).

Ian Bews was a calm and unassuming man, kind, conscientious, public-spirited and sincerely religious. Apart from his important service for OCCA, he became Deacon of Caterham Congregational Church and Chairman of the Caterham Urban District Council. He was not a man for lip-service; he supported his beliefs by practical involvement.

M.H.M.A.

Memorial Service

On Saturday 10 December 1977 the Association was represented at a Memorial Service for the late Ian Bews at the Caterham United Reform Church by Mr A. T. S. Rudram (Immediate Past President), Mr A. R. H. Tawn (Vice President), Mr A. G. Holt, Mr J. T. Tooke-Kirby and Mr H. C. Worsdall. The Church was crowded with representatives from the Church and the local community, demonstrating the great esteem in which Ian Bews had been held.

News of Members

Mr R. F. Page, an Ordinary Member attached to the London Section, has been appointed the Home Sales Manager of Horace Cory & Co. Ltd.

Mr P. H. Turner, Ordinary Member attached to the Manchester Section, has taken up another appointment with Kronos Titanium Pigments Ltd.

Mr P. H. Turner

Register of Members

Ordinary Members

ADAMS, TERENCE GRAHAME, 22 Victoria Avenue, Cliftonville, Kent. (*London*)

BROSWICK, GEORGE, 1041 North Service Road, Oakville, Ontario, L6H 1A7, Canada. (*Ontario*)

FISHER, JOHN EDWARD SEYMOUR, Shell Chemical SA (Pty) Ltd, PO Box 2068, Durban 4000, South Africa. (*Natal*)

GAGLIARDI, JOHN, 63 Hardings Street, Coburg, Victoria 3058, Australia. (*General Overseas*)

GORDON, NEALE, BSc, Shell Chemicals, PO Box 494, Johannesburg, South Africa. (*Transvaal*)

HAWORTH, PETER DUNCAN, 51 Circular Drive, Ewloe Green, Deeside, Clwyd. (*Manchester*)

HENTSCHEL, URS, Ciba-Geigy Canada Ltd, 858 York Mills Road, Don Mills, Ontario M3B 3A8, Canada. (*Ontario*)

HUGHES, DAVID WILLIAM, LRIC, 42 Barcheston Road, Knowle, Solihull, West Midlands. (*Midlands*)

JOY, WILLIAM JOHN, BSc, Dulux (NZ) Ltd, Hutt Park Road, PO Box 30-366, Lower Hutt, New Zealand. (*Wellington*)

LANGE, KLAUS DIETER WALTER, Hoechst (SA) (Pty) Ltd, PO Box 8692, Johannesburg 2000, South Africa. (*Transvaal*)

MEYER, ROGER MICHAEL, BSc, PO Box 80, Uitenhage 6230, South Africa. (*Cape*)

SKARSHOLT, OLAF HARALD, 203A Godley Road, Titirangi, Auckland, New Zealand. (*Auckland*)

SPARKES, BARRY AUSTIN, A C Hatrick (NZ) Ltd, PO Box 2359, Auckland, New Zealand. (*Auckland*)

Associate Member

BOOTH, WARWICK, 64 Wallace Road, Mangere Bridge, Auckland, New Zealand. (*Auckland*)

Registered Student

BEATTIE, DOUGLAS, 75 Lorraine Drive, Willowdale, Ontario, Canada. (*Ontario*)

Forthcoming Association Conference and Section Symposia

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

Midlands Section Symposium

The Midlands Section is organising a one day symposium entitled "The effects of modern technology and legislation on paint manufacture" to be held at the Metropole Hotel, National Exhibition Centre, Birmingham on Wednesday 8 March.

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

Thames Valley Symposium

Call for papers

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

Stratford-on-Avon

OCCA CONFERENCE

STRATFORD HILTON HOTEL • 20–23 JUNE 1979

Shakespeare's birthplace

Photograph by Herald Photographic Studio, Stratford-upon-Avon

CALL FOR PAPERS

The challenge to coatings in a changing world

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

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

Forthcoming 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.

1978

February

Thursday 2 February

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

Monday 6 February

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

Tuesday 7 February

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

Thursday 9 February

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

Scottish Section: "Microscopic studies of gloss of surface coatings" by Dr S. G. Lawrence, Ciba-Geigy (UK) Ltd, at the Bellahouston Hotel, Glasgow at 6.00 p.m.

Friday 10 February

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

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

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

Thursday 16 February

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

Friday 17 Febuary

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

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

Thursday 23 February

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

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

Thames Valley Section: "Cathodic protection" by Mr J. H. Morgan of Morgan, Berkeley & Co. Ltd, at the Beaconsfield Crest Motel (White Hart), Aylesbury End, Beaconsfield, Bucks at 6.30 for 7.00 p.m.

Friday 24 February

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

March

Thursday 2 March

Newcastle Section: "The vinyl approach to marine and maintenance coatings" by Mr J. Benson of Union Carbide to be held at St. Mary's College, University of Durham, Elvet Hill Road, Durham.

Monday 6 March

Hull Section: Ladies' Evening. Visit to the Ferens Art Gallery, Hull, conducted by Mrs L. Dunn, Senior Keeper at the gallery.

Tuesday 7 March

West Riding Section: "Calcium ferrite and zinc ferrite—two new active anti-corrosion pigments" by Dr P. Kresse of Bayer, West Germany to be held at The Mansion Hotel, Roundhay Park, Leeds 8, commencing at 7.30 pm.

Wednesday 8 March

Manchester Section: Student Works Visit to D. Macpherson Ltd.

Midlands Section: One day Symposium entitled "Modern methods of paint manufacture", at the Metropole Hotel, National Exhibition Centre, Birmingham.

London Section: "Metal decorating in the paint and printing industries". A day meeting in association with the Thames Polytechnic at Woolwich, SE18, commencing at 10.00 a.m.

Thursday 9 March

Scottish Section: Title, subject and lecturer still to be advised by National Corrosion Service.

Midlands Section—Trent Valley Branch: A joint meeting with Institute of Corrosion Science and Technology. Suggested title "Why paint it?" to be held at the Crest Hotel, Pastures Hill, Littleover, Derby at 7.00 p.m.

Monday 13 March

Manchester Section: "Toxicological problems in the pigment-using industry" by Mr M. T. Hobbs, ICI Ltd Organics Division, to be held at The Woodcourt Hotel, Sale, Cheshire, commencing at 6.30 pm.

Wednesday 15 March

Ontario Section: "The anatomy of the printed dot" by Mr P. Wyskowski of McLean Hunter Ltd at the Skyline Hotel, Toronto.

Thursday 16 March

Thames Valley Section: "Rheology of pigmented systems. A new low-shear instrument" by Dr M. L. Colclough of ICI Paints Ltd, to be held at The Beaconsfield Crest Motel (White Hart), Aylesbury End, Beaconsfield, Bucks at 6.30 for 7.00 pm.

Friday 17 March

Midlands Section: "Jewellery through the ages" a Newton Friend Lecture by Mr C. Reichsollner to be held at Birmingham Chamber of Industry and Commerce at 7.00 pm.

Wednesday 22 March

Scottish Section—Eastern Branch: Annual General Meeting followed by a Ladies' Evening which, by the kind permission of John Haig & Co. Ltd, will involve "whisky tasting" to be held at The Alfton Hotel, 6 Grosvenor Crescent, Edinburgh 12, at 7.30 pm.

Friday 31 March

Bristol Section: Annual Dinner Dance at the Mayfair Suite, New Bristol Centre at 7.30 for 8.00 p.m.

Irish Section: "Organic versus inorganic coatings" by Mr J. R. Lyon, Goodlass Wall Ltd, to be held at The Clarence Hotel, Dublin 2, commencing at 8.00 pm.

April

Wednesday 19 April

Ontario Section: Annual General Meeting.

Friday 21 April

Midlands Section: Members are asked to note that the Annual General Meeting will now be held on 21 April and not the 14th as previously notified, at the Crown Hotel, Broad Street, Birmingham at 6.30 p.m.

OIL & COLOUR CHEMISTS' ASSOCIATION

TECHNICAL EXHIBITION

18-21 APRIL 1978

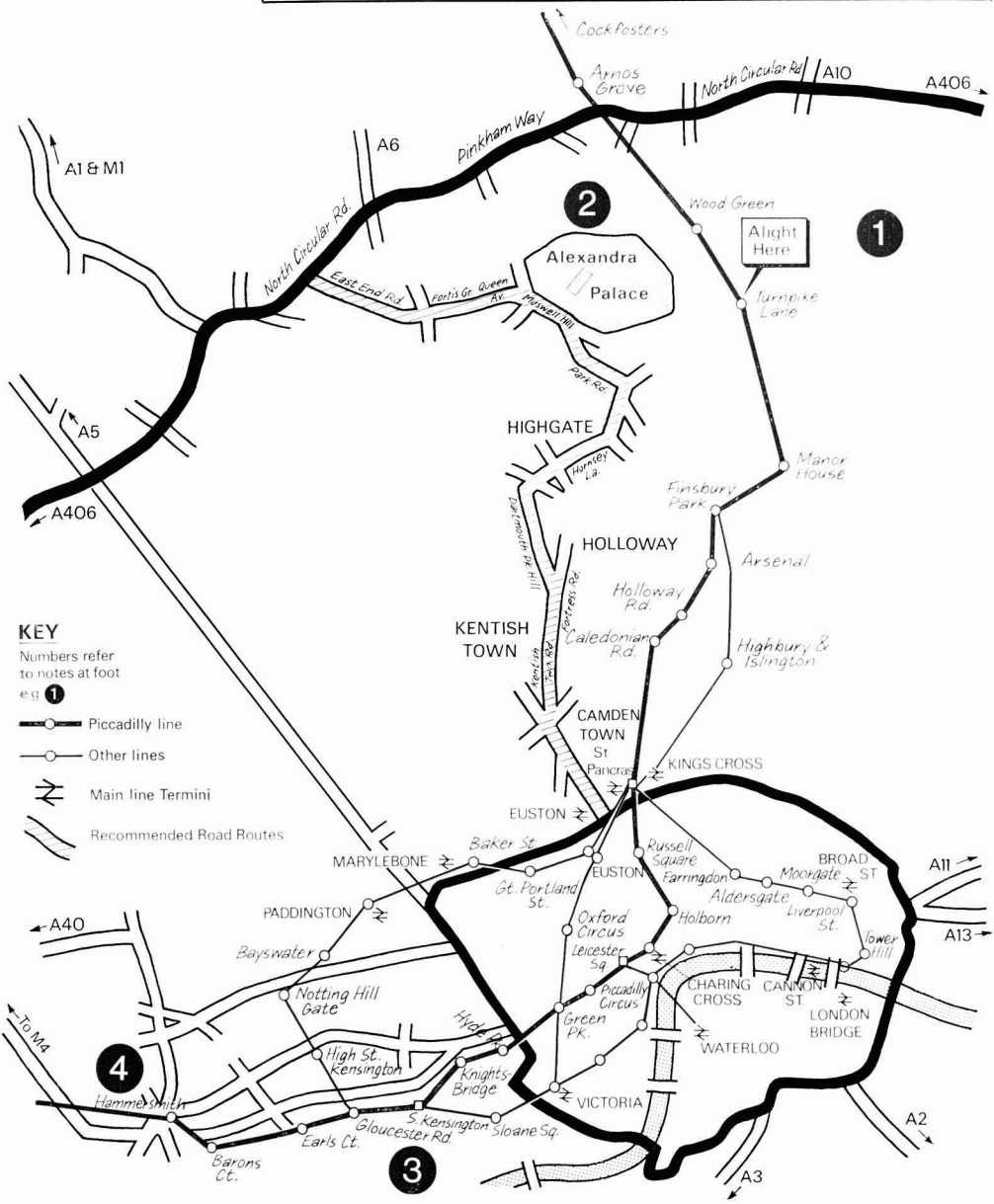
The motif, designed by Robert Hamblin, uses the symbol of a moving indicator on a calendar to emphasise the importance of the continuous dialogue year by year between suppliers and manufacturers. The inward-pointing letters recall the international aspect of this unique annual focal point for the surface coatings industries, which in 1977 attracted visitors from 50 countries.

TRAVEL ARRANGEMENTS FOR VISITORS TO OCCA-30

For those travelling to the Exhibition by car, ample free parking space is available in the grounds of Alexandra Palace, and recent improvements to the road system include the extension of the southbound carriageway of the M1 Motorway to the North Circular Road and the flyover on that road by the new Brent Cross Shopping Centre. By the 1978 Exhibition the extension of the Piccadilly Underground line to the Heathrow Airport Terminal will give a direct line to Turnpike Lane station from which the Association runs a free bus shuttle service to the Exhibition. The journey from central London to Turnpike Lane takes approximately 18 minutes.

OCCA-30

For the benefit of intending visitors to the Exhibition, a map is reproduced below of the area around Alexandra Palace showing the mainline stations in central London, the Piccadilly, Circle and Victoria Lines of the underground system and also the road links with the North Circular Road and motorways.



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

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

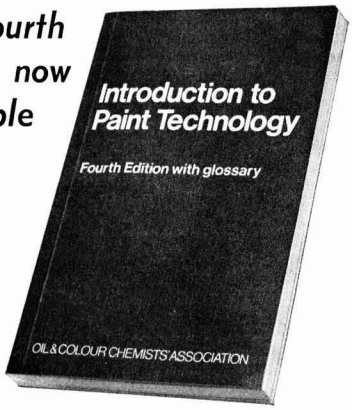
3. Visitors arriving at West London Air Terminal may board the Piccadilly Line trains at Gloucester Road station.

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

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

CURRENT PUBLICATIONS

New fourth edition now available



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

Price £5.00 (Registered Students of the Association £2.50) **Prepayment only.**

OIL AND COLOUR CHEMISTS' ASSOCIATION
Priory House, 967 Harrow Rd., Wembley HA0 2SF

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of Transactions and Communications

1966-1975
Volumes 49-58

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UV polymerisation

Newcastle Section Symposium 1975

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

UV

Paint Technology Manuals
Works Practice

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

Convertible Coatings

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

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

Biennial Conference
PREPRINTS

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

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

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

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

[See order form on back cover

CLASSIFIED ADVERTISEMENTS

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

APPOINTMENTS VACANT

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The Coatings Company of Tube Investments Ltd.

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Tel: 0602-609782

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6″ × 12″ Marchant Laboratory Triple Roll Mill.

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

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

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See above for details of rates for Classified Advertisements.

CLASSIFIED ADVERTISEMENTS

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Suitable applicants will probably be aged 25-35 years, possess a University degree or appropriate technical qualifications and have at least five years' experience in the Surface Coatings industry.

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Applications, in writing, giving details of qualifications and experience should be sent to:

LAPORTE

The Manager, Personnel Services,
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The same facility is available to non-members, who should notify their London address when sending in their application form for the season ticket (£2.00 each inc. VAT and copy of the "Official Guide").

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For details of rates see page xiv or contact:

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