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Veova 10 based mass polymers as binders for solvent-borne industrial coatings

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An interpretation of viscosity measurements on suspensions of powdered solids, in terms of the physical state of the suspended particles

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Yellowing of alkyd paint films

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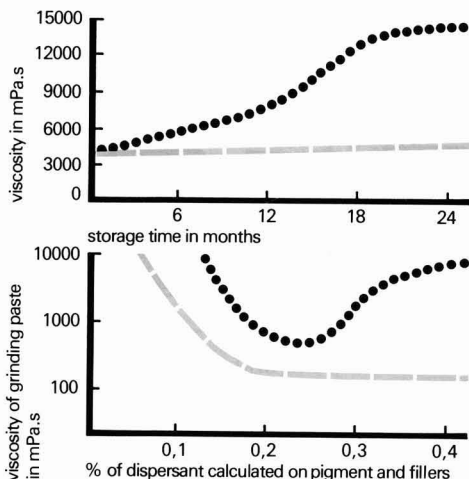
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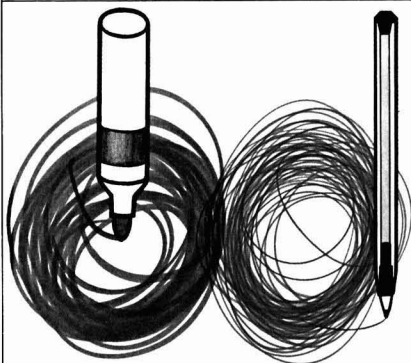
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Transactions and Communications

Veova 10 based mass polymers as binders for solvent-borne industrial coatings*

By W. C. Aten

Koninklijke/Shell-Laboratorium, Amsterdam, The Netherlands

Summary

Veova 10 has become a well-known co-monomer for vinyl acetate in emulsion polymers for the paint industry. More recently, work has been carried out to develop other Veova 10 based polymer types for industrial coatings applications. These investigations have led to the development of a bulk polymerisation technique by which Veova 10 can be copolymerised with styrene and optional functional monomers. Examples of how the Veova mass polymer can be formulated into powder coatings and non-aqueous or aqueous dispersion paints have already been given in the recent past. This paper deals with work on the development of Veova mass polymers as binders for solvent-borne coatings. The incorporation of hydroxyl-containing functional monomers permits crosslinking with amino resins or polyisocyanates. Two types of such

monomers are used: the usual hydroxyalkyl(meth)acrylates and the hydroxyl containing adduct of CARDURA E 10 and (meth)acrylic acid. The designing of a solvent system for such polymers using a modern solubility mapping technique will be discussed in detail.

The results of paint screening evaluations indicate that the performance of such Veova mass polymers is comparable with that of high-quality acrylic binder types. Possible outlets are in (metallic) automotive finishes and domestic appliance finishes when cross linked with melamine type resins. When cured with aliphatic polyisocyanates they might, for instance, be attractive for large vehicle coatings and car refinishing systems or for coatings on extruded aluminium.

Keywords

Types and classes of coatings and allied products

aluminium coating
appliance finish
automotive finish
industrial coating
solvent based coating

Processes and methods primarily associated with

manufacturing or synthesis
copolymerization
polymerization

Les polymères en masse à base de Véova 10 en tant que liants pour revêtements industriels non aqueux

Résumé

Dans le domaine de polymères en émulsion pour l'industrie de peintures, Véova 10 est devenu bien connu en tant que comonomère pour l'acétate de vinyle. Plus récemment une étude a été effectuée en vue de développer d'autres types de polymères à base de Véova 10 et destinés à être utilisés en revêtements industriels. Ces investigations ont abouti au développement d'une technique de polymérisation en masse au moyen de laquelle Véova 10 peut être copolymérisé avec styrène et d'autres monomères fonctionnels à option. Il y a peu de temps qu'on a déjà donné des exemples de la manière par laquelle le polymère en masse de Véova peut être incorporé en peintures en poudre et aussi en peintures-émulsionnées, soit aqueuse ou non aqueuse. Cet exposé traite de l'étude sur le développement de polymères en masse de Véova en tant que liants pour les revêtements industriels non aqueux. L'incorporation des monomères fonctionnels contenant du groupement hydroxyle permet le pontage avec les résines amines ou polyisocyanates. On utilise

deux types de tels monomères: les hydroxyalkyl (méth) acrylates usuels et l'adduct de CARDURA E 10 avec l'acide (méth) acrylique qui renferme également les groupements hydroxyles. La conception d'un système de solvants pour de tels polymères en se servant d'une technique moderne pour tracer la solubilité est discutée en détail.

Les résultats des essais d'évaluation sur peintures indiquent que le rendement de tels polymères en masse de Véova est comparable à celui des liants acryliques de haute qualité. Les débouchés éventuels sont les finitions (métalliques) pour automobiles et pour articles ménagers où les polymères de Véova sont durcis par les résines mélamines. Lorsqu'ils sont durcis par les polyisocyanates aliphatiques, ils puissent créer un vif intérêt dans le domaine de peintures pour véhicules utiles, pour les systèmes de repeinture à neuf des voitures ou pour les profils en aluminium.

Die auf Veova 10 gegründeten Massenpolymere als Bindermittel für nicht-wässrige Industrielacke

Zusammenfassung

Der Veova 10 wurde ein sehr bekannte komonomere für von der Lackindustrie angewandten Vinylacetatemulsionpolymere. Untersuchungen hatten sich kürzlich durchgeführt um andere

auf Veova 10 gegründeten Polymeretype für Industrielackanwendungen zu entwickeln. Diese Untersuchungen waren zur Entwicklung einer

*Paper presented at the South African Division's 8th National OCCA Symposium

Massenpolymerisationstechnik herbeigekommen, wodurch man den VeoVa 10 mit Styrol und anderen fakultativen Funktionsmonomeren copolymerisieren kann. Beispiele der Formulierung von dem VeoVa Massenpolymeren an Pulverbeschichtungen, non-wässrige oder wässrige Dispersionsfarbe waren schon früher ausgegeben worden. Diese Aufsatz befasst sich mit Entwicklungsuntersuchungen zu den VeoVa Massenpolymeren als Bindemitteln für non-wässrige Beschichtungen. Die Einverleibung der Hydroxyl erhaltenden Funktionelmonomere gestatten die von Aminohärzen oder polyisocyanaten verursachte Vernetzung. Zwei Typen solcher Monomere werden benutzt: die gewöhnlichen Hydroxyalkyl (meth) acrylate und das Hydroxy erhaltende Addukt von CARDURA E 10 und (Meth) acrylsäure. Die Entwicklung

eines für solche Polymere geeigneten Lösungsmittelsystems mittels einer modernen Lösungsmittelsausarbeitungstechnik wird eingehend diskutiert.

Die Resultate der Auswertungsproben zeigen, dass die Leistung der solchen VeoVa Massenpolymeren sind vergleichbar mit denen der hochwertigen Akrylbindermitteltype. Eventuelle Anwendungen befinden sich in (metallischen) Automobil- und Hausgerätlacken, wenn sie von Melaminharzen vernetzt würden. Wenn sie von aliphatischen Polyisocyanaten gehärtet würden, könnten sie, zum Beispiel, für Nutzfahrzeugglacke- und Autolackiersysteme oder für Aluminiumspritzgussteilbeschichtungsmittel interessant sein.

Introduction

VeoVa 10, the vinyl ester of VERSATIC 10 Acid, was introduced on the market some thirteen years ago. Since then it has been used with increasing success in the emulsion polymer market, usually in co- or ter-polymers with vinyl acetate. The main application of these latices so far has been in emulsion paints and plasters. The technical reasons for VeoVa 10 being so well received are its easy copolymerisation with vinyl acetate, its flexibilising properties, its hydrophobic character and the very good alkali resistance and excellent weathering resistance imparted to the ultimate binder.

Since the early 1970s, investigations have been undertaken to utilise these attractive properties in non-latex paint applications. This effort was mainly concentrated on the industrial coatings market. In retrospect, one factor can be identified which has played a crucial role in the various developments, namely: the reactivity of the VeoVa 10 monomer. The reactivity parameters of VeoVa 10 are approximately the same as those of vinyl acetate, which contributes to the ease of the copolymerisation with the latter in latex applications. For industrial coatings applications, however, vinyl acetate is not an ideal co-monomer, as it often does not provide sufficient chemical and water resistance at the optimal composition for balancing hardness and flexibility. Hence, for such applications there has been a gradual turning away from vinyl acetate towards other monomers which in general are more difficult to copolymerise with VeoVa 10. Due to the complications which arose as a result of their use, various elegant solutions have been found as will be discussed below.

Early VeoVa 10 based binders for industrial coatings

Despite the above-mentioned disadvantages of vinyl acetate, this monomer was still used in the first attempts to develop a binder for industrial coatings. The appropriate binder resistance properties were obtained by co-reacting the vinyl acetate with VeoVa 10 in a specific molar ratio and converting the ester groups most vulnerable to hydrolytic degradation into hydroxyl groups which could be used for crosslinking purposes. It was found that the best way to achieve this was to make the polymer in solution in isobutanol at 95-100°C and then subject it to a selective trans-esterification reaction with the solvent. In this way, hydroxyl functionality was introduced - via an inexpensive route - at the weakest ester sites in the polymer. The residual acetate groups were sufficiently sterically shielded by the bulky VERSATIC 10 ester groups for a binder with good chemical resistance properties to be obtained. Although this polymer (Figure

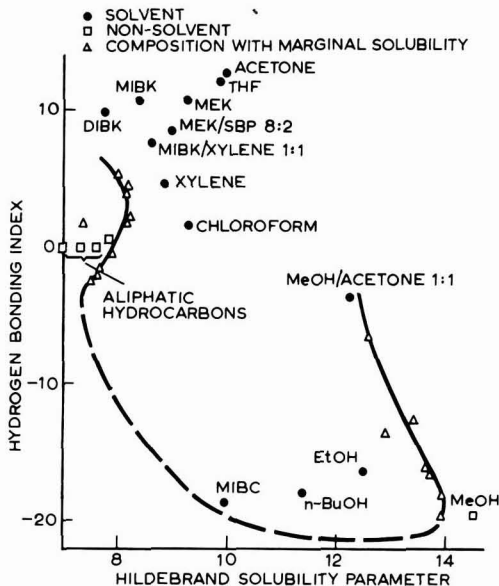


Figure 1. Solubility map for VeoVa mass polymer

2) made optimal use of the specific qualities of VeoVa 10 in combination with vinyl acetate, it has so far not been accepted for use on an industrial scale, owing to the practical complications presented by the lengthy transesterification step.

As a consequence, in further development work the use of vinyl acetate had to be avoided. The most attractive alternative monomer seemed to be styrene. Like vinyl acetate, styrene is hard, relatively cheap and readily available; in addition, its alkali resistance is much better than that of vinyl acetate. A serious drawback for its use in conjunction with VeoVa 10 is its completely different set of reactivity parameters. For instance, under normal emulsion polymerisation conditions around 70-80°C, the presence of a few per cent of styrene in VeoVa 10 monomer can have a strong inhibiting effect. If a reaction takes place at all, it is usually found that the VeoVa 10 monomer only starts to polymerise after nearly all of the styrene has been reacted. It was found that if the reaction temperature was brought to 140-150°C, so that the reactivity parameters of the two monomers come closer to each other, and use was made of the "bridging" action of acrylate monomers with reactivity parameters

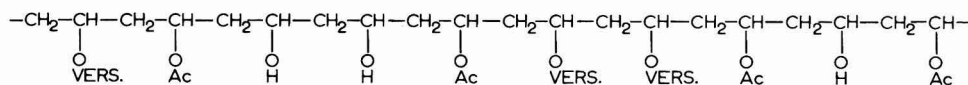


Figure 2

intermediate to those of VeoVa 10 and styrene, a polymer of sufficient homogeneity could be made in a solution polymerisation process:

	Parts by weight
VeoVa 10	35.00
Styrene	11.20
Methyl methacrylate (MMA)	11.97
Hydroxyethylmethacrylate (HEMA)	7.21
Acrylic acid (AA)	4.62
Polymer	70.00
ButylOxitol	30.00

Such a binder was developed for application in anodic electrodeposition paints and is being used in Western Europe for one-coat white electrodeposition finishes or light-coloured primers.

The "VeoVa" mass polymerisation technique

Refs. 1-2

To overcome the restrictions in styrene content of such polymers and to make the procedure independent of the presence of acrylic monomers, it was decided to polymerise at still higher temperatures (160-170°C) and in the absence of solvent. At around 170°C the reactivities of VeoVa 10 and styrene are still rather different (see below) and it was necessary to start the polymerisation with the monomer having the lowest reactivity (VeoVa 10) in the reactor, adding the more reactive monomers together with the initiator.

Monomer 1	Monomer 2	r ₁	r ₂	Temperature (°C)
VeoVa 10	Vinyl acetate	0.92	0.99	60
Styrene	Vinyl acetate	55	0.01	60
Styrene	VeoVa 10	25	0.04	140
Styrene	VeoVa 10	23	0.06	170

With this VeoVa mass polymerisation technique, polymers of the required homogeneity and molecular weight distribution could be produced.

By variation of the balance of "hard", "soft" and functional monomers in these mass polymers, a variety of paint binders can be made. On previous occasions, examples of such binders for powder coating applications and non-aqueous dispersions have been made, and a comparison was drawn between an aqueous dispersion, made via the mass polymerisation technique, and a VeoVa 10 based reactive latex¹. The application of mass polymers in solvent-borne paints has not yet been elucidated and it is the aim of this paper to compare the performance of such

hydroxy-functional VeoVa 10/styrene copolymers, with those of similar commercial all-acrylic or styrene/acrylic paint binders made by solution polymerisation.

For this purpose a series of polymers were prepared with the following basic composition:

VeoVa 10	20 - 50% w/w
Styrene	25 - 50% w/w
Hydroxy-functional monomer	15 - 45% w/w
Methacrylic acid (MAA)	0 - 2% w/w

As mentioned, VeoVa 10 was introduced into a reactor and heated to 165°C and a mixture of the other monomers and 2 phm* of initiator was added at a constant rate over a period of 5 hours. During the post-reaction lasting 1.5 hours, two additional portions of 0.2 phm initiator were added. After the post-reaction, the reactor contents were either dumped and allowed to cool to a solid mass which could be dissolved at a later stage, or thinned in the reactor whilst cooling. The free monomer content of these polymers ranged from 0.5 to 2.5 per cent, of which the greatest portion was free VeoVa 10 monomer; the amount of free styrene monomer was always less than 0.01 per cent and that of free acrylate monomer always less than 0.05 per cent†.

The polymers were almost odourless and ranged from colourless to very slightly yellow. When they were dissolved in a 1:1 (v/v) mixture of xylene and methyl isobutyl ketone (MIBK) to a concentration of 60 per cent w/w, low to medium viscosity solutions were obtained. The basic properties of these polymers and their solutions are shown in Table 1.

The ACE concept

In Table 1 the polymers have been divided into two groups. Those in the first group (nos. 1-6) contain either hydroxyethylmethacrylate (HEMA) or hydroxy-propylmethacrylate (HPMA) as the hydroxy-functional monomer; the use of these functional monomers does not need any further explanation. The second group of polymers (nos. 7-9) contain the adduct of acrylic acid with CARDURA E 10 (ACE). This hydroxy-functional monomer can be made by a simple reaction of the two raw materials in a 1:1 molar ratio at a temperature in the range 100-150°C. It is advisable to use a catalyst for the epoxy/acid reaction however, and to use a polymerisation inhibitor as well. In this way a clear liquid monomer is obtained with a viscosity at 23°C of about 0.1 Pa.s. (Figure 3.)

The use of the ACE structure in acrylic polymers is already known from the patent literature^{2,3}. The main reason for its acceptance in this application is probably that it offers an attractively priced alternative to the limited range of commercial hydroxy-functional acrylic monomers. Slight differences with these other monomers can be expected, for example, the OH group in ACE is less reactive than the one in e.g. HEMA or HMPA as it is not

*Parts per hundred parts monomer.

†No attempt was made to reduce these free monomer quantities.

Table 1
Polymer characteristics (60% w/w in MIBK/xylene 1:1 v/v)

Polymer composition (pbw)	1	2	3	4	5	6	7	8	9
Veova 10	43	34	49	41	39	29	34	23	23
Styrene	40	40	29	40	39	49	40	40	30
HEMA	15	25							
HPMA			20	17	20	20			
ACE							25	35	45
MAA	2	1	2	2	2	2	1	2	2
Polymer properties									
Free monomer content, Veova 10 (% w/w)	2.7	0.9	2.0	2.2	1.8	1.6	2.0	0.9	0.6
others (% w/w)	0.007	0.005	0.031	0.005	0.025	0.021	0.005	0.005	0.005
OH value (calc.) (meq/100 g)	115	192	139	118	139	139	78	109	140
Solution properties									
Colour	← none →						← v. slightly yellow →		
Haziness (10 = v. clear, 1 = v. hazy)	9	7	7	8	8	5-6	10	10	10
Approx. viscosity at 25°C (mm ² /s)	800	1200	550	630	370	900	500	850	1070

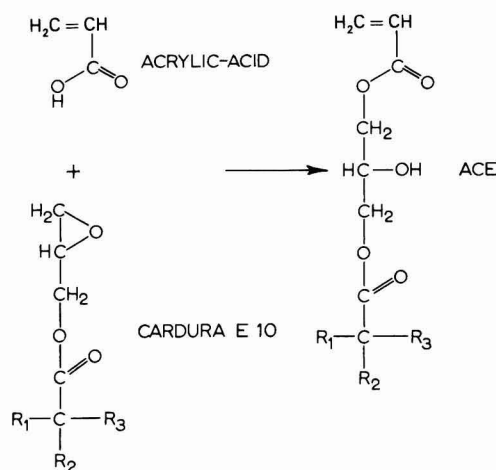


Figure 3

only a secondary OH group but, moreover, sterically shielded by the heavily branched VERSATIC Acid entity. On the other hand, this steric hindrance will also result in ACE being less easily hydrolysed than the above hydroxyalkylmethacrylates.

In acrylic polymers the ACE structure can be introduced via one of three possible routes:

- (1) By post-reacting the CARDURA E 10 with a previously made carboxy-functional solution polymer.
- (2) By making the pre-adduct ACE as described above and incorporating this monomer during the polymerisation process.
- (3) By introducing the CARDURA E 10 together with the free acrylic acid and allowing the addition polymerisation and the epoxy/acid reaction to take place simultaneously.

For the mass polymerisation process, routes (1) and (3) would be less than ideal, as the polymerisation is done in a highly viscous mass at 165-170°C, while the acid/epoxy reaction takes place at lower temperatures. Therefore, the second route was chosen which gave the three polymers 7, 8 and 9 in Table 1.

In making these polymers with ACE, a balance was maintained between the amounts of VERSATIC 10 Acid introduced via its glycidyl ester and via its vinyl ester in order not to obtain too soft an end product. Therefore, the styrene/Veova 10 ratio of the polymers was varied. In addition, as methacrylic acid acts as a built-in catalyst and influences the reactivity of the polymer with crosslinking resins, the MAA content was also varied.

Paint evaluation

A few polymers were first of all briefly screened in clear lacquer formulations to determine the influence of stoving conditions on some basic film properties. A hexamethoxymethylmelamine (HMMM) resin and an aliphatic polyisocyanate were used as crosslinking agents (tables 2 and 3). The results indicated that with the HMMM resin, a cure schedule of approximately 20 minutes at 160°C is required; with the polyisocyanate, a cure of 20 minutes at 130°C appears to be sufficient.

Subsequently, a series of simple white coatings based on each of the nine experimental polymers were formulated, again with either an HMMM resin or a polyisocyanate as crosslinker. Some commercial, high quality hydroxy-functional acrylates and an alkyd were also included in the evaluation. The pigment/binder ratio was 0.7 in all cases and, for the paints cured with the HMMM resin, 0.6 per cent of p-toluenesulfonic acid was added as an accelerator.

Table 2
Clear films, cure with Cymel 1141 (ratio 70:30)

Polymer	1	2	4	7	8
Hydroxy-functional monomer (% w/w)					
HEMA	15	25			
HPMA			17		
ACE				25	35
Film properties					
König hardness on glass (s)					
Cure schedule: 20 min/150°C	188	195	185	130	154
20 min/160°C	194	200	195	178	182
20 min/170°C	198	200	195	183	185
20 min/180°C	198	200	200	190	186
Xylene resistance on glass (s)					
Cure schedule: 20 min/150°C	60	280	45	10	20
20 min/160°C	190	>900	150	10	60
20 min/170°C	250	>900	250	15	70
20 min/180°C	310	>900	310	20	75
Ford impact resistance on mild steel after cure of 20 min/160°C,					
direct (in. lb)	12	9	12	10	12
indirect (in. lb)	3	1	1	2	2

Table 3
Clear films, cure with Desmodur N (ratio 70:30)

Polymer	1	2	4	7	8
Hydroxy-functional monomer(% w/w)					
HEMA	15	25			
HPMA			17		
ACE				25	35
Film properties					
König hardness on glass (s)					
Cure Schedule: 20 min/130°C	175	182	173	79	166
20 min/150°C	185	185	187	158	162
Ford impact resistance on mild steel					
Direct (in. lb)					
Cure schedule: 20 min/130°C	35	50	40	30	35
20 min/150°C	25	20	20	18	35
Indirect (in. lb)					
Cure schedule: 20 min/130°C	20	40	30	35	30
20 min/150°C	6	15	10	15	25
Potlife (h)	<4	5	5	>24	<24

The paints were spray-applied to various substrates and stoved at the above conditions. The basic film properties on glass and mild steel are given in tables 4 and 5, and various resistance data of the films on Bonder 130 are given in tables 6 and 7. From this evaluation the following conclusions could be drawn.

General impressions

The gloss and mechanical properties of the films based on the Veova 10 mass polymers were similar to, or marginally better than, those of the commercial products; only their resistance to xylene and shoe polish was generally lower. On average, their salt-spray resistance and resistance to alkali, acid and hot dishwashing powder solution were excellent. Paints cured with the polyisoc-

yanate showed a better overall performance than those crosslinked with HMMM resin; the latter films did, however, appear to have better weathering resistance.

Influence of Veova 10/styrene ratio

As expected, a high styrene content resulted in a more brittle, less flexible coating with higher xylene resistance. Low styrene content gave better resistance to UV light. At approximately 30 per cent w/w styrene, the gloss retention of the films based on the mass polymers is better than that of the best commercial products, at 40 per cent w/w styrene, it is on the same level or slightly lower, and at approximately 50 per cent w/w styrene, it is clearly poorer – after 2600 hours of exposure in the weatherometer these films start to chalk.

Table 4
White coatings, cure with Cymel 301 (ratio 70:30)

Polymer	1	2	3	4	5	6	7	8	9	Acrylic A	Acrylic B
OH-functional monomer (% w/w)											
HEMA	15	25									
HPMA			20	17	20	20				?	?
ACE							25	35	45		
Film properties on glass											
Gloss (45/45) (%)	94	99	91	93	90	103	94	93	92	88	85
König hardness (s)	186	195	165	180	165	175	134	171	140	146	149
Shoe polish resistance after 24 h*	2	7		2			0	0		4	4
Film properties on mild steel											
Ford impact, direct (in. lb)	25	15	12	15	12	12	6	20	12	8	8
indirect (in. lb)	15	3	1	3	2	2	1	15	1	1	1
Erichsen slow pen. (mm)	6.5	3	6	6	6	4	8	6	6	2	1.5
Con. mandrel bend (mm fail)	0	15	0	5	0	90	0	0	15	100	100
Crosshatch adhesion (GT)	2	4	3	2	3	4	0	1	4	3	4
Xylene resistance (min)	5	>15	10	6	7	>30	1/3	2 1/2	2	30	>30

*10 = no effect, 0 = very bad

Table 5
White coatings, cure with Desmodur N (ratio 70:30)

Polymer	1	2	3	4	5	6	7	8	9	Alkyd	Acrylic
OH-functional monomer (% w/w)											
HEMA	15	25									
HPMA			20	17	20	20				?	?
ACE							25	35	45		
Film properties on glass											
Gloss (45/45) (%)	100	104	100	100	102	104	96	98	95	105	97
König hardness (s)	174	174	160	180	160	171	135	172	151	175	166
Shoe polish resistance after 24 h*	7-8	10		8			3	5		10	10
Film properties on mild steel											
Ford impact, direct (in. lb)	>80	70	>80	>80	<80	70	>80	>80	>80	>80	50
indirect (in. lb)	80	55	70	80	80	70	80	80	70	80	30
Erichsen slow pen. (mm)	9.0	4.7	8.0	8.5	8.0	8.0	9.5	8.8	8.0	8.0	7.5
Con. mandrel bend (mm fail)	0	0	0	0	0	0	0	0	0	0	0
Crosshatch adhesion (GT)	0	4	0	0	0	1	0	0	0	0	1
Xylene resistance (min)	25	>30	30	35	17	>30	1 2/3	10	17	>30	>30

*10 = no effect, 0 = very bad

Influence of hydroxy-functional monomer

No great differences between the binders with ACE and those with hydroxyalkylmethacrylate could be detected, apart from the fact that ACE tended to give lower xylene resistance. A higher amount of hydroxyl monomer improved the solvent resistance but lowered the flexibility and adhesion of the coating; it also increased the viscosity of the polymer solution and slightly reduced the potlife in combination with polyisocyanate.

Influence of the carboxy-functional monomer

The presence of methacrylic acid had a marked influence on a number of properties. For instance, increasing its

content by 1 per cent caused a greater reduction in potlife than that brought about by a 10 per cent increase in hydroxyl monomer. A higher methacrylic acid content also resulted in a higher solution viscosity, an increased cure rate, and increases in film hardness and xylene resistance.

Outdoor durability

Some of the paint systems were also tested for their outdoor durability in Florida. Results after 1 year of exposure are given in Table 8. In general they confirm the data obtained with the weatherometer. Cure with an HMMM resin again gave better gloss retention than crosslinking with a polyisocyanate and weathering

Table 6
White coatings, cure with Cymel 301 (ratio 70:30)

Polymer	1	2	3	4	5	6	7	8	9	Acrylic A	Acrylic B
OH-functional monomer (% w/w)											
HEMA	15	25								?	?
HPMA			20	17	20	20					
ACE							25	35	45		
Film resistance properties on Bonder 130											
Salt-spray (mm rust creep)											
240 h	0	4	1-2	1-2	1	1-2	1-2	1	1-2	4	5
480 h	2	>10	2-4	2-4	2-3	1-4	2-8	2	2-4	5-6	10
5% Formic acid (blistering)											
10 d	←none→	←none→	←none→	←none→	←none→	←none→	←none→	←none→	←none→	6FM	6MD
20 d	none	6F	8D	none	8D	8MD	log ^c	log	8D	8D	2D
5% NaOH (blistering)											
28 d	←none→	none	none	7F	none	4F	5F	6-7F	←none→		
42 d	7F	6F	none	6F	6-7F	none	4F	5F	6-7F	none	6F
2% SUN (80°C) (blistering ^a)											
72 h	9D	none	←none→	←none→	9D	9D	none	none	none	none	9D
148 h	9D	9M	←9D→	←9D→	←9D→	←9D→	←9D→	←9D→	←9D→	none	9D
(adhesion) (GT)											
148 h	1-3	2-3	4	2-3	3	4	1	2	4	4	3-4
Weatherometer: gloss ^b (%)											
original	93	96	92	90	90	97	89	88	92	87	85
1000 h	92	97	93	93	89	96	88	88	92	88	83
2000 h	97	98	92	98	86	90	92	96	94	90	85
2600 h	98	96	94	88	75	30 ^d	88	83	93	92	85

a. at air/water interface, b. 45/45, c. loss of gloss, d. chalk

Table 7
White coatings, cure with Desmodur N (ratio 70:30)

Polymer	1	2	3	4	5	6	7	8	9	Alkyd C	Acrylic D
OH-functional monomer (% w/w)											
HEMA	15	25								?	?
HPMA			20	17	20	20					
ACE							25	35	45		
Film resistance properties on Bonder 130											
Salt-spray (mm rust creep)											
240 h	0-1	0-1	0-1	0	1	0-1	1	1	0-1	0	0
480 h	0-2	1-2	0-2	0-2	1-2	0-1	0-2	1-3	0-2	0-2	1-2
5% Formic acid (blistering)											
20 d	←6F→	←6F→	8F	←none→	←none→	←none→	←none→	←none→	←none→	←none→	←none→
30 d	←6F→	←6F→	6F	←none→	←none→	←none→	none	none	log ^c	none	7VF
5% NaOH (blistering)											
28 d	←none→	←none→	none	none	9VF	none	←none→	←none→	←none→	7VF	none
42 d	←none→	←none→	none	none	9VF	none	none	none	8VF	7VF	7VF
2% SUN (80°C) (blistering ^a)											
72 h	←none→	←none→	←none→	←none→	←none→	←none→	←none→	←none→	←none→	←none→	←none→
148 h	←none→	←none→	←none→	←none→	←none→	←none→	←none→	←none→	←none→	←none→	←none→
(adhesion) (GT)											
148 h	1	2-3	1	0-1	0	3	0	1	0	4	4
Weatherometer: gloss ^b (%)											
original	96	98	97	96	102	103	93	93	93	101	92
1000 h	96	97	93	91	95	98	85	90	91	88	91
2000 h	87	85	84	80	67	46	77	82	86	45	87
2600 h	55	62	80	45	40	15 ^d	34	43	80	18	71

a. at air/water interface, b. 45/45, c. loss of gloss, d. chalk

resistance seemed to increase in the order HMPA < ACE < HEMA. An increase in crosslinking density increased the gloss retention.

The mass polymers tested were those with approximately 40 per cent w/w styrene. They were found

to be on a par with the commercial acrylic systems. Although not tested in Florida, mass polymers with lower styrene contents can be expected - on the basis of weatherometer results - to give even better performance. The UV degradation mechanism of the commercial alkyd system is obviously different from that of the mass

Table 8
White coatings, Florida exposure trials

Polymer		1	2	4	8	Acrylic A	Acrylic B
						Alkyd C	Acrylic D
OH-functional monomer (% w/w)							
HEMA		15	25				
HPMA				17		?	?
ACE					35		
Cure with Cymel 301							
Gloss (45/45) (%)							
	original	95	100	95	93	87	85
	after 1 year exposure	42	54	27	32	31	40
Cure with Desmodur N							
Gloss (45/45) (%)							
	original	100	102	101	99	103	97
	after 1 year exposure	26	44	22	24	82	49

Table 9
Clear films, cure with Desmodur N (ratio 70:30)

Polymer composition (pbw)	4	10	8	11
Veova 10	41	41	23	23
Styrene	40	41	40	42
HPMA	17	17		
ACE			35	35
MAA	2	1	2	
60% w/w solution properties				
Approx. viscosity at 25°C (mm ² /s)				
	630	435	740	600
Potlife of binder/Desmodur N mixture (h)				
Unpigmented, undiluted	5	>8	5	>8
Unpigmented, diluted to spray visc.	±10	30	12	>72
Film properties				
On glass:				
König hardness (s)	173	199	182	122
On mild steel:				
Ford impact resistance, direct (in. lb)	40	35	35	35
indirect (in. lb)	30	30	30	35
Erichsen slow pen. (mm)	>8	>8	>8	>8
Conical mandrel bend (mm failure)	0	0	0	0
Xylene resistance (min)	>30	7	4	1/3

polymeric and acrylic binders: in artificial weathering tests the alkyd coating gave the poorest performance but in the Florida exposure tests it was by far the best.

Further details on polyisocyanate cure

Influence of carboxy-functional monomer

In order to look further into the possibilities of curing the mass polymers with an aliphatic polyisocyanate, the influence of acid content on potlife and clear film properties was studied with systems stoved at 130°C. From the results, given in Table 9, it can be seen that an acid-free polymer has a very long potlife but yields a film with relatively poor hardness and xylene resistance, which confirms earlier indications.

Cure at ambient temperature

Cure at room temperature was also studied with two polymers, one with HMPA and the other with ACE (Table 10). Despite its higher acid content, the ACE based polymer had a longer potlife and cured more slowly than the HMPA based one, which illustrates the lower reactivity of its hydroxyl groups. After full cure, however, which was achieved after approximately 6-7 days, the films from the two binders had properties which were similar and on a level with those of films cured for 20 minutes at 130°C. This performance means that these binders are, in principle, suitable for application in ambient-curing, two-component systems for large vehicle or car refinishing. Further optimisation of hydroxy- or carboxy-functionality would of course be required, as would more detailed study of polymer/polyisocyanate ratios and final paint properties.

Table 10
Clear films, ambient temperature cure with Desmodur N

Polymer composition (pbw)	10	8
Veova 10	41	23
Styrene	41	40
HPMA	17	
ACE		35
MAA	1	2
60% w/w solution properties		
Thinner per 100g polymer/Desmodur N solution (g)	34	60
Potlife (h)	12	>72
Film properties on glass		
Dust-free time (min) (cottonwool test)	20-25	30-35
Tack-free time (h) (finger touch test)	2-2.5	3-3.5
Hard through (thumb press test)		
after 24 h	OK	not OK
36 h	OK	nearly OK
48 h	OK	OK
König hardness (s)		
after 1 d	46	20
2 d	114	63
3 d	154	110
6 d	180	161
7 d	180	165
Film properties on mild steel		
Gloss (45/45) (%)	>100	>100
Ford impact resistance,		
direct (in. lb)	>80	60
indirect (in. lb)	>80	55
Erichsen slow pen. (mm)	>8	>8
Con. mandrel bend (mm failure)	0	0
Xylene resistance (min)	8	3.5

Coating for extruded aluminium

One binder was also screened in a pigmented system for its suitability for coating extruded aluminium. Curing was done at 130°C, again with an aliphatic polyisocyanate. In formulating the binder composition, some earlier findings were implemented with regard to a good balance of the various components; hence Veova 10, styrene, HPMA and MAA were used in the ratio 45:35:18.5:1.5.

To test the coating, a number of procedures were selected from those described by the Architectural Aluminium Manufacturers' Association (AAMA). The Veova 10 based paint was applied either directly to the aluminium or over an epoxy/phenolic precondensate based primer. For comparison, two different commercial acrylic binder/polyisocyanate based paint systems were included in the tests; one of these was applied over the same primer and the other directly to the aluminium.

Both the Veova based systems and the commercial binder systems for which tests were done met the AAMA requirements (Table 11). The hardness of the Veova based coatings was greater than that of the others, while the rest of the properties were on the same level as one of the acrylics and in some respects (gloss and adhesion) slightly better. Although the systems were not tested for weathering resistance, the results obtained in previous work indicate that this Veova based mass polymer would be a very promising candidate for outdoor applications. The

AAMA requires, for example, a coating to withstand 500 hours weatherometer and/or 1 year Florida exposure without loss of adhesion and only slight chalking, fading or colour change. Performance on this level has already been obtained with Veova mass polymers having an even higher styrene content than the one tested here.

Solvent system design

Refs. 4,5

All screening evaluations were done with 60 per cent w/w solutions of the Veova mass polymer in a 1:1 (v/v) mixture of xylene and MIBK, and a 1:1 mixture of xylene/SHELLSOL A was used as the spray thinner. These mixtures were chosen on the basis of general experience with the materials involved, but at this stage it was not specifically known whether these mixtures were in fact "good" or "bad" solvents. Therefore, experience from solvent design technology was used to optimise the solvent system in terms of cost and viscosity (solids content) for this new class of polymers.

Initially, a simple solvent mapping exercise³ was employed. The technique involved dissolving the polymer in a true solvent, then titrating this solution with a non-solvent until the point was reached where the binder just started to precipitate out of the solution. The solubility parameters of the solvent mixture at the point of precipitation could be calculated. In this case, the solubility of a

Table 11
White coatings on extruded aluminium, cure with Desmodur N

Primer (cure 30 min/180°C)	30% PVC ^a epoxy/phenolic	none	30% PVC epoxy/phenolic	none
Top coat (cure 30 min/130°C)	35% PVC Acrylic E/ IPDI ^b adduct	35% PVC Acrylic F/ Desmodur N	35% PVC Veova mass polymer/ Desmodur N	
Film appearance (1 = best)	3-4	2	3-4	1
Gloss (60°) (%)	88	92	92	93
König hardness (s)	114	140	173	175
Dry adhesion (GT)	4	0	2	1
Adhesion after 24 h in aerated water at 40°C (GT)	4	1	2	1
Impact resistance (2.5 mm)	← pass →			
Idem (max. deformation) (mm)	4	5	4	5.2
Resistance to:	← pass →			
10% HCl (15 min/65-75°F)	← pass →			
Mortar (24 h, 100% RH, 100°F)	← pass →			
Humidity (1000 h, 100% RH, 100°F)	← pass →			
Adhesion (tape test)	2	0	1	1
Blistering	← none →			
Salt-spray (1000 h)	← pass →			
Adhesion (tape test)	2	1	1-2	1
Blistering	← none →			

a. PVC = pigment volume concentration, b. IPDI = isophoronediiisocyanate

41:41:17:1 Veova 10/styrene/HMPA/MAA polymer was tested in the following solvents: tetrahydrofuran (THF), acetone (DMK), methyl isobutyl ketone (MIBK), diisobutylketone (DIBK), n-butyl acetate (NBA), methyl isobutyl carbinol (MIBC), chloroform, xylene and ethanol. Non-solvents for the polymer were found to be pentane, hexane, white spirit, SHELLSOL T and methanol. Subsequently, a series of 50 per cent w/w solutions of the polymer were titrated with the non-solvents. The results of this exercise are presented in Figure 1, where the Hildebrand solubility parameters are plotted against the hydrogen bonding index for the solvents, the non-solvents and the marginal solubility compositions as found by titration.

From the solubility map it appears that the polymer is soluble in a broad spectrum of solvent compositions. The fractional polarity, which is usually also taken into consideration, does not appear to play an essential role. It can be seen that the original solvent choice was very "safe" in terms of solvent power, but rather expensive. From the map it can be easily deduced that less costly solvents such as xylene, or even mixtures of xylene and white spirit, will give sufficient solvent power, although an eye must be kept on the solvent balance during film formation: the aromatic solvent must not evaporate more quickly than the aliphatic one. In practice it would, of course, always be wise to check the requirements for application and final paint properties before making such a drastic change in solvent blend.

With the solvent mapping technique it was possible to find, in a relatively simple way, that equally powerful but less expensive solvent systems can be obtained for the polymer. Indications on how a higher solids content can be achieved are less obvious however, and so use was made of an existing computer program. This program is

based on the general finding that for ideal solutions of a given binder, the viscosity of the binder solution is related to that of the solvent blend, and on Kendall's theory that the viscosity of a non-interacting solvent system can be calculated from the viscosity of its constituents in the following way:

$$\log \eta = \sum x_i \log \eta_i$$

where: x_i = mole fraction of each constituent

η_i = viscosity of each constituent

Rocklin and Edwards⁵ have refined this theory by introducing the concept of "effective viscosity", which corrects for cases where there are interactions between the solvent blend constituents, e.g. as a result of hydrogen bonding effects. This latter concept is in fact used in the program.

It was decided to look for solvent systems having a suitable set of solubility parameters which would give lower viscosities than the original 1:1 xylene/MIBK blend. The computer initially indicated blends of low-viscosity ketones with white spirit; ultimately pure acetone gave the lowest values. This outcome not only demonstrated once again the excellent viscosity-cutting power of ketones, but it also underlined the possibilities offered by blending ketones with paraffinic type solvents where no problems of compatibility exist.

From the computer derived data (Table 12), it was possible to make a comparison between the viscosities and relative costs of the xylene/MIBK starting blend, xylene on its own and an 80:20 (v/v) mixture of methyl ethyl ketone (MEK) and SPB 140:165. It is evident that the viscosity data calculated for the various solvent blends are

Table 12
Influence of solvent composition

Xylene (pbw)	50	100	
MIBK (pbw)	50		
SBP 140/165 (pbw)			20
MEK (pbw)			80
Solvent			
Viscosity at 25°C (mPa.s)	0.59	0.63	0.41
Evaporation time (relative)	313	415	89
Costs (relative)	143	96	131
Polymer solution			
Viscosity at 22°C (mm ² /s)			
60% w/w solution	370	630	140
65% w/w solution			320
Appearance of solution and dry film	clear	clear	clear

in good agreement with the actual viscosities of the polymer solutions. In all three cases no problems of compatibility were found, and the dried films were clear. It can be seen that the MEK/SBP blend is not only slightly less expensive than the original starting blend, but it also allows the solids content of the polymer solution to be increased from 60 to 65 per cent without an increase in the viscosity.

As this was only an exercise, the computer was not instructed to take into account other limitations such as those of evaporation time, flash point or conductivity, nor were legislative aspects such as threshold limit values considered. The examples do, however, illustrate how – in addition to work on the improvement of the polymer itself – modern solvent design technology can be used for the optimisation of VeoVa mass polymer solutions to be used as binders for industrial coatings.

Concluding remarks

On the basis of these screening evaluations it can be concluded that the VeoVa 10 based mass polymer, when used in solvent-borne paint formulations, yields films with attractive levels of gloss, gloss retention and mechanical and resistance properties. Judging from the comparisons with high-quality commercial binders, application of

VeoVa based mass polymers in areas such as automotive finishing and refinishing, domestic appliance coating and finishes for extruded aluminium seems to be feasible. Further optimisation of the polymers for specific applications will probably be required. The same is true for the solvent blends used in this investigation; ways to achieve this have been indicated.

Finally, investigations will have to be made to find out in which coating compositions and/or applications the VeoVa 10 based mass polymers will show price/performance advantages over existing high-quality solvent-borne binders for industrial applications, in order to determine their potential as coating binders for the eighties.

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An interpretation of viscosity measurements on suspensions of powdered solids, in terms of the physical state of the suspended particles

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Summary

The interpretation is based on a very simple relation which has been discovered between the viscosity of a suspension and the volume fraction of the suspended solid. The relation is empirical and is not based on theoretical reasoning, although the significance of the material parameter it contains is interpreted theoretically. It has the form:

$$\eta = \eta_0 \frac{(1-\alpha c)}{(1-1.75\alpha c)^2}$$

where: η is the viscosity of the suspension

η_0 is the viscosity of the suspending liquid or medium

c is the volume fraction of the suspended solid

α is a material parameter, defined as the ratio of the effective hydro-dynamic volume of the solid to its actual volume

α will be greater than unity for irregularly shaped aggregates and for floccules which, additionally, may contain internally immobilised liquid.

The equation was first applied to published viscosity measurements made on Newtonian suspensions of spherical particles and of synthetic aggregates of increasing complexity made from them, over a range of values of c . For each type of particle the value of α was found to be independent of c .

In the case of spherical particles, α should be unity if the equation is to reduce to that of Einstein at infinite dilution. In fact, the average value obtained was 1.07.

For the aggregates, the values of α were approximately in the range 1.2 to 2.3, increasing with increasing structural complexity.

It was also found that the values of α were related to the sediment volumes of the particles measured in air. From these observations, it was concluded that the quantity α is a significant material parameter related to the structure of the particles.

The next step was to apply the equation to suspensions of pigments in oils at different values of c , many of which were non-Newtonian. In these cases, the value of α decreased as the applied shear stress, F , increased; this would be expected if the suspended solid was in the form of floccules of mutually attractive particles, which would decrease in complexity as the disruptive stress on them increased. However, it was found that the magnitude of α did not depend only on the magnitude of F ; at a given value of F , α decreased as c increased. In fact, it was discovered that the controlling factor is the product, Fc . Plotting $1/\alpha$ against Fc , for a given pigment/oil system, gave effectively a single curve, regardless of the value of c at which the measurements were made.

No theoretical explanation of these observations is offered. It is proposed that the results are of value in comparing the basic characteristics of different pigment/oil suspensions, this being illustrated by some experimental results.

Keywords

Properties, characteristics and conditions primarily associated with

materials in general

cohesion
suspension
viscosity

raw materials for coatings and allied products

particle shape
particle size

Processes and methods primarily associated with

manufacturing or synthesis

dispersion
floculation

Une interprétation des mesures de viscosité effectuées sur des poudres en suspension en termes de l'état physique des particules en suspension

Résumé

L'interprétation se base sur un rapport très simple que l'on a découvert entre la viscosité d'une suspension et la fraction

volumique de la matière solide en suspension. Bien que la signification du paramètre de matériau α y renfermé sont interprétée

au point de vue théorique, le rapport est empirique, et ne se base pas sur des arguments théoriques, il a la forme suivante:

$$\eta = \eta_0 \frac{(1-\alpha c)}{(1-1,75\alpha c)^2}$$

ou: η est la viscosité de la suspension,

η_0 est la viscosité du liquide de suspension ou du milieu de suspension

c est la fraction volumique de la matière solide en suspension,

α est un paramètre du matériau que l'on définit comme le rapport du volume hydro-dynamique effectif au volume réel,

La valeur de α dépassera l'unité dans le cas des agrégats de forme irrégulière et aussi des flocons qui peuvent renfermer d'ailleurs à leur intérieur du liquide immobilisé.

L'équation a été appliquée en premier lieu aux mesures de viscosité, déjà publiées, sur des suspensions newtoniennes de particules sphériques et des agrégats synthétiques à partir de celles-la et d'une complexité plus en plus évoluée, et à travers une gamme de valeurs de c . Pour chaque type de particule on a trouvé que la valeur de α est indépendante de c .

Dans le cas des particules sphériques, α doit être l'unité, afin que l'équation puisse se diminuer à celle d'Einstein à la diffusion infinie. En effet, la valeur moyenne que l'on obtient était de 1,07.

Pour les agrégats, les valeurs de α se situaient dans l'éventail de 1,2 à 2,3 à titre approximatif, la valeur s'augmentant au fur et à mesure de la complexité structurale.

On a trouvé que les valeurs de α étaient en rapport avec les volumes sédimentaires des particules déterminées en air. En vue de ces observations on a conclu que la quantité α se montre un paramètre de matériau significant en rapport à la structure des particules.

L'étape subséquente était l'application de l'équation aux suspensions des pigments en huiles, à de différentes valeurs de c , dont plusieurs sont non newtoniennes. Dans ces cas, les valeurs de α se diminuaient à mesure que la force de cisaillement appliquée, F , s'augmentait; ce serait à être attendu où la matière solide en suspension était sous forme de flocons de particules mutuellement attirants, ce qui diminuerait la complexité à mesure que s'augmentait la force disruptive à laquelle les particules étaient subies. Toutefois, on a trouvé que la grandeur de α ne dépend pas seulement de la grandeur de F ; à une valeur donnée de F , α se diminuait à mesure que c s'augmentait. En fait, on a découvert que la facteur décisive c'est le produit, Fc . Dans le cas d'un système donné de pigment/huile, la courbe de $1/\alpha$ contre Fc était en grande partie unique, qui que ce soit la valeur de c à laquelle les mesures ont été effectuées.

On n'offre aucune explication de ces observations. On propose que les résultats ont une certaine valeur où l'on fait une comparaison des caractéristiques fondamentales de différentes suspensions pigment/huile, ce qui est démontrée à l'aide de quelques résultats expérimentaux.

Eine Auflegung, vom Standpunkt des physikalischen Schwebstoffzustands, der auf Schwebstoffe durchgeführten Viskositätsmessungen

Zusammenfassung

Die Auflegung gründet sich auf eine sehr einfache Beziehung, die zwischen der Viskosität einer Suspension und dem Raumbruchteil der geschwebten Festkörper. Die Beziehung ist empirisch und gründet sich nicht auf theoretische Beweisführung, obwohl die Beziehung des erhaltenden Materialparameters theoretisch aufgelegt wird. Die Beziehung besitzt die folgende Gestalt:

$$\eta = \eta_0 \frac{(1-\alpha c)}{(1-1,75\alpha c)^2}$$

wo: η ist die Suspensionsviskosität

η_0 ist die Viskosität der schwebenden Flüssigkeit,

c ist der Raumbruchteil des Schwebstoffs

α ist ein Materialparameter, der als das Verhältnis des effektiven Hydrodynamischvolumen zu dessen reellen Volumen

Im Falle irregulärformiger Aggregate and der Flocken, die ebenfalls okkludierte Flüssigkeit möglicherweise enthalten können, wird der α grösser als eins sein.

Die Gleichung wurde zunächst auf bekanntgemachte Viskositätsmessungen, über eine Reihe von c -Werte, an Newtonsche Suspensionen der kugelförmigen Teilchen und der synthetischen Aggregate von zunehmender Kompliziertheit, die aus diejenigen gebildet sein, angewandt. Für jeden Teilchentyp wurde es gefunden dass der α -Wert unabhängig von dem c -Wert war.

Im Falle kugelförmiger Teilchen, der α -Wert soll eins sein, wenn

die Gleichung an unendliche Verdünnung auf die Einsteinsche verwandelt werde. Tatsächlich war der erhaltende Durchschnittswert 1,07.

Im Falle der Aggregate, waren die α -Werte etwa im Bereich von 1,2 bis 2,3, und steigen als die Strukturkompliziertheit zunimmt.

Es wurde gefunden dass die α -Wert in Verhältnis dem in Luft gemessenen Absetzvolumen der Teilchenstand. Aus diesen Beobachtungen wurde es beschlossen, dass der α -Wert ein wichtiger mit der Teilchenstruktur bezogener Parameter ist.

Die nächste Etape wurde die Anwendung der Gleichung auf die in Öl geschwebten Pigmente, deren manche Suspensionen nicht Newtonsch wurden und die verschiedenen c -Werte besaßen. In diesem Falle verminderte sich der α -Wert als die eingesetzte Scherspannung, F , zunahm. Es würde darauf erwartet werden wenn der Schwebstoff aus wechselseitig anziehenden Teilchen bestehe, und dessen Kompliziertheit vermindert würde, als die auf ihnen angewandten Zerstörungsspannung zunahm. Es wurde jedoch gefunden, dass die Grösse des α -Werts nicht von der Grösse des F -Werts allein abhing; an einem bestimmten F -Wert verminderte sich der α -Wert, als der c -Wert zunahm. Tatsächlich wurde es entdeckt, dass der herrschende Faktor das produkt, Fc , ist. Im Falle eines bestimmten pigment-ölsystems wurde eine wesentlich einfache Kurve, ohne Rücksicht auf den c -Wert an dem die Messungen durchgeführt wurden, erhalten, wenn $1/\alpha$ gegen Fc gezeichnet Würde.

Keine theoretische Erklärung dieser Beobachtung wird angeboten. Es wird vorgeschlagen, dass die Resultate wertvoll seien, wenn die grundlegenden Eigenschaften verschiedener Pigment-ölsuspensionen verglicht wurden. Dieser Vorschlag wird durch einige Versuchsergebnisse erklärt werden.

Introduction

Ref. 1

An ability to relate the viscosity of a suspension to the physical state of the suspended particles is of practical value in two ways. If the main interest is in controlling the viscosity, it is possible, at least in theory, to vary the physical state of the particles to bring this about. Conversely, viscosity measurements could be used to obtain information about the state of the particles. For example, different liquids could be compared for their effect on the state of flocculation of the particles of a particular type of powder.

The effect of the suspended particles on the viscosity of a suspension is conveniently expressed by means of the mathematical relation between the viscosity and the volume fraction of the suspended particles, the parameters in the relation having significance in terms of the physical properties of the particles.

The first relation of this kind was the well-known equation derived by Einstein, for the case of a very dilute suspension of non-attracting spherical particles.

It has the form:

$$\eta = \eta_0 (1 + 2.5c) \dots \dots \dots (1)$$

where: η is the viscosity of the suspension
 η_0 is the viscosity of the suspending liquid
 c is the volume fraction of the suspended particles

Non-spherical particles would obey a similar equation, linear in c , but the value of the numerical constant would be different; its value would depend upon the actual shape of the particles.

Consequently, if the values were known for different shapes, the shape of the suspended particles could be deduced from the magnitude of the viscosity.

The constant would also appear to be greater than 2.5, even for non-attracting spherical particles, if they should carry an adsorbed layer of the suspending liquid, because this would increase their effective hydro-dynamic volume. If the particles were known to be spheres, then the thickness of the adsorbed layer could be calculated from the viscosity.

Unfortunately, Equation (1) does not apply to a very high proportion of the suspensions encountered in practice, because they have values of c which are higher than the values for which (1) was derived. In these cases, the observed viscosity is higher than that predicted, even for non-attracting spheres. This seriously limits the practical value of the equation; for this reason, a great deal of work has been done since its publication to extend it to higher values of c . Even for the simplest case of non-attracting spheres, the number of different solutions which have been proposed is very large¹. In addition, many of them are at least partially empirical, making it clear that an exact solution to this very complex problem has yet to be found.

A further complication exists for many industrially important suspensions, including paints and printing inks. In these systems, the particles are often mutually attrac-

tive, sticking together to form floccules. The structure of these floccules depends upon the existence of a balance between their cohesive strength and the magnitude of any disruptive forces they may experience. Such forces will arise when the suspension is subjected to a shear stress; consequently, as the value of the stress varies, so will the structure of the floccules, if equilibrium is to be maintained. As the structure changes, so will the viscosity of the suspension. Consequently the suspensions show non-Newtonian behaviour, the viscosity varying with the value of the applied stress.

In this paper, yet another equation is presented relating the viscosity of a suspension to the volume fraction of the suspended particles. It is intended to justify this presentation by showing that the one material parameter the equation contains is actually controlled by the structure of the suspended particles or floccules, whichever obtain under a given set of flow conditions.

It must be emphasised that the equation is completely empirical, having been arrived at from a study of experimental results. It has the form:

$$\eta = \eta_0 \frac{(1 - \alpha c)}{(1 - 1.75\alpha c)^2} \dots \dots \dots (2)$$

where: η , η_0 and c have the same meaning as in Equation (1)

α is the ratio of the effective hydro-dynamic volume of the suspended particles to their actual volume

The value of 1.75 for the numerical constant was chosen so that the equation would reduce to that of Einsteins when c is very small and $\alpha=1$, these being the conditions for a very dilute suspension of non-attracting spheres which are not carrying an adsorbed layer of the suspending liquid of significant thickness.

The effective hydro-dynamic volume would obviously be increased by an adsorbed layer; it would also be increased by liquid occluded by the irregular surface of a particle and by liquid trapped in the cavities formed between the particles in an open floccule.

Application to experiment

Refs. 2, 3

I. Newtonian suspensions

In this section, use is made of experimental results published by Lewis and Nielsen². They prepared a series of different types of glass particles, ranging from spheres of different sizes to aggregates of different degrees of complexity, which were synthesised from the spheres.

The particles were divided into classes and a number of the physical properties of the particles in each class were measured. For the spheres, these included the mean particle size and the size range; for the aggregates, in addition to their size, an estimate was made of the average number of primary spheres in an aggregate of each class. The sedimentation volume of each type of particle in air was also measured.

Suspensions of each class were made in a chlorinated biphenyl, a Newtonian liquid with a viscosity of 80 poise at 25°C, over a range of volume fractions, the viscosity of each suspension at 25°C being measured on a Haake Rotovisco Couette viscometer.

Table 1
Physical properties of Lewis and Nielsen's spheres and aggregates

Aggregates			
Aggregate class	Mean diameter (μm)	\bar{n}	$\bar{\phi}_m$
A	55	1.8	0.622
B	120	3	0.600
C	60	5	0.529
D	90	8	0.468
E	105	12	0.432
F	130	15-22	0.394
G	160	25-40	0.372
H	190	40-75	0.350
J	240	85-160	0.366
K	275	200-300	0.322
Spheres			
Mean diameter (μm)	Size range (μm)	$\bar{\phi}_m$	
95	90-105	0.64	
51	45-60		
54	30-40		
7	5-10		

In Table 1, the physical properties of the various classes of particles are listed; tables 2 and 3 give the values of the measured relative viscosities, η/η_0 , of each suspension and the corresponding values of the quantity α obtained from Equation (2). The equation can be rearranged to express α explicitly in terms of η/η_0 ; however, it was found more convenient to use a graphical method. Putting $\alpha c = c'$, a graph is drawn of η/η_0 against c' for a chosen range of values of c' ; for the experimentally determined value of η/η_0 , the corresponding value of c' is obtained from the graph by interpolation. The value of α is then simply determined by dividing c' by the actual volume fraction, c .

Results

Considering first the results obtained with the spherical particles, it can be seen that the value of α is substantially constant, being independent of the value of c and of the particle size range. The overall average value is 1.07, with an RMSD of ± 0.03 .

The form of Equation (2) requires α to be unity for spherical particles. Since it is not possible to alter the value of the numerical constant from 1.75, if the equation is to reduce to that of Einsteins at very low values of c , the value of 1.07 for α has to be accepted as the result of experiment. At present, no reason can be given for the discrepancy, but from a strictly practical point of view it is not very large and certainly small enough to justify continuing the study of the application of Equation (2) to other systems.

For the aggregates, the value of α is reasonably con-

Table 2
Lewis and Nielsen's viscosity measurements

Spheres						
Diameter range (μm)	5-10			30-40		
	c	η/η_0	α	c	η/η_0	α
	0.091	1.31	1.080	0.150	1.615	1.067
	0.197	1.95	1.070	0.306	3.89	1.098
	0.241	2.44	1.075	0.361	6.14	1.089
	0.246	2.46	1.057	0.366	6.58	1.090
	0.295	3.24	1.047	0.410	10.70	1.076
	0.345	4.95	1.067			
	0.397	8.50	1.066			
	Mean $\alpha = 1.06$			Mean $\alpha = 1.08$		
Diameter range (μm)	45-60			90-105		
	c	η/η_0	α	c	η/η_0	α
	0.05	1.155	1.120	0.100	1.33	1.030
	0.10	1.36	1.080	0.102	1.34	1.029
	0.15	1.56	1.000	0.155	1.67	1.090
	0.20	1.93	1.025	0.200	2.08	1.115
	0.25	2.58	1.076	0.250	2.72	1.120
	0.30	3.78	1.107	0.261	2.75	1.077
	0.35	5.23	1.069	0.298	3.60	1.091
	0.40	9.28	1.075	0.300	3.68	1.093
	0.45	20.80	1.071	0.350	5.45	1.083
	0.50	37.5*	1.013*	0.400	9.20	1.075†
					9.41†	
	Mean $\alpha = 1.07$			Mean $\alpha = 1.08$		
	Overall mean $\alpha = 1.07 \pm 0.03$					

*approximate values †calculated from mean of η/η_0 values

Table 3
Lewis and Nielsen's viscosity measurements

Aggregates					
Class A			Class B		
c	η/η_0	α	c	η/η_0	α
0.060	1.205	1.183	0.040	1.147	1.350
0.201	2.20	1.169	0.100	1.55	1.480
0.227	2.48	1.150	0.152	1.89	1.316
0.265	3.366	1.189	0.204	2.61	1.328
0.373	12.25	1.212	0.372	19.20	
Mean $\alpha = 1.18$			Mean $\alpha = 1.36$		
Class C			Class D		
c	η/η_0	α	c	η/η_0	α
0.035	1.15	1.57	0.044	1.21	1.64
0.098	1.52	1.46	0.0975	1.61	1.63
0.145	2.08	1.54	0.1275	1.91	1.59
0.187	2.79	1.52	0.193	3.51	1.66
0.240	5.47	1.58	0.204	3.67	1.61
0.264	7.81	1.58			
Mean $\alpha = 1.54$			Mean $\alpha = 1.63$		
Class E			Class F		
c	η/η_0	α	c	η/η_0	α
0.012	1.056	1.83	0.022	1.105	1.82
0.028	1.14	1.82	0.056	1.33	1.84
0.065	1.38	1.75	0.072	1.48	1.88
0.104	1.78	1.79			
0.163	3.06	1.84			
0.209	6.08	1.87			
Mean $\alpha = 1.82$			Mean $\alpha = 1.85$		
Class G			Class H		
c	η/η_0	α	c	η/η_0	α
0.050	1.325	2.04	0.0219	1.123	2.10
0.094	1.85	2.07	0.051	1.386	2.25
			0.064	1.51	2.20
Mean $\alpha = 2.06$			Mean $\alpha = 2.18$		
Class J			Class K		
c	η/η_0	α	c	η/η_0	α
0.022	1.12	2.05	0.039	1.264	2.23
0.052	1.36	2.10	0.077	1.647	2.14
			0.109	2.591	2.48
Mean $\alpha = 2.08$			Mean $\alpha = 2.27$		

stant for each class, independent of the value of c . Different values are obtained for each class; the more complex the aggregate the greater the value of α . This would be expected if the more complex aggregates occlude a proportionally larger volume of liquid than the simpler ones. There is, however, no direct way of assessing whether or not the values of α obtained are reasonable.

It is proposed that some indirect evidence can be obtained from the measurements of sediment volume, made by Lewis and Nielsen, using the following reasoning.

Irregularly shaped particles would be expected to pack together to give a more open sediment than would be obtained with an equal volume of smooth spheres. This is confirmed by the experimental results given in Table 1. Sediment volume (in air) is expressed by the quantity ϕ_m , which is the actual volume fraction of solid in the experimentally observed sediment; obviously, the more open the sediment the smaller the value of ϕ_m . The results show clearly that the value of ϕ_m decreases as the complexity of the aggregates increases.

The various values of ϕ_m which can be obtained with assemblies of smooth uniform spheres are known; they vary with the way in which the particles are arranged in the sediment. In random open packing the value is 0.58; for random close packing it is 0.64 and the highest possible value of 0.74 is obtained with closest packing. The experimental result of 0.64 for the spheres indicates that they are arranged in random close packing.

Suppose that a sediment of irregular particles can be modelled by a sediment of smooth, uniform spheres, the volume of each sphere being equal to the effective volume of each irregular particle, the effective volume being defined as that volume in space which is required to accommodate the particle at all orientations. Let it be further supposed that the effective volume is greater than the real volume of the particle by a factor α , where α is the quantity obtained from viscosity measurements. This involves assuming that the effective hydro-dynamic volume of an aggregate is the same as its effective volume in a dry sediment.

In the model, the equivalent spheres may be arranged in a variety of ways to give values of ϕ_m between 0.58 and 0.74. Consequently, for the irregular particles, the values of ϕ_m should be between $0.58/\alpha$ and $0.74/\alpha$.

Table 4 shows the values of these quantities calculated for each class of particle and the corresponding value of ϕ_m obtained from experiment. With the exceptions of the two classes of aggregate, B and C, ϕ_m agrees reasonably well with the ratio $0.74/\alpha$.

For the present this correspondence is offered only as evidence that the quantity α , obtained from viscosity measurements, has real significance as a measure of the structure of the suspended particles. This being the case, there is sufficient reason to investigate the application of the equation to flocculated, non-Newtonian suspensions, where the value of α will change with the flow conditions.

2. Non-Newtonian suspensions

The materials used to prepare these suspensions were commercial pigments and drying oils or resins of the types used in conventional printing inks which dry by oxidation. To prepare a set of suspensions from one pigment/oil com-

Table 4

Aggregate class	α	$0.58/\alpha$	$0.74/\alpha$	$\bar{\phi}_m$
A	1.18	0.49	0.63	0.622
B	1.36	0.43	0.54	0.600
C	1.54	0.38	0.48	0.529
D	1.63	0.36	0.45	0.468
E	1.82	0.32	0.41	0.432
F	1.85	0.31	0.40	0.394
G	2.06	0.28	0.36	0.372
H	2.18	0.27	0.34	0.350
J	2.08	0.28	0.36	0.336
K	2.27	0.26	0.33	0.322
Spheres	1.07	0.54	0.69	0.64

bination containing different volume fractions of the pigment, the solid was first dispersed in the liquid by one of the techniques normally used in printing ink laboratories, involving either a three roll mill or the manual technique using a palette knife on a glass slab.

In either case the actual dispersion was carried out at high pigment concentration, the suspensions of the required volume fractions being produced by subsequent dilution.

Rheological measurements were made on a standard Ferranti-Shirley cone and plate viscometer, the sample being maintained at constant temperature by passing water through the plate from a bath kept at 25°C. Increasing rates of shear were applied up to a predetermined maximum and then again in reverse, the corresponding shear stress being recorded in each case. With all the suspensions used in this work, the stress reached a steady value almost immediately; occasionally there was some slight hysteresis in that the up and down curves did not correspond exactly.

From the applied rate of shear, D , and the resulting shear stress, F , the viscosity at each value of D was calculated from the ratio F/D ; using the values of the pigment volume fraction c and the viscosity of the suspending liquid, the corresponding values of α were calculated using the interpolation procedure already described.

Because each pigment/oil combination gives up to 30 separate results, only those from the first combination have been given in full; the remainder are shown in the appropriate graphical form. Table 5 shows the details of the first combination and the full experimental results obtained from it.

Discussion of the results from the first combination

Considering first the range of values obtained for α , this is seen to be from 1.91 to 5.5. The largest value found for the aggregates of Lewis and Nielsen was 2.27. However, these were compact particles, so it would not be unreasonable to expect floccules to have a more open structure. At least the lower values overlap the higher values found for the aggregates.

In all cases, the values of α decrease as the rate of shear and the corresponding shear stress increase. This fits in with the idea of a balance existing between the cohesive strength of a floccule and the disruptive force it

experiences. The more open floccules must be weaker because there will be fewer inter-particle bonds per unit area of cross-section; assuming that the disruptive force increases with the rate of shear, the value of α must decrease as D increases.

The exact way in which α decreases as D increases will depend on how the cohesive strength of the floccules varies with α and how the disruptive force depends on D . To examine this further, use will be made of the quantity $1/\alpha$; this is the volume fraction of solid contained by a floccule. As the disruptive force increases, $1/\alpha$ will increase to a maximum value. This will be reached when the disruptive force is so great that even floccules of only two primary particles cannot exist as permanent entities. The value of $1/\alpha$ at this point will be that for the primary particles from which the floccules are made up. For rates of shear above this point, the suspension will be Newtonian.

It is assumed, initially, that a floccule with a particular value of $1/\alpha$ always has the same cohesive strength. If this is so, then that value of $1/\alpha$ will indicate that a disruptive stress of a certain magnitude is being applied to the floccules. Using these assumptions, it is possible to examine the results to investigate the factors controlling the disruptive stress.

In the simplest case, that of a spherical particle in a very dilute suspension, the maximum value of the disruptive stress experienced by the particle³ has been given as $2.5\eta_0 D$.

In more concentrated suspensions, the effect of the higher concentration might be obtained by replacing η_0 by η , the viscosity of the suspension. The value of the disruptive stress would then become $2.5F$, where F is the shear stress. These two possibilities have been investigated by plotting $1/\alpha$ against D (Figure 1), and F (Figure 2), respectively.

Figure 1 shows that $1/\alpha$ increases with D at a decreasing rate, as would be expected. However, it is quite clear that $1/\alpha$ is not controlled by D alone; at any value of D , the value of $1/\alpha$ also depends on the value of c ; the greater c the greater $1/\alpha$. Clearly the concentration contributes to the disruptive stress, as was anticipated for suspensions of finite concentration, when using F instead of D might be more appropriate.

However, Figure 2 shows a similar effect when $1/\alpha$ is plotted against F , although to a lesser degree. At a given value of F , the value of $1/\alpha$ still increases as c increases, although the values appear to be less dependent on c at the higher values of c . Consequently, it must be concluded that the shear stress F is not a sufficient measure of the disruptive stress experienced by the floccules, the effect of the concentration still being significant.

A considerable effort was made to try to resolve this difficulty by finding out the mechanism of the effect of the concentration. Because of the undoubtedly very complex nature of the systems, no satisfactory approach to the problem was found. However, the work was not entirely fruitless because a very simple relationship was discovered which should prove to be of considerable practical value.

This relationship is illustrated in Figure 3, where $1/\alpha$ is plotted against the simple product of the shear stress F and the concentration, c . It can be seen that, to a very

Table 5
System 1

Pigment: Monastral Blue BX (ICI Ltd, Organics Division)

Liquid: Hytex 1 (Alkyd resin, Croda Resins Ltd)

Method of dispersion: Manual, using palette knife and glass slab. The concentration of pigment in the grinding paste was the maximum possible.

Rheological measurements
(Rate of shear increasing)

Viscosity of the liquid $\eta_0 = 4.87 \text{ kg m}^{-1} \text{ s}^{-1}$

Suspension (a) $c = 0.213$						
D(sec ⁻¹)	F x 10 ⁻³ (Pa)	$\eta(\text{kg m}^{-1} \text{s}^{-1})$	η/η_0	α	$\frac{1}{\alpha}$	Fc(Pa)
31.4	2.64	84.1	17.3	2.22	0.450	562
62.1	3.45	55.6	11.4	2.09	0.478	735
91.5	4.06	44.4	9.11	2.02	0.495	865
122	4.67	38.3	7.86	1.96	0.510	995
153	5.28	34.5	7.09	1.91	0.524	1130
Suspension (b) $c = 0.174$						
9.5	1.00	105	21.6	2.77	0.361	174
20.4	1.27	62.3	12.8	2.66	0.376	221
38.6	1.73	44.8	9.20	2.47	0.405	301
76.0	2.41	31.7	6.51	2.29	0.437	419
113	3.09	27.3	5.61	2.20	0.455	538
150	3.64	24.3	4.98	2.12	0.472	633
186	4.19	22.5	4.63	2.06	0.485	729
Suspension (c) $c = 0.136$						
7.8	0.51	65.4	13.4	3.37	0.297	69
30.0	0.96	32.0	6.57	2.94	0.340	131
91.5	1.93	21.1	4.33	2.58	0.386	262
152	2.79	18.4	3.77	2.44	0.410	379
Suspension (d) $c = 0.100$						
10.2	0.271	26.6	5.46	3.79	0.264	27
40	0.722	18.1	3.71	3.29	0.304	72
118	1.65	14.0	2.87	2.89	0.346	165
194	2.44	12.6	2.58	2.69	0.372	244
Suspension (e) $c = 0.066$						
10	0.155	15.5	3.18	4.64	0.216	10
40	0.477	11.9	2.45	3.94	0.254	31
120	1.16	9.67	1.98	3.21	0.312	77
196	1.78	9.08	1.86	2.97	0.337	117
Suspension (f) $c = 0.032$						
10	0.077	7.70	1.58	5.50	0.182	2.5
40	0.284	7.10	1.46	5.00	0.200	9.1
120	0.787	6.56	1.35	4.38	0.228	25.2
196	1.29	6.58	1.35	4.38	0.228	41.3

acceptable approximation, all the points are on the same line, suggesting that the quantity Fc is the parameter which controls the floccule structure, independent of the individual values of either F or c. Should this relation prove to be widely applicable, it would be possible to predict the viscosities of suspensions of a particular pigment/liquid combination over a range of concentrations and values of the shear stress, from only one flow curve.

Although it is obvious that the factor controlling the disruptive stress must depend upon both F and c, it is remarkable that their combined effect can be expressed in such a simple way. No attempt is made in this paper to give any theoretical explanation of this observation; in any case, as with many empirical relations, it is likely that the product Fc is by coincidence sufficiently close in magnitude to the much more complicated expression which

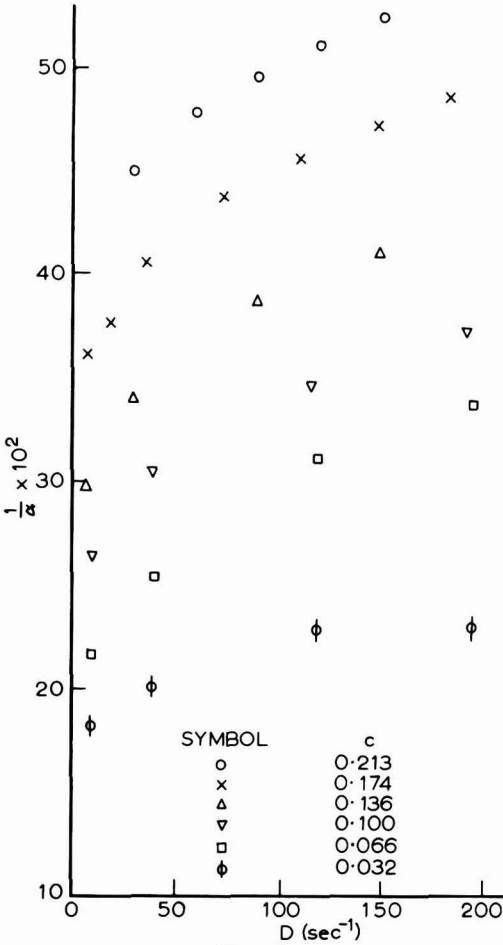


Figure 1.

would be derived were the mechanism known. Here it is sufficient to accept the very valuable empirical fact that the product F_c appears to control the structure of the flocules.

Results obtained on other systems

Ref. 4

As yet, no systematic work has been carried out to examine further the ideas presented here; however, some experimental results were already available, which can be used to illustrate how rheological measurements on suspensions might be interpreted on the basis of these ideas.

For these systems, the results are not given in full; they are shown in the form of plots of $1/a$ against F_c . Details of the composition and the method of preparation of each system are given on the graphs.

(a) Effect of different levels of dispersion

It is a well-known fact of dispersion technology that, for a

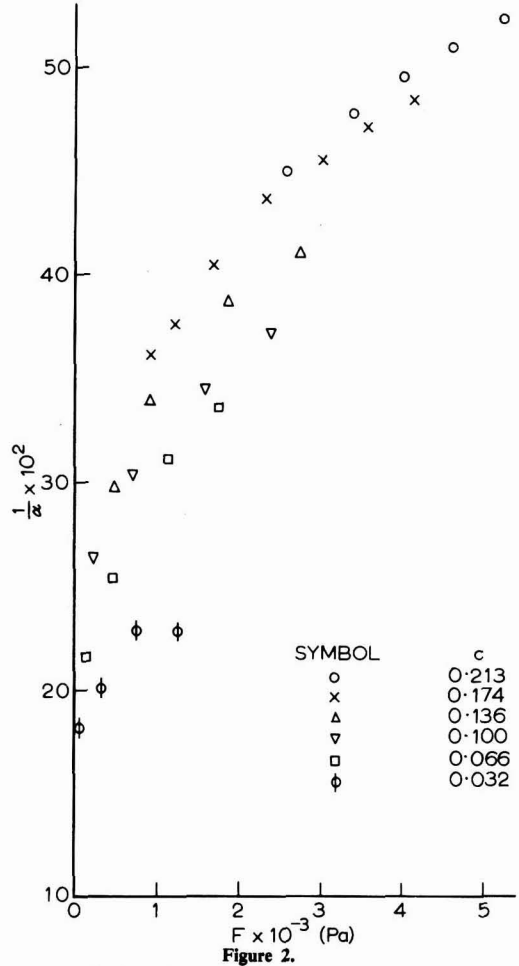


Figure 2.

given method of dispersion, some pigments are dispersed to a greater extent, producing a smaller average particle size the higher their concentration in the grinding paste, subject to the restriction that the paste is capable of being sheared in the grinding process.

The effect can be demonstrated practically by showing that the higher its concentration in the grinding paste, the more rapidly a pigment develops its colour strength.

Figure 4 shows the $1/a$ vs. F_c plots obtained from two systems, using the same pigment/oil combination but differing in the concentration of pigment used in the grinding paste.

Two very different curves are obtained; they show that, at the lower level of dispersion, much weaker flocules are produced, being much closer packed at a given value of F_c - which is a measure of their cohesive strength - and that they break down much more rapidly as F_c increases.

A similar conclusion could have been drawn merely from a comparison of relative viscosities of two suspensions, provided their pigment concentrations were the

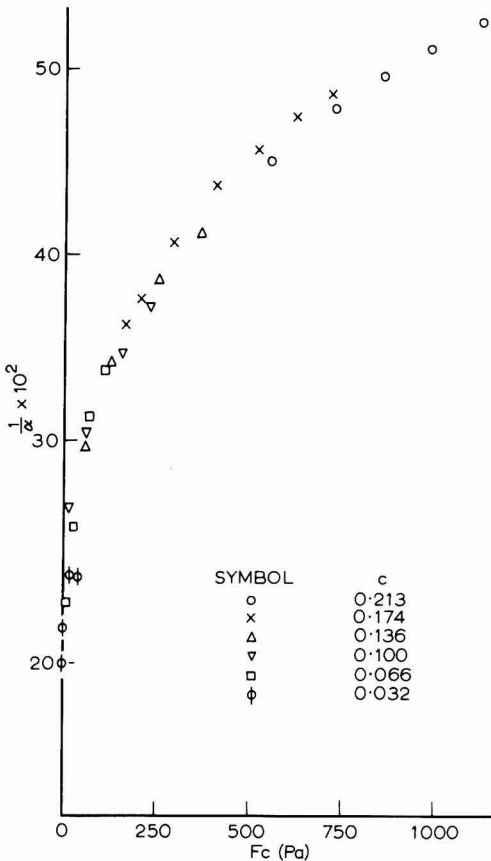


Figure 3.

same. The great advantage of the present method is that it allows a comparison of suspensions where the concentrations are different, a very important factor when practical systems are being compared.

In addition, the method allows a reasonable explanation of why different degrees of grinding produce differences in rheological properties. The results indicate that the better dispersed pigment produces stronger floccules which can consequently maintain a much more open structure at a given value of disruptive stress, leading to a higher value for the viscosity of the suspension.

Smaller particles would be expected to form stronger floccules if the attraction between them is due to Van der Waals' forces. For two spherical particles in a floccule at an equilibrium distance of separation d , the radius of the particles being r , the attractive force f between the particles is given by⁴:

$$f = Ar/12d^2 \dots \dots \dots (3)$$

where A is a constant, the value of which depends on the composition of the particles and the suspending liquid.

Since the cross-sectional area of the floccule is Πr^2 , the

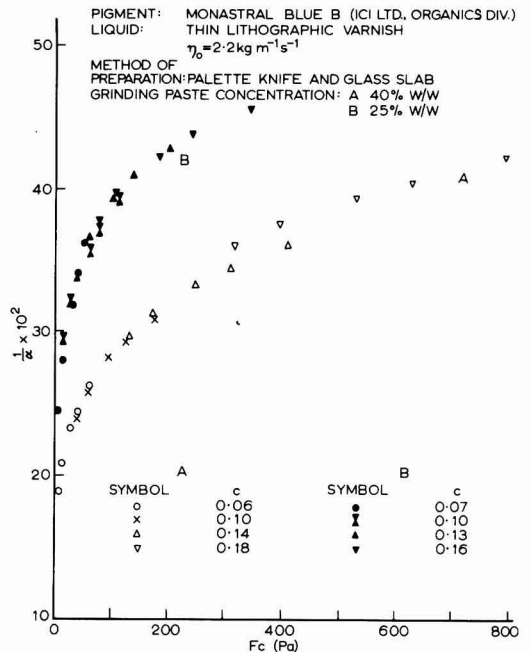


Figure 4.

attractive force per unit area of cross section, f_a , is given by:

$$f_a = A/12\Pi d^2 r \dots \dots \dots (4)$$

This relation shows that, other things being equal, f_a will increase as the radius of the particles decreases.

(b) Effect of different liquids

Figure 5 shows the $1/a$ vs. F_c plots for three systems in which the same pigment has been dispersed in different liquids. In each case the same grinding technique was used and the concentration in the grinding paste was the same. This does not guarantee that the dispersive effort in the three systems is the same but practically it is the nearest approach that can be made to this situation.

The three liquids used were:

1. Linseed oil, a drying oil of relatively low viscosity.
2. Thin lithographic varnish, a more viscous liquid, prepared from linseed oil by heat treatment.
3. Liquid paraffin, a non-polar liquid.

Both linseed oil and thin lithographic varnish are accepted as being efficient in the dispersion of pigments, being at one time widely used for the manufacture of paints and printing inks. On the other hand, liquid paraffin, because of its non-polar properties, is recognised as being very inefficient in this respect.

The plots indicate that the strongest floccules are formed by the dispersion in linseed oil: the thin lithographic varnish dispersion has weaker floccules and that in

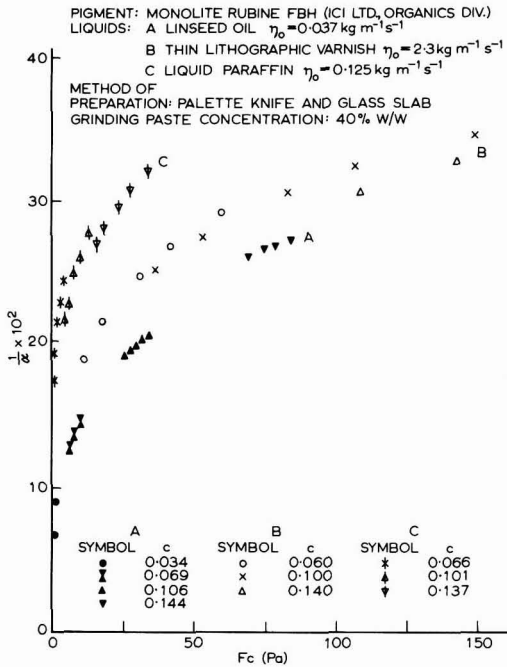


Figure 5.

liquid paraffin has the weakest of all. From the results of the previous experiment on the effect of degree of dispersion, it can be concluded that the linseed oil produces the finest dispersion, the thin lithographic varnish the next best and the liquid paraffin the worst dispersion of all. These findings are in agreement with the known properties of the

liquid paraffin in comparison with the other two liquids; it seems likely that the suggested difference between the other two is also valid. In a more extensive study, this could be checked by other means.

Discussion

With such a limited number of experimental results, most of which were not obtained with the present work in mind, it is clearly too soon to consider this method of interpreting rheological results to be of confirmed value. However, it would appear to be worthy of further investigation. In particular, it would be of interest to find out if the relation between $1/\alpha$ and F_c is independent of the individual values of F and c for a wide range of powder/liquid combinations.

Clearly, it cannot apply if basic changes occur during dilution, leading to, for example, permanent aggregation of the basic particles or changes in surface conditions which cause changes in the inter-particle forces. Equally, it cannot apply to systems which show marked hysteresis.

On the other hand, if sufficient evidence were to accrue to support the idea that F_c is a measure of the disruptive stress experienced by floccules, then the interpretation could be applied to the exceptional systems and used to make deductions about the changes taking place in them.

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Yellowing of alkyd paint films*

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Summary

The influence of the grade of titanium dioxide pigment on the yellowing of aged paint films has been studied. Using several yellowing environments, the results showed that an anatase pigmented film was more prone to yellowing than a non-coated rutile pigmented film, which in turn yellowed slightly more than a coated rutile pigmented film. The various surface treatments to which most rutile titanium dioxide pigments are subjected did

not affect the rate of yellowing, although a chloride route pigment and a fine crystal grade exhibited slightly less yellowing than a typical sulfate pigment.

From the point of view of retarding yellowing, the use of zinc oxide and acetoacetic ester was studied and it was evident that the former was more effective, particularly in the colloidal form.

Keywords

Types and classes of coatings and allied products

alkyd coating

Raw materials for coatings

prime pigments and dyes

titanium dioxide

Properties, characteristics and conditions primarily associated with

dried or cured films

yellowing

Le jaunissement des films de peintures alkydes

Résumé

On a étudié l'influence qu'exerce la variété du pigment de dioxyde de titane employé sur le jaunissement des films de peintures lors de leur vieillissement. Les résultats à partir de divers environnements, qui favorisent le processus de jaunissement, indiquent qu'un film de peinture pigmentée avec un type anatase est plus susceptible à jaunir qu'un film qui contient un type rutile sans enrobements, et ceci jaunit légèrement d'avantage qu'un film contenant du type rutile enrobé. Les divers traitements de surface que l'on accord à la plupart des pigments de dioxyde de titane du type rutile n'exerçaient aucune

influence sur la vitesse de jaunissement, bien qu'un pigment au chlore et un pigment du type cristal fin missent en évidence une tendance légèrement moins à l'égard du jaunissement qu'un pigment au sulfate ordinaire.

Au point de vue du ralentissement du processus de jaunissement, l'emploi de l'oxyde de zinc, et également de l'ester acétoacétique ont été étudiés et il était évident que l'oxyde de zinc, surtout sous forme colloïdale, était le plus efficace.

Die Vergelbung der Alkydanstrichstoffilme

Zusammenfassung

Der Einfluss des Titandioxidpigmenttyps auf die Vergelbung der gealterten Anstrichstoffilme wurden untersucht. Die Resultate unter verschiedenen Vergelbungsumgebungen zeigten, dass ein mit Anatas pigmentierter Film empfindlicher zur Vergelbung war als ein mit nicht nachbehandelten Rutil pigmentierter Film der etwa mehr als ein mit nachbehandelten Rutil pigmentierter Film vergelbete. Die verschiedenen Oberflächensbehandlungen, die die meisten Rutilpigmente bekommen,

wirkten keinen Einfluss auf die Vergelbungsgeschwindigkeit, obwohl ein Chlorpigment und ein feinkristaler Typ etwa wenige Vergelbung als ein typisches Sulfatpigment zeigten.

Vom Standpunkt der Vergelbungsverhinderung wurde der Anwendung der Zinkoxid und auch Acetoaceticester untersucht, und es war klar, dass der Zinkoxid, vor allem der Kolloidform, leistungsfähiger war.

Introduction

Refs. 1-7

The yellowing of white paint films on ageing, particularly indoors, is of considerable importance. Not only is yellowing unpleasant because of its appearance, it can also have a depressing psychological effect¹. As a result, considerable effort has been devoted to investigating the fundamental causes of yellowing². It is well known that:

1. Yellowing increases with the degree of chemical unsaturation of the base resin.
2. Choice of driers is important.
3. Contaminants such as the vapour of decomposed fats and tobacco smoke promote yellowing.
4. Darkness promotes yellowing.

It is generally accepted that the changes which cause

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Table 1
List of the various pigments evaluated

Pigment	Anatase or rutile	Crystal size (μm)	Process	Major inorganic constituents at pigment surface			Organic treatment
				$\text{Al}_2\text{O}_3(\%)$	$\text{SiO}_2(\%)$	$\text{ZrO}_2(\%)$	
A	anatase	0.15	sulfate				none
B	rutile	0.22	sulfate				none
C	rutile	0.23	sulfate	2.4	1.9		polyol (2)
D	rutile	0.23	sulfate	4.2	3.2	1.0	amine
E	rutile	0.17	sulfate	2.2	1.0		polyol (1)
F	rutile	0.24	chloride	5.4	2.0		polyol (1)
G	rutile	0.22	chloride	4.2	0.1		polyol (1)
H	rutile	0.22	chloride	4.2	0.1		polyol (2)
I	rutile	0.22	chloride	4.2	0.1		amine
J	rutile	0.22	chloride	4.2	0.1		none
K	rutile	0.24	chloride		1.4		polyol (1)
L	rutile	0.24	chloride	0.9	1.8		polyol (1)
M	rutile	0.24	chloride	1.8	0.9		polyol (1)
N	rutile	0.24	chloride	2.6			polyol (1)

yellowing occur in the base resin and, therefore, most investigations have primarily dealt with the binder. However, some results have indicated that the type of pigmentation can play a part in yellowing. For example, Chatfield³ observed yellowing when lithopone was used and this was attributed to the decomposition of zinc sulfide in lithopone to zinc and sulfur. Werthan *et al.*⁴ reported that the presence of zinc oxide has a tendency to reduce yellowing, and when titanium dioxide was also incorporated into the same paint, the paint film yellowed rather than bleached when exposed to light.

The term "titanium dioxide pigment" as used above is rather ambiguous since there are various commercial grades. For example, titanium dioxide is produced in two different crystalline forms - anatase and rutile. The latter exhibits better opacity and durability and is therefore used in most paint systems. Grades of titanium dioxide can vary in crystal size, type of inorganic coating and in the nature of the organic treatment they are subjected to prior to final plant milling. The crystal size plays a part in opacity and colour, whereas the surface treatment of titanium dioxide influences durability and dispersion characteristics⁵. Finally, the basic rutile crystal can be manufactured by either of two methods known as the "sulfate" and the "chloride" processes⁶. The latter process produces a pigment which is less yellow in tone because of the greater purity of the crystals. Thus there are various grades of titanium dioxide pigment which differ physically and chemically, and a programme of work was carried out in order to determine the extent to which the grade of titanium dioxide pigment affects the rate of yellowing.

As mentioned above it is well known that the addition of zinc oxide to an alkyd paint increases its resistance to yellowing. In 1977 Rakoff *et al.*⁷ reported that ozonized mono-olein and acetoacetic ester (ethyl-3-oxobutanoate) were also possible inhibitors of yellowing. Consequently, the performance of acetoacetic ester was compared with both colloidal and acicular zinc oxide with regard to inhibiting yellowing.

Experimental

Ref. 8

Method

Whilst considering the variables appertaining to titanium

dioxide pigment, it was decided to categorise the various factors in terms of the following:

1. Anatase versus rutile titanium dioxide.
2. A non-coated rutile versus a coated rutile grade of titanium dioxide.
3. The effect of inorganic coating composition.
4. The effect of organic treatment.
5. The influence of crystal size and method of manufacture (i.e. sulfate versus chloride process).

For this purpose, a variety of commercially available pigments were selected and in addition a few experimental grades were produced in order to cover all factors mentioned above. Consequently, fourteen grades of titanium dioxide pigment were considered and in Table 1 a list of their specifications is given.

For paint manufacture, four typical resins which were known to exhibit differences in yellowing were selected. The resins were as follows:

- (i) long oil soya modified alkyd
- (ii) urethane alkyd
- (iii) tall oil modified alkyd
- (iv) linseed modified alkyd

Paints (at a final pigment/binder ratio of 0.7:1) were prepared according to the formulation given in the Appendix. For all paints, a mixed drier solution (lead, cobalt and calcium naphthenate) was used such that the lead, cobalt and calcium metal levels were 0.5 per cent, 0.05 per cent and 0.1 per cent respectively on resin solids. Further paints based only on resin (i) were formulated using pigments B, C, F and G and zinc oxide (i.e. colloidal (0.2 μm mean size) and acicular (0.5 μm \times 2.0 μm)) such that the level of zinc oxide was 4 per cent and 8 per cent by weight on pigmentation. The pigment/binder ratio was 0.7:1. In addition, acetoacetic ester was added to the control paints (i.e. zinc oxide-free, with a pigment/binder ratio of 0.7:1) in order that levels of 2 per cent and 5 per cent on the weight of paint were obtained (i.e. equivalent to levels of 7 per cent and 18 per cent on the weight of pigment).

After ageing for one day, all paints were drawn down

on white cards measuring 10 cm × 15 cm with a No. 8 K bar applicator, to give a nominal wet film thickness of 100 μm. A second coat was applied the following day to ensure complete obliteration of the colour of the card's surface. The drawdowns were left to dry for one day in diffuse daylight before being measured for gloss, brightness and colour index, they were then exposed to the following environments which were known to cause some yellowing (NB paint films containing zinc oxide and acetoacetic ester were only exposed in 2 and 3).

1. A semi-dark room (max. temp. = 27°C)
(min. temp. = 19°C)
2. A dark oven (temp. = 60°C)
3. An atmosphere of ammonia (10 per cent concentration) in daylight
(max. temp. = 27°C)
(min. temp. = 20°C)
4. Natural daylight in a greenhouse
(max. temp. = 50°C)
(min. temp. = 9°C).

The paint films were removed at periodic intervals and measured for gloss, brightness and colour index, further details of which are given below.

Measurements

Gloss

Using a Byk-Mallinckrodt glossmeter conforming to BS 3900:Part D5: 1980, either 60° or 20° gloss was measured over several areas of each drawdown, the average value being calculated to the nearest integer.

Brightness and colour

Brightness and colour index (CI) values were measured using a Gardner XL-23 colorimeter where:

$$\text{colour index} = \frac{\text{reflectance (red)} - \text{reflectance (blue)}}{\text{reflectance (green)}} \times 100$$

Increasing positive values of CI indicate an increase in yellow tone, increasing negative values an increase in blue tone, a "neutral" white having a CI of zero. The reflectance in green light was taken as indicating the total brightness of the paint film.

Dispersion

In order to evaluate the degree of pigment dispersion in several of the paint systems, the flocculation gradient⁸ was determined as follows:

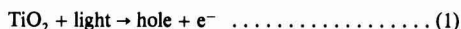
Four paint films of different wet film thicknesses were drawn down on polyester films using 3, 4, 5 and 6 K bar applicators. After drying, the reflectance of infrared radiation at a wavelength of 2.5 μm was measured using a Beckman spectrophotometer and the reflectance value plotted against film thickness. The gradient of the line produced gives an indication of the degree of flocculation in the dry film, the larger the value the greater the degree of flocculation.

Results and discussion

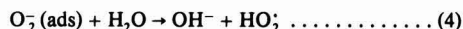
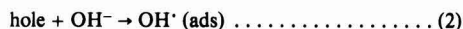
Refs. 9-14

Anatase versus rutile titanium dioxide

In order to simplify the large amount of information obtained from all exposures, the change in colour index (ΔCI) was taken to represent the degree of yellowing. Thus, ΔCI values for pigments A (non-coated anatase) and B (non-coated rutile) in all four resins and environments are given in Table 2. For the ammonia exposure, results were taken after three weeks, whereas with the remaining three environments results were recorded after 4 weeks. It can be seen from these results that irrespective of resin there is a significant difference in yellowing between pigments A and B in ammonia and in the greenhouse, a small difference in the oven, and no difference in the semi-dark room. The presence of light and heat thus seem to be relatively important factors in highlighting differences between anatase and rutile titanium dioxide. As mentioned in the introduction, it is well known that the anatase crystal is more photoactive (i.e. less durable) than the rutile. When titanium dioxide is exposed to ultraviolet radiation which is present in normal daylight, a photochemical reaction occurs in the crystal in which electrons are excited from the valence band to the conduction band, leaving positive vacancies (holes) in the valence band:



Both electrons and holes are free to move about the crystal and in doing so either recombine or reach the surface where they can initiate chemical reactions. On reaching the surface, positive holes react with surface hydroxyl groups to form adsorbed hydroxyl radicals, whilst electrons react with adsorbed oxygen to form the O_2^- radical ion which then attacks a water molecule to form a perhydroxy (HO_2) radical⁹:



Since the electron band gap for anatase is greater than that for rutile, the above reactions occur more readily in anatase and therefore it is most likely that more perhydroxy radicals will form in this grade. According to Elm¹⁰ the presence of hydroperoxides in a paint film can lead to the formation of yellow unsaturated ketones, and this is a possible reason why an anatase pigmented film is more prone to yellowing on exposure to light (NB it can be seen in Table 2 that the biggest difference between pigments A and B occurred in the greenhouse).

In order to determine if the absence of light affects the relative rate of yellowing of films (pigments A and B in resin (i)) exposed to ammonia, the same experiment as above was repeated but this time the chamber containing the ammonia and the films was kept in a dark room. It was found that the ΔCI values after 1 week for pigments A and B were 24 and 22 respectively, which illustrates that for this test, light is not a prerequisite for producing differences in the rate of yellowing between anatase and rutile titanium dioxide (cf. oven exposure results). This would therefore suggest that the difference in yellowing between anatase and rutile titanium dioxide cannot be completely attributed to differences in photoactivity, even

Table 2
Change in colour index (ΔCI) for anatase (A) and rutile (B)
grades of titanium dioxide

Resin	ΔCI in ammonia		ΔCI in semi-dark room		ΔCI in oven at 60°C		ΔCI in greenhouse	
	(3 weeks)		(4 weeks)		(4 weeks)		(4 weeks)	
	A	B	A	B	A	B	A	B
(i)	26.0	22.5	4.5	3.0	22.5	18.0	7.0	1.0
(ii)	25.5	22.0	3.0	3.0	20.0	19.5	4.5	1.0
(iii)	28.0	23.5	3.0	3.0	21.5	19.5	5.5	0.5
(iv)	29.0	26.0	5.5	5.5	22.0	21.0	6.0	0.5

Table 3
Change in colour index (ΔCI) for pigments B, C and D in resin (i)

ΔCI in ammonia			ΔCI in semi-dark room			ΔCI in oven at 60°C			ΔCI in greenhouse		
(3 weeks)			(4 weeks)			(4 weeks)			(4 weeks)		
B	C	D	B	C	D	B	C	D	B	C	D
22.5	21.0	20.5	3.0	3.0	3.0	18.0	16.5	16.5	1.0	-1.0	-1.5

though the order of performances in three of the environments corresponded to the photoactivity of each pigment.

From the point of view of brightness, there is a direct correlation with the extent of yellowing, in as much as brightness decreased as yellowing progressed. This is shown in Figure 1 where some typical results illustrate the decrease in brightness for two paint films (pigments A and B in resin (i)) exposed to ammonia. It is evident that the decrease in brightness for A is much greater than for B and this is primarily due to the fact that A has yellowed more than B. Similar results were obtained with the remaining three resins, with paints based on resin (iv) exhibiting the greatest decrease in brightness, which would be expected because a linseed modified alkyd is more prone to yellowing (see Table 2) than the other resins, due to its high linolenic acid content¹¹.

The effect of inorganic coating on rutile titanium dioxide and the influence of inorganic coating composition

It was shown above that a highly photoactive pigment such as anatase titanium dioxide is more prone to yellowing than rutile titanium dioxide. Since one of the primary purposes of applying an inorganic coating to titanium dioxide is to reduce its photoactivity, it would be expected from the previous results that this could influence the rate of yellowing if films were exposed to light and heat. In order to determine if this was the case, pigments B (non-coated), C, D and K to N were selected for evaluation. Since a considerable number of commercial pigments are coated with hydrated forms of alumina and silica, pigments K to N were chosen in order to illustrate the effect of changing the SiO_2/Al_2O_3 ratio. Considering initially pigments B, C and D in resin (i), it can be seen from the ΔCI values which are given in Table 3 that although both coated grades (C and D) are similar in terms of yellowing, pigment B (non-coated) has yellowed slightly more in ammonia, in the greenhouse and in the oven. The greenhouse results also show that with the coated grades

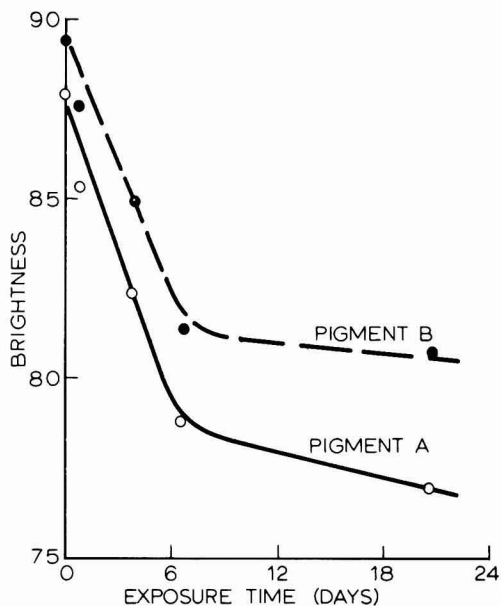


Figure 1. Change in brightness of two paint films (pigments A and B in resin (i)) exposed to a 10 per cent ammonia environment

of titanium dioxide the paint films have bleached, which is to be expected if titanium dioxide plays an inert role in yellowing since it is well known that oleoresinous films bleach when exposed to sunlight¹². Brightness measurements (Figure 2, pigments B and C in ammonia environ-

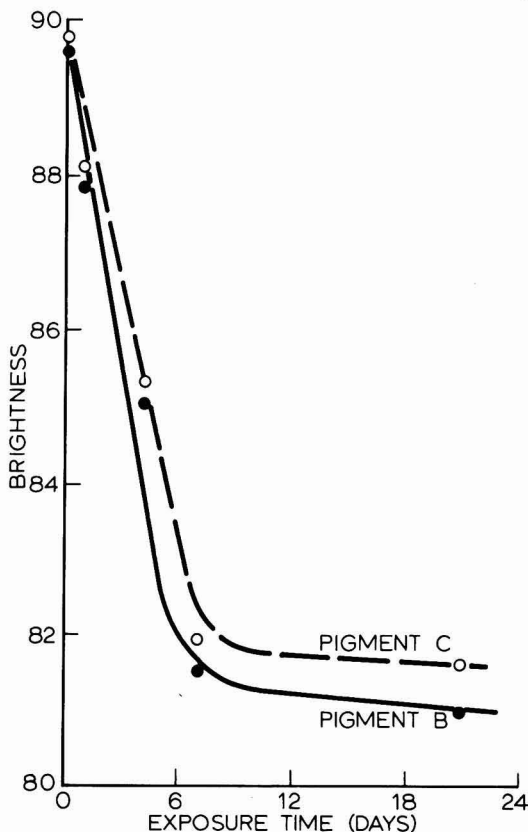


Figure 2. Change in brightness of two paint films (pigments B and C in resin (i)) exposed to a 10 per cent ammonia environment

ment) also illustrated the fact that for the uncoated grade of titanium dioxide, the decrease in brightness was slightly greater than with the coated grade.

Since the coating compositions of pigments C and D are different, the results in Table 3 suggest that the degree of yellowing has not been affected by the type of coating. To illustrate this further, pigments K to N and resin (iv) were used to produce paint films which were exposed to all four environments, and in Table 4 ΔCI values are listed. Although there is some scatter in the data, within experimental error there are no significant differences between

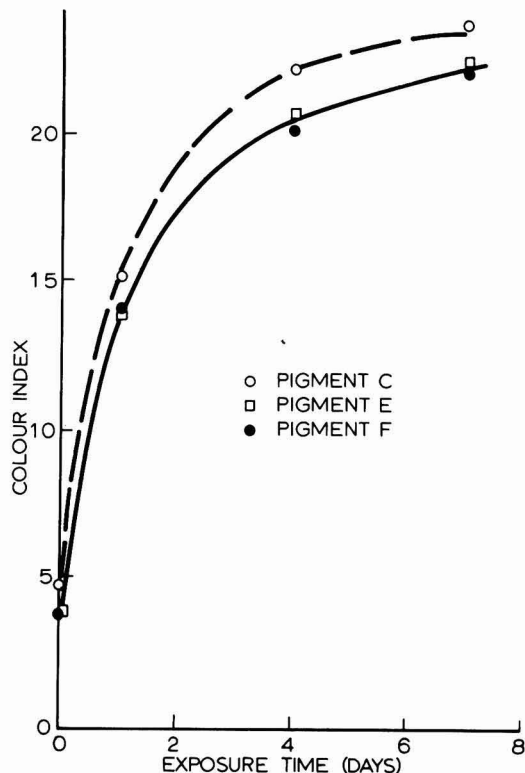


Figure 3. Change in colour index as a function of exposure time for films exposed to a 10 per cent ammonia environment (resin (ii))

the four pigments. (NB further results given below also indicate that the nature of the inorganic coating plays a negligible role in affecting the rate of yellowing.)

These results therefore suggest that as long as the grade of titanium dioxide exhibits moderate durability (i.e. it is coated), the type and amount of coating will not significantly affect the rate of yellowing.

The effect of organic treatment

Since it is thought that certain organic compounds react with drier catalysts and in doing so retard the auto-oxidation process, it is possible that the nature of an organic pigment treatment could affect yellowing; Robey

Table 4
Change in colour index (ΔCI) for pigments K to N in resin (iv)

Pigment	ΔCI in ammonia (1 week)	ΔCI in semi-dark room (4 weeks)	ΔCI in oven at 60°C (4 weeks)	ΔCI in greenhouse (4 weeks)
K	33.0	5.5	21.0	0.5
L	32.5	5.0	22.0	0.0
M	31.5	5.0	23.0	-0.5
N	31.5	5.0	23.0	-0.5

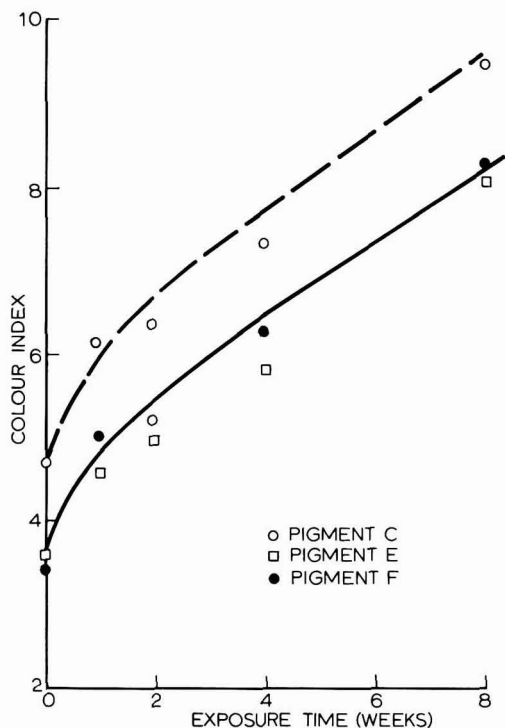


Figure 4. Change in colour index as a function of exposure time for films in a semi-dark room (resin (ii))

and Rybicka¹³ have suggested that products of advanced auto-oxidation are precursors of coloured species. Thus pigments G (polyol (1) treated), H (polyol (2) treated), I (amine treated) and J (non-organic) which had the same inorganic coating were selected and paints were made using the four resins. In Table 5 Δ CI values are given for these paint films after exposure to all four environments and it is evident that, within experimental error, there is little difference between the four pigments.

The influence of crystal size and method of manufacture

It was mentioned above that pigments produced via the chloride process generally exhibit better colour (i.e. bluer tone) than sulfate process pigments. However, a way by which a sulfate pigment can be made slightly bluer in tone, is to reduce its crystal size such that it preferentially scatters the shorter wavelengths (i.e. the blue end) of the light spectrum. Such pigments are often referred to as fine crystal grades and are primarily intended for the plastics industry. Thus it was decided to select a chloride process grade of titanium dioxide (pigment F), a fine crystal sulfate grade (pigment E) and a standard sulfate grade (pigment C) in order to see if a better coloured grade retards the rate of yellowing (NB inevitably all three pigments had different organic/inorganic coatings but it was shown above that the surface treatment does not affect the rate of yellowing). Since the results were similar irrespective of resin, only data which were obtained from one resin system (resin (ii)) are shown in figures 3, 4 and 5 where colour index is plotted against exposure time for three

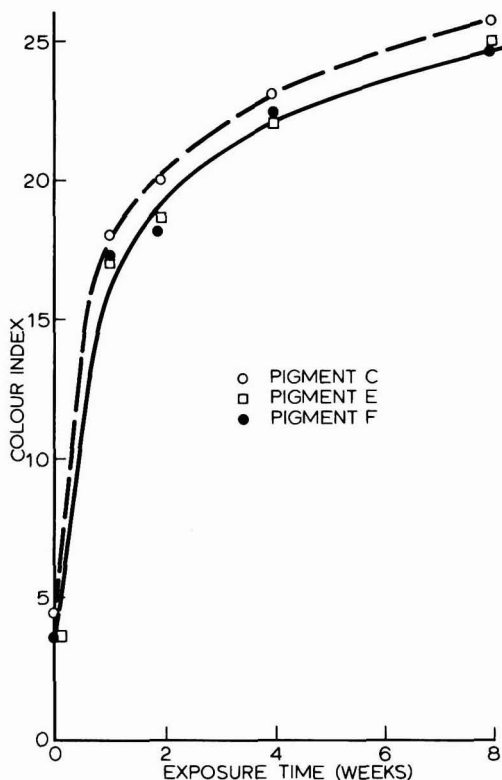


Figure 5. Change in colour index as a function of exposure time for films in an oven at 60°C (resin (ii))

environments (i.e. 10 per cent ammonia, semi-dark room and a dark oven at 60°C). It is evident from these results that the initial colour of films containing pigments F and E is similar, and that this colour is slightly better than the film pigmented with grade C. However, the rates of yellowing of all three pigmented systems are the same in all three environments. Consequently, pigments F and E still exhibit a slight colour advantage over C after yellowing.

Relationship between natural and accelerated yellowing conditions

From the results given so far (e.g. Table 5), it is clear that the order of performance of each environment in terms of the rate of yellowing is as follows:

1. 10 per cent ammonia
2. oven at 60°C
3. semi-dark room
4. greenhouse

Since most paint films bleached in the greenhouse, this condition could not be considered as a "yellowing" environment. However, films in a 10 per cent ammonia environment and a 60°C oven yellowed rapidly (e.g. figures 3 and 5) and therefore these tests would be termed accelerated. In order to compare the rates of yellowing in

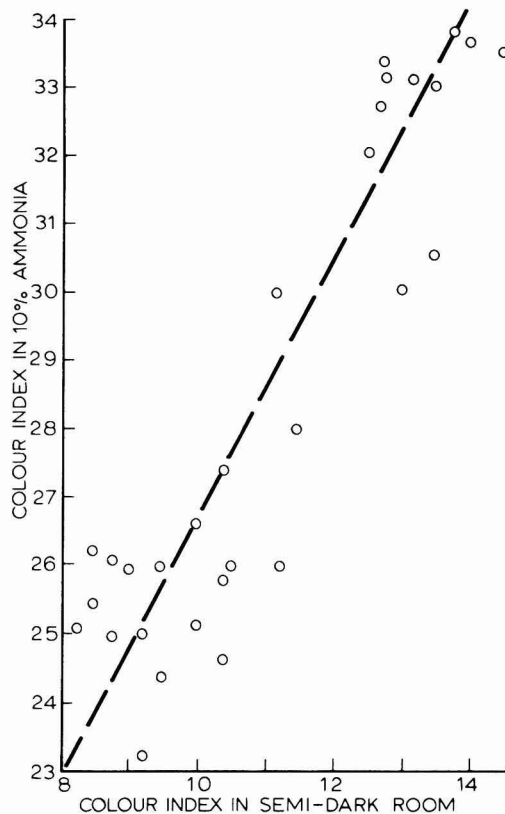


Figure 6. Comparison of colour index between a 10 per cent ammonia environment and a semi-dark room

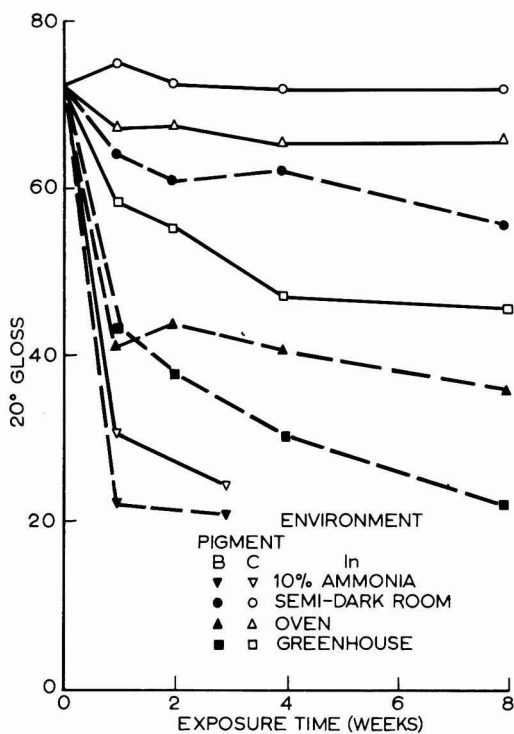


Figure 7. Change in gloss for pigments B and C in resin (i) as a function of environment

both of these environments with that of the semi-dark room (natural yellowing condition), regression lines were fitted for each comparison and correlation coefficients calculated. In Figure 6 a typical plot of colour index for films in a 10 per cent ammonia environment versus colour index in a semi-dark room is given. It was found that

Table 5
Change in colour index (ΔCI) for pigments G to J

Environment	Pigment	Resin (i)	Resin (ii)	Resin (iii)	Resin (iv)
ΔCI in ammonia (3 weeks)	G	23.0	21.0	24.0	26.0
	H	21.5	21.0	24.0	27.0
	I	22.0	22.0	22.0	26.0
	J	23.0	19.0	25.0	27.0
ΔCI in semi-dark room (4 weeks)	G	2.5	2.0	2.5	5.0
	H	2.5	2.5	3.0	5.0
	I	2.5	2.5	2.5	5.5
	J	2.5	3.0	3.0	5.0
ΔCI in oven at 60°C (4 weeks)	G	16.0	17.5	18.0	20.5
	H	16.5	17.5	17.5	19.5
	I	16.0	17.5	19.0	19.5
	J	17.0	17.5	18.0	19.0
ΔCI in greenhouse (4 weeks)	G	-1.5	-1.5	-1.0	-2.0
	H	-1.5	-1.5	-1.0	-2.0
	I	-1.5	-1.5	-1.0	-2.0
	J	-1.0	-1.0	-1.5	-2.0

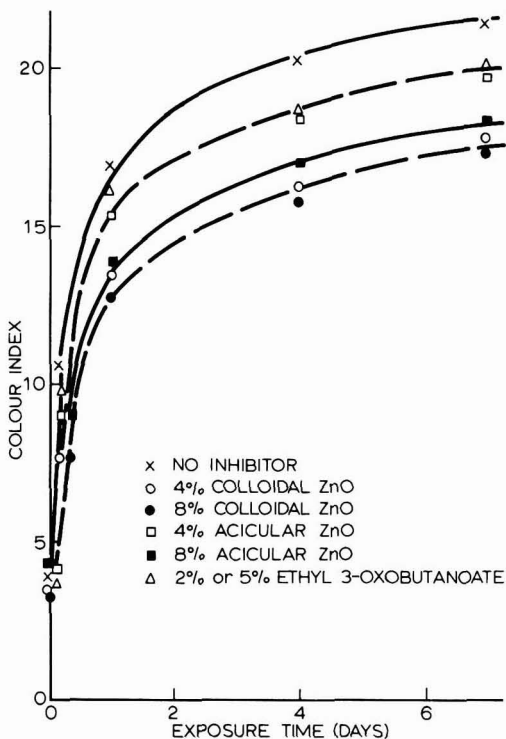


Figure 8. Change in colour index in a 10 per cent ammonia environment as a function of amount of yellowing inhibitor (resin (i))

values of the correlation coefficients for ammonia versus semi-dark room, and oven versus semi-dark room were 0.78 and 0.79 respectively which fall within a 99.99 per cent confidence factor. Thus, the results indicate that correlation between natural and "accelerated" yellowing is significant. In marked contrast, there were differences in the rate at which gloss changed as a function of environment. Generally, the grade of titanium dioxide did not affect the overall result, although it was apparent that if the grade was not well dispersed the decrease in gloss was much greater. This is illustrated in Figure 7 where gloss is plotted against exposure time for films (based on resin (i)) pigmented with grades B (non-coated rutile) and C (coated rutile). Flocculation gradient values for paints containing pigments B and C were 0.32 and 0.16 respectively, indicating that the non-coated grade of titanium dioxide was not as well dispersed as the coated grade. Since the decrease in gloss is to some extent attributable to paint film shrinkage which results in titanium dioxide having a greater influence on surface roughness and therefore gloss¹⁴, the degree of pigment dispersion becomes more important. With regard to the test environments, there was only a very small decrease in gloss in the semi-dark room compared with the 60°C oven and this was presumably due to greater film shrinkage caused by the latter environment. However, with the 10 per cent ammonia environment, a rapid decrease in gloss occurred which was possibly due to mild chemical attack of the surface causing the surface roughness to increase and therefore the gloss to decrease. In the greenhouse, paint films

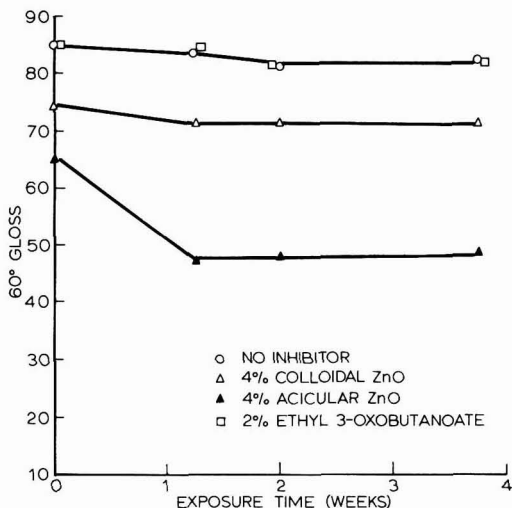


Figure 9. Change in gloss of films containing yellowing inhibitors in an oven at 60°C (resin (i))

reached temperatures of the order of 50°C and the relative humidity was as high as 60 per cent. Consequently in this environment, the decrease in gloss which was quite significant, was likely to be the result of film shrinkage and photodegradation (i.e. weathering). The latter mechanism was particularly evident with the anatase pigmented films where after eight weeks exposure, severe chalking was occurring.

The effect of yellowing inhibitors

It was evident from the above results that once titanium dioxide has an inorganic coating, the rate of yellowing is independent of the type of titanium dioxide pigment. Similarly, when the yellowing inhibitors were used (i.e. zinc oxide and ethyl-3-oxobutanoate) it was found that the nature of the titanium dioxide played a negligible role in influencing the rate of yellowing. Thus, results obtained from films containing only pigment C (coated rutile) will be given.

From the point of view of yellowing, it was apparent that the results were similar, irrespective of the environment (i.e. 10 per cent ammonia or an oven at 60°C). Consequently, only data from the 10 per cent ammonia environment are shown in Figure 8 where colour index is plotted against exposure time. It can be seen that the most effective inhibitor is colloidal zinc oxide and that there is little difference between the 4 per cent and 8 per cent levels. Acicular zinc oxide is second in performance, although at the 4 per cent level its resistance to yellowing is similar to ethyl-3-oxobutanoate (at both 2 per cent and 5 per cent levels). Thus, the inhibition of yellowing by acetoacetic ester is not particularly good but it does, to some extent, retard the rate of yellowing.

However, the advantage of using an organic liquid is that it is unlikely to impair the gloss of a paint (assuming it does not flocculate the pigment), whereas an inorganic oxide could if its particle size is relatively large. This effect is illustrated in Figure 9 where gloss is plotted against exposure time for films containing 4 per cent colloidal zinc

oxide, 4 per cent acicular zinc oxide and 2 per cent ethyl-3-oxobutanoate, exposed in a 60°C oven. It can be seen that the relatively large (i.e. 0.5µm × 2.0µm) acicular zinc oxide has produced a significant decrease in gloss which is further highlighted when the film shrinks during exposure. Similarly, the colloidal zinc oxide has reduced the gloss of the paint, although not to the same extent as the acicular grade, whereas the ethyl-3-oxobutanoate has not affected the gloss.

It is therefore clear from these results that yellowing inhibitors such as zinc oxide can be quite effective. Unfortunately, there are certain detrimental side effects such as poor gloss retention and possible reactivity of the paint during storage which must be taken into account when zinc oxide is used.

Conclusions

It has been shown that in a semi-dark room at ambient temperature (i.e. 25°C) the grade of titanium dioxide pigment does not significantly affect the rate of yellowing. However, if the temperature of the room is increased or films are exposed to light and atmospheric contaminants such as ammonia, then an anatase pigmented film yellows slightly more than a non-coated rutile pigmented film. The presence of an inorganic coating on the surface of a rutile titanium dioxide pigment slightly reduces the rate of yellowing, although the type of inorganic coating and organic surface treatment seems to have no influence on the rate of yellowing. If the total amount of yellowing is to be kept to a minimum then it is advantageous to select either a chloride route pigment or a fine crystal sulfate grade, since pigments of this type exhibit slightly better initial colour.

It was evident that zinc oxide and ethyl-3-oxobutanoate retarded the rate of yellowing, the former product being more effective particularly when it was in colloidal form.

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Appendix

Paint formulation

	Parts by weight
Titanium dioxide pigment	50.0
Resin (i) (20% n.v.)	20.0
Ballotini (8 mm)	130.0
Ballmill for 16 hours, then add	
Resin (i) 70% n.v. as received)	96.0
Mixed driers	7.0
White spirit	10.0
Methyl ethyl ketoxime	0.5

NB For resins (ii), (iii) and (iv), small modifications were made in order to take into account the differences in non-volatiles content of the received resins.

Next month's issue

The Honorary Editor has accepted the following papers for publication. They are expected to appear in the January issue of the *Journal*:

The truth about water dispersed epoxy coatings by *L. Field*

Foam control in the surface coatings industry by *K. M. Priestman*

Electrochemical impedance on coated metal electrodes. Part 4: The effect of overpotential by *L. M. Callow and J. D. Scantlebury*

Masonry paints and cleaning methods for walls affected by organic growth by *P. Whiteley and A. F. Bravery*

Problems involved in determining the solids content of amino resins, taking melamine resins as an example

By R. Stange, L. Klug and H. Klug

BASF Aktiengesellschaft, Ludwigshafen, West Germany

Summary

1. The tolerances laid down in national standards for determining the solids content of amino resins are by no means too wide and are fully accepted in practice. In fact, there is a likelihood that they may be exceeded by some experimental values that conform to a normal distribution.

2. The vacuum method yields a range of scattering that is wider than that from the stoving method. The IUPAC method now in preparation (Tentative Method, 3rd draft, March 1978) involves reducing the layer thickness, and this would appear to be a way out of the dilemma.

3. Gas chromatography (GC) yields data between the two extremes from the vacuum and the nonvolatiles methods. The more reactive the resin, the nearer the GC value to the nonvolatiles value.

4. The current West German and French standards (DIN 53 216 and AFNOR T 30-011) forbid the testing of amino resins and finishes containing them at temperatures above the reactivity threshold.

5. All previous methods have yielded data in "response" to certain specified conditions, i.e. they are not characteristic product figures.

6. Advantages of the new method are as follows.

Information with a degree of certainty r^2 of over 0.98 is obtained by determining the solids content of amino resins, particularly of melamine-formaldehyde resins, by statistical evaluation of weight loss curves.

Using the formula $y = a + b \cdot \ln x$, in which a and b are constants, it is possible to determine, by interpolation the solids content that would be expected with other stoving schedules (at the same temperature). It is also possible with this method to calculate the solids content expected from processing under certain conditions.

By extrapolating the curve to $t \rightarrow 0$, the solids content of the product as supplied can be reliably calculated. This figure could then be used in labelling. The exact solids content can be found by extrapolating to between $t = 0.1$ h and $t = 0.01$ h.

The constant b of the logarithmic equation corresponds to the slope of the weight loss curve. However, b is not a direct measure of the amino resin's reactivity, which must be interpreted from the entire curve. The work described in this paper will be continued with the aim of producing a quantitative description of the speed of curing.

Keywords

Properties, characteristics and conditions primarily associated with

bulk coatings and allied products

nonvolatile content
volatile content

Specifications, standards and regulations

test method

Raw materials for coatings

binders (resins, etc.)

alkyd resin
melamine resin

Miscellaneous terms

statistics

Une étude, en utilisant les résines mélamines à titre d'exemple, sur les problèmes associés à la détermination de la teneur en extrait sec des résines aminées

Résumé

1. Les tolérances pour la teneur en extrait sec citées dans les normes nationales ne sont pas les valeurs extrêmes, mais celles qui sont utilisées dans la pratique. Au contraire, afin d'être en conformité à la répartition normale, il est assez certain que quelques données doivent se trouver au-delà de la tolérance prescrite.

2. La méthode à vide rend, forcément semble-t-il, une répartition des données qui est plus large que celle rendue par la méthode à l'étuve. La méthode de l'IUPAC qui est actuellement en cours de préparation (Méthode Tentative, 3^{ème} Projet du mars 1978) prévoit la diminution de l'épaisseur de la couche de résine. Il semble que cette modification offre la possibilité de résoudre ce dilemme.

3. La chromatographie en phase gazeuse donne les valeurs qui se trouvent entre les valeurs rendues par la méthode à vide et celles de la teneur en extrait sec rendues par la méthode à l'étuve. Plus la résine est réactive, plus la valeur donnée par la chromatographie en phase gazeuse est proche de la valeur donnée par la méthode à l'étuve.

4. Les normes actuelles (DIN 53216 et AFNOR T 30-011) interdisent les essais, aux températures qui dépassent le seuil de réactivité sur les résines aminées et également sur les finitions contenant ces résines.

5. Toutes les méthodes déjà en vigueur ne donnent des chiffres

qu'en réponse aux questions spécifiques, c'est-à-dire, qu'ils ne sont pas des chiffres typiques au produit.

6. Les avantages de la nouvelle méthode:

Les résultats ayant un degré de certitude r^2 supérieur à 0,98 sont obtenus par la détermination de la teneur en extrait sec des résines aminées, et en particulier les résines mélamine-formols, grâce à une évaluation statistique des courbes de la perte de poids. En se servant de la formule $y = a + b \cdot \ln x$, où a et b sont des constantes définies, il est possible de calculer par interpolation les teneurs en extrait sec auxquelles on doit s'attendre en utilisant des différents temps d'étuvage à la même température. De cette manière il est possible d'ailleurs de calculer la teneur en extrait sec à laquelle on doit s'attendre sous certaines conditions dans la pratique.

Problematik der Festkörperbestimmung bei Aminoharzen, vorgetragen am Beispiel von Melaminharzen

Zusammenfassung

1. Die in den nationalen Normen angegebenen Toleranzen der Festkörperbestimmung sind keine Extremwerte, sondern werden in der Praxis voll in Anspruch genommen. Im Gegenteil, es besteht eine gewisse Wahrscheinlichkeit, daß wegen der Gesetzmäßigkeiten der Normalverteilung ein Teil der Meßwerte außerhalb der Solltoleranzen liegen muß.

2. Die Vakuum-Methode liefert – offenbar grundsätzlich – einen Streubereich für die Meßwerte, der größer ist als der bei der Einbrennmethode. Einen gewissen Ausweg aus diesem Dilemma scheint die in Ausarbeitung befindliche IUPAC-Methode (Tentative Method, 3. Draft v. März 1978) in einer Verringerung der Schichtdicke zu suchen.

3. Die Gaschromatografie liefert einen Wert, der zwischen den beiden Extremen Vakuum und nFA liegt. Je reaktiver das Harz, um so dichter liegt der GC-Wert beim nFA.

4. Die gegenwärtig existierenden Normen (DIN 53 216 und AFNOR T 30-011) verbieten die Prüfung von Aminoharzen und aminoharzhaltigen Lacken bei Temperaturen oberhalb der Reaktivitätsschwelle.

5. Alle bisherigen Methoden liefern nur jeweils eine bestimmte Fragestellung beantwortende Ergebnisse. Sie sind nicht charakteristische Produktangaben.

Determining the solids content of an aqueous or organic solution is not peculiar to paint technology but is well known in inorganic or organic analytical chemistry. However, in the latter case the materials involved are usually crystalline and rarely amorphous; prior to quantitative determination, they are converted into a form in which there are practically no bonds with the solvent. Drying must be carried out only under such conditions as are suitable to remove quantitatively the solvent, usually adhering to the surface, and the only limit placed on the usual treatment at elevated temperatures is that the solids content must remain intact. Drying is continued until the weight is constant.

The peculiarities of paint resins are caused by two features. The first is that the solid substances must be subjected to very much lower temperatures, either because the components at the lower end of the molecular weight distribution range are volatile, or because the substances themselves begin to decompose or react together. In all these instances, the solids content (SC) is no longer a constant but a function of time (t) and temperature (T) i.e.,

La teneur en extrait sec du produit prêt à la livraison peut être calculée, en faisant l'extrapolation de la courbe $t \rightarrow 0$. Cette valeur peut être utilisée pour répondre aux besoins légaux d'étiquetage. La teneur précise en extrait sec se trouve entre $t = 0,1h$ et $t = 0,01h$.

La pente de la courbe de la perte de poids est gouvernée par la constante b . Cependant, b ne donne pas de mesure précise de la réactivité de la résine aminée qui fait l'objet de la détermination, mais cette valeur doit être dérivée à partir du tracé entier de la courbe.

L'étude décrite dans cet article sera continuée en vue d'élaborer une description quantitative de la vitesse de cuisson.

6. Vorteile der neuen Methode:

Die Bestimmung des Festgehaltes von Aminoharzen, insbesondere von Melamin-Formaldehydharzen, durch statistische Auswertung von Gewichtsverlustkurven ermöglicht Aussagen mit hoher Sicherheit (bei einem Bestimmtheitsmaß r^2 von über 0,98).

Mit Hilfe der Formel $y = a + b \ln x$, wobei a und b bestimmte Konstanten sind, ist es möglich, durch Interpolation Festgehalte zu berechnen, die bei anderen Einbrennzeiten (bei gleicher Temperatur) zu erwarten sind. Hierdurch läßt sich auch der Festgehalt berechnen, der bei der Verarbeitung unter bestimmten Praxisbedingungen zu erwarten ist.

Durch Extrapolation der Kurve $t \rightarrow 0$ kann der Festgehalt in Lieferform berechnet werden. Dieser Wert kann für die Kennzeichnungspflicht verwendet werden. Der exakte Festgehaltswert liegt zwischen $t = 0,1h$ und $t = 0,01h$.

Die Konstante b der logarithmischen Gleichung ist für die Steilheit der Gewichtsverlustkurve verantwortlich. Allerdings ist b nicht direkt ein Maß für die "Reaktivität" des gemessenen Aminoharzes, sondern muß aus dem ganzen Kurvenverlauf interpretiert werden. Die hier beschriebenen Arbeiten werden mit dem Ziel einer zahlenmäßigen Beschreibung der Häufigkeit fortgesetzt.

$$SC = f_{(t,T)}$$

The second feature is that strong forces of association take effect between the resins and the solvents and thus make it extremely difficult to expel the final solvent residues from the solidifying resin.

Figure 1 shows typical curves for the evaporation of ethanol from various resin solutions.

The curves were obtained on an automatic balance at room temperature. All resins have practically the same softening range.

Without going into details on the interactions between solvent and resin, the curve can be divided into three different functional sections (Figure 2).

Phase (a), which could be described as that in which vapourisation takes place under normal conditions, is linear. The slope is governed by external conditions, i.e. temperature, pressure, air movement etc., and depends on

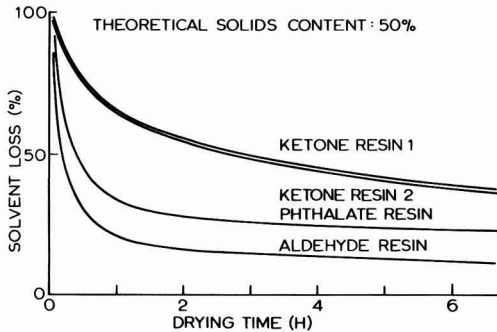


Figure 1. Solvent loss from 50% resin solutions

whether the test surface (usually a tin lid) is completely covered by the specimen. The slope is the same if pure solvent is used instead of resin solution. The viscosity of the resin solution is still quite near to that of the solvent. The release of solvent at the surface, which would normally result in a concentration gradient within resin solutions, is compensated by convection within the resin solution, which occurs so quickly that the concentration of solvent at the surface remains constant.

Phase (b) is distinguished by the fact that it is increasingly difficult to equalise the concentration between the surface and the interior as a result of the rising viscosity. Solvent release is greatest, therefore, at the outermost surface, which means that in extreme cases, e.g. with certain acid-curing paints, a solid skin forms on the paint while it is still liquid in the interior. However, solvent is still being transported to the surface by convection.

Phase (c) begins when the material being tested is no longer free-flowing. From this point, the solvent still remaining must be transported to the surface by diffusion. This is also not a constant factor and also decreases in speed as drying proceeds. With chemically reacting resins and paints, the crosslinking density can be such that the final solvent residues are enclosed as in a cage and can no longer diffuse.

These examples show the factors which can influence the expected measurements to some degree. The length of phase (a) is governed decisively by the change in viscosity as a function of the rise in concentration at the test temperature. Resins which have particularly low melting points and/or solution viscosities at high concentrations display a particularly long phase (a) at the expense of (b) and (c) (in Figure 1, an aldehyde resin). In phase (b) great importance is attached to the viscosity and to the rate at which the concentration is equalised by it. In contrast, the viscosity in phase (c) is no longer of importance and the rate at which solvent is given off is governed by diffusion of solvent to the surface. An over proportional influence due to the layer thickness is also to be expected as the diffusion pressure potential is distributed over a greater or lesser film thickness.

From this it can be seen that the ascertainable solids content is not merely (as stated above) a function of t and T , but, within the scope of laboratory conditions, it also becomes a function of the layer thickness, the viscosity, the drying oven and prevailing air circulation, the position of the specimen in the oven, the solvent concentration in

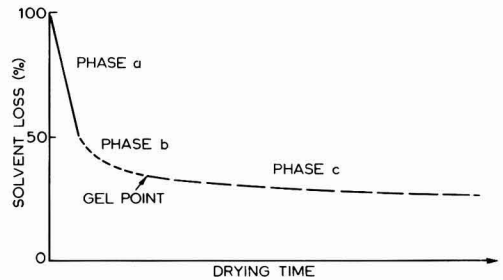


Figure 2. Solvent liberation (schematic)

the oven atmosphere, etc. If an exact comparison of two resin solutions is made, it is shown that the figures obtained are really only comparable when both resins are weighed in almost exactly the same amounts (because of the overproportional influence of the layer thickness), and, of course, when the other conditions are kept as similar as possible. Nevertheless, particular national test standards still permit errors that would be unacceptable in a scientific/chemical laboratory. The German standard, DIN 53 216, allows for an error of 2 per cent (comparability) and 4 per cent (reproducibility); the French standard, T 30-011, stipulates that no individual figure may vary by more than 2 per cent from the average figure.

All these statements apply to resins that do not change during testing. A further complication exists in the testing of resins that give off volatile products of decomposition under the measurement conditions. During phase (a), i.e. whilst there is still an excess of solvent, there is usually no reaction, this does not set in until the bulk of the solvent has already evaporated and the specimen is nearly at the prescribed temperature. This requires 10-20 minutes, the actual time depending on the type of solvent. (Incidentally, a difference of 10 minutes in the time taken to reach the specified temperature has, in fact, exactly the same effect as a corresponding change in the entire period for which the temperature is applied.)

When volatiles commence to be given off to the full extent, phase (c) is already attained, i.e. before vaporisation, and the products of elimination must diffuse through the solid resin. Equilibrium is then established between the solvent still present and the products continually supplied by the reaction.

If, as is the case with amino resins, the product of the elimination reaction is identical to the solvent in which the resin has been dissolved, it can never be said at which point solvent evaporation ends and elimination begins. The question as to whether it is possible to differentiate between original and eliminated solvent is dealt with below.

In addition to all the factors already mentioned, an important part is also played by the reactivity of the resins (different cleavage rates), the crosslinkage density (varying degrees of hindrance to diffusion) and also the diffusion properties of the elimination products (molecular weight, polarity etc.). The measured values obtained for various resins under these circumstances not only involve a great deal of error but are also not comparable. For example, it could not be expected that two samples of melamine formaldehyde resin with the same solvent content, one

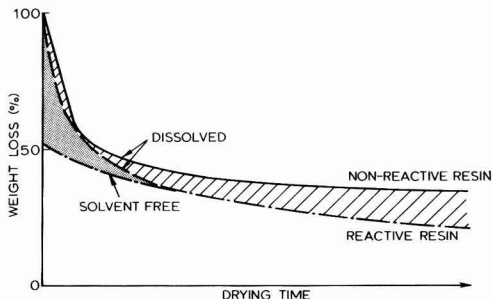


Figure 3. Weight loss with reactive and non-reactive resins

etherified with methanol and the other etherified with butanol, would have the same nonvolatile content.

This is obviously one of the reasons why testing of resins and coatings which undergo elimination is not permitted in DIN 53 216 or in AFNOR T 30-011.

However, these two standards do differ. In the DIN standard, testing of such resins is simply not permitted; in contrast, in the AFNOR standard it is stipulated that the test must be carried out in such a manner that no elimination can occur (sans toutefois entraîner une dégradation du produit étudié).

In view of this fact, there have been no lack of attempts to produce suitable methods for testing amino resins. Many have been devised by manufacturers, all of which involve low temperatures and working in vacuum. At present, IUPAC is preparing a method in which an attempt is being made to find a compromise between the contradictory requirements of low test temperature and short testing time in line with the requirements of practice.

Such methods have so far only been applied by raw materials manufacturers and not by the coatings industry because of the unfavourable compromise and apparently because of a lack of vacuum apparatus.

However, a basic change has now been brought about by the new European Community regulations on including the solvent content on labels. In the past, particularly with amino resins, habit led people to ignore the contradiction that there are considerable differences between the solids content and the nonvolatiles (for example, urea formaldehyde resins with 60 per cent nonvolatiles can contain up to 95 per cent solids). Many people were first reminded of this contradiction when it was seen that the sum of the nonvolatiles and the declared amount of solvent was much less than 100 per cent.

Although it may come as a surprise, there are still no test methods for ascertaining the "true solids content" of amino resins with any degree of certainty, if in fact, there is such a thing. The current situation is absurd: the method of determining the nonvolatiles used almost exclusively in practice is not only impermissible, but certainly yields the most inaccurate values for the solids content. There is no doubt about it: the method has been used to good effect in practice in the laboratory, it allows for production control at reasonable expense, it is clearly superior to the vacuum methods with regard to comparability and reproducibility, but it yields false data. As is shown by the measurements put forward for discussion below, the figures are also of no

use in estimating the solids content which results when the paints are used in the prescribed manner. What can be done? Should the entire complex consisting of the solids content, the elimination rate at a given time, temperature and reactivity be split into a number of individual tests, each of which covers one special facet without obtaining an overall result? Or would it not be better to search for a method of testing which yields a comprehensive result and perhaps leads to a more profound understanding of the events taking place during the reaction?

The nonvolatile content is determined by choosing at random, one of the possible time/temperature groupings, but the point thus selected has no practical implication. This point differs for every resin. Only in comparison tests with the same resin can it be expected that the same "condition" will always be tested. This is a reason for the great success of the method in production or acceptance control tests.

If two resins have the same nonvolatiles content, this can mean that they are similar. It can also mean that one resin has a higher solids content and a higher elimination rate, i.e. it is more reactive, whilst the other resin has a lower solids content and lower reactivity. If testing had been carried out with some other time/temperature co-ordinates, the two resins would have yielded different values. It is evident from this that single-point measurements in systems with several variables yield data that require extremely careful interpretation.

Just as a viscosity curve plotted over a large shear gradient range yields more detailed information on the rheological behaviour of a paint than a single measurement of the flow time, so is it apparent that the changes in weight should be measured from the beginning over a longer period of time in order to obtain information that allows better differentiation.

This practice was followed with a number of resins and paints and it was found that, in principle, it is possible to obtain adequate answers to the questions of solvent content, solids at the surface, and nonvolatiles from a series of measurements. In addition, information was obtained on other special properties.

Planning the experiments

The aim was to evaluate critically, the efficiency of the various methods used in practice to determine the solids (or solvent) content of a number of characteristic melamine formaldehyde resins. A new method which had been used in the authors' laboratories for some time was also to be compared. This method is based on DIN 53 216. A new feature is that weight decrease is permanently measured and plotted by means of an automatic balance and recorder in the form of a curve. By means of a logarithmic curve fit, the constants a and b , defining the shape of the curve, and the square of the correlation factor r^2 , can be obtained. The mathematical formula thus derived for the curve allows any desired intermediate values to be calculated with the exception of the discontinuity at the beginning. The authors therefore feel confident that in future it will be possible to obtain from a single curve all the information mentioned above that must be derived from the determination of the solids content.

Results and interpretation

Table 1 shows the original values obtained using the con-



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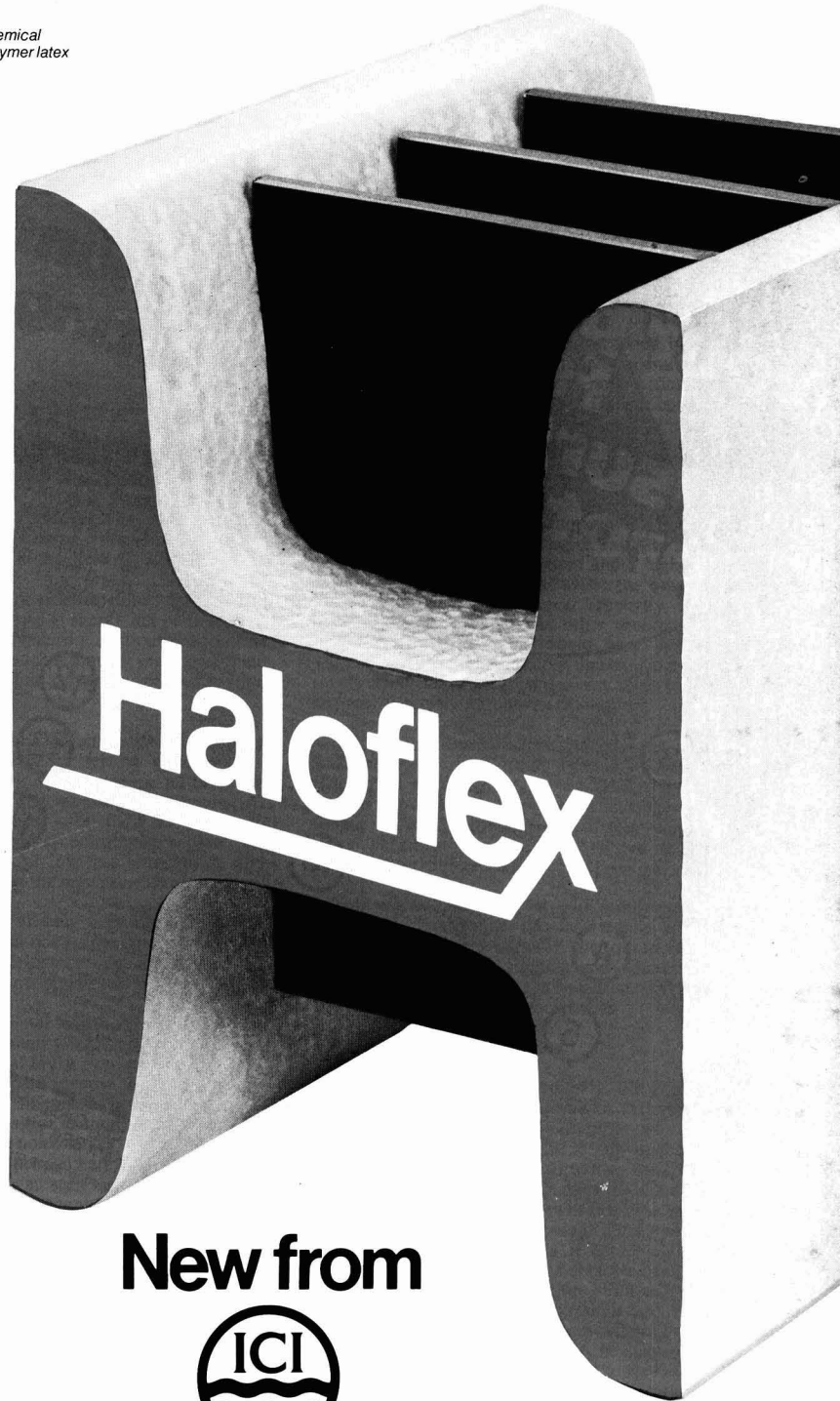
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Table 1
Comparison of nonvolatiles

Specimen	Method		
	2h/125°C (%)	4h/40°C vacuum (%)	GC (%)
Alkyd resins Melamine resins B = etherified with butanol M = etherified with methanol			
Synthetic fatty acid alkyd	60.4 60.5 60.6	61.2 61.4 61.2	63.2
DCO alkyd	74.1 74.2 74.6	78.2 78.4 78.0	76.5
Melamine resin B1	56.1 56.8 56.4	63.0 61.8 61.8	62.9
Melamine resin B2	61.3 60.8 60.7	68.4 68.4 69.4	68.6
Melamine resin B3	55.7 55.4 55.0	62.8 63.2 63.0	62.3
Melamine resin B4	54.8 54.5 54.6	60.4 61.8 62.4	56.6
Melamine resin B5	56.5 56.1 56.6	59.8 60.2 60.2	62.5
Melamine resin B6	78.5 78.2 78.8	88.8 88.6 88.6	81.7
Melamine resin M1	81.1 79.8 81.0	96.6 96.0 96.8	91.8
Melamine resin M2	72.4 71.9 72.2	87.4 87.4 87.4	83.3
Melamine resin M3	75.6 75.9 75.8	87.2 86.2 86.6	80.6

ventional method of determination of the solids contents of two alkyd resins and eight different typical melamine resins. The two alkyd resins were used by the authors for technical testing in white paints. Melamine resins B1 to B5 were etherified with butanol or isobutanol; M1 to M3 were etherified with methanol.

The first column shows values obtained in testing to DIN 53 216: 2g of material is distributed over the tin lid with the aid of 2ml of butanol and stoved in a circulated-air oven for 2 hours at 125°C. These figures are based on BASF's standard leaflet information.

The second column shows the solids content obtained in vacuum at low temperature. The resins were diluted with their own volume of butanol, and 2g of the mixture was weighed out. The dilute resin solution must completely cover the bottom of the lid. Drying was carried out to weight constancy in a vacuum cabinet at approximately

Table 2
Mean values, standard deviation from Table 1

Specimen	Method		
	2h/125°C (%)	4h/40°C vacuum (%)	GC (%)
Alkyd resins Melamine resins B = etherified with butanol M = etherified with methanol			
Synthetic fatty acid alkyd	\bar{x} 60.5 s 0.10	\bar{x} 61.3 s 0.12	63.2
DCO alkyd	\bar{x} 74.3 s 0.27	\bar{x} 78.2 s 0.20	76.5
Melamine resin B1	\bar{x} 56.4 s 0.35	\bar{x} 62.2 s 0.69	62.9
Melamine resin B2	\bar{x} 60.9 s 0.32	\bar{x} 68.7 s 0.58	68.6
Melamine resin B3	\bar{x} 55.4 s 0.35	\bar{x} 63.0 s 0.20	62.3
Melamine resin B4	\bar{x} 54.6 s 0.15	\bar{x} 61.5 s 11.03	56.6
Melamine resin B5	\bar{x} 56.4 s 0.27	\bar{x} 60.1 s 0.23	62.5
Melamine resin B6	\bar{x} 78.4 s 0.30	\bar{x} 88.7 s 0.12	81.7
Melamine resin M1	\bar{x} 80.6 s 0.72	\bar{x} 96.5 s 0.42	91.8
Melamine resin M2	\bar{x} 72.2 s 0.25	\bar{x} 87.4 s 0.00	83.3
Melamine resin M3	\bar{x} 75.8 s 0.15	\bar{x} 86.7 s 0.50	80.6
F	2844	2133	
Q	3166	5625	
Q ₁	3163	5619	
Q ₂	2.45	5.79	
F _{95%;22;10}	2.30	2.30	

30 mbar and 40°C over a period of 4 hours. The doubled figures are quoted.

The last column shows the solids content calculated on the percentage of solvent which was ascertained by gas chromatograph (GC). The melamine resins were injected straight into the block at 175°C. In spite of the high temperature, the GC values were usually so near to the vacuum method values that the differences were within the limits of experimental error.

Table 2 shows the statistical evaluation of the data in Table 1. As expected, with both alkyd resins all three values, i.e. the two mean values from the gravimetric measurement and the GC value, are comparatively close together. The situation with the melamine resins is very different. Here, there are considerable differences between the vacuum method values and the stoving method values, viz., 6-8 per cent for butanol-etherified resins and up to 16 per cent for methanol-etherified resins. For the "normal reactive" B1 to B3 and B5 resins the GC values correspond to the vacuum values; the B4 and B6 resins are highly reactive, and the GC values are between those

Table 3
Comparison of nonvolatiles

Specimen	Method		
	2h/125°C (%)	4h/40°C vacuum (%)	GC (%)
Melamine resin B1	64.1 63.6 63.7	73.6 72.6 62.8	64.5
Melamine resin B2	63.2 63.9 64.9	72.2 72.6 72.4	67.3
Melamine resin B3	62.6 62.2 62.0	74.0 72.2 72.2	67.2
Melamine resin B4	62.1 61.4 61.1	72.0 71.6 72.4	67.4
Melamine resin B5	62.3 62.0 62.8	73.2 73.0 73.8	66.9
Melamine resin B6	69.0 70.1 69.9	81.2 79.8 79.8	82.5
Melamine resin M1	74.0 73.4 72.9	80.1 82.6 83.4	74.0
Melamine resin M2	69.4 69.2 70.4	82.0 81.4 83.2	67.3
Melamine resin M3	70.1 69.1 69.6	79.0 79.2 78.8	72.8

of the stoving method and those of the vacuum method. It would appear that the high temperature of the injection block is of some importance. With the M1 to M3 resins the GC values are almost exactly in the middle between the vacuum and the stoving test values.

These figures clearly show the problems that arise in declaring the solvent content on an obligatory label. The values that the authors quote for labelling purposes are obtained using the vacuum method.

A study of the standard deviation gives the impression that a figure of around 0.3 to 0.4 is inherent in the method, whereas in individual cases deviations upwards (0.7) and downwards can arise. A mean standard deviation of approximately 0.35 must be taken as the basis. The variance analyses for each set of figures embraced 27-33 individual measurements, and the sum of squares in each case was 3-8, corresponding to the mean standard deviation quoted.

However, it should not be forgotten that a standard deviation of 0.35 implies that only around 65 per cent of all measurements lie within the range $x \pm 1s$ (s = standard deviation). Put in another way: if the same resin sample is repeatedly tested, only about 65 per cent of all the (average) findings are within the range $x \pm 1s$. Approximately 95 per cent of the values are found in the

Table 4
Mean values, standard deviation
from Table 3

Specimen	Method		
	2h/125°C (%)	4h/40°C vacuum (%)	GC (%)
Melamine resin B1	\bar{x} 63.8 s 0.27	\bar{x} 73.0 s 0.53	64.5
Melamine resin B2	\bar{x} 64.0 s 0.85	\bar{x} 72.4 s 0.20	67.3
Melamine resin B3	\bar{x} 62.3 s 0.31	\bar{x} 72.8 s 1.04	67.2
Melamine resin B4	\bar{x} 61.5 s 0.51	\bar{x} 72.0 s 0.40	67.4
Melamine resin B5	\bar{x} 62.4 s 0.40	\bar{x} 73.3 s 0.42	66.9
Melamine resin B6	\bar{x} 69.7 s 0.59	\bar{x} 80.3 s 0.81	82.5
Melamine resin M1	\bar{x} 73.4 s 0.55	\bar{x} 82.0 s 1.72	74.0
Melamine resin M2	\bar{x} 69.7 s 0.64	\bar{x} 82.2 s 0.92	67.3
Melamine resin M3	\bar{x} 69.6 s 0.50	\bar{x} 79.0 s 0.20	72.8
	F	207	43
	Q	459	489
	Q ₁	454	479
	Q ₂	4.93	9.52
	F _{95%;18;8}	2.51	2.51

range $x \pm 2s$, and only in the interval $x \pm 3s$ is a figure of approximately 99 per cent found, which represents a highly significant degree of confidence. In concrete terms, this means that the error to be expected in measurement alone, i.e. neglecting other factors is $\pm 3s \approx \pm 1$ to 1.2. This is also confirmed by the limits of experimental error stipulated in the above-mentioned standards. The standard deviation in the vacuum method is distinctly higher. This is only partly due to the fact that the values obtained by actual measurement must be doubled, and the reliability of the measurement itself is less. Daily laboratory routines rarely go to the extent of weighing out three specimens per resin type. However, it is not possible to obtain statistically valid data from two weighed samples only, and in this situation everything is then left to chance.

The extraordinarily high F values compared with the threshold values corresponding to the degrees of freedom df_1 and df_2 , indicate that, in principle, the two methods, vacuum and stoving, can be very readily compared although it must be noted that this applies only for the same type of oven. This became evident in other trials which are not dealt with in this paper.

When the 9 melamine resins were combined with the alkyd resin modified with synthetic fatty acids in the ratio 3:7 and the same tests were carried out, the figures shown in Table 3 were obtained. The summary in Table 4 shows

that the reactivity of the melamine resins increases in the presence of alkyd resin. This also follows from the fact that the GC values are distinctly nearer to the stoving values. The standard deviation is greater than that for the pure resins, a fact which was not expected.

The authors are not aware of any published literature which states that melamine resins appear to be more reactive in the presence of alkyds, i.e. that the amount eliminated is greater than that obtained by testing separately. Therefore, the expected loss in weight was calculated from the individual values measured, assuming that it was purely additive and that there were no interactions.

Figure 4 shows the theoretical values thus calculated as compared with the actual values found (2h, 125°C). With butylated resins, the increased reactivity resulted in a 5-7 per cent (by weight) increase in loss; with methylated resins the increase in loss was only around 3-5 per cent (by weight).

To avoid misunderstandings, it should be mentioned that, chemically, it is incorrect to equate reactivity with weight loss. Nevertheless, in practice it can be assumed that resins with a high elimination rate require a shorter stoving schedule.

Table 5 shows combinations of DCO alkyds with the melamine resins in the same arrangement. The summary in Table 6 confirms the results previously obtained, i.e. the vacuum value is the highest and the stoving value is the lowest (with one exception). There is a distinctly higher standard deviation with the vacuum method.

The comparison of the theoretical/actual loss on stoving (Figure 5) shows that there is also an increase in loss here, although this is slightly less than that with the synthetic fatty acid alkyd.

Figure 6 shows the different accelerating effects of the two alkyd resins.

New method, procedure

As in DIN 53 216 and in analogy to AFNOR T 30-11, approximately 2g of substance is weighed, diluted with butanol in order to give better flow on the test lid, and then placed on an automatic balance with accurate temperature adjustment to 125°C.

The balance is connected to a recorder (Figure 7). However, it is intended to extend this test method for routine production control by installing an automatic scale with a capstan head device for the samples. Evaluation can be simplified by feeding the digital data into an in-line computer. There is then no need to draw and evaluate a curve.

To evaluate the curves (Figure 8), the data obtained after 1, 2, 3 hours etc. are fed into a suitable pocket or desk computer, such as Hewlett-Packard HP 65 or HP 97 etc. With the appropriate program, the input data yield a curve equation of the form $y = a + b \cdot \ln x$ where y is the solids content and x is the time in hours. Numerical values are obtained for the constants a and b , which define the curve, and the square of the correlation factor r^2 , which is a measure for the curve fit and is unity in the ideal case, i.e., when all points of measurement lie exactly on the calculated curve. Thus, r^2 is not evidence of exact

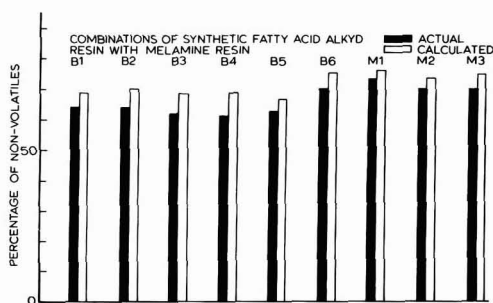


Figure 4. Comparison of actual and calculated values

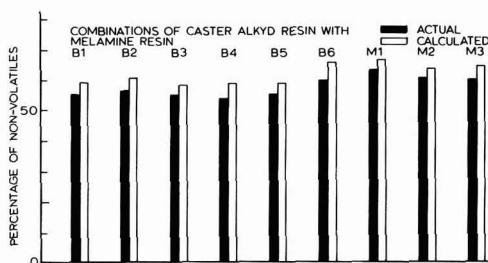


Figure 5. Comparison of actual and calculated values

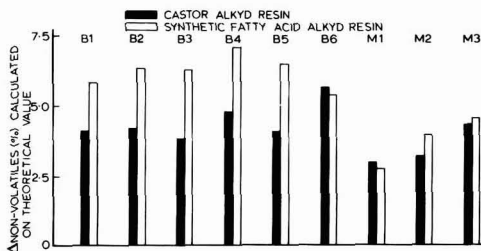


Figure 6. Difference between the theoretical and actual figures for stoving finishes, resin ratio 70:30 solid on solid, 2h/125°C

measurement, but is confirmation that the experimental and calculated curves agree. Also, it cannot be assumed that the events taking place during stoving follow a logarithmic reaction rate law. The information received is that the sum of all events taking place during stoving is a weight loss curve that can be described by the formula $y = a + b \cdot \ln x$ with a degree of certainty of practically, 1.

If the points obtained during the first 30 minutes are included in determining the fit, r^2 assumes a value which indicates that two independent curves have been added. One curve, which is only effective at the very beginning, describes the process of pure solvent evaporation. There is then a smooth transition to the logarithmic fit. If the exponential curve is extrapolated in the direction $x = 0$, i.e. $\ln x \rightarrow \infty$, points are obtained that should correspond to the course of events taking place when solvent-free solid resin is cured. Theoretically, if it tends completely to $x = 0$, the exact resin content of the solution should be obtained. Unfortunately, $\log 0$ does not exist, but if the curve is extrapolated to $x = 0.1$ or 0.01 , the values thus

Table 5
Comparison of nonvolatiles

Specimen	Method		
	2h/125°C (%)	4h/40°C vacuum (%)	GC (%)
Melamine resin B1	55.0	60.2	54.6
	55.4	60.1	
	55.0	60.4	
Melamine resin B2	56.1	63.8	55.4
	56.6	61.2	
	56.1	61.8	
Melamine resin B3	55.2	58.8	58.8
	54.9	60.6	
	55.5	58.8	
Melamine resin B4	54.0	58.6	54.4
	54.4	58.4	
	54.1	59.0	
Melamine resin B5	54.9	63.4	58.0
	55.0	63.2	
	55.4	64.2	
Melamine resin B6	59.8	66.0	67.7
	60.5	66.2	
	60.1	65.6	
Melamine resin M1	63.1	67.2	62.6
	63.5	67.0	
	63.8	65.6	
Melamine resin M2	61.0	66.0	62.0
	60.7	66.4	
	60.5	66.8	
Melamine resin M3	60.1	66.0	63.0
	60.6	66.6	
	61.2	67.0	

obtained for the solids content are valid for labelling purposes and agree very well with the vacuum and GC figures.

The figures calculated for 2h at 125°C should conform to the figures normally found within the limits of experimental error. A study of the figures shown in Table 7 shows that, at first sight, all values for r^2 are nearly unity. A repeat of individual curves with the same test material showed that the curves were identical and that slight apparent differences in the calculated values for a and b were due to the fact that the graphical evaluation did not allow accurate determination of significant figures after the decimal points. It was therefore decided not to pursue the reproducibility aspect in this study. The information obtained as compared to that with previous methods was more important.

To this end, figures for the solids content as determined at 2 hours/125°C were calculated from the numerical values for a and b and were compared with the corresponding figures determined by conventional means. There was excellent agreement. As the nonvolatiles content was expressed as a mean value, which can also lie within the range $\pm 2s$ (= 95 per cent) with 95 per cent probability, the deviations were due mainly to the nonvolatiles value. The method of curve fit for describing the product thus yielded a

Table 6
Mean values, standard deviation from Table 5

Specimen	Method		
	2h/125°C (%)	4h/40°C vacuum (%)	GC (%)
Melamine resin B1	\bar{x} 55.1	\bar{x} 60.2	54.6
	s 0.23	s 0.15	
Melamine resin B2	\bar{x} 56.3	\bar{x} 62.3	55.4
	s 0.29	s 1.36	
Melamine resin B3	\bar{x} 55.2	\bar{x} 59.4	58.8
	s 0.30	s 1.04	
Melamine resin B4	\bar{x} 54.2	\bar{x} 58.7	54.4
	s 0.21	s 0.31	
Melamine resin B5	\bar{x} 55.1	\bar{x} 63.6	58.0
	s 0.27	s 0.53	
Melamine resin B6	\bar{x} 60.1	\bar{x} 65.9	67.7
	s 0.35	s 0.31	
Melamine resin M1	\bar{x} 63.5	\bar{x} 66.6	62.6
	s 0.35	s 0.87	
Melamine resin M2	\bar{x} 60.7	\bar{x} 66.4	62.0
	s 0.25	s 0.40	
Melamine resin M3	\bar{x} 60.3	\bar{x} 66.5	63.0
	s 0.55	s 0.50	
F	252	15.58	
Q	202	281	
Q ₁	201	245	
Q ₂	1.79	35	
F _{95%;18,8}	2.51	2.51	

reliable value for the nonvolatiles obtained after 2 hours at 125°C.

As was already stated, extrapolation of the stoving curve towards very short periods of time is restricted by the discontinuity at $x=0$. There could also possibly be objections to the test procedure. For example, several minutes are required before the specimens reach the prescribed temperature. Data obtained during this time should not be included in the exponential curve. The predominating effect of the solvent in section a completely invalidates the curve there and care must therefore be taken to ensure that extrapolation is not carried out too near to zero.

Table 7 gives a comparison of the vacuum-solids content values with the calculated 0.1 and 0.01h values. In this case, agreement was not as good as with the values for 2h/125°C. However, comparatively high standard deviations were determined from the vacuum figures, and the question therefore arises as to whether the differences are due more to the vacuum method or to extrapolation. In view of the very high r^2 , the authors are inclined to think that the calculated values are more reliable.

They would, however, put forward the following two points.

1. In view of the obligation to include details of

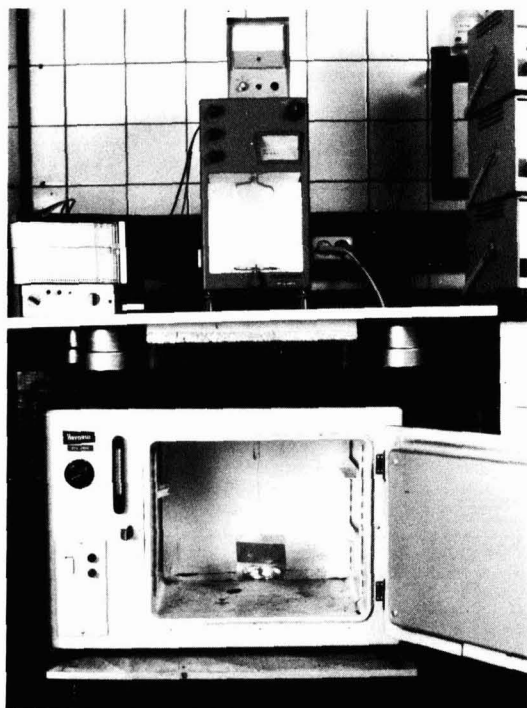


Figure 7. Apparatus for determining weight loss curve

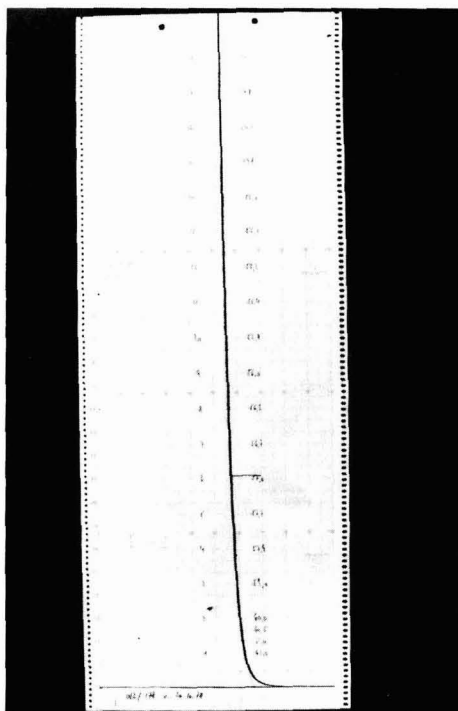


Figure 8. Example of a weight loss curve

solvents in labelling, the question on the "true" solids content of amino resins and the contribution made by elimination must be gone into in more detail, i.e. the potential methods of testing must be examined and compared with each other in regard to reproducibility and repeatability and (this must be clearly stated again) in regard to their usefulness in determining what is to be called the "true" solids content. The inverted commas have been inserted because the changing equilibria between many of the bonds concerned make it impossible to say which of the solution's ingredients are accounted

for by the resin. Amino resins do not have a "true" solids content.

2. If, however, amino resins do not have a true solids content and the real solids that remain depend upon the drying, curing or processing conditions, then all statements in regard to the solvent content of resins as supplied are only estimates. If this is the case, the question arises as to which estimated value is obtained if the weight loss curve is extrapolated as described above. Here also, it was found that extrapolation to 0.01h yielded values which

Table 7
Evaluation of logarithmic functions

Specimen	Logarithmic functions						Nonvolatiles	
	r^2	b	$x=2$	a $x=1$	$x=0.1$	$x=0.01$	2h/125°C (%)	4h/40°C vacuum (%)
Alkyd resins, Melamine resins, B = etherified with butanol M = etherified with methanol								
Synthetic fatty acid alkyd resin	0.9822	-1.05	78.1	78.8	81.2	83.6	74.3	78.2
DCO alkyd resin	0.9281	-0.49	61.8	62.1	63.2	64.4	60.5	61.3
Melamine resin B1	0.9919	-1.94	53.8	55.1	59.6	64.0	56.4	62.2
Melamine resin B2	0.9893	-2.51	57.7	59.5	65.2	71.0	60.9	68.7
Melamine resin B3	0.9779	-2.31	54.6	56.2	61.6	66.9	55.4	63.0
Melamine resin B4	0.9919	-2.10	55.3	56.8	61.6	66.5	54.7	61.5
Melamine resin B5	0.9935	-1.70	56.6	57.8	61.7	65.6	56.4	60.1
Melamine resin B6	0.9930	-3.22	78.1	80.4	87.8	95.2	78.5	88.7
Melamine resin M1	0.9958	-2.69	82.7	84.6	90.7	96.9	81.0	96.5
Melamine resin M2	0.9972	-2.79	73.4	75.4	81.8	88.2	72.5	87.2
Melamine resin M3	0.9985	-2.89	75.8	77.8	84.5	91.1	74.5	86.7

Table 8
Evaluation of logarithmic functions

Specimen Combination of DCO alkyd resin with	Logarithmic functions						Nonvolatiles	
	r^2	b	$x=2$	a $x=1$	$x=0.1$	$x=0.01$	2h/125°C (%)	4h/40°C vacuum (%)
Melamine resin B1	0.992	-1.10	55.5	56.3	58.8	61.4	55.2	60.6
Melamine resin B2	0.972	-1.27	55.9	56.7	59.6	62.5	56.4	62.3
Melamine resin B3	0.989	-1.12	55.5	56.3	58.9	61.4	55.2	59.4
Melamine resin B4	0.969	-1.06	53.9	54.6	57.0	59.4	54.2	58.8
Melamine resin B5	0.991	-1.23	55.3	56.2	59.0	61.8	55.2	63.6
Melamine resin B6	0.995	-1.48	59.6	60.6	64.9	68.3	60.2	65.9
Melamine resin M1	0.989	-0.99	62.2	62.9	65.2	67.5	63.5	66.6
Melamine resin M2	0.984	-0.96	60.7	61.4	63.6	65.8	60.8	66.4
Melamine resin M3	0.989	-0.90	61.9	62.5	64.6	66.0	60.3	66.5

Table 9
Evaluation of logarithmic functions

Specimen Combination of synthetic fatty acid resin with	Logarithmic functions						Nonvolatiles	
	r^2	b	$x=2$	a $x=1$	$x=0.1$	$x=0.01$	2h/125°C (%)	4h/40°C vacuum (%)
Melamine resin B1	0.994	-1.50	62.6	63.6	67.0	70.5	63.9	73.0
Melamine resin B2	0.990	-1.71	65.6	66.8	70.7	74.6	63.9	72.4
Melamine resin B3	0.991	-1.50	61.5	62.5	65.9	69.4	62.3	72.8
Melamine resin B4	0.994	-1.55	63.5	64.6	68.2	71.7	61.6	72.0
Melamine resin B5	0.995	-1.63	63.3	64.4	68.2	71.9	62.4	73.3
Melamine resin B6	0.997	-1.93	72.9	74.2	78.6	83.1	69.7	80.3
Melamine resin M1	0.993	-1.46	76.8	77.8	81.2	84.5	73.4	82.3
Melamine resin M2	0.986	-1.36	73.8	74.8	77.9	81.1	69.7	82.3
Melamine resin M3	0.981	-1.26	73.8	74.7	77.6	80.5	69.8	79.0

agreed very well with the vacuum data that were formerly used as reference for the solvent content.

In other words, both the vacuum data and the non-volatiles determined after 2 hours at 125°C can be produced from a single measurement. In addition, all intermediate values for stoving periods at the same temperature are obtainable.

Tables 8 and 9 show the data for combinations of alkyd and melamine resins. Here also, there was very good agreement between the 2h/125°C values and the calculated 2h values. The deviations lie within the error allowable with the standard methods. If, as shown in Figure 4 and Table 5, the anticipated non-volatiles for the combinations are calculated and these are compared against the values actually found, this will also confirm

that weight loss typically more than expected occurs with butanol- or methanol-etherified resins.

The great advantage of the above method of determining the weight loss by means of a curve becomes evident. With the aid of the equation, provided that the constants a and b for a paint are known, the actual amount of solids remaining after any desired stoving time at a prescribed temperature and layer thickness can be determined without additional measurements. This also yields a very real estimate for spent air calculations. Although the following was not the intention of the study, the figures shown above prove that if the amount of solids remaining is calculated from the 2h/125°C values for alkyd and melamine resins, it results in false data. The deviations are quite considerable.

[Received 14 August 1981]

Short Communication

Preparation of steel substrates before application of paint and related products

By A. N. McKelvie

Paint Research Association, Waldegrave Road, Teddington, Middlesex TW11 8LD, England

A seminar on the above topic was held by the International Standards Organisation (ISO) Sub-Committee TC35/SC12 on 30 January 1981, details of which appear in *ICorrTBull.*, 1981, March, 19 (2).

The Chairman, Mr K. C. Chandler, opening the seminar, defined the scope of his committee as standardisation in the field of preparation of steel surfaces previously coated or otherwise before application of paint or related products; he listed the programme of work as:

1. Establishment of a necessary and sufficient series of test methods for assessing surface qualities required for acceptable performance of coatings.
2. Establishment of acceptable standards for surface quality related to the types of coating when assessed by the test methods established.
3. Means of achieving the surface qualities established.

This work is being carried out by three working groups:

- Group 1. Surface profile led by UK (Convener Mr A. N. McKelvie, Paint Research Association).
- Group 2. Surface cleanliness led by Sweden (Convener Prof E. Mattsson, Swedish Corrosion Institute).
- Group 3. Surface preparation techniques led by Germany (Convener Mr A. W. H. Capell, Heinrich Mühlhan GmbH).

Following this seminar and meetings of working groups (WG) 1, 2 and 3 on 6 and 7 May 1981, a third meeting of ISO TC35/SC12 took place at the DIN headquarters in Berlin on 8 May 1981.

Agreement was reached on the basic format of the projected standard, ISO 7167, which would consist of four groups entitled:

- Group A - Grades of assessment before and after preparation of steel substrates (WG2 responsible).
- Group B - Test methods for assessment of surface cleanliness (WG2 responsible).
- Group C - Test methods for assessment of surface roughness (WG1 responsible).
- Group D - Techniques for surface preparation (WG3 responsible).

Each group will consist of a number of separate parts and it will be the responsibility of the appropriate working

group to suggest titles for each part. It was also agreed that attached to each part would be a short general introduction, which could be issued and sold separately, indicating the principles and format of ISO 7167, it would also list those parts already published and indicate other work under consideration.

For group A, working group 2 are still considering the best format for presenting rust grades and preparation grades. There has been considerable opposition to the retention of the present Swedish Pictorial Standards as the sole criteria for visual cleanliness, some consideration is being given to describing and assessing the amount of visible contaminants on a percentage basis. For example, it has been pointed out that the Swedish Pictorial Standards A Sa 2½, B Sa 2½ and C Sa 2½ are exactly the same picture, repeated; whereas by definition the contamination visible on A Sa 2½ should only be millscale and on C Sa 2½ should only be rust. This has led to a re-assessment of the whole position, some countries consider that a verbal description clause would be preferable and should take precedence over the Swedish photographic references. The Swedish photographs represent sand-blasted steel (use of sand is not allowed in some countries), other types of abrasives give different visual appearances. It is hoped that working group 2 will have sufficient additional data available at its next meeting to make a unanimous recommendation to TC35/SC12 on these points.

Group B is also the responsibility of working group 2. Since the overall task is very large it has been agreed to delegate some of the work to small task groups. One overall problem is devising a practical, simple, surface sampling technique which can be used on-site and the UK has agreed to study this through its national committee (PVC/21/-/2). Another problem is deciding on acceptable and practical levels of the various contaminants. While each test method will have limiting values, the significance of these can only be determined when working group 3 applies the suggested tests in their study of the various surface preparation techniques. Despite these difficulties, a considerable amount of test method work is in hand and by the next meeting of SC12 it is hoped that draft proposals will be available for assessment of soluble iron contaminants, detection of rust and millscale, detection of surface moisture, detection of residual dust and detection of oil, grease and wax. The use of these methods by working group 3 should characterise the levels of contaminants remaining after applying the various cleaning techniques to steel representative of rust grades A, B, C and D and to previously coated steel requiring cleaning prior to maintenance.

For group C, significant progress has been made on the assessment of surface roughness after blast cleaning,

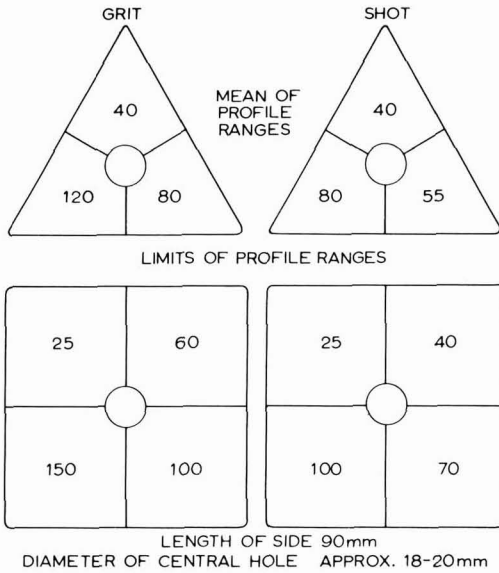


Figure 1. Proposed 3 and 4 segment comparators
(Profiles quoted as R_{max} or H_{max} in μm)

Using the 3 segment comparators the fine, medium and coarse grades of roughness correspond to the three mean profile ranges, each characterised by an R_{max} (H_{max}) value. Using the 4 segment comparators the fine, medium and coarse grades of roughness are intermediate between the four profile ranges, each characterised by an R_{max} (H_{max}) value.

which produces a random profile. At the third meeting of working group 1, the specifying of three levels of roughness (fine, medium and coarse) assessed by visual or tactile comparison was considered adequate and existing comparators were studied. At the fourth meeting, a three segment (triangular) comparator was proposed together with three levels of roughness based on ISO 2632/11 "Roughness Comparison Specimens", a document issued by ISO Technical Committee TC/57. At the fifth meeting, a four segment comparator was proposed which would indicate the limits of the three levels of roughness and perhaps make the assessment easier. The original levels in ISO 2632/11 were also queried and new levels agreed. It was also established that no suitable stylus measuring instrument was widely available at the moment for checking the accuracy of comparators over the range of random profiles required; it was agreed that a focusing microscope method with clearly defined limits for field of view and magnification, and based on the difference between the

highest peak and the lowest valley in the field of view, shall be the method adopted for standardising comparators and for referee purposes. In order to make a field study of the relative usefulness of 3 segment and 4 segment comparators, a quantity of both the grit and shot type are being made and will be assessed by various member body countries of TC35/SC12. This assessment will also establish the suitability of the levels of roughness chosen for both types. These test comparators will conform to the shapes, sizes and profile levels shown in Figure 1. It is hoped that as a result of the work envisaged, a definite comparator design and levels of roughness can be agreed, and that draft proposals for a method of assessing surface roughness of a blast cleaned surface using a comparator, together with a definitive method (focussing microscope method) for standardising comparators, will be submitted at the next meeting of TC35/SC12.

Group D, techniques for surface preparation, is the responsibility of working group 3 which held its third meeting in Berlin on 7 May 1981. There is still some debate as to the details required in describing techniques and their applicability. It seems appropriate not only to describe each technique in general terms but also to establish the levels of cleanliness likely to be attained when cleaning each of the four rust grades of steel (A, B, C and D). Study documents have already been prepared, or are in the course of being prepared, covering the various abrasive blast cleaning techniques, flame cleaning and manual, and mechanical derusting methods; these will be circulated for comment, together with data (as they become available) on the levels of cleanliness that can be obtained when using the test methods from group B. It is hoped that some progress can be reported at the next meeting of TC35/SC12. Working group 3 is also considering terms and definitions relating to surface preparation and will liaise with TC35/SC1 "Terminology". The intention is that specific terms and their definitions will be incorporated in the particular parts of ISO 7167 and that the more general terms will be considered for ISO 4618 "Paints and Varnishes Terminology".

In their deliberations at Berlin, TC35/SC12 discussed the proposal, accepted by TC35, that consideration should be given to characterising paint systems for the protection of steel structures from corrosion. It was agreed that SC12 would not undertake such work at present but suggested, should work start outside SC12, it would be appropriate to keep them informed.

The next meetings of TC35/SC12 and its working groups are planned for the period 18-21 March 1982 in Houston, Texas and will just precede the Annual NACE Conference Corrosion 82 (22-26 March 1982).

[Received 25 June 1981]

Further information on any of the publications reviewed may be obtained by circling the appropriate *Reader Enquiry Service* number on the form at the back of the *Journal*. Enquiries will be forwarded to the publisher.

Printing on textiles by direct and indirect techniques

Editor R. W. Lee

Noyes Data Corporation, New Jersey, USA

Chemical Technology Series No. 189

3 August 1981

pp. 410 + 3 indexes, price \$48.00

This book details 264 US patents with direct relevance to the title. The processes in the first four chapters are grouped according to textile substrates, but it should be stressed that a number of dye compounds are applicable to more than one substrate, especially where these can be modified to give improved affinity. It is clear, therefore, that it has been necessary to associate the compounds with the substrates to which they are most compatible. It is of importance to point out that the majority of the processes in these chapters cover both dyeing and printing, since the basic difference between the two lies in formulating the compounds for the two modes of application.

The next two chapters are concerned with the formulation of dyes into pastes, and printing processes including fixation, treatment and techniques to facilitate the printing process.

Subjects covered in the transfer printing section include dyes suitable for transfer, formulation of dyes into printing inks, the temporary transfer media and treatment processes associated with the transfer printing operation.

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

Midlands

Living with lead legislation

The second technical lecture of the Midlands Section was held on 15 October 1981 at the Warwickshire County Cricket Ground, Edgbaston, Birmingham.

Members and guests heard Mr A. C. D. Cowley of ICI PLC give a talk entitled "Living with lead legislation". The speaker opened his talk by saying that the most recent legislation to be introduced was the "Control of lead at work regulations of 1980". These regulations do not introduce any new controls but tidy up the jungle of legislation that has arisen since 1903. Many of the older regulations dealing with specific industries were outdated and have been revoked. The new regulations have been designed to cover all lead workers in Great Britain. Cases of lead poisoning in the UK have dropped from 123 in

reviews

The last chapter presents patents of equipment for transfer printing.

Reader Enquiry Service No. 21

D. S. Newton

Techniques in visible and ultraviolet spectrometry. Volume 1. Standards in absorption spectrometry

UV Spectrometry Group, edited by C. Burgess and A. Knowles

Chapman & Hall Ltd, London and New York, 1981

pp. xii + 142

The UV Spectrometry Group (originally known as the Photoelectric Spectrometry Group) was formed in Cambridge in 1948 with the aim of standardising methods for UV spectrometry. Chapters based on working party and workshop reports, together with others written by specialists, constitute this monograph. It covers the design of the cell, liquid and solid absorbance standards, calibration and standardisation, and other aspects concerned with instruments and procedure; it is essentially a working manual for the chemist practising UV or visible spectrometry.

Reader Enquiry Service No. 22

L. A. O'Neill

occa meetings

1971 to only 12 in 1977 and most of these occurred in the demolition or scrap metal recovery industries.

Mr Cowley went on to give results of a survey carried out to answer the question "To what extent will these regulations affect the paint industry?" The survey was carried out at four companies and covered different processes and procedures. The tests carried out covered a warehouse, a fork lift truck driver, an operative charging both mixers and mills and a paint sprayer. The lead-in-air determinations were observed both as personal tests and static tests. In each case the lead-in-air level decreased rapidly as soon as the operation was completed.

Finally, Mr Cowley briefly outlined the regulations that

occa meetings

must be complied with, depending on which classification the lead-in-air level falls in. There are three classifications, namely:

1. non-significant exposure – less than 0.075 mg/m³ as lead
2. significant exposure – 0.075-0.15 mg/m³ as lead
3. exposure above lead-in-air standard – greater than 0.15 mg/m³ as lead

During the question time that followed the talk, Mr Cowley's colleague, Mr F. Chapman, gave details of the equipment used for the lead-in-air sampling. The meeting finally closed with a vote of thanks proposed by Mr L. P. Goodale.

B. E. Myatt

Trent Valley

Computers in colour prediction and formulation control

The first technical meeting of the Trent Valley Branch at the new venue, The Sutton Centre, Sutton in Ashfield, Notts, was held on 8 October 1981, when the speaker was Dr J. P. Ferguson whose paper was entitled "Computers in colour prediction and formulation control".

Dr Ferguson explained that an instrumental colour-matching system comprised three parts, a spectrophotometer, a computer and programs. The spectrophotometer incorporated a diffraction grating and filters with which sixteen points are measured along the spectrum. The computer translates these into numbers and, by means of the software, manipulates the numbers to produce the results.

The discoveries made in colour physics, the adoption of analogue computers in the textile industry to predict colour matches and the major developments in computers in the last decade, coupled with improvements in spectrophotometers, have led to much smaller units for use in instrumental colour matching systems.

To predict a colour match it is necessary to have a linear relationship between reflectance and pigment/dye concentration; the mathematics are based on the work of Kubelka and Munk.

Instrumental colour matching systems are widely used by major UK paint manufacturers, both as production aids and in laboratory formulation. They can be used for matching, mix correction and colour difference measurements in all types of paints, but due to the single-angle measurement are not entirely satisfactory for metallics.

In the future it is envisaged that systems may be linked to automatic dispensing and weighing equipment to provide fully automatic paint factories.

A lively discussion period followed, the points raised being the importance of good samples, the versatility of these systems, further possible developments and size reductions, possible improvements for the accurate measurement of metallics and the use of instrumental colour matching systems in non-paint industries.

The vote of thanks was proposed by J. P. Bourne and heartily endorsed by all members present.

J. C. Ellis

Ontario

Quality control for raw materials in a printing ink plant

The first meeting of the season was chaired by Mr Alan Gray, who welcomed members and friends and introduced the new Section Committee.

Mr Shirish Patel introduced the evening's speaker, Mr Robert Edmonds, who is the technical co-ordinator of Sinclair & Valentine Company of Canada Ltd. Mr Edmonds has been in continuous service with the firm for some 34 years and has earned the abiding respect of his many past and present colleagues in the ink industry.

Mr Edmonds presented his view on "The establishment of a quality control programme for raw materials in a printing ink plant". The aim throughout was to provoke thought as to the desirability and methods of such a programme. He began by reminding the audience that coatings (including inks) are designed at a great cost in time and effort to meet specific end-uses, and that they are invariably quality-checked as finished products to ensure that they will perform as intended. Since every ingredient in the formula is meant to contribute some specific property or properties, the inadvertent use of any significantly off-grade raw material is likely to ruin the entire batch. This costly consequence is the main rationale for routine testing of incoming materials.

While suppliers normally provide materials which meet their published specifications, the coatings manufacturer must also ensure that these materials will produce a finished product which will meet his criteria, batch after batch. This may depend on properties which are outside the supplier's normal quality control tests; or in some cases the acceptable range from an end-user's point of view may be much narrower than the specified range. Thus, even materials which are nominally within specifications may not give the anticipated results. In these cases, it is wholly incumbent on the user to confirm suitability of the received material before releasing it to production.

The various types of raw material were considered separately. For each type, basic tests were suggested which would determine suitability. These were of two kinds: tests for chemical/physical properties (such as melting point, colour and viscosity), and tests for particular end-use properties (such as dispersibility, bleed resistance and adhesion); the latter relating directly to the specific application intended. Special attention was paid to pigments because they were felt to be the most critical ingredients in most inks, certainly for appearance and often for performance as well.

In closing, it was pointed out that most of the raw material properties which are important to the ink-maker can be determined by relatively simple testing, and that the

prevention of production problems and spoiled batches makes the effort fully worthwhile.

During the subsequent discussion, it was agreed that the supplier should be consulted when test methods are being established; the ink manufacturer should understand what degree of consistency can be expected, given practical considerations, while the supplier should understand what properties are significant for the application intended.

Additional discussion dealt with the interpretation of

Further information on any items mentioned below may be obtained by circling the appropriate Reader Enquiry Service number on the form at the back of the *Journal*. Enquiries will be forwarded to the organisation concerned.

Receivership of LB Holliday & Co. Ltd

A receiver has been appointed to L. B. Holliday & Co. Ltd, the Huddersfield manufacturer of dyestuffs and intermediates. Holliday employs 450 people and is one of the three main manufacturers of dyestuffs in the United Kingdom.

In 1980, the turnover of the company was more than £14m, of which 70 per cent was for export. The company occupies a 33 acre site in Huddersfield and over the last 12 years has invested £10m in new and modernised plant. L. B. Holliday & Co. Ltd received the Queen's Award for Exports in 1978.

The receiver, P. R. Copp of Stoy Hayward & Partners, is seeking a buyer for the business as a going concern.

Reader Enquiry Service No. 31

£2.3m scheme for ICI dyes intermediates

ICI is to spend a total of £2.3 million over the next 18 months in modernising dyes intermediates manufacturing operations by the Organics Division at Huddersfield, Yorkshire.

The project is the culmination of changes in the pattern of manufacture, supply and demand of dyes and their intermediates which have developed over recent years. The investment is intended to safeguard the manufacture by ICI of a key range of intermediates through the 1980s and beyond.

It involves integrating into three units, the manufacture of intermediates currently carried out in five plants. The aim is to protect the competitiveness of manufacture by more efficient plant utilisation, reducing costs through more effective use of modern assets and savings in maintenance on older and under-occupied plants.

The 40 products to be re-sited, many of them ICI specialities, are used mainly in the manufacture of the major ICI ranges of Dispersol dyes for polyester and Procion fibre-reactive dyes for cellulose. More than 85 per cent of these dyes are exported.

Reader Enquiry Service No. 32

Camrex secures 2,000,000 sq ft

Following its continued success over the past ten years with the four coat Camkote N Polyurethane system, Camrex was pleased to receive, earlier this year, a contract for four 38,650 dead weight tons product tankers being built for Nedlloyd bulk, a division of Koninklijke Nedlloyd Groep NV, Rotterdam.

The total surface area to be coated in these four product tankers is approximately 2,000,000 sq ft. The vessels, which are due for delivery in 1982/83, are being built at Van Der Giessen, De Noord.

They will be used for the transportation of a wide range of chemicals, solvents and oils, all of which can be carried successfully in cargo tanks protected by Camrex Camkote N.

Reader Enquiry Service No. 33

"Hospital" line for Range Rovers

Land Rover Ltd has improved the painting conditions and increased the efficiency of energy usage by the installation of a Haden Drysys paint-repair line at Solihull.

Range Rovers are assembled from a large number of preprepared components, unlike most vehicles whose entire body shell is painted.

In any paint plant it is inevitable that some paint damage occurs to the vehicle components during assembly. It is to rectify this, plus blemishes from main paint line malfunctions and accidental scratches, that a paint repair line was specially installed. The quality of the finish on Range Rovers, including the new four door version, is assured by the so-called "hospital" line.

The Haden Drysys line, installed as part of BL's £85 million modernisation programme for Land Rover, replaces a unit on the same site in which both Range Rover and Land Rover repair work was carried out.

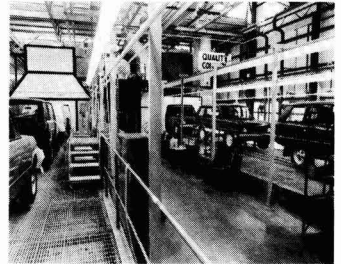
The principle feature of the line is a Haden Drysys Hydrospro paint booth, whose efficiency of operation and con-

test results and the training of quality control personnel, the necessity of re-checking negative results, the wisdom of agreeing (with supplier and/or within the industry) on certain standard test methods, the involvement of the purchasing department in the raw material quality control system and the general consistency of raw materials quality over the past several decades.

In response to the motion by Mr Gray, Mr Edmonds was given a most enthusiastic vote of thanks.

J. Ambury

news



The Range Rover "Hospital" line

trolled airflow mean that overspray fog in the booth is eliminated.

Consequently, painters need not wear aspiration equipment or bulky protective clothing. The booth walls may be made of glass, further improving working conditions. Different colours may be applied to cars on the line separated by only a few feet without the fear of cross-colour contamination.

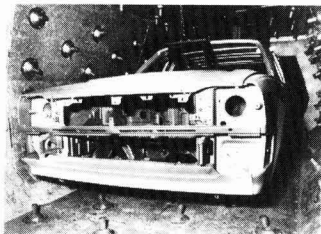
Reader Enquiry Service No. 34

High-solids paint for gas appliances

Valor Newhome Ltd is pioneering the use of a new paint system which is expected to cut costs, reduce pollution and conserve energy, with the potential to save millions of litres of oil-based solvents annually. Valor Newhome is claimed to be Europe's first gas-appliance manufacturer to introduce high-solids paint, adopting Intersol acrylic, a material developed by International Paint after three years research.

Mr Jack Tiley, Valor Newhome's manufacturing director, states "quality has increased and material costs have fallen. In addition, the faster process enables 16 hours of painting production to be equalled in eight, bringing manpower savings of about 30 per cent".

Reader Enquiry Service No. 35



A phosphated Triumph Acclaim being dried with air blown from the nozzles

Triumph Acclaim pretreatment

Launched at the beginning of October, BL's Triumph Acclaim four door family saloon is being built to meet the Canadian code for corrosion resistance, generally regarded as the toughest in the world.

The pretreatment relies on plant designed, manufactured and installed by Durr Ltd of Warwick. It ranges from an initial preclean to remove heavy deposits of oil and grease, through a seven stage spray-dip-spray zinc phosphating process to cathodic electro-dip and underseal application.

Four Pyrene processes are used in pretreatment, starting with high pressure spray cleaning of the body shell using a knock-off cleaner. On exit from the pre-clean section, the body enters the first of its seven stage pretreatment operations passing into a spray-dip-spray alkaline cleaner. The spray dip process involves passing the body through cleaning sprays at the beginning of the stage, followed by immersion in a tank of the cleaning solution. The body travels horizontally through the solution, emerges the other end of the tank and then passes through another bank of cleaning sprays.

After cleaning, any residual chemical cleaner must be removed prior to zinc phosphating and this is achieved with two rinse stages, a spray-dip cold water rinse – following the same spray-immersion-spray pattern as the cleaner – and a straight-forward cold water spray rinse.

The zinc phosphate conversion coating is applied at the next stage, again by spray-dip-spray, using Pyrene Bonderite 399 low temperature zinc phosphate. This is followed by a further spray-dip-spray rinse. The body then passes to the chromate stage where it is rinsed with a Pyrene Parcolene 86 trivalent chromate rinse. This has been found to be highly beneficial in further improving the adhe-

sion performance of paint finishes applied by the cathodic electropaint method. The final pretreatment stage is a deionised water spray rinse which removes residual Parcolene.

The car body is then dried and cooled before it passes to the cathodic electropainting stage, which is the ED3002 process supplied by International Paints. The application, by airless spray, of underseal and chip-resistant sill coat is carried out by robots from Hall Automation of Watford. Finally surfacing and finishing take place on the floor above.

Reader Enquiry Service No. 36

Advanced formaldehyde plant on stream

A new plant for the manufacture of formaldehyde has now been commissioned at the manufacturing and distribution centre of the Chemical Division of Blagden & Noakes (Holdings) Ltd.

Claimed to be the most modern of its type in Europe, the plant has an initial capacity of 30,000 tonnes per annum and utilises the low energy Formax process. The high strength formaldehyde solution produced can be diluted with water or methanol to suit customer requirements.

The process is totally enclosed and the latest waste gas equipment ensures virtually nil emission of gases into the environment.

Reader Enquiry Service No. 37

Pigments agency

H. Haeffner & Co. has been appointed sales agent for Forthfast Organic Pigments, part of Croda Colours' wide range of pigments for the paint industry and allied trades.

Haeffner is well known in the paint industry and has had a long involvement selling inorganic pigments, extenders and fillers on behalf of a number of manufacturers.

Reader Enquiry Service No. 38

Mistron supplies

Cyprus Industrial Minerals Corporation (CIMC) is now supplying its range of speciality mistron products directly to the paint industry.

This action has the full approval of Compounding Ingredients Ltd (CIL), the CIMC distributor in the UK. According to Peter Raymond, CIL sales director, "this move is designed to ensure the best possible response to the special problems of the paint industry". He stressed that CIL will continue to distribute CIMC products to all other industries.

Reader Enquiry Service No. 39

Heat recovery to save dyestuffs firm £100,000 pa

A £250,000 scheme for the integration of site services with heat recovery will save a Manchester dyestuffs manufacturer £100,000 a year, it is claimed.

The scheme has been monitored by National Industrial Fuel Efficiency Services Ltd as part of the Government's Energy Conservation Demonstration Projects Scheme. Applied nationally, the engineering principles which are demonstrated in this project, particularly by users of inert gas or thermal fluid heaters, could save 80,000 tonnes of coal-equivalent a year.

Clayton Aniline has a site requirement for steam, inert gas and thermal fluid for high temperature process heating and has devised an integrated system which generates these products. Heat recovery is practised at all stages of the process and steam is generated as a means of balancing the heat flows within the plant.

Reader Enquiry Service No. 40

Portuguese resin agency

Charles Tennant & Co. Ltd has been appointed agent for the UK and Eire by the Portuguese resin company Resinas Naturais Lda. A brochure has been published on the available products, which are based on rosin.

Reader Enquiry Service No. 41

Water-borne coating system wins award

A coating system produced by Deft Inc. of California, was selected for an I-R 100 Award by a panel of renowned scientists and engineers. The award, which was presented in Chicago on 24 September, recognises innovators and organisations for work in outstanding practical technical developments.

Deft's revolutionary system produces primers and topcoats that meet the United States Clean Air Act requirements and are environmentally acceptable to every state. The system meets current and all foreseeable regulations designed to reduce volatile organic compound (VOC) emissions.

This is claimed to be the only water-borne coating system able to pass the requirements of the Military's MIL-P-23377D primer specifications. In addition, air force and navy industrial hygienists have established that during application there is a ten-fold reduction in solvents, chromates and particulate matter surrounding the painting operation, making application safer and more pleasant.

To date, three commercial applications of the water-borne primers have been developed. Firstly, high-performance aircraft primers, secondly, a corrosion-resistant military primer for steel vehicles and ground equipment, and thirdly, transportation primers for heavy-duty trucks.

Reader Enquiry Service No. 42



The official opening of CVP's head office. The Mayor of Bromley, Councillor M. B. Kenward, is seen having unveiled the Machen stone. On the Mayor's left is Sir Richard Meyjes, chairman of Coates Brothers & Co. Ltd and CVP, Graham North, CVP's managing director and Ivor Stanbrook, MP for Orpington. The Machen stone was a gift from Machen quarry; Machen is the village where the largest CVP manufacturing plant is situated

CVP open research and administrative centre

The official opening of Cray Valley Product's research and administrative centre took place on 20 October 1981. The building is situated in Farnborough, Kent and houses some of the most advanced equipment in the world.

Cray Valley Products Ltd was formed in 1938 to supply resins to the parent company Coates Brothers and subsidiaries, at that time it was registered as a company in its own right. Today, Cray Valley Products is not only the biggest subsidiary of the group, it is also the largest manufacturer of synthetic resins within the United Kingdom and one of the leading manufacturers worldwide. CVP has agents or representatives in more than fifty countries, resulting in licensing agreements in many territories.

In addition to Cray Valley Product's head office situated in Farnborough (England), the three main manufacturing plants are located at Machen (South Wales), Stallingborough (South Humberside) and Isipingo (South Africa).

Reader Enquiry Service No. 43

new products

New coil and sheeting coating

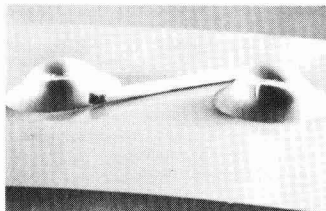
The range of coatings for coil and sheeting application supplied by Drynamels has been extended with S70. It contains a high performance modified polyester and is formulated to give excellent flexibility.

Tests during development work have shown that the film produced from S70

remains intact, even when the metal substrate has been deformed to breaking point.

S70 is said to be ideal for coil-coating purposes, especially for deep draw applications. An example of a use to which it is put, is for the manufacture of automotive oil filter cans. These are pressed from a flat coil of precoated metal.

Reader Enquiry Service No. 44



The S70 polyester film remains intact past the metal fracture point

New Tioxide pigment

Tioxide has announced a new pigment produced at the chloride plant in Greatham.

Tioxide R-TC50 is designed primarily for use in matt emulsion paints, it may also be used to advantage in other types of decorative products.

The new pigment is available in bulk tankers and in 800 kilo Tiotainer bags as well as in standard 25 kilo paper sacks.

Reader Enquiry Service No. 45

Water toxicity testing

Beckman's Microtox is claimed to be the first commercially available instrument

news

for monitoring acute water toxicity in coloured aqueous samples. The Microtox Model 2055 Toxicity Analyzer System is also claimed to be the first instrument to utilise luminescent bacteria for determining water toxicity. The assay is simple, reliable and reproducible requiring only a few millilitres of sample. Results are obtained in minutes and correlate with other bioassay techniques requiring days.



The Beckman Microtox

Microtox can be used in industrial, municipal and environmental laboratories. It has immediate application in the bio-monitoring of waste effluents from pulp and paper mills, oil refineries, chemical processing plants, plating operations and marine paint manufacturers. In upstream monitoring applications, it helps prevent toxic wastes from contaminating waste treatment systems and, ultimately, from spilling untreated toxicants into rivers, streams and lakes.

Instrument operation is based on measuring the influence of toxicants on the light output of a special strain of luminescent bacteria. Like other living cells, these bacteria consist of diverse enzyme systems. Unlike most other organisms, a natural metabolic product is light. Disruption of the metabolic process results in an alteration of light emission. By using a specially designed photometer to sense changes in light output, it is possible to determine the presence and relative concentration of toxic materials.

Reader Enquiry Service No. 46

news



Dust collector from Dustcheck

Dust collector

A range of compact dust collectors are being marketed in the UK by Dustcheck Ltd after having been used throughout mainland Europe for many years. They are currently being evaluated for UK factory inspectorate approval.

The heart of these collectors is a washable single element pleated cartridge filter, manufactured from a synthetic resin based micronic corrugated paper. These are specially treated to create an optimum filtration medium with high dust retention and low flow resistance. They are claimed to be rot proof and highly compatible with most industrial and chemical filtered products as well as cleaning solvents.

A combination of filter plates can be fitted to accommodate 9, 13, 19, 25 or 37 standard diameter filter cartridges, with five nominal lengths to achieve delivery throughputs from 900 to 5000 m³/hour. Two grades of filter media can also be provided with filtration down to 2 and 0.45 microns respectively. For demanding applications, a scavenging or after-filter can be fitted for operation at negative pressures. Two methods of *in situ* filter cleaning can be offered: type RF has a motor operated mechanical shaker system which dispenses with mechanical linkages and offers what is possibly a unique feature for this type of product. A type SF introduces a reversed flow air jet and in both cases the residue filter cake is collected in a low level dust container with optional bagging facilities, rotary feeders or product separators.

Reader Enquiry Service No. 47

Automated colourant dispensing

Applied Color Systems Inc. (ACS) has made available new precision metering pumps for low volume applications of colourant, they are intended for use with ACS's automated colourant Production Dispensing System. The pumps are available in air and hydraulic models. Air operated pumps are designed for volumes of 0.92 oz. per cycle, hydraulic pumps are designed for 1.3 oz. per cycle.

The new pumps enable the Production Dispensing System to dispense colourants in small quantities in a variety of container sizes. In operation the system is entirely computer controlled and usually linked to an ACS colour system which enters colour formulas in to the computer memory. A control terminal in the production area communicates with the colour system to assure that colourants are precisely dispensed according to computer determined formulations.

Reader Enquiry Service No. 48

Big bag discharger

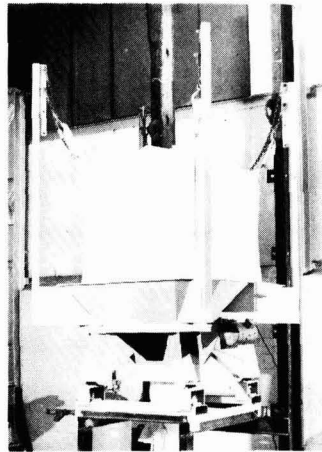
Flomat Ltd has just introduced a machine for the discharging of big bags.

Called the Flomat Big Bag Discharger, the machine quickly and efficiently empties bulk materials from all sizes of IBCs up to 2 cubic metres, into either secondary storage bins or directly into processes.

The Big Bag Discharger is available in three models, the choice of machine being determined by the flow characteristics of the material involved.

Although simple in design, the discharger is extremely robust. It consists of a conical shaped hopper into which the big bag is lowered while being suspended by bag-loops, a liner tensioning mechanism is then applied to establish the correct discharge mode.

Reader Enquiry Service No. 49



The Flowmat Big Bag Discharger

Volstatic launch modular powder booths

Volstatic Coatings Ltd has developed a new modular spray booth for manual and automatic powder coating. It has been designed for fast installation and easy cleaning.

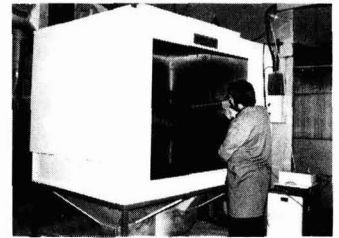
The new booth is available in modules of 1 metre, with the size and shape of the opening determined by the nature of individual applications.

All booths can be installed for on-line or off-line working and they are fully compatible with the range of Volstatic gun handling equipment for automated powder coating, notably the new MK III waggler.

Vestibule work silhouettes are included for conveyorised, on-line installations.

The interior design of the new booth has been kept very simple so that clean-outs and colour changes can be carried out quickly.

Reader Enquiry Service No. 50



A two metre spray booth

New resin

BIP Chemicals Division has announced a new high-amino content urea formaldehyde (UF) resin. Amino resins, particularly UF resins, are claimed to be the least expensive surface coatings resins currently available.

The new resin, Beetle L6104, is an etherified urea formaldehyde which can be combined with a second component, such as caprolactone, to create cold-cure wood finishes and other surface coatings with a medium-to-high solids content.

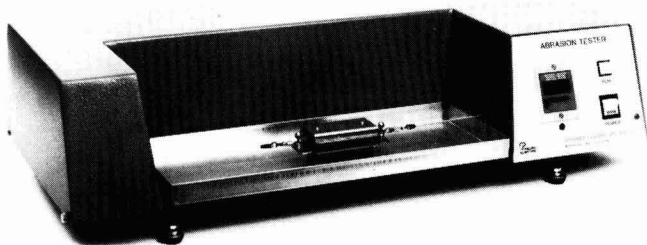
Minimal use is made of petroleum based chemicals during the production of Beetle L6104, making the resin extremely cost-effective.

Reader Enquiry Service No. 51

Air purifier

Astec Environmental Systems Ltd has just introduced a new model to its range of air purifiers. Astecair 150, a wall mounted unit, does not merely mask unpleasant smells and fumes, it removes particulate contamination, physically and chemically destroying odours and harmful fumes through a unique filtration system.

Reader Enquiry Service No. 52



The improved Gardner Abrasion Tester

Abrasion tester

Westlairs Ltd, representative of Pacific Scientific Company (Gardner Laboratory Division) for physical test equipment, has made available an improved Gardner Abrasion Tester.

Based on the well-known Gardner Washability Machine, the Abrasion Tester provides a reciprocal movement of a brush, sponge or other abrasive device in a straight line across the test surface.

Improvements include constant speed at 37 cycles per minute across the entire travel of 10 inches, increase in load capacity, enclosure of components, redesigned electronic circuitry and a "count down" counter with automatic shut-off to preset the number of cycles required.

Reader Enquiry Service No. 53

New one-pack pigmented etching primer

Sonneborn & Rieck Ltd has announced a new one-pack pigmented etching primer designated 08:25.

Developed with flexibility of use in mind, this primer is claimed to provide excellent adhesion to a variety of substrates – particularly when stoved – and improves the technical performance of the system in which it is incorporated.

Available in black, dark grey, mid-grey, light grey, red oxide and white, it is suitable for conventional spraying (at a rate of 7-8m² per litre), electrostatic spraying (9-10m² per litre), airless spraying and dipping.

The new primer does not have to be separately stoved before applying a stoving finish, provided the substrate surface is dry the application can be made and the system then fully cured as recommended.

Reader Enquiry Service No. 54

New from Synthese

Synthese has recently developed two new hydroxyl functional acrylic resins for

curing with polyisocyanates. Firstly, Setalux C-1181 SS-60, the main advantages of which are claimed to be extremely low polyisocyanate usage, good hardness, good mechanical properties, good adhesion, very high gloss and excellent flow.

Secondly, Setalux C-1186 SS-60, the advantages claimed are low polyisocyanate usage, excellent solvent resistance – which is unique for an acrylic resin with such a low hydroxyl content – excellent hardness, excellent mechanical properties, very high gloss and excellent flow.

Synthese has also developed a new thermosetting acrylic copolymer solution, Setalux C-1388 VV-60, for combination with melamine resins, it is claimed to have excellent outdoor durability, good hardness, good colour retention, good adhesion and durable flexibility on base-coat.

Reader Enquiry Service No. 55

literature

BSI publications

The British Standards Institution has made the following literature available:

Revision of "BS 476 Fire tests on building materials and structures Part 6 Methods of test for fire propagation for products". This work has been undertaken to improve the reproducibility of test results by clarifying procedures and more closely specifying the apparatus to be used.

A revision of the 1972 standard, "BS 4800 Paint colours for building", which brings the ideal of total colour coordination within the reach of the entire building industry.

"BS 6044 Specification for pavement marking paints", a new British standard which has been specially developed to accommodate modern requirements associated with the marking of roads, parking bays, airfield runways and similar surfaces. It will be of benefit to the

news

paint industry, local government and other purchasing authorities, and the various categories of road transport.

Reader Enquiry Service No. 56

Colour card from Sikkens UK Ltd

A new colour card is available for the Sikkens Cetol Transparent/Rubbol THB system of transparent low-maintenance finishes for exterior softwood and hardwood.

The colours are shown on 80 × 35mm wood chips to give an accurate impression of the finish. The new card also demonstrates how the final colour depends on the colour and type of timber used.

Reader Enquiry Service No. 57

New coatings and linings brochure

The newly formed Ensecote (Holdings) Group, specialists in the manufacture and application of protective industrial coatings and linings, has published a new brochure describing the services and facilities offered by the companies in the group.

A wide range of coatings systems is listed together with technical data on typical applications. Case histories are illustrated in colour.

Reader Enquiry Service No. 58

Particle size analyser

New product literature on the Micromeritics SediGraph-L Particle Size Analyser is now available from Coulter Electronics Ltd. The SediGraph-L complements Micromeritics X-ray SediGraph 5000D by employing light extinction to track sedimentation of both organic and inorganic particles. This automatic instrument features a diameter range of 100 to 0.1 microns and high resolution with better than ±2 per cent reproducibility.

Reader Enquiry Service No. 59

meetings, etc.

BPVLC programme

The Birmingham Paint, Varnish and Lacquer Club has published a programme of meetings to be held during the 1981-1982 session.

Reader Enquiry Service No. 60

OCCA news

OCCA-34 Exhibition

27-29 April 1982

Cunard International Hotel
Hammersmith, London W6

INNOVATIONS AT OCCA-34

ABC

A member of the Audit Bureau of
Circulations

Innovations at OCCA-34 welcomed

The Annual Exhibition of the Oil & Colour Chemists' Association has long been known as the international forum for display and discussion for the surface coatings industries and – in pursuance of this theme – the Exhibition Committee has made innovations in the arrangements for OCCA-34 which have already been welcomed. These are set out below and intending exhibitors are asked to send in their applications as soon as possible so that adequate time is available for making all arrangements and publicity.

1. Discourse sessions

The opportunity is offered without charge to exhibitors to present short discourses on **commercial or technical developments**. These sessions will take place in separate rooms; full publicity will be given in *JOCCA* and in the *Official Guide* to the Exhibition.

2. Demonstrations/competitions

The Association wishes to extend the



The Cunard International Hotel

æo

A member of the Association of
Exhibition Organisers

scope of the exhibition, particularly in the application field, and encourages exhibitors to arrange demonstrations, to hold competitions and to take advantage of presenting their company image and products before visitors who, in recent years, have been drawn from between 40-50 countries as shown on the Association's Audit Bureau of Circulations Exhibition Data Form.

3. Dinner dance

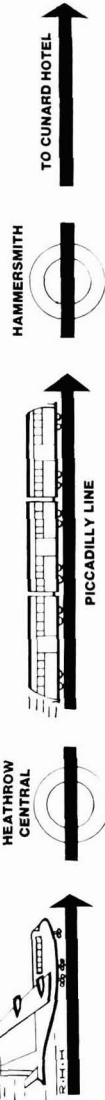
As the social highlight of the Exhibition, the Association has arranged for its prestigious dinner dance at the **Savoy Hotel**, London, WC2 to be held on the middle day of the Exhibition (Wednesday 28 April) thus affording exhibitors an excellent chance to entertain their customers. Exhibitors who usually hold social functions during OCCA Exhibitions are asked not to hold them on this evening.

4. Stands and rooms

Exhibitors can exhibit on stands of the shell scheme pattern or in special



Motif designed by Robert Hamblin



Dynamit Nobel

CHEMICALS

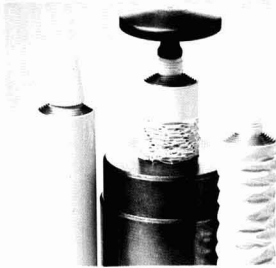
DYNAPOL[®] L
and lacquers stay in brilliant shape



DYNAPOL® L

A range of high molecular, linear, saturated polyesters, based on terephthalic acid. DYNAPOL L lacquer films have extremely high adhesive strength; they are highly elastic, scratch-resistant and impact-resistant, and they do not yellow with age. Can be stamped and deep-drawn, even under extreme loads. Also tasteless and completely safe in terms of foodstuffs legislation (BGA and FDA). Suitable for use as external and internal lacquer on packagings, and for sterilizable applications.

DYNAPOL L is available with different elasticity and viscosity properties as DYNAPOL L 205, L 206, L 411 and L 850. Main applications: roll-coating on sheet and strip, decorative lacquers for packagings, internal protective lacquers for packagings, coilcoating primers and top-coats, foil lacquers.



DYNAPOL® LH

A range of linear, saturated polyester resins containing hydroxyl groups. For use in combination with cross-linking resins to obtain top-quality, full-bodied industrial stoving finishes. DYNAPOL LH lacquer coatings are flexible, have good deformation properties and excellent adhesion to a metal substrate, such as aluminium, steel or galvanized steel. Good surface hardness and resistance to weathering and chemicals. Especially suitable for outdoor use particularly in the building industry.



Also suitable for use on packaging materials.

DYNAPOL LH 812 and LH 818 are fully compatible with the high molecular polyesters of the DYNAPOL L range. Their properties may be modified and rendered suitable for specific applications by combined use of DYNAPOL L/LH grades. Main applications: coilcoating lacquers, decorative lacquers for packagings; also suitable for UV printing methods.

DYNAPOL polyesters at a glance:

DYNAPOL	L 205	L 206	L 411	L 850	LH 818	LH 812
Form supplied	Granules	Granules	Granules	Granules	Solution	Solution
Hydroxyl No.	10	5-10	5-10	5-10	15	35
Molecular weight	15000	18000	16000	14000	7000	3000
Glass temperature	70°C	70°C	50°C	40°C	40°C	30°C

Further products in development.

DYNAPOL® L DYNAPOL® LH

Our range of products also includes:

DYNAPOL® H

Branched polyester containing hydroxyl groups

DYFLOR® 2000

Polyvinylidene fluoride for EPS and WS coatings

DYNASIL®

Silicic acid ester

DYNATRI® LQ

Specially stabilized trichloroethylene

RHENOFLEX®

Chlorinated polyvinylchloride

ICDAL® TE/TI

Electrical insulating lacquer resins

WITAMOL®

Plasticizers, phthalic acid ester

WITACLOR®

Plasticizers, chloroparaffin

* = registered trade mark

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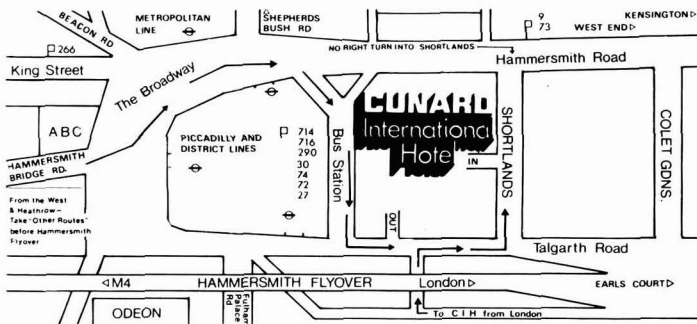
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CHEMICALS

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Location of the Cunard International Hotel



mation relating to raw materials, plant and equipment used in the paint, polymer, printing ink, colour, adhesive and allied industries, both in their manufacture, processing and application.

The Exhibition Committee will be particularly pleased to welcome exhibits from companies relating to the new energy efficient, low-polluting technologies, including powder coatings, high solids coatings, radiation curing, water-based coatings and other developments.

OCCA-34 will take place at the Cunard International Hotel, Hammersmith, London, W6 on 27, 28 and 29 April 1982 from 9.30 a.m. to 5.30 p.m.

Those organisations intending to exhibit are urged to submit their applications as quickly as possible.

individual rooms of varying sizes. Exhibitors are permitted to serve alcoholic refreshments on stands but some may feel that they wish to have private talks in separate rooms and for exhibitors taking both a stand and a room the Association offers substantial reductions of 25 or 50 per cent on the room charges only.

5. Special advertising rates

The OCCA Exhibition is a first class medium to project your spring sales campaign and to help exhibitors who wish to reinforce this by advertising in

JOCCA, the Association has recently introduced a special 3 insertion rate which can, if so desired, cover the *Official Guide* as well as the *Preview and Review* issues of the *Journal*. JOCCA won the 1980 ABC Reed International Award for its *Media Data Form* and this shows that advertisements reach over 22,000 readers in 80 countries at a cost of less than 1p each!

Aim of the Exhibition

The aim of the Exhibition is the presentation of commercial and technical infor-

Manchester Section

1981 OCCA National Golf Tournament and Manchester Section Jubilee Golf Trophy

Fifty-one members and their guests competed in the 1981 event held at the Stockport Golf Club, Cheshire on Wednesday 9 September. Numbers were lower than expected for this the premiere event in the OCCA golf calendar. This was due to distribution difficulties with the *Journal* which were beyond the control of OCCA. The scoring was based on a Stableford competition in which the handicap allowance was 7/8 and the maximum stroke allocation was 18. Weather conditions this year were favourable and less variable than the golfing styles displayed.

An excellent round of golf was followed by an equally satisfactory dinner during which the Manchester Section Chairman, Frank Redman, welcomed members from visiting sections who included Graham Fowkes, Bristol Section Chairman, and Norman Cochrane, organiser of the West Riding Golf Trophy.

The presentation of prizes by Frank Redman, assisted by Jim Jackson – the 1980 OCCA National Golf winner – followed the dinner and they were distributed thus:

1981 OCCA National Golf Trophy plus replica – D. Thomas



(Left to right) Frank Redman and Dave Thomas – the "crowning ceremony"

1981 Manchester Section Jubilee Golf Trophy plus tankard – D. Thomas

Dave Thomas – a famous golfing name – is a new Manchester Section member employed by Crown Paints. He has a 17 handicap and returned a score of 38 points in this year's competition, a memorable maiden attempt. In addition to the two trophies mentioned above, Dave "collected" a golf trolley donated by BTP Ltd.

Second prize went to John Everett, a previous Manchester Section trophy winner, with a score of 35 points. Return-

ing the same score was the winning guest, P. Vallance. The prizes were then distributed on a diminishing points basis until the booby prize winner, who shall remain nameless, collected a single ball for single points. Finally the prizes for "twos" were awarded to Messrs Gill, McGuinness, Crossley, Leadbetter and Cochrane.

The Section's gratitude is extended to those companies and individuals that generously donated prizes.

It is hoped to follow this successful event with a 1982 event at Stockport, complete with improved publicity.

F.B.W.

Ontario Section

The following meetings have been arranged for 1982 by the Ontario Section:

Wednesday 20 January, 1982
 Wednesday 17 February, 1982
 Wednesday 17 March, 1982
 Wednesday 21 April, 1982 – AGM
 Wednesday 19 May, 1982

Programme details will be sent to all Section members in advance of each event. All meetings will be held at 6.00 p.m. in the York Suite of the Cambridge Motor Hotel, 600 Dixon Road, Rexdale (Toronto), Ontario. Dinner tickets are Cdn \$15.00 per person. Overseas members and friends will be welcome.

OCCA news

Midlands Section

Ladies' night

On Friday 25 September 1981, the annual ladies' night of the Midlands Section was held in the Westbourne Suite of the Botanical Gardens, Edgbaston, Birmingham.

The guests, welcomed by the Midlands Section Chairman, Mr R. L. Devenish, included the President of OCCA, Mr D. J. Morris, the President of the Birmingham Paint Varnish and Lacquer Club, Mr R. Inglesstone and Mrs Inglesstone, the Director & Secretary, Mr R. H. Hamblin and various section chairmen and their ladies. Unfortunately the President's wife was unable to attend owing to ill health and the Section wished her a speedy recovery.

In his speech proposing the toast to the ladies and guests, the Chairman thanked all the members' companies concerned for their continuing support.

In his reponse on behalf of the ladies and guests, the President said that this was his first official function since coming to office and kept the audience amused with some lighthearted quotations.



Pictured from left to right: Don Clement (Hon. Secretary of BPVLC, member of OCCA's Technical Education Committee), Roy Inglesstone (Immediate Past President of BPVLC, member of OCCA Midlands Section), John Green (President of BPVLC, past OCCA Midlands Section committee member), Ray Tennant (President-elect of BPVLC, past OCCA Midlands Section committee member) and Harry Griffiths (Treasurer of BPVLC, past Chairman of OCCA Midlands Section)



Shown at the Midlands Section ladies' night (left to right), Mr R. Inglesstone (President, BPVL Club), Mrs Inglesstone, Mr T. L. M. Humphrey (Chairman, Scottish Section), Mrs Humphrey, Mr V. Moore (Chairman, Thames Valley Section), Mrs Moore, Mr R. L. Devenish (Chairman, Midlands Section), Mrs Devenish, Mr D. J. Morris (President), Mrs Redman, Mr F. Redman (Chairman, Manchester Section) and Mr R. H. Hamblin (Director & Secretary)

Afterwards the members and guests adjourned to the ballroom where dancing to the Smokey Bears Big Band continued until 1.00 a.m.

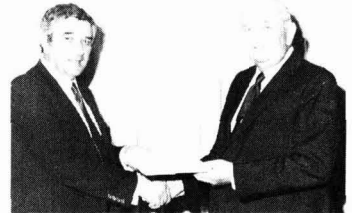
B.E.M.

News of members

Mr. C. Butler retires as SPL personnel director

Mr C. Butler attended a dinner held at the Walton Hall Country Club to mark his retirement as personnel director of SPL (Holdings) Ltd of Batley.

At the dinner were over 60 SPL directors, colleagues and their wives, as well as members of Mr Butler's family. Leslie Silver, chairman of SPL and a past President of OCCA, presented Mr Butler with a luxury cruise for two, other guests



(Left to right) Mr L. Silver and Mr C. Butler

marked the occasion with the gift of a colour television.

Cecil Butler joined SPL in 1968 after working for many years with International Paints in Ripon where he still lives with his wife, Louise.

Mr Butler served as Chairman of the West Riding Section, 1959-61, and is currently an Elective Council Member and Representative of the Auckland Section.

John Green of BIP elected BPVLC president

John Green, who is Coating Resins marketing manager with the Chemicals Division of BIP and who has served on the Midlands Section Committee a number of times, has been elected President of the Birmingham Paint, Varnish & Lacquer Club - "The Paint Club" as it is known in the industry.

In addition to bestowing a personal honour upon himself, John considers that the Paint Club has also honoured BIP. Traditionally, Paint Club presidents were elected only from amongst representatives of paint manufacturers. This is the first time a resin manufacturer has been so honoured; the club recently changed its rules to permit this wider outlook.

John is married and lives in Sutton Coldfield; he has been with BIP for over 25 years.

News of members

Founder Chairman of Zimbabwe Branch on worldwide tour

Mr R. E. G. Johnson, the founder chairman of the Zimbabwe Branch of the General Overseas Section, is undertaking a worldwide tour in November 1981-January 1982 to Rio de Janeiro, Miami, Honolulu, Auckland, Sydney, Melbourne, Perth and Johannesburg.

He will return to Zimbabwe at the end of January 1982 and hopes, during his tour, to meet other OCCA members and members of OCCA Australia.

Member of Council visits Japan

Members who attended the Association's Conference at Bath in June 1981 had the pleasure of meeting Dr Kenjiro Meguro (Vice-President of the Japan Society for Colour Material) and his wife and will be interested to learn that, in a recent visit to Japan, Mr J. R. Bourne (Hon. Secretary of the Association) met Dr and Mrs Meguro and had a traditional meal with them.

On 1 October, Dr Meguro was promoted to director of the Science University of Tokyo.

Obituary

Dr Sydney Hector Bell, OBE

President 1965-67

Honorary Member June 1976

Sydney Bell, who died in Teddington Memorial Hospital on 15 August 1981, was widely known to OCCA members in the UK and overseas as an outstanding scientist, lecturer, participant in technical discussions and after-dinner speaker.

He was educated at Battersea Grammar School and the Royal College of Science, obtaining an honours degree in chemistry in 1931 and a PhD two years later for work on "adsorption, retentivity and heat of wetting of active charcoal". He joined the Paint Research Station of the Research Association of British Paint, Colour & Varnish Manufacturers (as it then was) as a physical chemist in 1933, working initially on the structure of lithopone. This led, naturally, to a study of pigment/medium relationships which were a major interest for the rest of his life. The opening of an extension to the Paint RA's Teddington Laboratories in 1936 by Ramsay MacDonald and the associated open days demonstrated his organising abilities and the following year Dr L. A. Jordan, Paint RA's founder Director, made him his personal assistant.

The war was a time of intense activity by the Research Association. Sydney Bell

was concerned with many technical projects, notably the numerous applications of luminous compositions. He was also much involved with the many attendant negotiations and discussions with armed services and departments of Government, particularly the Ministry of Supply (on protection of equipment, etc.) and the Ministry of Home Security (on camouflage, etc.).

In 1945 he became Secretary of the Research Association, combining with this the post of Development Officer which included responsibility for the production of all the Association's technical publications. In 1953 he was made deputy Director & Secretary, the position he held until his retirement in 1975.

He was a great believer in looking at things (both abstract and physical). He was greatly interested in developments in electron microscopy and especially the studies of paint pigments etc., carried out at Paint RA.



Dr S. H. Bell, OBE

He was keenly interested in technical education in a very wide sense. He helped the Paint Industry Technical Education Study Group in preparing its report and was the author of "More than meets the eye", the Paintmakers Association's guide to the scientific and technical background of the paint industry and the technical training and careers it offered. He was also Chairman of the City & Guilds of London Institute's Advisory Committee on Paint Technology, an external examiner for the Polytechnic of the South Bank and the Royal Institute of Chemistry's Postgraduate Research Diploma, and a member of OCCA's Professional Grade Committee. He also gave practical advice to several generations of young people at Paint RA including new recruits preparing papers for subject panels, sandwich course students undergoing training or people from industry attending the courses in paint technology introduced in 1956.

He was a highly accomplished writer and lecturer and contributed numerous lectures, articles and papers particularly on pigment/medium relationships, the structure of paint films and electron microscopy and other techniques. He

OCCA news

gave a plenary lecture (on behalf of OCCA) at the 1964 FATIPEC conference in Vichy on "Pigment/medium relationships". In 1967 he toured Scandinavia as a guest lecturer of the Scandinavian Federation for Paint Technology with a paper on "Paint films are not homogeneous", he also lectured at a postgraduate course in Utrecht and at a meeting of the German Chemical Society's Pigment and Paint Study Group (of which he was made a member). He also lectured at several meetings including those of OCCA sections on another of his great interests, heraldry, and was much appreciated as an after-dinner speaker.

He recognised at an early stage the importance that health & safety matters would have, and was one of the original members of the Paint Industry's Health & Safety Committee, later a sub-committee of the Technical Committee.

He served on a number of OCCA committees and on Council and in 1950 was asked to make a survey of the Association's rules. His principle in this was that rules should be "the servants not the masters", and the new set of rules adopted as a result in 1953 not only covered procedures but set a policy pattern with scope for change and development. During his Presidency (1965-67), he took this a stage further by asking his fellow officers to join him in a "forward thinking" exercise. This did a great deal to stimulate the re-thinking of OCCA's future role. He greatly valued being made an Honorary Member of the Association.

He was a member of many other societies and served on numerous committees. He served on the Council and Publications Committee of the Society of Chemical Industry and was Chairman of the Polymer Group from 1955 to 1957, an important time for the paint industry in deciding which polymers would be used in coatings technology. He also served on the Colloid and Biophysics Committee of the Faraday Society, the Library Committee of the Chemical Society and the Royal Society's National Committee for Chemistry.

In addition to technical matters, Sydney had a wide range of interests including music, art (he was also an amateur painter), literature and natural history. His many friends will greatly miss his wit, kindness, warmth and erudition but his influence for good-will, will long continue.

M.K.J.

Hull Section

Commendation Award

The Session opened with the cancellation of the Section's annual dinner and dance. For some years attendance at this function has declined and in view of the limited support likely for this year's function, it was felt by the Committee to be better if the dinner and dance were abandoned and a less formal evening event programmed for later in the session. Detailed planning has now begun.

The Section presented the Association's Commendation Award to Mr E. Armstrong at a special meeting of the Section on 24 September. The venue



Mr P. W. Munn (left), Chairman of the Hull Section, presenting a Commendation Award to Mr E. Armstrong who has twice held the office of Chairman of the Section

for this presentation was the Duke of Cumberland Hotel in Cottingham, near Hull, and it was decided that in future the Section's meetings would also take place at this hotel, rather than the venue published in the joint programme. The first technical meeting at the new venue took place on 2 November 1981.

The Section held a very informal evening at the Hull Bowl on Saturday 7 November which was open to friends and members. The Committee welcomed as many as possible to a rather more strenuous evening than the Section usually indulges in.

P.W.M.

Association dinner dance

Next year's Association dinner dance will be held, as on previous occasions, at the Savoy Hotel, London. As an innovation it is scheduled to take place on 28 April 1982 – the middle day of the forthcoming Exhibition, OCCA-34. This will afford exhibitors an opportunity of entertaining their guests at this prestigious function. The guest of honour will be Mr N. Mischler, chairman of Berger Ltd and Hoechst UK Ltd.

Tickets are priced at £25 + VAT each and forms of application are available from the Director & Secretary at the Association's offices.

Association notices

Binding of the Journal

Members will be pleased to know that J. S. Wilson & Son, 14a Union Road, Cambridge CB2 1HE, will undertake the binding of back volumes of the Association's *Journal* sent in by individual members. Quotations will be given on request.

Members wishing to avail themselves of this facility should send the parts, securely wrapped, direct to J. S. Wilson & Son, enclosing the remittance for each volume, and ensuring that notes bearing names and addresses are enclosed with the parcels. It is particularly important that packets are sufficiently wrapped to negate the possibility of damage in the post.

1982 members subscriptions

Members are reminded that the 1982 membership subscriptions to the Association are payable on 1 January 1982. Forms were despatched to members in October or November, depending upon address.

The Commissioners of Inland Revenue have approved of the Association for the purpose of the 1970 Income and Corporation Taxes Act Section 192, so that a member subject to United Kingdom income tax is entitled to a deduction from the amount of his emoluments assessable to income tax under Schedule E for the whole of the annual subscription to the Association, provided that the subscription is defrayed out of

the emoluments of his office or employment and that the interests covered by the objects of the Association are relevant to such office or employment.

Members resident in the United Kingdom are reminded that if there is any change in the standard rate of Value Added Tax announced before they send in their 1982 subscription, the VAT payable on membership subscriptions is the amount applies on the date of payment.

1982 library subscriptions

The *Journal* subscription rate to non-members, including libraries, for 1982 will be £45.00 (\$100) post free by surface mail, home and abroad. Individual copies can be purchased for £4.00. Remittances should be sent with an order to the Association's offices.

Retired members

Council also wishes it to be known widely that in 1962 it introduced a reduced membership subscription rate for members who have retired from Business. This applies to a member who has completed 20 years as an Ordinary or Associate Member and has retired from business and normally has reached the age of 60; he may apply for his name to be retained on the register of members at an annual subscription rate of £3.00 and he will retain the same rights of membership as the class of membership to which he was attached upon retirement.

Members wishing to avail themselves of this concession should write, in confidence, to the Director & Secretary at the address on the contents page, giving the relevant information under the four headings: (a) name, address and section, (b) date of election, (c) date of retirement, (d) age.

Situations wanted

Any member who wishes to place a small advertisement (with Box Number) in the situations WANTED section of *JOCCA* can do so *without charge* by sending details to the Director & Secretary, marking the envelope "CONFIDENTIAL".

Monographs and student reviews

Council wishes to publish in the *Journal* occasional monographs or student review type articles for the benefit of younger technologists to act as introductions to some specialised fields in the industry. It is intended that such articles would then be reprinted as separate booklets. Council wishes to invite suggestions from members of suitable topics for such a series of articles, *together with the names of persons who would be willing to write the articles.*

Any members who would be willing to write such a monograph, or who can suggest another person who might be willing to do so, should write to the Director & Secretary at the address on the contents page giving full details.

Optional Professional Grade for Ordinary Members



Anyone who has allowed his membership of the Association to lapse and now desires to rejoin the Association is reminded that previous service as an Ordinary Member (or Registered Student) can be counted towards the qualifying period of membership set out in the regulations.

The attention of senior members of the Association is particularly drawn to the Licentiate Grade and they are asked to encourage younger technical personnel to take advantage of this important Association activity. Several colleges are now willing to help suitable candidates with the preparation of dissertations and a list of Colleges was given in the August 1981 issue of the *Journal* (p. 332).

Reprints of the regulations covering the Professional Grade are obtainable from the Association's offices, together with application forms.

List of successful candidates

As laid down in the report of the Working Party on Education, Training and Qualifications which was adopted in the institution of the Professional Grade, a list of those members in the Grade is published in the December issue of the *Journal* each year. The tenth such list appears below and includes the names of members resident in 35 countries.

The section to which the member is attached is given in italics.

The certification fees at present are: Fellows £10.00 + VAT, Associates £6.00 + VAT, and Licentiates £3.00 + VAT. The regulations for admission to each grade appear on page 526 of this issue of the *Journal*.

Fellows

- Addenbrooke, Brian John (*Midlands*)
- Aitken-Smith, Frank Joseph (*Auckland*)
- Anneveldt, Jan Johan Willem (*Natal*)
- Apperley, Thomas William James (*West Riding*)
- Archer, Harold (*Manchester*)
- Arnold, Michael Henry Miller (*London*)
- Ashworth, Norman (*Manchester*)
- Astfalk, Anthony Noel (*Transvaal*)
- Atterton, Donald (*Scottish*)
- Austin, Denis Leonard (*Bristol*)
- Bailey, John Noel (*Newcastle*)
- Banfield, Thomas Arthur (*London*)
- Bayliss, Derek Arthur (*London*)
- Beachen, John Frederick (*Auckland*)
- Beere, Andre Jaimie (*Thames Valley*)
- Bell, Richard Thomas (*General Overseas—Australia*)

- Bennett, Norman Arthur (*General Overseas—Malta*)
- Bester, Lawrence Percy (*Transvaal*)
- Bhumkar, Chidanand Jayram (*General Overseas—India*)
- Birrell, Peter (*Ontario*)
- Bishop, Eric Harold Abbott (*West Riding*)
- Bohringer, Eberhard (*London*)
- Boroky, Joseph Stephen (*General Overseas—Australia*)
- Bosman, Herman Izak (*Transvaal*)
- Bourne, John Robert (*Midlands—Trent Valley Branch*)
- Brooks, Leo James (*London*)
- Brown, Arthur Ernest Girdlestone (*London*)
- Butcher, George Alfred (*Midlands*)
- Butcher, Kenneth William George (*Manchester*)
- Butler, Cecil (*West Riding*)
- Caldwell, David George (*Wellington*)
- Campbell, George Alexander (*Manchester*)
- Carr, William (*Manchester*)
- Carter, Eric Victor (*London*)
- Chatfield, Herbert Walter (*London*)
- Chessman, Clifford Reginald (*Transvaal*)
- Clarke, Harry James (*Midlands*)
- Colborn, Douglas Charles (*Thames Valley*)
- Cole, Derek (*General Overseas—Australia*)
- Cole, Reginald Joseph (*London*)
- Collier, Claude William (*Midlands—Trent Valley Branch*)
- Collings, Arthur Geoffrey (*London*)
- Cook, Harold Gilbert (*London*)
- Coupe, Raymond Richard (*London*)
- Courtman, Frank (*Manchester*)
- Coverdale, Peter Frederick Muir (*Midlands*)
- Cutter, John Outram (*London*)
- Davidson, John Dixon Wilson (*Scottish*)
- Davidson, Sigismund Leonard (*General Overseas—USA*)
- de Jong, Jan Lauwrens (*Transvaal*)
- Dowsing, George Frederick (*London*)
- Draper, Patrick Albert (*Natal*)
- Duckworth, Samuel (*Manchester*)
- Duligal, Eric Arthur (*Transvaal*)
- Dunkley, Frederick George (*Midlands—Trent Valley Branch*)
- Durrant, George Geoffrey (*Hull*)
- Easton, James Douglas (*Ontario*)
- Eglington, Roland Alexander (*Natal*)
- Ellinger, Marianne Livia (*London*)
- Entwistle, Thurston (*Newcastle*)
- Ernst, Joel (*London*)
- Finn, Stanley Russell (*London*)
- Froggatt, Joshua John (*London*)
- Fullard, John Edward (*Transvaal*)
- Furuhjelm, Viktor Henrik (*General Overseas—Finland*)
- Gate, Peter Atholl Jackson (*Transvaal*)
- Gay, Philip James (*Hull*)
- Geddes, Kenneth Raymond (*Manchester*)

- Gellay, Victor Peter (*London*)
- Gellman, Alexander (*London*)
- Ghosh, Sunil Kumar (*General Overseas—India*)
- Giesen, Mathias Franz (*General Overseas—Germany*)
- Gillan, James Graham (*Manchester*)
- Gollop, Percy Lionel (*London*)
- Gooch, Colin (*Wellington*)
- Gosling, Harry (*Manchester*)
- Graham, Thomas (*Manchester*)
- Grainger, William Alan (*Irish*)
- Gray, Denis Roy (*West Riding*)
- Grover, Donald Henry (*London*)
- Haken, John Kingsford (*General Overseas—Australia*)
- Hamburg, Herman Rudolf (*London*)
- Hanson, Robert Philip (*Newcastle*)
- Hawkey, John Albert Lawrence (*London*)
- Hill, Derek Alfred Wheeler (*London*)
- Hill, Gilbert Victor Geoffrey (*Thames Valley*)
- Hill, Lawrence Albert (*General Overseas—Australia*)
- Hipwood, Hubert Allan (*London*)
- Hodgson, Kenneth Vickerson (*Newcastle*)
- Holbrow, Gordon Leonard (*London*)
- House, Dudley James (*Natal*)
- Hutchinson, Geoffrey Herbert (*Scottish—Eastern Branch*)
- Inshaw, John Leslie (*Thames Valley*)
- Iyengar, Doreswamy Raghavachar (*General Overseas—USA*)
- Jacob, Basil (*Thames Valley*)
- Johannsen, Ralf Peter (*Cape*)
- Johnson, Roland Emanuel George (*General Overseas—Zimbabwe Branch*)
- Jolly, Anthony Charles (*Manchester*)
- Kalewicz, Zdzislaw (*General Overseas—France*)
- Kane, Joseph Richard (*London*)
- Keenan, Henry Wilfred (*London*)
- Kotwal, Hoshidar Peshotan (*General Overseas—Pakistan*)
- Kut, Sigmund (*London*)
- Landmann, Axel Wolfgang (*London*)
- Lasser, Howard Gilbert (*General Overseas—USA*)
- Lewin, John Buckingham Grey (*London*)
- Lewis, Fred (*Manchester*)
- Ley, John Barry (*London*)
- Lomas, Harold (*Ontario*)
- Lunt, Walter Richard (*West Riding*)
- McKelvie, Archibald Neil (*London*)
- McLean, Angus (*Scottish*)
- McQuirk, Peter John (*London*)
- Mitchell, John Edmund (*Manchester*)
- Mitchell, Seward John (*Midlands*)
- Moll, Ivor Stuart d'Anvers (*Manchester*)
- Monk, Cyril James Henry (*Bristol*)
- Moon, William Robert (*Manchester*)
- Morgans, Wilfred Morley (*London*)

Morris, David
(*General Overseas-Nigerian Branch*)
Munn, Raymond Henry Edward
(*London*)
Newnham, Herbert Alan (*London*)
Newton, Dennis Sydney (*London*)
Newton, Donald Stringer (*Bristol*)
Nutt, William Owen (*London*)
Oostens, Emile Elie Eugene
(*General Overseas-Belgium*)
Parfitt, Geoffrey Derek (*Newcastle*)
Pienaar, Dirk Jacobus (*Transvaal*)
Piggott, Kenneth Elliot (*Natal*)
Poborca, Stefan (*Midlands*)
Polaine, Sidney Alan (*London*)
Raaschou Nielsen, Hans Kristian
(*General Overseas-Denmark*)
Ray, Stanley Arthur (*Midlands*)
Rechmann, Heinz
(*General Overseas-Germany*)
Reid, John Rodney Stanford
(*Natal*)
Rleigh, Albert Kenneth
(*General Overseas-Australia*)
Roe, David Edwin (*London*)
Rose, Charles (*Manchester*)
Rouse, Robert Earnshaw (*Transvaal*)
Rubin, Wallace (*London*)
Rudram, Arthur Thomas Stephen
(*London*)
Saunders, Laurence Frederick (*Natal*)
Seymour, Norman Henry (*Manchester*)
Sharpe, Eric Edward Victor
(*Transvaal*)
Shepherd, Joseph (*Newcastle*)
Silsby, Denys John (*Midlands*)
Sim, Richard Alastair
(*General Overseas-Australia*)
Simon, Raymond (*Irish*)
Slade, Harold Aitken (*Midlands*)
Slinn, Thomas Walter (*Wellington*)
Smith, Francis Mark (*Manchester*)
Smith, Harry (*Manchester*)
Smith, John George Nixon (*Newcastle*)
Sowerbutts, Frank (*London*)
Sreeves, John Ernest (*Midlands*)
Stoodley, Keith Herbert (*London*)
Stoyle, Francis Wilbert (*Irish*)
Talbot, Ernest Alexander
(*Thames Valley*)
Tatton, William Henry
(*Thames Valley*)
Tawn, Alec Richard Hornsey (*London*)
Taylor, John Roberts (*Bristol*)
Thukral, Prem Sager (*London*)
Tickle, Trevor Cyril Kenneth
(*Manchester*)
Tooke-Kirby, John Theodore
(*London*)
Tooth, John Henry Collins (*London*)
Touchin, Herbert Roy (*Manchester*)
Turner, John Harry Wallace
(*Manchester*)
Valentine, Leslie (*London*)
Walker, Alan Gordon (*Newcastle*)
Wall, Dennis Charles (*Manchester*)

Warner, Eric Albert Andrew
(*Wellington*)
Watkinson, Leonard James
(*West Riding*)
Westwood, George Ernest (*London*)
White, Robert Arthur (*Auckland*)
Whiteley, Peter (*London*)
Whitfield, Thomas (*Auckland*)
Wilkinson, Thomas William (*Hull*)
Willis, Gervase Hewitson (*Manchester*)
Wood, George (*London*)
Woodbridge, Richard John (*Bristol*)
Worsdall, Herbert Charles (*London*)
Yorath, Robert Stanley (*Wellington*)

Associates

Abel, Adrian George (*Manchester*)
Acey, John Arthur (*London*)
Adams, John Charles (*Midlands*)
Adams, Terry Ernest (*London*)
Anthony, Alan Sydney (*London*)
Armstrong, Edward (*Hull*)
Armstrong, Herbert Walter Maynard
(*London*)
Arnold, Frank (*Manchester*)
Ashton, Ronald (*Manchester*)
Assink, Jo (*Auckland*)
Awan, Mumraiz Khan
(*General Overseas-Kuwait*)
Bains, Ranjit Singh (*London*)
Baldwin, George William (*Manchester*)
Bannington, Donald Bertram (*London*)
Bargrove, Kenneth Laurence (*London*)
Barnes, Peter James (*London*)
Barton, James Francis (*London*)
Batch, Alan James Edward (*London*)
Bax, John Charles
(*General Overseas-USA*)
Beadle, Roy William (*London*)
Beckley, Albert Henry (*London*)
Bell, Brian Robert (*Midlands*)
Bell, Eric James (*London*)
Belsham, Barry Michael
(*General Overseas-Cyprus*)
Bentley, Major Gordon (*West Riding*)
Bird, George Donald Chaplyn
(*Midlands*)
Bloomfield, Kenneth Vincent (*London*)
Bluck, Ross Steele (*Wellington*)
Bolam, Ion Barrow (*Newcastle*)
Bose, Sunil Kumar (*London*)
Bowler, Kenneth Ernest (*Midlands*)
Boxall, John (*Thames Valley*)
Brooke, Leslie John (*Bristol*)
Brown, Peter Thomas (*London*)
Byrns, Arthur Robin (*Cape*)
Caffery, George Francis (*London*)
Calder, Robert Malcolm (*Auckland*)
Campbell, Douglas Shaw (*Transvaal*)
Campey, Leslie John Randall
(*Ontario*)
Canterford, Barry Albert (*London*)
Cartwright, Jeffrey (*London*)
Catchpole, David Thomas (*Scottish*)
Catherall, Kenneth David (*Midlands*)
Chambers, Anthony (*London*)
Chebsey, Maurice (*Manchester*)
Chellingsworth, Horace Thomas
(*Midlands*)
Chippington, Kenneth Alan (*Bristol*)
Churchman, Anthony Edward
(*London*)
Clark, Laurence Norman (*London*)
Clark, Michael Denis Thomas
(*Wellington*)
Clarke, Raymond John (*Ontario*)
Clayton, David Walter Norbury
(*Manchester*)
Clausen, Hans Christian (*Natal*)
Clement, Donovan Harry (*Midlands*)
Coates, John Allen (*Manchester*)
Cole, Francis William (*Midlands*)
Constantinides, Erricos (*London*)
Cordwell, Terrence Allan
(*Midlands-Trent Valley Branch*)
Cowie, Edward Bruce (*Natal*)
Cox, Garth Anthony (*West Riding*)
Craske, Anthony John
(*General Overseas-Indonesia*)
Cunnington, Robin Roy Carol
(*London*)
Daggett, Wilfred Francis (*London*)
Dalton, Frank
(*General Overseas-Denmark*)
Davies, Frank Watkin (*Manchester*)
Davis, Brian Frederick James
(*London*)
Davis, Reginald Albert (*Bristol*)
de Waal, Tielmann Johannes
(*Cape*)
Dennis, Reginald Herbert (*London*)
Devine, James (*Transvaal*)
Donkersley, Brian (*London*)
Downham, Stephen Airey
(*Manchester*)
Drew, Harold Henry Lennox
(*Midlands*)
Dunn, Paul Alan (*London*)
Durant, Leslie Arthur William
(*London*)
Dury, Ian Clifford James
(*Thames Valley*)
Eaton, Michael George
(*Thames Valley*)
Ekeh, Godwin Chukoemeka
(*General Overseas-Nigerian Branch*)
Elliott, Peter (*London*)
Eltringham, James Norman (*Auckland*)
Fell, Alan William (*Thames Valley*)
Fernandes, Larry Raphael Francis
Joseph (*London*)
Field, Lawrence Edward (*Natal*)
Finlay, Cecil Newton (*Newcastle*)
Fisher, Leslie Alexander
(*General Overseas-Malaysia*)
Flood, Geoffrey Terence (*Manchester*)
Ford, Keith Sydney (*Manchester*)
Formanek, Leopold
(*General Overseas-Czechoslovakia*)
Freund, Kurt Alfredo
(*General Overseas-Ecuador*)
Fry, Jack Ian (*Wellington*)
Garratt, Peter Garth
(*General Overseas-Austria*)
Gascoyne, John (*Auckland*)
Gibson, Frank (*Manchester*)
Gibson, John Carrington (*Hull*)
Gilliam, Brian Frederick (*London*)
Goodman, Robert John
(*General Overseas-Spain*)
Green, Basil Ray
(*General Overseas-Trinidad*)
Green, Brian James (*London*)
Greenall, Brian John (*Wellington*)
Greenfield, Eric (*Midlands*)
Griffiths, Henry James (*Midlands*)
Grime, David (*London*)
Gunn, David John (*Midlands*)
Gunn, Reginald (*Thames Valley*)
Hackney, Thomas (*Auckland*)
Hamilton, Alexander (*Scottish*)
Hardie, Ian William (*London*)
Harrison, Cyril Geoffrey (*Hull*)
Harty, David Basil
(*General Overseas-Australia*)
Hasnip, John Anthony (*Hull*)
Heald, Desmond (*Manchester*)
Herriott, Charles Edward (*London*)

Hickman, Edwin Peter (*Midlands*)
 Hill, Raymond Forsyth (*Scottish*)
 Hitchmough, Rex Henry (*London*)
 Hodge, Robert Alexander Paul
 (*Auckland*)
 Holden, William Desmond
 (*Manchester*)
 Holt, Clifford (*West Riding*)
 Homden, Kenneth James Arthur
 (*London*)
 Honiball, Alan Edward (*Scottish*)
 Hopper, Derek Edgar (*Midlands*)
 Howard, Eric (*Manchester*)
 Howells, Barry John (*Hull*)
 Howes, Edward John (*London*)
 Hughes, Anthony Harold (*Manchester*)
 Humphrey, Thomas Lawson Myles
 (*Scottish*)
 Hutchinson, Martin Alfred
 (*West Riding*)
 James, Tudor Herbert (*Irish*)
 Jangbahadur, Shyam Sharan
 (*General Overseas-India*)
 Jenkins, Brian Gordon Allan
 (*Auckland*)
 Johnsen, Svend
 (*General Overseas-Denmark*)
 Johnstone, James William
 (*Manchester*)
 Jones, Derek Frederick Arthur
 (*Thames Valley*)
 Jones, Geoffrey Peter (*Wellington*)
 Kaye, Dennis David (*London*)
 Kenna, Frank William (*Manchester*)
 Kerr, Michael Anthony (*Manchester*)
 Khan, John Mohammed (*Manchester*)
 King, Charles William Henry
 (*Midlands*)
 King, Raymond John (*Midlands*)
 Kirkwood, Thomas (*Scottish*)
 Kirlew, Charles Wesley
 (*General Overseas-USA*)
 Kitchen, John Robert
 (*Midlands-Trent Valley Branch*)
 Knight, Richard Charles (*London*)
 Laker, Bernard George (*London*)
 Lakshmanan, P. R.
 (*General Overseas-USA*)
 Lander, Wilfred Terence (*London*)
 Lang, Robert (*Scottish*)
 Langford, Henry (*London*)
 Langley, Robert (*Scottish*)
 Lawrence, Simon Gayner (*Scottish*)
 Lawton, Cyril Victor (*Midlands*)
 Leathley, George Derek (*Auckland*)
 Le Maistre, Paul Francis (*Midlands*)
 Lever, Colin (*Manchester*)
 Lewis, Geoffrey John (*Midlands*)
 Lewis, John David (*Thames Valley*)
 Low, Charles
 (*General Overseas-Australia*)
 Macdonald, Alan (*Auckland*)
 Malik, Javed Haider
 (*General Overseas-Pakistan*)
 Mandagi, Willy
 (*General Overseas-Indonesia*)
 Mandelson, Jack (*Scottish*)
 Maple, Donald Peter (*London*)
 Martin, Christian Pierre
 (*General Overseas-France*)
 Maynard, Albert William David
 (*Overseas*)
 McCallum, Ian Robert (*Scottish*)
 McCapra, Ronald (*Auckland*)
 McClean, Michael Dennis (*London*)
 McDonald, Kenneth Roy (*Natal*)
 McDonnell, Christopher Robin Stack
 (*West Riding*)
 McFetridge, John Henry (*Wellington*)
 McKay, Alan Gordon (*London*)

McKean, James Newlands (*Auckland*)
 McMillan, James (*Manchester*)
 Mephram, Brian Edwin (*London*)
 Mikucki, Wiktor (*London*)
 Moore, Frank Roden (*West Riding*)
 Moore, James (*Thames Valley*)
 Moore, William Alexander (*Auckland*)
 Morpeth, Frederick Johnson
 (*Manchester*)
 Moss, Noel Sydney (*London*)
 Mouallem, Mouhammad
 (*General Overseas-Syria*)
 Mun-Gavin, Patrick William James
 (*Natal*)
 Munro, Hugh Anderson (*Scottish*)
 Murray, David John (*Manchester*)
 Myers, Gordon (*Natal*)
 Mynett, Raymond John (*Midlands*)
 Naess, Erik
 (*General Overseas-Norway*)
 Ness, Robert Alexander (*Auckland*)
 Newman, Derek George (*Transvaal*)
 Nolan, Michael Melvyn (*Irish*)
 Norton, Douglas Kent (*Midlands*)
 Oakley, Ernest (*Newcastle*)
 O'Connor, Eugene Daniel
 (*Manchester*)
 Ohene-Kwadade, Kofi
 (*General Overseas-Ghana*)
 Oswitch, Stanley (*Ontario*)
 Pace, Graham (*Thames Valley*)
 Parry, Martin Gerald (*London*)
 Patel, Shriish Manibhai (*Ontario*)
 Patrick, Alan Clive (*Irish*)
 Payne, Edward James (*Manchester*)
 Payne, Kenneth (*Bristol*)
 Pemberton, Joseph James (*London*)
 Pessall, Robert George (*Midlands*)
 Piper, Norman William (*Manchester*)
 Porteous, Barrie Milroy (*Ontario*)
 Potter, Francis
 (*General Overseas-Zimbabwe*)
 Proudley, Philip Miles (*West Riding*)
 Provan, Andrew Wilson (*Wellington*)
 Pyett, Albert Lawrence (*Manchester*)
 Quorn, Peter James (*Cape*)
 Rackham, John Michael (*Newcastle*)
 Rampley, Dennis Neil (*London*)
 Redman, Frank Benson (*Manchester*)
 Reeve, Frank Nicholson (*Transvaal*)
 Roberts, Peter David Mynwy
 (*London*)
 Robinson, Arthur Graham
 (*Manchester*)
 Robinson, Francis Derrick (*Hull*)
 Rothwell, Gerald William (*London*)
 Rout, Peter George (*West Riding*)
 Routley, Alan Francis (*London*)
 Russell, Frederick Charles (*London*)
 Rycroft, Christopher Peter (*London*)
 Scheinost, Bernd (*Natal*)
 Semple, James William (*London*)
 Sharp, Peter Frank (*Auckland*)
 Sharpe, David (*London*)
 Sheikh, Saeed
 (*General Overseas-Pakistan*)
 Shepherd, David William (*Bristol*)
 Silverwood, David (*Manchester*)
 Smith, Brian James (*London*)
 Smith, David Dorman (*Scottish*)
 Smith, Harry Bertram (*London*)
 Soman, Chettiparambil
 (*General Overseas-Spain*)
 Sowerby, Alan Hope (*Auckland*)
 Spaargaren, Albert Arend (*Natal*)
 Spargo, Robert (*Auckland*)
 Springett, Robert Arthur Edward
 (*London*)
 Staples, Peter Graham (*London*)
 Stephenson, Robert Perry (*Auckland*)

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Stone, James Bryan (*London*)
 Stott, Raymond (*Manchester*)
 Stretton, Elizabeth (*Manchester*)
 Surinphong, Julian Suriya
 (*General Overseas-Thailand*)
 Sutton, Peter Michael (*London*)
 Talwalkar, Vinayak Sakharan
 (*London*)
 Tape, Brian William Charles
 (*General Overseas-USA*)
 Taylor, Richard Anthony John
 (*London*)
 Taylor, Terence (*Manchester*)
 Thomas, Anthony
 (*General Overseas-Brazil*)
 Thornton, Philip Joseph Martin
 (*Irish*)
 Tillyer, Richard Brian (*London*)
 Topping, George David (*London*)
 Toovey, John (*Scottish*)
 Trevitt, Edwin William (*London*)
 Triggs, Francis Cyril (*London*)
 Troparevsky, Alejandro
 (*General Overseas-Argentina*)
 Tye, Terence Thomas (*Midlands*)
 Van der Merwe, Hendrik Albertus
 Johannes (*Natal*)
 Van Londen, Anton Matthijs
 (*General Overseas-Holland*)
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 Webb, John Peter (*London*)
 Webb, Laurence (*Manchester*)
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 Whalley, James
 (*General Overseas-Nigerian Branch*)
 Whating, Allan (*Manchester*)
 Wheatley, Kenneth Valentine (*Irish*)
 Whetstone, Peter John (*London*)
 Whiteside, Alexander Edward
 (*General Overseas-Australia*)
 Wilkes, David Graham (*Bristol*)
 Williams, Adrian Arthur Owen
 (*London*)
 Williams, Cyril (*Manchester*)
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 Wu, Andrew Chi Kit
 (*General Overseas-Hong Kong*)
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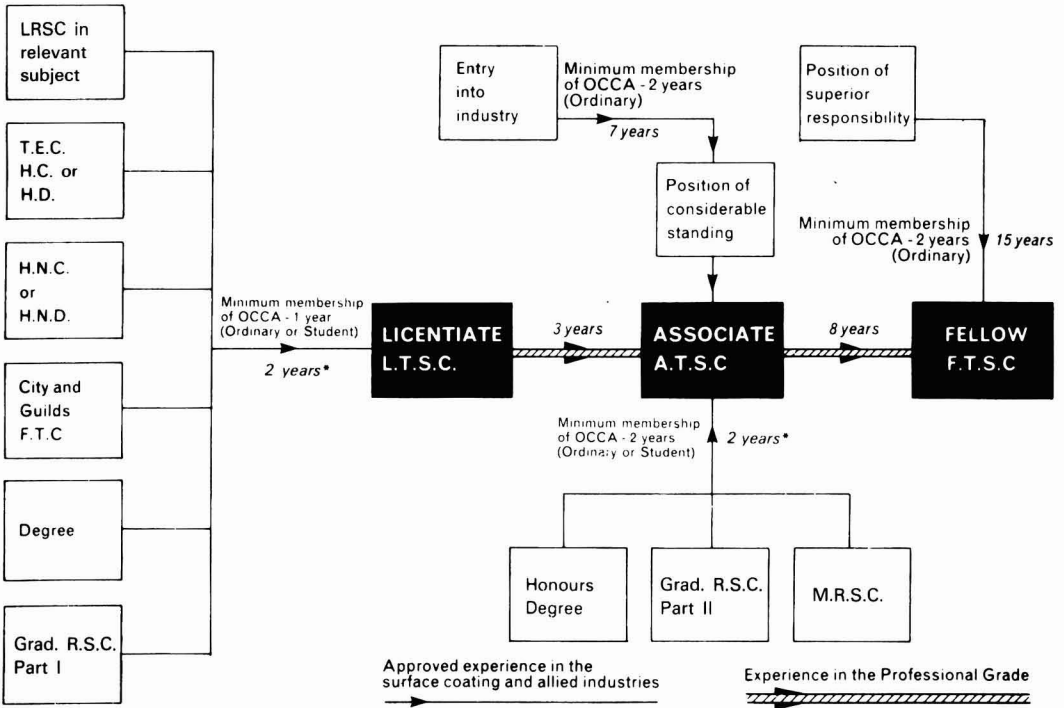
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Asiedu-Dompheh, Johnathan (*London*)
 Hayles, John William (*London*)
 Leonard, Michael William (*London*)
 Lodge, David William (*London*)
 Richardson, Robert Keith (*London*)
 Rogers, Michael Ambrose
 (*General Overseas-Trinidad*)
 Rowntree, Randal Peter (*Manchester*)
 Saggur, Anoop Kumar
 (*General Overseas-Kenya*)
 Sawyer, Olatunji Pekun
 (*General Overseas-Nigerian Branch*)

Optional Professional Grade for Ordinary Members

Regulations for admission to the Professional Grade—Amended February 1981

Routes to the Professional Grades



*Not necessarily after qualification – see regulations.

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.

A. Licentiate

1. Shall be an Ordinary Member of the Association and have been an Ordinary Member or Registered 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 Society of Chemistry in Coatings Technology or another relevant subject, such as advanced analytical chemistry, colour chemistry or polymer science.
- OR (b) Shall have passed the Higher Certificate or Higher Diploma of the Technician Education Council in Coatings Technology or other relevant subjects (or equivalent SCOTEC qualification).

- OR (c) Shall have passed Higher National Certificate or Higher National Diploma in a relevant subject (or equivalent SCOTEC qualification).
- OR (d) Shall hold the Full Technological Certificate of the City and Guilds of London Institute in a relevant subject.
- OR (e) Shall be a graduate in a relevant subject.
- OR (f) Shall have passed Part I of the examination for the Graduateship of the Royal Society of Chemistry or Council of Physics.
- OR (g) Shall have passed such other qualifications as approved by the Professional Grade Committee from time to time.
4. Shall have attained approved

- experience in the science or technology of coatings. It is not expected that sufficient experience would be gained in a period of less than two years in the industry. Approved experience may be gained before, during or after the qualifications in paragraph (3) above have been attained.
5. 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 subject directly associated with the science and technology of Surface Coatings or allied materials previously approved by the Professional Grade Committee.
6. Shall normally be sponsored by three Ordinary Members of the Association in the Professional Grade (either

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

Associate or Fellow at least one of whom is a Fellow). A sponsor will usually be a person who has knowledge of the career of the applicant. The candidate shall be in a position to furnish the name of a referee acceptable to the Committee, who can be contacted in confidence, if required. The referee will have a full knowledge of the candidate's technical and scientific achievements to date and could be the applicants employer.

7. 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 Licentiate, 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 increased professional skill and maturity since his election as a Licentiate.
3. Shall have published work which, in the opinion of the Professional Grade Committee, is of a sufficiently high standard OR may be required to submit a thesis or dissertation on a topic previously approved by the Professional Grade Committee OR shall hold the City & Guilds of London Institute Insignia Award.
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.

The nomination of a referee for a *viva voce* examination will normally be for those whose work could be of a highly confidential nature or for overseas candidates.

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.

A sponsor will usually be a person who has knowledge of the career of the candidate. The candidate should be in a position to furnish the name of a referee acceptable to the Committee, who can be contacted in confidence if required. The referee will have a full knowledge of the candidate's technical and scientific achievements to date and could be the applicant's employer.

6. Shall have paid the fee stipulated by 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 Registered Student of the Association for not less than two years.
3. Shall hold the Graduateship of the Royal Society of Chemistry or Council of Physics or a University or Council of National Academic Awards degree recognised by the Royal Society of Chemistry or Institute of Physics as giving full exemption from the Graduateship examination.
4. Shall have attained approved experience in the science or technology of coatings. It is not expected that sufficient experience would be gained in a period of less than two years in the industry. Approved experience may be gained before, during or after the qualifications in paragraph (3) above have been attained.
5. Shall normally be required to satisfy the Professional Grade Committee or some other body approved by the Professional Grade Committee in a *viva voce* examination.

The nomination of a referee for the *viva voce* examination will normally be for those whose work may be of a highly confidential nature or for overseas candidates.

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.

A sponsor will usually be a person who has knowledge of the career of the candidate. The candidate must be in a position to furnish the name of a referee acceptable to the Committee, who can be contacted in confidence if required. The referee will have full knowledge of the candidates technical and scientific achievements and could be the applicant's employer.

7. Shall have paid the fee stipulated by 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, with appropriate responsibility for technical and scientific matters within the company.

It would be helpful if he had contributed to the knowledge and understanding of surface coatings technology by lecturing or by the publication of articles. He will also have shown himself to take a keen interest in the work of the Association by being an active member of his Section and by attendance at lectures etc., whenever and wherever possible.

11. Shall normally be required to satisfy the Professional Grade Committee in *viva voce* examination of his professional competence and also be required to submit a dissertation on a subject agreed by the Committee. In cases where the subject is of a highly confidential nature the use of a referee agreeable to the Committee may be asked to examine the dissertation.
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.

A sponsor will usually be a person who has knowledge of the career of the candidate. The candidate must also be in a position to furnish the name of a referee acceptable to the Committee who can be contacted in confidence, if required. The referee will have a full knowledge of the candidate's technical and scientific achievement and could be the candidate's employer.

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. 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 ten years.
3. Shall have made outstanding contributions to the science and technology of coatings or reached a position of eminence in the industry through the practice thereof.

4. EITHER (a) shall have been an Associate of the Professional Grade for at least eight years and shown continued development.

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

5. Shall submit, with the application, an account of 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, (who should submit a supporting commentary to the Professional Grade Committee).

7. It would be helpful if he had con-

tributed to the knowledge and understanding of surface coatings technology by lecturing or by the publication of articles. He will also have shown himself to take a keen interest in the work of the Association by being an active member of his Section and by attendance at lectures etc., whenever and wherever possible.

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

The fees payable with the application are as follows:

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

Cape, Natal, Transvaal, Wellington, Auckland and Ontario 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.

new members

Ordinary Members

BIRKETT, STEPHEN, 20 Valley Ridge, Kippax, Leeds, West Yorkshire, LS25 7DQ (West Riding)

DENNING, GORDON H, 24 Woodside Avenue, Wrenthorpe, Wakefield, West Yorkshire, WF2 0LG (West Riding)

DEVENDRA RANJ, BSc, MSc, 180/4 Polhensoda Road, Kirillapone, Colombo 5, Sri Lanka (General Overseas)

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

OWEN, DAVID JOHN, Croda Inks Ltd, 170 Glasgow Road, Edinburgh (Scottish - Eastern Branch)

VAN DER WERF, SIERK J., Ing. Chem. Eng., HIM Chemie BV, PO Box 1, 1950 AA Velsen-Noord, The Netherlands (General Overseas)

WOODWARD, ROBERT ALAN, 49 Lime Avenue, Camberley, Surrey (Thames Valley)

Associate Member

SAWERS, ALEXANDER, 96 Menzies Road, Aberdeen AB1 3AP (Scottish)

occa diary

December

Tuesday 1 December

West Riding: "The storage and uses of flammable materials in industry" by D. Gill, assistant chief officer, Fire Service Dept., Birkenshaw. To be held at the Mansion Hotel, Roundhay Park, Leeds 8, commencing 7.30 p.m.

Details are given of Association meetings in the United Kingdom and Ireland up to the end of the third month following publication.

Thursday 3 December

Bristol: "Solubility parameters" by P. Kershaw of Shell Chemicals (UK) Ltd, a joint meeting with the Birmingham Paint, Varnish and Lacquer Club in Birmingham.

Newcastle: "Durability of decorative gloss paints" by E. A. Oakley of BTP Tioxide Ltd at the Students' Common Room, St. Mary's College, Elvet Hill Road, Durham, commencing 6.30 p.m.

Friday 4 December

Irish: "Purchasing and quality control" by D. Donnelly Irish Institute of Purchasing and Materials Management at the Clarence Hotel, Wellington

Quay, Dublin 2, commencing 8.00 p.m.

Monday 7 December

Hull: Second ordinary meeting. D. Wilson of Vinyl Products Ltd will be discussing "Some recent developments in pressure polymer emulsions". to be held at the Duke of Cumberland Hotel, Cottingham, Nr. Hull, commencing 6.45 p.m.

Thursday 10 December

Scottish: "Pigment dispersion" to be given by a lecture from Ciba-Geigy at the Albany Hotel, Glasgow, commencing 6 p.m.

Friday 11 December

Manchester: Lecture "Identifying vehicles for paint transfer" by Dr Wright, NW Forensic Science Laboratories at Manchester Polytechnic, New Administration Building, All Saints commencing 6.30 p.m.

January 1982

January

Midlands - Trent Valley Branch: Joint dinner lecture with Midlands Section. *Details to be announced.*

Thursday 7 January

Newcastle: "Occupational health & safety - corporate and personal commitment and accountability" by F. Riddell of Crown Decorative Products Ltd, to be held at the Students' Common Room, St Mary's College, Elvet Hill Road, Durham, commencing 6.30 p.m.

Monday 11 January

Hull: Third ordinary meeting, F. J. Morpeth of Foscolour Ltd will be talking on the subject of "The manufacture and uses of pigment chips", to be held at the Duke of Cumberland Hotel, Cottingham, Nr. Hull, commencing 6.45 p.m.

Manchester: Lecture "Radiation curing of polymers" by K. O'Hara or M. Holder, Cray Valley Products Ltd, to be held at the Lord Daresbury, Warrington, commencing 6.30 p.m.

Thursday 14 January

Scottish Section - Eastern Branch: Joint Scottish Section/Eastern Branch meeting in the Maybury Roadhouse, Edinburgh, "Mossmorran in relation to the North Sea" by J. R. Alywin of Esso Chemical Ltd, commencing 7.30 p.m.

London: "Optimised TGIC levels in polyester powder" by D. Lawlor, Grilon (UK) Ltd, at the Great Eastern Hotel, Liverpool Street, EC2, commencing 6.30 p.m. Refreshments will be available from 6.00 p.m.

Friday 15 January

Scottish: Annual Dinner Dance, to be held at the St. Andrew Suite, Albany Hotel, Glasgow, commencing 6.45 p.m.

Tuesday 19 January

Bristol: "Colour instrumentation" by D. A. Plant, supported by BOC

(Software Science) Ltd, to be held at the George and Dragon, High Street, Winterbourne, Nr. Bristol, North Avon, commencing 7.15 p.m.

Wednesday 20 January

Manchester: Student lecture "Cellulose esters for liquid inks" by K. Walker, Eastman Chemicals Ltd, to be held at the Manchester Polytechnic, New Administration Building, All Saints, commencing 4.30 p.m.

Thursday 21 January

Midlands: M. Leveté, Paintmakers Association. *Details to be announced.*

Irish: Technical and commercial business forum to be held at the Clarence Hotel, Wellington Quay, Dublin 2, commencing 8.00 p.m.

Thames Valley: "Electron beam curing in the 80s" by P. Cahill of Otto Durr Ltd to be held at Beaconsfield Crest Motel (White Hart), Aylesbury End, Beaconsfield, Bucks, commencing 6.30 p.m. for 7 p.m.

February

Monday 1 February

Hull Section: Fourth ordinary meeting. P. W. Munn, an independent speaker, will be discussing "Paints for concrete". To be held at the Duke of Cumberland Hotel, Cottingham, Nr. Hull, commencing 6.45 p.m.

Tuesday 2 February

West Riding Section: "The financing, organisation and work of the Paint Research Association" by G. de W. Anderson, managing director of the PRA. To be held at the Mansion Hotel, Roundhay Park, Leeds 8, commencing 7.30 p.m.

Thursday 4 February

Newcastle Section: "OCCA past, present and future" by R. H. Hamblin, Director & Secretary of the association. To be held at the Students' Common Room, St. Mary's College, Elvet Hill Road, Durham, commencing 6.30 p.m.

Thursday 11 February

Midlands Section - Trent Valley Branch: "Primers for difficult surfaces" by J. R. Bourne of Mebon Paints Ltd. To be held at the Sutton Centre, High Pavement, Sutton-in-Ashfield, Nottinghamshire, commencing 7.15 p.m.



Scottish Section: "Haloflex for water based paints". A speaker from ICI Ltd. To be held at the Albany Hotel, Glasgow, commencing 6 p.m.

Friday 12 February

Manchester Section: Lecture "Polymers without carbon" by B. R. Currell of Thames Polytechnic. To be held at ICI Ltd, Piccadilly Plaza, commencing 6.30 p.m.

Scottish Section - Eastern Branch: "Burns supper", to be held as usual in the Commodore Hotel, Marine Drive, Edinburgh, commencing 7.30 p.m.

Thames Valley Section: Annual dinner dance to be held at Great Fosters, Egham, Surrey.

Wednesday 17 February

Manchester Section: Student lecture, "Carbon black for paint and printing ink systems" by P. Gallagher, Cabot Carbon Ltd. To be held at Manchester Polytechnic, New Administration Buildings, All Saints, commencing 4.30 p.m.

Thursday 18 February

London Section: "Developments in artists' colours" by P. Staples, technical director of Winsor and Newton Ltd. To be held at the Great Eastern Hotel, Liverpool Street, EC2, commencing 6.30 p.m., refreshments available from 6.00 p.m.

Midlands Section: "Water thinnable epoxies" by A. Schnelle, Emser Werke AG. To be held at the Calthorpe Suite, County Cricket Ground, Edgbaston, Birmingham, commencing 6.30 p.m.

Thames Valley Section: *Details to be announced.*

Friday 19 February

Newcastle Section: Ladies' night at the Five Bridges Hotel, Gateshead. *Details to be announced.*

Friday 26 February

Bristol Section: "Pigments paint and protection" by D. Bishop, British Rail. To be held at the Post House, Cardiff.

Irish Section: Ladies' evening, to be held at the Clarence Hotel, Wellington Quay, Dublin 2, commencing 8.00 p.m.

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