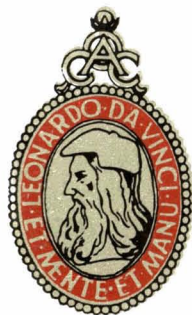




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Aluminium compounds as major components of surface coatings

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Dry-on-wet—A new concept in low pollution coatings utilising cellulose acetate butyrate

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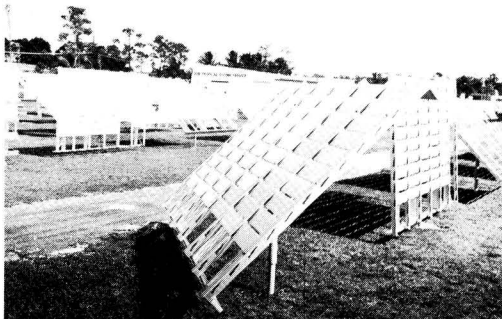
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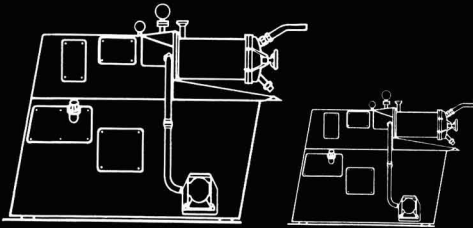
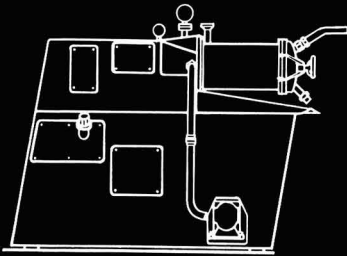
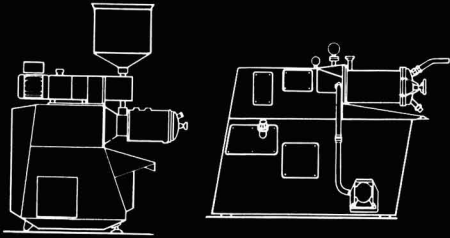
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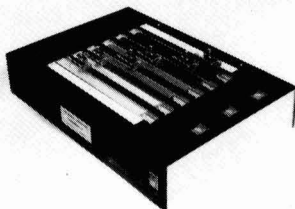
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Contents

Vol. 62 No. 11

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Transactions and Communications

Aluminium compounds as major components of surface coatings	419
<i>J. H. W. Turner and W. K. H. Lakin</i>	
Dry-on-wet—A new concept in low pollution coatings utilising cellulose acetate butyrate	426
<i>B. P. Windibank and R. W. Mahar</i>	
The changing role of polymer chemistry in organic coatings technology	432
<i>M. F. Kooistra</i>	
Scanning electron microscope study of a coating component deposited from solution into wood	441
<i>M. H. Schneider</i>	
<i>Section Proceedings</i>	445
<i>Information Received</i>	447
<i>Notes and News</i>	450
<i>Register of Members</i>	453
<i>Forthcoming Events</i>	453

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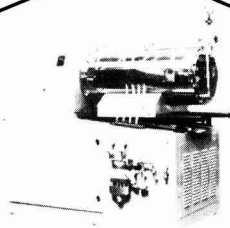
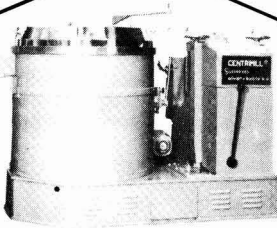
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Transactions and Communications

Aluminium compounds as major components of surface coatings*

By J. H. W. Turner and W. K. H. Lakin

Manchem Ltd., Ashton New Road, Manchester M11 4AT

Summary

The formation of composites of aluminium compounds with alkyd and other resinous media and their properties and potential applications are envisaged. The preferred aluminium compounds are derivatives of aluminium alkoxides in which a proportion of the alkoxide content is replaced by other substituents such as enolate, acyloxyde or phenoxide which are less volatile and more strongly held than the alkoxide radical. The alkyd or other preferred resinous compound is selected to contribute desirable physical properties for the required application and to associate chemically with the aluminium compound to which it is added by reaction with carboxyl, hydroxyl or other reactive groups it may contain.

The reaction product is thought to be a composite in equilibrium with its components which include the volatile alcohol released in its formation. Exposure of the applied film allows this component to escape and be replaced by water from the atmosphere or the substrate which initiates the primary co-ordination drying mechanism. In the case of unsaturated composites, the dried film may be reinforced by oxidation initiated polymerisation.

The properties of the composites are considered and possible applications envisaged.

Keywords

Types and classes of coatings and allied products

alkyd coatings
polyester resins

Raw materials for coatings driers

aluminium complex
lead drier
calcium drier
manganese drier

Properties, characteristics and conditions primarily associated with materials in general

viscosity
compatibility
package stability
coatings during application
drying rate
dried or cured films
hardness
flexibility

Les composés d'aluminium en tant que constituants importants de revêtements de surface

Résumé

On envisage à la fois la mise au point des complexes à base des composés d'aluminium en combinaison avec des alkydes ou d'autres milieux résineux et leurs caractéristiques et applications éventuelles. Les composés d'aluminium de préférence sont les dérivés des alcoolates d'aluminium ou une proportion de la teneur en alcoolate a été remplacée par d'autres substituants, tels que énolate, acyloxyde ou phénolate, qui sont tous moins volatils et plus fortement liés que le radical alkoxyde. On choisit l'alkyde ou d'autre composé résineux préféré en vue des propriétés physiques souhaitées qu'il apporte à l'application requise et de son aptitude à s'associer chimiquement avec le composé d'aluminium, auquel il a été ajouté, par réaction avec carbonyle, hydroxyle ou autres

groupements réactifs qu'il puisse renfermer. On considère que le produit de réaction est un complexe en équilibre avec ses constituants qui comprennent entre autres l'alcool volatil dégagé lors de la formation du complexe. L'exposition du film permet le dégagement de ce constituant-ci et son remplacement par de l'eau en provenance de l'atmosphère ou du support et c'est elle qui déclenche le mécanisme de séchage par coordination primaire. Dans le cas des complexes non saturés, le film sec peut être renforcé par la polymérisation covalente initiée par oxydation.

On considère les caractéristiques des complexes et l'on envisage leurs applications éventuelles.

Aluminiumverbindungen als wesentliche Bestandteile von Beschichtungsmitteln

Zusammenfassung

Die Rezeptierung von Mischungen von Aluminiumverbindungen mit Alkyd- und anderen harzartigen Bindemitteln und deren Eigenschaften und Verwendungsmöglichkeiten werden betrachtet. Die vorzugsweise verwendeten Aluminiumalkoxide, in welchen ein Teil des Alkoxidgehalts durch andere Substituenten, wie Enolat, Acyloxyd oder Phenoxid ersetzt wird, die weniger flüchtig und fester gebunden als die Alkoxidradikale sind. Die Alkyde- oder bevorzugte andere Harzverbindung werden ausgewählt, um gewünschte physikalische, für die beabsichtigte Anwendung erforderliche Eigenschaften zu erhalten, sowie sich chemisch mit der Aluminiumverbindung, zu welcher es zugefügt wird, durch Reaktion mit Karboxyl-, Hydroxyl- oder anderen reaktiven

Gruppen, zu verbinden. Es wird angenommen, dass das Reaktionsprodukt ein im Gleichgewicht mit seinen Komponenten befindliches Mischprodukt ist, einschliesslich dem flüchtigen, bei seiner Bildung freigesetztem Alkohol. Bei der Exponierung des aufgetragenen Films kann sich diese Komponente entfernen und wird durch Wasser aus der Atmosphäre oder dem Substrat, wodurch der primäre Koordinations-Trocknungsmechanismus in Gang gesetzt wird. Wenn die Zumischnungen ungesättigt sind, kann der getrocknete Film durch oxidativ initiierte copolymerisation verstärkt werden.

Die Eigenschaften der Kompositen werden betrachtet, ebenso wie die Möglichkeiten ihrer Verwendung.

Introduction

The paper which the authors presented to the Scarborough Conference in 1975¹ was concerned, primarily, with the reaction of aluminium compounds with stabilised alkyd resins using concentrations of Al (as metal) on alkyd not

exceeding 2 per cent². However, that paper did include a reference to other alkyd composites with a much higher content of aluminium³. Similar alkyd composites with aluminium compounds as predominant components were disclosed in paper by the author (JHWT) to the 1972 FATIPEC Congress in Florence⁴ and on other occasions⁵ but their development

*Paper presented at the Association's Conference held at Stratford upon Avon from 20-23 June 1979.

has tended to be overshadowed and delayed by the priority of effort given to the more conventionally formulated paint media using, mainly, proprietary alkyds of long oil length with lower contents of aluminium. The time now seems to be appropriate to review the composition and properties of composites with a relatively high content of aluminium and to consider their future role in paint technology as coatings which dry, primarily, by the action of moisture and contribute other properties which may be seen as relevant to the requirements of the changing world.

Background

The composition postulated for these composites of aluminium compounds with alkyd resins, for which the name *Alumedium* has been registered, is shown diagrammatically in Fig. 1. To obtain the composite, the alkyd resin, represented by *A*, is dispersed, usually as a solution in a non-reactive diluent such as white spirit or xylene, in a stoichiometric excess of aluminium alkoxide which may be modified by the substituents *X* and *Y* and may also be diluted with non-polar or polar solvents. The postulated reaction mechanism of the alkyd, through its available reactive carboxylic acid groups and the less reactive hydroxyl groups, with the substituted aluminium alkoxide is slightly exothermic and may result in the formation of the aluminium alkoxo acids shown in Fig. 1(b). By using an adequate excess of aluminium alkoxide to saturate the reactive groups of the alkyd, multiple substitution of the aluminium alkoxide by the alkyd is prevented and there is formed what may be an equilibrium mixture of composite solution and reactants which remains stable on storage.

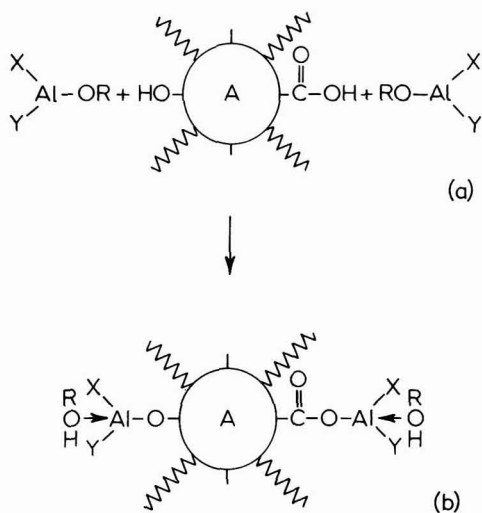


Figure 1. Alumedium reaction

When the composite solution is applied the equilibrium is thought to be disturbed in the manner postulated in Fig. 2. Solvents, which include by-product alcohol, escape from the film and allow the ingress of water from the atmosphere or from the substrate to which the film is applied and cause some of the weaker substituents to be hydrolysed. The low steric impedance of the resulting hydroxyl groups facilitates the formation of the complex co-ordinate gel structure which is well known to be characteristic of basic aluminium

soaps. The final stage of the drying process is the re-reinforcement of the moisture-set film by co-valent polymerisation of the unsaturated drying oil component through the normal cobalt or manganese catalysed oxidation mechanism.

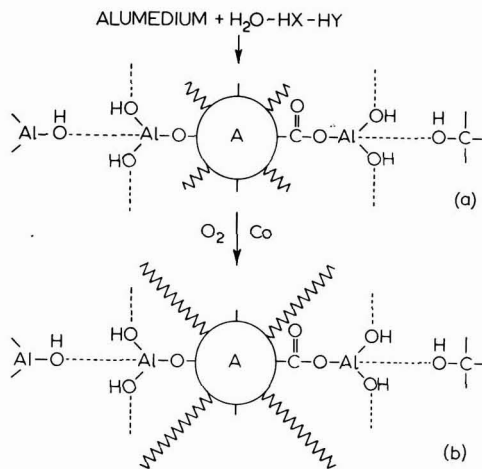


Figure 2. Alumedium drying

No doubt, this is a considerable over simplification of the complex of mechanisms in which activated methylene groups and hydroperoxide groups are also likely to be involved. In the pigmented system, too, the hydroxyl and other active groups which may be available at the pigment interface with the medium are considered to be potential reactants.

Components

The principle components of the composite medium are the aluminium compound and the alkyd resin, the diluent and the drier. Any pigment and/or filler dispersed in the composite medium must also be regarded as potentially reactive components. All contribute to the performance of the composite but not, necessarily, in a strictly additive fashion. Each will be considered in turn.

Aluminium compounds

Aluminium alkoxides, notably the tri-isopropoxide and tri-ethoxyethoxide were used in much of the initial development work but, unmodified, they are excessively sensitive to moisture and often too difficult to control in their reaction with some of the resins with which they may have to be used. It is, generally, desirable to replace one or more of the alkoxide groups by radicals more strongly held by the aluminium atom than are the alkoxides. This has the effect of reducing the functionality and, therefore, the complexing reactivity of the aluminium compound. Besides reducing reactivity, the substituent may provide steric protection and enhance compatibility with other components of the composite. Some useful substituents are exemplified in Table 1, with an indication of their relative bond energies with aluminium, the steric protection they provide and the contribution they make to compatibility with the resin component of the composite. An indication is also given of their relative lability as components of the applied film.

Table 1

Substituent	Al.bond energy	Compatibility	Lability	Steric Protection
Isopropoxide	low	good	v. high	fair
Sec.butoxide	low	good	high	good
Ethoxyethoxide	low	good	high	v. good
Pentachlor phenoxide	medium	v. good	low	v. good
2,6.ditert.butyl	medium	v. good	low	v. good
4.methyl phenoxide				
Ethyl acetoacetate	high	v. good	f. low	v. good
Methacrylate	high	good	f. low	good
Versate	high	f. good	low	v. good
Linoleate	high	v. good	low	fair
Mono isopropyl phthalate	high	good	low	good
Mon sec.butyl maleate	high	good	low	good

Table 2

Contribution of substituent X to performance of $X_nAl(OR)_{3-n}$ *

X =	Contribution to Film Solids %	Drying Rate	Film Properties
Isopropoxide	38	v. fast	hard and brittle
Sec.butoxide	32	fast	hard and brittle
Ethoxyethoxide	27	fast	hard and brittle
Pentachlorphenate	80	medium	hard and brittle
2,6.di.tert.	79	slow	softish
4.methyl phenol			
Ethylacetoacetate	70	medium	hard
Methacrylate	64	medium/fast	hard/brittle
Versate	74	medium	f. hard/tough
Linoleate	81	medium	hard/tough
Mono isopropyl phthalate	76	medium/fast	hard
Mono sec.butyl maleate	73	medium	f. hard

*When $n = 1$ and R is C_3H_7 .

The extent of substitution and the nature of the substituent group determine the properties of the aluminium compound and the contribution it makes to the performance of the composite. It is, usually, desirable to retain at least one alkoxide substituent to facilitate reaction with polar groups in the resin component. The other alkoxide groups may be replaced by one or more substituents to achieve the combination of properties desired. Table 2 indicates the type of contribution expected from each substituent but, to a considerable extent, this must also depend on the resin with which it is used.

Primary aliphatic substituents, whether they be alkoxide or acyloxy, are sterically less protective than branched substituents and are, therefore, less effective as stabilising components. Thus, for example, the methacrylate confers greater storage stability than the propionate substituent; and the α,α' dialkyl substituted versatic acid confers much greater protection than the longer chain, but unbranched, linoleic acid. The structure of the aromatic substituents makes them relatively more effective than the primary aliphatics, especially when they are substituted in the ortho position. Some steric impedence is a desirable means of enhancing composite stability, but too much may obstruct the reaction of the aluminium compound with the alkyd resin. For example, the screening effect of the versate radicle in aluminium monoethoxyethoxide diversate is sufficient seriously to impede its forming a homogeneous composite with some alkyd resins at ambient temperature.

The application of heat is usually sufficient to reduce the steric barrier to reaction, but it is preferable to employ a less protective combination of substituents.

Apart from the contribution they make to the storage stability of the composite and its susceptibility to hydrolytic action, the substituents can fulfil a number of other functional roles. Some, like the stearate and versate, increase water resistance and reduce water vapour permeability; others, such as the non-ionic surfactant polyethylene glycol half ethers and esters may be incorporated to increase water absorption and facilitate water vapour transmission through the applied film. Unsaturated substituents, such as methacrylate, half maleate esters and linoleate introduce a copolymerisation potential with the unsaturated fatty acylate component of the alkyd. Phenates, such as 2,6 ditert.butyl 4 methyl phenate may function both as plasticiser and antioxidant; other phenates such as pentachlorphenate and orthophenyl phenate contribute fungicidal activity. And there are many other functions which appropriate substituents can fulfil.

Alkyd resins

The use of alkyd resins with the more reactive aluminium alkoxides and their monosubstituted derivatives of the type considered in the Scarborough paper, is restricted by considerations of storage stability to those of long oil length and low reactivity, or to alkyds of shorter oil length and

greater reactivity, which have been reduced in their reactivity with aluminium compounds by reducing their content of available carboxyl groups as, for example, by post reaction with oxirane compounds.

This limitation need not only apply when the functionality of the aluminium compound is reduced by appropriate substituents and there is no upper limit to the proportion of aluminium compound to alkyd which can be used. The critical factor is the performance of the composite as a coating and this will depend both upon the composition of the aluminium compound and of the alkyd used with it.

The use of aluminium compounds with alkyd resins inevitably results in additional cross linking, which usually causes the applied film to be harder and less flexible. This may or may not be offset sufficiently by the contribution of the substituents as plasticising or softening agents. There is, therefore, the probability that the preferred alkyd resin for use in these composites will be designed to contribute more flexibility and less cross linking through co-valent drying oil polymerisation. This might be achieved by a more linear polyester composition with a lower content of oxidisable and polymerisable fatty acylate. Suitable polyesters might use polyols, such as trimethylol propane, glycerol and even glycols and polyglycols to replace higher polyols, such as pentaerythritol. They might also use some aliphatic dibasic acids instead of the usual content of phthalic acid.

Some conventional, proprietary alkyds, particularly those of medium to short oil length which are excessively reactive and may lack compatibility with the preferred aluminium compound, can be modified by reaction with oxirane compounds, such as phenyl glycidyl ether and glycidyl versate and thereby converted into useful resins for some composite applications. However, it seems unlikely that remedial treatment of this sort will generally be justifiable as a practical alternative to the direct reformulations already suggested.

Diluent

The role of the diluent in aluminium/alkyd composite media is to ensure the solubility of the components and provide a solution appropriate in non-volatile content and viscosity for the required application. But unlike the diluent for some conventional finishes, it has other important functions. The diluent may be volatile or non-volatile, is usually of low polarity, but containing some alcohol by-product from the composite reaction which has a significant peptising role and, in addition, may facilitate the ingress of the water needed for the hydrolytic film gelling mechanism.

Potential non-volatile diluents include liquids or resinous solids which are compatible with the composite, but do not react with the aluminium component. However, there may be a latent potential reactivity with the composite which may be activated, after application, by the drying process. Such non-volatile solvents include liquid and solid hydrocarbons, such as olefinic polymers; chlorinated paraffin wax; and esters, both saturated and unsaturated, which may include dioctyl phthalate, maleic and fumaric esters, higher vinyl esters and fatty esters.

These non-volatile diluents contribute a number of apparent advantages which have not yet been evaluated fully. By remaining in the film as non-volatile components they contribute to ease of application and increased film build. Through their presence in the gel matrix they appear to enhance the drying process, possibly by reducing steric impedence

and, if unsaturated, by participating directly in the drying mechanism as copolymerisable monomers. There is also evidence of a plasticising action which counteracts the hardening action of aluminium compounds when used with conventional alkyds. This may contribute some improvement in durability when the composite is applied to dimensionally unstable substrates, such as wood.

The limited work so far undertaken indicates that the probable optimum content of non-volatile solvent is between 10 and 20 per cent of the total binder weight.

Driers

The accelerated weathering of films of pigmented composites and of corresponding paints using the same alkyd resins with conventional driers has shown that film deterioration, notably loss of gloss, is aggravated by conventional driers and is least when all catalytic driers are excluded from the coating. Lead and calcium have proved to be the most damaging driers. The effect of manganese and cobalt is less damaging and there is consistent, but limited, evidence that of these the manganese is less damaging than cobalt. The action of conventional driers has been particularly apparent in expediting the onset of chalking of paints containing titanium pigments.

The rate of drying of aluminium composites is dependent on the type of alkyd it contains and on the substituent groups present in the aluminium compound used with it. With composites based on the more reactive medium and short oil length alkyds, the inclusion of any conventional drier may be unnecessary. With the longer oil length alkyds, forming composites of low viscosity and high solids, some addition of conventional drier is likely to be needed to reduce tack and increase hardness within the required time limits. With such composites, recent work has shown manganese to be no less efficient than cobalt. The reason for this is not yet understood. But it is possible that the hardening of composites is dependent more on the formation of groups, such as hydroperoxides, capable of reacting directly with the aluminium, than it is with free radicles being formed to initiate the conventional co-valent polymerisation processes of the drying oil component.

Pigmentation

Many pigments and fillers contain adsorbed water and have active hydroxyl groups available at their interface with the medium in which they are dispersed. If the medium is an aluminium/alkyd composite there is the risk that hydrolytic action by this water could cause instability and structural changes.

This risk can be eliminated by drying the pigment. Or it may be contained by dispersing the pigment in a solvent, such as white spirit, incorporating a suitable excess of aluminium alkoxide derived water scavenger. The wetting action of the aluminium compound for the wet pigment is soon apparent and there is often a substantial improvement in the rate of deflocculation and in the degree of dispersion.

The potential reactivity of the aluminium compound with the pigment surface and with the alkyd may account for the apparent increased contribution by the pigment to the performance of paints based on composites. There is extensive evidence of improved colour and opacity and of reduced chalking when titanium pigments, dispersed in composites, are subjected to weathering. But so far there has been no

serious attempt to quantify other effects which may be attributable to enhanced pigment/medium association.

Composition/property relationships

By utilising all the possible combinations of aluminium compounds with drying oils and alkyd resins of all types, it should not prove impossible to provide composites with some properties relevant to most coating, printing and related applications. The scope of this paper is too broad to consider in detail which compositions are most appropriate to which applications. But a few examples may illustrate the more obvious composition/property relationships and provide a pointer to formulations conferring specific properties.

The chosen examples are of composites in which aluminium compounds of varying composition are used with alkyds of long, medium and short oil length. This selection excludes a number of potentially important components, such as epoxy esters which have already established their value as coatings. Nevertheless, they should indicate the implications of the chemistry and technology involved, which others with a more detailed understanding of coatings technology could apply to a wider range of composition and performance requirements.

Long oil compositions

When a polymerised drying oil, such as a 35 poise linseed stand oil, is added to one of the more reactive aluminium alkoxides, the composite formed may be unstable, especially at low aluminium contents. If the reactivity of the aluminium component is decreased by substitution and the Al content of the composite increased, it becomes more stable and, with the usual addition of cobalt, dries well to a film showing improved colour, water resistance and gloss retention when compared with a film of the same oil, but containing only the conventional combination of cobalt and lead driers. In mechanical properties, the dried film of the stand oil composite

is generally inferior to alkyd composites of similar aluminium content, although the use of certain substituents can upgrade it considerably.

The better performance of composites based on long oil alkyds may reflect their intrinsic superiority as coating materials. It may also be attributable to the more stable and compatible composite formed by the reaction of the aluminium compound with the carboxylic acid and hydroxyl groups available in the alkyd. The results of reaction between the long oil alkyd and aluminium compound which are most noticeable are the greatly improved through drying and reduced surface tack. But a number of other changes become apparent in due course. The applied film has much greater resistance to water, better gloss retention and colour stability. If the aluminium component has versatate or fatty acylate substituents which contribute high solids and low viscosity, the composite with a long oil alkyd, also of high solids and low viscosity, will combine these qualities of high build and low solvent loss with very good through drying and durability.

Proprietary alkyds of lower oil length in the range 60/70 per cent will usually be more viscous, more reactive and require higher contents of more substituted aluminium compounds to provide composites of satisfactory stability. Table 3 shows the effect on solids content, viscosity, storage stability and drying rate of using a di-substituted aluminium compound with a well known and widely used proprietary alkyd of 68 per cent oil length. The stability of formulation No. 3 may be considered to be the minimum acceptable for industrial or decorative coatings. These composites were unpigmented and it is possible that pigmentation would have reduced their stability further. On the other hand, storage tests over a period of 5 years have shown that pigmented composites employing the same proprietary alkyd post treated with an oxirane compound to reduce its reactivity remain as stable as the paints employing the same alkyd with conventional driers and no aluminium compound.

*Table 3
Effect of Al content of composite on stability and drying performance*

Components and composition	Al.C:	1	2	3	4	5	RS 50
Aluminium Compound Al.C.	100	100	100	100	100	100	--
Alkyd Sol.RS/50	—	100	200	300	400	600	100
Alkyd Solids %	0	25	33	37.5	40	42.9	50
Al.content %	5.3	2.7	1.8	1.3	1.1	0.8	0
Al/Alkyd %		10.6	5.3	3.5	2.7	1.8	0
Total Solids %	68.2	60	57	55	54	52.6	50
Viscosity poises							
30.7.75	2.5	0.5	0.5	0.8	1.0	1.4	1.5
2.8.75	2.5	0.6	0.7	1.1	1.4	3.2	2.0
7.6.76	2.7	0.6	1.0	1.5	2.7	15	2.2
2.2.79	3.0	0.7	1.3	1.6	3.4	gelled	2.9
Drying Time (Hrs.) (Beck Koller)							
Stage 1	1.7	1.1	1.1	1.0	0.6	0.5	1.2
2	6.9	1.9	2.5	2.6	2.1	1.6	1.8
3	9.6	3.3	4.0	4.2	3.6	4.2	10.4
4	17.3	7.2		4.6	4.3	4.8	14.2

Aluminium compound Al.C	%	Alkyd resin solution RS/50
Al	5.3	Type—Linseed, P.E. P.A.
Alkoxide	11.8	O.L.—68%
Substituent X enol	25.6	A.V.—10
Substituent Y acylate	34.4	OH Value—38
Diluent	23.0	Co (on alkyd solids) 0.06%, White Spirit—50%

The better drying of the composite reflects the different drying mechanism, which is thought to involve additional cross linking by co-ordinate bonds. However, excessive cross linking can reduce film flexibility, impair its ability to adapt to dimensional changes in the substrate and cause its premature failing as a protective coating.

Another property not revealed in the table is the greatly enhanced water resistance of the composite film. This may result in the absorption of water by the immersed film of composite being reduced from the normal saturation level of an alkyd which may be as high as 70 per cent to 130 per cent of the dry film weight to no more than 10 per cent of the dry film weight. This again is a great advantage in such applications as anti-corrosive metal coatings, marine topside paints and many other industrial applications. On the other hand, for application to wood, coatings with low water absorption and low water vapour permeability often lack the flexibility to adjust to dimensional change in the substrate and may prove too great a barrier to the escape of water vapour from the substrate through the film. For such an application a composite with a too low water absorption and high impermeability to water vapour would be at a disadvantage. Fortunately, these particular requirements can be satisfied by replacing a hydrophobic substituent, such as the versatate radicle, by one which is more hydrophilic, such as a polyglycol half ether. This ability to adjust composite properties by a simple compositional change could be one of the more important assets of this type of coating composition.

Table 3 shows that although the composite has a higher effective binder content than the corresponding unmodified alkyd medium, it is low compared with the composites based on the low viscosity long oil alkyds which can have binder contents of the order of 75 to 85 per cent. The diluent white spirit is relatively cheap but ultimately, the cost of no waste product polluting the atmosphere can be justified if it can be replaced by another product with a more positive contribution to make. In fact, part of the white spirit content of these composites can be replaced by one or other of the non-volatile diluents mentioned earlier. For example, dioctyl maleate, used at about 15 per cent of the total binder weight, can replace an equal weight of white spirit and improve the performance of the composite in application, drying, build and flexibility.

Medium oil compositions

With alkyds of lower oil length, the viscosity and the polarity increase, solubility and compatibility become more critical and a higher proportion of the substituted aluminium compound, as much as 6 to 9 per cent, is needed to ensure storage stability. The applied film is highly dependent on moisture activated co-ordination as the primary drying mechanism. This is subsequently re-inforced by normal oxidative polymerisation through the small content of unsaturated fatty acylate the composite contains.

Some interesting compositions have been based on proprietary medium oil length alkyds dilutable with white spirit. Used with sufficient di-substituted aluminium compound to provide a minimum of 6 per cent Al/alkyd, it is possible to obtain composite media with much higher binder content than the alkyd alone could provide. The composite medium takes pigments well and, on application, the paint air-dries quite rapidly or can be flash dried at 70 to 100 C to provide hard, tough and adherent coatings, which appear to merit investigation and development for a number of quick setting, energy conserving industrial applications.

More linear polyesters might be used with, or instead of, the conventionally formulated medium oil alkyd, to contribute greater flexibility to the composite film. There is also the possibility of using reactive diluents which, in certain circumstances, such as curtain coating applications, might include styrene. This is an area of great interest and potential achievement in which the mechanisms of co-ordinate and co-valent cross linking might be integrated in a synergistic fashion.

Superficially, there appears to be some similarity between the hardening role of the aluminium compound in the composite and the contribution made by amino resins to the hardness of some baking finishes. But the resemblance is limited and the operative mechanisms of the two compounds are widely different. Here, too, the possibility of combining the two polymerisation mechanisms may merit examination.

Short oil compositions

When aluminium alkoxides are exposed to moist air they "skin" at once and on prolonged exposure the mass of alkoxide is converted into non-cohesive powder. Substituted alkoxides such as Al.C. (Table 3) react with moisture relatively slowly and form films which are more cohesive, but still inadequate in strength for most applications. It is possible to obtain a useful compromise between setting rate and strength of the applied film by using partially substituted, but moisture sensitive, aluminium compounds with minor amounts of compatible alkyds of high functionality and short oil length. Separately and combined the components are usually soluble in alcohols or mixtures of alcohols with hydrocarbon solvents.

The rapid, but controlled, moisture setting of these composites is useful in a number of coating and printing applications. Perhaps the most obvious potential is in low viscosity inks which are dried by contact of the composite with moisture in the paper printed. The same reaction can be employed in paper coating, veneer and chip board sealing and, perhaps, in some wood finishes.

Composites of this type looked promising as binders for sand in the formation of foundry cores and moulds for metal casting. Cold cured moulds of more than adequate strength were produced, but the heat resistance of the alkyd modified composite was not sufficient to prevent some "spalling" during the metal casting process. Similar composites have been considered as binders for road marking paints and in this application they show greater promise.

Conclusion

Since the time of the cave paintings, paintmakers as artists, as craftsmen and now, as scientists and technologists forming a sizeable part of the chemical industry of the world, have found or been given many new products and tools to use. It is right that the craftsman should not, too readily, abandon his skills and turn to new things. And, certainly, change in the paint industry was slow enough until well into this century. Since then, things have happened more quickly, and it would be understandable if the paintmakers of today were bewildered by the changes they have seen and, eventually, adopted. The evolutionary pattern continues to develop under pressure from ecological considerations, diminishing natural resources, and the more critical utilisation of available energy. Aluminium compounds and others with new and useful properties to contribute may be seen as part of this pattern.

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Discussion at Stratford Conference

MR R. H. WALLINGTON commented that the authors had stressed the use of accelerated weathering techniques for assessing the durability of the coatings containing alumed, but work at both the PRA and MQAD, Woolwich, had indicated that results of accelerated weathering techniques were not always borne out on natural exposure. He asked if the authors would like to comment on this.

MR TURNER affirmed that only accelerated weathering tests had so far been carried out, but stressed that at this stage the work was really only a feasibility study rather than product development tests. It had, however, been confirmed that at the lower levels of aluminium concentration there was difficulty obtaining a relationship between accelerated and natural weathering. He said that accelerated weathering had been used as a method of screening a large number of experimental products, but they were aware of the need for all types of natural weathering tests in the full assessment of the products. He emphasised the importance of the crosslinking in designing media for gelation by co-ordination, as well gelation by polymerisation, which he hoped would lead quickly to more linear structures.

MR LAKIN added that the difference between natural and accelerated weathering related to the behavioural performance which was not only related to the aluminium compounds. Some of the properties which are extremely important to the surface coatings industry, such as gloss, freedom from chalking, whiteness and colour retention are shown during both accelerated and natural weathering.

MR T. R. BULLETT added that one of the problems with this type of material was that after the initial curing, some oxidative crosslinking continued to occur resulting in a film with too much crosslinking which then became subject to cracking, especially on substrates such as wood.

MR D. A. KALWIG asked the lecturer to comment on the cost/benefits of the use of aluminium alkoxides in the light of increasing raw materials costs and shortages. He asked if such chemical mechanisms could enable the industry to use materials of previously unacceptable grades.

MR TURNER said that this was really a commercial question that he was unable to answer completely, but one of the factors they had in mind when introducing this new type of technology was to make available to the industry materials which have a low cost, but are not usable with normal techniques. The materials they had produced were of comparable quality to conventional systems, and the new technology might certainly help in the future to alleviate shortages of some traditional raw materials.

MR LAKIN stated that it was very difficult to predict costs in new areas, but the general trend was towards high labour costs and so a naturally occurring source of raw materials, whether from oil or coal based products, was important, and the crosslinking process provided by metals, such as aluminium, was perhaps one way in which the industry could provide coatings that are both ecologically and economically acceptable.

MR R. L. J. MORRIS referred to the effect of water on the curing mechanism of the aluminium co-ordinated media. In view of the apparent sensitivity of the rate of crosslinking to moisture content, he asked whether it would be possible to design systems which could be applied in outdoor conditions.

MR TURNER stated that in most cases this was possible, although in some cases restrictions on drying rate would be necessary to prevent unduly rapid crosslinking by formation of a gel before much of the volatile component had been released. In an early experiment, a product had been airless sprayed in an ordinary atmosphere and the product gelled between the time it left the gun and when it hit the surface, enabling a coating of gel to be built up equivalent to four or five coats in one application. The problem had been that it was not then possible to release all the solvent wanted before the gel formed, which produced problems of stress cracking. Normally it is possible to control the resistance to hydrolysis, so that the ingress of moisture is slow enough to allow film formation at a rate which allows the required amount of solvent to escape. This is often easier to achieve with films of a high solids content.

"Dry-on-wet"—A new concept in low pollution coatings utilising cellulose acetate butyrate*

By B. P. Windibank and R. W. Mahar

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Summary

The paper describes a novel method of producing low pollution coatings, whereby a clear powder coating is applied over a thin, pigmented, solvent-containing base coat, the two being stoved together without any form of intermediate stoving. The system overcomes the twin major deficiencies of powder coating, of inability to make rapid colour changes and to produce attractive metallic finishes. Furthermore, the dry-on-wet system is a marriage of two existing and well-proven technologies and unlike some

of the alternative proposals for low polluting coatings, does not require expensive and largely untried equipment or use toxic ingredients.

Formulation, application and physical property details of the proposed system are given in the paper, together with the advantages over other low polluting forms of coating.

Keywords

Types and classes of coatings and allied products
metallic pigmented paint
powder coating

Raw materials for coatings
binders (resins, etc.)
cellulose acetate butyrate

Process and methods primarily associated with application of coatings and allied products
dry-on-wet

Miscellaneous terms
energy requirement
effluent
investment cost
pollution

"Sec-sur-frais"—Un nouveau concept dans le domaine de revêtements à faible teneur en matière polluante

Résumé

On décrit dans cet exposé une nouvelle méthode de production des revêtements à faible teneur en matière polluante, et qui comprend l'application d'une couche de revêtement en poudre non pigmenté sur une couche mince de peinture de base, contenant pigment et solvant, l'ensemble étant cuit au four sans aucun étuvage intermédiaire. Le procédé élimine les deux défauts principaux des revêtements en poudre, à savoir, l'impossibilité de faire rapidement des changements de couleur et de produire des finitions métalliques attrayantes. D'ailleurs le système "sec-sur-frais" est une fusion de deux techniques fiables, déjà en vigueur et contrairement à

certaines propositions alternatives pour mettre au point des revêtements à faible teneur en matière polluante, il n'exige pas de matériel et coûteux et en grande partie non essayé, ni de constituants toxiques.

Dans cet exposé on donne des détails sur la formulation, l'application et les caractéristiques physiques du système proposé, ainsi que les avantages qu'il possède par rapport à d'autres types de revêtements à faible teneur en matière polluante.

"Trocken auf Nass"—Ein neue Art von Anstrichen, auf der Basis von Celluloseacetatbutyrat mit niedriger Umwelt belastung

Zusammenfassung

Die Arbeit beschreibt eine neuartige Methode von niedriger Pollution verursachenden Anstrichen, bei welcher ein klarer Pulverlack über eine dünne Schicht pigmentierten, Lösungsmittel haltigen Grundlackes aufgebracht wird, und beide zusammen ofengetrocknet werden ohne irgendwelche dazwischenliegende künstliche Trocknung. Dieses System bewältigt die zweifachen Hauptmängel der Pulverlackierung, nämlich, die Unmöglichkeit schnelle Farbtonwechsel vorzunehmen und attraktive Metalllackierungen zu erzeugen. Ausserdem ist das "Trocken auf Nass" System eine Vereinigung zweier bestehender und wohlbewährter Technologien

und im Gegensatz zu einigen der anderen Vorschläge für Anstrichmittel niedriger Pollution, erfordert es keine teure und grossenteils unerprobte Ausrüstung oder Benutzung giftiger Bestandteile.

Es werden Angaben hinsichtlich Rezeptierung, Anwendung und physikalischer Eigenschaften des vorgeschlagenen Systems gemacht, ebenso über die Vorteile gegenüber anderen Arten von Anstrichsystemen niedriger Pollution.

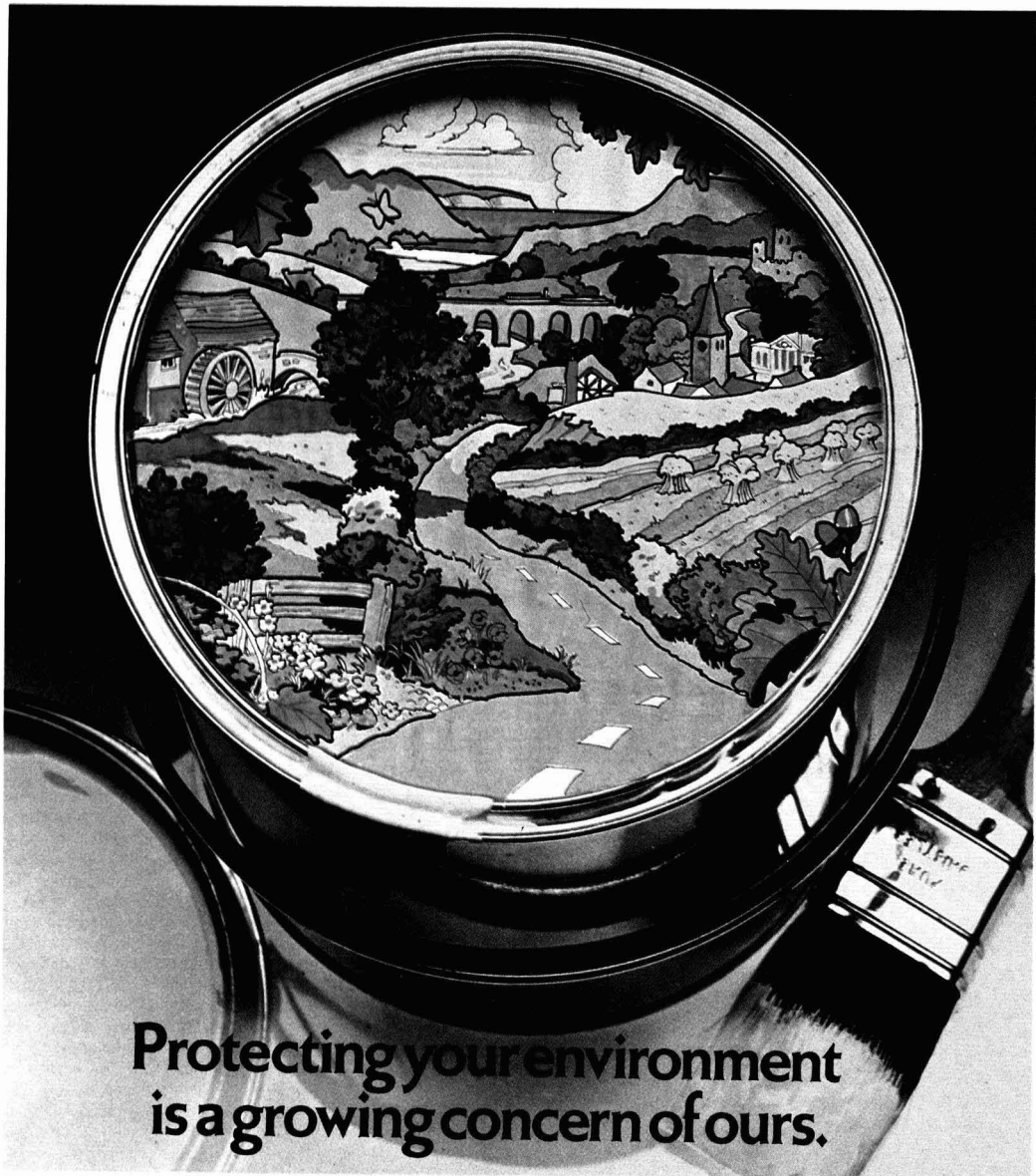
Introduction

In recent years the coatings industry has come under increasing pressure to restrict its use of solvents. This pressure is twofold, being brought about by ever increasing solvent costs and legislation designed to protect the environment. It is predicted that solvent costs will continue to increase, whilst strong environmental protection laws, at present in force in Germany, Holland and some parts of the USA, will be adopted in other countries, particularly those in the EEC.

The coatings industry has made, and is continuing to make, strenuous efforts to meet these new requirements by developing new coatings systems. Each of these systems has its own advantages and deficiencies, resulting in different systems being used for different end-use applications. Currently there is no new single system suitable for the majority of coating applications (as solvent based coatings are), with the result that the industry is tending to become fragmented.

A list of the most important types of coating, both existing

*Paper presented at the Association's Conference held at Stratford upon Avon from 20-23 June 1979.



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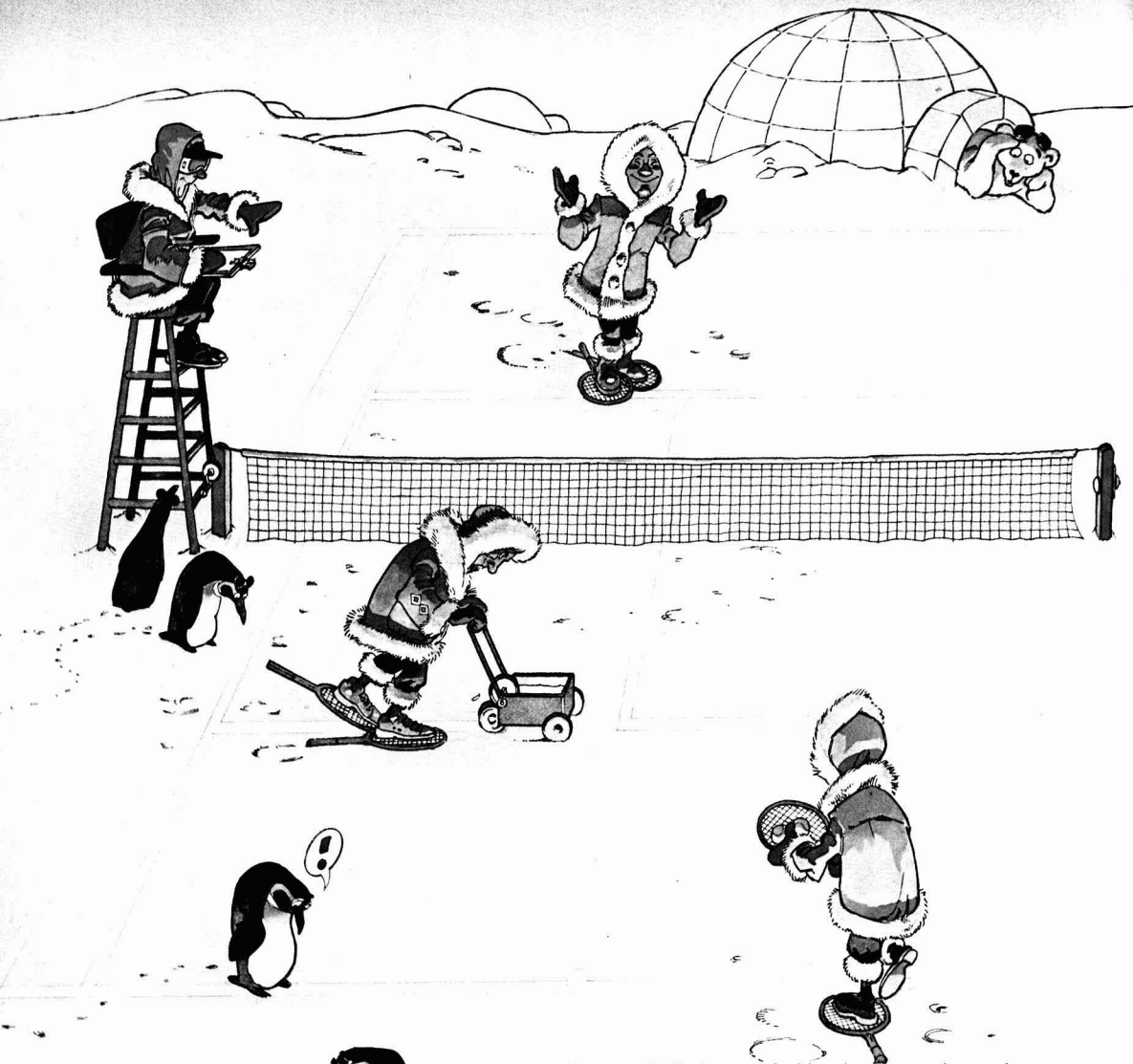
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Table 1

Comparison of existing and projected coating systems

System	Advantages	Disadvantages
Conventional solvent	Established technology	Environmental pollution, cost of solvent, high energy requirements
Water-borne (excluding electrodeposition)	Water is inexpensive and ecologically desirable	Pollution of air and water by solubilising amine, high energy requirements (but less than for conventional solvent systems), high capital expense, very sensitive to substrate cleanliness
Powder	Solventless, high utilisation efficiency, low energy consumption, tough coatings resistant to handling damage	Difficult and expensive to carry out rapid colour changes, cannot produce satisfactory metallic finishes, high capital expenditure
High solids (single pack)	Reduced solvent usage and emission, can be used on existing spray equipment, low energy consumption, extension of existing technology	Sticky nature prevents use on disc equipment, reduced electrostatic properties, low level of coating properties, sticky overspray builds up on extraction equipment, high level of dirt pick-up
High solids (two pack)	Very low energy consumption, reduced solvent usage and emission, high "build" enables number of coats to be reduced	High cost and toxicity of isocyanate components, metallic finishes limited to <50% volume solids, high cost of sophisticated application equipment
UV Curing	Low energy consumption, solventless, high cure speed	Pigmented coatings thicker than 25µm cannot be cured, limited to flat stock and simple shapes, toxicity problems of acrylate types

and under development, is given in Table 1, together with the major advantages in each case.

One new coating system not listed in the table, because it is only a variation on conventional solvent types, is the "wet on wet" technique, also known as base coat/clear or clear over base. This method of application is used exclusively in the automobile industry for the preparation of metallic finishes and is gaining increasing acceptance, principally because of its superior appearance compared with conventional single coat metallics.

It can be seen from Table 1 that, if the elimination of solvent were to be the only criterion in the selection of a new coating system, then powder coatings would be the obvious choice for many, or even perhaps most, applications. However, this disregards the two major disadvantages of powder coating, *ie* (a) the difficulty and expense of carrying out rapid colour changes on a multi-colour product line, such as automobile production and (b) the lack of satisfactory metallic finish powders. For these reasons the application of powder coatings has to date principally been in single colour, long run products, such as domestic appliances. However, work carried out in the Eastman Kodak Coatings Chemicals Research Laboratory, Kirkby, has demonstrated that it is possible to combine the desirable features of powder coatings with those of conventional solvent based systems to produce a coating having most of the advantages of both. This system has been called "dry-on-wet". The present paper describes the formulation and application of the "dry-on-wet" system, and highlights the advantages and opportunities for the system relative to existing compositions.

The dry-on-wet principle

The dry-on-wet system involves the application to a pre-treated metal substrate of a thin metallic or straight-shade base coat, followed immediately by a clear powder top coat without any intermediate stoving. The two coatings are then stoved according to the cure requirements of the powder.

As well as having the advantages of powder coatings listed in Table 1, there are two very important additional

benefits in using the new system:

- Only a clear powder coating is required, eliminating the need for the expensive colour changing equipment, together with all the problems encountered when different coloured powders are to be used.
- Attractive metallic finishes can be produced, comparable with those obtained when using a wet-on-wet system, thus opening up the automobile top coat market to powder coatings.

The key to satisfactory operation of the dry-on wet system lies in correct formulation of the base coat and linking this with the fusion properties of the powder. It is of critical importance that the bulk of the solvent in the base should be removed before application of the powder. Extensive work has shown that cellulose acetate butyrate (CAB) is uniquely suited to this requirement.

In the metallic base coat, for example, CAB retains its open structure and facilitates rapid removal of the solvent, at the same time 'fixing' the aluminium flake in the base coat in the correct orientation (parallel to the substrate) and, assuming the formulation to be correct, preventing the film and flake from sagging.

Method of application

For industrial application the base coat is best applied using a high spinning disc, such as the Sames Gigastatron, or by using air assisted electrostatic spray equipment, such as the De Vilbiss AGC High Voltage spray gun. Conventional spinning disc electrostatic spray equipment, such as the Ransburg type, can also be used provided satisfactory atomisation can be achieved.

With all the above types of application the base coat is touch dry when it emerges from the spray booth and is ready for application of the clear powder, followed by stoving at the normal conditions required for the powder.

In laboratory application the base coat is sprayed on in thin layers (allowing time for the solvent to evaporate from

each layer before addition of the next), until the base coat thickness reaches 15–20 μ m (ideally this is achieved by a low throughput of liquid sprayed at a high air pressure ~ 55–60 psi).

The total spraying and drying time to achieve a tack-free, smooth base is of the order of 3 minutes with a solvent system based on butyl acetate/xylene solvent. Straight-shades are a little slower due to the need to use a less volatile solvent system. A smooth finish on the base coat is important if the optimum appearance of the powder top coat is to be achieved.

Immediately the base coat is dry, the clear powder coating is sprayed on electrostatically to a thickness of ~ 50 μ m and the two coatings are then stoved at the curing temperature of the powder.

Types of material used

Base coats

It has been possible to produce satisfactory results utilising both thermoplastic and thermosetting resin types in the base coat, although thermoset materials have generally been preferred, because of their better intercoat adhesion properties.

Top coats

Good results have been achieved with both polyester and acrylic powder top coats, although the best finish to date has been achieved by the use of polyurethane powder which consistently gave less orange peel.

Selection of components for base coat

Cellulose acetate butyrate

Compatibility is an important consideration in the system. It is desirable to maintain compatibility in the base coat to improve adhesion of the base coat to both the substrate and to the powder top-coat.

All resins and cross-linkers used in combination with CAB should be checked for compatibility prior to use, by applying the unpigmented three-component system on to a glass plate, air drying and then stoving under normal conditions. The cured film is then examined for clarity, any opacity or "miliness" being indicative of incompatibility. For dry-on-wet coatings, the system should be fully compatible or show only very slight incompatibility.

CAB 551-0-2 and CAB 531-1 have been found to be suitable for use with polyester and acrylic/melamine systems, principally because of their good compatibility. Because of its lower inherent viscosity, which enables a higher solids level of the base coat to be maintained, CAB 551-0-2 is preferred.

If the base resin being used is of a low viscosity type, then it may be necessary to balance this with a higher viscosity grade of cellulose acetate butyrate in order to obtain a satisfactory dry surface on which to apply the clear powder. CAB 381-2 has been used satisfactorily in such a system.

Resin

Several resins have been used. The best results, however, have been obtained from a hydroxy-substituted acrylic, Synedol 2263XB, from Synres BV. Acrylics, in general, have better

compatibility with CAB than alkyds and this particular resin has been found to be most suitable. The thermoplastic types produced were based on a Rohm and Haas acrylic resin, Paraloid B-66.

Cross-linking agent

The selection of this component requires considerable care as it must have good compatibility in the three-component system, CAB/resin/cross-linker, as well as imparting good flow properties and retaining good reactivity at the cure temperature of the powder (160–180 C), as well as being thermally stable at this temperature.

It also contributes towards achieving satisfactory intercoat adhesion. In the case of some polyester top coats it is found that a ratio of resin to cross-linker of 80:20 is needed in the base coat to achieve satisfactory intercoat adhesion, whilst with polyurethane top coats a ratio of only 90:10 may suffice.

Pigment

Metallic coatings

Two types of non-leafing, acid resistant aluminium flake have been used, these are:

- (a) Sparkle Silver (Silberline (UK) Ltd)
- (b) Stapa Mobilux (Eckart-Werke (BRD))

These metallic flakes have also been used in combination with coloured pigment *eg* phthalocyanines and transparent iron oxides to give further decorative effects.

Straight-shade coatings

At present, only a white base coat containing titanium dioxide has been made, essentially in order to establish the feasibility of this system. A variety of pigments supplied by BTP Tioxide Ltd, and Kronos Titanium Pigments Ltd, have been used without any significant differences being noted.

Here, the problem is one of achieving satisfactory hiding power in a thin base coat without needing too long a time for evaporation. In practice, slower evaporating solvents are required resulting in a slight lengthening of the spraying and flash-off time compared with that required for metallic coatings. It may, however, be necessary to increase the base coat thickness to 20–25 μ m to obtain satisfactory hiding.

Solvent system

Metallic coatings

A butyl acetate/xylene mixture in the ratio 70/30 is normally used and this gives satisfactory flow-out and simultaneous rapid evaporation; however, the solvent blend can be varied to adjust the rate of evaporation.

Straight-shade coatings

These may necessitate a higher boiling solvent blend to achieve satisfactory flow-out, whilst maintaining a satisfactory evaporation rate without sacrificing hiding power.

Powder top coat

All the early work on the dry-on-wet system was carried out using commercial clear powder coatings. However, more recently, specially formulated powders have been used and examples of these are given in the Appendix. A study of the

flow and curing characteristics has shown the following:

- (i) Powders should have low melt viscosity in order to obtain a low level of orange peel in the 45–50 μ m thick coatings
- (ii) The softening point of the powder should be as high as possible, consistent with (i) above, to prevent trapping of volatiles from the cross-linking reaction in the base coat
- (iii) The cross-linking reaction should preferably be of the delayed action type to allow maximum time for flow out and the escape of base coat volatiles.

Benefits of the dry-on-wet systems

Reduction in air pollution

The "first generation" metallic base coats for the dry-on-wet system require a solids content of ~ 24 per cent by weight for satisfactory spraying. If a composite dry-on-wet system having a 15 μ m base coat and a 45 μ m clear powder is assumed, then the following calculation can be made:

	Thickness	Relative amounts by weight for any given area of coating	
		Total applied	Total dry
Base coat (24% solids)	15 μ m	62	15
Top coat (100% solids)	45 μ m	45	45
Overall solids =	$\frac{\text{Total applied}}{\text{Total dry}}$	$\frac{45 + 62}{45 + 15}$	56%

(For simplicity an average specific gravity of 1.0 has been used in this calculation. If actual values are taken, the percentage overall solids by weight is even higher).

This figure is not very far removed from the solids level quoted for two pack "high solids" metallic finishes currently under development, and is achieved without using expensive (and as yet unproven) computerised spray equipment and costly and toxic liquid isocyanates.

Work is under way at present to develop higher solids base coats for the dry-on-wet system to reduce solvent emissions still further.

To extend this argument on emission savings, comparison is made with the wet-on-wet system. Assume in both cases a 15 μ m base coat and a 45 μ m top coat with a 35 per cent solids top coat for the wet-on-wet system, and a 24 per cent solids base coat for the dry-on-wet and a 19 per cent solids base coat for the wet-on-wet. (These solids levels correspond to a Ford Cup No. 4 viscosity of 24 seconds).

Wet-on-wet

	Thickness	Relative amounts by weight for any given area of coating	
		Total applied	Total dry
Base coat (19% solids)	15 μ m	78	15
Top coat (35% solids)	45 μ m	129	45
Relative amount of solvent emitted	$\frac{\text{Total applied} - \text{Total dry}}{\text{Total applied}}$		147.

Dry-on-wet

	Thickness	Relative amounts by weight for any given area of coating	
		Total applied	Total dry
Base coat (24% solids)	15 μ m	62	15
Top coat (100% solids)	45 μ m	45	45
Relative amount of solvent emitted	$\frac{\text{Total applied} - \text{Total dry}}{\text{Total applied}}$		47.

$$\text{Emission saving} = \frac{147 - 47}{147} \times 100 \text{ per cent} = 68 \text{ per cent}$$

By a similar reasoning for a 20 μ m base and 40 μ m top coat the emission saving would be 60.4 per cent. Thus, a dry-on-wet system shows an emission saving of 60–68 per cent when compared with the currently used wet-on-wet method. Similar calculations can be made for straight-shade finishes.

Production of metallic and straight-shade finishes

Unlike conventional powder systems, the dry-on-wet system is suitable for both metallic and straight-shade finishes. Different colours can be alternated rapidly, as is currently carried out in the production of automobiles, and expensive colour changing equipment is not required. It also enables short runs to be made which are currently not economic.

Improved coating properties

Because the powder top coat forms the outer protective layer, the properties of the coating are largely a function of the powder. Experience of manufacturers who have switched from the solvent based coatings to powders has shown a significant reduction in losses due to handling damage in the factory and subsequent transit. A similar improvement can be confidently predicted for the dry-on-wet system.

Some wet-on-wet automobile coatings have suffered from poor intercoat adhesion, but none of the dry-on-wet systems tested have shown this deficiency even after 2000 hours Weatherometer (xenon arc) exposure, followed by immersion in water for 48 hours.

Reduced capital costs

The capital costs for installing a dry-on-wet system are much lower than those for water-borne or two pack urethane systems which are the two frequently discussed types of low pollution coatings.

Combination of proven technologies

The technologies for both the wet base and powder top coats are well understood and have been proven for a number of years. There are no problems of toxicity associated with liquid isocyanates, nor is the cost and maintenance of computerised spray guns involved, as with the two-pack high solids coatings. Neither are there the difficulties experienced with water borne coatings of effluent pollution by solubilising amines nor the costs of stainless steel pipework and air conditioning equipment.

Reduced operating costs

In common with some other low polluting systems, operating

costs are much lower than when using conventional solvent based systems. The quantity of "make-up" air required to replace that removed in keeping the solvent concentration below the lower explosive limit is reduced with consequent energy savings.

Potential applications of the dry-on-wet process

In terms of market size, the most significant potential market for the dry-on-wet system lies in the automobile industry. Because of capital investment and space requirements, a change of this nature is obviously a medium to long term consideration. Nevertheless, the large quantities of paint consumed in automobile manufacture make it a prime target for anti-pollution legislation and there is no doubt that ultimately it will be obliged to conform to very strict emission requirements. Dry-on-wet offers a means of meeting these requirements, whilst at the same time having the flexibility for colour change and production of metallic finishes required by the industry. Additional benefits are reduced energy costs and the properties of the finished coatings normally associated with powder.

There has already been interest expressed in the use of dry-on-wet for coating auto-parts, bicycle frames, toys, motor cycles and hospital furniture.

In all these examples, dry-on-wet has the advantage that all the required colour changes can be made in the thin decorative base coat, which is then overcoated with the clear protective powder coating.

Conclusion

It has been demonstrated that, using cellulose acetate butyrate, a novel type of coating system can be prepared, which largely retains the benefits of powder coating, whilst at the same time overcoming some of its major deficiencies.

[Received 26 January 1979]

Appendix

Typical base coat formulations for dry-on-wet coatings

(a) Silver metallic finish

	% by weight
Synedol ¹ 2263XB (50% in xylene butanol)	19.2
Eastman ² CAB 551-0.2	7.2
Cymel ³ 300	2.4
Silberline ⁴ Sparkle Silver 3000 AR (65% in xylene)	2.5
Silberline ⁴ Sparkle Silver 5000 AR (65% in xylene)	5.0
n-Butyl acetate	44.6
Xylene	19.1
	100.0
Spray viscosity (Ford Cup No. 4) - secs	24
Spray solids - wt %	24

(b) Blue metallic finish

	% by weight
Synedol ¹ 2263XB (50% in xylene butanol)	16.2
Eastman ² CAB 551-0.2	0.0
Cymel ³ 300	2.0
Irgalite ⁵ Blue BCS	4.0
Silberline ⁴ Sparkle Silver 3666 (65% in xylene)	6.0
n-Butyl acetate	46.1
Xylene	19.7
	100.0
Spray viscosity (Ford Cup No. 4) - secs	24
Spray solids - wt %	24

(c) White straight-shade finish

	% by weight
Synedol ¹ 2263XB (50% in xylene butanol)	4.0
Cymel ³ 300	1.0
Eastman ² CAB 551-0.2	5.0
Tioxide ⁶ R-TC4	15.0
Toluene	14.7
n-butyl acetate	26.1
Cyclohexane	8.1
Ektasolve EF acetate	12.5
Methyl ethyl ketone	13.6
	100.0
Spray viscosity (Ford Cup No. 4) - secs	15
Spray solids - wt %	25

For electrostatic spraying, the conductivity of the base coat can be increased to the value specified for the particular application equipment by the addition of a quaternary ammonium compound e.g. Querton KKES (Oleo-Fina (UK) Ltd).

Typical powder top coats and typical properties

(a) Polyurethane powder

	Parts by weight
Uralac ⁷ P2504	80
Vciba Adduct ⁸ B 1065	20
Stannous Octoate	0.7
Resilow P ⁹	1.0
Benzoin	0.8
Mixed on a Buss Ko-Kneader at 100 C and sieved through a 106µm sieve.	

Typical properties of a dry-on-wet coating using the above powder

Curing conditions	15 minutes at 180 C (metal temp)
Base coat/clear coat thickness	15µm/50µm
20/60 gloss	80/100
Pencil hardness	HB
Orange peel	trace
Front impact strength	6.8 joules (60 inch lbs)
Reverse impact strength	2.3 joules (20 inch lbs)
Flexibility $\frac{1}{8}$ " conical mandrel	no cracking
Overall adhesion - cross-hatch	Gt 0 (pass)
Intercoat adhesion - peel	excellent

(b) Acrylic powder

	Parts by weight
Cray Valley resin ¹⁰ X8401	40
Cray Valley resin ¹⁰ X8494	60
Dodecanedioic acid	17.5
Benzoin	0.2
Triphenyl tin chloride	0.3
Mixed on a Buss Ko-Kneader at 120 C and sieved through a 106µm sieve.	

Typical properties of a dry-on-wet coating using the above powder

Curing conditions	20 minutes at 170 C (metal temp)
Base coat/clear coat thickness	15µm/52µm
20/60 gloss	60/94
Pencil hardness	F
Orange peel	slight
Front impact strength	4.5 joules (40 inch lbs)
Reverse impact strength	<0.2 joules (<2 inch lbs)
Flexibility $\frac{1}{8}$ " conical mandrel	fail
Overall adhesion - cross-hatch	Gt 0 (pass)
Intercoat adhesion - peel	excellent

(c) Polyester powder

	Parts by weight
Scado ⁷ resin P2400	89
10% Acronal ¹¹ 4F in Scado P2400	12
Trisglycidyl isocyanurate ¹²	7.5
Benzoin	0.8

Typical properties of a dry-on-wet coating prepared from a powder made to the above formulation

Curing conditions	20 minutes at 180 C (metal temp)
Base coat/clear coat thickness	20µm/45µm
20/60 gloss	68/98
Pencil hardness	HB

JOYCE
LOEBL



APPLICATION REPORTS

DISC CENTRIFUGE MARK III

The physical and chemical properties of many materials often depends upon their particle size and distribution. The Joyce Loebel Disc Centrifuge and its associated hydrodynamic stability technique, the Buffered Line Start, have been used with invariant success to determine the particle size distribution of a very wide variety of particulate materials; these are listed on the back cover.

One of the most important ways in which we have been able to convince prospective customers of

the potential of the instrument within their own particular technology, has been for us to process samples, of their materials, in our Applications Laboratory.

These reports are re-printed from a selection of those sent back to the customer following such trials, and represent applications from the following major end-user groups:

- * POLYMER EMULSIONS
- * INKS AND PIGMENTS
- * TITANIUM DIOXIDE
- * MAGNETIC IRON OXIDE
- * CARBON BLACK



THE INSTRUMENT

The Joyce Loebel Disc Centrifuge Mark III is an instrument used to produce absolute particle size against weight distribution curves in the range 0.01 micron to 30 microns, by separating the sample at pre-selected sizes and quantitatively analysing the collected fractions. The instrument, with Photosedimentometer attachments offers the facility to produce:

- a) Absolute cumulative weight against particle diameter distribution curves by the standard centrifuge technique. In this method, all particles of less than a pre-determined size are collected from the disc after a pre-set time using a special sampling probe (fig. 1).

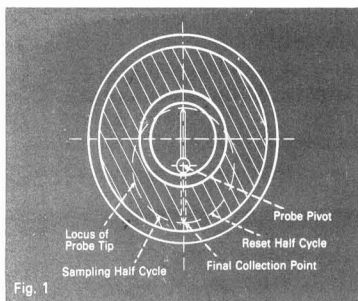


Fig. 1

The weight of particles in each collected fraction is determined by a suitable quantitative chemical analysis, and the absolute size against weight curve plotted (fig. 2).

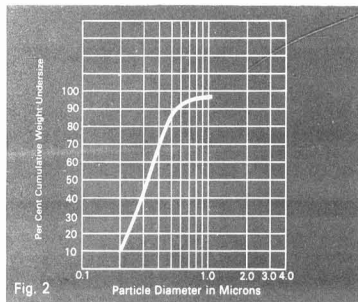


Fig. 2

- b) Rapid comparative frequency against particle size diameter distribution curves by the photosedimentometer method. In this method size distributions are produced by measuring the attenuation of a beam of white light, passed through the disc cavity at the same radius as the probe final collection point. The attenuated signal is displayed on a Chart Recorder, which produces a curve (fig. 3) representative of the size distribution of the sample under test. If required, the photosedimentometer traces can be calibrated from the results obtained using the absolute size distribution technique.

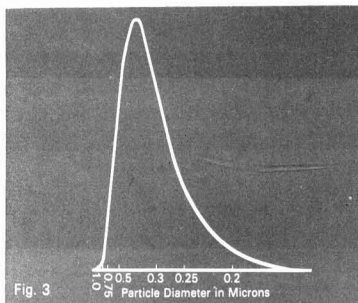


Fig. 3

LABORATORY PROCEDURES

There are two standard centrifugal sedimentation procedures, the homogeneous method and the line-start or two-layer technique. Of the two techniques, a stable line-start method is by far the most accurate and precise and is normally used in preference to the dubious homogeneous technique.

The standard two-layer technique, where the dispersion is floated directly onto the surface of the sedimentation fluid, suffers from inherent hydrodynamic instability problems. This problem is resolved by the Joyce Loebel Buffered Line Start Technique which can be observed through the transparent disc, illuminated by the synchronized stroboscope.

THE BUFFERED LINE START TECHNIQUE

The Joyce Loebel Buffered Line Start guarantees the particles will settle into the spin-fluid with streamline motion giving stable sedimentation; the progress of the particles can be derived using a modified form of Stokes' equation:

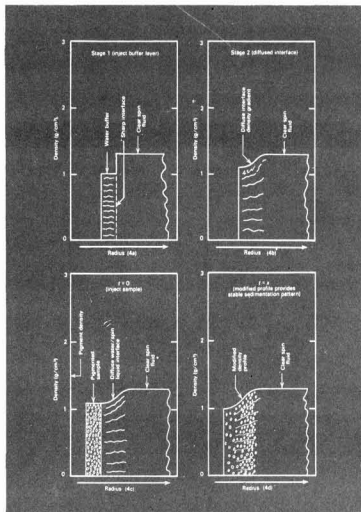
$$t = [(18\eta/d^2 \omega^2 (\rho - \rho_0))] \ln (R_2/R_1)$$

where t is the time taken for a particle of density P and size d to move from a radius R_1 to radius R_2 through a fluid of density P_0 and viscosity η where ω is the angular velocity of the centrifuge.

The Buffered Line Start Technique is a Joyce Loebel patent; it consists in physically replacing the inter-face between the dispersion and spin-fluid by a region of gradually increasing density and viscosity.

In practise a thin layer (buffer layer) of the particle dispersing fluid (normally water) is injected onto the higher density and higher viscosity sedimentation fluid, as shown in fig. 4a. This is done with the Centrifuge disc spinning at a constant speed. Then, by using special 'cut' and 'boost' controls incorporated in the instrument, the disc is subjected to a transient acceleration or deceleration sufficient to diffuse the buffer layer into the first 1 or 2 mm. of the sedimentation fluid. This technique sets up a gradually increasing density and velocity gradient in this region, as shown in fig. 4b.

When the disc returns to synchronous speed, the dispersion is injected onto the free surface (fig. 4c.) Since there is now no 'sharp' inter-face, the particles are able to move evenly through the gradient (fig. 4d) and hence stable sedimentation is reliably and reproducibly obtained.



PROBE METHOD

For each particle size fraction required, a precise volume of the dispersion is injected onto the surface of the appropriate volume of buffered spin fluid. After the pre-set time has elapsed an undersize fraction (spin fluid and particles below the programme size) is removed into a volumetric flask with vacuum assistance, by a hollow stainless steel probe rotating inside the disc cavity down to the fixed radius. The probe system is then washed through with spin fluid into the flask, which is then filled to the required graduation point with more spin fluid.

The disc is then cleaned and if required, a different group of parameters is set-up to collect a different undersize fraction and the procedure repeated.

For each sample reference fractions are produced by injecting 1 ml. of the appropriate dispersion into a volumetric flask and filling this up to the required graduation with spin fluid.

A variety of methods are used to determine the weight of the material in each of the undersize and reference fractions, amongst these are:

- Colorimetric absorption
- Volumetric estimation
- Gravimetric estimation
- Polarographic determinations et cetera

In all methods the average of the weights of material in the reference fraction is equated to 100% cumulative weight undersize, and hence the percentage weight below each collected size is calculated and the weight/size distribution curve plotted.

PHOTOSEDIMENTOMETER METHOD

Using the photosedimentometer attachment in conjunction with the computer Stokes' Law tables supplied with the instrument, it is possible to produce rapid comparative particle size diameter distribution curves.

Study of the Stokes' Law tables will indicate that as all the variables, including initial and measuring radii are known, the times for different particle sizes to reach the measuring radius can be calculated from the proportionality:

$$T \propto \frac{1}{d^2}$$

It will be appreciated that as the centrifuge speed is increased and/or the spin fluid volume is lowered (thereby reducing the distance which the particles must travel from their start radius to the measuring radius), the analysis time is reduced, but so also is the resolution achieved at the coarser end of the curve. In practise, a centrifuge speed/spin fluid volume would be empirically established to give results either in a required time or of a certain resolution.

PROCEDURE

Using the Joyce Loebel Buffered Line Start technique to ensure precise and stable sedimentation, the spin fluid is injected into the disc cavity, then the buffer layer is injected onto the surface of the spin fluid and is dispersed using the 'cut' and 'boost' controls. The disc is allowed to regain synchronous speed.

In this condition, the pen of the Chart Recorder, depicting a signal from the detector of a light beam passing axially through the disc cavity, is set on the chart baseline. The pre-selected chart drive speed is then initiated and the sample injected.

As the particles sediment past the sensing zone, set at a radius near the outer periphery of the disc, the light extinction caused by the passing particles is displayed in graphical form on the Chart Recorder.

When the run is completed, the supernatant liquid is extracted from the disc by use of the probe collecting system. The results are frequency curves of light extinction (turbidity) against time and represent the particle size distribution of the sample.

POLYMER EMULSIONS · MATERIAL-LATEX

METHOD OF ANALYSIS USED

MATERIAL PROBLEM SAMPLE

PROBE

Latex
The determination of the particle size distribution.
Latex in dispersed form - 49.87% solids,
density 1.1 gm/ml.

PHOTOSEDIMENTOMETRY

Latex
Production of particle size distribution curves.
Latex in dispersed form - 38.5% solids and powder,
density 1.25 gm/ml.

DISPERSION PREPARATION

The dispersion was diluted down to the required level of concentration with a Methanol/water solution using ultrasonic agitation for 30 seconds.

The sample containers were given ultrasonic agitation for 30 seconds in order to ensure complete dispersion, then 1 ml. was placed in a beaker and distilled water mixed with 2 drops of non-ionic surfactant (NONIDET P60) was added 5 mls at a time until the 1% level was reached. The beaker was vigorously swirled to mix the constituent parts after each water addition, and the whole was given 30 seconds of ultrasonic agitation after the dispersion was complete.

N.B. If the samples were diluted too quickly, 'dilution shock' caused agglomeration of the particles.

PARAMETERS

Dispersion Spin Fluid

2% w/v Latex/20% Methanol/Distilled Water.
Distilled water.

0.75 ml. of 1% by weight dispersion.
30 mls. of 10% w/v Glycerol/Distilled water -
density 1.022 g/ml.

Spin Fluid Viscosity Buffer Layer Density Difference Chart Speed

1.0 cP
1 ml. Distilled water.
0.1 gm/ml.

1.31 cP
1 ml. Distilled water.
0.228 gm/ml.
20 seconds/cm.

PROGRAMME

Equiv. Stokes Dia. (Microns)	Speed R.P.M.	Spin Fluid Vol. (mls)	Centrifuge Time (Secs)
0.500	6000	—	—
0.300	8000	20	555
0.200	8000	20	1249
0.100	8000	10	1459
0.075	8000	10	2590

Spin Fluid Vol. (mls)	Centrifuge Time (Secs)	Chart Travel (cms)
30	403	20

RESULTS

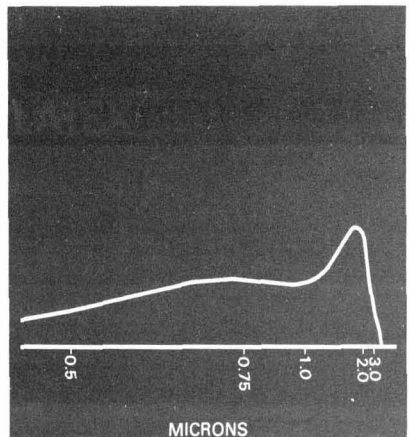
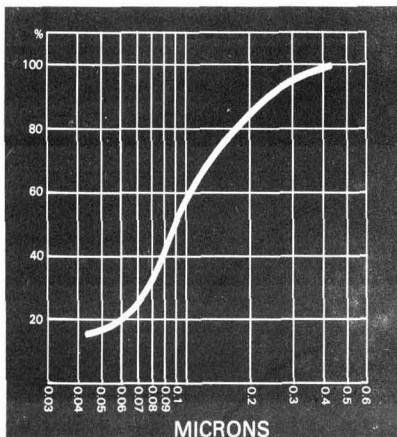
Equiv. Stokes Dia. (Microns)	Resultant Weight (Mgms)	Cumulative Weight Undersize %
0.500	—	—
0.300	20.60	94.5
0.200	18.48	84.7
0.100	11.05	50.6
0.075	5.65	25.8
Ref. sample 1	21.70)	
Ref. sample 2	21.50)	100.0

Coarse	(Microns)	Fine
1.68		0.71

COMMENTS

The use of Methanol in the dispersion (to allow sedimentation through a water spin fluid) did not appear to introduce any flocculation effects, since the maximum and minimum sizes present were not exactly comparable to those obtained when sedimenting an aqueous dispersion through a glycerol/water solution as spin fluid.

The curve shows an inflexion (marked X) prior to its first peak. In our experience this phenomenon is normally due to the presence of a small percentage of the particles which have agglomerated to form larger particles.



INKS AND PIGMENTS · MATERIAL - PHTHALOCYANINE BLUE

METHOD OF ANALYSIS

MATERIAL PROBLEM

PROBE
Phthalocyanine Blue.
Determination of the particle size distribution.

SAMPLE

Phthalocyanine Blue in powder form; particle density 1.6 gm/ml.

DISPERSION PREPARATION

The dispersion was prepared as a paste, with the surfactant then diluted with distilled water. The sample was agitated on an ultrasonic probe system for 10 seconds prior to sample run to maintain the dispersion standard.

PARAMETERS

Dispersion

0.5% w/v Phthalocyanine/Distilled water +0.05% v/v Lissapol N.

Spin Fluid

10% v/v Glycerol/Distilled water.

Spin Fluid Viscosity

1.31 cP

Buffer Layer

Distilled water.

Density Difference

0.5783 gm/ml.

Chart Speed

PHOTOSEDIMENTOMETRY

Printing Ink.

Production of particle size distribution curves of a sample of ink.

Ink sample 2% dispersion in aliphatic hydrocarbon; density 1.4 gm/ml.

Tested as submitted.

1 ml. of 2% dispersion in aliphatic hydrocarbon.

20 ml. of 9:1 v/v White Spirit/Carbon Tetrachloride.
0.58 cP

1 ml. White Spirit.

0.88 gm/ml.

20 secs/cm.

Disc speed 6000 r.p.m.

PROGRAMME

Equiv. Stokes Dia. (Microns)	Speed R.P.M.	Spin Fluid Vol. (mls)	Centrifuge Time (Secs)	Spin Fluid Vol. (mls)	Centrifuge Time (Secs)	Chart Travel (cms)
1.0	3000	30	159	20	248	12.4
0.75	4000	30	159			
0.50	6000	30	159			
0.30	8000	30	248			
0.20	8000	30	560			

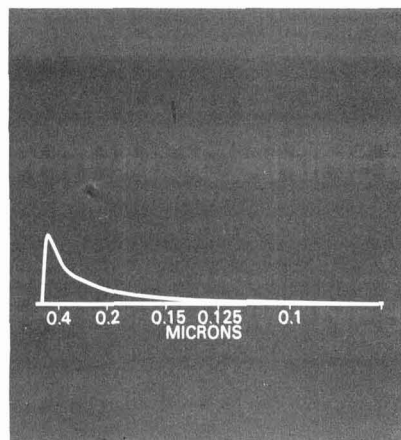
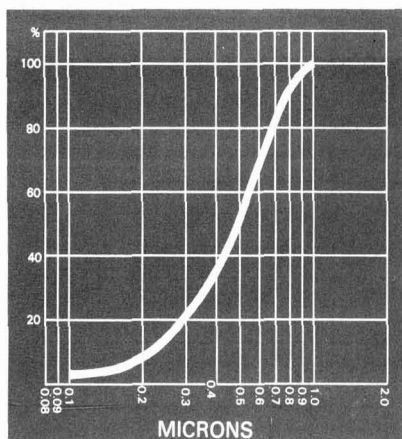
RESULTS

Equiv. Stokes Dia. (Microns)	Optical Density	Cumulative Undersize %
1.0	0.550	98.3
0.75	0.498	89.0
0.50	0.250	44.7
0.30	0.130	23.2
0.20	0.044	7.9
Ref. 1	0.570)	
Ref. 2	0.550)	100.0

COMMENTS

- Analysis was carried out colorimetrically
- To 10 ml. of the fraction was added 20 ml. of concentrated Sulphuric Acid.
 - The container was then allowed to cool.
 - The solutions were then measured on a spectrophotometer at the absorption wavelength 605 nm.
 - The average of the reference optical densities was equated to 100% cumulative weight undersize and hence the % cumulative weight below each particle size calculated.

As the sample contained a fairly wide range of particle sizes, scanning at a lower speed would resolve the coarser particle curve with greater accuracy.



TITANIUM OXIDE · MATERIAL-TITANIA T₁O₂

METHOD OF ANALYSIS

MATERIAL PROBLEM

PROBE
Titania T₁O₂
Production of size distribution curves for sample Titanium Dioxide.

SAMPLE

Powdered T₁O₂ Assumed density 4.2 gm/ml.

DISPERSION PREPARATION

An initial period of 30 seconds agitation by ultrasonic probe was used, followed by an additional 10 seconds agitation prior to run.

PHOTODEMENTOMETRY

T₁O₂
Production of size distribution curves for sample Titanium Dioxide.
Rutile Titanium Dioxide in powder form. Assumed density 4.2 gm/ml.
0.2 gm of the Titania powder was weighed into a beaker, 100 ml. of distilled water was added - this had previously been mixed with 2 drops of a non-ionic surfactant (NONIDET P60). The total time for the water/surfactant addition was around 90 seconds during which the dispersion was agitated by a 70 watt ultrasonic probe. Prior to taking an aliquot for injection into the disc, the dispersion was stirred manually for 15 seconds.

PARAMETERS

Dispersion

0.5% w/v T₁O₂/distilled water +0.05% w/v Sodium Hexametaphosphate +0.1% v/v Lissapol N dispersion.

0.5 ml. of 0.2% T₁O₂/Distilled water/Surfactant.

Spin Fluid

30% w/v Glycerol/Distilled water.

30% w/v Glycerol/Distilled water.

Spin Fluid Viscosity

2.51 cP

2.51 cP

Buffer Layer

1 ml. Distilled water.

1 ml. Distilled water.

Density Difference

3.128 gm/ml.

3.128 gm/ml.

Chart Speed

Nominal 20 secs/cm.

Measured 21.43 secs/cm.

PROGRAMME

Equiv. Stokes Dia. (Microns)	Speed R.P.M.	Spin Fluid Vol. (mls)	Centrifuge Time (Secs)
0.75	2000	30	226
0.50	4000	30	127
0.40	4000	30	199
0.30	6000	30	157
0.20	8000	30	199

Spin Fluid Vol. (mls)	Centrifuge Time (Secs)	Chart Travel (cms)
30	354	16.52

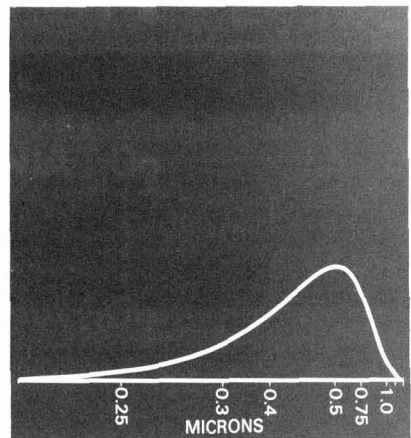
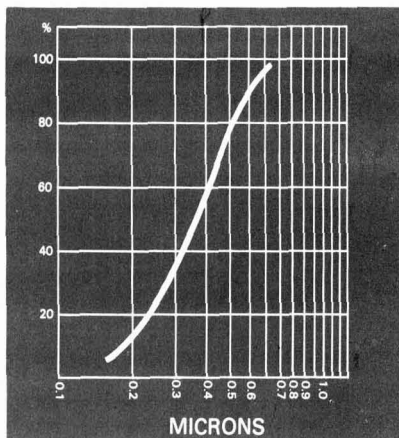
RESULTS

Equiv. Stokes Dia. (Micron)	Optical Density	Cumulative Undersize %
0.75	0.141	100.0
0.60	0.126	90.0
0.50	0.105	75.1
0.40	0.079	56.5
0.30	0.050	35.7
0.20	0.019	13.6
Ref. 1	0.139	
Ref. 2	0.141	100.0

COMMENTS

The determination of the cumulative weights of Titania in the undersize and reference fractions was carried out colorimetrically. The average of the reference optical densities was equated to 100% cumulative weight undersize and hence the % cumulative weight below each particle size was calculated.

Using the synchronised stroboscope and transparent disc the sedimentation pattern was observed as very stable.



MAGNETIC IRON OXIDE · MATERIAL - RED IRON OXIDE

METHOD OF ANALYSIS

MATERIAL PROBLEM SAMPLE DISPERSION PREPARATION

PROBE
Red Iron Oxide.
Particle size distribution in sample.
Powdered red iron oxide; density 5.24 gm/ml.
Stable sedimentation was realised using half per cent dispersion of the sample in Sodium Pyrophosphate with a wetting agent. The dispersion was mixed for 30 seconds before each run, its temperature being kept as close as possible to spin fluid temperature.

PHOTOSEDIMENTOMETRY
Magnetic Iron Oxide.
Determination of particle size distribution.
Magnetic Iron Oxide; assumed density 4.0 gm/ml.
The dispersion was prepared as a paste with a surfactant and then diluted to the required concentration level under carefully controlled conditions of ultrasonic agitation.

PARAMETERS

Dispersion

½% w/v iron oxide/0.005mol/ltr Sodium Pyrophosphate solution/1 drop Lissapol N.

0.125% w/v Iron Oxide/0.1% Truton X-100/ Distilled water +0.1% Sodium Pyrophosphate.

Spin Fluid

2.5 cP

6.05 cP

Spin Fluid Viscosity

1 ml. Distilled water.

1 ml. Distilled water.

Buffer Layer

4.168 gm/ml.

2.87 gm/ml.

Density Difference

Chart Speed

20 secs/cm

Disc Speed 6000 r.p.m.

PROGRAMME

Equiv. Stokes Dia. (Microns)	Speed R.P.M.	Spin Fluid Vol. (mls)	Centrifuge Time (Secs)
2.0	1000	30	105
1.0	1000	30	426
0.75	2000	30	189
0.50	2000	30	426
0.40	2000	30	664
0.30	4000	30	295
0.25	4000	30	426
0.20	4000	30	664

Spin Fluid Vol. (mls)	Centrifuge Time (Secs)	Chart Travel (cms)
30	149	1.2

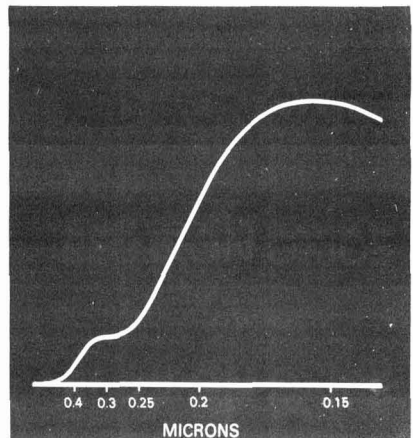
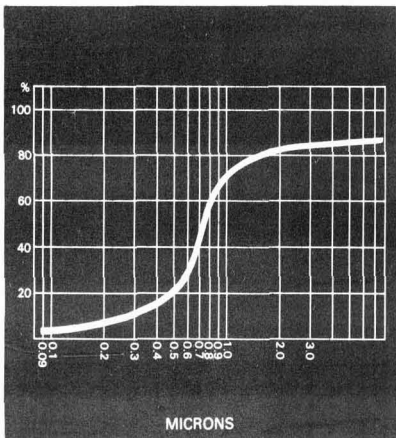
RESULTS

Equiv. Stokes Dia. (Microns)	Optical Density	Cumulative Undersize %
2.0	0.2337	81.0
1.0	0.1994	70.0
0.75	0.1313	46.0
0.50	0.0551	19.0
0.40	0.0408	14.0
0.30	0.0276	10.0
0.25	0.0216	8.0
0.20	0.0165	6.0
0.10	0.0088	3.0
Ref. sample	0.2871	100.0

COMMENTS

To 3 mls. of each fraction and the reference sample. 7 mls. of concentrated Hydrochloric Acid (36 HCL) was added to dissolve the iron oxide. The solutions were then measured by a Spectrophotometer at 410 m in a 1 cm. cell. The equivalent optical density of the reference sample was equated to 100% and by comparison the optical density of each fraction represented the percentage cumulative weight.

Using the synchronised stroboscope and transparent disc the sedimentation pattern was observed as very stable.



CARBON BLACK·MATERIAL-CARBON BLACK IN OIL

METHOD OF ANALYSIS

MATERIAL PROBLEM

PROBE

Carbon Black in Oil
Determination of the particle size distribution of carbon black in oil dispersions.

SAMPLE DISPERSION PREPARATION

Carbon black in oil; density 1.8 gm/ml.
The dispersion used was as received after using ultrasonic treatment to break agglomerates

PHOTSEDIMENTOMETRY

Carbon Powder.
Production of particle size distribution curves by photosedimentometry.
Carbon in powder form; density 1.8 gm/ml.
Each dispersion was prepared as a paste with a surfactant, then diluted with distilled water to the required concentration level under ultrasonic agitation.

PARAMETERS

Dispersion 1% w/v Carbon Black/Oil and Arobis.

Spin Fluid 30% v/v Carbon Tetrachloride/White Spirit.

Spin Fluid Viscosity 0.75 cP

Buffer Layer 75% v/v White Spirit/Arobis.

Density Difference 0.75 gm/ml.

Chart Speed

0.05% w/v Carbon/Methanol/Distilled water
20:80+0.1% non-ionic surfactant.

Distilled water.

1 cP

1 ml. Methanol/Distilled water 20:80.

0.8 gm/ml.

60 secs/cm

PROGRAMME

Equiv. Stokes Dia. (Microns)	Speed R.P.M.	Spin Fluid Vol. (mls)	Centrifuge Time (Secs)
0.75	3000	30	126
0.50	4000	30	158
0.30	6000	30	196
0.20	8000	30	247
0.15	8000	20	238
0.10	8000	20	500

Spin Fluid Vol. (mls)	Centrifuge Time (Secs)	Chart Travel (cms)
20	156	2.6

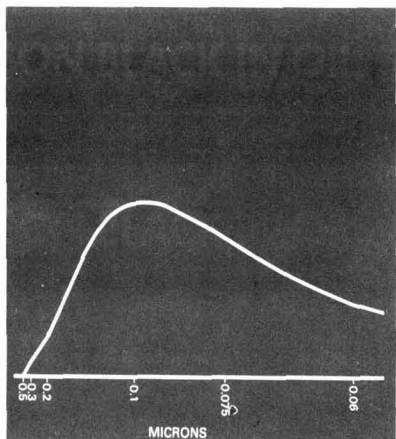
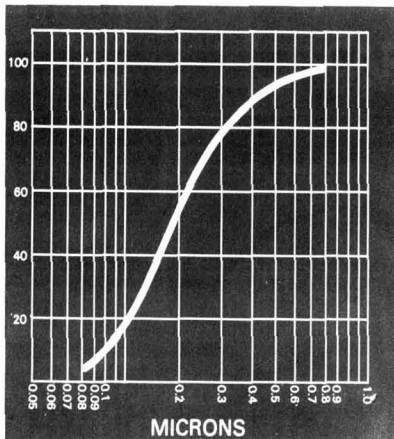
RESULTS

Equiv. Stokes Dia. (Microns)	Resultant Weight (Mgs)	Cumulative Weight Undersize %
0.75	9.41	98.7
0.50	8.78	92.0
0.30	7.64	80.0
0.20	5.37	56.2
0.15	3.07	32.2
0.10	0.96	10.5
Ref. sample 1	9.42)	
Ref. sample 2	9.69)	100.0

COMMENTS

The analysis method used was 'filtering and weighing'. The second filter used in the analysis caught any particles which permeated the top filter, the bottom filter being used as the actual control filter.
In this way accurate and reproduceable results were obtained using only normal laboratory care and technique.

The quality of the dispersion did not appear to be affected by the addition of the Methanol as the same distribution was obtained when running a straight-forward water dispersion. The addition of the Methanol allowed a water spin fluid to be used, so reducing the sedimentation time.



DISC CENTRIFUGE MARK III CAPABILITY

These reports represent only a few of the many trials carried out on behalf of customers, and are published to illustrate the variety of the systems which have been studied using the Disc Centrifuge, and to show the essential experimental details and the typical programmes and parameters involved.

We will be delighted to supply further details of the Disc Centrifuge and to answer any questions you may have regarding the instrument and its associated technology.

If the material you are interested in sizing is not contained in these reports or listed here, our Applications Department will be pleased to carry out trials on your material to prove to you the capability of the Joyce Loebl Disc Centrifuge Mark III.

Materials for which detailed Application Reports are available:

MATERIAL	METHOD OF ANALYSIS USED		MATERIAL	METHOD OF ANALYSIS USED	
	PHOTOSEDI-MENTOMETER	PROBE		PHOTOSEDI-MENTOMETER	PROBE
Aluminium	.	.	Ilmenite	.	.
Aluminium Oxide	.	.	Kaolin	.	.
Aluminium Silicate	.	.	Latex Emulsion	.	.
Asbestos	.	.	(PVA/PVC/Acrylic etc.)	.	.
Ammonium Perchlorate (fuel)	.	.	Lead Oxide	.	.
Anthracene (pigment)	.	.	Lead Telluride	.	.
Bentonite	.	.	Molybdantie	.	.
Barium/Strontium Carbonate	.	.	Molybdenum Bisulphide	.	.
Barium Sulphate	.	.	Mica	.	.
Biological Cells	.	.	Oil in Water Emulsion	.	.
Cadmium Sulpho-Selenide (pigment)	.	.	Polystyrene	.	.
Clays	.	.	Resin (phenolic)	.	.
Calcium Oxide	.	.	Rubber Latex	.	.
Carbon Black	.	.	Pharmaceuticals	.	.
Carbon in Engine Oil	.	.	Organic Resin	.	.
Calcium Carbonate	.	.	Pulverised Fuel Ash	.	.
Caffeine (in tea)	.	.	Silver Halide Emulsions	.	.
Calcium Sulphate (gypsum)	.	.	Quartz	.	.
Cement	.	.	Steroids	.	.
Corn Starch	.	.	Sugar	.	.
Copper Oxychloride	.	.	Silts/soils	.	.
Copper Silicate	.	.	Sodium Fluoride	.	.
Cobalt	.	.	Silica	.	.
Chocolate	.	.	Silicon Nitride	.	.
Cellulose	.	.	Silicon Dioxide	.	.
Carbon Tantalum (C Ta)	.	.	Silicon Powder	.	.
Calcium Phosphate	.	.	Pigment/Dyestuffs	.	.
Fly Ash	.	.	Piperazin Derivative	.	.
Fluorescent Powders	.	.	Phthalocyanine Blue (pigment)	.	.
Flue Dust	.	.	Paint	.	.
Graphite	.	.	Telc	.	.
Ferrite	.	.	Tungsten	.	.
Gold	.	.	Tungsten Carbide	.	.
Glass	.	.	Titanium Dioxide	.	.
Hormone	.	.	Whiting	.	.
Insecticide	.	.	Zinc Oxide	.	.
Ion Complex	.	.	Vanadium Carbide	.	.
Ion Oxide (magnetic)	.	.	Urea Formaldehyde Resin	.	.
Ink (printing)	.	.	Uranium Dioxide	.	.
			Zircon	.	.



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Crosfield
SILICAS

**Consider where our other silicas are used
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Crosfield Silicas. **What we've done for others,** **we'll happily do for you.**

Crosfield matting agents do a very good job.

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Above all, Crosfield HP Silicas* offer excellent matting efficiency and achieve the required effect with exceptional economy.

They give you outstanding clarity and a superb film smoothness like silk.

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*Crosfield HP is a proprietary brand name.

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experience tells in the end

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Orange peel	moderate	3. Cyanamid (UK) Ltd.
Front impact strength	6.8 joules (60 inch lbs)	4. Silberline (UK) Ltd.
Reverse impact strength	4.5 joules (40 inch lbs)	5. Ciba-Geigy Ltd, UK.
Flexibility—1" conical mandrel	20 mm cracking	6. BTP Tioxide Ltd.
Overall adhesion—cross-hatch	Gt 0 (pass)	7. Scado B.V. Holland
Intercoat adhesion—peel	excellent	8. Veba-Chemie AG, BRD.
		9. Worlee-Chemie GmbH, BRD
		10. Cray Valley Products, UK.
		11. BASF AG, BRD.
		12. Ciba-Geigy Ltd, UK.

References

1. Synres BV Holland.
2. Fastman Chemical International

Discussion at Stratford Conference

MR J. R. TAYLOR asked about the metallic finishes applied by the dry-on-wet method, which had been claimed to be excellent. Normally difficulties arise with geometric metamerism in this type of paint. Mr Taylor asked if the "flip tone" was consistent, and less than that obtained with the wet-on-wet system.

MR WINDIBANK stated that provided enough CAB was used in the formulation, the "flip-flop" effect was very good, and indicated that CAB was widely used on the Continent in wet-on-wet systems for this reason. The dry-on-wet system required the use of more CAB, but provided sufficient was present, then the effect with metallic paints was good.

MR MAHAR added that the commercial spray equipment mentioned in the paper had actually been used successfully for this type of application and that the results, examples of which he had available, were very good.

MR R. H. WALLINGTON asked if using the dry-on-wet system would present any greater problems for touching up damaged metallic finishes than the current metallic systems.

MR WINDIBANK indicated that if repair of the coating was necessary, then the same problems would be experienced using the dry-on-wet system as with the current wet-on-wet systems. It would be necessary to take the car to a proficient repairer, but it should be possible to achieve a good match.

MRS. T. HARRIS pointed out that the powder coatings formulations for the dry-on-wet system appeared to have almost stoichiometric quantities of curing agents. He suggested that it might be possible to improve the mechanical properties of the dry-on-wet system, particularly the acrylic system, by increasing the proportion of curing agent to achieve some measure of intercoat adhesion.

MR WINDIBANK agreed that this might be so, and reported that with the polyurethane systems, greater than stoichiometric amounts of the isocyanate had been incorporated without the catalyst, resulting in an improvement in certain properties. He agreed that it might be rewarding to follow up this suggestion with the acrylic system.

MR MAHAR explained that one of the problems with the acrylic system was that very few firms in the UK were currently using acrylic powder systems, and it was very difficult to arrange for co-operation. The polyurethane systems were far more widespread and here it was possible to collaborate with many firms both in the UK and Europe.

MR T. R. BULLETT asked if the authors could offer an explanation of the remarkable effect of CAB in accelerating the loss of solvent from the film.

MR WINDIBANK said that it was thought that the cellulosic nature of the CAB gives an open structure in the same way as does nitrocellulose, which allows the rapid removal of the solvent.

The changing role of polymer chemistry in organic coatings technology*

By M. F. Kooistra

Akzo Coatings, Amstelveen, Holland

Summary

The course of the reactions occurring during the stoving of high solids polyester-melamine finishes is followed by using a variety of modern analytical techniques. Low molecular weight neopentylglycol adipates and fatty acid modified alkyl resins are used as model polyesters and hexamethoxy methyl melamine and a butylated melamine resin are used as typical amino resins.

It is stressed that in normal stoving finishes, the greater proportion of the total chemical reactions occur during the preparation

of the resins and relatively little during stoving. The reverse is the case with high solids and solvent free coatings, where relatively little chemical reaction occurs during the formation of the low molecular weight polymers, and the greater part of the reactions occur during stoving.

The analytical techniques used enable the amount of self condensation occurring in the prepolymers to be determined, as well as the types of linkage present in the final completely cured finish.

Keywords

Types and classes of coatings and allied products

high build coatings
stoving finish

Raw materials for coatings binders (resins, etc.)

alkyd resin
polyester resin
amino resin
melamine resin

Processes and methods primarily associated with analysis, measurement or testing

gas chromatography
nuclear magnetic resonance spectroscopy
gel permeation chromatography
gradient elution liquid chromatography
high pressure liquid chromatography
radio chemical method

Le rôle évolutif de la chimie polymères dans la technologie des revêtements organiques.

Résumé

Au moyen d'une gamme de techniques d'analyse modernes, on suit le cours des réactions qui se produisent pendant le séchage au four de finitions "high solids" à base de résines polyester-mélatmine. On utilise en tant que polyesters types les adipates de néopentylglycol de faible poids moléculaire et une résine alkyde modifiée par des acides gras, et en tant que résines amines typiques la hexaméthoxy méthyle mélatmine et une résine mélatmine butylée.

A l'égard des finitions usuelles qui séchent au four, on souligne que la plupart des réactions chimiques se produisent au cours de

la préparation des résines, et en comparaison les réactions lors du séchage au four sont peu nombreuses. Il est tout à fait au contraire dans le cas des revêtements "high solids" ou exempts de solvant, où il n'arrive pas beaucoup de réaction chimique au cours de la formation des prépolymères à faible poids moléculaire, et la plupart des réactions se produisent pendant le séchage au four.

Les techniques d'analyse utilisées permettent la détermination du degré d'autocondensation qui se produit dans les prépolymères, aussi bien que les types de liaison qui se présentent dans la couche de finition après son durcissement complet.

Die sich verändernde Rolle der Polymerchemie in der Technologie organischer Beschichtungsmittel

Zusammenfassung

Der Reaktionsverlauf bei Ofentrocknung von Polyester-Melaminlacken hohen Festgehaltes wird unter Benutzung einer Anzahl moderner analytischer Techniken verfolgt. Als Modell-Polyester werden niedrig molekulare Neopentylglykoladipate und fettsäuremodifizierte Alkydharze angewandt, und als typische Aminoharze werden Hexamethoxymethylmelamin und ein butyliertes Melaminharz benutzt.

Es sei betont, dass bei normalen Ofenlacken der grössere Teil der gesamten chemischen Reaktionen während der Herstellung der Harze, und ein verhältnismässig kleiner während der

Ofentrocknung stattfindet. Der Fall ist bei lösungsmittelfreien Lacken mit hohem Festgehalt umgekehrt, wobei wenig chemische Reaktion stattfindet, wenn die Präpolymere niedrigen Molekulargewichtes gebildet werden, und der Hauptteil der Reaktionen während der Ofentrocknung stattfindet.

Die verwendeten analytischen Techniken ermöglichen die Bestimmung der Höhe von Selbstkondensation in den Präpolymeren ebensowie der Art der im endgültig, völlig gehärteten Lackfilm anwesenden Arten von Bindungen.

*Paper presented on behalf of FATIPEC at the Association's biennial Conference "The challenge to coatings in a changing world" held at Stratford upon Avon from 20-23 June 1979.

Introduction

Modern organic coatings of the "High solids" or "Solvent free" types are characterised primarily by the low initial molecular weight of the vehicle resins used, as compared to conventional solvent based coatings. This is necessary for reasons of application viscosity, assuming that the same application techniques are being used.

This is true for various classes of high solids or solvent free coatings, such as two pack epoxy or polyurethane coatings, acrylic oligomer and monomer based coatings for electron beam curing, unsaturated polyester plus monomer based coatings for UV curing, and particularly so, for modern oven-baked thermosetting coatings, based upon low molecular weight OH-functional polyester or acrylic resins, which are cured with a low molecular weight cross-linking resin such as hexamethoxy methyl melamine (HMMM).

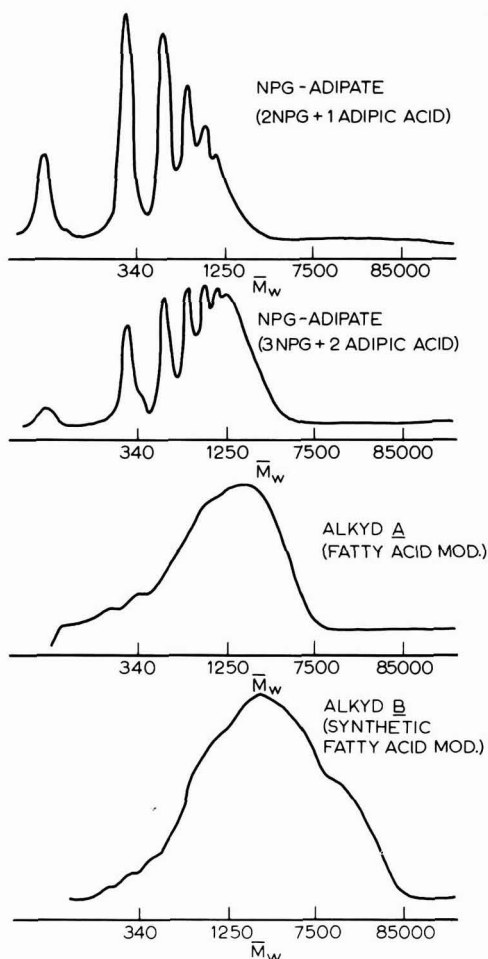


Figure 1. GPC chromatographs of ester diols and conventional alkyd resins

For practical reasons the discussion here will be limited to one particular type of high solids coating, i.e. the class of oven-baked coatings from low molecular weight OH-functional polyesters in combination with HMMM as a cross-linker. This type of coating shows interesting possibilities as an automotive topcoat, and comparisons will be made with conventional solvent-based polyester or alkyd-melamine types of automotive topcoats.

As an example of a model compound which may be used as the OH-functional component of such a system, di(neopentylglycol)adipate or tri(neopentylglycol)di(adipate) will be chosen as ester diols.

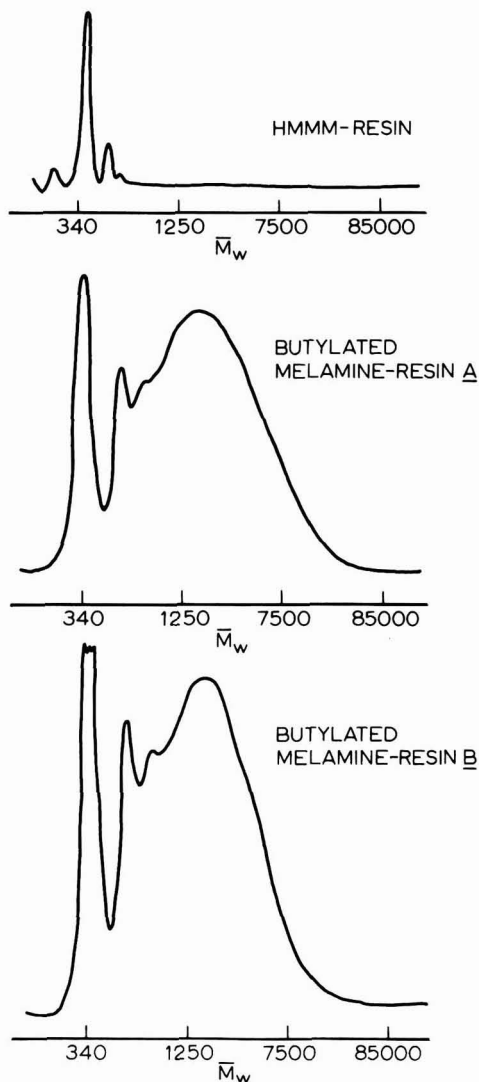


Figure 2. GPC chromatographs of a commercial HMMM and butylated melamine resins

Fig. 1 shows gel permeation chromatograms of such ester diols in comparison with commercial conventional (synthetic) fatty acid modified alkyd resins used for the same type of application. Fig. 2 shows gel permeation chromatograms of a commercial HMMM resin as compared with two conventionally used butylated melamine resins.

Table 1 shows some elementary information on molecular weight and molecular weight distribution of these resins.

Table 1
Molecular weights and molecular weight distributions \bar{M}_w/\bar{M}_n of high solids and conventional resins

	\bar{M}_n	\bar{M}_w	$\frac{\bar{M}_w}{\bar{M}_n}$
Di-(NPG)-adipate	320	540	1.7
Tri-(NPG)-di-(adipate)	580	910	1.6
Alkyd A	925	1670	1.8
Alkyd B	1390	5610	4.0
HMMM-resin	458	526	1.15
Butylated melamine resin A	1070	2730	2.55
Butylated melamine resin B	867	1860	2.15

Scope of study

Curing (baking) of alkyd-melamine resin combinations leads to the formation of a three-dimensional network of molecules having a certain cross-link density, necessary to produce the desired properties, such as hardness, flexibility, adhesion, resistance and durability.

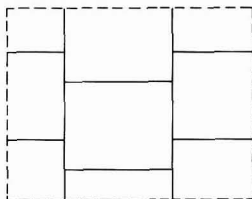
A. CONVENTIONAL SYSTEM

ALKYD $\bar{M}=1500$
BUTYLATED MELAMINE $\bar{M}=1200$

SIZE OF NETWORK A:

8 CROSS-LINKS
3-DIMENSIONAL:

$$8^{\frac{3}{2}} = 23 \text{ CROSS-LINKS}$$



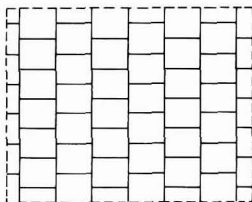
B. HIGH SOLIDS SYSTEM

ESTERDIOL $\bar{M}=500$
HMMM $\bar{M}=400$

SIZE OF NETWORK B:

84 CROSS-LINKS
3-DIMENSIONAL:

$$84^{\frac{3}{2}} = 770 \text{ CROSS-LINKS}$$



IN HIGH SOLIDS EXAMPLE DURING CURING
10-30 x AS MUCH CHEMISTRY AS
COMPARED TO CONVENTIONAL EXAMPLE

Figure 3. Hypothetical networks, starting from conventional and high solids resins

Hypothetical networks are constructed in Fig. 3, one starting from conventional resins with molecular weights of 1500 and 1200, the other starting from high solids type resins with molecular weights of 500 and 400.

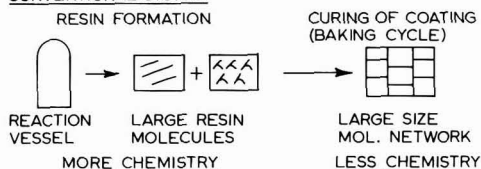
The sole purpose of these hypothetical networks is to compare the order of magnitude of size and dimensions of cross-linked structures. It is obvious, even without bothering about exact chemical processes, functionalities etc., that with such high solids resins the cure reaction roughly involves 10-30 times more chemical reactions as compared to comparable conventional coating resins.

It is, therefore, postulated that the chemistry occurring during curing of high solids (and solvent free) coatings is of much more importance than that of conventional solvent based coatings.

In other words, a greater part of the chemistry necessary to form a coating's end product, i.e. a polycondensed insoluble three-dimensional polymer network, takes place during the curing cycle with HS/SF coatings, whereas a greater part of the chemistry, playing a role in conventional, solvent-based coating resin technology, takes place when making the resin (Fig. 4).

It is recognised, that the role of polymer chemistry is changing in modern organic coatings technology, shifting from the resin cooking to the coating curing stage.

CONVENTIONAL SYSTEM



HIGH SOLIDS SYSTEM

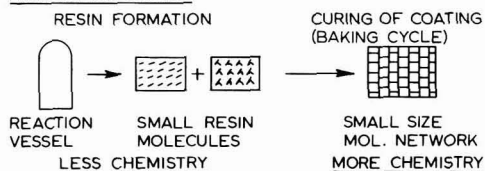


Figure 4. The polymer chemistry shifts from resin formation to the resin curing stage for high solids coatings

Clearly, the resin making process will still always be an essential part of the whole process, both with low molecular weight resins or oligomers for HS/SF coatings. For example, this is necessary to control chemical structural details, molecular weight distribution (narrow), distribution of functional groups etc.

Characterisation of cured coatings

Refs. 1, 2

In characterising the structure of cured coatings, the concept of cross-link density is often used. Cross-link

density is generally described in terms of \bar{M}_c , i.e. the average molecular weight of chains between cross-links, which can be calculated from the initial molecular weight \bar{M}_0 , the initial average functionality f_0 , and the average extent of the polycondensation reaction, expressed as r (the fraction of "used" functionalities), as follows:

$$\bar{M}_c = \frac{\bar{M}_0}{r f_0 - 2}$$

assuming that two functional groups do not contribute to cross-linking, but simply serve for chain extension (Huntjens, Berger¹).

The cross-link density may be an important parameter by which to judge cross-linked polymer performance properties (dimensional strength and stability, creep rate, solvent resistance, heat distortion or softening temperature, etc.) (Nielsen²). It has the disadvantage, however, of being only an average value, if calculated as above.

In general, the study of cross-linked polymers by such techniques as swelling by solvents, elastic moduli and creep measurements, and mechanical damping (for a coatings' resin network torsional braid analysis or vibrational spring methods could be considered), is very difficult to interpret exactly in chemical terms. This certainly is true for coatings, where relatively little work has so far been done.

Chemical structural analysis of cured coatings to elucidate its cross-linked structure appears to be very difficult and is always of a destructive nature, thus making a correct interpretation very uncertain.

In the author's opinion, more can be learned by following the chemical reactions during curing, measuring the kinetics of separate reactions, and using expert interpretation to reach a conclusion on the real chemical structure of the end product - the cured coating.

It is postulated here, that following chemical reactions during curing and measurement of the reaction kinetics are of prime importance in gaining an insight into cross-linked structures such as cured coating resins.

Polycondensation of multi-functional resins

Refs. 3-6

Multi-functional polycondensation and gelation of cross-linking resins are very complex processes, characterised by an accelerating growth of branched polymer chains progressing into networks, and ending in complete precipitation as a gel. Conditions for gelation and examples of molecular weight calculations were presented by Flory³ and Stockmayer⁴. In these calculations, the probability of chemical reactions leading to molecular weight increase, was the basic starting point (statistical method), in fact, disregarding the kinetics of the chemical reactions involved.

Such a statistical approach in more complicated situations, such as with coating resins with their non-homogeneous chemical composition, and consequently varying functionality, is of great practical difficulty.

Pis'men and Kuchanov⁵ derived an exact solution of the kinetic equations describing multi-functional polycondensation. Their method makes possible a general calculation of the main statistical distribution characteristics.

Similarly, Andrianov and Emel'yanov⁶ analysed the roles of kinetic, thermodynamic and structural factors in the reactions of polyfunctional compounds which lead to the formation of such gel polymer structures. The emphasis on reaction kinetics is striking, but not really surprising, because a true chemical approach cannot dispense with the basic chemical reaction kinetics.

It is essential, when trying to study reaction kinetics of such multi-functional polycondensation reactions (which in such cases as ester diol resins with HMMM or OH-functional acrylic resins with HMMM is the main curing reaction), to have an exact knowledge of the chemical composition of the starting reactants.

Practical approaches

Ref. 7

The high solids industrial topcoats now under development on the basis of low molecular weight ester diols and HMMM-types of cross-linking resins, promise attractive performance properties.

Such a system will be considered in some more detail. Fig. 5 shows the major possibilities for chemical reaction in the system ester diol plus HMMM.

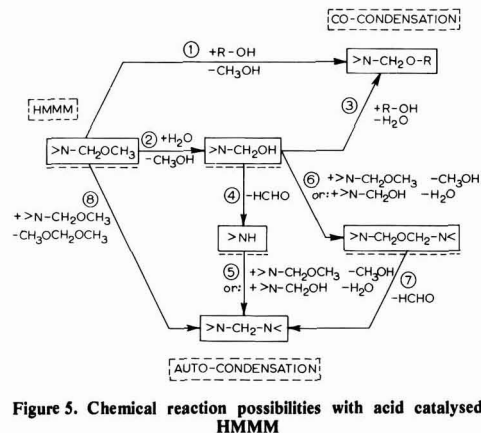


Figure 5. Chemical reaction possibilities with acid catalysed HMMM

The essential reaction paths which produce the desired cross-linking between the two resins (acid catalyst is assumed to be present in all cases) are reaction 1 (direct trans-etherification) and reactions 2 and 3 (hydrolysis of a methoxy-group, followed by etherification with an OH-group of the ester diol).

Reaction 2, however, yielding $>N-CH_2OH$ as an intermediate step, may easily lead to autocondensation of the HMMM, via reaction paths 4 and 5 or 6 and 7.

Reaction 8 shows the direct autocondensation of two molecules of HMMM, producing $CH_3OCH_2OCH_3$ (dimethoxymethane or methylal) as a volatile by-product. In all cases of autocondensation of HMMM, the final linkage is $>N-CH_2-N<$.

It is generally accepted (a good review of acid-catalysed alkyd-melamine reaction possibilities was presented by

Holmberg⁷), that self-curing (autocondensation) of melamine resins is favoured by high concentrations of $>NH$ and $>N-CH_2OH$ groups. This also holds for HMMM resins.

It is, moreover, interesting to note the influence of water (vapour) on reaction 2, producing the intermediate $>N-CH_2OH$ structure, also an intermediate for the co-condensation reaction 3 and for autocondensation of the HMMM resin. It is generally accepted that water vapour, which is always present in practical curing ovens, participates to a large extent in such curing reactions.

It can be concluded that in the curing of a relatively simple reaction mixture, such as an ester diol with HMMM, both autocondensation of HMMM and co-condensation of HMMM with ester diol will contribute to the construction of the cross-linked molecular network.

In Fig. 6, the differences between two extreme possibilities (reactions which are largely by autocondensation or by co-condensation) are illustrated. From the

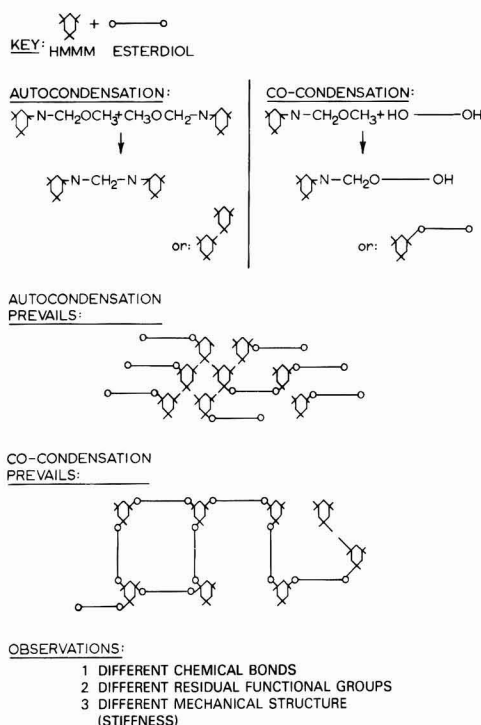


Figure 6. Effects of HMMM autocondensation and co-condensation with ester diol resin

differences indicated in ultimate structure of the cured resin combinations, it becomes apparent how essential is a knowledge of the curing mechanism to be able to assess a coatings' properties. It is postulated here, that knowledge about the chemical structure of a cured coating is of paramount importance in order to predict performance properties after ageing, which is of direct interest in the early development stage of such products.

Identification of initial resins

Ref. 8

In order to indicate the complexity of the curing process of a relatively simple coatings composition, based upon an ester diol and HMMM, and to underline the essential identification of the resinous compounds present, the composition of commercial resins of the HMMM-type may serve as a perfect example.

Characterisation of melamine resins has been an intriguing activity for many research workers. Christensen⁸ has presented a survey of methods for the analysis of functional groups in amino resins, demonstrating the complexity of the subject.

By using analytical techniques, such as gel permeation chromatography (GPC), gradient elution liquid chromatography (GrLC), high pressure liquid chromatography (HPLC), 1H NMR or ^{13}C NMR (proton or carbon-13 nuclear magnetic resonance), the analysis of commercial HMMM resins presents no major difficulty.

Fig. 7 shows GPC and GrLC curves of four commercial HMMM resins, Fig. 8 shows an example of a ^{13}C NMR spectrum of such a resin, and Table 2 shows some characteristic data on functional groups present in these resins.

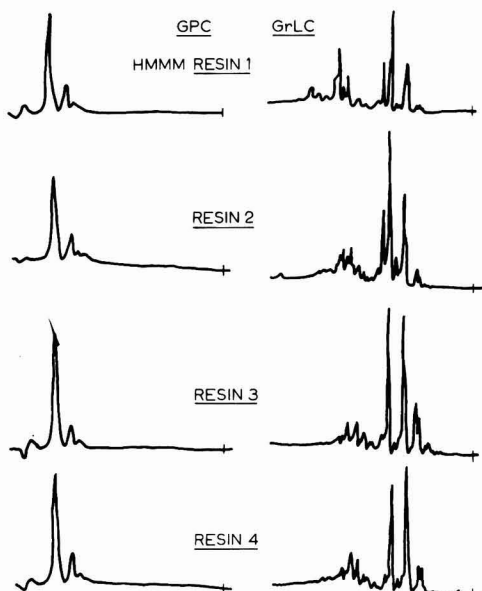


Figure 7. GPC and GrLC chromatographs of commercial HMMM resins

Table 3 shows the amount of oligomers in these resins, as determined with GPC.

It is obvious from this information that because of differences in composition and functionality, careful analysis of a certain HMMM resin should always precede

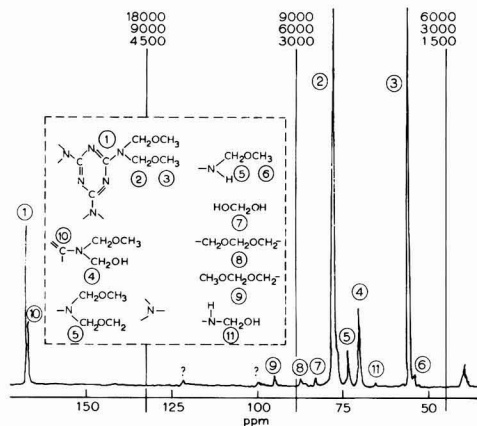
Figure 8. ^{13}C NMR spectrum of a commercial HMMM resin

Table 2
Functional groups per triazine ring in
commercial HMMM resins

	$-\text{CH}_2\text{OCH}_3$	$-\text{CH}_2\text{OH}$	$>\text{NH}$
HMMM Pure	6	0	0
Resin 1	5.0	0.6	0
Resin 2	5.0	0.3	0.4
Resin 3	4.4	1.2	0.2
Resin 4	4.7	0.8	0.3

any measurement of reaction kinetics in a combination with, for example, an ester diol resin. The same holds good for this type of resin as well.

Measurement of HMMM cure reactions

To simulate practical conditions and to have the ability to examine changes under these conditions, HMMM cure reactions can be conveniently followed in a flow-cell or mini-oven type of experimental set-up, where a layer of the material under investigation is cured under pre-determined conditions, and where volatile reaction products are collected and fed directly into a connected gas chromatograph.

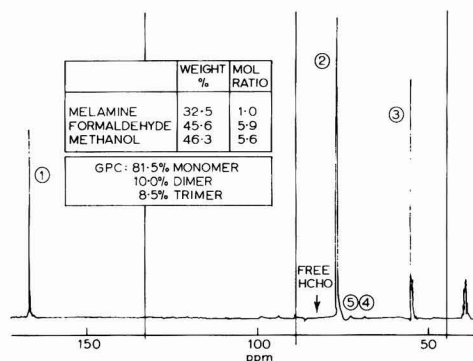
From the reaction schemes in Fig. 5 it can be seen that the emission products to be expected under certain curing conditions, are the following:

dimethoxymethane (methylal)	$\text{CH}_3\text{OCH}_2\text{OCH}_3$	- in the absence of $R-\text{OH}$ or water (reaction 8)
methanol	CH_3OH	- in the presence of $R-\text{OH}$ (reaction 1)
methanol	CH_3OH	- in the presence of water (reaction 2)
formaldehyde	HCHO	- in the presence of water (reactions 2 + 4)
water	H_2O	- when initially water or $>\text{N}-\text{CH}_2\text{OH}$ was present (reaction 4)
methanol + water + formaldehyde)	- in the presence of $R-\text{OH}$ and water (reactions 1 + 2 + 3 + 4 reactions 5, 6 + 7)

Table 3
Oligomer distribution as a percentage in commercial HMMM resins

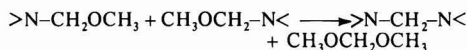
	Monomer	Dimer	Trimer	Tetramer	Pentamer
HMMM Pure	100	-	-	-	-
Resin 1	72	18	6	3	1
Resin 2	57	23	11	6	3
Resin 3	82	14	3	1	-
Resin 4	73	20	6	1	-

Experiments were carried out with model-HMMM of known composition (Fig. 9) in a mini-curing oven under a He-atmosphere and always in the presence of 1 Mol. per cent of para-toluene sulfonic acid, dissolved in 3.4 Mol. per cent of CH_3OH , as a catalyst.

Figure 9. ^{13}C NMR spectrum of a purified HMMM resin

Reaction rates at different curing temperatures under dry conditions, in the absence of $R-\text{OH}$ (ester diol) or water, can be deduced from dimethoxymethane emission as shown in Fig. 10.

The comparison of emission product curves, when curing HMMM dry or in the presence of water vapour (Fig. 11) is interesting. In the first situation, dimethoxymethane emission is dominant, indicating that the major reaction is:



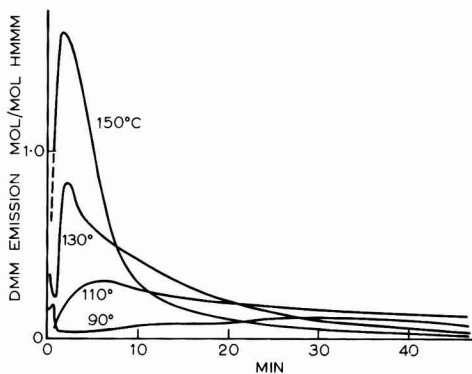
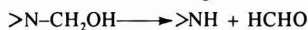
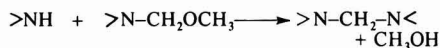


Figure 10. DMM (dimethoxymethane) emission at "dry" curing of HMMM at different temperatures

In the second case very little dimethoxymethane, a nearly constant amount of HCHO and a large amount of methanol are produced, which is indicative of the formation of intermediate $>N-CH_2OH$ groups, followed by autocondensation according to reactions 4 and 5 (Fig. 5):



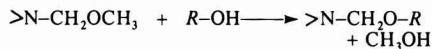
or



Alternative autocondensation reactions, according to reaction pathways 6 and 7, may also take place.

The dominant role of H_2O (as water vapour) for the autocondensation of HMMM becomes evident.

On comparing the curing of HMMM under dry conditions on its own or in combination with an ester diol, the transesterification reaction



obviously dominates in the second situation (Fig. 12).

A different, but very convenient approach to the measurement of such curing reactions will be briefly mentioned here: the use of Fourier Infrared analysis. The Fourier transform infrared spectrometer normally produces a spectrum within one second (absorption) or within one minute (emission). Thus, many spectra can be obtained during curing of such a coating film, thus following the curing reactions more or less continuously. Following the decrease of the ether band at 915 cm^{-1} , with the triazine band at 815 cm^{-1} as a reference (Fig. 13), the disappearance of HMMM $>N-CH_2OCH_3$ groups can be recorded as a function of time, and it is convenient to plot temperatures as a function of time simultaneously (Fig. 14).

Discussion

The purpose of this work is to indicate the possibilities of studying reaction mechanisms and reaction rates of multifunctional polycondensation reactions, such as with high solids ester diols with HMMM, as a tool to obtain an understanding of the role of polymer chemistry in the curing phase, which from a chemical point of view is of such major importance in constructing films of high solids and solvent free coatings.

It is hoped to present further information on the subject of auto- and co-condensation of high solids resin combinations for industrial topcoats on the basis of ester diols with HMMM in a future publication.

Evaluation results of performance properties of different types of high solids and solvent free coatings will then com-

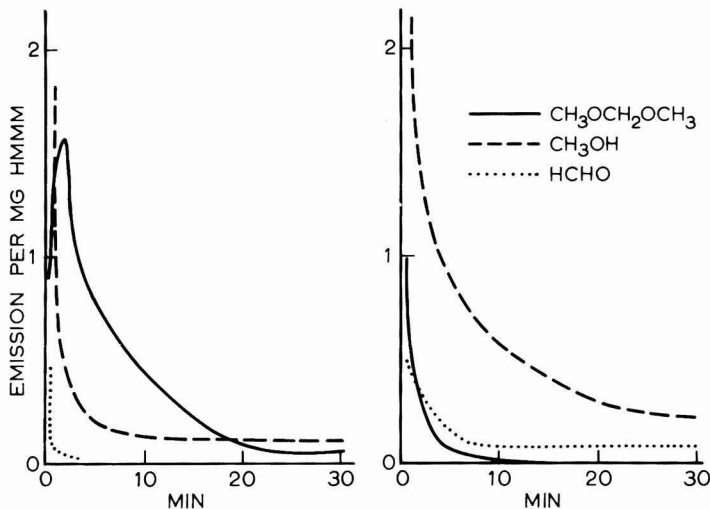


Figure 11. Left: "Dry" curing of HMMM resin at 130°C . Right: Curing of HMMM resin under water vapour at 130°C

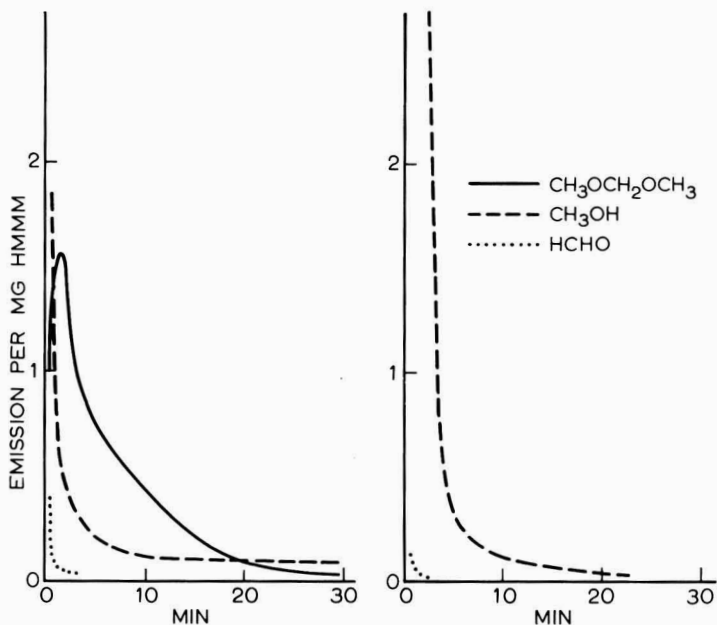


Figure 12. Left: "Dry" curing of HMMM resin at 130°C. Right: "Dry" curing of HMMM with ester diol resin at 130°C

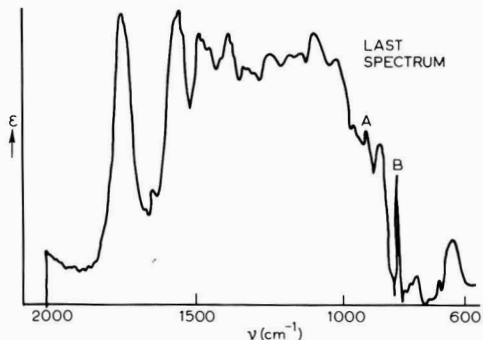
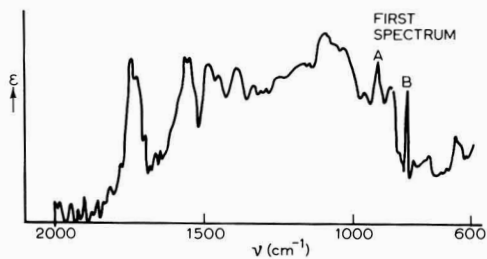


Figure 13. Fourier infrared emission spectra of HMMM with ester diol resin curing at 130°C

plete the picture, demonstrating the feasibility of high solids or solvent free coatings having remarkably good mechanical, resistance and durability properties.

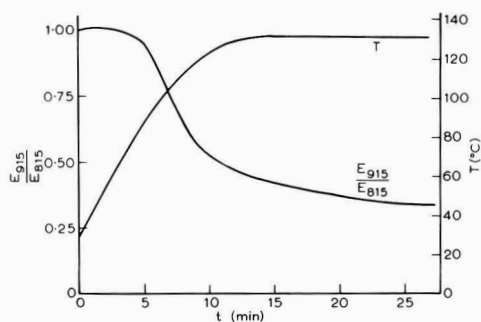


Figure 14. Curing of HMMM with ester diol resin followed with Fourier infrared (E_{915}/E_{815}) and temperature as a function of time

Acknowledgements

The author wishes to thank his colleagues in the Akzo Corporate Research Laboratories, Arnhem, for their contribution and help, and for the presentation of their research work results, and in particular to Mr J. H. van Dijk and his colleagues from the Analytical Chemical Department, Dr R. Buter and his assistants from the Organic Chemistry Department, and Dr P. van Woerkom from the Physical Research Department, and also Mr P. Wagenaar from Synthese Kunststoffsabrieken, Bergen op Zoom. He also wishes to thank the Management of Akzo Coatings and of Sikkens Lakfabrieken, Sassenheim, for their support and encouragement in presenting this paper.

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Discussion at Stratford Conference

DR J. RAMSBOTHAM asked to what extent the Fourier transform infrared spectroscopy technique could be interpreted quantitatively, and how accurate a technique it was for following the progress of the curing mechanism. In his experience, great care was needed when making quantitative measurements with this system.

IR KOOISTRA stated that this was not his speciality, but that the experts who performed the measurements were confident that they had achieved acceptable accuracy in measuring the reaction rate by this technique.

DR T. A. EGERTON, continuing on the subject of the infrared spectroscopy, asked if the films used in the work

had been pigmented or unpigmented, and if the transmission of infrared had been sufficient with pigmented films.

IR KOOISTRA stated that the examples he had shown were of pigmented films, showing emission spectra.

MR S. VOUT asked if there was any intention to study the use of aziridine crosslinkers in this research work.

IR KOOISTRA said that some work was being done on the use of aziridine crosslinkers, but they had not been used yet in his studies, because the aziridine crosslinking mechanism is not directly relevant to the ester diol-HMMM curing reactions involved.

Next month's issue

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

Characterisation of the interaction between pigments and binder solution using rheological measurements by A. Saarnak

A study of the effects of increasing crystallinity in polyvinylidene chloride coated polypropylene (Prop C) and cellulose (mxxT/s) by M. W. Leonard

Coatings based on fatty acid modified cellulose phthalate resins by B. P. Singh, S. Chandra and A. K. Vasishtha

Raft trials of anticorrosive paints of high resistance by V. J. D. Rascio, J. J. Caprari, B. del Amo and R. D. Ingeniero

Scanning electron microscope study of a coating component deposited from solution into wood

By M. H. Schneider

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Summary

Linseed oil was deposited from solution into wood and then viewed with the scanning electron microscope in order to obtain information about solution coating's distribution in wood, and to obtain the oil content of wood cell walls. The oil was non-

uniformly distributed in the wood, and first appeared in cell lumens at approximately 5 per cent oil content. Below 5 per cent it appeared to be in cell walls.

Keywords

Types and classes of coatings and allied products

sealant
wood finish
impregnating varnish

Types and classes of structures or surfaces to be coated

wood

Raw materials for coatings

oils

linseed oil

Processes and methods primarily associated with analysis, measurement or testing

electron microscopy

Properties, characteristics and conditions primarily associated with materials in general

permeability

structures or surfaces being coated

wood structure

Une étude a l'aide du microscope électronique à balayage de la déposition d'un constituant soluble d'un revêtement appliqué au bois.

Résumé

L'huile de lin était déposée en bois à partir d'un solvant, et ensuite examinée par un microscope électronique à balayage, afin d'obtenir de l'information sur la répartition dans le bois du revêtement en solution, et d'obtenir la teneur en huile des parois des

cellules du bois. L'huile était répartie dans le bois dans une manière inégale et elle ne se présentait dans la lumière des cellules qu'à une teneur en huile de 5% à peu près. A moins de 5%, il paraissait qu'elle se trouvait dans les parois des cellules.

Abtastelektronenmikroskopische Untersuchung einer aus Lösung auf Holz niedergeschlagenen Beschichtungsmittelkomponente

Zusammenfassung

Leinöl wurde aus einer Lösung auf Holz ausgeschieden und darauf mit dem Abtastelektronenmikroskop betrachtet, um die Verteilung der Lösung im Holz kennenzulernen und den Ölgehalt der Zellenwände des Holzes festzustellen. Das Öl war im Holz

ungleichförmig verteilt und wurde zunächst mit etwa 5 Prozent Ölgehalt in den Zellenlumen festgestellt. Unter 5 Prozent schienen sich in den Zellwänden zu befinden.

Introduction

Ref. 1

Coatings for wood are usually applied as solutions in volatile solvents. Little information is available concerning the effects of the concentration of the solution upon the distribution of the coating in the wood. One objective of this study, therefore, is to generate such information using a rather simple coating. In this study, an equilibrium approach was desirable because it allows ready measurement of oil content and it can give a large sample. In the equilibrium approach, wood samples are immersed in and allowed to come to equilibrium with an excess of solution.

It has been speculated¹ that penetration of wood cell walls by coatings could be very important to adhesion between the coating and the wood, particularly under adverse conditions. The second objective of this study is to obtain the approximate oil content at which the linseed oil component of coatings first becomes visible in lumens, since there is some question as to how much oil penetrates cell walls¹.

Experimental procedure

Ref. 2

White spruce (*Picea glauca* Moench. (Voss.)) sapwood

was crosscut into 2 cm lengths using a precision, smooth-cutting saw. It was then split into 0.5 cm square samples, 2 cm long. These were air dried and then oven dried at 103°C to constant weight.

Dry samples were placed in solutions of raw linseed oil in carbon tetrachloride for several days. Concentrations ranged from controls in neat solvent to 80 per cent linseed oil, solvent weight basis. The carbon tetrachloride does not swell wood² and thus it is the oil which should dominate adsorption interactions.

Solvent was evaporated from the samples using moving, inert gas. Their oil content was then determined gravimetrically, knowing the dry wood weight. Oil was cured in an oven maintained at 60°C for one week.

After the curing process, each sample was cut in half across the grain, and one newly exposed surface moistened. This surface was smoothed with a fresh razor blade. The other half of the sample was split radially. The prepared cross sectional and radial surfaces were coated with gold and viewed in a Cambridge S4-10 scanning electron microscope.

Results and discussion

Figures 1 and 2 are scanning electron micrographs of the untreated wood. They provide a basis for comparison of oil-containing samples. Figure 3 is representative of wood containing 2.5 per cent oil. There is no obvious difference from the untreated wood, suggesting that any oil present has been sorbed into the cell wall.

At 5 per cent oil, Figures 4 and 5, the oil is evident on the cut surfaces, but the cell lumen interiors appear to be little changed from the untreated condition. This suggests that

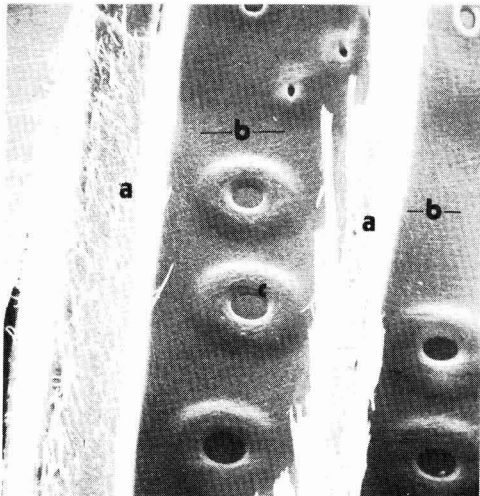


Figure 1. Intertracheal bordered pits and tracheid lumen interior from split radial surface of untreated white spruce (x1714).

- (a) Fractured intercellular walls
- (b) Cell cavities with wall sculpturing
- (c) Intercellular bordered pits (the cells are tracheids, hence the pits are often called intertracheal)

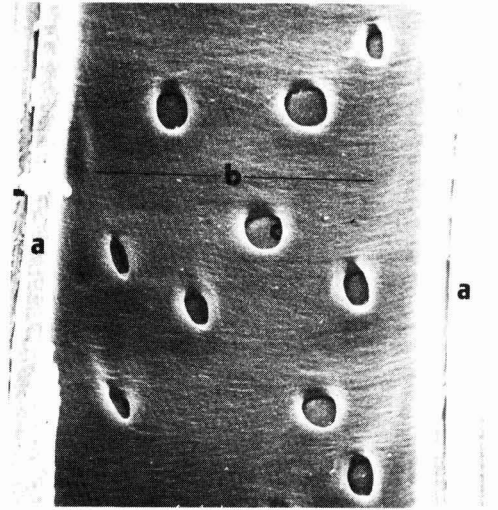


Figure 2. Interior of tracheid of untreated white spruce in ray crossing area (x3428).

- (a) Fractured intercellular walls
- (b) Cell cavities with wall sculpturing
- (c) Pits between tracheid (shown) and ray cells (behind)

most of the oil is sorbed in cell walls, and only exposed by the cutting process.

At 7 per cent oil, Figure 6, oil is visible as a film on two of the lumen interiors and as droplets on several of the intercellular pits. Figure 7 is spruce containing 17 per cent oil. One pit contains crinkled oil, and there is a film of oil

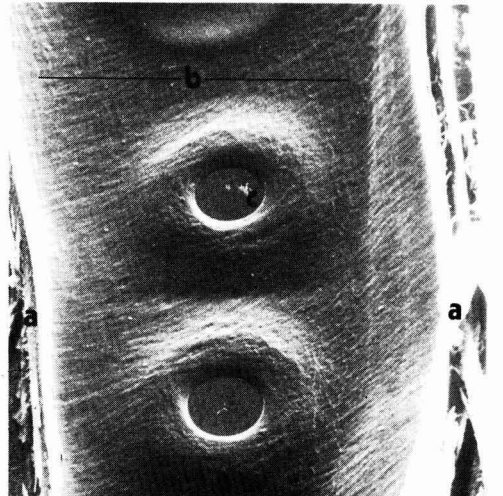


Figure 3. Intertracheal bordered pits and lumen interior of spruce containing 2.5 per cent linseed oil. Change in cell wall or pit structure, compared to untreated wood, is not noticeable (x1971).

- (a) Fractured intercellular walls
- (b) Cell cavities with wall sculpturing
- (c) Intercellular border pits (intertracheal)

over the cell lumen interiors. These two figures do not show as great a difference in visible oil as their measured oil contents would indicate. Also, oil distribution within the viewed

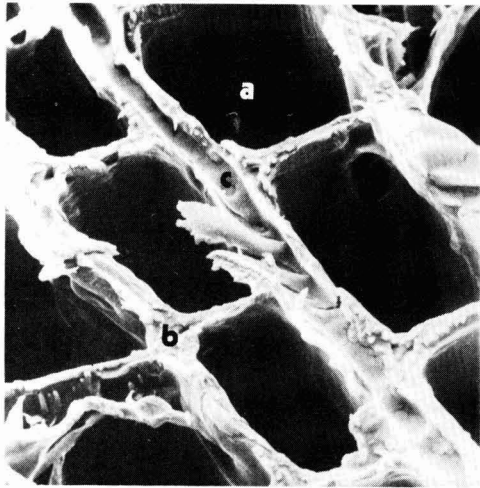


Figure 4. Cross sectional surface of spruce earlywood containing 5 per cent oil. Some oil is evident on cut surfaces. Lumen interiors may be slightly smoothed compared to untreated wood (x1200).

- (a) Cell cavity
- (b) Cut cell wall containing oil on surface
- (c) Exposed ray cell cavities containing oil (probably squeezed from walls during cutting)

Note: This wood is from the fast growing part of the annual ring where cavities are large and walls thin.

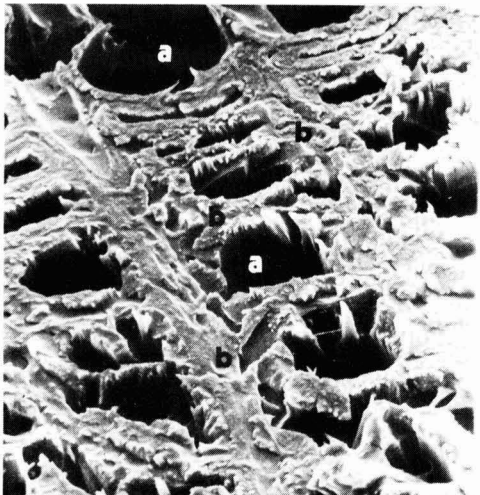


Figure 5. Latewood adjacent to earlywood of Figure 4 (5 per cent oil). Again, effects on cell interiors are not very noticeable, but cut surfaces exhibit oil (x1200).

- (a) Cell cavity
- (b) Cut cell wall containing oil on surface

Note: This is from the denser wood laid down later in the growing season when walls are thick. Many coniferous woods show this annual pattern.

areas is non-uniform, with some pits full of oil and others containing little.

Scanning electron micrographs of spruce containing 30 per cent linseed oil are shown in Figures 8 and 9. Copious amounts of oil are visible in both micrographs. The radial

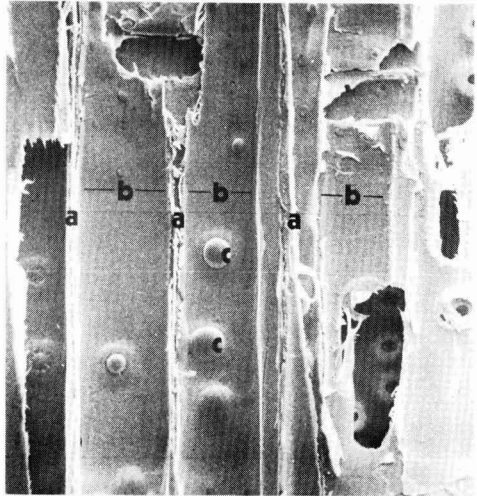


Figure 6. Split radial surface of spruce containing 7 per cent oil. Oil droplets are associated with some of the pits, and a film of oil covers the lumen interior surfaces (x463).

- (a) Fractured intercellular walls
- (b) Cell cavities coated with film of oil
- (c) Oil droplets in intertracheal bordered pits

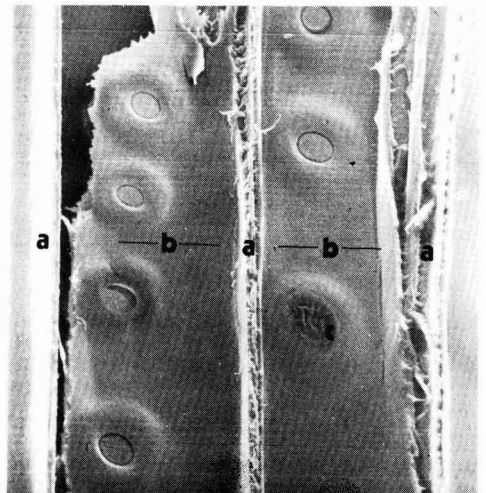


Figure 7. Split radial surface of spruce containing 17 per cent linseed oil. One pit contains crinkled oil. The remainder of the surface has only a thin film of oil, if any (x951).

- (a) Fractured intercellular walls
- (b) Cell cavities coated with film of oil
- (c) Intertracheal bordered pit containing oil which crinkled during curing

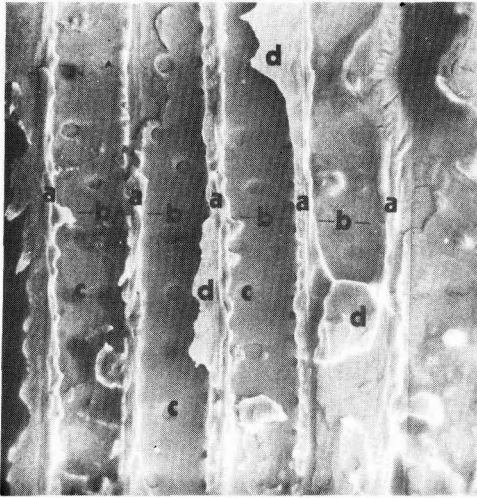


Figure 8. Split radial surface of spruce containing 30 per cent linseed oil. The oil is abundant in lumens, allowing only impressions of pits to be seen (x960).

- (a) Fractured intercellular walls
- (b) Cell cavities coated with film of oil
- (c) Bordered pits nearly filled with oil
- (d) Excess oil as "globs" in cell cavities

Note: Complete surface is covered with oil in this micrograph.

view (Figure 8) shows a non-uniform distribution of oil. There are places where lumens are nearly full, and others where an oil film covers the lumen interior.

This series of scanning electron micrographs shows that differing concentrations of oil in wood are visible, and that there is not an even distribution of oil within each sample. The uneven distribution may be a result of the sample preparation technique in this particular instance. The technique employed however, is similar to the application of thinned coatings to wood. A more idealised technique, employing low and fluctuating pressure, would probably give a more uniform oil distribution.

The micrographic evidence suggests that small amounts of oil are sorbed in walls. Below approximately 5 per cent oil content, the oil appears to be located in cell walls, but not in lumens. A more precise estimate of the point at which oil first appears in lumens could perhaps be made by obtaining more uniform oil distribution within the sample, and working with small increments of oil content near 5 per cent.

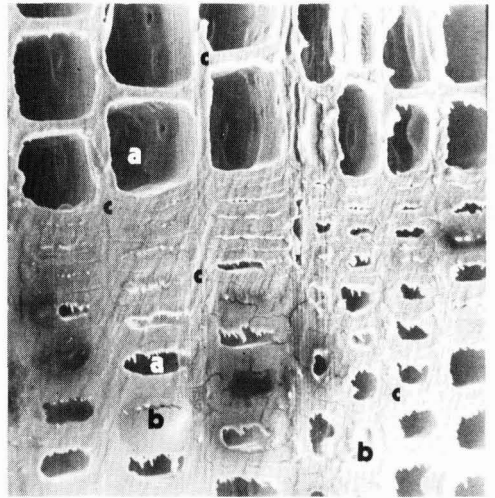


Figure 9. Cross sectional surface of spruce containing 30 per cent oil. Oil is abundant both on cut surfaces and on lumen interiors (x497).

- (a) Open cell cavities
- (b) Cavities filled with oil
- (c) Cut walls swollen with oil

Note: This is a latewood area.

Conclusions

Various concentrations of linseed oil in wood are differentiable in scanning electron micrographs of the wood structure. Oil distribution within wood is non-uniform using the method of uptake employed in this experiment.

Oil is not visible in cell lumens at concentrations below approximately 5 per cent oil, but is visible on the cut surface and perhaps as a smoother texture of cell walls. This indicates that the oil is sorbed by the walls. Because of non-uniform oil distribution, and few data points near 5 per cent, a refined experiment would be required to obtain a more reliable estimate of the oil content at which oil first appears in lumens.

[Received 14 May 1979]

References

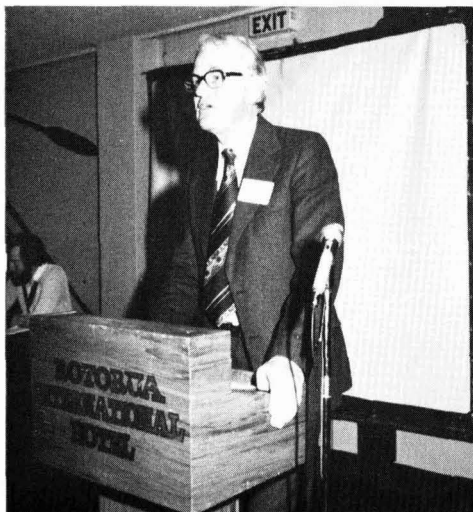
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New Zealand Division

Annual Convention 1979

The seventeenth annual convention of the New Zealand Division of OCCA was held at the International Hotel, Rotorua, on 26-29 July. The theme for this year's papers was "On the surface" and emphasis was made throughout the sessions on the importance of understanding substrates as part of the total surface coatings technology. Fifteen speakers addressed delegates during the three day convention and the six overseas lecturers contributed valuable experience of current developments.

Following the investiture of Colin Gooch as the 1979-80 NZ Vice-President of OCCA, Dr D. Kear, Assistant Director General of the Department of Scientific & Industrial Research New Zealand, addressed the full conference on the current energy reserves in this country. While it is true that liquid fuels are lacking in New Zealand, Dr Kear reviewed the massive reserves of alternative energy available. The combined resources of gas, coal, geothermal steam, electricity and biomass energy potential are more than sufficient to meet the needs well into the future.



Dr D. Kear delivering his address to the Convention

During the remainder of the sessions, two lectures were presented simultaneously in adjacent theatres. Mr D. R. Martin of Mintech (NZ) Ltd described indigenous fillers and extenders for use in the paint industry. The development of this local industry has been fascinating to follow, to the point where today the degree of product sophistication is remarkable. Comparison of geological origins helped explain problems in matching overseas formulations, but in most cases reformulation around NZ minerals produced acceptable products. Dr V. H. Vedda, Tenneco Chemicals, USA, spoke of the development and merchandising of multiple-choice colour systems for paints. Colour matching, preparation, formulation and prediction of market preferences are all fully computerised in the USA and on the Continent. A lively discussion on the technical difficulties of formulating base vehicles for the colour systems followed.

Mr T. J. Rickerton of Ciba-Geigy Australia spoke on organic pigment technology. The organic pigment manufacturers' problems are daunting when the endless permutations and combinations of chemical substitutions which can be made in a few basic pigment structures are considered. Optimising existing pigment types has considerably upgraded pigment performance. The future moves towards high-solids, water-based, powder and UV curing systems, assures the organic pigment technologist of a constant supply of headaches. Dr T. J. Sprott discussed analytical techniques for surface coatings and underlined the need to know the basic chemistry involved in order to get the best from sophisticated instruments. He considered that the testing and quality control of NZ paints could be improved.

The third session was a panel discussion with four speakers presenting short papers on particular substrates, followed by a protracted question and answer session on some of the controversial issues raised. Dr John Harris of the Forest Research Institute spoke about wood as a substrate and advocated a tighter control of kiln drying techniques to minimise surface coating problems. Mr Don McIntyre of James Hardie & Co. discussed asbestos/cement products, relating curing techniques to paint performance for building applications. Mr Joe Bognar of NZ Forest Products Ltd described a wide range of wood panel products and their particular surface coating properties. He warned of the encroachment of laminated finishes into the traditional paint market, where liquid coatings had failed to provide end-user requirements.



The panel for the discussion during the third session of the Convention (l-r): Dr J. Harris, Mr J. Bognar, Mr P. Sharp (Panel Chairman), Mr D. McIntyre and Dr D. Christian

Dr David Christian from NZ Steel discussed galvanised steel as a substrate and emphasised that when painted, the paint must assume the role of the barrier coat. He rejected the two-coat acrylic paint system for galvanised roofing and sparked off a heated discussion amongst delegates. This panel session was one of the most important of the convention, as the opportunity for dialogue between paint chemists and substrate manufacturers occurs, regrettably, only infrequently.

During the fourth session, Mr W. A. Vandenbrink of Samson Paints gave an entertaining review of grinding equipment from the 14th Century. His colourful descriptions were well laced with anecdotes and were suitably illustrated with slides of the cumbersome mills used in paint manufacture. Mr F. Glasl of Hoechst Australia discussed

new opaque organic pigments used for decorative industrial finishes and automotive coating. Use of various particle sizes to improve the rheology of paint systems was illustrated and it was shown that much progress has been made towards replacing older toxic pigments. Organic/inorganic blends of chrome, titaniums and synthetic pigments are performing well in current trials.

During the fifth session, Mr R. Reti from Dow Corning Australia presented a paper on silicone alkyds. He gave a brief history of silicone chemistry and used slides to demonstrate the incredible properties of silicone polymers in the formulation of durable surface coatings. Mr C. F. Francis from Du Pont Australia discussed PETTCA with its implications for the paint industry in New Zealand. The Pigments Ecological and Toxicological Technical Committee of Australia is actively engaged in setting acceptable levels for colourants in a variety of products, including paints and other surface coatings. It was evident that a similar body was needed in NZ.

In the final session Mr E. V. Carter discussed the painting of steel bridges in the UK. His talk was highlighted by a film of current developments and showed how virtually maintenance-free surface coatings can be achieved, in contrast to the continuous painting programmes usually associated with bridges in marine environments.

Michael and Yvonne Edwards are both psychologists concerned with inter-personal relationships, particularly those in marriage and within the family. They jointly addressed OCCA members and their wives in a stimulating and thought provoking session. An animated question and answer period rounded off the convention.

The social activities at "Convention '79" provided some memorable highlights for all those who attended. The sports tournament between the Auckland and Wellington Sections was again a keenly contested affair, although the golfers found their playing surface coated with rather too much rain. Enthusiasm was not dampened, and the weekend's activities ended with the official dinner and the presentation of trophies.

J.G.G.

Transvaal

South African Corrosion School 1979

The role of protective coatings

The third Corrosion School was held on 2-3 July 1979 at the University of Witwatersrand with the theme "The role of protective coatings". It was organised jointly by the Transvaal Section, the South African Corrosion Institute and the South African Chemical Institute. The organising committee was under the chairmanship of Mr P. A. J. Gate, South African Vice-President of OCCA.

The School was opened by a Vice-President of the South African Corrosion Institute, Mr H. Snow, on behalf of the President, Mr M. Brett who was overseas. The first lecture dealt with the theory and principles of metallic coatings. This was followed by a lecture on surface preparation and application of coatings. The coating system and its components was then dealt with. The final lecture of the first morning detailed the factors affecting the selection of a

protective system. The afternoon was devoted to metallic coatings including galvanising and rubber lining, except for the last lecture which dealt with the subject of primers.

The first three lectures of the second day were concerned with mild and heavy duty paint coatings. A lecture on tape systems followed and the second morning was concluded by a lecture on the philosophy underlying specification compilation for particular environments.

The final papers covered the execution of a contract and inspection methods, coating failures and maintenance coating. The school was concluded by an open forum which was chaired by Mr D. J. Pienaar. Due to the large number of questions submitted by delegates, only a small proportion of which could be answered at the open forum, a further meeting of the school is being organised for the evening of 30 August. This will be held at Kelvin House, in Johannesburg, at which it is hoped to deal with the remaining questions.

The School was attended by 249 delegates, 56 of whom travelled from outside the Johannesburg area. Delegates were present from Zimbabwe Rhodesia and South West Africa. Fifty-three per cent of delegates could be classed as users of anti-corrosion products and coatings, and represented inter alia electricity supply authorities, chemical plant engineers, mining engineers, municipalities, consultants and representatives from industries ranging from brewing to property development. Some 30 per cent of the delegates were from material and equipment suppliers, 7.5 per cent could be classed as contractors in the application of coatings (paint, rubber linings, metal spraying and galvanising). The remainder represented Universities, technical colleges, standards and research organisations.

The lectures have been published in book form to provide a permanent record for delegates.

Rare earth driers and the cobalt story

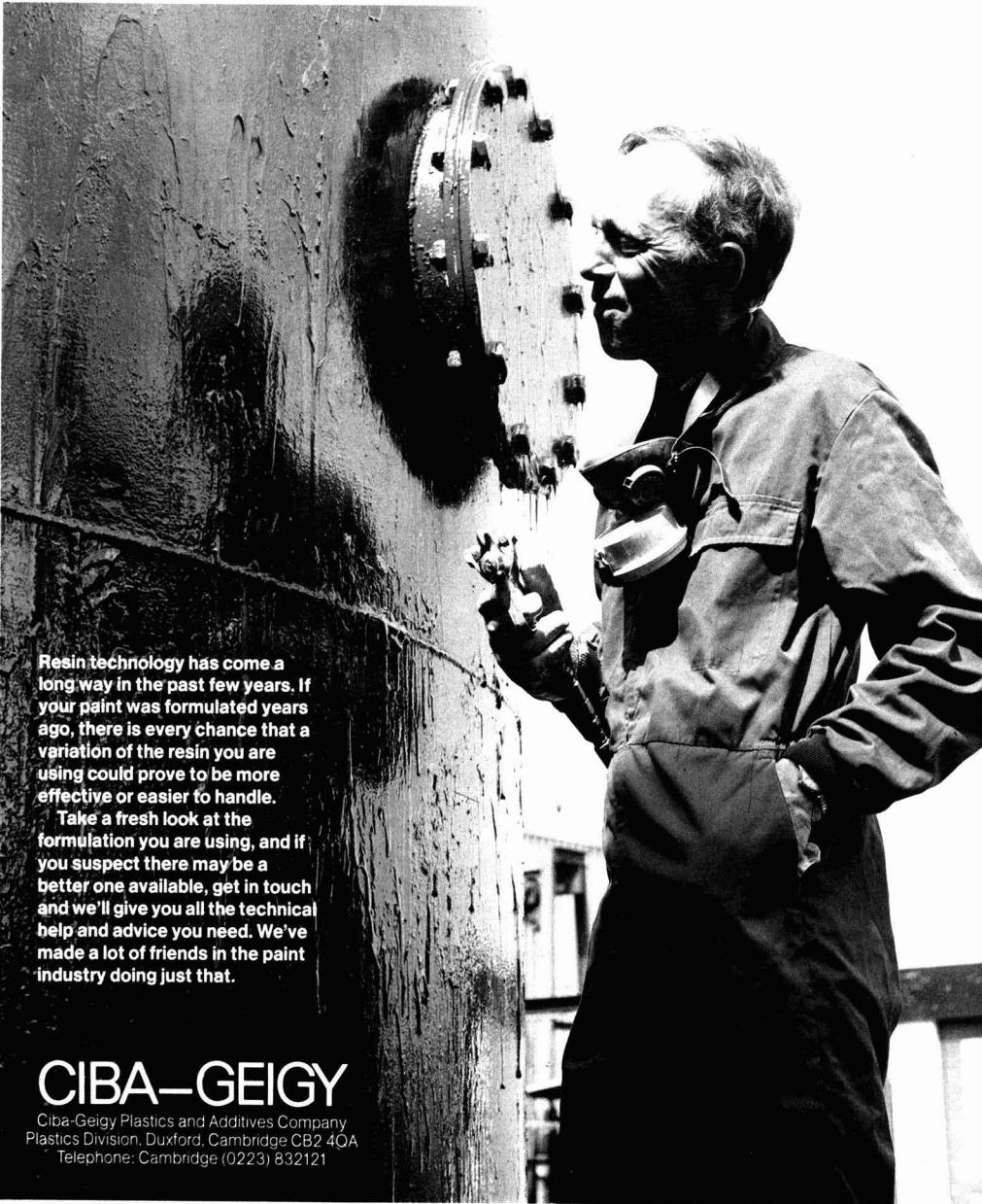
A meeting of the Transvaal Section was held on 26 July at the Sunnyside Park Hotel, Johannesburg, when Mr C. R. Chesman of Chelac Chemicals delivered a lecture entitled "Rare earth driers and the cobalt story". The meeting was sponsored by Protea Chemical (Pty) Ltd.

Mr Chesman introduced his talk by explaining that due to the large number of guests who were non-technical, he would not delve too deeply into the known chemistry of the subject, which had been detailed in his lecture given to OCCA in 1961.

The theory of the air-drying of paints and varnishes was briefly outlined. Paint driers are oil-soluble metallic soaps which, when added to resinous media, are capable of introducing oxygen by catalytic reactions. This results in the formation of hard dry films. The common metals used in various proportions were summarised and the normal metal/resin ratios detailed.

A background to the rare earth driers was given. Paint chemists had for many years been searching for a replacement for lead, especially in enamels for toys and paints for children's furniture. Work has been carried out on the uncommon metals, such as cerium, lanthanum, thorium, zirconium and vanadium. Cerium and zirconium were found to be useful, but only one company in South Africa is

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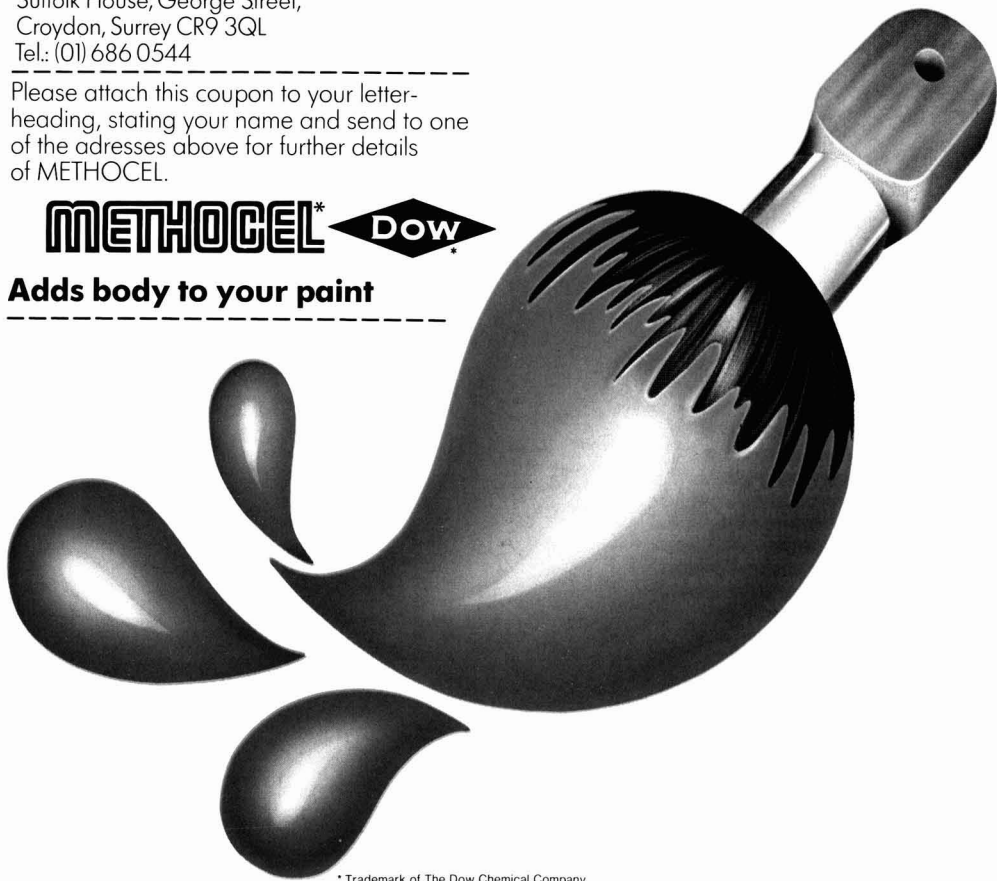
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using cerium in quantity, to replace lead and to reduce the amount of cobalt used.

Details of the metal to solid resin ratios were given and discussed. This indicated that low levels of driers based on cerium/cobalt mixtures could be incorporated in paint formulations.

As a result of the high price and shortages of cobalt, Mr Chesman explained that in conjunction with an officially recognised standards laboratory, he had carried out detailed tests on a metal combination (not disclosed) to replace cobalt. The work performed covered touch dry and

hard dry times, gloss and yellowing of a standard specified white paint and a specified gloss varnish. The results of the tests were most encouraging. The only difference between an equivalent amount of cobalt and the sample submitted was that cobalt gave a four hour touch dry time as compared with the mixed sample which gave a five hour touch dry time. All other results were equivalent. The point of the exercise was to introduce an equivalent drier to cobalt at approximately half the cost.

A vote of thanks was proposed by Mr Bosman.

P.A.J.G.

Information Received



Some of the range of bathroom ware which has been produced using Beetle resins

Beetle in the bathroom

Linfordware Ltd has developed and patented a new process for decorating and protecting acrylic and ceramic bathroom ware using Beetle polyester resins supplied by the Chemicals Division of BIP. Baths, basins and shower trays are made from vacuum formed 5 mm transparent acrylic shells, to the backs of which any desired patterns and colours are applied. Linear motifs, distinctive stripes or personal monograms or logos are applied as dry transfers to the shells, which are then coated with coloured acrylic resin.

After the shells have been decorated they are strengthened and stiffened with a

coating of glass fibre-reinforced Beetle type 874 polyester resin, and it is claimed that this process prevents delamination, a problem which has occurred in previous GPR-coated acrylic bathroom ware.

The shells are said to be so strong that they can be scrubbed, kicked or jumped on without causing damage. Because the decoration is applied to the backs of the shells and not to their exposed surfaces, it is protected against abrasion and fading.

Agents for Sauter

MSE Scientific Instruments Ltd, a member of the Fisons group, has been

appointed sole importers of Sauter Weighing Equipment. The new Sauter "KM" range, which covers programmed digital industrial weighing platforms that can be dormant, part of a conveyor system or free-standing, has recently been introduced and is now available. These industrial platforms are programmed for either gross net weighing, counting and weighing, mixture weighing with gross, net and total net weight totalising. Other equipment in the Sauter ranges cover both mechanical and electronic weighing from 30 g to 6000 kg.

Dynamic flow ovens

Following an agreement with Blu-Surf Inc. of Michigan, USA, the Chal Division of Wellman Incandescent Ltd are now able to incorporate the Blu-Surf patented Dynamic Flow oven into their complete finishing systems.

Dynamic Flow is a process in which a mixture of air and products of combustion are forced at high velocity against all surfaces of a product. The inside of the oven is constructed with alternate baffles and jet pipes, and Blu-Surf burners fire into an insulated plenum in the oven. A pressure blower then forces air through the many small orifices in the jet pipes at very high velocity, which because of the Venturi effect, pulls the hot flue gases from the cavity behind the jets and the air and hot gases blend together as they flow around the work. The result is a very rapid rate of heat transfer to the surface of the work.

Corrosion resistance with powder coatings

Glynwed Leisure are using powder manufactured by TI Drynamels Ltd to coat their range of domestic steel shower cabinets which were previously stove enamelled.

A vital requirement of the shower cabinet finish is that it must be durable under hot and wet conditions to prevent metal corrosion. The electrostatic application of powder means that all edges, holes and angles are covered, and results in a harder glossier coating.

New products

New laboratory exposure system

Atlas Electric Devices Co. have introduced, as a follow-up to their free-standing UVCON Cabinet, a compact bench top laboratory exposure system using alternate cycles of fluorescent ultraviolet light and condensation, which is known as the UV2 and is available in the UK through their agents, Westlairs Ltd. This unit provides a screening test for coatings, plastics and pigments which may be specifically affected by the UV portion of the sunlight spectrum, together with moisture penetration.



The new Atlas UV2 bench top laboratory exposure system

The Atlas UV2 retains all of the operating principles and controls, including the important lamp to sample geometry, of the Atlas UVCON. The system forms a simple and relatively inexpensive means of screening materials for degradation due to moisture and UV radiation as a guide to outdoor weatherability.

Reactive diluent

Eastman have developed a new reactive diluent for high solids enamel coatings called RG-82, which is claimed to reduce the level of enamel viscosity for efficient spray application, and which is also relatively non-volatile during the cure.

The low vapour pressure and reactive hydroxyl groups of RG-82 enable it to be cross-linked into melamine and isocyanate coatings, resulting in coatings with good hardness, gloss and gloss retention, and excellent resistance to colour change, solvents, weathering and humidity.

Typical formulations utilising RG-82 for melamine enamels and polyisocyanate cross-linked enamels are given in a leaflet available from Eastman.

Liquid crystal totaliser

Veeder-Root has available the new Series 7997 LCD Totaliser, which has advanced circuitry and a built-in power source rated for 10 years typical life. The unit is ideal for replacing existing electromechanical totalisers, and has operating speeds up to 3000 counts per minute.

The 7997 is designed for continuous duty counting of production units, machine cycles, revolutions and other in-house totalising applications. With appropriate input sensors, it also measures material length or volumes.



The Series 7997 LCD Totaliser from Veeder-Root

Digital thermometers

Channel Electronics (Sussex) Ltd have introduced the 7600 Series digital thermometers, which are suitable for bench top or laboratory work, to complement their range of hand-held instruments. The new Series covers the range -220°C to 1750°C , and can be supplied with a linearised recorder output socket and a built-in selector switch to control up to six thermocouples.

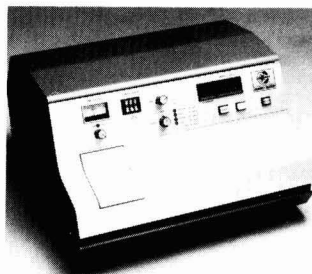
Stainless steel filter elements

Microfiltrex Ltd has released details of a full range of high-flow cleanable filters constructed from stainless steel, which are available in pleated or cylindrical format. A choice of welded, brazed or epoxy resin bonded assemblies are available to meet the numerous different applications of the elements, which when used for liquids have 100 per cent removal ratings in the range 1 to 280 micrometers.

Butyl reinforced sealant

The building chemical division of Blundell-Permoglaze has launched its first sealant, a butyl reinforced product formulated to give a long life, even without being overpainted. The sealant has been designed for multi-purpose application on exterior and interior work, such as for sealing, bedding in, pointing etc.

The product, which has been tested to BS 3712, is applied with a cartridge gun which is also available from Blundell-Permoglaze.



The Nano-Sizer particle sizer and counter from Coulter Electronics

Particle size measurement

Coulter Electronics Ltd, who have long standing expertise in particle counting and sizing in both medical and industrial fields, have announced an entirely new instrument for the measurement of very small particles in industrial laboratories.

The Coulter Nano-Sizer determines average particle size in suspensions and emulsions in the overall range 0.04–3.0 micrometers. This, together with an index of the width of the size distribution, is presented typically within 2–4 minutes, with no operator attention, calculation or calibration.

The Nano-Sizer combines the natural phenomenon of Brownian motion with auto-correlation spectroscopy of scattered laser light. For the analysis, no material properties of the sample such as shape or refractive index are required, but only the refractive index and viscosity of the suspending liquid.

The built-in micro-computer ensures that no calculations are required and the results are displayed digitally to three significant figures.

Portable infrared thermometer

Land Pyrometers Ltd have introduced the Cyclops, a new portable infrared thermometer which focuses the infrared radiation emitted from the surface of an object, amplifies the electrical signals produced by a pyro-electric detector, and displays a digital temperature reading in a viewfinder.

Two models cover the ranges 0 to 400°C (Cyclops 31) and 200 to 800°C (Cyclops 32), and Fahrenheit versions are also available. Accuracy is within 0.75 per cent plus one digit centigrade, and best operation is obtained at a range of between one and five metres.



The Cyclops portable infrared thermometer from Land Pyrometers Ltd

The pyro-electric detector used in the Cyclops avoids the practical difficulties of using photon detector devices at these relatively low temperatures, does not need to absorb a high proportion of the incident radiation in the form of heat as thermopile devices do, and avoids the elaborate zero-setting procedures necessary for thermistor systems. Accurate measurements can be made from very small targets; the target circle viewed by Cyclops is only 35 mm at a distance of one metre.

New from Perkin-Elmer

Hitachi/Perkin-Elmer have announced the addition of the S405A scanning electron microscope to the popular S400 range.

It offers high resolution (70 Angstroms) and high vacuum, 5×10^{-6} Torr. A complete compensation allowing correct and direct magnification readout in digital format plus many operational modes are available. Dual magnification is standard and automatic data display optional.

New Kremlin pump

The major problem with airless spraying of high solids zinc rich paints is the damage caused to the piston packings by the impaction of zinc particles, which results in excessive wear causing leaking and loss of efficiency.

Kremlin Spray Painting Equipment Ltd claim to have solved this problem for the first time with the introduction of their 10-102 pump, in which the paint is drawn from the paint container through the suction hose to the suction cylinder. The air motor, on the return stroke, then transfers the paint to the pressure cylinder, and the following stroke this paint is delivered to an Airmix spray gun. The advantage of this type of pumping system is that the piston is being continuously washed by the solvent, which prevents the build-up of zinc in the packings. Hardening is impossible because any paint leakage can only occur in an area where there is a solvent solution.

Pocket microscope

Cobonic Ltd has available a pocket sized powerful 30x magnifying tool with its own integral battery and illumination which has many applications, particularly in quality control.

Dosing/blending system

The Ranger Instrument Co. has developed an inexpensive dosing/blending system, originally designed to dispense pre-set amounts of oil and other lubricants into cars on a production line, which has been found to have applications in many other industries.

The device uses positive displacement meters to measure the liquid to within an accuracy of one per cent, and there is a wide range of bore sizes available which increases the flexibility of applications. Operation is by push button, and once the dialled amount of liquid has been dispensed, the unit automatically re-sets itself.

Conferences, courses etc.

Inklings

Coates Brothers Inks Ltd has recently published an authoritative review of all aspects of ink drying, in an extended version of the company's technical publication, *Inklings*. The review explains the methods used to dry printing inks and examines the latest techniques and their significance to the field of printing as a whole.

Measurements in chemistry

Roth Scientific has recently published a new edition of "Measurements in chemistry" which contains full technical specifications on a very wide range of high quality electrochemical titration equip-

ment, including the recently introduced Metrohm range.

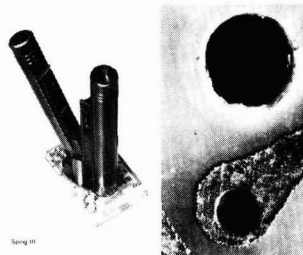
Low level nitrogen analysis

A new elemental analysis application study is available from Perkin-Elmer, which deals with the applications of the Model 240B Elemental Analyzer for low level nitrogen elemental analysis of crude oils, lubricating oils, and surfactants etc.

Epoxy powder coatings

R. H. Chandler Ltd has published a new booklet entitled "New epoxy powder coatings" which reviews the many advances which have been made in the six years since the first epoxy powder coating bibliography was published.

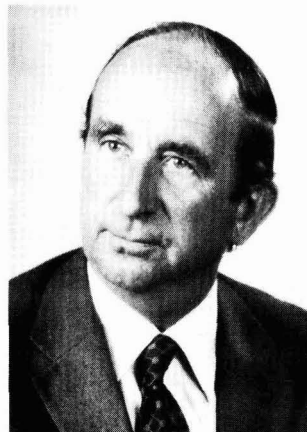
The bibliography provides statistics on powder consumption and examines in detail powder formulation, selection of curing agents and the best organic and inorganic pigments and extenders.



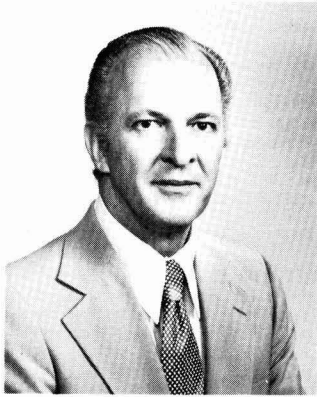
The powerful pocket-sized microscope from Cobonic Ltd

Appointments

Mr J. A. W. Gibson has been appointed Director and General Manager of Hythe Chemicals Ltd, based at the company's ethylene oxide and derivatives processing plant in Hampshire.



Mr J. A. W. Gibson



Mr A. A. Ostrowski

Engelhard's Minerals & Chemicals Division has appointed Mr A. A. Ostrowski as director of international sales for Europe. Previously international marketing manager, Mr Ostrowski now assumes responsibility for pigment, extender and sorbent products.

Mr W. H. Avery, formerly Managing Director of Croda operations in New Zealand, has been appointed Chief Executive of Croda Australia Group Ltd to succeed Mr P. S. Hudson, who is returning to the UK to take a new appointment with Croda.

Mr J. Neil has been appointed Technical Sales Co-ordinator for Mebon Paints Ltd, to provide technical liaison between customers and the factory. With 18 years experience of specifying paint, he is well qualified to provide customers with a high level of technical advice.



Mr J. Neil

Notes and News



OCCA-32 Exhibition

13-15 May 1980 Cunard International Hotel
Hammersmith, London W6

Many applications received from UK and overseas in enthusiastic response to new arrangements

**Closing date for applications
30 November 1979**

Since the Invitation to Exhibit at the 1980 OCCA-32 Exhibition was despatched in July, there has been a continuous stream of enquiries from companies and organisations which have expressed their intentions to participate at the Exhibition, and already the list of organisations both from the UK and overseas which have firmly booked space has grown to considerable proportions.

Amongst those which have already taken space, including many multinational organisations, are some that have not shown at the exhibition for several years, ensuring that the 1980 OCCA Exhibition will have something on display to interest everyone.

The closing date for applications to exhibit was given in the Invitation to Exhibit as 30 November 1979, and organisations wishing to participate at the Exhibition are urged to ensure that their applications are received by that date so that full consideration may be given to their preferences. Shortly after the closing date, the Exhibition Committee will be meeting to allocate space in the exhibition areas, and exhibitors will then be notified

of details of their positions and stand numbers. Any applications for space received after this initial allocation of space has taken place will be considered, and may be allocated any space remaining. Intending exhibitors are reminded, however, that space in the Cunard International Hotel is limited and that late applications will have to be accommodated around those sites already allocated.



The Cunard International Hotel

The new venue for the 1980 Exhibition will be the Cunard International Hotel, Hammersmith, London W6, and the main part of the Exhibition will be in two sections: on the ground floor, forming the entrance to the Exhibition, traditional style stands will be accommodated in the New Exhibition Hall where exhibitors of heavy machinery, plant and equipment will be located; on the first floor of the hotel, in the Queen Mary Suite, the stands will be of a simplified nature with the objective of allowing exhibitors to use modular display systems. These stands will have no platform, being erected directly on to the carpeted Suite floor, and so it will not be possible for heavy equipment to be displayed in this section.

Access between these two areas will be through the intermediate Mezzanine floor, where there are a number of rooms in the Armada Suite for companies to display free standing exhibits. These rooms were amongst the first to be requested, and have now all been allocated to companies, the deposits already having been received.

In addition there will be several suites and syndicate rooms on the third floor of the hotel, either for companies who wish to use this type of facility to exhibit, or for those who wish to have somewhere convenient to entertain their visitors in addition to their main stands elsewhere in the Exhibition. Already, several organisations have taken advantage of this opportunity to have a main stand for general enquiries, and the more private room for detailed discussions.

* * * * *

Companies intending to apply for these hospitality suites are reminded that although the main allocation of space in the Queen Mary Suite and the New Exhibition Hall will take place after the closing date, these suites and rooms are allocated upon application in strict order of receipt. Immediate application is, therefore, advised, as the number of these suites available is limited.

* * * * *

Dates and times

The thirty-second annual OCCA Exhibition, a three day event, will be open as follows:

Tuesday 13 May 1980 09.30 to 17.30
 Wednesday 14 May 1980 .09.30 to 17.30
 Thursday 15 May 1980 09.30 to 17.30

Theme for the Exhibition

Motif: The motif, designed by Robert Hamblin, uses the compass to symbolize the unique attraction of the OCCA exhibitions which annually draw exhibitors and visitors from numerous countries.



The aim of the Exhibition is the presentation of technical and commercial information relating to raw materials, plant and equipment used in the paint, polymer, printing ink, colour, adhesive and allied industries, both in manufacture, processing and application.

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



A view of part of the Queen Mary Suite at the Cunard International Hotel

Invitations to Exhibit

Invitations to Exhibit, giving details of the various types of exhibition facilities available at OCCA-32 have been despatched, together with application forms, to those companies who have exhibited at previous OCCA Exhibitions, or have requested information for the first time for OCCA-32. Any organisation which has not yet received a copy of the Invitation to Exhibit and wishes to do so, should write to the Director & Secretary at the address on the Contents page.

The Annual Exhibition

The OCCA Exhibition is recognised as the world's most important international exhibition for the surface coatings and related industries, and annually provides companies with the opportunity to display their products and services and discuss them with visitors from many parts of the world.

"... the accomplishments of the first day alone were enough to justify our participation in the Exhibit, and I should also point out that the subsequent days were even better ..."

In recent years, the Exhibition has regularly attracted visitors from over 50 countries, with a remarkably high proportion of top personnel from both the UK and overseas attending. The value of exhibiting need hardly be stressed when it is considered that it is possible, at this one annual event, to contact and maintain contacts with companies involved in the surface coatings industries from all over the world.

"Anyone visiting this exhibition (OCCA-31) could not have failed to be impressed by the spacious and comfortable layout and the ease with which one could identify individual stands. For those whose range of interests includes all of the varied displays, to be able to see at a glance individual stands and the personnel available for discussion and yet at the same time, while promenading, enjoy the many re-unions is recognised as another of the unique characteristics of this annual event. The atmosphere in this exhibition has never been bettered ... the deserved claim to be the international forum for the surface coatings industries."

"The exhibition was well attended and seemed to need all of the vast space in the crowded middle periods when so many overseas visitors dominated."

The British Ink Maker, May 1979

Official Guide

It is intended, as in previous years, to publish the *Official Guide* to the Exhibition several weeks in advance so that it may be sent to visitors to enable them to plan the itinerary for their visits. The *Official Guide* will contain descriptions of all the exhibits together with much other useful information for visitors, such as maps of the exhibition areas, details of facilities, travel information and an analysis of the exhibits.

Many thousand copies of the *Official Guide* will be printed and distributed on a world-wide basis. Advertising space will be available in this publication, and details of availability, rates, special positions etc., may be obtained from the Assistant Editor at the address on the Contents page.

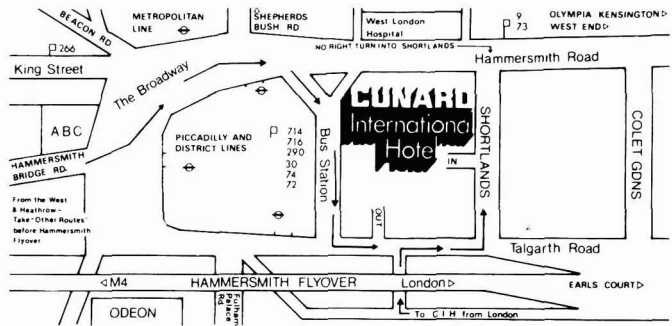
Travel arrangements

The Exhibition Committee has decided to move the venue of the 1980 Exhibition to the Cunard International Hotel, as it is felt that the more central site and greater range of types of exhibiting facilities it offers will be welcomed both by exhibitors and visitors to the Exhibition.

"... we obtained countless inquiries from representatives from nearly 30 different countries"

The Hotel has a selection of restaurants, shopping facilities and bars. There is a coffee shop immediately adjacent to the Queen Mary Suite (which itself contains a bar), an Exhibitors' Lounge on the Mezzanine floor, and there are plans for a coffee bar to be erected in the New Exhibition Hall for the Exhibition.

The Hotel is situated near Hammersmith Station on the Piccadilly Underground Line between Heathrow Airport and the centre of London. Visitors from overseas may board the Piccadilly



Line in the Airport complex, which will take them direct to Hammersmith Station or to central London where they may be staying. Hammersmith Station is also served by the Metropolitan and District Underground Lines (the latter of which connects to Victoria Station for those arriving at Gatwick Airport). The Hotel is adjacent to the Hammersmith flyover on the M4 Motorway which links Heathrow Airport by road. Car parking at the Hotel is limited, but there is a large NCP car

park close by in King's Mall off King Street.

Admission to the Exhibition will be free, and visitors will be asked to complete registration cards which will be available from the Association with copies of the *Official Guide* in advance. Copies of the *Official Guide* will be charged at £1.50 each, and both registration cards and copies of the *Official Guide* will also be available at the entrance to the Exhibition.

Berger Grand Prix

Shown below is Scott Autrey (centre), currently riding with Exeter's speedway team, collecting the Berger Speedway Grand Prix Trophy and a cheque for £1250 from Ray Malt, Berger's general manager decorative trade paints.

Second in the Grand Prix, held at Wimbledon in August, was Cradley Heath's Bruce Penhall (right) with Reading's John Davis (left) in third place. Twenty top riders qualified for places in the final after a season's qualifying heats at tracks around the UK. The Grand Prix was backed jointly by Berger's Vehicle Refinishing and Decorative Trade divisions, and organised in conjunction with the *Daily Mirror*.



News of Members

Mr W. F. A. Thorpe, an Ordinary Member attached to the Midlands Section and a Fellow in the Professional Grade, has retired from his position as Director of Research for Arthur Holden & Sons Ltd. Mr Thorpe joined the company in 1934 and has contributed greatly to the technical strength and growth of the company over the years. He was also closely connected with the development of the new University of Aston.

Call for papers

The Plastics and Rubber Institute is organising a one-day conference for Wednesday 12 November 1980 with the theme

"Plastics and paints against corrosion", and has issued a call for papers to cover the subjects of the economics of corrosion, the mechanisms of protection, and the replacement of traditional building materials.

D. G. Soar

It is with regret that the death in August of Mr D. G. Soar, a former member of the London Section Committee, is recorded. It is hoped that an obituary will appear in a future issue of the *Journal*.

ICI Paints Division Blaze

An electrical fault is thought to have started a fire in a building used to prepare powder for metal cleaning at the ICI Paints Division at Slough on midday Thursday 11 October. At the height of the blaze, a thick cloud of smoke drifted from the building, and police evacuated two nearby housing estates due to the possibility of release of toxic gases.

ICI's own fire crews were the first to tackle the fire as it spread through the building, only yards from the adjacent old Slough gas works, but within minutes, more than 50 Berkshire firemen were at the scene, including a special incident tender designed for chemical and toxic substance fires.

The firemen, wearing special contamination suits, had to be washed down as they left the scene, and Thames Water Authority scientists began testing all sewers and drains in the area to ensure that no chemicals had seeped into the drainage system. None were found. The fire was brought under control within half an hour.

A statement issued by ICI was circulated to local residents, stating that there had been a risk of a small quantity of toxic gas being released—the building concerned had contained chemicals such as potassium ferricyanide, chromic acid and potassium fluoro zirconate. Tests had not detected any such toxic gas, however, and the smoke had been due to the plastic roof of the building burning.

ICI have not said how much the fire would cost the company, but production in other parts of the plant continued even during the height of the blaze.

Midlands Section

Shown (right) at the annual Dinner Dance of the Midlands Section held on Friday 28 September are (l-r): Dr F. M. Smith (President), Mrs Smith, Mrs Burns, Mr J. A. Burns (Chairman, Midlands Section).



Register of Members

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

Ordinary Members

BEALE, MICHAEL ANTHONY, 1 Gossops Green Lane, Gossops Green, Crawley, West Sussex. (*Thames Valley*)

HUDSON, TREVOR ANTHONY, OBS Machines Ltd, Unit 34 Heathfield, Stacey Bushes, Milton Keynes MK12 6HR. (*Thames Valley*)

LAWLOR, DAVID PETER, 9 Littlecote, Petworth, Sussex GU28 0EF. (*London*)

MITCHELL, WAYNE MARTIN, BSc, Anchor Chemicals Co Ltd, Clayton Lane, Clayton, Manchester M11 4SR. (*Manchester*)

POWELL, MARK TERENCE, Grad RIC, 5 Welsford Road, Stapleton, Bristol BS16 1BS. (*Bristol*)

RUSTON, BARRY KENNETH, MRIC, 37 Hartsbourne Drive, Halesowen, West Midlands B62 8ST. (*Midlands*)

SILK, BERNARD CLIVE, 66 Cornwall Road, Greenhill, Herne Bay, Kent. (*London*)

Associate Member

DICKINSON, MARTIN, 118 Paterson Place, Shephed, Loughborough, Leicestershire. (*Manchester*)

Registered Students

EARLEY, JULIA, 85 Lyndhurst Avenue, Higher Irlam, Manchester M30 6HS. (*Manchester*)

MANKU, KAMALJIT SINGH, 117/653 Pandu Nagar, Kanpur 208005, India. (*General Overseas*)

TURNER, ANDREW, 49 Eastleigh Road, Heald Green, Cheadle, Cheshire. (*Manchester*)

Forthcoming Events

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

November

Thursday 1 November

Newcastle Section: "Antifoulings" by J. D. Symonds of International Marine Coatings, in the Students Common Room, St. Mary's College, Elvet Hill Road, Durham, commencing at 6.30 p.m.

Monday 5 November

Hull Section: "Instrumental colour

control in the paint industry" by Miss S. Williams, Director (Applications Department), Instrumental Colour Systems, at the George Hotel, Land of Green Ginger, Hull, commencing at 6.30 p.m.

Tuesday 6 November

Scottish Section: "The effect of instrumentation on management" by J. M. Scadlock of J & P Coats UK Ltd. Joint meeting with the Society of Dyers & Colourists, at the North British Hotel, Glasgow, commencing at 7.30 p.m.

West Riding Section: "The biology of paint and paint films" by E. Allsop of the University of Aston, at the Mansion Hotel, Roundhay Park, Leeds 8, commencing at 7.30 p.m.

Thursday 8 November

Thames Valley Section: "The pigmentation of non-polluting coatings" by T. Entwistle of British Titan Products Ltd, at the Beaconsfield Crest Motel (White Hart), Aylesbury End, Beaconsfield, Bucks, commencing at 6.30 p.m.

Friday 9 November

Irish Section: Annual Dinner Dance, at the Clarence Hotel, Dublin, commencing at 8.30 p.m.

Monday 12 November

Manchester Section: "Photography—Do you need help?" by N. Webster of Nicholas Webster Photography Ltd, at the Woodcourt Hotel, Sale, commencing at 6.30 p.m.

Wednesday 14 November

Scottish Section—Eastern Branch: "The language of colour" by J. Hunter of the Design Department, Scottish College of Textiles, at the Maybury Road House, Maybury Road, Edinburgh, commencing at 7.30 p.m.

Thursday 15 November

Midlands Section: "Solvent system design" by R. L. Mawer of Shell Chemicals Ltd, at the Calthorpe Suite, County Cricket Ground, Edgbaston, Birmingham, commencing at 6.30 p.m.

Wednesday 21 November

London Section: "Pigments". Day meeting at the Thames Polytechnic, Woolwich, SE18, commencing at 10.00 a.m. *Details to be announced.*

Manchester Section: Students' Lecture, "Weatherability of surface coatings" by E. Oakley of BTP Ltd, at the Manchester Polytechnic, New Administration Building, All Saints, commencing at 4.30 p.m.

Friday 30 November

Bristol Section: Ladies' Evening. "Furs" by D. Bauers of F. K. Bauers of Bristol, at the Royal Hotel, Bristol, commencing at 7.15 p.m.

December**Monday 3 December**

Hull Section: "Adhesion of surface coatings: Fact and fiction" by M. Wilkinson of Blundell-Permolaze, Decorative Division, at the George Hotel, Land of Green Ginger, Hull, commencing at 6.30 p.m.

Tuesday 4 December

West Riding Section: "Quality assurance in process industries, with special reference to paint" by D. J. Murray, at the Mansion Hotel, Roundhay Park, Leeds 8, commencing at 7.30 p.m.

Wednesday 5 December

Scottish Section—Eastern Branch: "Fire and hygiene hazards associated with the surface coatings industry" by W. W. Ashcroft-Hutton and R. U. Khan of the Health & Safety Executive, Edinburgh, at the Murrayfield Hotel, 18 Corstorphine Road, Edinburgh, commencing at 7.30 p.m.

Thursday 6 December

Newcastle Section: "Setting up a paint company" by J. R. Bourne of Mebon Ltd, at the Students Common Room, St. Mary's College, Elvet Hill Road, Durham, commencing at 6.30 p.m.

Thames Valley Section: "Dispersants for emulsion paints" and a film on dispersants by A. S. Doyle of IMC Chemie GmbH, at the Beaconsfield Crest Motel (White Hart), Aylesbury End, Beaconsfield, Bucks., commencing at 6.30 p.m. for 7.00 p.m.

Friday 7 December

Irish Section: "Progress in printing inks" by G. H. Hutchinson, Technical Director, Croda Inks, at the Clarence Hotel, Dublin, commencing at 8.00 p.m.

Thursday 13 December

Scottish Section: "100 years of colour chemistry" by I. D. Ratee, School of Colour Chemistry, University of Leeds, at the Albany Hotel, Glasgow, commencing at 6.15 p.m.

Friday 14 December

Manchester Section: "Matting silicas and their use in UV cured systems" by R. C. Chapman of Joseph Crosfield & Sons Ltd, at the Manchester Polytechnic, New Administration Building, All Saints, commencing at 6.30 p.m.

January 1980**Thursday 3 January**

Newcastle Section: Chairman's lecture, by J. Clark of BTP Tioxide Ltd, at the Students Common Room, St. Mary's College, Elvet Hill Road, Durham, commencing at 6.30 p.m. *Details to be announced.*

Monday 7 January

Hull Section: At the George Hotel, Land of Green Ginger, Hull, commencing at 6.30 p.m. *Details to be announced.*

Thursday 10 January

Bristol Section: "Coal as a replacement

for oil in the chemical industry". Joint meeting with the Birmingham Paint Varnish & Lacquer Club, to be held in Birmingham. *Details to be announced.*

Friday 11 January

Scottish Section: Annual Dinner Dance, details to be announced.

Monday 14 January

Manchester Section: "Powder, high solids, water" by M. Bus, L. C. Kwakman and A. J. Van Der Werff of Scado BV, at the Crest Motel, Bolton, commencing at 6.30 p.m.

Thursday 17 January

London Section: "International standardisation of methods of test for paints" by H. A. Hipwood, MQAD, at the Princess Alice, Romford Road, Forest Gate E7, commencing at 6.15 p.m. To be followed by a buffet supper.

Scottish Section: "Applied colour measurement in surface coatings" by D. A. Plant, at the Albany Hotel, Glasgow, commencing at 6.15 p.m.

Friday 18 January

Irish Section: Ladies' Evening. Travel film and Cheese and Wine evening, at the Clarence Hotel, Dublin, commencing at 8.00 p.m.

Midlands Section: "The role of the technologist in marketing" by R. Fidler of International Paint Ltd, at the Birmingham Chamber of Commerce & Industry, commencing at 6.30 p.m.

Wednesday 23 January

Manchester Section: Student Lecture "Physical testing of paints and coatings" by R. H. Wallington of MQAD, MOD, at the Manchester Polytechnic, New Administration Building, All Saints, commencing at 4.30 p.m.

Scottish Section—Eastern Branch: At the Murrayfield Hotel, 18 Corstorphine Road, Edinburgh, commencing at 7.30 p.m. *Details to be announced.*

Thursday 24 January

Thames Valley Section: Works visit to Rondec Ltd, Egham, Surrey at 7.00 p.m. Demonstration of screen printing for labelling.

Friday 25 January

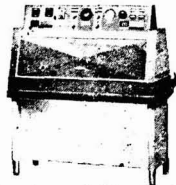
Bristol Section: "Inks for packaging" by H. J. Duffin of Irlam Inks Ltd, at the Royal Hotel, Bristol, commencing at 7.15 p.m.

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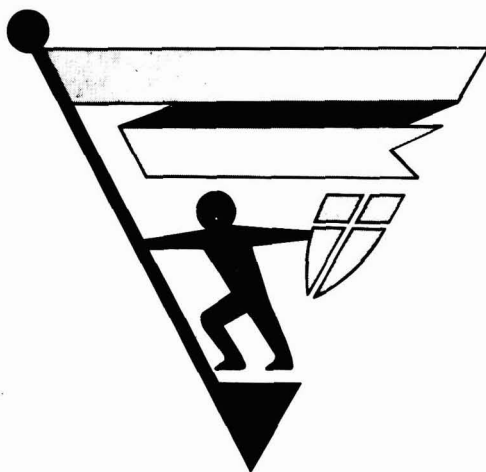
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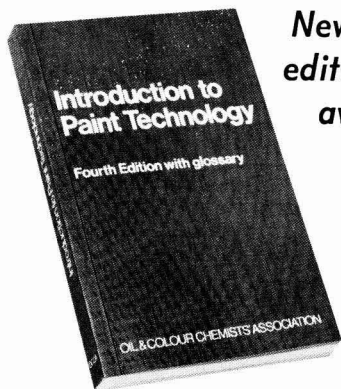
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The Association organises an international Conference every two years and preprints of the papers are prepared for delegates. A strictly limited number of the following are available to those who wish to have the complete bound sets of papers.

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Editor: DR. S. PETER PAPPAS

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The unique balance between theoretical and practical presentations, makes this book a vital part of any research and development organization currently involved (or desiring to get involved) in the rapidly growing technology of uv curing. Contents of this book are not found in any other publication.

ABOUT THE EDITOR

Dr. S. Peter Pappas, Professor of Chemistry at North Dakota State University, received his B.A. degree from Dartmouth College in 1958 and his Ph.D. degree in Chemistry from the University of Wisconsin in 1962.

Dr. Pappas has authored (or co-authored) over 30 journal articles on synthesis, reaction mechanisms, photochemistry, and coatings technology, including a recent review paper on photochemical aspects of ultraviolet curing. His papers on the photochemistry of pigments as related to chalking and uv curing, received first prize in the Roon Award Competition, sponsored by the Federation of Societies of Coatings Technology in 1974, 1975 and 1976.

ABOUT CO-AUTHORS

In preparation of "Ultraviolet Curing: Science and Technology" Dr. Pappas has been assisted by several internationally recognized authorities from major industrial firms. The editor and each co-author have contributed at least one chapter, in their respective field of specialty, to "UV Curing: Science and Technology." Co-authors and their affiliations are listed below.

Robert W. Bassemir, Sun Chemical Corporation
Graphic Arts Laboratories

Anthony J. Bean, Sun Chemical Corporation
Graphic Arts Laboratories

Dr. James V. Crivello, General Electric Company
Research and Development Center

Dr. Eugene D. Feit, Bell Laboratories

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Dr. James E. Moore, General Electric Company

Research and Development Center

Dr. S. Peter Pappas, Polymers and Coatings Department

North Dakota State University

Dr. Zeno W. Wicks, Jr., Polymers and Coatings Department

North Dakota State University

TABLE OF CONTENTS

The following represents the table of contents:

1. PHOTINIATION OF RADICAL POLYMERIZATION
2. PHOTOINITIATED CATIONIC POLYMERIZATION
3. EFFECTS OF PIGMENTATION ON UV CURING
4. LIGHT SOURCES
5. CALORIMETRIC ANALYSIS OF UV CURABLE SYSTEMS
6. RESIN DESIGN
7. UV CURING OF COATINGS
8. UV CURING OF PRINTING INKS
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Mr Gordon Holt, a past Chairman of the Thames Valley Section, has recently left the paint industry and has started a new career in pharmacy. He would like to convey his good wishes for the future to all his many friends in the industry, both at home and overseas.

West Riding Section Annual Dinner Dance

The Annual Dinner Dance of the West Riding Section will be held on Friday 30 November 1979 at the Crown Hotel, Harrogate.

Further details from the Hon. Social Secretary, Mr N. Cochrane, The Coach House, Red Holt, Hainsworth Wood Road, Ingrow, Keighley, West Yorks. BD21 5BJ.

London Section PIGMENTS

The London Section is organising a day meeting in association with the Thames Polytechnic on Wednesday 28 November 1979 on the subject of "Pigments". The meeting will commence at 10.00 a.m. at the Thames Polytechnic, Wellington Street, Woolwich, London SE18.

The registration fee (£10.00 for members; £15.00 for non-members) includes morning coffee, lunch with sherry and wine, and afternoon tea.

The programme will include the following lectures:

"Health and safety aspects" by Mr B. F. Gilliam, Cowan Colours Ltd.

"Pigmentation of paints—Current theories of dispersion" by Mr G. A. Gunn, Courtaulds Ltd, Synthetic Fibres Laboratory.

"Pigmentation of printing inks" by Mr R. Hagger, Usher-Walker Ltd.

"Pigmentation of plastics" by Mr F. J. Morpeth, Foscolor Ltd.

"The colour makers view" by a BCMA spokesman.

Further details may be obtained from the Hon. Secretary, London Section, or from Dr R. C. Denney at the Thames Polytechnic at the above address.

Applications must be received by 21 November 1979.

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INDEX TO ADVERTISERS

B			M	
Banner, Samuel, & Co. Ltd	xi		Mastermix Engineering Co. Ltd	iii
C			Mickle Laboratory Engineering Co. Ltd	iv
Ciba-Geigy Plastics & Additives Co.	ix			
Crosfield, Joseph, & Sons Ltd	Inserts		N	
D			Nicholson's (Overalls) Ltd	vi
Dow Chemicals-Methocel	x		O	
E			OBS Machines Ltd	vi
ECC International	i		Q	
G			Q-Panel Co.	xi
Glen Creston Machinery Ltd	Cover		S	
H			Sachtleben Chemie GmbH	viii
Highgate & Job Ltd	iv		Sanyo-Kokusaku Pulp Co. Ltd	iii
I			Sheen Instruments (Sales) Ltd	Cover
ICI Ltd-Organics Division	vii		Sub-Tropical Testing Service Inc.	Cover
J				
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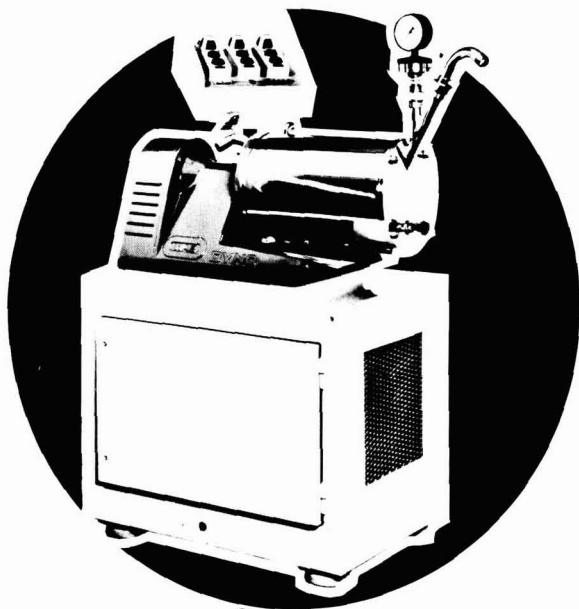
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