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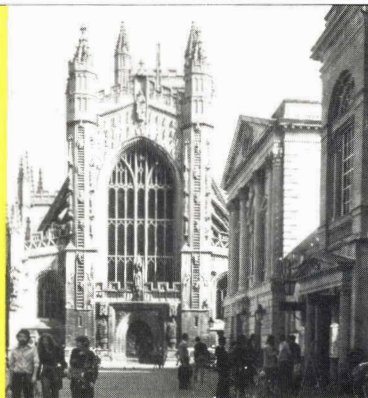
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A. J. Burgess, D. Caldwell and J. C. Padget

Skin irritation due to hexanediol diacrylate; problems in the evaluation
of results from animal studies
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Tel: 01-908 1086
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Transactions and Communications

A new approach to the design of latex paints for the protection of steel

By A. J. Burgess, D. Caldwell and J. C. Padgett

ICI Mond Division, Runcorn Heath, Cheshire WA7 4QF, England

Summary

The objective of the work described in this paper was to prepare and evaluate a high performance air drying anti-corrosive primer paint which would overcome many of the disadvantages associated with current aqueous anti-corrosive systems. The design of a vinyl acrylic copolymer latex and its formulation into an anti-corrosive primer is described and discussed. The vinyl acrylic copolymer latex gave a film which was highly impermeable to water. It was shown that formulation additives such as stabilisers and thickeners could be selected such that the intrinsically good barrier properties of the copolymer were essentially retained. The vinyl acrylic copolymer latex was formulated into a primer paint at an acidic pH of 5, in order to

avoid the dehydrochlorination reactions which occur with chlorine containing polymers in the alkaline pH region. The performance and properties of this acidic paint based on the vinyl acrylic copolymer latex were compared with those of conventional anti-corrosive latex paints. The vinyl acrylic system exhibited an excellent balance of mechanical properties. In accelerated testing the vinyl acrylic system performed substantially better than the conventional latex systems and a selected solvent based proprietary alkyd system. Exterior exposure trials are in progress and to date the vinyl acrylic system is giving superior performance to the latex systems and comparable performance to the alkyd system.

Keywords

Types and classes of coatings and allied products

corrosion resistant coating
primer

Raw materials for coatings

binders (resins etc.)

latex

Processes and methods primarily associated with drying or curing of coatings

air drying

Un nouvel abord à l'élaboration des peintures-émulsions pour la protection de l'acier

Résumé

Le but des travaux décrits dans cet exposé était la préparation et l'évaluation d'une peinture primaire anti corrosion de haut rendement, séchant à l'air, qui pourrait surmonter en grande partie les inconvénients des actuels systèmes aqueux anti corrosion. On a décrit et discuté l'élaboration d'un latex à base d'un copolymère acrylique/vinyle chloré et son incorporation dans la composition d'une peinture primaire anti corrosion. Le copolymère acrylique/vinyle chloré donnait des films qui étaient extrêmement imperméable à l'eau. On a démontré que les adjuvants tels que stabilisateurs et épaississants peuvent être choisis de sorte que les bonnes caractéristiques de barrage du polymère étaient largement retenues. Le latex du copolymère acrylique/vinyle chloré était incorporé dans une peinture primaire à pH 5, afin d'éviter les réactions de déshydrochlorina-

tion qui se produisent à la région alcaline dans le cas des polymères chlorés. Le rendement et caractéristiques de cette peinture acide à base du latex du copolymère acrylique/vinyle chloré ont été comparés auprès de ceux des peintures-émulsions anti corrosion habituelles. Le système acrylique/vinyle chloré présente un excellent bilan de caractéristiques mécaniques. Au cours des essais accélérés le système acrylique/vinyle chloré s'est comporté fort mieux que les systèmes émulsionnés habituelles et également qu'une peinture alkyde de commerce. Les essais de résistance aux intempéries sont en train et à présent le système acrylique/vinyle chloré donne un rendement supérieur à ceux des systèmes émulsionnés et comparable à celui d'un système alkyde.

Ein neue Weg zur Ausführung der Stahlschutzdispersierfarben

Zusammenfassung

Das Ziel der Untersuchung die in diesem Aufsatz beschrieben wird, war die Herstellung und die Auswertung eines luft-trocknenden Hochleistungskorrosionsschutzgrundanstrichstoffs der manche der mit gegenwärtigen wässrigen Korrosionsschutzsystemen verbundenen Nachteile überwinden würde. Der Bauart eines Vinylchlorid/Acrylcopolymerlatex und seine Einverleibung in einem Korrosionsschutzgrundanstrichstoff werden beschrieben und diskutiert. Das Vinylchlorid/Acrylcopolymer brachte höchstwasserundurchlässige Filme hervor. Es wurde gezeigt dass solche Additive als Stabilisatoren und Thixotropierungsmittel gewählt werden könnten, so dass, im

grossen und ganzen, die guten Sperrereigenschaften des Copolymers behalten wurden. Das Vinylchlorid/Acrylcopolymerlatexsystem wird zu einem pH-Wert von 5 in einem Grundanstrichstoff einverleibt, um die Entsalzsäuerungreaktionen zu vermeiden, die zu alkalischen pH-Werten im Falle Chlorpolymeren passieren. Die Leistung und die Eigenschaften dieses säurehaltigen und auf Vinylchlorid/Acrylcopolymerlatex gegründeten Anstrichstoffs wurden mit jenen konventioneller Korrosionsschutzdispersierfarben verglichen. Das Vinylchlorid/Acrylsystem zeigte eine ausgezeichneten Bilanz von mechanischen Eigenschaften. Bei

Kurzbewitterungsproben war die Leistung des Vinyl/Acrylsystems wirklich besser als der konventionellen Latexsysteme und als eines geschützten auf Lösungsmittel gegründeten Alkydsystems. Bewitterungsproben sind in Gang

und das Vinylchlorid/Acrylsystem bringt eine bessere Leistung als die Latexsysteme und eine vergleichbare Leistung dem Alkydsystem hervor.

Introduction

Air drying paints have been used for many centuries initially for decorative purposes but more recently for the protection of substrates. Throughout this period change has taken place as new materials such as resins, pigments and solvents have become available, or new processes have been developed requiring specific paint characteristics and as the need has arisen for more sophisticated systems to afford protection under severe conditions. With the advent of vinyl type monomers and the development of aqueous polymerization techniques synthetic latices became available to the paint industry. Initially latices were developed for use in domestic paints, particularly for interior use, where decorative qualities are of importance. Later developments included air drying water based paints for the more demanding area of steel protection which has to date been mainly the preserve of solvent borne coatings.

The reasons for a move to develop water based paints as protective coatings for use in situations where solvent based paints are currently used can be attributed to a number of motivating factors. These include air pollution restrictions, increasing cost and possible restricted availability of solvents, reduced personal exposure to solvents, reduced fire hazard which can result in lower costs and greater safety, easier waste disposal, and the ecological acceptability of water based compositions.

In this paper a new approach to latex and paint formulation is outlined with the specific aim of providing air drying coatings for the protection of steel.

Current latices on steel

Ref. 1

The air drying latices available for use in paints for steel are principally copolymers where the vinyl monomers may comprise alkyl acrylates, methacrylates, unsaturated carboxylic acids, styrene and in some cases acrylonitrile. The monomer combination and copolymer molecular weight will affect the mechanical properties and performance of the resultant coatings.

Although advances have been made in the development of latices and paint formulated from them for air drying applications to steelwork, such paints have achieved small penetration in this area up to the present time, and this could be due to a number of recognised effects:

Flash rusting	This is an unsightly spotty effect that may be evident very soon after paint application.
Early rusting	Similar effect to flash rusting but occurring after the film is touch dry but not fully coalesced ¹ .
Rheology	Inferior to solvent based coatings, resulting in poor levelling particularly when brush applied.

Drying characteristics This will depend on the humidity and air flow of the environment and the dew point. The ambient temperature is important as there will be a lower limit for film formation.

Adhesion Adhesion to metal surfaces and also intercoat adhesion has been commented on as less acceptable than with solvent borne paints.

Permeability Water and oxygen permeabilities of films from current latices are generally higher than solvent cast films.

Accelerated testing In accelerated tests such as salt spray and water soak less than satisfactory results have been obtained.

Permeability and adhesion considerations play an important part in the authors' approach to latex design. Elimination of flash and early rusting and satisfactory performance in accelerated tests have been prime targets for paint formulation in this work.

Permeability considerations

Refs. 1-5

The primary function of paint in an industrial or marine environment is to ensure that the structures are adequately protected against corrosion.

Paints must therefore have good adhesion, resistance to a wide range of chemicals and excellent weathering properties. For corrosion to occur, anodic and cathodic reactions must be possible at the metal surface, e.g. on steel:



It is apparent therefore that corrosion would not occur if steel could be coated with a film that was completely impervious to water or to oxygen, since both water and oxygen are required for corrosion reactions.

Calculations have indicated that typical paints are so permeable to water and oxygen that they cannot inhibit corrosion by preventing either from reaching the metal surface¹. However, the passage of charge across the polymer metal interface is also necessary for corrosion reactions, and paint films could suppress corrosion by virtue of their high electrical resistance.

It is known that the rate of ionic transport through hydrophilic membranes is critically dependent on the water content of the membrane because that quantity determines the diffusion characteristics. In addition it has been shown that small amounts of absorbed water can

markedly affect film properties and ionic permeability³. Also, it has been suggested that there is a direct relationship between conductivity of coatings and the rate of corrosion⁴.

The current generation of air drying emulsion polymers sold as binders for anti-corrosive paints are generally regarded as being inferior in performance when compared to the best solvent borne systems. It is proposed that the reasons for this inferior performance are:

- (i) Due to the choice of co-monomers the emulsion polymers provide a poor barrier to the diffusion of water and oxygen.
- (ii) The polymerization techniques employed involve the use of relatively high concentrations of surfactants and/or what are generally known as colloids (typically 2-4 per cent on latex solids) to ensure colloid stability during polymerization. It has been shown⁵ that colloids (e.g. polyvinyl alcohol, hydroxyethyl cellulose) added during polymerization can form graft copolymers with the monomers employed in the polymerization process and give a cross linked grafted outer surface to the polymer particles which impairs particle coalescence during the film forming process, with a resulting deleterious effect on the barrier properties of the resultant film. It is also generally considered that the typically high concentrations of surfactant/s employed in conventional emulsion polymerization can, unless carefully selected, increase the water sensitivity of the final film.

Polymer design

Refs. 6-10

The object of the work described in this paper was to attempt to overcome the above limitations by:

- (a) Synthesising an emulsion copolymer with a low permeability to water and oxygen, and which at the same time provided good adhesion, a good balance of mechanical properties and resistance to a range of chemicals.
- (b) Performing the polymerization essentially in the absence of colloids and in the presence of a minimum quantity of surfactant, whilst at the same time

carefully selecting the paint formulation additives (notably stabiliser, thickener and dispersant) such that the benefits of using a polymer possessing intrinsically good barrier properties were not lost by the formulation of the polymer into a paint.

A good starting point for the design of a binder for an aqueous anti-corrosive paint might be to base the polymer on a solvent borne polymer binder which is known to give good performance in the field. Such a polymer is chlorinated rubber with its low permeability to oxygen and water^{6,7}, good adhesion and good resistance to a wide range of chemicals. It was decided therefore to investigate high chlorine content air drying latex polymers.

In general air drying emulsion copolymers are based on at least one "soft" monomer and at least one "hard" monomer. It is then possible to exercise control over copolymer T_g, and hence MFT, by varying the co-monomer ratio. Typical examples of the main chemical classes of copolymer currently used as the basis for anti-corrosive emulsion paints are summarised in Table 1. It is apparent that in all cases at least one of the principal co-monomers is an ester of acrylic or methacrylic acid. These co-monomers are known to produce polymers having relatively high permeability to water⁷ and hence the high water vapour permeability values shown in Table 1 for latex cast films of these polymers is to be expected. When vinyl chloride and vinylidene chloride were used as the principal hard and soft monomers respectively, a copolymer with a very low water vapour permeability relative to that of the conventional emulsion polymer types was obtained, whilst as might be expected, an intermediate value was obtained for the VDC/acrylate/methacrylate copolymer.

The acrylate modified vinyl chloride-vinylidene chloride copolymer referred to in Table 1 is Haloflex 202, one of a range of copolymer latices developed in ICI Mond Division Laboratories⁸. This will be referred to as a vinyl acrylic copolymer throughout the present paper. The acrylic co-monomer was introduced in order to exercise fine control over film formation characteristics, and was present at a concentration selected such that the good barrier properties of the VC-VDC class of copolymer were retained. A further function of the acrylic monomer was to split up VDC sequences in the polymer chain since the presence of such sequences tends to adversely affect the thermal stability of this class of polymer. The average VDC sequence length was also minimised by performing

Table 1
Water vapour permeability of latex films

Hard monomer	Soft monomer	wt ratio hard monomer/ soft monomer	Water vapour permeability g/25μm ² /day
Styrene	2-ethyl hexyl acrylate	55/45	920
Methyl methacrylate	butyl acrylate	50/50	1270
Acrylonitrile	butyl acrylate	45/55	1290
(Butyl acrylate + methyl methacrylate)	vinylidene chloride	55/45	200
Vinyl chloride	vinylidene chloride	34/66	12*
Vinyl chloride	vinylidene chloride + acrylate		11†

Water vapour permeabilities determined at 38°C, 90% RH according to BS 3177.

*Data from Reference 7 for solvent cast film

†Haloflex 202

the polymerization in such a way that a homogeneous polymer of the desired composition was formed throughout the polymerization, thus avoiding the formation of a VDC rich polymer (which has an increased probability of containing long VDC sequences) at any stage of the polymerization. The polymer also contained a small proportion of a co-monomer introduced to improve the adhesion characteristics of the film to various substrates.

A good demonstration of the effect of the presence of chlorine in a copolymer was provided by a series of vinyl acrylic copolymers. Figure 1 shows the pronounced decrease in water vapour permeability with increasing chlorine content obtained for films cast from solution in toluene. Again, the very low water vapour permeability of the vinyl acrylic copolymers is apparent, especially in the high chlorine content region.

In practice the rate of liquid water permeation is also relevant. It might be expected that there would be no difference between water liquid and water vapour permeation rates, and indeed Vanderhoff⁹ showed that for a 70/30 vinylidene chloride/n-butyl acrylate copolymer there was no observable difference between liquid water and water vapour transmission rates. Morgan⁷ has reported the liquid water permeability for a simple VDC/VC copolymer (12g/25 μ m²/day for 66/34 VDC/VC copolymer) which is very similar to the water vapour permeability shown in Table I for the more complex vinyl acrylic copolymer. Thus the low permeability of the VDC/VC class of copolymer to both liquid water and water vapour can be regarded as being well established.

The water permeability results discussed above refer specifically to the diffusion of water liquid and vapour

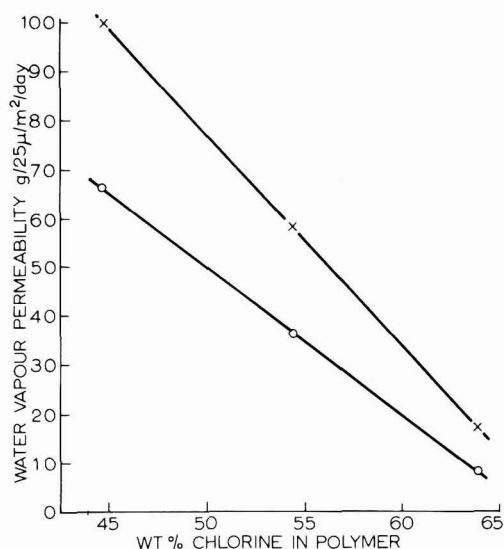


Figure 1. The effect of chlorine content on the water vapour permeability of a series of vinyl acrylic copolymer films

O = solvent cast films

x = latex films (latex containing 3% Syneronic PE39/70 and 0.5% hydroxy propyl methyl cellulose on latex solids)

through detached polymer films. However, when considering a binder polymer for use in protective paints, it is the permeability of the film when coated on the substrate to be protected which must be considered. In this latter situation additional factors which may affect permeability become important, notably the osmotic driving force for the diffusion of water through the film and the rate of diffusion of ions through the film. It was therefore relevant to establish whether the vinyl acrylic copolymer latex discussed above also exhibited good barrier properties when coated onto a metal substrate and immersed in water containing dissolved ions. In order to establish this point, the change in capacitance with time of coated substrates immersed in 2.5 per cent sodium chloride solution was measured using an AC impedance technique. The rate of water uptake of the film was calculated from the measured values of capacitance by the method of Brasher and Kingsbury¹⁰. The results, which will form the basis of a future publication, confirmed the low rate of water uptake of the vinyl acrylic copolymer when compared to the conventional latex types.

Latex colloid stabilisation

Refs. 11, 12

In recent years a number of workers^{11,12} have demonstrated that emulsion polymerization can be carried out in the complete absence of either conventional surfactants or what are usually termed as protective colloids (e.g. polyvinyl alcohol, hydroxyethyl cellulose). In such polymerizations colloid stability can be provided by the charged polymer end groups which arise from the polymerization initiator¹¹, some of which are located on the outer surface of the polymer particles and hence give rise to charge stabilisation of the system. A similar procedure was employed in the preparation of the vinyl acrylic copolymer latex discussed in this paper, with only a very low proportion of surfactant being employed in the polymerization. This procedure has the advantage that the presence of protective colloids, which can impair latex coalescence³, was avoided.

Although the addition of surfactants and dispersants is generally necessary when formulating a practical paint from the latex, it may be argued that there can be advantages in using as a starting point, a latex which has a very low surfactant content. One such advantage is that it is then possible to select formulation surfactants purely on the grounds of obtaining optimum paint properties, notably storage stability and protective properties. The behaviour of the formulation surfactants in the polymerization need not be considered, which can be a distinct advantage in that certain limitations in the choice of surfactant are imposed when it is required to introduce the surfactant at the polymerization stage. For example, the optimum surfactant from the point of view of paint properties may be above its cloud point under the conditions of pH, temperature and ionic strength pertaining during the polymerization.

From the point of view of paint storage stability it was found that a block copolymer surfactant of the ethylene oxide/propylene oxide/ethylene oxide type (e.g. Syneronic PE39/70) was extremely effective. It was considered to be necessary to establish whether this class of stabiliser impaired the latex film forming process or final film properties. A number of experiments were performed in order to establish this point.

Water vapour permeability measurements were performed on both solvent cast films and films obtained by casting latex films containing block copolymer surfactant (3 per cent by weight on latex solids) and a small proportion of water soluble thickener (0.5 per cent by weight of hydroxypropyl methyl cellulose). The solvent cast films were prepared by casting films for test from toluene solution. It was found (Figure 1) that although the water vapour permeability of each latex cast film was higher than that of the corresponding solvent cast film, which can be assumed to have a very high degree of integrity, the permeability was of the same order of magnitude. Thus the latex cast films containing 3 per cent added block copolymer surfactant and added thickener, were sufficiently well coalesced to give a very low water vapour permeability by the standards of typical emulsion polymers used for preparing anti-corrosive paints. Indeed, evidence was obtained by electron microscopy to suggest that the addition of the block copolymer surfactant increased the rate and extent of coalescence of Haloflex 202 latex cast film. Figure 2 shows transmission electron micrographs of the surface of latex cast films which had been replicated 4 hours after casting the film. Figure 2 shows quite clearly that as the concentration of block copolymer surfactant increased, a progressive improvement in the extent of coalescence of the latex particles took place. In the absence of surfactant the boundaries between the polymer particles are still clearly visible after 4 hours drying. That these boundaries do in fact correspond to the individual latex particles was confirmed by measuring the particle size of the latex in question using a Joyce Loeble disc centrifuge which gave a particle diameter of $0.21 \mu\text{m}$, in agreement with the dimensions visible in Figure 2. In the case of the film containing 3 per cent added block surfactant (Figure 2c) the boundaries between the individual latex particles had virtually disappeared. There is evidence that this acceleration in the rate of coalescence of the latex film, caused by the addition of the block copolymer surfactant, was due to plasticization of the latex polymer particles by the surfactant.

Polymer stability considerations

Although the results and arguments given in the previous section indicate the potential of high chlorine content copolymer latices for steel protection, it is necessary to translate this potential to a practical paint system. It is in this respect that chloropolymer latices present features not found in conventional latices. The main feature which must be considered is the tendency of high chlorine content polymer latices to undergo dehydrochlorination reactions at alkaline pHs, the higher the pH the higher the rate of dehydrochlorination. This dehydrochlorination reaction results in a number of effects, the main ones being a progressive drift to lower pHs and a progressive increase in the chloride ion concentration in the latex (or corresponding paint) aqueous phase. Although the rate of dehydrochlorination can be reduced by reducing the chlorine content of the polymer, this reduction was found to be at the expense of the barrier properties of the polymer.

Typical data showing the downward drift in pH and the increase in chloride ion concentration for a chlorine containing copolymer latex held at 40°C , is shown in Figure 3 in which the initial latex pH at the commencement of the experiment was 8. Chloride ion concentrations were measured by an electrometric titration method using a silver electrode.

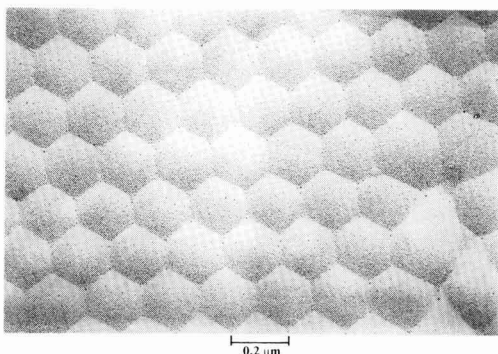


Figure 2a. Latex containing no added block copolymer surfactant

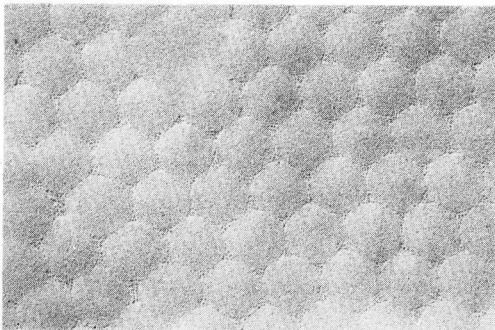


Figure 2b. Latex containing 1% added block copolymer surfactant



Figure 2c. Latex containing 3% added block copolymer surfactant

Figure 2. Transmission electron micrographs of pre-shadowed replicas of films cast from vinyl acrylic copolymer latex. Film surface replicated 4 hours after casting

Normally, latex paints are formulated at alkaline pH, usually in the range 7-9. Formulation of a high chlorine content latex into a paint in this pH range was found to give the expected pH drift and increase in chloride ion concentration. Typical data obtained for the zinc phosphate primer formulation shown in Table 2 and based on

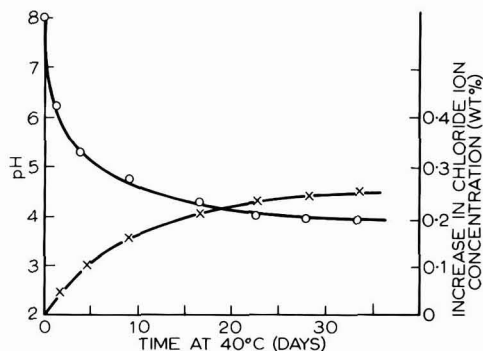


Figure 3. Change of pH and chloride ion concentration for a neutralised vinyl acrylic copolymer latex at 40°C

O = pH
x = chloride ion concentration

Haloflex 202 is shown in figures 4 and 5. These figures relate to a set of identical paints differing only in their initial pH. It can be seen that the higher the pH of the paint, the greater the rate of dehydrochlorination of the polymer. As the dehydrochlorination reaction proceeded, the pH fell and hence the rate of dehydrochlorination decreased. When stored at 40°C, the pH of each paint fell to a final steady value of 4.0 irrespective of the initial pH, although the time taken to achieve this steady pH of 4.0 decreased with decreasing initial pH. By the stage at which each paint had reached this steady pH value of 4.0 the increase in chloride ion concentration had virtually ceased, showing that polymer dehydrochlorination had ceased at this pH even at 40°C. The actual final chloride ion con-

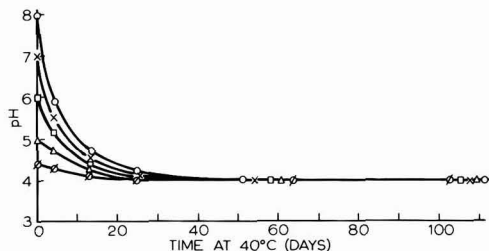


Figure 4. Change of pH at 40°C for a set of vinyl acrylic latex paints with varying initial pH

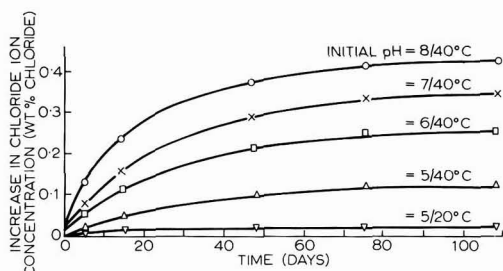


Figure 5. Increase in chloride ion concentration at 40°C for a set of vinyl acrylic copolymer latex paints with varying initial pH

centration of the stored paints naturally decreased with decreasing initial paint pH (Figure 5), demonstrating the benefit of formulating the paint at a low pH (<5). As might be expected, for any given formulation pH, the rate of chloride ion evolution decreased with decreasing temperature (Figure 5).

Although in principle it is possible to buffer the paints at an alkaline pH, in practice it was found that at the higher chlorine contents (as is required for good barrier properties) the rate of dehydrochlorination was such that unrealistically high concentrations of buffer were required if the pH was to be maintained in the alkaline region during storage. The use of such high concentrations of water soluble buffer was found to impair the protective properties of the final paint. Furthermore, the higher chloride ion concentration generated in the heavily buffered paints is likely to be a disadvantage in an anti-corrosive primer paint because the chloride ion is an aggressive ion in a corrosion sense.

It was found that the variation of pH with time during paint storage depended strongly on the nature and purity of the pigments used. When basic pigments were used the pH of a paint formulated at pH 5 was found to rise, an effect which was regarded as disadvantageous because of the consequent increased rate of chloride ion evolution.

It can be seen from figures 4 and 5 that when the paint formulated at pH 5 was stored at 40°C it underwent only a small increase in chloride ion concentration, the chloride ion evolution virtually stopping when the pH fell to its final steady value of 4.0; when stored at 20°C the increase in chloride ion concentration was negligibly small (Figure 5) throughout the entire period. It is for this reason that an acidic paint formulation was developed.

Design of an acidic paint formulation

The initial attempts at preparing a storage stable primer paint at a pH of 5 using the classes of conventional surfactants and thickeners generally used in conventional alkaline formulations proved unsuccessful, and it became apparent that some care was required in the choice of the additives and the design of the formulation. All the paint components must obviously be chemically stable in the pH range 4-5, and the water soluble components such as surfactant, dispersant and thickener must be selected on the basis of their ability to be effective in this pH range. One very storage stable paint which was eventually developed is shown in Table 2 and merits a number of comments. The particular formulation makes use of an ethylene oxide/propylene oxide block copolymer (Synperonic PE39/70) as latex stabiliser which provides a strong degree of steric stabilisation to the system in the pH range 4-5. This particular latex stabiliser was chosen for a number of reasons:

- (i) It conferred on the latex a very high degree of stability to added multivalent ions in the relevant pH range. Such stability is especially important in an anti-corrosive paint due to the solubility (albeit small) of the anti-corrosive pigments.
- (ii) It aided film coalescence, with good barrier properties being obtained in its presence as discussed above.
- (iii) It eliminated the tendency of the thickener (hydroxypropyl methyl cellulose) to progressively bridge-flocculate the latex during storage.

It was found that the effectiveness of a series of ethoxylates in providing paint storage stability increased with increasing ethylene oxide chain length, with the block copolymer being particularly effective in that it produced viscosity stability for a period well in excess of 12 months at 40°C.

The paint formulation shown in Table 2 also makes use of Synperonic PE39/70 as pigment dispersant. This selection was made not on the basis of its efficiency as a pigment dispersant, but on the basis of its ability to maintain the state of the pigment dispersion during storage. Other pigment dispersants in fact proved to be capable of producing a storage stable paint but when the stored paints were re-tested they demonstrated inferior performance in, for example, the salt spray test when compared to the corresponding fresh paints and when compared to the stored paint containing Synperonic PE39/70 as pigment dispersant. This deterioration during paint storage was shown to be due to pigment flocculation.

It will be noted that in this particular formulation the non-toxic pigment zinc phosphate was used.

Table 2

Primer formulation based on Haloflex 202.
The following formulation was prepared by high speed cavitation dispersion

Ingredient	% w/w
Haloflex 202 (a)	59.7
Synperonic PE39/70 (30% w/w solution) (b)	3.1
Methocel J12MS (c)	0.2
Zinc phosphate PZ40 (d)	5.8
Micronised barytes	15.6
Micronised red iron oxide	2.6
Bevaloid 642 (e)	0.2
Butyl Ethoxol (f)	2.0
Water	10.8
PVC	20%
Volume solids	44%
pH	5
Viscosity	2 poise (180 s ⁻¹)
(a) Haloflex 202 – vinyl acrylic binder (60% solids)	– ICI Ltd, UK
(b) Synperonic PE39/70 stabiliser/dispersant	– ICI Ltd, UK
(c) Methocel J12MS – thickener	– Dow Corning, USA
(d) Zinc phosphate PZ40	– Societe Des Couleurs Zincique, France
(e) Bevaloid 642 – defoamer	– Bevaloid Ltd, UK
(f) Butyl Ethoxol – coalescing solvent	– ICI Ltd, UK

Paint performance

Having established that latices can be prepared from selected monomers to give films with low permeability, and that stable paints can be formulated from these latices at acidic pH, it is necessary to show that these paints give satisfactory performance on steel. Results will be presented in confirmation, comparing this system to paints from other available latices in a variety of tests. The paints used for comparison are to formulations recommended for the type of latex in question.

Table 3
Paints under test

Paint system	Polymer type	Primer paint pH	Primer paint PVC %
1	Vinyl acrylic*	5.0	20
2	Styrene acrylic	9.5	22
3	Acrylic	9.0	20
4	Acrylic	9.0	30
5	Acrylic	9.5	30
6	Styrene acrylic	9.0	22

*Haloflex 202

Where top coats were used in accelerated and exterior exposure testing, these were white paints prepared from similar polymer types to those used for the corresponding primers. Top coat PVC's were 20 per cent for all but systems 1 and 5 (15 per cent PVC).

Tests and procedures

The following tests were performed on paint systems according to the procedures outlined:

(i) Flash rusting tendency

Paints were brush applied to mild steel blasted with G17 grit to Sa2.5 according to Swedish Standard SIS 055900-1967 to give a 40µm dry film thickness in one coat. Panels were examined for flash rusting.

(ii) Mechanical properties

Flexibility – primers only were applied to abraded aluminium panels to give 100µm dry films and after 7 days drying were tested for flexibility using a 3 mm mandrel as detailed in BS 3900: Part E1:1970.

Scratch hardness – primers only were applied by applicator to 150 × 100 mm degreased Pyrene test panels to give 100µm dry films. After 7 days drying the films were tested for maximum load without failure by the test outlined in BS 3900: Part E:1970.

Impact – primers only were brush applied to 150 × 100 mm degreased Pyrene test panels to give 40µm dry films and allowed to dry for 7 days before testing. Panels were tested using a 3 mm indentation according to the procedure in BS 3900: Part E3:1973 with the paint film tested upwards and downwards.

(iii) Adhesion

Cross cut test – primers were brush applied to 150 × 100 mm test panels to give 120µm dry films in two coats on shot blasted steel to Swedish standard Sa2.5 and degreased cold rolled steel. Tests for cross cut adhesion were then performed after 7 days drying of the films according to BS 3900: Part E6:1974, results being recorded after cross cutting and also using adhesive tape over the test area.

Pull-off adhesion – the same panels after the same drying period were subjected to pull-off adhesion testing. For this the Erichsen pull-off adhesion tester was used, but using 12.6cm² dollies affixed to the paint surface with two pack epoxy adhesive which was allowed to dry for a further day

before test. The pressure required to cause failure and the type of failure and its extent were recorded.

(iv) *Accelerated testing*

Salt spray – primers were brush applied to the right hand halves of shot blasted steel 150 × 100 mm panels cleaned to Swedish Standard Sa2.5 (50 μm profile) to give 3 × 40 μm dry films, allowing 24 hours drying between coats. On the left hand half of each panel the system was 2 × 40 μm dry films of primer and 2 × 40 μm dry films of appropriate topcoat. Twenty-four hours drying was allowed between coats.

All panels were cross cut to expose metal.

After 7 days drying the panels were subjected to continuous salt spray testing according to BS 3900: Part F4:1968.

Distilled water soak – panels prepared as for salt spray testing above were subjected to immersion in non-aerated distilled water, with only half the panel immersed.

(v) *Exterior exposure*

300 × 100 mm panels were blast cleaned and the paints brush applied to give 2 × 30 μm DFT of primer and 2 × 30 μm DFT of the appropriate finish and exposed at a coastal site (Gladstone Dock, Liverpool, UK), Rural (Holford, Cheshire, UK), and an Industrial site (Widnes, Cheshire, UK).

Accelerated testing

In Table 4 below are recorded the comparative results for the paints under investigation after salt spray and distilled water soak testing. Blister ratings are recorded according to ASTM D714-56. For the distilled water soak test, results are recorded for films both above and below the liquid level.

From the data presented it is evident that in the salt spray test the vinyl acrylic gave ideal protection across the film in general, some blistering occurring at the cross cut only. All of the acrylic and styrene/acrylic binders broke down to some extent in the bulk film in terms of blistering and in most cases rust staining of the surface of the paint film. In distilled water soak testing similar trends have been observed for the immersed parts of the paint films.

In addition photographs of the test panels are shown in Figure 6.

Taken together, it can be seen that the vinyl acrylic produced much superior results compared to the other copolymer types.

The vinyl acrylic paint was compared to a proprietary solvent borne alkyd primer in the more severe American salt spray test (ASTM B117-75), with the results shown in Table 5.

Table 5
American salt spray test

Paint system	500h Salt spray primer only
1 alkyd	8M 4M/MD

From the results in Table 5 it can be seen that the vinyl acrylic compared favourably with the solvent alkyd in this test.

Flash rusting tendency

Ref. 13

Table 6 details the results obtained for flash rusting of primers when tested according to the procedure that has been outlined.

Table 6
Flash rusting results

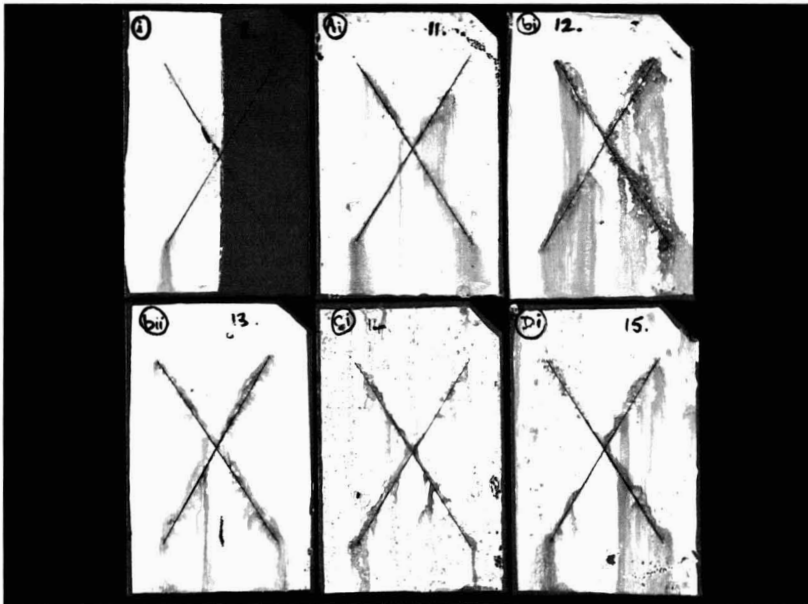
Paint system	Flash rusting
1	none
2	flash rusted
3	none
4	none
5	flash rusted
6	flash rusted with severe film cracking

Table 6 shows that mixed results were obtained from the existing types of latex which were tested at alkaline pH. Paint 1 which was at pH 5 has shown no flash rusting

Table 4
Accelerated test results

Paint system	1000h salt spray		1000h distilled water soak			
	Primer only	Primer/topcoat	Primer only		Primer/topcoat	
			Above	Below	Above	Below
1	none	none	none	none	none	micro B/M
2	7D	7D	8F	2D	none	3D
3	3MD	none	none	4MD	none	2cm dia. blisters
4	6F	none	none	2D	none	2D
5	4MD	4MD	6MD	4MD	6MD/D	2MD
6	7MD	7D	none	4D	8F	4D

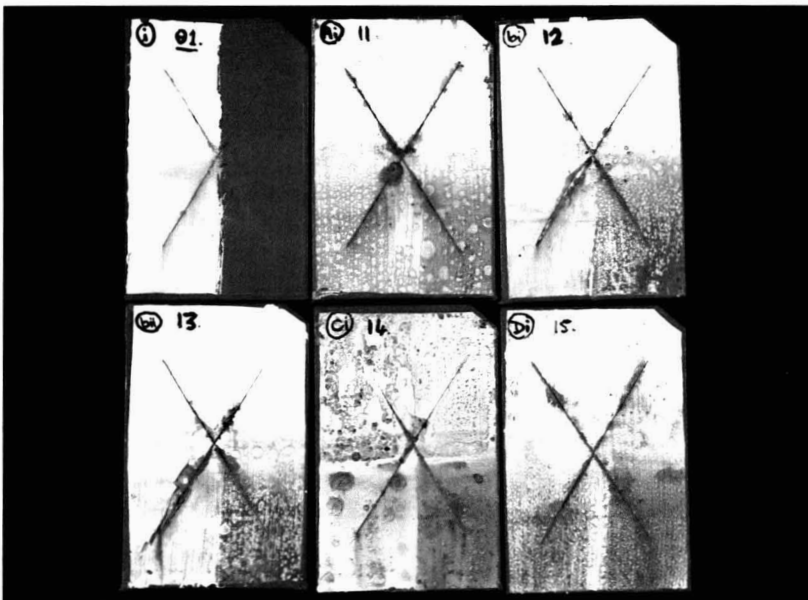
B/M – along brush marks



1000 h BS salt spray

Plan showing the paint systems used on the respective panels (same for top and bottom)

1	2	3
4	5	6



1000 h distilled water soak

Figure 6. Accelerated testing. Left hand side of each panel, primer plus topcoat; right hand side of each panel, primer only

and this was achieved without the addition of specific inhibitors other than might be afforded by the anti-corrosive pigmentation. The same paint prepared to a pH of 6 or higher would be expected to show flash rusting.

A consequence of formulating paints at an acid pH was that, surprisingly, the tendency to give flash rusting was very much reduced¹³. There is at present no explanation as to why there is a much reduced tendency to exhibit flash rusting at the more acidic pHs. No convincing mechanism to explain the flash rusting process has to date been proposed, although it is presumably an electrochemical reaction involving the formation of iron salts which migrate to the paint film upper surface where they are clearly visible. There are a number of fundamental differences between operating at an acidic pH and an alkaline pH, for example the solubility of the anti-corrosive pigment and the change in the balance of reactions taking place at the cathodic sites. These two factors obviously need to be taken into account in any explanation of the reduced tendency to produce flash rusting at acidic pHs.

Mechanical properties

In order to maintain paint film integrity on steel during handling of the coated steel and in service, good mechanical properties are necessary. Comparative results are shown in Table 7 from which it is evident that all paints tested had good flexibility but only the vinyl acrylic in paint 1 shows the desirable combination of good impact resistance and high scratch hardness.

Table 7
Mechanical properties of paint films

Paint system	Flexibility	Scratch hardness (g)	Impact	
			Film up	Film down
1	pass	3300	pass	pass
2	pass	1100	pass	pass
3	pass	700	pass	fail
4	pass	400	fail	pass
5	pass	1100	pass	pass
6	pass	3100	fail	fail

Adhesion

The results on primers subjected to adhesion testing are shown in Table 8.

All systems produced good results on shot blasted steel. On cold rolled steel a more variable level of results was obtained.

It has been shown that the vinyl acrylic produced good adhesion to cold rolled steel when tested as a latex film. Paint formulation ingredients obviously also affect adhesion. Further investigations have shown adhesion to be influenced by type and level of extender and coalescing aid, and work along these lines is continuing.

Exterior exposure

The ultimate test for any steel protection system is its performance in true user conditions. The vinyl acrylic latex was compared with an acrylic latex, styrene acrylic latex, and solvent borne proprietary alkyd at a number of exposure sites. Identical dirt collection and retention were recorded for all the systems, so only data for blistering, rusting and chalking has been detailed.

The acrylic latex system overall, had more incidence of blistering than the styrene/acrylic system whereas the vinyl acrylic latex was free from this defect. At the two most severe sites rust damage was evident more from the acrylic than the styrene acrylic system tested, again the vinyl acrylic being free from defect. The styrene/acrylic and acrylic results show a fall-off in overall performance with test site in the order rural > industrial > coastal, but the vinyl acrylic and solvent borne alkyd gave excellent performance to date at all sites. These results confirm the promise shown by accelerated testing of the vinyl acrylic.

Conclusion

Environmental and cost pressures on solvent based paint systems have led the industry to look at alternative coating systems.

The approach to latex and paint formulation described has shown a number of advantages. These include films of

Table 8
Adhesion test results

Paint system	Cross cut test				Pull off test			
	Shot blasted		Cold rolled		Shot blasted		Cold rolled	
	A	B	A	B	Pressure PSI	Main failure	Pressure PSI	Main failure
1	0	0	0	5	450	CP	350	PS
2	0	0	0	4/5	450	CP	300	AD
3	0	0	0/1	5	425	CP	350	PS
4	0	0	2/3	5	325	CP	350	PS
5	0	0	0	4	450	CP	400	PS
6	0/1	0/1	0	0	450	CP	400	CP

A – before adhesive tape
B – after adhesive tape

CP – cohesion of primer
PS – primer to substrate

AD – adhesive failure
0-5 scale where 0 = perfect

Table 9
Exterior exposure results

Paint system	1.25 years coastal			1.5 years rural		1.5 years industrial	
	Blistering	Rusting	Chalking	Blistering	Rusting	Blistering	Rusting
1	10	10	8	10	10	10	10
2	8M	8	8	9M	10	8M	9
3	6M	5	8	8M	10	8M	7
Alkyd				10	10	10	10

0-10 scale where 10 is perfect

low permeability to oxygen and water, and air drying paints with reduced tendency to flash rust, good mechanical properties and excellent performance in accelerated laboratory tests on steel. Exterior exposure results on steel are following similar trends.

The indications from the promising results obtained are that this type of system could provide a significant advance in the area of aqueous anti-corrosive paints for steel.

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Skin irritation due to hexanediol diacrylate; problems in the evaluation of results from animal studies*

By H. P. Gelbke and H. Zeller**

Summary

The skin irritation due to hexanediol diacrylate (HDDA) was investigated in the rabbit by various test establishments according to the method of Draize. The testing of technical HDDA from different manufacturers and of distilled HDDA as a standard substance led to the following results:

1. A wide range of variation was observed for the primary irritation indices (PII) determined by the different test establishments. This applied not only to the technical HDDA samples but also to the standard substance, which originated from the same batch in all tests. Therefore, the reliability of conclusions drawn only from the numerical values of PIIs is questionable. In contrast, HDDA was detected as a "skin irritant" in all animal tests, and this would have resulted in a recommendation of preventive measures for the protection of the worker (avoid contact with skin). This also applies to the

lowest PII (1.46), since special findings on the skin were obtained which clearly emphasised the irritant action without being expressed as numerical values. The problems of a strictly numerical assessment of subjective findings thus becomes apparent.

2. In the comparative study of technical HDDA types with distilled HDDA as the standard substance on the same animals, no difference in skin irritation was found between the HDDA products from different manufacturers and the distilled material. Thus differences in the PIIs are probably not due to the specific HDDA types but to the wide range of variation encountered in biological test results. If irritant effects of different substances are to be compared with each other, a strictly comparative study on the same animal is recommended.

Keywords

Raw materials used in manufacture or synthesis of ingredients for coatings

acrylate

Miscellaneous terms

health and safety
skin irritation

L'irritation épidermique provoquée par hexanediol diacrylate, les problèmes associés à l'évaluation des résultats donnés par les essais sur animaux

Résumé

L'irritation épidermique provoquée en lapins par hexanediol diacrylate (HDDA) a été étudiée par la méthode de Draize dans divers instituts de recherche. Les essais de HDDA de qualité commerciale fournis par divers fabricants, et où on s'est servi du HDDA distillé en tant que matière de contrôle, a conduit aux résultats suivants.

1. On a noté une large variation parmi les indices d'irritation primaire (PII) déterminés par les divers instituts de recherche. Cette variation s'applique non seulement aux qualités commerciales de HDDA, mais aussi à la matière de contrôle, dont les échantillons pour tous les essais ont été pris du même lot. Ainsi la fiabilité des conclusions tirées exclusivement des valeurs numériques des PII est douteuse. Par contraste, HDDA se montre comme irritant épidermique dans tous les essais sur animaux, et ce fait aurait dû aboutir à l'établissement des mesures de prévention pour protéger les ouvriers (par exemple éviter tout contact avec la peau). Ces considéra-

tions s'appliquent également à la plus basse valeur du PII (1.46), puisqu'on a noté des effets sur la peau ce qui ont indiqué nettement l'action irritante en dépit du faible PII. Ce désaccord met fortement en évidence le problème créé par une appréciation exclusivement numérique des observations subjectives.

2. Dans l'étude comparative sur la même espèce d'animal des types commerciaux de HDDA, et où on s'est servi du HDDA distillé en tant que matière de contrôle, aucune différence n'a été trouvée entre les variétés de HDDA fournies par divers fabricants et le produit distillé. Ainsi les différences parmi les PII sont dues probablement non aux types spécifiques du HDDA, mais à la variation éendue des résultats donnés par les essais biologiques. Dans le cas où il faut comparer l'action irritante de diverses substances, une étude rigoureusement comparative est à conseiller.

*The following companies participated in the comparative study with a standard substance: Bayer AG Leverkusen; Degussa, Frankfurt; Kunststoffsabrik Synthese BV, Bergen op Zoom; Röhm GmbH, Darmstadt; UCB SA, Drogenbos; BASF Aktiengesellschaft, Ludwigshafen.

**BASF Aktiengesellschaft, Department of Toxicology, D-6700 Ludwigshafen, Federal Republic of Germany
The following companies submitted test data on their products: Ancomer Ltd, Clayton, Manchester; CdF Chimie, Paris, Defense; Diamond Shamrock Europe Ltd, Eccles, Manchester.

Die Hautreizwirkungen von Hexandiodiacrylat, Auswertungsprobleme der tierexperimentellen Untersuchungsergebnisse

Zusammenfassung

Es wurde die Hautreizwirkung von Hexandiodiacrylat (HDDA) tierexperimentell nach der, "Draize-Methode" von verschiedenen Prüfinstituten untersucht. Die Prüfung von technischen HDDA-Qualitäten verschiedener Hersteller und von destilliertem HDDA als Standardsubstanz führte zu folgenden Ergebnissen:

1. Es fand sich eine breite Streuung für die primären Reizindizes (sogenannte "Draize-Werte"), die von den verschiedenen Prüfinstituten ermittelt würden. Dies galt nicht nur für die technischen HDDA-Qualitäten, sondern auch für die Standardsubstanz, die bei allen Untersuchungen ein und derselben Charge entstammte. Die Aussagekraft exakter Zahlenangaben zum primären Reizindex muß daher in Frage gestellt werden. Dagegen wies jede Untersuchung HDDA als "hautreizende" Substanz aus und hätte zur Empfehlung von Schutzmaßnahmen (Vermeidung eines Hautkontaktes) geführt, so daß sich der Wert der tierexperimentellen Untersuchung für den vorsorgenden Schutz am Arbeitsplatz

bestätigen ließ. Dies gilt auch für die Untersuchung mit dem niedrigsten PII (1.46), da hierbei deutliche Hautveränderungen verbal umschrieben wurden, die nicht in die numerische Berechnung eingingen. Die Schwierigkeit einer zahlenmäßigen Erfassung subjektiv einzustufender Befunde wird dadurch besonders deutlich.

2. Bei der streng vergleichenden Prüfung der technischen HDDA-Qualitäten gegen destilliertes HDDA als Standardsubstanz an denselben Tieren konnte kein Unterschied in der Hautreizwirkung zwischen den HDDA-Produkten verschiedener Hersteller und dem destillierten HDDA gefunden werden. Unterschiede in den PII-Werten dürften bei diesen Produkten also nicht substanzbedingt sein, sondern auf der großen Streubreite der biologischen Prüfmethode beruhen. Sollen Reizwirkungen verschiedener Substanzen gegeneinander abgeschätzt werden, so muß daher eine streng vergleichende Prüfung dieser Stoffe an denselben Tieren empfohlen werden.

Introduction

Refs. 1-6

Multifunctional acrylates, such as hexanediol diacrylate (HDDA), trimethylolpropane triacrylate or pentaerythritol triacrylate, are used widely in radiation-curable finishes, printing inks and adhesives. When handling these products with inadequate precautions, substance-induced skin changes were observed repeatedly¹⁻³. The considerable skin irritation potential of these substances was also confirmed in animal studies, mainly by using the method of Draize⁴ for rabbits. This test method is now used worldwide and was also published in the US "Federal Register" as a standard procedure⁵. Here, skin irritation is determined under extreme conditions that are often not encountered in industrial practice (24-hour duration of contact under an occlusive dressing, testing of intact and abraded skin with destruction of the upper skin layer). The substance-induced erythema and oedema of the skin are scored using a numerical scale at defined times; the primary irritation index (PII) is subsequently calculated by a given formula. On the basis of the PII and defined limit values, the substance is then often classified as "non-irritating", "slightly irritating", "moderately irritating" or "severely irritating" (Table 2).

In this investigation, the validity of exact numerical values for the PII was to be demonstrated for multifunctional acrylates using hexanediol diacrylate (HDDA) as an example. Two questions were to be answered in particular:

1. To what extent do different PIIs actually indicate differing irritation potentials of the substances tested? Additionally, the considerable variations in the PIIs of technical HDDA products from different manufacturers needed explanation. It was to be clarified whether differences in PIIs were due to the actual qualities of the technical HDDA products or because the animal experiments were carried out in different establishments.
2. Is there an animal test model which can be used to reliably compare the irritation potentials of different substances? Such a method could prove extremely useful for developing and using new products with a low irritant effect.

Exact figures derived from animal studies pose their own problems. The numerical values for the PIIs, which are often given to three significant places, may be used erroneously as if they were measured physical quantities. Here, the fact which is well-known to toxicologists that animal data always vary considerably, is not taken into account; this has been proved impressively by a round-robin study⁶.

However, potential hazards can be reliably detected by animal studies for skin irritation. In practice it is often observed that differences in PIIs, which are absolutely irrelevant from the toxicological point of view, serve users as a criterion for the selection of their products and manufacturers as a criterion for further development of substances with a low skin irritation potential. This leads to a feeling of safety, which does not correspond to the conditions of practice, and often results in misinterpretations. Sometimes PIIs are even used to calculate the PII of a mixture from the PIIs of the individual components. Furthermore, the argument is often put forward that in a mixture only the component with the severest skin irritation determines the overall effect. Of course these considerations are inadmissible from the scientific point of view. Basically, it is impossible to foresee whether the individual components in a mixture have synergistic or antagonistic biological effects.

Method

Ref. 5

Test establishments 1-12 examined technical HDDA from various manufacturers (A-F) and/or a distilled product (HDDA dist.), which always originated from the same batch, for skin irritation. The tests were carried out in accordance with the method given in the Federal Register⁵; further specifications were not given since each establishment was to follow its own standard procedures with regard to the experimental details. However, for the comparative study the method had to be slightly altered (see below). The outline of the test methods is shown below (for further details see reference No. 5).

A defined quantity of the test substance was applied to intact and abraded skin areas of a prescribed size for 24

hours under occlusive dressing, 24 and 72 hours after the beginning of application, erythema and oedema were recorded and scored using a defined scale from 0 to 4. Thereby, 48 individual values were obtained which were divided by 24 to give the primary irritation index. Eight days after application a further reading was carried out but these results were not used to calculate the PII, they were taken to assess the reversibility of the skin changes.

If only one substance was to be examined, six rabbits were used. Each animal received two applications, one on the intact and the other on the abraded skin.

The comparative study of two substances (HDDA dist. as a standard compared with technical HDDA from different manufacturers) was carried out with 12 rabbits, each of which again received two applications. In half of the animals both substances were applied to the intact skin, in the other half to the abraded skin. The substances were applied to opposite sides of the animal's body at application sites exactly corresponding to each other. Thus the effects of the substances could be compared with each other under identical conditions.

Table 1
Skin irritation due to HDDA in rabbits

Test establishment	PII values of		
	HDDA dist.	HDDA tech. [manufacturers]	
1 CT	1.46	1.54	[A]
2 IT CT	3.33	3.25	[B, I]
2 IT		2.83	[B, II]
3 CT	5.2	5.2	[C, I]
4 IT CT	5.0	4.9	[D, I]
4 IT		4.5	[C, II]
4 IT		5.0	[F, I]
4 IT		6.0	[D, II]
5 IT CT	6.0	5.6	[E, I]
5 IT CT	2.9	3.5	[F, II]
5 MT	4.6		
5 MT	4.7		
5 MT	4.8		
5 MT	4.8		
5 MT	4.9		
5 MT	5.1		
5 MT	5.1		
5 IT		6.0	[F, III]
5 IT		3.4	[E, II]
5 IT		5.0	[G]
5 IT		5.0	[H]
6	4.8		
7	5.8		
8	6.25		
9	6.3		
10		3.81	[D, III]
11		2.17	[D, IV]
12 IT		3.0	[K, I]
12 IT		4.2	[K, II]

Key:
PII — primary irritation index
HDDA — 1,6-hexanediol diacrylate
dist. — distilled
tech. — technical
IT — these values were determined in independent single-substance tests by the same test establishment
MT — these values were determined in a multiple test series by the same test establishment
CT — comparative test of HDDA dist. and tech. on the same animal
I-IV — different batches from the same manufacturer.

Table 1a
Primary irritation indices of comparative skin irritation studies using HDDA dist. and tech. in rabbits (extract from Table 1)

Test establishment	PII values of	
	HDDA dist.	HDDA tech. [manufacturers]
1	1.46	1.54 [A]
2	3.33	3.25 [B, I]
3	5.2	5.2 [C, I]
4	5.0	4.9 [D, I]
5	6.0	5.6 [E, I]
5	2.9	3.5 [F, II]

Key as Table 1

Results and discussion

Refs. 3, 4, 6

The primary irritation index (PII) was determined by test establishments 1-12 for technical HDDA and/or distilled HDDA from the same batch. In some cases, the PII of technical HDDA from different manufacturers (A-K) was compared with distilled HDDA using the same test animals (for details see under "Method"). Tables 1 and 1a show the results obtained from single-substance and comparative tests, which are discussed in more detail below.

Testing of the same batch (HDDA dist.) by different test establishments (Table 1)

When investigating HDDA dist. as a standard at different test establishments, a wide variation in the PIIs between 1.46 and 6.3 was found. These results agree with those of the round-robin study mentioned above⁶ and the disparities are probably due to the numerous variables in animal studies which may influence the experimental outcome, such as: differences in the sensitivity of the animals used by the different establishments, seasonal variations in the sensitivity of the rabbits' skin, differences in the subjective evaluation by individual observers, and slight variations in the experimental techniques. However, it's hardly possible to eliminate this wide range of variation in test results by means of a strict, worldwide standardisation of test methods.

Independent testing of different batches (HDDA tech.) (tables 1 and 1a)

Similarly with the results obtained from HDDA dist., a wide range of variation in the PIIs was also found when technical HDDA types from different manufacturers (A-F) were tested by different test establishments (1.54-6.0), as expected. Furthermore, even when different technical HDDA types were investigated by the same test establishment in independent single-substance tests (IT), the PIIs still varied considerably (test establishment 2: PII 2.83-3.25; test establishment 4: PII 4.5-6.0; test establishment 5: PII 3.4-6.0).

Testing of the same batch (HDDA dist.) by the same test establishment (tables 1 and 1a)

HDDA dist. was investigated by test establishment 5 in independent single-substance tests (IT). Test materials were submitted by various sponsors; thus the test establishment was not able to establish a direct connection

between the substances. It was found that even tests carried out by the same establishment with HDDA dist. resulted in considerably different PIIs (2.9 and 6.0).

In contrast, if HDDA dist. was investigated in a multiple test series (MT), there was an almost negligible variation in the PIIs, between 4.6 and 5.1. In this case, the substance was examined under the same name in seven individual tests carried out within a short time-span. However, the good agreement of the PIIs may possibly be connected with a certain bias, since the investigator was aware that the same substance was examined in the different tests.

Classification and evaluation

In Table 2 some proposed or prescribed limits for scoring skin irritation as "not" to "severely" irritating according to the PII are listed. If these are compared with the wide variation in the PIIs which were recorded by the various test establishments using the same substance (HDDA dist.), different classifications would be obtained depending on the particular investigator. This may be confusing for the manufacturer and user, and reduces the validity of rigid classification limits and exactly defined numerical PIIs for skin irritation. However, the results derived from animal experiments are well suited to the actual problems that occur in practice, i.e. differentiating between irritating and non-irritating substances. The main purpose of an animal experiment is to obtain information on potential hazards in handling a substance, so that the necessary precautions may be taken in advance. All investigations presented here clearly indicate an irritating potential for HDDA in spite of the differing PIIs and regardless of the test establishments. In every case the toxicologist would classify the substance as irritating to the skin and would recommend that contact with the skin is to be avoided. This also applies to the results from test establishment 1, which obtained a PII of 1.46: in this particular case, special findings were described which clearly emphasised the irritant action, although they could not be expressed numerically.

Comparative testing of different batches (HDDA dist. and tech.) by the same test establishment (tables 1 and 1a)

Technical HDDA from different manufacturers (A-F) was tested in comparison with HDDA dist. as a standard. Since in this test distilled and technical HDDA were applied to opposite sides at corresponding sites of the animal's body, the reasons for the variations in the PII, which depended exclusively on the experimental details (sensitivity of the animals, details of the test procedures, subjective evaluation by the observer, as described above), did not apply here. In this test the PIIs of HDDA dist. and HDDA tech. which were determined by the same test establishment showed only a slight variation up to a maximum of 0.6. This difference has no practical relevance from the toxicological point of view. These results led to the following conclusions:

1. In this test there was no difference between the skin irritation due to HDDA dist. and that due to the technical samples supplied by different manufacturers (A-F).
2. If the skin irritation potentials of different substances are to be related to one another, comparative studies of the substances should be carried out by applying them to opposite sides of the body of the same animal, under identical conditions.

Time course of skin irritation

Recently it has been shown that HDDA leads to delayed skin irritation in man³. The first skin changes were observed only about 12-24 hours after contact, and the maximum was reached on day two or three. A close examination of the present data obtained from the animal experiments led to similar conclusions: A comparison of the findings obtained with HDDA 24 and 72 hours after the beginning of application showed that the severity of skin irritation remained the same or even increased. However, for the majority of other skin irritants, changes during this period of time normally subside. Furthermore, one week after the application of HDDA distinct skin changes were still observed, but these are not included in calculating the PH. This must be regarded as a further disadvantage if skin irritation is expressed only numerically, since slowly subsiding skin changes must be considered very critically.

Conclusions

1. Technical HDDA from different manufacturers did not show any significant differences in the levels of skin irritation.
2. The numerical values for PIIs vary considerably when determined by different investigators. A wide range of variations in PIIs may also occur when independent tests are carried out by the same establishment.
3. This shows that the comparison of skin irritation of different substances, if based exclusively on the

Table 2
Limits for the classification of skin irritation potential on the basis of the PII

VCI/ETAD	Draize ⁴	France	USA (1969)
		0	
		non irritant	
0-0.5 nicht reizend	0-2 mildly irritating	0-2 légèrement irritant	
0.6-3.0 leicht reizend			
	2-5 moderate irritants	2-5 moyennement irritant	
3.1-5.0 mäßig reizend			≥ 5 irritant
	> 6 severe irritants		
5.1-8.0 stark reizend		5-8 sévèrement irritant	

- Key:
- VCI/ETAD — Proposal of "Verein der Chemischen Industrie" and "Ecological and Toxicological Association of the Dyestuffs Manufacturing Industry".
- France — Textes législatifs Français des arrêtés du 5/4/1971 publiés au JO du 21/4/1971.
- USA (1969) — Proposed rule making of the Department of Agriculture; *Fed. Reg.*, 34, 6106 (4 April, 1969).

- numerical value of the PII, may lead to misinterpretations. Additionally, the classification of a substance as "not" to "severely" irritating on the basis of rigid limit values, taking only the PII into account, may result in controversial assessments.
4. Although animal studies into skin irritation lead to a wide variation in the numerical data, the information obtained is completely sufficient for the requirements in practice. In these studies the potential hazard of skin irritation due to HDDA was detected in every case, and a toxicologist would, under such circumstances, always give appropriate warning. This also corresponds with the many years of experience in animal studies for skin irritation; non-irritant and severely irritant substances can almost always be detected; problems arise in the intermediate "grey zone".
 5. It could be shown that exact figures for PII are of little value in data sheets and may lead to misinterpretations. Serious consideration should be given as to whether numerical values for PII can be omitted from data sheets. A rough classification such as, "non-irritating" or "irritating" would be completely sufficient in practice and for the recommendation of precautions. Furthermore, a differentiation between "slightly irritating" and "severely irritating" could also be useful, provided that not only the numerical value of the PII is taken into account but also the overall mode of action.
 6. A PII determined by different investigations cannot be used to compare the skin irritation of different substances with one another. For this purpose, a strictly comparative test on the same animal is necessary. If only a limited number of substances is to be examined, one or two defined standards may be selected for comparative purposes. As a safeguard against wrong decisions this comparative test should be used if skin irritation is the criterion for the selection of certain products by the user or the criterion for the development of new substances by the manufacturer.

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Thermal stability of microvoid coatings

By M. S. Ramaiah and W. Funke*

Regional Research Laboratory, Hyderabad, India

Summary

Microvoid coatings were prepared both from thermoplastic and alkyd resins using the solvent-non-solvent technique. In the case of alkyd resins it was necessary to replace part of the resin with a thermoplastic to obtain microvoid coatings. The contrast

ratios of the films were measured before and after heating with a reflectance photometer. The thermal stability of the films was observed from opacity measurements and from the electron-micrographs of the films taken before and after heating.

Keywords

Processes and methods primarily associated with analysis, measurement or testing
thermal degradation

Types and classes of coatings and allied products
microvoid coating

La stabilité à la chaleur des revêtements de structure microcellulaire

Résumé

Des films microcellulaires sont préparés au moyens de la technique solvant – non solvant à partir de résines alkydes ou thermoplastiques. Pour obtenir des revêtements microcellulaires, il est nécessaire, dans le cas des résines alkydes, d'en remplacer une partie par une résine thermoplastique. A l'aide d'un

photomètre à réflexion on a mesuré les rapports de contraste, avant et après l'exposition à la chaleur. La stabilité à la chaleur des films est déterminée à partir des mesures du pouvoir opacifiant et aussi des élection micrographies des films prises avant et après l'exposition à la chaleur.

Die Thermostabilität der mikrozelligen Beschichtungen

Zusammenfassung

Mittels einer Löser – Nichtlösertechnik werden Filme von mikrozelligen Beschichtungsstoffen aus Thermoplastischen- und Alkydharzen hergestellt. Im Falle der Alkydharze ist es nötig um mikrozelligen Beschichtungen zu bekommen, einen Teil dieses Harzes mit einem Thermoplastischen zu ersetzen. Die

Kontrastverhältnisse werden, vor und nach Heizung, mittels eines Reflexionsphotometer bestimmt. Die Thermostabilität der Filme werden durch Deckvermögensmessungen und auch durch vor und nach Heizung aufgenommenen Elektronenbildern bestimmt.

Introduction

Refs. 1-7

In conventional paint systems opacity is achieved by the dispersion of pigments with high refractive indices, such as titanium dioxide. Recently opacity has been achieved by the introduction of microcellular structures into the coatings which act as light-scattering loci. Such coatings are called microcellular coatings. They are also known as bubble, pitted or microvoid coatings. There is a fundamental difference between conventional coatings and microvoid coatings. In conventional coatings no incompatibility or phase separation should occur during film formation, whereas in microvoid coatings the opacity is obtained by the principle of phase incompatibility.

There are many methods by which microvoid coatings can be prepared:

- (i) The solvent-non-solvent technique¹⁻³
- (ii) The emulsion technique
- (iii) The solvent extraction technique⁴
- (iv) Using preformed air or air and pigment containing

- capsules⁵ and dispersing them in the liquid coating as a permanently dispersed phase
- (v) Using blowing agents to produce voids^{6,7}.

Normal microcellular coatings have the drawback of being thermally unstable, due to the thermoplastic nature of the binder with which the coatings are usually prepared. Hence attempts have been made in the present investigation to develop thermally stable microvoid coatings using thermosetting resins.

Experimental

Raw materials

Resins

1. Long oil linseed modified alkyd (AL 67)
2. Medium oil linseed modified alkyd (AL 52)
3. Short oil alkyd modified with mixed fatty acids (AF 25)
4. Vinyl acetate/vinyl chloride copolymer (Vinylite R)
5. Chlorinated rubber
6. Cellulose nitrate† (0.5 s⁻¹ viscosity)

All the above resins are commercially available.

*Forschungsinstitute für Pigmente und Lacke, Stuttgart, West Germany

†The cellulose nitrate used in all compositions was dry and free from any solvent which is usually used for safety reasons

Solvents

The solvents used were of commercial grade: ethyl acetate, acetone, toluene, xylene, n-butanol, diacetone alcohol, Shellsol R (a mixture of aromatic hydrocarbons with an evaporation number 550) and Kristalol 60 (a mixture of aliphatic hydrocarbons with an evaporation number 200).

Plasticizers

Dimethyl glycolphthalate (palatinol 0).

Formulation technique

The solvent-non-solvent technique was employed for the development of microvoid coatings. The method consists of dissolving the polymer or the mixture of polymers in a solvent system and then gradually adding a suitable amount of the non-solvent. The solvent should have a higher vapour pressure compared to the non-solvent and the polymer should set to a solid state rapidly, before the non-solvent leaves the film completely. To improve the flexibility of the films 10-30 per cent by weight of dimethyl glycolphthalate, based on the thermoplastic resin, was added as a plasticizer.

Application technique

Preparation of the films

The coating composition was applied onto tin foil supported by a glass plate using an automatic film applicator to get the required uniform film thickness. The film was then detached from the tin foil by amalgamation.

Films on Moresst charts

To measure contrast ratios the films were applied at a suitable film thickness over standard black and white Moresst charts, with the corresponding reflectance (R_y) values of 2.5 per cent for black and 80 per cent for white surfaces.

Testing of films, apparatus

Measurement of opacity

An Elrepho reflectance instrument (Carl-Zeiss) was used for measuring the reflectance values and contrast ratios.

Electron micrographs

The electron micrographs of the free films were taken with a Leitz-AMR 1000 electron microscope.

Evaporation number

The evaporation number of diethyl ether was taken as unity. The evaporation numbers of the other solvents were calculated with respect to this number. Thus, an increase in the evaporation number means that the particular solvent evaporates slower.

Preparation of microvoid coatings using the thermoplastic resin as the only film-forming material

Microvoid coatings were prepared using the thermoplastic resins: Vinylite, cellulose nitrate, and chlorinated rubber as the film forming materials.

The formulations of microvoid coatings from these three resins are shown below:

From Vinylite	Percentage by wt.
Vinylite R	20
Acetone	60
Butanol	20
<i>From cellulose nitrate</i>	
Cellulose nitrate	15
Ethyl acetate	55
Butanol	5
Kristalol 60	25
<i>From chlorinated rubber</i>	
Chlorinated rubber	22
Ethyl acetate	52
Butanol	26

Microvoid coatings from cross-linkable polymers

Preliminary experiments to prepare microvoid coatings using long, medium or short oil alkyds alone as the film forming media have not been successful as they are soluble in all commonly used solvents (with the exception of n-butanol) which have a fairly high evaporation number. When n-butanol (evaporation number 33) was used as the non-solvent together with either toluene (evaporation number 6.4) or acetone (evaporation number 2.1) as the solvent, even though the film appeared opaque, immediately after the evaporation of the true solvent (acetone or toluene) the dried films were transparent. This was due to the fact that the alkyd films, unlike the thermoplastic films, take more than an hour to dry; in the meantime the non-solvent may leave the film before it has dried completely.

In order to obtain microvoid films from alkyd resins, it is therefore necessary to modify them suitably by blending with other thermoplastic resins so that they set to dry films more rapidly. Experiments carried out by blending alkyd resins with Vinylite, chlorinated rubber or cellulose nitrate showed that a minimum of 1:1 (w/w) parts of thermoplastic resin are required to obtain opacity, when the thermoplastic content is below this ratio the films are either clear or translucent. It is also observed that with further increase in the thermoplastic content, the increase in opacities of the films is not appreciable. Thus, in further experiments the thermoplastic resin was added to the alkyd resin in the proportion 1:1 (w/w) and the resin solutions were prepared with 20 per cent resin solids. These solutions were applied on both tin foil and Moresst charts at a wet film thickness of 300 microns. Coatings without microvoids possessed a dry film thickness of about 60 microns; when microvoids were present, the film thickness was found to be almost double. The contrast ratios and the sizes of the microvoids are shown in Table 2. The sizes of the microvoids were measured from electron micrographs.

Thermal stability and hiding power of microvoid coatings

In order to determine the thermal stability of the microvoid coatings based on thermoplastic resins alone and thermoplastic resins mixed with cross-linkable polymers the following compositions, based on Vinylite and cellulose nitrate shown in tables 3 and 5 respectively, were used; their free films were prepared as mentioned earlier.

Free film strips measuring 5 cm × 6 cm of all the ten

Table 1
Contrast ratios of microvoid coatings from thermoplastic resins

Film	Dry film thickness	R _y value over black	Size of microvoids in microns	Contrast ratio
Vynlite	70	70.55	3-5	0.85
Cellulose nitrate	75	68.22	3-6	0.82
Chlorinated rubber	95	84.50	2-3	0.95

Table 2
Contrast ratios of microvoid coatings from a mixture of thermoplastic and thermosetting resins in the ratio 1:1 (w/w)

Composition	Dry film thickness	R _y value over black	Size of microvoids in microns	Contrast ratio
Chlorinated rubber and alkyd L67 in ethyl acetate, toluene and butanol	110	82.20	3-5	0.96
Vynlite and alkyd L 67 in acetone, toluene and butanol	115	77.85	3-6	0.92
Cellulose nitrate and alkyd F 26 in ethyl acetate, xylene and Kristalol 60	85	81.50	2-3	0.97

Table 3
The compositions of microvoid lacquers based on Vynlite and alkyd resin (the thermal stability and other properties of these lacquers are shown in Table 4)

Composition No. of lacquers	Vynlite	Alkyd L 67	Acetone	Toluene	Butanol
1	25		70		30
2	15	10	45	5	25
3	15	15	40	5	25
4	10	20	30	10	30

compositions were prepared. The thicknesses, specific gravities and contrast ratios of the films were measured both before and after the films were kept at the specified temperatures for the specified time interval (tables 4 and 6). The films were kept stretched whilst in the oven. Electron micrographs of the cross-sections, substrates and bottom surfaces of the films, were taken both before and after heating.

The results are shown in Table 4 and Table 6.

Results and discussion

Thermoplastic resin content in microvoids from cross-linkable polymers

In order to obtain a microvoid film from cross-linkable polymers such as alkyd resins, it was found that a minimum ratio of 1:1 by weight of a thermoplastic resin

had to be mixed with the cross-linkable polymer. It was also observed that the resin system had to set to a non-tacky solid state before the non-solvent evaporated completely.

Opacity of microvoid films

By comparing the contrast ratios of the microvoid films obtained with the thermoplastic resins Vynlite, cellulose nitrate and chlorinated rubber when used alone as the film forming material (Table 1) and in combination with alkyd resins (Table 2), it was observed that the contrast ratios of films from thermoplastic resins increased when in combination with alkyd resins.

Thickness of the microvoid coatings

Due to the presence of microvoids the thickness of the

Table 4
Thermal stability of microvoid coatings (free films)

Composition No. of lacquers* and treatment	Dimensions in cm, area in cm ²	Thickness in microns	Volume in cc	Weight in g	Specific gravity g/cc	R _v over black	Contrast ratio
1 Before heating	5.0×6.0 = 30.00	35	0.105	0.0769	0.7324	70.55	0.8459
After heating† at 100°C for 1 hour	2.9×3.5 = 10.15	80	0.081	0.0523	0.6441	29.77	0.3675
2 Before heating	5.0×6.0 = 30.00	120	0.360	0.2068	0.5744	77.85	0.9137
After heating at 160°C for 1 hour	4.8×5.7 = 27.36	120	0.325	0.1997	0.6145	72.70	0.9070
3 Before heating	5.0×6.0 = 30.00	93	0.279	0.1747	0.6262	65.05	0.7931
After heating at 210°C for 1 hour	4.9×5.9 = 28.91	93	0.268	0.1716	0.6381	57.70	0.7346
4 Before heating	5.0×6.0 = 30.00	92	0.276	0.2527	0.9156	58.27	0.7346
After heating at 160°C for 1 hour	4.9×5.9 = 28.91	92	0.270	0.2460	0.9111	55.80	0.7256

*For the composition of the lacquers see Table 3

†Above 100°C the film became first clear then lost its shape and finally changed to a lumpy black mass

Table 5
Composition of the clear and microvoid lacquers based on Alkyd L 52 and cellulose nitrate (the thermal stabilities of these lacquers are shown in Table 6)

Composition No. of lacquers	Alkyd 52	Cellulose nitrate	Ethyl acetate	Kristolol 60	Palatinol 0
1	20		70	30	
2		20	70	30	3
3	4	16	70	30	3
4	5	15	70	30	3
5	7	13	70	30	3
6	10	10	70	30	3

microvoid coatings was observed to increase to almost twice that of the clear film, applied at the same wet film thickness.

Thermal stability of the microvoid coatings, physical study

Microvoid films with cellulose nitrate as the film-forming material with or without an alkyd resin as the second resin component were found to be stable at the relatively high temperatures of 160-170°C for one hour. No significant change in the thickness, specific gravity, weight or the volume of the films was observed. Even the opacity of the films remained almost the same. The films did however, shrink slightly (Table 6).

In the case of microvoid films obtained from Vinylite as the only film-forming vehicle, it was observed that the microvoids started collapsing at almost 100°C. Thus around 100°C the films began to lose their opacity turning almost clear; then they began to lose their shape. Above 100°C the films turned into a lumpy black mass,

completely losing their shape due to melting (Table 4, composition 1). On the other hand, the results of the experiments conducted with mixtures of long oil alkyd and Vinylite showed that in these films there was no significant change in the physical properties of the films, even when they were heated to 160°C for 1 hour (Table 4, compositions 2 and 4); the opacity was only slightly reduced. In the case of composition 3 (Table 4), where the film was heated to 210°C for 1 hour, the physical properties remained almost the same even though the volume decreased slightly with a corresponding increase in the specific gravity; the thickness remained the same. The shrinkage of the film was not very significant compared with composition 1 where only Vinylite was used.

Study of the electron micrographs

The electron micrographs of the cross-section (figures 1 and 2), bottom surface (figures, 3, 4, 5 and 6) and top surface (figures 7 and 8) of the film of Vinylite mixed with long oil alkyd (composition 3, Table 4) were taken before and after heating at 210°C and show that the film

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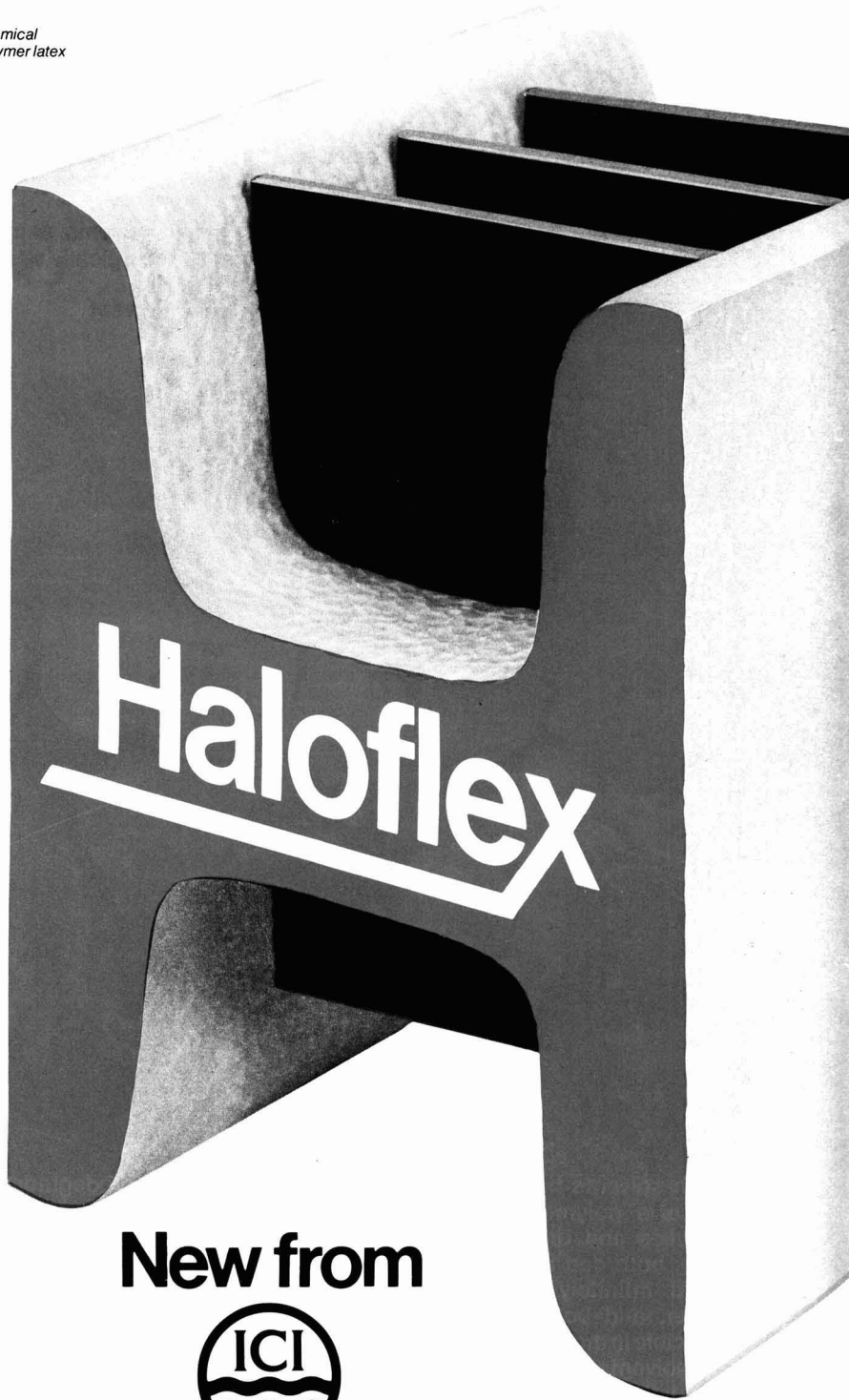
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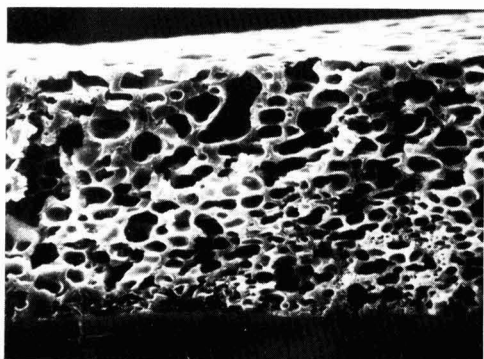


Figure 1. Electron micrograph of the cross-section of a Vinylite and long oil alkyd film (1:1) before baking, magnification ca. $\times 380$

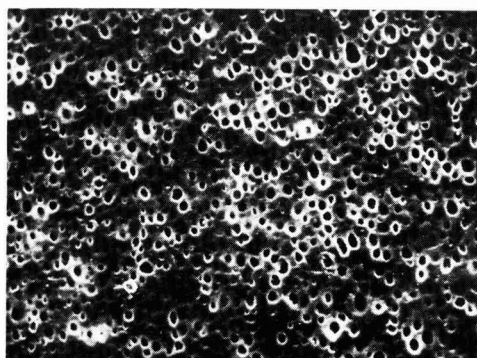


Figure 3. Electron micrograph of the bottom surface of a Vinylite and long oil alkyd film (1:1) before baking, magnification ca. $\times 140$

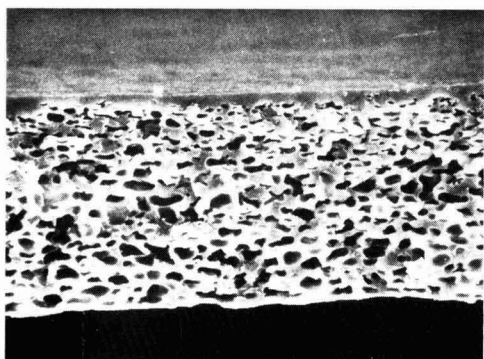


Figure 2. Electron micrograph of the cross-section of a Vinylite and long oil alkyd film (1:1) after baking, magnification ca. $\times 380$

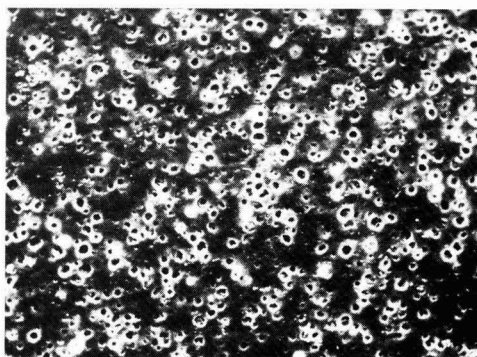


Figure 4. Electron micrograph of the bottom surface of a Vinylite and long oil alkyd film (1:1) after baking, magnification ca. $\times 140$

Table 6
Thermal stability of microvoid coatings (free films)

Composition No. of lacquers	Dimensions in cm area in cm ²	Thickness in microns	Volume in cc	Weight in g	Specific gravity g/cc	R _v over black	Contrast ratio
1 Before heating	5.0 \times 6.0 = 30.00	17	0.051	0.0648	1.2706	6.65	0.1106
After heating	5.0 \times 6.0 = 30.00	17	0.051	0.0638	1.2510	6.65	0.1101
2 Before heating	5.0 \times 6.0 = 30.00	55	0.165	0.1263	0.7655	57.80	0.7062
After heating	4.9 \times 5.9 = 28.91	55	0.159	0.1225	0.7704	57.85	0.7211
3 Before heating	5.0 \times 6.0 = 30.00	85	0.255	0.1478	0.5798	87.00	0.9753
After heating	4.9 \times 5.9 = 28.91	85	0.246	0.1431	0.5824	86.35	0.9757
4 Before heating	5.0 \times 6.0 = 30.00	73	0.219	0.1468	0.6705	83.60	0.9686
After heating	4.9 \times 5.9 = 28.91	73	0.211	0.1422	0.6734	83.40	0.9614
5 Before heating	5.0 \times 6.0 = 30.00	85	0.255	0.1669	0.6545	84.60	0.9658
After heating	4.9 \times 5.9 = 28.91	85	0.246	0.1621	0.6597	81.82	0.9624
6 Before heating	5.0 \times 6.0 = 30.00	93	0.279	0.1677	0.6012	88.80	0.9810
After heating	4.9 \times 5.9 = 28.91	93	0.269	0.1630	0.6063	87.10	0.9739

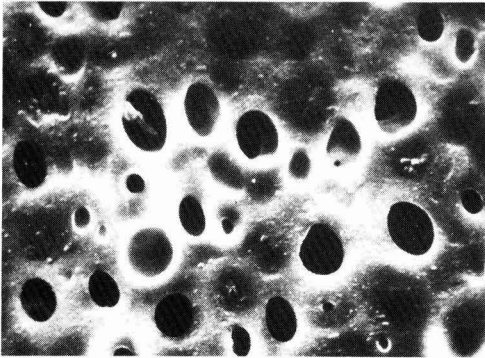


Figure 5. Electron micrograph of the bottom surface of a Vinylite and long oil alkyd film (1:1) before baking, magnification ca. $\times 700$

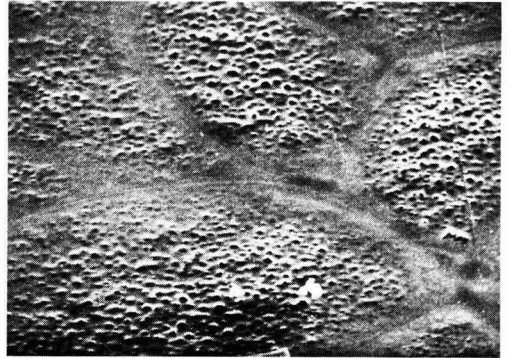


Figure 7. Electron micrograph of the substrate of a Vinylite and long oil alkyd film (1:1) before baking, magnification ca. $\times 140$

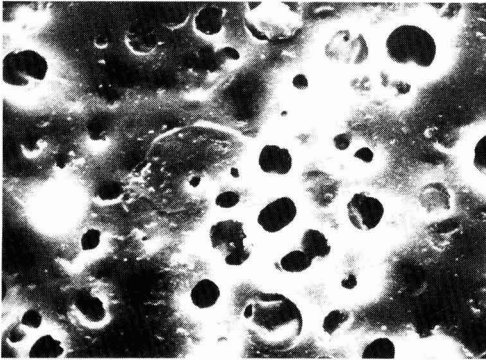


Figure 6. Electron micrograph of the bottom surface of a Vinylite and long oil alkyd film (1:1) after baking, magnification ca. $\times 700$

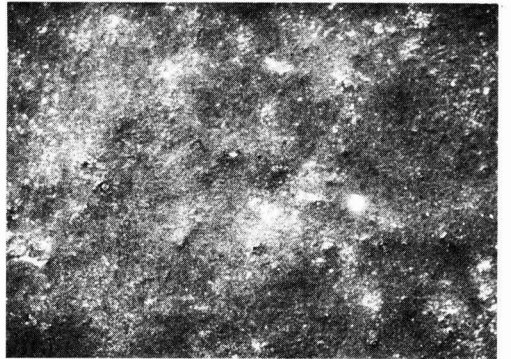


Figure 8. Electron micrograph of the substrate of a Vinylite and long oil alkyd film (1:1) after baking, magnification ca. $\times 140$

morphology did not change significantly on baking. The microvoids did not collapse and the microvoid structure was found to remain almost the same, both at the bottom surface and cross section, even after baking. However, the top surface of the microvoid film changed significantly (figures 7 and 8). The open pores found on the substrate before baking were completely filled with the melted thermoplastic resin after baking. This is readily observed in figures 1 and 2, where the cross-section of the microvoid film is seen and the top surface is partially visible (the bright portion above the cross-section in Figure 1, and above the microvoids in Figure 2).

Thus, from both a study of the physical properties of the films and a study of the electron micrographs, it may be stated that in the presence of alkyd resins the microvoids formed in the vinyl copolymer are not deformed even at temperatures around 210°C , whereas without alkyd resins the film melts into a lump. Obviously, when thermoplastics are mixed with cross-linkable polymers such as alkyd resins, there is a protective action by the cross-linkable polymers on the structural stability of the thermoplastic resin. Presumably this stability is due to the morphological structure of the two resin phases in the microvoid films as shown in figures 9 and 10. The

structural arrangement might either be as in the case of Figure 9 or as in the case of Figure 10 or both.

If the arrangement of the microvoids after baking were as in figures 9c and 10b, then much more shrinkage, a significant increase in the specific gravity and a decrease in the contrast ratios of the films would be expected. Such shrinkage and reduction in contrast ratio, though observed (tables 4 and 6) was found to be insignificant. Also, the electron micrographs did not show any significant collapse of the microvoids. Thus it can be concluded that the arrangement of the microvoids throughout the film was as shown in figures 9a and 9b, with the possibility of a minor proportion of the structure being arranged as shown in figures 9c and 10b. The loss in weight on heating may have been due to the loss of the retained solvent in the film. In the case of the clear varnish film (composition 1, Table 6) the loss in weight was 1.5 per cent and in the case of the microvoid coatings it was about 3 per cent. Thus it may be concluded that microvoid coatings retain more solvent than clear coatings.

Acknowledgements

The authors acknowledge with thanks, the assistance

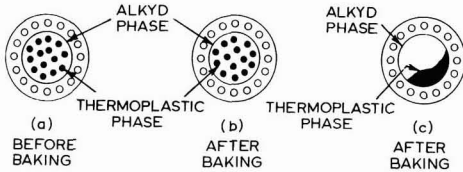


Figure 9. Hypothetical arrangement of the resin phases

given by Mrs Gretel Handloser and Mr Horst Zattloulal of Forschungsinstitute für Pigmente und Lacke, Stuttgart, West Germany.

[Received 8 December 1980

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1. Belgian patent; 760,690; 1971.

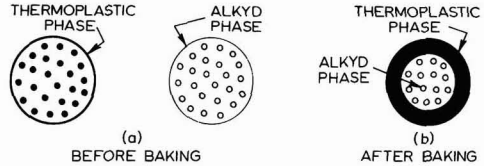


Figure 10. Hypothetical arrangement of the resin phases

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3. French patent; 2,083,687; 1972.

4. British patent; 1,193,865; 1970.

5. Rosenthal, W. S. and McBane, B. N., *J. Paint Techn.*, 1973, **45**, 584.

6. US patent; 3,615,972; 1971.

7. US patent; 2,978,340; 1961.

Next month's issue

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

Organotins in wood preservation by *C. J. Evans and R. Hill*

Recent formulation developments in chlorinated rubber paints by *R. G. Humphries*

Further investigation of the photo-induced oxidation of normal primary alcohols by anatase titanium dioxide by *M. A. Malati and N. J. Seager*

Estimation of shellac by *N. Prasad and B. B. Khanna*

Corrigendum

The outlook for the eighties

By F. M. Smith, *JOCCA*, 1981, **64**, 144-155.

The numbering of figures 11 and 12 were inadvertently transposed and they should have read as follows:

Figure 11. Cyclical movement of pigment sales

Figure 12. Cyclical launching of products

Short Communication

The analysis of alcohols using N-bromosuccinimide

By J. K. Haken and D. Srisukh

The University of New South Wales, PO Box 1, Kensington, NSW 2033, Australia

Introduction

Refs. 1-4

A recent paper¹ has reported the use of N-bromosuccinimide for the volumetric analysis of n-butanol in nitrocellulose thinner. While the paper indicates that the reagent does not react with the other solvents present in the mixture, i.e. butyl acetate and toluene, it should be stressed that the method is not generally applicable to thinners for nitrocellulose lacquer as a procedure for the analysis of n-butanol. Many thinners for nitrocellulose materials contain substantial amounts of alcohols of lower molecular weight than n-butanol and all of these will react with N-bromosuccinimide similarly, iso-butanol is also used and to lesser extents high boiling solvents containing a hydroxyl functional group. The general reactivity with alcohols is not unexpected as N-bromosuccinimide has been studied for several decades¹. The present work further shows the widespread use of the reagent and it is suggested that simple gas chromatography provides a rapid procedure for the identification of all of the components of a simple thinner blend, which may be supplemented by other chemical or instrumental methods for complex mixtures²⁻⁴. With complex mixtures abstraction of alcohols with N-bromosuccinimide might be employed, however testing to ensure that reaction with other functional groups did not occur would be necessary.

Results and Discussion

Refs. 5-10

The general reactivity was demonstrated by preparation of 1 ml samples of 5 per cent of each alcohol in n-paraffin in a 2 ml glass vial fitted with a serum cap (Reaction Vials No. 6602A, Alltech Associates, USA). n-hexane was generally used as solvent, however depending on the gas chromatographic column overlap occurred with some C₃ and C₄ alcohols and heptane was substituted. A sample of the solution i.e. 0.1 μl, was injected into the chromatograph to ensure separation after which 1 ml of N-bromosuccinimide saturated solution and 0.1 ml glacial acetic acid were injected into the vial which was vigorously shaken. The reaction time varied and tended to be longer for the branched chain alcohols. The reaction time, where necessary, was greatly reduced by agitation in a stream of warm air. The samples, after reaction, were re-injected into the chromatograph i.e. 2 μl, and the decrease in peak height or area was measured. Complete removal of all of the alcohols was shown to occur in 2-10 minutes by the absence of the alcohol peak.

Table 1 shows the range of alcohols removed where it is apparent that hindered alcohols such as tertiary butanol are reactive as are glycol ethers i.e. ethylene glycol monoethyl ether, diethylene glycol monoethyl ether and a ketoalcohol.

Figure 1 shows a chromatogram of all of the alcohols considered which may be resolved in boiling point order by temperature programming on a non-polar dimethyl polysiloxane (SE-30) column of reasonable length, i.e. 20 feet. Separation of all of the simple alkanols, Figure 2, may be carried out isothermally using Armeen SD (Mixed primary amines, Armour Chemicals, USA) as the stationary phase^{5,6}. The low temperature limit, i.e. 75°C, of the materials prevents separation of the higher molecular weight compounds which may be separated using Z6020 (N-β-aminoethyl-γ-aminopropyl trimethoxy silane) (Dow Chemical Co., Midlands) as the stationary phase⁷. The separation of alcohols considered is shown in Figure 3. Figure 4 shows a chromatogram with ethanol, n-butanol and cellosolve resolved on a dimethylpolysiloxane column before and after addition of N-bromosuccinimide.

The use of gas chromatography in solvent analysis has found almost universal acceptance and has been widely reported⁸ over the last 25 years. An early report being that of Whitham² in 1956, who supplemented the usual chemical and physical tests for solvent mixtures with gas chromatography, examining both the sample and the fractions obtained by the use of the fluorescent indicator absorption technique developed by Ellis and Le Tourneau^{9,10}.

[Received 23 January 1981]

Table 1
Alcohols examined (the reaction with N-bromosuccinimide was complete in every case)

Chemical name	Trade name or synonyms	b.p. °C
Methanol		64.5
Ethanol		78.3
2-propanol	iso-propanol	82.3
2-methyl-2-propanol	tert-butanol	82.5
1-propanol	n-propanol	97.0
2-butanol	sec-butanol	99.5
2-methyl-1-propanol	iso-butanol	107.2
1-butanol	n-butanol	117.7
1-pentanol	n-amyl alcohol	138.0
4-hydroxy-4-methyl-2-pentanone	diacetone alcohol	169.2
2-ethoxy ethanol	ethylene glycol monoethyl ether Cellosolve*	135.6
2-butoxy ethanol	ethylene glycol monobutyl ether butyl Cellosolve*	176.1
Ethoxydiglycol	diethylene glycol monoethyl ether Carbitol*	201.0

*Union Carbide trade names. Equivalent products are offered internationally by The Shell Company and Imperial Chemical Industries.

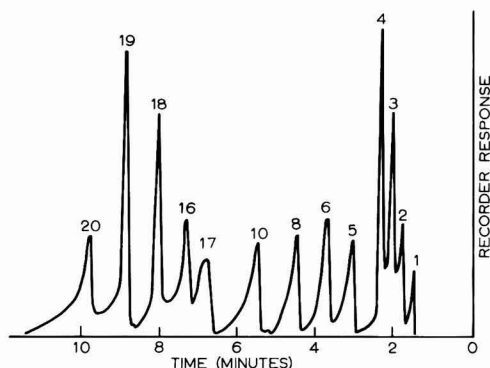


Figure 1. Chromatogram showing separation of alcohols using 12 ft \times 1/4 in. OD column packed with 10 per cent SE-30 on Chromosorb W. Temperature programme 30-200°C at 15°C/min. post injection 4 minutes. Alcohols (1) methanol, (2) ethanol, (3) 2-propanol, (4) tert-butyl alcohol, (5) 1-propanol, (6) 2-butanol, (7) 2-methyl-2-butanol, (8) isobutyl alcohol, (9) 2-methyl-3-butanol, (10) 1-butanol, (11) 3-pentanol, (12) 2-pentanol, (13) 2,2-dimethyl-1-propanol, (14) 3-methyl-1-butanol, (15) 2-methyl-1-butanol, (16) 1-pentanol, (17) Cellosolve, (18) diacetone alcohol, (19) butyl Cellosolve, (20) Carbitol

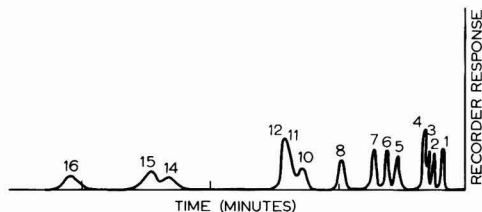
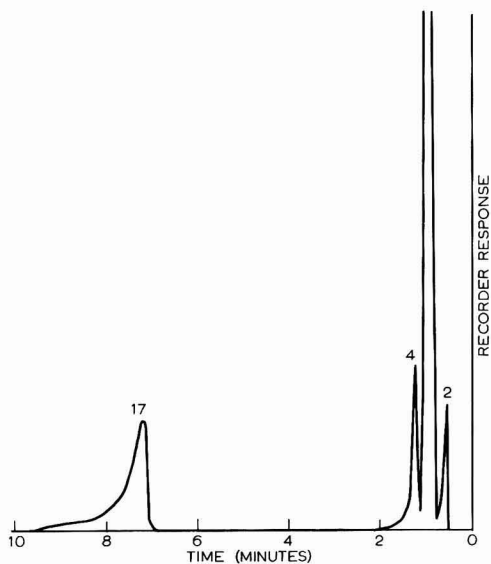


Figure 2. Chromatogram showing separation of alcohols using 20 ft \times 1/8 in. OD column with 10 per cent Armeen SD operated isothermally at 75°C. Compounds as Figure 1

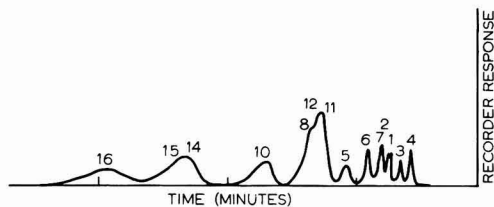


Figure 3. Chromatogram showing separation of alcohols using 24 ft \times 1/4 in. OD column packed with 10 per cent Z6020 on Celatom AWDACS operated isothermally at 100°C. Compounds as Figure 1

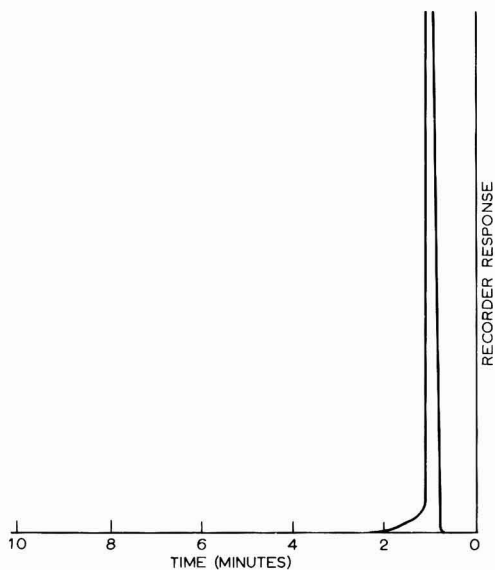


Figure 4. Chromatograms using SE-30 column (as Figure 1) showing separation of alcohols (top) and removal of alcohols on addition of N-bromosuccinimide (bottom)

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3. Haken, J. K. and McKay, T. R., *JOCCA*, 1964, **47**, 517.
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6. Zarembo, J. E. and Lysyl, I., *Anal. Chem.*, 1959, **31**, 1833.
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reviews

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

Paint Chemistry and Principles of Paint Technology

By G. P. A. Turner
Science Paperbacks, Chapman & Hall Ltd
pp.213 + App. & Index Price £6.50

This book is clearly stated to be on paint chemistry and technology, not one on pigment or resin chemistry or technology. Part one covers theoretical organic and inorganic chemistry from the surface coatings aspect with sufficient physical chemistry and physics included to explain the mechanism of colour formation in organic compounds.

Part two deals with science applied to surface coatings. There are ten chapters, beginning with the constituents of paints of various well-known types, their application, methods of drying, and properties.

The author then proceeds to describe the various ingredients in as much detail as one could justifiably

expect in a book of this size, with a strong bias towards media. These are dealt with under the headings of lacquers, emulsion paints and non-aqueous dispersions, oil and alkyd paints, thermosetting paints, epoxy coatings, polyurethanes and unsaturated polyesters and acrylics.

There is also one chapter on additives, one on solvents, and one on pigmentation.

Having written the above, what of its value to the person to whom it is obviously addressed, the trainee/student? The approach is logical, and part one could almost be used as a primer (in the printed sense) for anyone wanting a general approach to technical chemistry.

Part two does suffer slightly by a neglect of more detailed descriptions of the pigments compared with the resins, but with the proliferation of resins during the last 10 years, and the need to keep up to date, this can be forgiven. The chapter on solvents leaves little to be desired, it deals with the solubility parameter concept of Burrell, and includes a table of solvent properties, and one of solubility parameters. Thus anyone reading this book will obtain a good basic knowledge of the "hows and whys" of what is an extremely complex technology, produced in a very readable way.

Reader Enquiry Service No. 21

D. S. Newton

occa meetings

For information on membership of OCCA, enquiries should be sent to the Association's offices, see front cover for address.

various pieces of sophisticated equipment were seen operating.

The whole visit was very impressive, and showed the way microchip technology was changing our approach to chemistry.

A. W. Fell

Thames Valley

Works visit to Perkin Elmer

The Thames Valley Section meeting on Thursday 22 January was a works visit to Perkin Elmer. The party was initially given light refreshment, followed by a general talk on the major instrumental analytical techniques available from Perkin Elmer, which are IR and chromatography. The major advances have been in better resolution achieved by the equipment, and the introduction of computer interpretation of results.

Computers can now give probable interpretations of IR spectra, or produce a visual display on a VDU, from which known spectra can be removed or areas of differences between known and unknown spectra can be highlighted. This technique could be used for quality control to compare relative purity, or for the comparison of similar materials containing slightly different amounts of minor additives.

The party was shown round the display area where

Manchester

Pigments for printing inks

Dr J. D. Sanders of Ciba-Geigy, P & A Division, delivered his lecture to 75 members and guests at the Manchester Polytechnic, All Saints, Manchester on Wednesday 21 January 1981. Commencing his lecture with the statement that the "printing ink industry tends to solve its problems by formulation techniques". He stressed that the pigment chemist must have a working knowledge of ink technology to solve the problems in the printing ink industry. The importance of the latter to the pigment industry in commercial terms can be illustrated by the relative pigment loading values as stated by Dr Sanders:

printing ink industry	15-20 per cent
paint industry	5-8 per cent
plastics industry	0.5-1.0 per cent

General properties of pigments which included

strength/flow/lithographic performance were listed under the following headings:

Ink production; dispersibility, flow, heat stability, storage stability and gloss promotion

Printing operation; litho performance flow and strength

Applied properties; hue/brightness, gloss, resistance properties i.e. soap, and transparency

The lecturer then discussed the following subjects which can be summarised thus:

Organic pigments

1. Pigmentary colour – insoluble in water
2. Toners – soluble but converted into insoluble salts, i.e. Na,Ca
3. Lakes – water soluble dye precipitated onto aluminium hydrate

Pigment types described were as follows:

Azo pigments – i.e. hansa yellow 10G/5G/yellow G
Diarylide yellow pigments – double molecular weight – limited solubility in toluene

Diarylide orange pigments – orange 13 and 34

Azo reds – limited use in printing inks, used in paint industry

Arylamide reds – pigment red 112, 2, 7, 5, 12

Red toners – calcium 4B toner – main line process red in PI

Calcium 2B toner – increased lightfastness/yellow end of structure

Phthalocyanine blue – excellent heat/lightfastness

Phthalocyanine green – excellent heat/lightfastness plus stability to action of solvents

Fanals – violets and green

Methyl violet – phosphomolybdic acid – pigment violet 3 – poor bleed resistance

Methyl violet – copper ferrocyanide – pigment violet 27 – poor bleed resistance

Performance factors and the ink chemist

Transparency – to convert opaque type to transparent type, modification with rosin or derivatives

Dispersibility – method of manufacture of pigments – cohesive forces/crystal growth, methods of improvement include resination or flushing

Ink stability – surface active agents – improve flow, chemical treatment of pigments during manufacture

Gloss properties – improvement achieved by use of pigment chips

Bronzing – caused by particles of pigments (fines) on the surface and partial reflection

Resistance properties – lightfastness, increase chlorination alkali fastness – increase complexity

Dr Sander's lecture was concluded by questions raised in relation to subjects as diverse as bronzing, resination of benzidines, fluorescent pigments, toxic properties of organic pigments including the use of benzidine which is carcinogenic with dichlorobenzidine present in diarylamide yellow pigment at 2 ppm as a viable alternative. R. Stott proposed a vote of thanks which terminated this very interesting lecture of one and a half hours duration.

Water based printing inks and their related problems

The above lecture was delivered at the joint meeting of the North Western Branch of the Institute of Printing and the

1981(5)

The logo for OCCA meetings is enclosed in a rounded rectangular border. The word 'OCCA' is written in a large, bold, black, sans-serif font. Below it, the word 'meetings' is written in a smaller, lowercase, black, sans-serif font.

Manchester Section of OCCA on Thursday, 15 January 1981 at the Woodcourt Hotel, Sale. Seventy-five members and their guests heard Geoff Hutchinson, Technical Director of Croda Inks Ltd, Edinburgh and an OCCA member commence his lecture by stating the reasons for the advent of water based inks. These included such factors as packaging odours, safety, environmental pressures and the conservancy of expensive solvents.

Compliance with the maxim of "necessity being the mother of invention" began in 1974 with the "great toluene shortage". Gravure inks were particularly affected and football coupons were one of the innovative users of water based inks (WBI). The following year, 1975, saw the return of toluene in abundance and a relative decline in the WBI.

The lecturer discussed the notable Rule 66 of Los Angeles which particularly affects users of solvent based gravure/web offset inks and predicted that Rule 66 will be in operation throughout USA in 1982. Mention of UK regulations was in reference to the H and S Act, 1974, Section 5 which states that "no manufacturer shall emit obnoxious materials into the atmosphere".

Alternative options to the use of WBI are:

1. Solvent recovery plant – expensive
2. After burners

Geoff Hutchinson continued his lecture by describing the four main types of printing as:

1. Letterpress
2. Offset litho
3. Flexographic
4. Gravure

Plus silk screen printing showing increasing usage.

The incorporation of up to 20 per cent water in the inks used in methods 1 and 2 was discussed, the advantage of cost reduction was balanced by the evaporation rate of water in relation to roller temperatures.

The flexographic method of printing gives several advantages in relation to WBI including relatively low roller temperatures. The history of flexographic ink types was presented as follows:

1. Shellac – good resin – shellac/rosin maleate + ammonia – water solubility – acid board – insolubility.
2. Early 1970s – styrene acrylic copolymers.
3. 1974 – conventional flexo inks – alcohol – expensive – low flash point.
4. Basic dyes – tannic acid/high b.p. glycol – water reducible.

Further subjects discussed in Geoff Hutchinson's 90 minute lecture could be summarised thus:

1. 1980 – corrugated box industry – gloss inks, litho 4 microns, flexo 7-8 microns and gravure 10 microns.

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OCCA meetings

2. WB gravure – problems of drying in the cell – depth 37 microns with the shellac/alcohol amine type.
3. Reaction in gravure recess between shellac and chromium.
4. 1974 – WBI plus toluene overcoating lacquer.
5. WB gravure ink – inferior to 2 pack and urethane solvent system especially in relation to freezer packs.
6. Claimed LH of water 4 times greater than organic solvents. Remedial procedure is to apply higher energy or reduce roller speeds.
7. WBI – absorption on paper/board satisfactory but application to polythene and foil very difficult – post treatment using alternative drying methods such as radio wave, microwave, infrared are necessary.
8. WBI generally eliminated from the letterpress and litho methods but use of a WB varnish over a partially set litho ink is a valid alternative to the use of spray powders which were claimed to be a health hazard.
9. WBI – American developments – chemistry – carboxyl groups and amines added to emulsion polymer to give “solubility in alkaline water”. pH control is all important. Water resistance – improved by the addition of zinc ammonium carbonate. Unfortunately sensitivity to ammonia remains.
10. Drying – 1966 – *Coventry Telegraph* – webb offset – microwave 2450 MHz – suitable for polar materials only – oversold – “Star Trek thinking”. Radio frequency – 27 MHz/4 KW unit – improvement on microwave type.
11. Reference to the 1977 OCCA Conference – Hardisty, Bath University paper on WB gravure varnishes – radio frequency and air flow very important.

The lecture was followed by a discussion period during which questions on the following subjects were answered by the lecturer.

1. Use of dyes vs. pigments.
2. WB metallic inks – stabilised gold powder plus wetting agent – claimed no real instability problems!
3. Foaming/cissing.
4. Corrosion.

John Inskip, Chairman of the NW Branch of the IOP concluded this comprehensive lecture by delivering a vote of thanks which was warmly applauded.

Colour match prediction

Friday 13 February 1981, was the date that Dr Ian Bridgeman of Ciba-Geigy, Pigments Division gave his lecture to the Manchester Section at the Manchester Polytechnic, All Saints.

The lecturer defined his approach to the subject as the kind of system that can be used, the ways in which it can assist and some of the problem areas. The system used at Ciba-Geigy is based on the use of a G.E. Hardy spectrophotometer possessing a degree of antiquity combined with a relatively slow speed. The data obtained from this instrument, consisting of pattern measurement, is passed

into the second half of the operation which consists of a Hewlett-Packard 2100 series disc computer with a 32K core memory which houses the match prediction programs. Before any match prediction system can operate it needs to know how strongly a colourant absorbs and scatters light at each wavelength across the visible spectrum. This involves making calibration patterns which consist of various concentrations of the colourant dispersed into the appropriate medium. Normally five different concentrations for each colourant are prepared, this number is not mandatory but was described as realistic in relation to the range over which the colourant might be employed. This allows for the exclusion of one or even two patterns that might produce doubtful results.

Dr Bridgeman continued his lecture with the use of slides and the various subjects covered can be summarised thus:

Pigment Library: it relates to one batch of pigment, one dispersion technique, one substrate and one application (e.g. spray, brush, drawdown etc.). It is very important to fully understand the consequences of altering these parameters in terms of the numbers of libraries that are required to achieve the desired degree of accuracy for any given substrate.

Plastics: basically straight forward, except for spread coating applications, one library has been found to be adequate.

Speed: by using the first prediction and subsequent correction routines in place of manual matching the average times to match a colour can be reduced by 50-73 per cent.

Pigment Choice: variable number of different colour combinations, maximum use of six pigments in each formulation, produces a broad information base on how to match a given shade in terms of cost, metamerism and application properties before requiring practical work.

Cost: the target is that formulation which just succeeds in hiding a moresat chart at the required film thickness. The white content is treated as a standard pigment, this makes it possible to obtain the optimum hiding pigmentation levels very quickly and keeps the cost of the formulation down to an absolute minimum.

Correction routine: this facility provides a means of correcting variations from standard that might exist in the pigment, dispersion technique and substrate.

Metamerism: when a non-metameric match is required selection of the correct pigments is often “hazardous” and matchings are often dependant on type of light source. It follows that mis-matching can occur with variation in illuminant, CMP provides a fast efficient method of quantifying the degree of metamerism involvement before practical work is performed.

Pigment inventory: visual matching usually involves an addition to the users colour library by adding an extra colour to an existing pattern. CMP drastically reduces the need to use more than two colours.

Rationalisation: the lecturer claimed that CMP provided a faster solution to the problem of pigment withdrawal by the manufacturer.

Versatility: this claimed asset of CMP can be itemised as follows:

1. Substrate variation
2. Input of tri-stimulus values or chromaticity coordinates to replace reflectant values
3. Mismatches – deliberate prediction to increase use of white, black and oxides to reduce total costs

Dr Bridgeman then discussed the problems associated with CMP and his observations are listed below:

Human fear: people, rightly or wrongly, are afraid that their jobs or their job interest will be threatened by the introduction of CMP. Against this background, mistakes made by CMP are usually of monumental proportions, caused by operator error, but exaggerated and often made memorable by many human opponents.

Accuracy: good with pastel shades, decreasing value as colour depth increases.

Heavy shades: still problematical in relation to colour matching, caused by the relatively low levels of illumination which in turn requires greater accuracy from the instrument, this is the main area of deficiency.

In conclusion Dr Bridgeman stated that companies cannot afford to be without CMP and a professional colourist. Colour match prediction, certainly in the short-medium term, can only be an aid to the colourist and never a complete replacement.

A comprehensive question period followed this interesting colourful lecture and included reference to a 10 pigment library for BS4800 matching, 150 pigments for ball milling and 50 pigments for sand mills. The vote of thanks given by Stanley White was unanimously received.

F. B. Windsor

London

Low odour hydrocarbon solvents

The third meeting of the 1980/81 session was held on 15 January 1981 at the Princess Alice, Forest Gate E7. Mr Alan Cumbers of Carless Solvents Limited presented a talk on "Low odour hydrocarbon solvents" to an audience of 40 members and guests.

Following a general introduction to solvents and an illustrated presentation on Carless's refinery, Mr Cumbers discussed in more detail the various types of low odour solvents available.

Of the three main classes of hydrocarbon solvents widely used – aromatic, naphthenic and paraffinic, the former exhibited the greatest solvent power and the strongest odour. Paraffin hydrocarbons, particularly isoparaffins were of the lowest odour, but were also weakest in solvent power. They were not widely used in coatings and allied products because of their poor solvency. There was some use in polishes.

The general low odour solvents which had been introduced some years ago to the coatings industry were essentially naphthene rich solvents. Solvent suppliers were able to provide a range of solvent blends which matched the solvent power of equivalent distillation range aromatic hydrocarbons. As well as being of lower odour, these solvents generally had higher threshold limit values (TLVs) than the aromatics and this was advantageous where solvent toxicity was of concern.

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The third type of low odour solvents were low odour printing ink distillates. Traditionally three distillation ranges (220-240°C, 260-290°C and 280-320°C) had been used. They had approximately 18 per cent aromatic hydrocarbon content. These aromatic solvents could cause taint in foods and confectionery and odours from drying ovens. Low odour versions had been manufactured by acid washing these grades and reducing the aromatic content to 2 per cent. With increasing controls on waste disposal, dumping of spent acid became more difficult and hydrogenation of the solvents was now employed. Whilst this resulted in higher aromatic content (5-6 per cent) they were acceptable for taint and odour.

The aspect of TLVs were discussed in more detail, particularly the importance placed on them by customers and the relevance of the values set out by the "experts" for particular solvents. To illustrate this point the TLVs set by different countries for the same solvents were shown to be widely varied. It was also possible to influence and change the TLVs set by e.g. The American Conference of Industrial and Governmental Hygienists.

Mr Cumbers then went on to describe and illustrate the facilities and test equipment used by Carless in their laboratories for control and analysis of their products.

Following a question time, a vote of thanks was proposed by Mr Farrow to which the audience warmly responded.

A. J. Newbould

West Riding

Some aspects of microprocessors

A meeting of the West Riding Section was held on Tuesday 3 February 1981, at the Mansion House, Leeds.

Mr D. Hamilton of the Chemical and Allied Products Industrial Training Board gave 24 members and guests a clear insight into microprocessors.

Mr Hamilton opened his discussions by mentioning the dramatic reduction in costs of computers in the last decade. This was due to rapid strides in development and technology.

There were three types of computers mentioned – main frame, minicomputers and microcomputers. Mr Hamilton concentrated his discussion on the latter computer.

The basic functions of a computer was said to be control, input, storage, output and arithmetical.

Mr Hamilton suggested that many of these functions had been greatly influenced by the advent of the microchip. He went on to discuss the manufacture of the silicone chip.

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OCCA meetings

The microcomputer was said to do nothing different from ordinary computers, it is the way in which they do it that makes them exceptionally powerful and gives rise to the vast range of applications to which they can be applied. They are cheap, compact and reliable.

Mr Hamilton went on to introduce the many uses of the microcomputer some of which he demonstrated with the use of a typical system.

D. V. Maltman

Newcastle

Industrial relations

The February meeting of the Newcastle Section (5 February 1981) was held at the usual St. Mary's College, Durham venue, and proved to be one of the most well attended (48 members and guests) and most enjoyable in recent years, with a controversial lecture by two good speakers, an interesting formal discussion before the usual excellent free buffet, followed by further informal discussions which went on until almost 10.00 p.m.

The speakers for the evening were Mr J. McLoughlin (personnel manager, Berger, Newcastle) and Mr P. Hooper (paint tin filler, convener and TUC lay tutor, of Berger, Bristol), who presented a joint lecture on "Industrial relations with particular reference to the paint industry and Berger".

With audience participation, Mr McLoughlin first established that "industrial relations is the formal means whereby changes are negotiated so as to lessen the conflict between company and employee aspirations". He pointed out the perpetual conflict of the corporate aim of survival and expansion by profit, and the employee aim of improved status and security by enjoying as much of the corporate profit as possible.

Mr McLoughlin then outlined the formal negotiating structure within Berger. Approximately a dozen unions were broken up into five main bargaining groups (i.e. staff unions, maintenance unions, transport unions, production unions in the London factories and the production unions of the remaining Berger UK sites). Senior stewards or conveners from each of these groups, and senior management from each of the sites, negotiated major issues, such as pay and conditions, on a national basis. To ensure that Berger was in line with the rest of the industry these negotiating groups were linked through the Paint Makers' Association to its National Joint Industrial Council (NJIC), which made minimum recommendations for wages and conditions.

The grievance and disputes procedure was then outlined, beginning with the individual and his supervisor and rising by set stages and time stages through department manager/shop steward, site manager/convener, group

manager/full-time official, to conciliation and the NJIC Disputes Committee if not settled at any of the intermediate stages.

Mr Hooper then outlined the structure of the TGWU, from its individual members up through the local branch, to the Regional Committee, to the National Committee and the TUC. He outlined the role of the lay members and officials and the full-time officials. Mr Hooper reviewed some of the more recent union history and changes that had taken place within the last ten years, the most significant being that while at one time a full-time official would sit down with senior management and finalise agreements and then dictate them to the union members, now the members dictated their requirements to the official, who had to refer back to them before he could accept anything on their behalf.

During the joint lecture Mr McLoughlin was self-critical of management and management attitudes, while Mr Hooper was self-critical of unions and union attitudes, and thereby on the night the perpetual conflict between management and unions was cleverly avoided.

Some of the interesting snippets from the lecture and discussion were as follows:

"Managements tend to look after money and machines but not men."

"Managements who criticise union decisions made by a handful of 'fanatics' at branch meetings in the back room of a pub on a Friday night should be prepared to allow all union members to attend branch meetings at work in work's time, so that every member is involved in policy decision making." Berger reputedly do this.

"While management reserve the right to manage, to achieve optimum results they should manage by consent."

"A worker director who said 'sorry lads, that's the best pay and conditions that we can have', would lose credibility with his members and be considered 'one of them'."

"Bad communications are not the problem. 'We want more'; 'You cannot have it' is good clear communications, but each side of the conflict can only hear what it wants to hear."

"More is achieved by persuasive discussion than by table thumping."

"Closed shops are helpful to both management and unions in that all individuals can readily make a formal contribution should they wish."

"Closed shops can lead to poor shop stewards, as they need no longer convince their members of the advantages of being in the union."

"If closed shops are undesirable, why do lawyers, solicitors, etc., operate them?"

"A consensus of opinion has shown that 80 per cent of interviewees thought the unions were too strong but that their own union was too weak."

"If secret ballots give the only true feeling of the voters, why does everyone in Parliament know how every MP votes and why do the CBI make their decisions with a show of hands?"

"With gradual build-up to national negotiations and agreements, the limitations of the 'big monster' are becoming more apparent and the future trend could be back to local negotiations and agreements, which were much more specific to particular sites and localities."

It was amusing to note that, in general, the trade unionists present thought the personnel manager, Mr McLoughlin, was the more interesting of the two speakers, while the managers present thought the TUC lay tutor, Mr Hooper, the more interesting.

I. Maugham

Scottish

Water-borne stoving finishes

The following is a report of a lecture on "Water-borne stoving finishes" given on Thursday 12 February 1981, by Mr T. Entwistle of Tioxide International Ltd.

The lecture dealt primarily with the dispersion stability of titanium dioxide pigments in water soluble stoving paints and considered what factors could contribute to obtaining a paint free from pigment flocculation.

The dispersion stability of pigments in conventional solvent based paints has been thoroughly investigated in the past and it is generally accepted that two mechanisms, steric hindrance and electrostatic repulsion, contribute in such systems. The lecturer considered how these mechanisms could be applied to water-soluble paints and how they would be affected by replacing organic solvents by water.

As both resin polymer and TiO_2 pigment will each carry a negative charge at alkaline pH the possibility of resin absorption and hence steric repulsion will be reduced. Although the increased electrical charge on the pigment surface could be expected to increase the degree of electrostatic repulsion, the results obtained showed that this was not the case and that, as with solvent based systems, the major contribution to dispersion stability was by steric repulsion.

The effect of pH on the zeta potential of titanium oxide pigments of different surface treatment (both inorganic and organic) and how these factors affected pigment flocculation was discussed. Flocculation was assessed by film gloss, electron microscopy and determination of flocculation gradient.

The talk concluded by showing that the order of gloss retention and chalk resistance on exterior exposure of different pigments in water soluble paints was similar to that obtained in paints diluted with conventional organic solvents.

Water-borne systems for wood

The following is a report of a lecture on "Water-borne systems for wood" given on Thursday 12 February 1981, by Mr P. Coventry of Hill Son & Wallace.

The first significant use of acrylic emulsion systems on wood was in joinery primers conforming to BS 5082. These have gained acceptance particularly for factory pre-priming of softwood.

Recent advances in emulsion technology has resulted in



a new range of acrylic and styrene/acrylic emulsions which produce tack free films with good dirt and stain resistance, have improved water resistance and good wetting and flow properties. Many of these new polymers have free hydroxyl or carboxyl groups which allows crosslinking with melamine resins (acid catalysed) or poly-functional aziridines respectively.

This has enabled other areas of woodfinishing, such as furniture, to be tackled and considerable success has been achieved in this area. Successful production has been carried out using spray, curtain coating and dipping techniques and drying times have proved acceptable. The resultant finishes have equivalent and often superior properties compared with solvent based acid catalysed and pre-catalysed finishes particularly when crosslinked with aziridines.

Further use of acrylic emulsion on wood is likely to result due to increased interest in water-borne stains and paints for exterior use. The basic weathering properties such as resistance to degradation by UV light, water resistance and maintenance of flexibility have been demonstrated but there is some concern regarding application properties and the build achieved. On site application tests and monitoring of trial sites, including a comparison with alkyd systems of maintenance painting schemes, are now necessary for further progress to be made in this area.

W. L. MacDonald

Trent Valley

Dispersants

Approximately 20 members of the Trent Valley branch of the Midlands Section held their monthly meeting on the night of Thursday 12 February 1981, at the Derby Crest Motel. Dr R. D. Harding of Bevaloid Ltd gave a lecture on the subject of "Dispersants". Dr Harding drew upon his considerable academic background from Imperial College, London and practical experiences gained from both sides of the Atlantic Ocean to outline the principal types of dispersant available for the incorporation of powders into water.

The necessity for dispersants, advantages and disadvantages of the principal types, effect of molecular weight, dosages and pH on their effectiveness, how they are affected by dissolved electrolytes and solvents such as glycols were all neatly outlined and illustrated graphically.

There followed an interesting question period, during which it was discovered that some members had travelled from as far afield as Wolverhampton, Manchester and Bingham to hear Dr Harding's address.

The meeting was concluded with the customary free buffet supper.

J. R. Kitchen

occa meetings

Midlands

Pretreatment for painting

The final technical meeting for this session of the Midlands Section was held on Thursday 19 February 1981, at the County Cricket Ground, Edgbaston, Birmingham.

Members and guests heard Mr M. J. Danks of W. Canning Materials Ltd give a talk entitled "Pretreatment for painting".

Mr Danks opened his talk by saying that the first stage in the pretreatment process was to ensure that the metal surface was free from all traces of oil, grease, rust and mill-scale. This can usually be achieved by one or more of the following:

1. Vapour and abrasive blasting
2. Organic solvents
3. Emulsion and emulsifiable cleaners
4. Alkali cleaners
5. Acid pickles

Of the above processes the alkali cleaners are the most important and most commonly used and consist of alkali

salts such as caustic soda or sodium silicate in combination with sequestering agents and organic surfactants.

The lecturer continued by saying that the phosphate coating consisted of inorganic crystalline compounds reacted with and chemically bonded to the surface or substrate. The pretreatment improves corrosion resistance by forming a physical barrier to prevent water reaching the metal surface. It also forms a good base for painting by giving the surface a rougher texture which will hold more paint. The pretreatment improves adhesion by forming a bond between the metal surface and the paint film.

The various types of phosphate coating that are available are set out in BS 3189:1973. After phosphating the final stage is the drying process. This is necessary in order to remove the water of crystallisation from the phosphate coating and is usually carried out at 150°C for 3 to 5 minutes. In order to speed up the phosphating process various accelerators such as nitrates, nitrites and chlorates are used. These accelerators remove by-products that are formed during the phosphating process thus ensuring that the chemical reactions that are taking place proceed in the required direction.

Mr Danks concluded his talk by comparing the advantages and disadvantages of spray phosphating and immersion phosphating. The latter method must, however, be used for the heavyweight phosphate coatings. Throughout his talk the lecturer showed numerous slides to illustrate the various chemical reactions that take place during phosphating, also the different types of plant that are used.

A very lively question time followed the talk and the meeting was finally brought to a close with a vote of thanks proposed by Mr D. G. Williams and endorsed by the audience.

B. E. Myatt

news

Haeflner takeover

H. Haeflner and Company, a member of Wolstenholme Rink Group, recently took over Proctor Johnson Limited. Proctor Johnson are processors and manufacturers of a range of specialist pigments primarily for use in the construction related industries. Some years ago Proctor Johnson made the decision to concentrate on the export market and today some 80 per cent of their turnover is exported from the UK to a large number of overseas destinations, and despite the high valued pound they currently have a full order book. Proctor Johnson will operate as a subsidiary of H. Haeflner but will retain its management team with Mr Peter Rathbone as managing director.

Reader Enquiry Service No. 31

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

Can coating licensed

International Paint have been licensed by Du Pont to manufacture and sell their water-based can coating material. The product, marketed as Corlar WBE can coating, is used as an inside coating for beverage cans.

Reader Enquiry Service No. 32

More Volstatic labs

Volstatic Coatings Limited, who recently opened an American subsidiary, have now increased their international marketing effort by the installation of new test coating facilities in Europe and Scandinavia.

The company already has powder coating laboratories in Canada, the USA and Italy as well as three in the UK, of which the one at their London headquarters is claimed to be the best in Europe.

The latest are at their Spanish agents, Sum-Ind-Spray, Barcelona, and at the

factory of Swedish oven and pretreatment equipment manufacturers, Tri-Innovations AB - TRIAB.

Reader Enquiry Service No. 33

Prayon SA appoint distributor

Prayon SA have appointed Alfa Chemicals Limited as distributors of certain anti-corrosive and colour pigments for the UK and Ireland. The anti-corrosive pigments which will be stocked in the UK are zinc tetraoxochromate, strontium chromate and barium chromate, the colour pigments are yellow lead chromates.

The anti-corrosive pigments find application in etch wash primers and ferrous and non-ferrous metal primers. Red iron oxide and lead chromate are also available in the initial range that Alfa is handling for Prayon.

Alfa Chemicals is a successor to Burrell Colours Limited.

Reader Enquiry Service No. 34

Coloured flakes

Steeley Minerals Limited have been appointed exclusive distributors in the UK and Ireland, for the Dutch Company Coldec BV's range of coloured flakes.

Coldec flakes are available in a variety of sizes from 1 to 12 mm mean diameter; the finer flakes being used in wall coating systems whereas the larger sizes are used in flooring systems. They can be supplied in 25 standard colours giving virtually unrestricted decorative possibilities from full colour to pastel shades.

Reader Enquiry Service No. 35

ACS computer colour control

Applied Color Systems Inc., has appointed Elcometer Instruments Limited as its exclusive UK representative for the range of ACS digital computer spectrophotometer colour control systems.

Reader Enquiry Service No. 36

On-line inorganic thermodynamic information

MTDATA is a specialist computer system developed by the National Physical Laboratory (NPL) now available as an on-line computer service from Scicon Computer Services.

It comprises a database of thermodynamic information on inorganic substances with associated computer programs for the calculation of applications relating to inorganic chemical reactions.

Reader Enquiry Service No. 37

Simon-Rosedowns appoint agent in Mexico

Desarrollo Industrial-Ingenieros SA has been appointed official representative in Mexico for Simon-Rosedowns Ltd of the UK.

Desarrollo are experienced engineers in the vegetable oilseed processing industry and will specifically promote the sales of the full range of screw press/cooker units, in particular the "G" type pre-press which has already achieved considerable success in the sunflower and rapeseed growing areas of North America.

Reader Enquiry Service No. 38

Powder 81 postponed

In view of the economic recession, the Paintmakers' Association has decided to postpone the conference and exhibition on powder coatings scheduled for 2-4 November 1981.

While remaining convinced of the bright long-term prospects for powder coatings, the association considers that it is in the best interests of all concerned to delay this year's event.

Reader Enquiry Service No. 39

new products

New water based binder for primers for steel protection

ICI Mond Division has announced an air drying water based vinyl acrylic copolymer called Haloflex 202 which has been primarily designed as a binder for use in primer paints for the protection of steel. Haloflex 202 produces tough films of very low permeability to water vapour and affords excellent protection of steel under a wide range of environmental conditions. Tests have shown it to be better at steel protection than any other water borne primer system.

Haloflex primers dry very rapidly and do not exhibit flash rusting. The coatings are hard and flexible and retain these properties on ageing. The primers have good adhesion to steel and multiple coats show excellent intercoat adhesion. They can be applied over a wide range of blast primers and can be overcoated with water based acrylics and solvent coatings such as chlorinated rubber, vinyl and alkyl.

When applied at a dry film thickness of 50 microns over the profile peaks on blast cleaned steel, the primer gives excellent protection in salt spray and water immersion tests demonstrating the good barrier properties afforded by the polymer. These results have been confirmed by long term exterior exposure trials.

Being water based and non-flammable, Haloflex 202 is an ideal product for use in paints to be applied under factory conditions or in other situations where the use of solvents is undesirable.

ICI are offering free samples of coated panels, paint and the Haloflex 202 latex to those interested in testing the formulated primer or trying out their own formulations with the latex.

Further details are contained in the paper entitled "A new approach to the design of latex paints for the protection of steel" and in the centre fold advertisement, both in this issue.

Reader Enquiry Service No. 40

Miniature float switches

The RSF 51, from FR Electronics, is a range of miniature fluid level float switches for vertical mounting. These units incorporate a reed switch in the fixed stem and an operating magnet in the ultra-sonically welded float.

The devices are usually installed and are designed for mounting in the top of a tank for high-level control and the bottom of a tank for low level control.

Reader Enquiry Service No. 41

A new pigment from Hoechst

Hoechst has introduced a new pigment into its Hostaperm H Series, Hostaperm

news

Yellow H6G. It is reported as being a very pure shade and is the greenest of the series. It shows very good flow properties and is of particular interest for producing brilliant green shades in combination with phthalocyanines. Chemically, it belongs to the benzimidazolone group of pigments.

Its high fastness properties make it ideal for automotive finishing and other high quality applications.

Reader Enquiry Service No. 42



The Power 2000 Electronic glue gun

New glue gun

A new hot melt adhesive hand gun, the Power 2000 Electronic, is being introduced by Power Adhesives, specialists in hot melt product assembly and packaging equipment. The gun is suitable for use wherever two surfaces need to be bonded together instantly, including fabric, wood, brick, ceramics, paper, plastics, board, rubber, foam, leather, light-gauge metals, etc.

The new gun has several unique features. Chief amongst these is an electronic control system using a specially designed printed circuit board, the first time, it is claimed, such an advanced system has been incorporated into a tool of this type. The main virtue of the PCB is in providing a stabilised adhesive temperature and heater housing. Previously, glue guns have used a simple thermostat which cuts in and out over a temperature range of some 25°C. The Power 2000 is temperature controlled to within five degrees of the set temperature. In addition, a three-way switch allows the operator to select another unique feature, a pre-set standby temperature intended for use where the gun needs to be at constant readiness, yet is used intermittently.

Reader Enquiry Service No. 43

Twin-shell grinding mill

OBS have introduced the Duplex super-mill, a machine which features two interconnected grinding shells on a common frame.

This new unit will offer a major benefit to manufacturers dispersing magnetic iron oxides, gypsum and dyestuffs. Previously, to achieve satisfactory refinement it was often necessary to process the product twice. The Duplex offers quicker and more convenient processing by automatically transferring the material from the first grinding shell to the second for further dispersion prior to discharging the finished product.

Features of interest to users include a totally enclosed grinding shell which eliminates solvent evaporation and is of obvious benefit when processing toxic materials, a choice of two peripheral disc speeds, and quick shell removal for easy cleaning and routine maintenance. The Duplex is available with shell capacities from 5 to 200 litres.

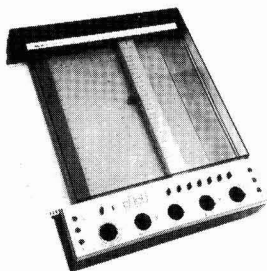
Reader Enquiry Service No. 44

Chart recorders

J. J. Lloyd Instruments Limited have recently announced three new chart recorders amongst which is the battery/mains operated, portable XY/t flat bed recorder. This new instrument, known as the PL4-BX has an A4 format and self-contained NiCd rechargeable batteries.

Also announced were the 1000 series with an A3 format designed for applications requiring high sensitivity, exceptional resolution and linearity, and the CR 650S Mk II a competitively priced model with battery/mains operations local/remote pen lift and eventmarker.

Reader Enquiry Service No. 45



The PL4-BX XY/t flat bed recorder

Re-chargeable sprayer

Pollybin have added a new 6 oz re-chargeable sprayer to their Sure Shot range of chemical spraying equipment. This model, the "C" unit, can be pressurised from any normal compressed air supply, and through its range of nozzles and extension pieces can efficiently and economically jet or spray almost any fluid.

Reader Enquiry Service No. 46

New level gauges

A new range of level gauges specially designed for use with chemicals in both pressurised or open vessels is being marketed by Brooks Instrument.

The Maglink 5400 range which has been accorded PTB approval for use in zone 0 (hazardous areas), offer high accuracy, require no calibration, provide linear (and remote) transmission of readings and are particularly suitable for use in severe conditions of corrosion, temperature and pressure.

All parts of the level indicator in contact with the liquid are made from stainless steel, plastic or other special materials (PVC, Teflon, polypropylene etc., according to use).

Capable of handling level changes of up to 11 meters, the Maglink gauges operate using a flux linkage between a float magnet and a follower magnet contained in a guide tube. Level variations are transmitted by means of a wire to the linear indicating mechanism in the head. The magnetic coupling is capable of withstanding the severest fluctuations in tank liquid level and the Brooks design also eliminates backlash in the indicator mechanism by way of a gearbox with spring motor which compensates for the weight of the magnet.

Reader Enquiry Service No. 47

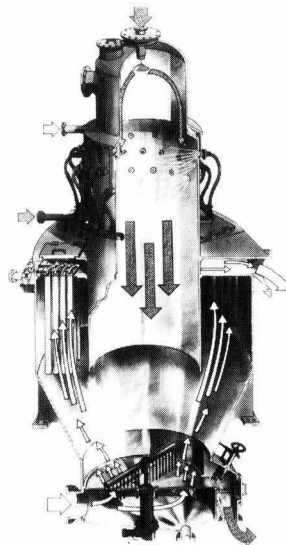
Benzo(a) pyrene determination

Baird & Tatlock have developed a specific TLC method to determine benzo(a) pyrene quantitatively.

Benzo(a) pyrene, together with other PAH, is present either as a normal component in mineral oil or is formed through processes involving the heating or incomplete combustion of organic substances. It is generally regarded as a carcinogen and therefore its determination is very important to quality control for obvious health reasons.

Baird & Tatlock also announce a new accessory, a special delay titrate timer, for the AF3 Karl Fischer Titrator, used for the automatic determination of moisture content. This timer will delay the automatic titration by a pre-set time (up to 15 minutes) while the moisture in solid samples is extracted into the solvent.

Reader Enquiry Service No. 48



The Spraymixer; powder fed in at the top mixes with liquid sprayed in just below

New spray mixing process

By adopting a novel approach to the mixing of powders with liquids, the Claudius Peters group has developed a practical and economically attractive process that is applicable to many industrial requirements.

Known as Spraymixer, the new processing system is unusual in that the mixture is produced in air by spraying solid and liquid components into a mixing chamber. Droplets of the liquid become coated with the powdered solid. The result is a homogeneous, dry and free-flowing product which is easy to handle.

When compared to the conventional method of mixing all components as liquids and subsequently drying the mixture, Spraymixer saves the heat that would be required for the drying process. Again, its energy consumption is low in relation to a mechanical process with mixing blades.

Owing to the lack of mechanical shear, the new process is gentle and causes no product degradation. Also contributing to the low running costs is the fact that the process is continuous and fully automatic, so only the minimum of operating personnel is required.

All process variables can be controlled precisely to obtain exceptionally accurate mixes. Spraymixer is a versatile processing system; not only has this design of plant proved to be successful in mixing applications, but it also shows considerable promise for re-wet instantizing and chemical reactions.

Reader Enquiry Service No. 49

Packaging replacement for drums

Chempak is a new concept in packaging for the chemical industry, designed principally for powder chemicals.

It replaces the traditional metal and polythene drums and consists of an extra strong rectangular, corrugated outer carton and a specially designed and treated inner container. Being rectangular it packs neatly onto standard pallets with no wasted spaces in between. Up to 27 cartons may safely be carried on one pallet (1.00 x 1.20 m), and pallets may be stacked 3 high.

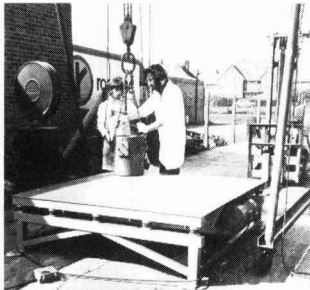
The inner and outer cartons are stacked flat when empty, saving on transport costs and requiring much less storage space when not in use.

The cartons are designed for automatic opening, filling and closing, but are also suitable for manual or semi-automatic processes.

The specially treated and re-inforced PVC liner is safe for toxic powders. It is also impervious to harmful light rays, an important feature for pigment manufacturers.

Chempak is currently available in 45 litres capacity.

Reader Enquiry Service No. 50



Vibrating table

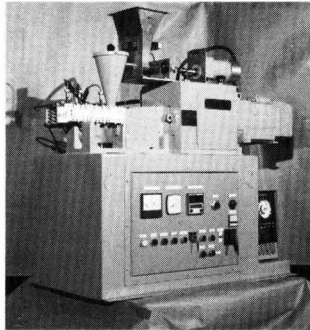
Vibrating table

A new 4m² vibrating table introduced by Triton Engineering Company Limited complements an existing range of equipment designed to improve and speed compaction processes.

The new 3 tonne gross capacity machine is a stressed-design unit comprising a single piece mild steel table top 2 m x 2 m, with heavy section steel under-stiffening and ten 70 mm diameter anti-vibration rubber mountings spaced at equal distances on the under side.

Power source is from two contra-rotating L/125/2 electric vibrator motors strategically mounted beneath the table to transmit direct vertical and linear forces to the table top, providing uniform amplitude over the entire surface area avoiding the possibility of dead spots.

Reader Enquiry Service No. 51



The MPC/V 30 compounder

Laboratory compounder

A new size of twin screw compounder, the MPC/V 30, has been added by Baker Perkins Chemical Machinery Limited to the company's range. It has 30 mm twin bores and is intended for laboratory and pilot-scale use.

The new machine can be tailored to suit particular applications, the whole spectrum of technical thermoplastics compounding, devolatilising and pelletising duties, or compounding thermosts, powder coatings, toners and universal masterbatches.

The flexibility of the design means that the machine can be supplied for a particular use: customer service, colour matching, formulation development, production control, basic research, sample production or combinations of these.

All key features of the production range of machines have been retained. These include the quick opening barrel, fully segmented screw construction and co-rotating, fully self-wiping and inter-meshing screw design. A useful feature is that the machine can be dead-stopped when full of material and opened up so that the compounding process can be observed. Another point of major importance is that accurate scale-up to larger machines, it is claimed, can be guaranteed.

Reader Enquiry Service No. 52

The Pipo 2 spray gun

The latest addition to Kremlin Spray Painting Equipment's range of spray guns is the Pipo 2. A distinguishing feature is the wide range of uses that the new spray gun can be put to, as it is supplied with two different types of air caps and the projector can be adjusted to give a variety of fan shapes.

The Pipo 2 can be used, for example, in the building industry, for general industrial finishing work including delicate retouching work, and for car bodywork.

Reader Enquiry Service No. 53

news

New colourants for inks from BASF

BASF have introduced two new pigment preparations for printing inks and four new low dusting dyes. The pigment preparations, Enceprint Violet 6071 and Basoflex Red 3750 are intended for alcohol based and aqueous printing inks respectively and the pigment contents are approximately 40 per cent and 45 per cent respectively.

BASF have also introduced four new dyes, Rhodamine F5G-NS, Rhodamine 6GD-NS Type 8016, Rhodamine FB-NS and Victoria Blue FB-NS, these are primarily recommended for the preparation of flexographic inks and are all low-dusting.

Reader Enquiry Service No. 54

Packaged chillers

Carter Cooling Towers have introduced a comprehensive range of packaged water chillers to satisfy the demands of small to medium industrial process cooling duties.

Ten water cooled and five air cooled models are available as standard units from 12-112 kW capacity.

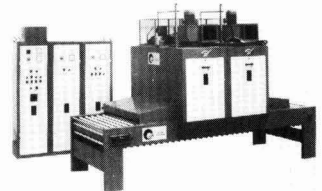
Reader Enquiry Service No. 55

UV curing tunnels

The new Comil STL range of UV curing tunnels, designed for the rapid and effective curing of filling materials, grounding and photo-sensitive lacquers, is now available in the UK from the Machinery Division of Macpherson Components Ltd.

These tunnels incorporate high power lamps which generate a minimum of heat yet ensure fast curing. The lamps are fitted to the tunnel roof in specially shaped supports, with reflectors which give optimum and uniform distribution of the rays over the entire panel surface.

Reader Enquiry Service No. 56



New UV curing tunnel

OCCA news

UV gloss control

Fusion Systems Corporation has recently developed and patented a simple process providing variable control of gloss for clear UV coatings.

Gloss control is gained through on-line adjustment of the curing system rather than by changing coating formulation, application conditions or curing atmosphere. Initial exposure to long wave length UV light initiates curing of the lower layers of the coating only. This causes flattening agents to migrate to the surface of the coating. Subsequent exposure to the short wave length UV region of the standard mercury lamp completes the cure.

All coatings are air curable, thus expensive inert gas blanketing is not required. The user is free to choose coatings from many suppliers. Users gain on-line adjustment capability with minimal use of flattening materials, and complete control of coating finish from matt to satin to glossy.

Reader Enquiry Service No. 57

Two new portable detectors

Built to the same basic design, two new portable gas and combustible hydrocarbon detectors are available from Rotheroe & Mitchell Limited.

Called the CGS-8M and CGS-10M units, these instruments are equipped with meter display for direct reading of gas concentrations. A two position switch provides capability for detection of either toxic gases or combustible hydrocarbons, with the CGS-10M additionally providing constant monitoring for oxygen deficiency. A continuous alarm is triggered when the level of oxygen drops below a pre-set level.

Designed to provide rugged and dependable operation in field conditions, the units sample gas using a convection/diffusion process, negating the use of pumps, tubes or squeeze bulbs.

Reader Enquiry Service No. 58

Pressure sensitive coatings

Mansanto has announced the publication of a new technical brochure for Gelva multipolymer resin solutions, including information on three new products.

Gelva multipolymer solutions are used to make pressure sensitive coatings and laminating adhesives for surfaces that are

difficult to bond. They are also used in formulating surface coatings and for speciality paper coatings. Gelva products are supplied as water white or slightly yellow solutions and are based on vinyl acetate, acrylic and other co-monomers.

New Gelva products include: RA 2045, when cured with an isocyanate it is an acrylic solution resin that dries to form a permanent tacky pressure sensitive film, with controlled bond stress and easy removability after bond ageing. Particularly suited for protective films and medical tapes. RA 2258, is designed to provide outstanding cohesive strength combined with well balanced tack and peel properties. It has an excellent resistance to high temperatures and to plasticizer migration. Particularly suited for double-sided tape.

Reader Enquiry Service No. 59



Dry film thickness gauge

New dry film thickness gauge

Elektro-Physik recently introduced a dry film thickness gauge called the Certotest. It measures, non-destructively, the thickness of non-magnetic coatings applied to ferro-magnetic substrates, e.g. paint, plastic, zinc, chromium and most electroplated deposits (except electroplated nickel) on steel.

The Certotest is suitable for all types of inspection and quality control work, both in the factory and the field. The logarithmic scale in the meter makes accurate readings possible throughout the scale length.

This economy priced instrument is simple to use, is small and lightweight. The single scale is calibrated both in thousandths of an inch (mils) and microns, and ranges from zero to 500 microns (20 mils).

literature

CEFIC Tremcards Group Texts

Following the successful publication last year of CEFIC Tremcards, the individual transport emergency data cards carried in

chemical road vehicles, a complementary reference volume covering groups of chemicals has been published entitled "CEFIC Tremcards Group Texts".

While the 1980 reference edition contains more than 500 cards referring to individual substances representing a high proportion of chemicals regularly carried in bulk quantities, the new 64 page Group Texts make provision for the many other substances conveyed, often less regularly and in smaller quantities, and which can be categorised into groups. These will enable a manufacturer to select an appropriate text for those products not covered by individual cards.

Reader Enquiry Service No. 60

American health study

The American *Journal of Occupational Medicine (JOM)* has just released the complete results of a three-year, half-million-dollar study which concludes that paint and coatings manufacturing workers face "no major hazard to health" from the wide variety of chemical substances they are exposed to in their jobs.

The study was based on the job histories and personnel records of over 15,000 workers in 32 production plants. It was commissioned in 1978 from the research firm SRI in the first instance by the National Paint & Coatings Association of the USA (NPCA). It was subsequently supported in substantial financial terms both by the European Committee of Paint and Printing Ink Manufacturers' Association (CEPE) and by the Canadian Paint Manufacturers' Association (CPMA).

The study showed that the white male paint industry workers used in the study actually had a lower mortality rate than the general white male population (white males were used in the study because women and minorities made up only a small part of the US industry's work force during the study period). However, because industry workers are generally healthier than the population as a whole, *JOM* states that the overall result of the study compares favourably with "about what might be expected" of a normal industry study group.

Reader Enquiry Service No. 61

15th anniversary catalogue

International Light Inc. has announced its new 15th anniversary catalogue. The catalogue contains spectral curves and dynamic performance graphs for most of its light monitoring systems as well as new products and over 100 product pictures. Systems featured, with application comments, include spectroradiometers, UV measurement systems, photoresist, UV curing, UV hazard monitors, photometers and laser power meters. Also amply displayed is a wide range of filters, accessories and fibre optic cables.

Reader Enquiry Service No. 62

Electrophoretic painting

"Advances in electrophoretic painting 1978-80. Bibliographies in paint technology No. 36" is published by R. H. Chandler Ltd and provides reviews on the literature and patents, and references on the following.

The growth in cathodic electropainting has been substantial in the three years, 1978-80, but there has also been a detectable swing away from electropainting as a whole for the first time as other environmentally satisfactory coatings have become available. All the large automotive firms now run cathodic tanks and though better corrosion resistance is obtained, this is now thought to be due to the better quality of the resin binder rather than the intrinsic nature of the process. It has been proved that metal dissolution does take place during deposition, as with anodic electropainting. One key feature of the recent literature has been the investigation into the phosphating stage which is clearly seen as the cause of scab corrosion. Several research reports appear amongst the 196 literature references and 168 patents reviewed.

Reader Enquiry Service No. 63

Glossary of colour terms

The Federation of Societies for Coatings Technology has announced publication of a Glossary of Colour Terms.

The 96 page booklet is a compilation of over 500 colour related terms and their definitions which have been extracted from the Federation's Paint/Coatings Dictionary. Included are colour difference equations, as well as terms for such topics as optical phenomena, gloss, hiding, and colour instrumentation.

A thesaurus and bibliography are also included.

Reader Enquiry Service No. 64

Micro-algae a source of industrial chemicals and drugs

Micro-algae have been little exploited up till now, apart from a few instances in the area of health food or diet supplements. However it is believed that micro-algae have a large potential as primary sources of industrial foods, fats and oils, proteins, gums, colouring agents, industrial chemicals, drugs and plant growth promoters, and several research developments are presently under way in different countries aiming at identifying the most promising applications of micro-algae growth systems.

The Battelle-Northwest laboratory has been particularly active over the last few years in studying a total systems approach to micro-algae culture, with a view to minimising culture and harvesting costs. As a result of this internally funded programme, Battelle is now proposing a multi-client study which will collate and evaluate data on: the history of micro-algae development world wide, current industrial activities and potential future developments.

Reader Enquiry Service No. 65

PRA publications

The PRA has published its first quarterly bulletin on "Hazards, pollution and legislation in the coatings field" which is designed to keep industry abreast of legislation and toxicological information.

Number 11 in the series Notes to industry "Lead in paint" has also been published and is available free of charge from the Paint Research Association.

Reader Enquiry Service No. 66

Corrosion and protection of metals

The Institution of Corrosion Science and Technology has published "A bibliography of books on corrosion and protection

news

of metals". This revised publication contains 450 individual references subdivided into five sections on aspects of corrosion and five sections on aspects of protection, it is supplemented by an authors index for easy reference. Price: members £3.00, non-members £5.00.

Reader Enquiry Service No. 67

Chemical plant contractors

Chemical Data Services has published the third edition of its directory "Chemical Plant Contractor Profiles".

Thoroughly revised and updated, the main body of this reference book gives details of 108 of the world's major contracting companies.

Reader Enquiry Service No. 68

meetings, etc.

Surface and colloid science

The Israel Chemical Society is holding the Fourth International Conference on Surface and Colloid Science in Jerusalem on 5-10 July 1981. Further information is available from: The Secretariat, Fourth International Conference on Surface and Colloid Science, c/o Conventions (Kopel Tours) Ltd, PO Box 3054, Tel Aviv 61030, Israel. Tel: (03) 22217/8 Telex: 341132.

For information on membership of OCCA, enquiries should be sent to the Association's offices, see front cover for address.

Jack Smethurst

At the time of going to press, it was learnt with regret, of the death of Jack Smethurst. An obituary notice will appear in a forthcoming issue.

Professional Grade

At a meeting of the Professional Grade Committee held on 25 February 1981, the following Ordinary Members were admitted to the Professional Grade. The Section or Branch to which members are attached is shown in brackets:

Fellows (FTSC)

Courtman, Frank (*Manchester*)
Furuhjelm, Viktor Henrik (*General Overseas - Finland*)
Sharpe, Eric Edward Victor (*London*)

Associates (ATSC)

Mouallem, Mouhammad (*General Overseas - Syria*)
Potter, Francis (*General Overseas - Zimbabwe Branch*)

At a further meeting of the Professional Grade Committee held on 8 April the following Ordinary Members were admitted to the Professional Grade:

Associates

Ashton, Ronald (*Manchester*)
Lawrence, Simon Gayner (*Scottish*)
Hutchinson, Martin Alfred (*West Riding*)
Webb, John Peter (*London*)
Bell, Eric James (*London*)

OCCA news

Licentiate

Hayles, John William (*London*)

Jordan Award

Members of the Association will be pleased to learn that the Jordan Award for 1979-80 has been conferred upon Mr M. W. Leonard LTSC, for his paper published in the December 1979 issue of the *Journal* which was based upon his dissertation for Licentiate status.

OCCA news

Ontario Section

Colour panel/Ladies' Night

At the annual Ladies' Night in December the Ontario Section was treated to a presentation by Mrs Eleanor Brown, a professional designer and colour expert who has made a career of decor and colour consultation.

The formal topic was "The psychology of colour, design and decor". Mrs Brown quickly set the informal tone of the meeting by relating anecdotes from her experience and giving a preview of upcoming trends in interior and exterior colours. The emphasis of the talk was on the effect that various hues and saturations have on people, and thus how they are used in shops to influence buyers and how they can be used in the home or workplace to establish any desired mood. Mrs Brown also demonstrated that colour preferences are influenced by eye colour and gender. When volunteers representing blue-eyed and brown-eyed men and women selected their most liked and disliked colours from a gamut of paint cards, she predicted and explained their choices with considerable accuracy and much humour. The volunteers were Jack Marshall, Ken Abbott, Iris Purnell and Arunika Patel. A lively discussion followed.

J.A.

News of Members

Mr. H. T. (Ted) Chellingsworth, deputy managing director of Eagle Industrial Paints Limited, of Bridgnorth, and a member of the Midland Section of OCCA, joined the delegation to the Spanish Exhibition at Zaragoza in April, in support of Lord Ferrers, Minister of State and Ministry of Agriculture.

Eagle Industrial Paints Limited are specialist manufacturers of paints for the agricultural machinery industry and the visit is in line with the company's policy of keeping abreast of worldwide developments in this field, a policy which has taken their managing director, Mr F. Pursglove, and technical manager, Dr G. C. Simmons (also a member of the Midland Section) on recent trips to the United States, France and the Middle East.

Eagle Industrial Paints Limited were looking into the possibility of establishing a manufacturing unit in Spain, in view of that country's impending entry into the EEC.



Shown at the Ontario Section colour panel/Ladies' Night (from left to right), Jack Marshall, Ken Abbott, Iris Purnell and Arunika Patel

Scottish Section

Annual Dinner Dance

The Annual Dinner Dance of the Scottish Section was held in the Albany Hotel on 9 January 1981. As always, the event proved to be the highlight of the Section's social calendar and a most enjoyable evening was had by all.

On this occasion those present had the unique distinction of having a sassenach propose the toast to the haggis, as the Section Chairman this year is Mr Tom Humphrey. He did not, however, show the slightest signs of fear of the large gathering of Scottish clansmen around him and performed his task with equanimity.

On a slightly more serious note, it must be said the whole evening passed with

everything going off very smoothly, and this of course must be attributed to the hard work put in prior to the event by Mr J. Toovey, the Vice-Chairman.

W.L.M.



Shown at the Scottish Section's Annual Dinner Dance are (from left to right) Dr and Mrs F. M. Smith, and Mr and Mrs T. L. M. Humphrey



Shown at the Scottish Section's Annual Dinner Dance are the following (from left to right) Glasgow police piper, Mrs L. Brooke, Mr L. Brooke (Bristol Section Chairman), Dr F. M. Smith (President), Mrs F. M. Smith, Mr J. Clark (Newcastle Section Chairman), Mrs A. McKendrick, Mr A. McKendrick (Eastern Branch Chairman), Mrs J. Clark, Dr T. Banfield (London Section Chairman), Mrs G. Bentley, Mr G. Bentley (West Riding Chairman), Mrs T. L. M. Humphrey and Mr T. L. M. Humphrey (Scottish Section Chairman)



OCCA CONFERENCE 1981

The Beaufort Hotel,
Bath, England
17-20 June 1981

Alternative technologies in coatings

As already announced in the *Journal*, the next Biennial Conference of the Association will take place at Bath from Wednesday 17 to Saturday 20 June 1981. The headquarters will be the Beaufort Hotel, with overflow accommodation at the Francis Hotel and the Royal York Hotel. The title for the Conference will be "Alternative technologies in coatings". Summaries of most of the papers and biographies of the lecturers are given below. The programme for the technical sessions is as follows:

Session I

Thursday 18 June 9.15 a.m.–12 noon

Chairman: Mr C. N. Finlay, ATSC (Hon. Research & Development Officer)

Alternative technologies in coatings – The challenge and the response (Keynote address) *By Dr L. Valentine*
(Director of Research & Development, Berger Jenison Nicholson Ltd)

Pigmented UV dual cure coatings (Paper presented on behalf of FATIPEC) *By Mr A. Noomen (Sikkens BV, Netherlands)*

Developments in aqueous powder systems *By Mr A. G. North (Managing Director, Cray Valley Products Ltd)*

Modern practices in formulating powder coatings *By Mr S. T. Harris (Consultant)*

Session II

2.00 p.m.–4.30 p.m.

Chairman: Dr G. de W. Anderson (Managing Director, Paint RA)

Caprolactone in surface coatings *By Dr B. E. Bailey and Mr J. Lister (Laporte Industries Ltd)*

Obtaining opacity with organic pigments in paints *By Dr H. Schäfer (Hoechst AG)*

Alternative means of controlling paint viscosity/temperature phenomena *By Dr N. Reeves (NL Chemicals Europe Inc., Belgium)*

Precision spectral ultraviolet measurements and accelerated weathering (Paper presented on behalf of FSCT)

By Dr M. L. Ellinger and Mr G. A. Zerlaut (DSET Laboratories, USA)

Session III "Alternatives to coatings"

Friday 19 June 9.15 a.m.–12 noon

Chairman: Mr F. D. Timmins (Mebon Paints Ltd)

How not to paint bridges *By Dr R. R. Bishop and Mr M. A. Winnett (Transport and Road Research Laboratory)*

Short presentations—To be followed by discussion period

(i) Don't paint—use wood stains

By Dr F. W. Brooks (Hickson Timber Products)

(ii) Don't paint—galvanize

By Dr J. Wilcock (The Galvanizers Association)

(iii) Don't paint—use plastics

By Dr D. Gardiner (Building Research Establishment)

(iv) If you must paint—coil coat

By Mr N. S. Makins (European Coil Coating Association)

Session IV

2.15 p.m.–4.15 p.m.

Chairman: Mr A. C. Jolly (Synthetic Resins Ltd)

Plastic pigments: A novel approach to microvoid hiding. Part IV: Effect of composition on latex paint performance

By Dr A. Ramig (Gliden Coatings and Resins, USA)

Flow in coatings and orientation in metallics (Paper presented on behalf of SLF)

By Mr P. Fink-Jensen (Consultant)

Prospects for automation in the paint industry

By Mr M. Camina (Paint RA)

Conference preprints

Preprints of the papers are now being prepared and it is expected that these together with details of accommodation, badges, programmes etc, will be sent to those who have registered early in June 1981. It is a feature of the Association's Conferences that preprints are sent well in advance of the function, in order to enable delegates to read them before the Conference, at which the lecturers illustrate their topics, but do not read the papers in their entirety. This allows for a much longer discussion period than at many other conferences, and has proved of immense value to those attending previous Association Conferences.

Registrations

The closing date for registrations for the Conference was given as 1 April 1981, primarily for the benefit of the hotel managements who would be in a position to let any accommodation not taken by that date. Registrations have been received from delegates in the United Kingdom and several overseas countries. However, applications can still be accepted and delegates are urged to write in to the address shown on the contents page or phone 01-908 1086.

Council has fixed the registration fees for the Bath Conference at £80.00 (plus Value Added Tax at the standard rate)

for Members, £105.00 (plus VAT) for non-members, and £25.00 (plus VAT) for wives. A daily registration fee for Members of the Association of £45.00 (plus VAT) and of £25.00 (plus VAT) for Registered Students of the Association has been set.

Non-members wishing to avail themselves of the preferential Conference fee for Members should request application forms from the Association's offices and these should accompany registration forms.

A list of papers to be presented together with their summaries and biographies of the lecturers was published in the

OCCA news

January 1981 issue of the *Journal* and copies of the Conference brochure and registration form were enclosed in copies sent to Members attached to the General Overseas Section and non-member subscribers to the *Journal*. The brochures have been sent to all Members of the UK and Irish Sections with Section circulars. Any non-member who has not yet received a copy of the brochure and wishes to register for this important Conference should apply for the necessary form to the Association's offices.

Social events

Bath, with its eighteenth century and Ancient Roman history, together with the unrivalled surroundings of some of the most picturesque countryside in England, is expected to prove to be one of the most popular venues the Association has arranged for one of its Conferences. The varied social programme will afford Conference goers an opportunity to visit places of historical interest including the Roman Baths, the Pump Room, the Abbey and Royal Crescent in addition to the coach tours across some beautiful countryside to Chepstow Castle, Tintern Abbey, Wells Cathedral and Glastonbury; a tour has also been arranged to Longleat House, seat of the Marquess of Bath, where tea will be taken and which is set in a beautiful park created by Capability Brown.

As an innovation, on the Wednesday afternoon a Works Visit has been arranged to Torrance & Sons Ltd, Bitton, where delegates will have an opportunity



Pulteney Bridge, Bath

of inspecting the manufacturing equipment for the surface coatings industries produced by this long established West Country firm.

Following the success of the river trip during the last Conference at Stratford, delegates will this time have an opportunity to travel on the Kennet and Avon Canal on board the *Jane Austen*, during which coffee and biscuits will be served.

In addition to theatre parties on the Wednesday and Thursday evenings, arrangements have been made for delegates to tour the John Harvey Wine

Museum on Thursday evening where they will be given a lecture, shown a film and have an opportunity of tasting the various types of sherry – transport has been arranged for this function.

For those wishing to play in the golf tournament (for the OCCA Conference Trophy donated by Mr S. Sharp) facilities will be available at the Lansdown Golf Club.

Further information may be obtained from the Director & Secretary at the address on the Contents page of this issue (Tel: 01-908 1086; Telex 922670 OCCA G).

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

BERRISFORD, PETER GEORGE, 14 Milford Close, Westergate Road, Upper Abbey Wood, London SE2 0DT (*London*)

CARROLL, BRIAN, 4 Rimsdale Walk, Ladybridge, Bolton, Lancashire (*Manchester*)

CONLEY, STEPHEN, BA, 58 Hall Moss Lane, Bramhall, Cheshire SK7 1RD (*Manchester*)

FOSTER, HARRY, BSc, 27 Lynmouth Drive, Sully, South Glamorgan CF6 2TP (*Bristol*)

JACK, WILLIAM WATSON, 71 Curloss Grove, Fairfield, Stockton on Tees, Cleveland TS19 7SQ (*Newcastle*)

new members

MORRELL, CHRISTINE ANN, BSc, 7 Ormskirk Rise, Spondon, Derby DE2 7NN (*Trent Valley Section*)

Registered Students

DEARDEN, LYNDA MARY, Industrial Research Laboratory, Crown Decorative Products, Hollins Road, Darwen, Lancs (*Manchester*)

OWEN, STEPHEN MICHAEL, 57 Church Green, Handsworth Wood, Birmingham B20 2HP (*Midlands*)

CLASSIFIED ADVERTISEMENTS

Classified Advertisements are charged at the rate of £5.00 per single column cm. Advertisements for Appointments Wanted are charged at £1.50 per line. A box number is charged at £1.00. They should be sent to D. N. Buddles, Assistant Editor, Oil & Colour Chemists' Association, Priory House, 967 Harrow Road, Wembley, Middlesex HA0 2SF. JOCCA is published EVERY month and Classified Advertisements can be accepted up to at least the 12th, and in exceptional circumstances the 20th of the month preceding publication. Advertisers who wish to arrange for an extension of the copy deadline should contact the Assistant Editor, D. N. Buddles at the address given above (telephone 01-908 1086, telex 922670 OCCA G).

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Hons. degree paint chemistry, LRIC, Professional Grade OCCA, 16 years experience surface coatings and pigment dispersions; senior R & D, quality control, technical service, works liaison, technical management. N. England preferred.
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Qualified paint chemist (graduate, 29) with substantial experience in quality control, formulations, production, laboratory set-up, product development, colour match, technical service, etc. seeks a challenging position (UK), most locations considered, SE England preferred. NB also experience of plastics, electroplating and teaching.
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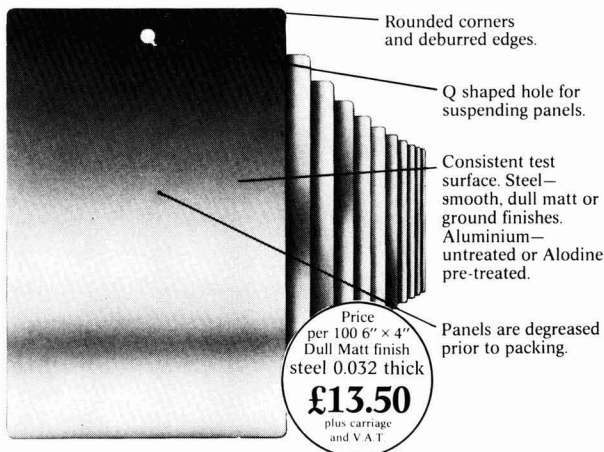
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Q~Panels have arrived

The Q-Panel Company of Cleveland, U.S.A., probably the worlds largest manufacturer of steel and aluminium panels for coating evaluation have now opened a European Branch Office. The office is a marketing, distribution and technical advisory centre for the well established range of Q-Panel products.

Q-Panels—coating test panels are available in a full range of sizes and surface finishes at a remarkably low price.

Q.U.V.—Accelerated Weathering Tester simulates the damaging effects of sunlight and moisture. Advantages of the design include faster tests, closer control, and reduced operating costs.



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NOVAMET's water grade stainless-steel flake contains a special lubricant. A lubricant that will not saponify with the vehicles to reduce lustre in your finished coating.

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Get to know more about NOVAMET's new bright leafing and water grade stainless-steel flake pigments.

Typical Characteristics of NOVAMET Stainless Flake

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Average Flake Thickness	~1.0 micron
Approximate Bulk Value	0.067 L/kg
Approximate Specific Gravity	3.68
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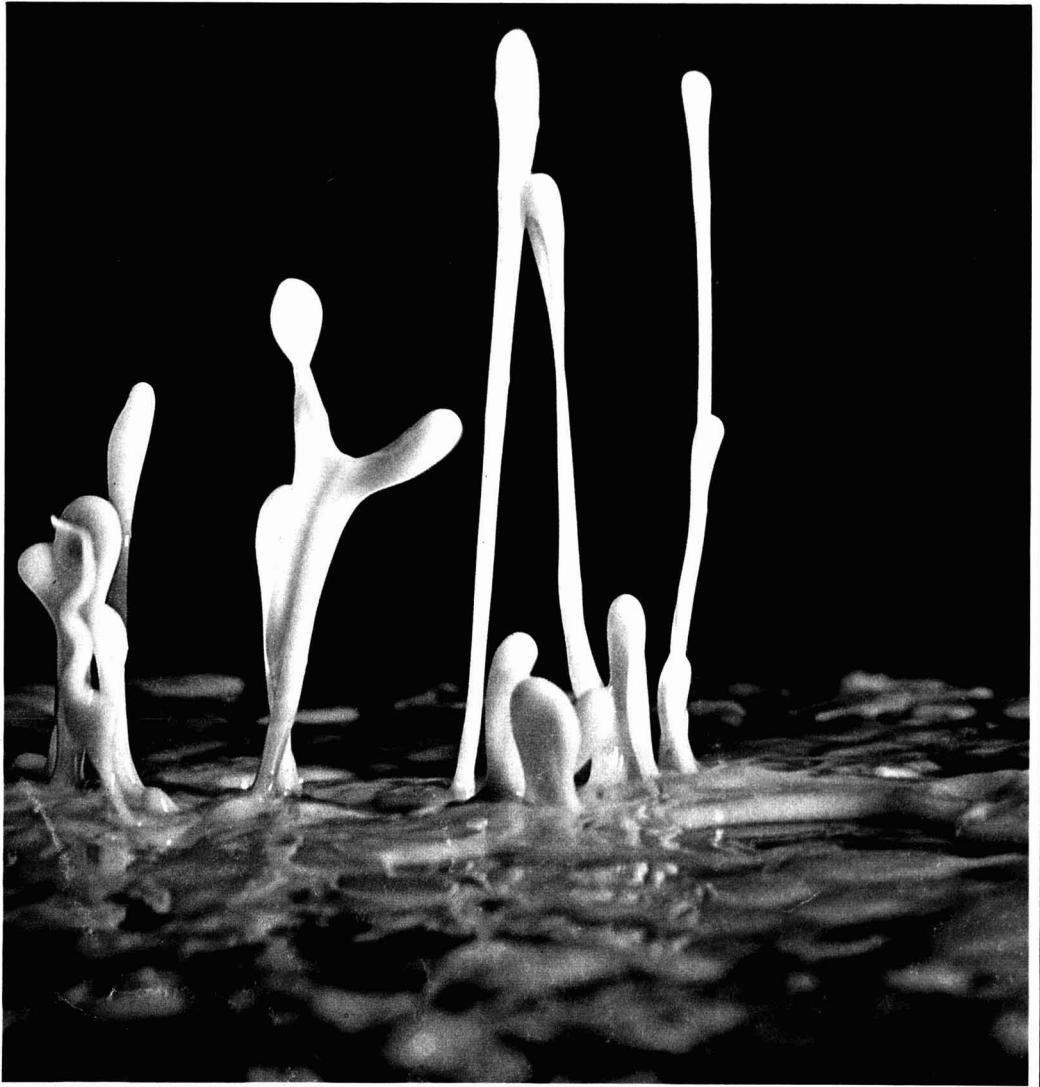
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