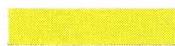




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*J. Pelgrims*

The design and construction of ultraviolet lamp systems for the curing of coatings and inks

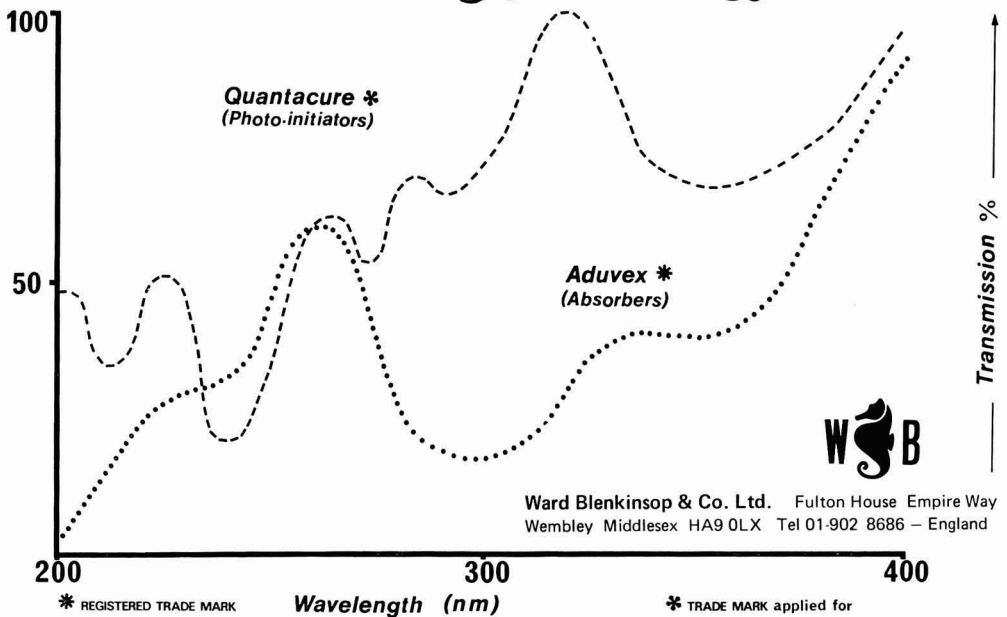
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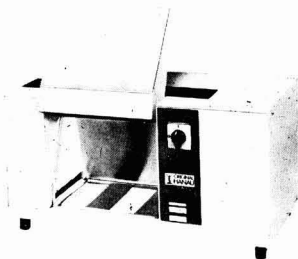
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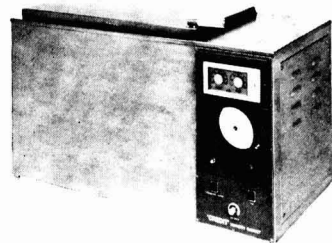
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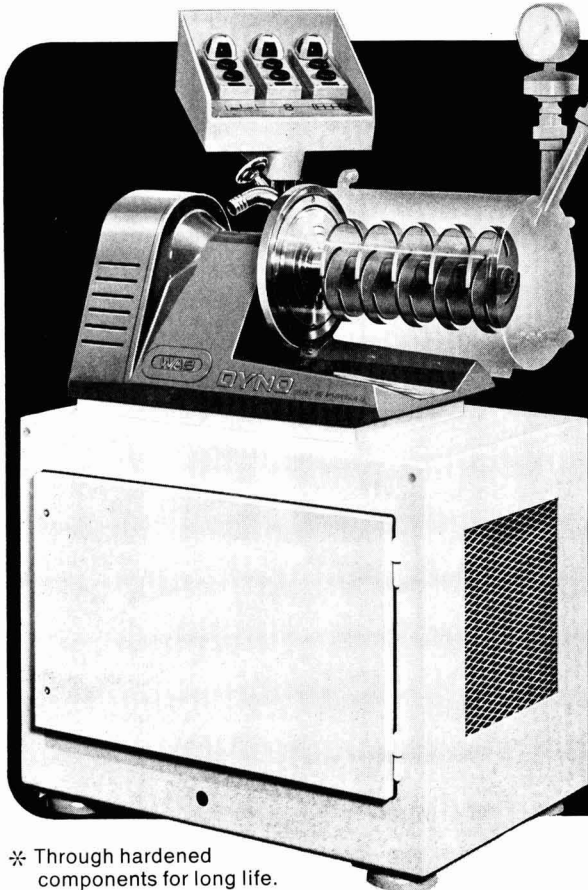
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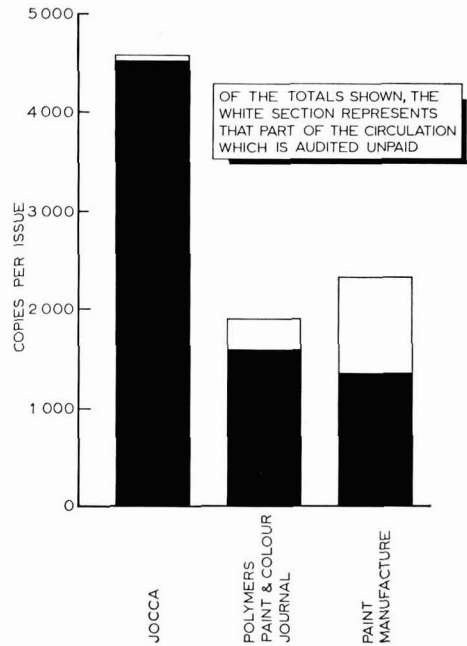
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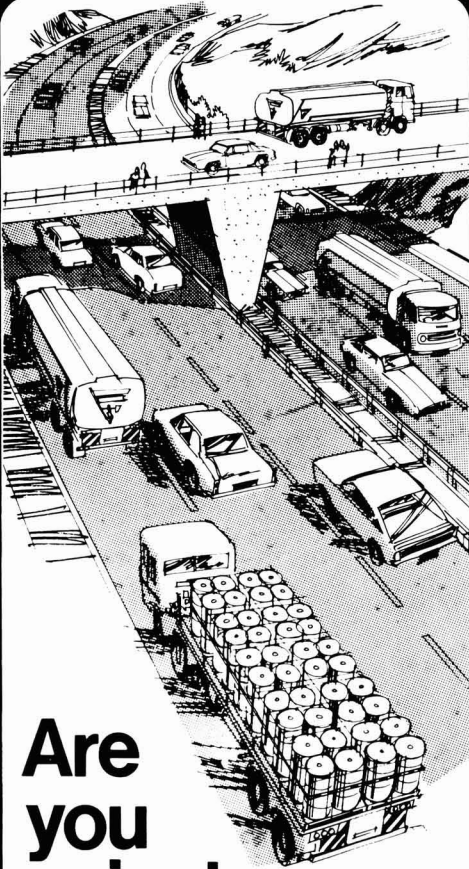


Celebrations

Details of the functions arranged for 11 and 12 May are given on page 137 of this issue. Application forms for both functions can be obtained from:

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# Exciplex interactions in photoinitiation of polymerisation by fluorenone-amine systems\*

By A. Ledwith, J. A. Bosley and M. D. Purbrick

Department of Inorganic, Physical and Industrial Chemistry, University of Liverpool, Liverpool L69 3BX

## Summary

Fluorenone-amine mixtures find commercial application in photocure processes prompting detailed studies of the kinetics of polymerisation and molecular weights of polymeric products for methyl methacrylate homopolymerisation in benzene at 30°C, photoinitiated by fluorenone and several derivatives. Triethylamine, *N,N*-dimethylethanolamine, and indole-3-ylacetic acid were used and gave widely disparate results, although in every case, increasing the amine concentration produced a beneficial effect on rate only up to a certain point, after which excited state deactivation-processes become dominant. Initiating free radicals are suggested to arise by chemical processes subsequent to initial formation of exciplexes between photoexcited ketone and ground state amine. Exciplex quenching by methyl methacrylate is important in many cases and, taken with the more general effects of solvent (including monomer and amine components) on the competition between reactions leading to the formation of free radicals and those regenerating amine and ketone components, serves to illustrate the extreme difficulty of generalising on structure-activity relationships for

ketone amine photoinitiator systems.

Of particular interest is the apparent reduction in transfer or termination processes on replacing fluorenone by 2-methylfluorenone. The latter serves as a model for the chromophore in copolymers of 2-vinylfluorenone and methyl methacrylate, has an absorption spectrum slightly red shifted from, and is much more strongly fluorescing than, fluorenone.

Photoinitiation of methyl methacrylate polymerisation utilising copolymers with pendent fluorenone units has been studied to help clarify the very complex effects of amines on initiation and termination processes. It is concluded that initiation by the fluorenone derived (semipinacol) radical is not important and hence graft copolymer formation (and presumably increased rates of photocure) for given systems is likely to be more effective when using polymers with pendent amine units rather than pendent ketone rings.

## Keywords

*Processes and methods primarily associated with drying or curing of coatings*

photochemistry  
ultraviolet curing  
photoinitiation  
reaction mechanism  
photoactivation  
ketone initiation  
exciplex formation  
ketone-amine initiation

*Raw materials*

binders (resins, etc)  
acrylic resin

*Miscellaneous terms*

reaction mechanism

## Le rôle des interactions exciplexes dans la photo-initiation de polymérisation par les systèmes fluorénone-amines

### Résumé

Les mélanges fluorénone-amines ont les applications industrielles dans le domaine de photo-durcissement, et par conséquent, ils ont suggéré les études détaillées de la cinétique de polymérisation et des poids moléculaires des produits polymères dans le cas de l'homopolymérisation en benzène à 30°C, photo-amorcée par fluorénone et par plusieurs de ses dérivés. On a utilisé triéthylamine, *N,N*-diméthylamine, et l'acide indole-3-ylacétique. Tous ces composés ont rendu des résultats fortement différents, mais en chaque cas, une augmentation de la concentration d'amine provoque un effet favorable sur la vitesse de polymérisation jusqu'à un certain point, et au delà duquel les processus de déactivation à cause des états activés deviennent prédominants. On suggère que les radicaux libres amorceurs sont dégagés au cours des processus chimiques avant de la formation initiale des exciplexes entre la cétone photo-activée et l'amine primitive. Dans plusieurs cas l'extinction des exciplexes par méthyle méthacrylate joue un rôle important et avec les effets plus généraux du solvant (y compris les constituants monomères ou amines) sur la concurrence entre les réactions qui conduisent à la formation des radicaux libres et celles qui régénèrent les constituants amines ou cétones, ils servent à démontrer l'extrême difficulté à laquelle on fait face en proposant des généralisations sur les rapports entre la structure et l'activité à l'égard des systèmes de

photo-amorceurs cétone-amines.

D'un intérêt particulier c'est la diminution vraisemblable des processus de transfert ou de terminaison dans le cas où l'on remplace la fluorénone par 2-méthylfluorénone. Celle-ci sert de modèle pour l'agent chromophore présente en copolymères de 2-vinylfluorénone et méthyle méthacrylate, et elle possède un spectre d'absorption qui en comparaison de celui de fluorénone s'est déplacé légèrement dans le rouge et également est plus fortement fluorescent.

Afin d'élucider les effets très complexes qu'exercent les amines sur les processus d'initiation et de terminaison, on a étudié la photo-initiation de la polymérisation de méthyle méthacrylate au moyen des copolymères contenant des groupements de fluorénone pendants. On conclut que l'initiation par le radical dérivé de la fluorénone (semipinacol) n'est pas importante, et ainsi il est probable que la formation du copolymère greffé (et probablement les vitesses de photo-durcissement augmentées) dans le cas des systèmes donnés serait plus efficace lorsqu'on utilise des polymères aux groupements amines pendants plutôt que ceux aux noyaux cétoniques pendants.

\*Presented at the Second European Symposium on "Ultraviolet Polymerisation and the coatings industry" held by the Newcastle Section at Durham University on 14-15 September 1977.

## Exciplex Reaktionen bei der Photoinitiation der Polymerisation mittels Fluorenon-Amin Systemen

### Zusammenfassung

Fluorenon-Amin-Mischungen werden in Photohärtungsverfahren allgemein angewandt. Sie regen ins Einzelne gehende Untersuchungen der Polymerisationskinetik und Molekulargewichte polymerer Produkte für die Homopolymerisation von Methylmethakrylat in Benzol bei 30°C an, photoinitiert durch Fluorenon und verschiedene Derivate. Triäthylamin, N,N-Di-methyläthanolamin und Indol-3-Ylessigsäure wurden benutzt und ergaben voneinander abweichende Resultate, obwohl in allen Fällen Erhöhung der Aminkonzentration sich nur auf die Geschwindigkeit bis zu einem gewissen Punkt günstig auswirkte, wonach Deaktivierungsvorgänge im Empfindlichkeitszustand vorherrschen. Es wird vorgeschlagen, dass die Reaktion anregenden freien Radikale durch chemische Vorgänge gebildet werden, im Anschluss an die anfängliche Bildung von Exciplexen zwischen lichtempfindlichen Ketonen und Aminen im Grundzustand. Exciplex Auslöschung durch Methylmethakrylat ist in vielen Fällen wichtig und im Zusammenhang mit den allgemein auftretenden Auswirkungen von Lösungsmitteln (einschliesslich Monomer- und Amin-Komponenten) auf die Konkurrenz zwischen Reaktionen, die zur Bildung freier Radikale führen, und denen, welche Amin- und Ketonverbindungen regenerieren, hilft die ungewöhnliche Schwierigkeit zu illustrieren, die Beziehungen

Struktur-Aktivität in Keton-Amin-Photoinitiator-Systemen zu verallgemeinern.

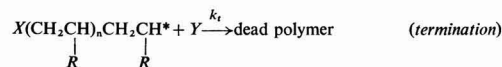
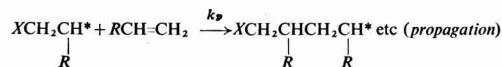
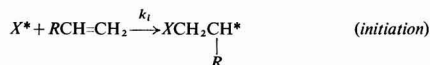
Von besonderem Interesse ist die scheinbare Reduktion bei Transferierungs- oder Endprozessen durch Ersatz von Fluorenon mit 2-Methylfluorenon. Letzteres dient als Modell für den Chromophor in Kopolymeren von 2-Vinylfluorenon, und Methylmethakrylat hat ein etwas nach Rot zu verschobenes Absorptionsspektrum und ist viel stärker fluoreszierend als Fluorenon.

Die Photoprimärreaktion bei der Methylmethakrylatpolymerisation unter Benutzung von Kopolymeren mit Seiteneinheiten von Fluorenon wurde untersucht, um zur Klärung des so komplexen Einflusses von Aminen auf die Primär- und Endprozesse zu verhelfen. Es wird geschlossen, dass Einleitungsreaktion durch von Fluorenon abgeleitete (Semipinacol) Radikale unwichtig ist, und somit Pfropfkopolymerbildung (und vermutlich Verstärkung der Photohärtung) für ein gegebenes System aller Wahrscheinlichkeit nach wirkungsvoller ist, wenn Polymere mit angehängten Amineinheiten benutzt werden, als mit angehängten Ketonringen.

### Introduction

Refs. 1, 2, 3

Chain reaction polymerisation processes predominate for olefinic monomers<sup>1</sup>. They usually involve propagating intermediates which are free radical, carbocationic, carbanionic or complex organometallic in nature and, in highly simplified form, may be represented as follows:



Mechanisms for termination differ markedly according to the nature of the propagating species as do the variety of chain transfer reactions, deliberately omitted from the simple scheme. However, it is important to remember that the same competing processes control the more complex chain polymerisations leading to formation of insoluble, cross-linked resins from systems comprising multifunctional monomers.

Initiation of chain polymerisation processes is consequent upon generation of the reactive intermediate ( $X^*$ ) by thermal, photochemical or other types of activation or decomposition of a suitable labile molecule. Photochemical methods of activation of labile systems may lead to formation of radicals or ionic species, although to date, it is mainly the former which have proved of real value and which will, therefore, occupy the bulk of the following discussion.

The photoinitiation of radical chain polymerisations has become increasingly important during the past five to ten years largely because of the ever increasing requirements for photoactive relief printing plates, printed circuits and a wide variety of photochemically cured surface coatings. Even more recently, there has been an upsurge of interest in development of photochemically curable printing inks for use in applications where environmental or economic considerations demand the use of solvent free systems. In all these processes

the final polymeric product is usually a crosslinked resin produced by photoinitiated polymerisation of mixtures of suitable prepolymers with mono- and polyfunctional olefins. As has already been noted, the basic chain steps are similar, irrespective of the degree of functionality of the polymerising components and, for photochemically active free radical systems, polymerisation depends upon initial light-induced formation of free radicals from a suitable photoinitiator. Assuming the usual radical termination processes, the overall rate of polymerisation ( $R_p$ ) is given by:

$$R_p = \frac{k_p}{k_t^{1/2}} R_i^{1/2} M$$

and the rate of initiation ( $R_i$ ) is approximated by:

$$R_i = I_0 \phi \epsilon [\text{initiator}]$$

where  $I_0$  = intensity of incident radiation,  $\phi$  = quantum yield for initiation of radical chains, and  $\epsilon$  = molar extinction coefficient of initiator at the wavelength employed.

Thus, given an appropriate light source, important requirements for efficient photoinitiation include suitable absorption coefficients and wavelength sensitivities for the initiator molecule, together with quantum yields for initiation in the range 0.1–1.0. Another extremely important criterion is that the initiator molecule, or any of its photofragments, should not function as chain transfer or terminating agents. In recent years the authors have been engaged in a systematic study of a wide range of important photoinitiator systems in an attempt to determine these properties<sup>2</sup>. Estimates of the chain termination and transfer effects of components and photofragments in any particular polymerising system are readily available from studies of rates of polymerisation and polymer molecular weights, which yield values of the ratio  $k_p/k_t^{1/2}$ . Since  $k_p$  is essentially constant for the polymerisation of any given olefin, variations in values of  $k_p/k_t^{1/2}$  arise from termination and transfer processes not apparent in comparable conventional polymerisations initiated (thermally and photochemically) by e.g. AIBN. Molecular weights can only be measured for polymers which are truly soluble and, for convenience, the homopolymerisation of methyl methacrylate in bulk or in various solvents has been chosen as a model system. The majority of commercially applicable photocure systems depend upon the formation of insoluble crosslinked resins as products, and so caution must be applied when comparing data for relative initiator effects in the model,

soluble polymer system and the more viscous, multicomponent, commercially applicable systems.

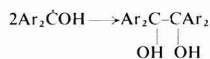
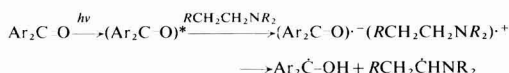
Free radical initiators are extensively employed as curing agents or promoters in a wide variety of polymerisation processes, especially those required for decorative or protective surface coatings. A large proportion of the useful range of photoinitiators<sup>3</sup> possess, as the light absorbing chromophore, an aromatic carbonyl group ( $\text{ArCOX}$ ). These classes of compound have light absorption characteristics in the 300–400 nm region with high quantum yields for the appropriate radical forming reactions. Aromatic carbonyl compounds may have lowest lying  $\pi$ ,  $\pi^*$  or  $n$ ,  $\pi^*$  excited states according to the nature of the aryl group and frequently exhibit inter-system crossing to the appropriate lowest energy triplet states.

### Photoreduction of aromatic carbonyl compounds by amines

Refs. 3–13

In contrast to the well documented<sup>3, 4</sup> homolytic fragmentation and hydrogen abstraction reactions of aromatic carbonyl compounds which normally involve  $n$ ,  $\pi^*$  excited states, a very wide range of so-called exciplex interactions involving both  $n$ ,  $\pi^*$  and  $\pi$ ,  $\pi^*$  excited states have been observed. Notable among these are the wide variety of photo-induced oxidations of aminocompounds by aromatic carbonyl compounds<sup>5</sup>.

Singlet and triplet states of aromatic ketones are efficiently quenched by a wide variety of aminocompounds and a general correlation exists between quenching efficiency and ionisation potential of the amine, for a particular class of amine. In many cases tertiary amines are more effective than corresponding secondary and primary derivatives, although specific solvation phenomena may change this order of reactivity. Reduction of aromatic ketones by amines proceeds at rates which are substantially faster than those observed for corresponding photo-induced hydrogen abstractions from, say, alcohols. Products from photo-oxidation of amines, may be generalised as follows:



The authors have preferred to utilise ketones thought to have lowest lying  $\pi$ ,  $\pi^*$  triplet excited states because, although the point is still uncertain in some cases,  $\pi$ ,  $\pi^*$  excited triplets do not generally undergo direct hydrogen abstraction reactions with alcohols, alkanes *etc.* Fluorenone (FLO) is an aromatic ketone having a lowest lying  $\pi$ ,  $\pi^*$  triplet excited state and its photophysical characteristics have been extensively studied by several groups of workers<sup>6–8</sup>.

Photoreduction of fluorenone does not occur in alcohol, ether or alkane solvents, but occurs readily in the presence of amines, with tertiary amines being most effective<sup>9, 10</sup>. A rather special feature of the photochemistry of fluorenone is the dramatic effect of solvent on the rates of inter-system crossing from singlet to triplet manifolds. Increasing solvent

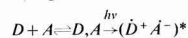
polarity decreases the facility for inter-system crossing and simultaneously increases the quantum yield for fluorescence. Thus triplet state activity is maximised in solvents such as benzene and cyclohexane, for which values of triplet yields are 0.93 and 1.03 respectively<sup>11</sup>. In contrast, triplet yields in solvents such as alcohols, acetone, acetonitrile are substantially less than unity with a concomitant increase in quantum yields for fluorescence<sup>7, 8</sup>. Ionisation potentials of amines are important in determining the quenching ability for both singlet and triplet excited fluorenone but it now appears that *photoreduction* is consequent upon interaction of triplet excited fluorenone with amine donors<sup>5</sup>, the main effect of singlet state quenching being to reduce triplet yields. Evidence for the existence of both ion-radical pairs and free radicals is now available from detailed CIDNP studies of photoreduction of aromatic carbonyl compounds by several amines<sup>12, 13</sup>.

### The nature of exciplex interactions

Refs. 4, 14–20

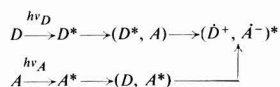
The simple reaction scheme outlined for photoreduction of aromatic carbonyl compounds by amines pre-supposes direct formation of a pair of ion radicals on the collisional interaction of an excited carbonyl compound and a ground state amine molecule. It is now well known that this is a gross oversimplification, and that ultimate production of a pair of ion radicals in these, or other systems, is subsequent to the formation of a number of possible intermediates, of which the most important is generally referred to as an exciplex.

Charge transfer complexes formed by interaction of a donor and an acceptor molecule, both in their ground states, are well characterised and have excited states with a substantial degree of electron transfer when compared to the corresponding ground state, *e.g.*



Nevertheless, the excited states of a charge transfer complex should not be equated with a thermally-equilibrated pair of ion radicals.

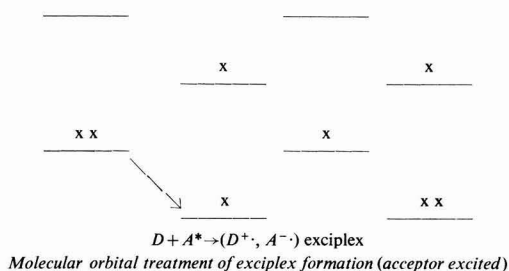
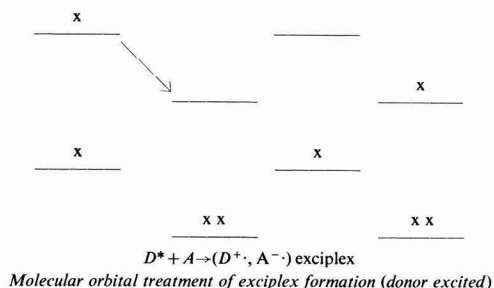
More recently, mainly as a result of the pioneering studies of Weller and his associates<sup>14</sup>, it has become apparent that excited states, having structures equivalent to those of photoexcited charge transfer complexes, may be formed by local excitation of one or the other component (*i.e.*  $D$  or  $A$ ) in systems which do not give evidence of ground-state complex formation, *e.g.*



Here the excited state having the most electron-transfer character  $(\dot{D}^+, \dot{A}^-)^*$  is termed an exciplex and its formation may be preceded by a variety of collisional complexes (encounter complexes) between an excited donor and ground-state acceptor or vice versa, *e.g.*  $(D^*, A)$  and  $(D, A^*)$ .

A simple molecular orbital treatment due to Weller<sup>14</sup> assumes that an electron is transferred from an excited state of one component to the ground state of another as illustrated on page 98.

The ionisation potential ( $I_D$ ) and electron affinity ( $E_A$ ) are measures of the energies of the highest bonding and lowest antibonding molecular orbitals respectively, and hence



$\Delta G$ , the energy of formation of the separated ions  $\dot{D}^+$  and  $\dot{A}^-$  from  $D$  and  $A$  in their ground states, is given by  $I_D - E_A$ . Since the process actually starts with an excited state (e.g.  $A^*$ ), the energy of formation is diminished by the excitation energy of  $A^*$ . Bringing the separated ions  $\dot{D}^+$  and  $\dot{A}^-$  to their equilibrium distance ( $r$ ) in the exciplex reduces  $\Delta G$  by an electrostatic term ( $-e^2/r$ ). It follows, therefore, that the free energy of exciplex formation in free space is given by:

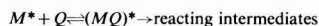
$$\Delta G = I_D - E_A - hv - \frac{e^2}{r}$$

where  $hv$  is the excitation energy of  $A^*$  or  $D^*$  as appropriate. Because a molecule in its excited state is both a better electron donor and a better electron acceptor than in its ground state, exciplex formation should be a widespread phenomenon<sup>15</sup>. A particular advantage of ion radical formation *via* exciplexes rather than ground state charge transfer complexes is that a much wider range of substrates may be employed, including many compounds which do not interfere with commercially important polymerisation systems. However, it must be stressed that molecules having ionisation potentials or electron affinities typical of those observed in formation of ground state charge transfer complexes can equally well participate in exciplex formation with partners which do not give rise to ground state adducts. Likewise it is not necessarily the case that the photoexcited state of a ground state charge transfer adduct is the same as an exciplex formed by collisional interaction of an excited (uncomplexed) component<sup>16</sup>.

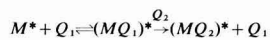
Exciplex intermediates are now commonly invoked to explain quenching of singlet and triplet excited states where orbital energies of the excited state and of the quencher preclude the common exothermic electron exchange quenching process. Detailed studies<sup>17, 18</sup> of the quenching of carbonyl compounds by a wide range of ethylenically unsaturated molecules are useful as an indication of potential problems to be encountered in initiation of olefin polymerisations, and serve to illustrate the dangers of oversimplifying correlations between quenching efficiencies and ionisation potentials (or electron affinities).

In addition to their widespread occurrence in simple fluorescence and phosphorescence quenching, exciplexes may sometimes be properly identified as intermediates by their characteristic luminescence spectra<sup>14, 15</sup>. Such emitting exciplexes may then, in appropriate systems, be quenched by addition of suitable third component molecules. The studies of Caldwell and his collaborators<sup>19</sup> are of particular significance in this respect and help to establish important further reaction consequences of exciplexes: ( $M^*$  represents the appropriate excited species,  $Q$  represents any quenching species:

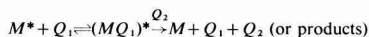
(i) Exciplex formation is a thermodynamically reversible process in competition with subsequent formation of useful chemical intermediates.



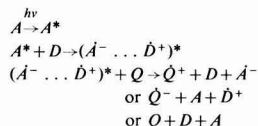
(ii) Exciplexes may undergo substitution with a second quenching component, e.g.



(iii) Exciplexes may be quenched by reaction with a second type of quencher, e.g.



In considering the possible role of exciplexes in initiation of vinyl polymerisations the conclusions of Caldwell *et al.*<sup>19</sup> are especially important, since they indicate that both useful photochemistry and unwanted quenching processes may result mainly from termolecular phenomena which, for the particular case of ketone ( $A$ ) and amine ( $D$ ) combinations may be represented:



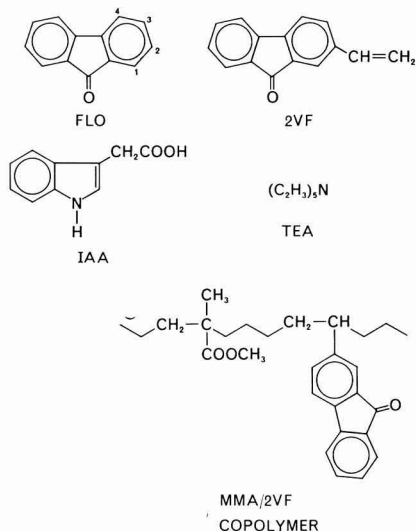
Generally  $Q$  may be a polymerisable olefinic component, or any other additive or impurity present in the particular system. It is also probable that exciplexes play an important role in the self quenching of ketone excited states and for all systems exhibiting exciplex interactions, polarity of the medium and any specific solute-solvent interactions may influence the respective deactivation pathways. Michler's Ketone has both aromatic carbonyl group and amino groups in the same molecule and is a component of many photo-initiation systems<sup>4</sup> utilised in commercially important photo-cure processes. Very recent detailed studies<sup>22</sup> of its photochemical and photophysical behaviour amply illustrate the rather complex nature of exciplex interactions and solvent effects in ketone-amine systems.

#### Photoinitiation of methyl methacrylate polymerisation by fluorenone-amine combinations

*Refs. 5, 21*

Methyl methacrylate was chosen as a convenient model for the polymerising components of typical photocure systems and its homopolymerisation was studied in bulk and in benzene solvent. Fluorenone was selected as the most convenient carbonyl compound because although there are complexities associated with medium effects in the relative energy levels of  $n$ ,  $\pi^*$  and  $\pi$ ,  $\pi^*$  excited states, and on rates of inter-system crossing, the chemical products of photoreduction by amines are well characterised<sup>5</sup>. Selection of the amine component

was determined mainly by the demands of experimental convenience in vacuum line manipulations. Triethylamine (TEA) was chosen so as to permit comparison of photo-reduction data with that existing in the literature<sup>5</sup>, *N,N*-dimethylethanolamine ((CH<sub>3</sub>)<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>OH, DME) represents an important class of amine used in commercial systems, and indole-3-ylacetic acid (IAA) is a crystalline material with excellent electron donor properties and a high propensity to photoinduced decarboxylation with formation of the indole-3-ylmethyl radical.



At low fluorenone concentrations, DME and IAA gave rise to initial rates of photoinitiated polymerisation of methyl methacrylate in benzene solution which could be fitted to the following kinetic expression, provided that the amine concentration remained constant:

$$R_p = -\frac{d[\text{MMA}]}{dt} = k_p[\text{FLO}]^{\frac{1}{2}}[I_0]^{\frac{1}{2}}[\text{MMA}]$$

The effects of varying amine concentration on rates of polymerisation were complex and differed for the three amines, although in each case, there is a limit to the increase in rate which may be accomplished by increasing the amine concentration. Typical data are given in Figures 1 and 2. For initiation involving DME and IAA the variations in rate with increasing [amine] were accompanied by the expected changes in molecular weight (Figures 6, 7), indicating that the effects of [amine] were associated with initiation rather than propagation.

In contrast, the data for TEA (compare Figures 3–5) give clear evidence for unusual effects of amine and fluorenone on both initiation and termination processes. For example, values of the kinetic parameter  $k_p/k_t^{\frac{1}{2}}$  decrease in the range 0.046–0.018 litre<sup>1</sup> mol<sup>-1</sup> s<sup>-1</sup> as the concentration of fluorenone is increased from  $3.6 \times 10^{-5}$  M to  $4.0 \times 10^{-3}$  M, and there is no simple dependence of rate on [FLO]<sup>1</sup>. Figures 1–7 indicate that IAA is effective as a coinitiator at concentrations 1–2 orders of magnitude lower than those required for TEA and DME respectively and this could reflect increased efficiency of exciplex formation in FLO/IAA systems.

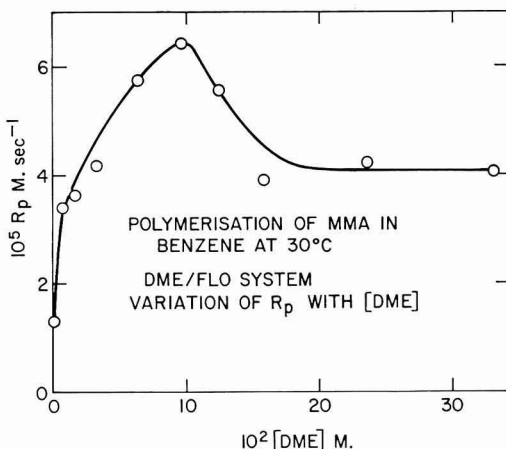


Fig. 1. Polymerisation of methyl methacrylate in benzene at 30°C photoinitiated by fluorenone/DME  
[FLO] =  $1.14 \times 10^{-4}$  M [MMA] = 3.9 M

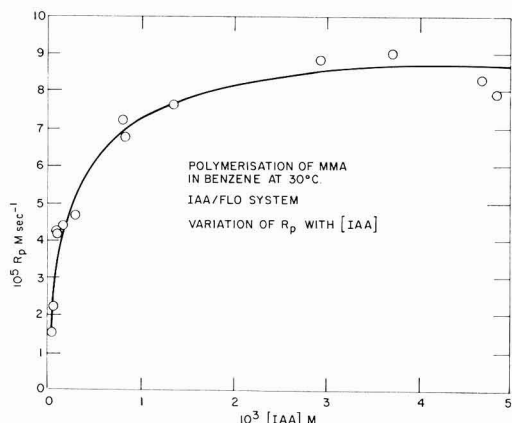


Fig. 2. Polymerisation of methyl methacrylate in benzene at 30°C photoinitiated by fluorenone/IAA  
[FLO] =  $2 \times 10^{-4}$  M [MMA] = 6.04 M

Generally speaking, quantum yields for initiation by FLO/amine systems fall in the range 0.1–0.3 depending on substrates and conditions and it is important that values of  $k_p/k_t^{\frac{1}{2}}$  may be significantly less than the normal value ( $\sim 0.06$ ). Thus for IAA/FLO and DME/FLO combinations at 30°, typical values of  $k_p/k_t^{\frac{1}{2}}$  for polymerisation of methyl methacrylate in benzene are 0.036 and 0.038 respectively. Such low values of  $k_p/k_t^{\frac{1}{2}}$  imply termination or transfer by the initiator components or, more probably, by primary radicals produced on irradiation. In order to gain insight into these effects, a number of substituted fluorenones were evaluated as photoinitiators when activated by IAA. After correcting for relative light absorption characteristics at the wavelengths employed for activation (366 or 405 nm), relevant data ( $10^4 R_p$  M.sec<sup>-1</sup>,  $k_p/k_t^{\frac{1}{2}}$  M<sup>-1</sup>sec<sup>-1</sup>) are as follows: fluorenone (1.04, 0.036); 2-methylfluorenone (0.92, 0.066); 1-methylfluorenone (1.06, 0.042); 2-methoxycarbonylfluorenone (0.73,

0.066); 2-methoxyfluorenone (0.30, 0.042). These data indicate a great deal of complexity in the various processes competing to deactivate intermediate exciplexes, prior to radical formation and to scavenge radicals once they are formed.

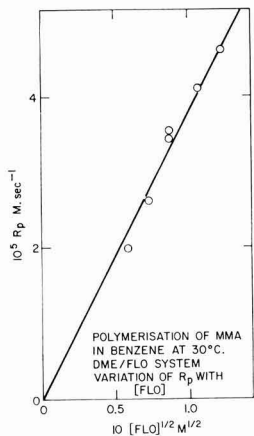


Fig. 3. Polymerisation of methyl methacrylate in benzene at 30°C photoinitiated by fluorenone/DME  
 [DME] = 0.33 M [MMA] = 3.9 M

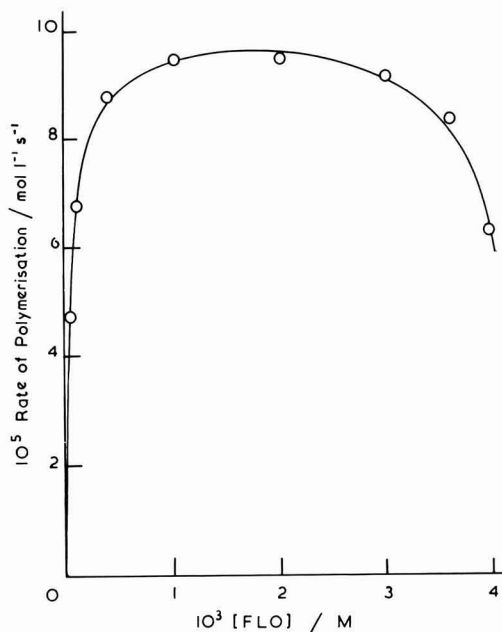


Fig. 5. Polymerisation of methyl methacrylate in benzene at 30°C photoinitiated by fluorenone/TEA  
 [TEA] = 0.1 M [MMA] = 5.0 M

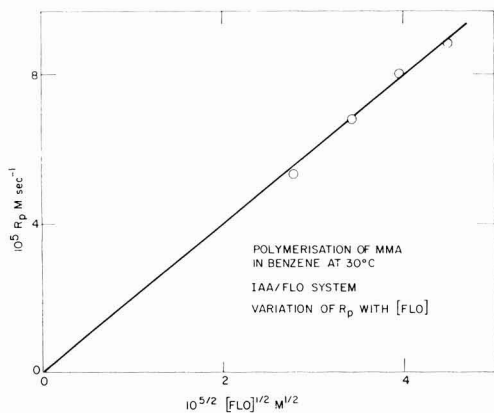


Fig. 4. Polymerisation of methyl methacrylate in benzene at 30°C photoinitiated by fluorenone/IAA  
 [IAA] =  $2.9 \times 10^{-3}$  M [MMA] = 6.1 M

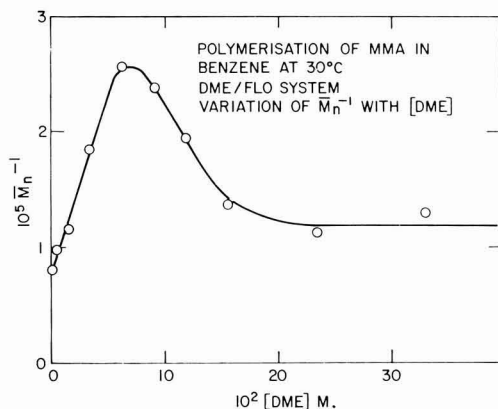


Fig. 6. Polymerisation of methyl methacrylate in benzene at 30°C photoinitiated by fluorenone/IAA  
 [FLO] =  $2 \times 10^{-4}$  M [MMA] = 6.0 M

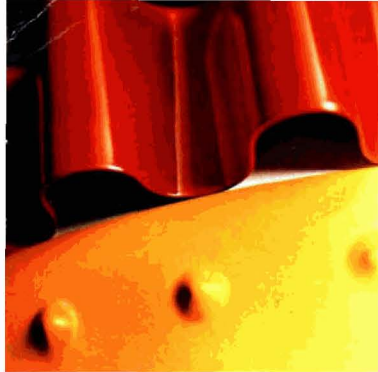




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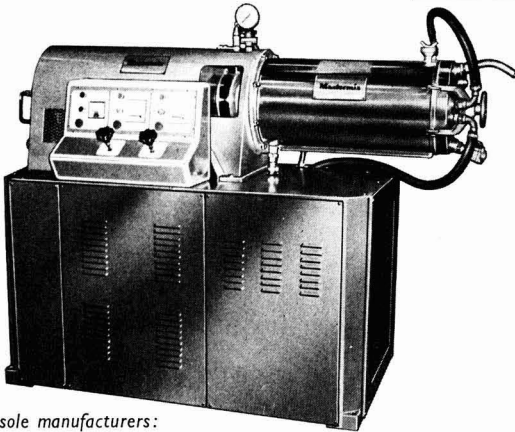
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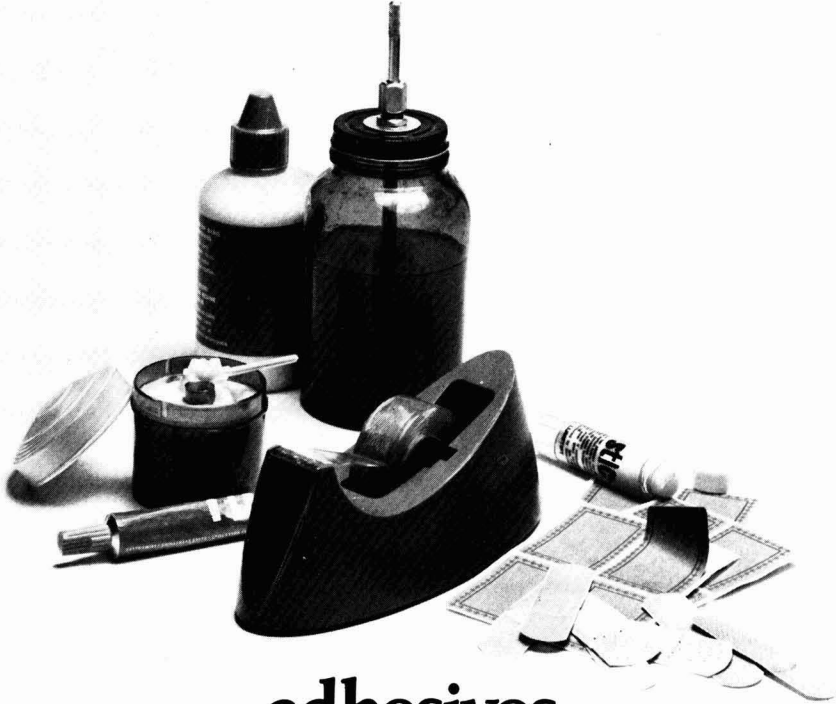
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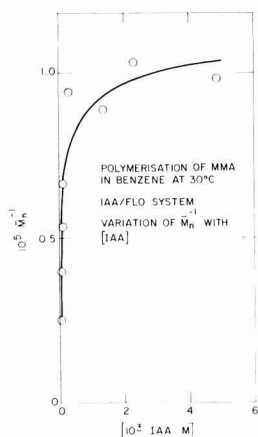
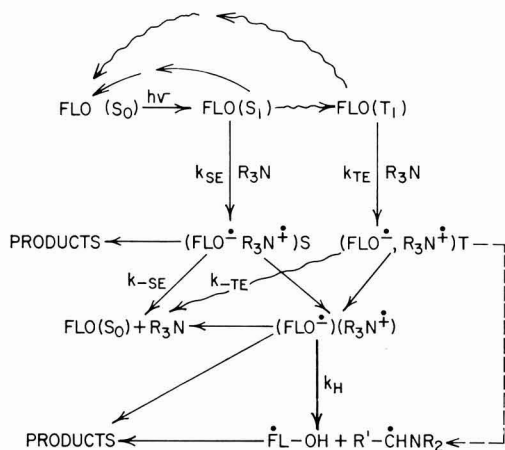


Fig. 7. Polymerisation of methyl methacrylate in benzene at 30°C photoinitiated by fluorenone/DME

$$[\text{FLO}] = 1.14 \times 10^{-4} \text{ M} \quad [\text{MMA}] = 3.91 \text{ M}$$

A reaction scheme which can be used to account for many of the experimental observations, especially those resulting from changes in efficiency of initiation, is given below:



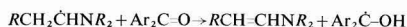
Here the quenching and/or energy wastage mechanisms for excited fluorenone not involving amine are represented, for clarity, by the curved arrows at the top of the diagram. Singlet (*S*) and triplet (*T*) exciplexes ( $\text{FLO}^{\cdot-} \text{R}_3\text{N}^{\cdot+}$ ) are produced from the appropriate excited states by collisional encounters with amine molecules ( $k_{SE}$  and  $k_{TE}$  respectively) and may undergo radiationless decay to ground states by the reverse processes ( $k_{-SE}$ ,  $k_{-TE}$ ). Chemical products are seen as arising directly from the triplet exciplex or, *via* a pair of ion-radicals ( $\text{FLO}^{\cdot-}(\text{R}_3\text{N}^{\cdot+})$ ), formed by further relaxation of either singlet or triplet exciplexes, between which the proton transfer ( $k_H$ ) is kinetically important. Solvent effects previously noted suggest that, for production of radical species, relaxation of singlet exciplexes ( $\text{FLO}^{\cdot-} \text{R}_3\text{N}^{\cdot+} S$ ) does not compete as favourably with  $k_{-SE}$  as does the comparable relaxation of triplet exciplexes ( $\text{FLO}^{\cdot-} \text{R}_3\text{N}^{\cdot+} T$ ) with  $k_{-TE}$  or direct formation of the radicals from the triplet exciplex.

Photoinitiation of polymerisation by fluorenone-amine systems is accompanied by consumption of fluorenone, as evidenced by the progressive diminution in the characteristic yellow colour of reaction mixtures. It was of considerable interest, therefore, to examine the quantum yields for reduction of fluorenone by the three amines used as coinitiators, under typical polymerisation conditions. Representative data are given in Table 1.

Table 1  
Quantum yields for photoreduction of fluorenone at 30°C

Solvent	DME	IAA	TEA
Benzene	1.22	0.19	0.69
Benzene/MMA (1 : 1)	0.37	0.28	0.18

It has been noted earlier that quantum yields for photoreduction of ketones by amines take account of a ground state radical reaction such as:



and would, therefore, be expected to decrease under conditions where the intermediate free radicals initiate polymerisation. Similar radical reactions are not likely for the special case of IAA and this may explain the lower quantum yields observed for this particular amine.

The results of Table 1 are in broad agreement with these ideas in that for DME and TEA, MMA actually appears to retard photoreduction of fluorenone, whereas the presence of MMA is beneficial to the reduction of fluorenone by IAA. This may go some way towards explaining the very much higher activity (per mole) in photoinitiation of IAA compared with DME and TEA. Why, or how, methyl methacrylate monomer is apparently able to quench excited states, or otherwise retard processes involved in photoreduction of fluorenone is not clear. The most likely explanation would be that methyl methacrylate (and presumably other acrylic monomers) quenches either the singlet or triplet exciplexes formed between fluorenone and the amine, *e.g.*



In an attempt to clarify these problems, similar experiments were performed with various ring substituted fluorenones, of which 2-methylfluorenone was the most important, since it served as a model alkyl fluorenone chromophore for copolymers derived from 2-vinylfluorenone<sup>21</sup> (2VF) and methyl methacrylate. Fluorenones having 2-alkyl substituents are much more intensely fluorescing than the unsubstituted ketone and exhibit a slight red shift in their absorption spectra (Figure 8). The latter property could make alkyl fluorenones more useful than fluorenone in photocure applications where pigmented systems are required. A number of 2VF/MMA copolymers were investigated, but there was little effect of composition on rates of photoinitiation and ketone photoreduction. Consequently, data for the copolymer MMA/2VF having 18 mole per cent 2VF may be taken as representative of the entire series.

Table 2 gives data for quantum yields of photoreduction of 2-methylfluorenone and the copolymer under a variety of conditions.

Comparison of the data of Tables 1 and 2, and of much independent work on the fluorenone/TEA systems reported

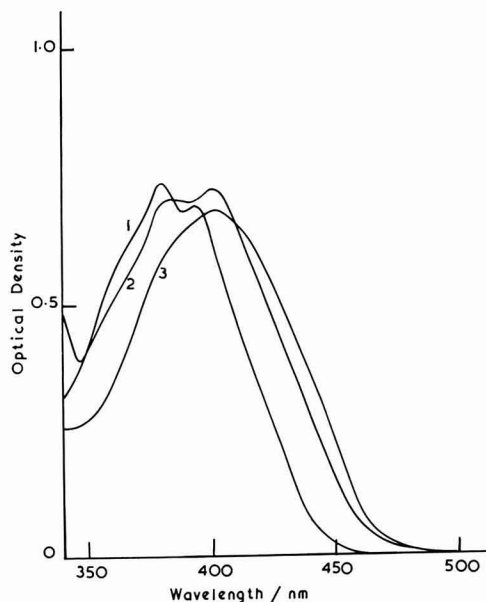


Fig. 8. Long wavelength absorption spectra:

1. Fluorenone. 2. 2-methylfluorenone. 3. Copolymer of MMA and 2-vinylfluorenone

[Ketone] =  $\sim 2.5 \times 10^{-2}$  M

Table 2

Quantum yields for photoreduction in benzene at 30°C

[Ketone] =  $3 \times 10^{-3}$  M, [MMA] = 5.0 M

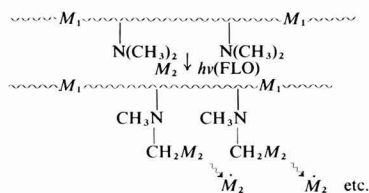
Ketone	IAA $3 \times 10^{-3}$ M	TEA 0.10 M	TEA 0.01 M
2-methylfluorenone	0.25	0.27	—
2-methylfluorenone+MMA	0.28	0.13	0.20
MMA/2VF (18%)	0.15	0.29	—
MMA/2VF (18%)+MMA	0.21	0.15	0.21

in the literature<sup>5</sup>, shows that decreasing the concentration of TEA brings about an improvement in the quantum yields for photoreduction. In addition, as observed for fluorenone (Table 1), methyl methacrylate quenches photoreduction of the alkyl fluorenones by TEA, but not that induced by IAA.

#### Photoinitiation by copolymers containing fluorenone units

Refs. 3, 5, 16, 22-27

The great variety of substrates participating in exciplex formation may be readily extended to polymer-based systems. Of these, ketone-amine combinations offer most scope since it is comparatively easy to prepare a range of copolymers containing 2-dimethylaminoethyl methacrylate units as electron donor entities. Such copolymers, when allowed to interact with photoexcited fluorenone, are effective in causing rapid crosslinking and gelation, e.g.:



More recently the authors have studied the utilisation of copolymers of methyl methacrylate and 2-vinyl-fluorenone as photoinitiators. In combination with suitable amine donors, these polymeric carbonyl compounds are effective crosslinking and gelling agents for methyl methacrylate, styrene and acrylonitrile monomers, and in this respect, behave in a manner similar to that of the polymer-based amines. However, detailed examination of rates of polymerisation and molecular weights of products at conversions below the gel-point have indicated new mechanistic features for amine-ketone systems.

Table 3

Photoinitiated polymerisation of MMA<sup>a</sup> by 2VF/MMA copolymers

Mole % 2VF in copolymer	Amine	[Amine] M	$10^4 R_p$ M s <sup>-1</sup>	$k_p/k_t^{1/2}$ M <sup>-1/2</sup> s <sup>-1/2</sup>
1.1 <sup>b</sup>	TEA	0.1	1.2	0.033
2.2 <sup>b</sup>	TEA	0.1	1.3	0.037
18.4 <sup>b</sup>	TEA	0.1	1.2	0.035
38.3 <sup>b</sup>	TEA	0.1	1.1	0.037
67.9 <sup>b</sup>	TEA	0.1	1.3	0.042
(2-MeF) <sup>b</sup>	TEA	0.1	0.86	0.034
(FLO) <sup>b</sup>	TEA	0.1	0.98	0.022
18.4 <sup>b</sup>	TEA	0.01	1.75	0.041
38.3 <sup>b</sup>	TEA	0.01	1.50	0.054
18.4 <sup>c</sup>	IAA	$10^{-3}$	1.15	0.066
(2-MeF) <sup>c</sup>	IAA	$10^{-3}$	0.92	0.066
(FLO) <sup>c</sup>	IAA	$10^{-3}$	1.04	0.036

a. [MMA] = 5.0 M; Temp 30°C; Solvent benzene

b. [Ketone] =  $3 \times 10^{-3}$  M

c. [Ketone] =  $1 \times 10^{-3}$  M

Again using methyl methacrylate as the model system in benzene solvent ([MMA]=5.0 M) at 30°C, a range of copolymers of 2-vinyl fluorenone with methyl methacrylate (containing from 1-70 per cent 2-vinyl fluorenone) in the presence of IAA or triethylamine, were found to give polymerisation rates and photoreduction characteristics (per mole of ketone) very similar to that given by 2-methylfluorenone (Tables 2, 3). Polymers isolated before gelation were examined by G.P.C. in THF solution and gave clear evidence of both homopolymethylmethacrylate, the expected main reaction product, and a distinct polymeric product having an average molecular weight substantially greater than that of the starting initiator-polymer (Figure 9). Rather similar results were observed for reactions in which IAA was replaced by TEA in the 0.01 M concentration range (Figure 10). In contrast, related experiments in which the TEA concentration was maintained at approximately 0.1 M gave polymeric products having only one component, corresponding to the expected homopolymerised methyl methacrylate (Figure 11).

Before considering the mechanistic implications of these data it is worthwhile to note the nature of the polymeric products obtained by photolysis of the VF/MMA copolymers in the presence of IAA, but without added monomer. Typical G.P.C. curves are outlined in Figure 12 and indicate that



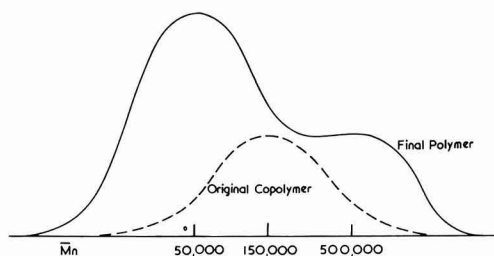


Fig. 9. G.P.C. curves for reactants and products of polymerisation of MMA (5.0 M) in benzene photoinitiated by MMA/2VF copolymer (18% 2VF) and IAA ( $10^{-3}$  M) in benzene at  $30^\circ\text{C}$  ( $[\text{Ketone}] = 6 \times 10^{-3}$  M)

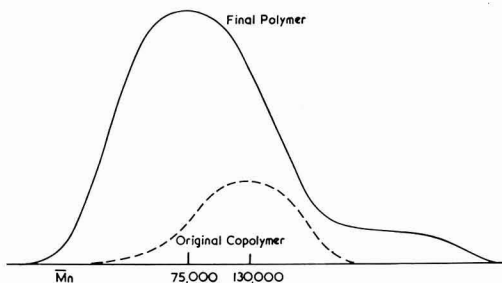


Fig. 10. G.P.C. curves for reactants and products of polymerisation of MMA (5.0 M) in benzene at  $30^\circ\text{C}$  photoinitiated by MMA/2VF copolymer (18% 2VF) and TEA (0.01 M) ( $[\text{Ketone}] = 6 \times 10^{-3}$  M)

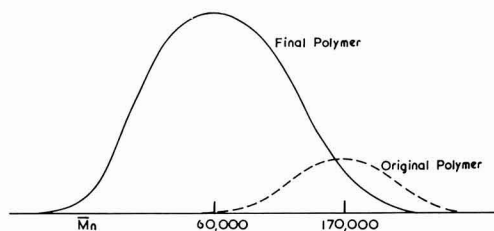


Figure 11. G.P.C. curves for reactants and products of polymerisation of MMA (5.0 M) in benzene at  $30^\circ\text{C}$  photoinitiated by MMA/2VF copolymer (18% 2VF) and TEA (0.1 M)

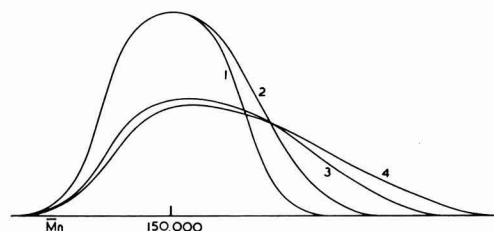


Fig. 12. G.P.C. curves of reactants and products of photolysis of MMA/2VF copolymer (18% 2VF) in the presence of IAA ( $6 \times 10^{-3}$  M) as a function of solvent ( $[\text{Ketone}] = 6 \times 10^{-3}$  M)

1. Original copolymer. 2. Benzene. 3. Acetone. 4. Ethyl acetate

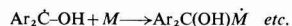
formation of high molecular weight polymeric products, which for these combinations can arise only by polymer-polymer combinations, is strongly solvent dependent. In the

extreme case, photolysis of the 18 per cent VF/MMA copolymer in tetrahydrofuran containing  $10^{-3}$  M IAA, induced gelation at a rate which precluded sampling of the initial soluble fraction. Inspection of all G.P.C. traces, and comparison of reaction conditions, suggests strongly that the high molecular weight products obtained (Figures 9, 10) by photolysis of polymeric ketone-amine combinations in the presence of monomeric methyl methacrylate similarly arise mainly from polymer-polymer combination processes.

This important conclusion is further reinforced by values of the kinetic parameters  $k_p/k_t^{1/2}$  for polymerisations initiated by the various ketone-amine combinations (Table 3). In particular, it should be noted that for co-initiation by IAA, both 2-methyl fluorenone and the 18 per cent 2VF/MMA copolymer gave identical values ( $k_p/k_t^{1/2} = 0.066 \text{ M}^{-1/2} \text{ s}^{-1/2}$ ) which are similar to that anticipated for "normal" polymerisations of MMA (e.g. for initiation by AIBN<sup>3</sup>,  $k_p/k_t^{1/2} = 0.06 \text{ M}^{-1/2} \text{ s}^{-1/2}$  at  $30^\circ\text{C}$ ), whereas the value obtained for fluorenone ( $0.036 \text{ M}^{-1/2} \text{ s}^{-1/2}$ ) is indicative of significant additional transfer or termination processes. Here again, the 2-alkylfluorenone derivatives appear to be more useful photoinitiators than fluorenone, a fact which is borne out by similar data for co-initiation by triethylamine (Table 3), although in these cases the data indicate additional transfer or termination processes for all the ketones studied.

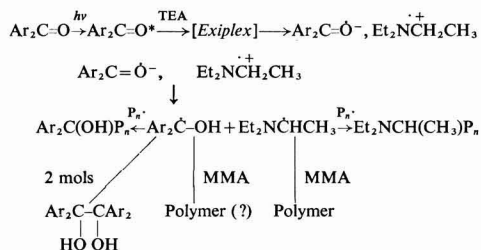
It must be concluded, therefore, that, at least for co-initiation by IAA, formation of high molecular weight graft products from the ketone copolymer photoinitiator, cannot arise by termination of growing MMA chains with initiator-polymer derived radicals.

Coupling of polymer based radicals to growing chains, or to monomer, would logically involve termination or initiation respectively by the semipinacol radicals produced from the ketone component:

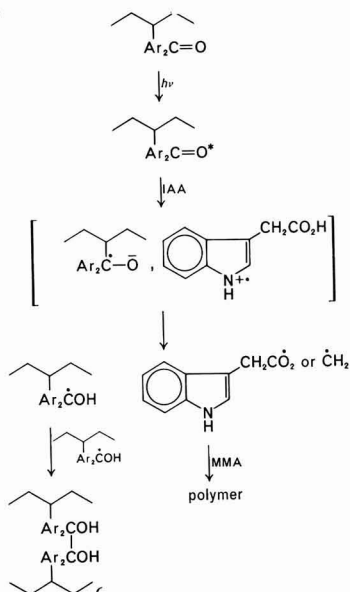


Independent studies<sup>22, 23</sup> indicate that FLOH and related semipinacol radicals are not especially efficient in initiation of polymerisation at ambient temperatures (but see below) and the important initiating species will, therefore, be amine derived radicals such as  $\text{RCHNR}_2$ . On the other hand, low molecular weight semipinacol radicals are effective terminating species for polymeric radicals<sup>22</sup>.

A generalised reaction scheme embracing the important reaction possibilities for radical intermediates produced in amine promoted photoinitiation for both small molecule and polymeric ketones, is indicated for the case of TEA. Deactivation pathways in which either the exciplex or the ion radicals return to starting components are omitted for clarity.



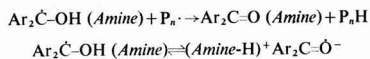
For the specific case of initiation by polymer-based ketones a suggested reaction scheme involving IAA is given below:



It is difficult to explain fully the results obtained for co-initiation by TEA and, considering that this simple tertiary amine is a very good model for many systems employed in commercial photocure processes, the complex nature of the kinetic and molecular weight data serve to indicate the difficulties of generalising structure-activity relationships in photoinitiation by ketone-amine combinations.

In contrast to the situation in co-initiation by IAA, polymerisations photoinitiated by polymeric ketone and triethylamine (TEA) showed no evidence for graft polymer formation at  $[\text{TEA}] = 0.1 \text{ M}$  and formed gelled systems only after prolonged irradiation. Identical polymerisations, but keeping  $[\text{TEA}] = 0.01 \text{ M}$ , showed slightly higher rates of polymerisation, gave clear (G.P.C.) evidence for grafting and formed gels readily. The conclusion must be that the higher concentrations of TEA retard, or prevent, coupling of the growing chains or reactions with polymer based radicals.

Possible explanations for the effects of amine concentration on rates and products of photoreduction of benzophenone have been discussed independently by Parola *et al.*<sup>5</sup>, but for the present work an additional process is required whereby triethylamine at 0.1 M suppresses or modifies self-interaction of polymer based semipinacol radicals or their reactions with growing chains or monomer. It is tempting to speculate that hydrogen bonding of semipinacol radical to the amine is important, so providing a mechanism for either amine mediated hydrogen transfer to growing radicals or monomer, or a diminished capability of  $\text{Ar}_2\text{COH}$  (Amine) to react by self combination perhaps because of initiation *e.g.*:



In any case this result would be extremely important for the design of systems involving polymer based photoinitiators having anticipated rapid cure characteristics.

Utilisation of polymer-bound initiator components is an obvious means of improving gel times or cure times in photo-initiated polymerisations as the authors<sup>16</sup> and others<sup>24, 25</sup> have demonstrated previously. Photoreductive coupling<sup>26</sup>, and attempted photoinduced grafting<sup>27</sup> of polymers containing aryl carbonyl groups have also been reported previously. Taken with the present results, these facts suggest that graft efficiencies in model polymer studies, and curing rates for commercial systems, may be improved more easily by employing polymers having pendent amine groups rather than pendent ketone groups.

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# Recent developments in photoinitiators\*

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## Summary

A survey is presented of the photoinitiators which are commercially used today. Information is given on the products of their photolysis and their performance in different UV curing systems. The prob-

lems encountered in the light stabilisation of UV curing systems are discussed.

## Keywords

*Processes and methods primarily associated with manufacturing or synthesis*

ultraviolet curing

*Processes and methods primarily associated with drying or curing of coatings*

radiation curing  
photoinitiation  
exciplex formation  
ketone initiation

*Raw materials*

binders (resins, etc)

acrylic resin  
polyester resin

*Properties, characteristics and conditions primarily associated with coatings during application*

curing rate

*Types and classes of coatings and allied products*

pigmented coating

## Développements récents dans le domaine de photo-amorceurs

### Résumé

On présente une revue au sujet des photo-amorceurs utilisés actuellement par les industries intéressées. On donne des renseignements sur leur comportement dans de différents systèmes durcissables

par les rayons ultra-violet. On discute les problèmes que l'on rencontre en assurant la solidité à la lumière des systèmes durcissables par les rayons ultra-violet.

## Neue Entwicklungen auf dem Gebiet der Photoinitiatoren

### Zusammenfassung

Eine Übersicht von gegenwärtig gehandelten Photoinitiatoren wird gegeben, ebenso wie Information über die Photolyse der Produkte und ihr Verhalten in verschiedenen UV Härtungssystemen. Die

hinsichtlich Licht - Stabilisierung bei UV Härtung vorkommenden Schwierigkeiten werden besprochen.

## Introduction

*Refs. 1-3*

In the past few years UV curing has found a broad field of application in the area of coatings and inks for metal, paper, plastics, in the manufacture of printing plates, and adhesives. The many advantages of the UV curing technology compared to the conventional means has been the subject of many papers and needs no further comment.

In general, a system which is cured by UV light may consist of the following ingredients, where the first three are found in almost all formulations:

*UV formulation scheme*

- prepolymer (polymer)
- monomer
- photoinitiator

- coinitiator
  - reducing agent
  - chain transfer agent
  - spectral sensitiser
- light stabiliser
- thermal stabiliser
- colourants, plasticisers, and additives.

Large numbers of compounds have been patented as photoinitiators, but those which have obtained commercial significance belong mainly to the following classes:

- (a) benzoin ethers
- (b)  $\alpha$ -acyloxime esters
- (c) acetophenone derivatives
- (d) benzil ketals
- (e) ketone-amine combinations

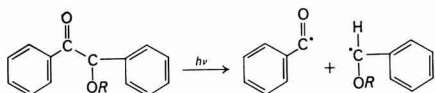
\*Paper presented at the Newcastle Section Symposium on "Ultraviolet curing" held at the University of Durham on 14-15 September 1977.

The first section of this paper will cover the photochemistry of these photoinitiators. In principle, the role of the photoinitiator is to form radicals by the absorption of UV light. The manner in which this is achieved has been the subject of numerous reviews<sup>1,2,3</sup>. This paper will concentrate on the reaction schemes established according to the reaction products detected by various analytical methods.

### Benzoin ethers

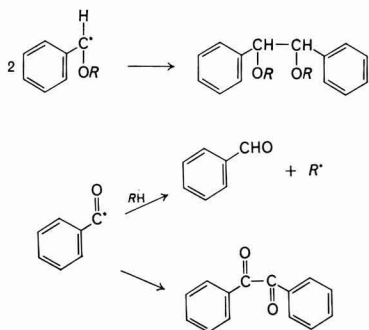
Refs. 4, 5, 11, 14

The photochemistry of benzoin ethers has been reported by Heine *et al.*<sup>4</sup> and Pappas *et al.*<sup>5</sup>. These types of photoinitiators dissociate from the excited triplet state to form free radicals according to Norrish type I cleavage.



From theoretical considerations it should be assumed that both radicals are of different reactivity. A recent study with <sup>14</sup>C labelled benzoin ethers by Pappas and co-workers gave evidence that the two radicals are equally effective in the polymerisation of acrylates and methacrylates<sup>14</sup>. On the other hand, evidence for the greater reactivity of the benzoyl radical with styrene has been presented<sup>11</sup>.

In addition to starting polymerisation, the two radicals may undergo dimerisation, H-abstraction or chain termination:

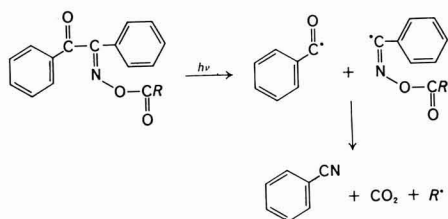


It was shown that the reactivity of benzoin derivatives as photoinitiators is strongly dependent on the substitution pattern of the carbon atom  $\alpha$  to the carbonyl group. Substituents capable of stabilising an adjacent positive charge are the most effective in accelerating  $\alpha$ -cleavage.

### $\alpha$ -Acylxime esters

Ref. 6

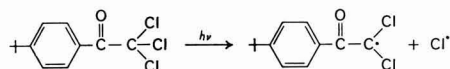
The photochemistry of  $\alpha$ -acyloxime esters as photoinitiators for acrylics and unsaturated polyesters was studied by Delzenne *et al.*<sup>6</sup>. A Norrish type I reaction leads to two radicals: A benzoyl radical and a second radical which undergoes the further cleavage:



### Acetophenone derivatives

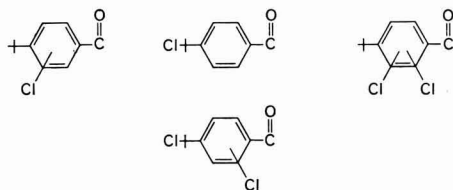
#### Chlorinated acetophenone derivatives

Commercial representatives of this class of compounds are substituted di- and trichloroacetophenones. The main reaction during irradiation is a  $\beta$ -cleavage as shown for *p*-tert-butyl-trichloroacetophenone.

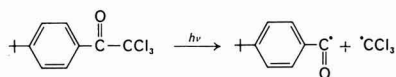


The chlorine radical so formed is highly reactive and is effective in initiating polymerisations. On the other hand, this radical may form hydrochloric acid by hydrogen abstraction from a hydrogen donor. Consequently this photoinitiator (PI) is also effective in acid catalysed vehicles.

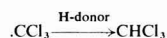
By mass spectrographic (MS) examination of the photolysis products of this compound it was possible to characterise the following benzoyl partial structures:



By chemically induced nuclear polarisation (CIDNP) measurements it was shown that chlorinated acetophenones are also cleaved to some extent by Norrish type I reaction:



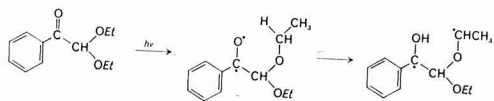
The trichloromethyl radical forms chloroform by hydrogen abstraction.



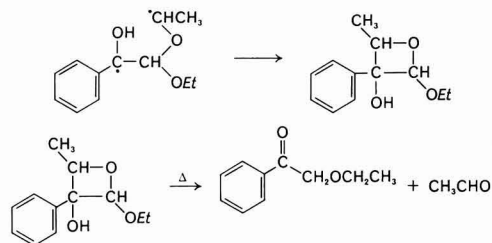
### Dialkoxyacetophenones

Refs. 7, 8

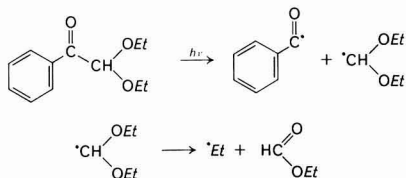
The photoproducts of 2,2-diethoxyacetophenone (DEAP) have been analysed by chemical means by Sandner and Osborn<sup>7,8</sup>. According to these experiments the first step is internal H-abstraction, a Norrish type II cleavage, yielding a biradical as the chain initiating species:



In the absence of reactive double bonds the biradical undergoes internal coupling to form an oxetanol intermediate which disproportionates thermally to acetaldehyde and  $\omega$ -ethoxyacetophenone.



The authors have studied the photochemistry of 2,2-diethoxyacetophenone in benzene solution by  $^{13}\text{C}$  and  $^1\text{H}$  chemically induced nuclear polarisation (CIDNP) experiments. According to these experiments there is evidence that in the photolysis of 2,2-diethoxyacetophenone a Norrish type I reaction also takes place:



Benzaldehyde, propiophenone and ethylene have been detected as possible reaction products between the benzoyl and the ethyl radical.

The CIDNP experiments show that the Norrish type I and Norrish type II pathways are of comparable importance. The H-NMR integrals of a typical reaction mixture at room temperature after relaxation of CIDNP leads to the following weight ratio:

Norrish I : Norrish II : oxetanole intermediate = 2 : 1 : 2

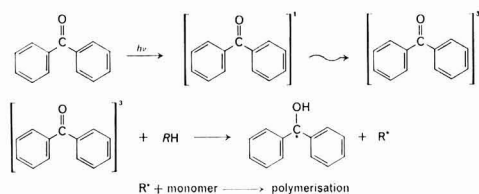
### Ketone/amine-combinations

Ref. 9

#### Photoreduction of ketones

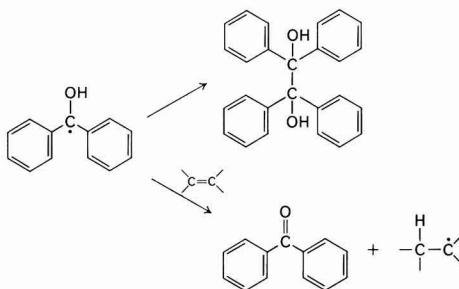
The photoreduction of benzophenone to benzpinacol in the presence of hydrogen donors is a well-studied reaction.

Triplet excited benzophenone abstracts hydrogen from hydrogen donors and the radicals so formed initiate polymerisation chains of acrylates<sup>9</sup>.



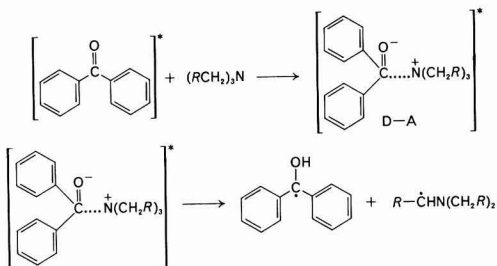
The ketyl radicals may undergo:

dimerisation to benzpinacol, initiation of polymerisation by H transfer, or chain termination by recombination with the growing radical species



#### Photoreduction of ketones via "exciplex"-formation

A special case of photoreduction of excited arylketones involves the use of amines as hydrogen donors. Amines have a high affinity to excited carbonyl groups forming donor/acceptor complexes, either by a charge transfer or an electron transfer process.



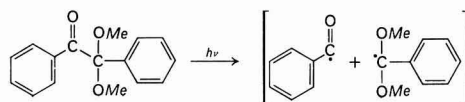
The donor acceptor complex collapses to form the amine derived radical which is an efficient polymerisation initiator. Another example for this mechanism is the exciplex formation of benzophenone/Michler's ketone, a well known system for the UV curing of printing inks.

### Benzilketals

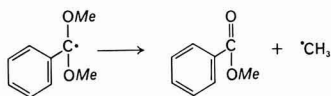
Ref. 10

To obtain an insight into the photochemistry of this class of compound the authors have studied the photoreaction of benzildimethylketal in solution by  $^{13}\text{C}$  and  $^1\text{H}$  chemically induced nuclear polarisation (CIDNP) experiments at different temperatures. Some photoproducts of these compounds have been analysed chemically by Sandner and Osborn<sup>10</sup>.

The primary reaction is a Norrish type I cleavage of the photoinitiator furnishing a benzoyl and a substituted benzyl radical:



The substituted benzyl-radical decays into the highly reactive methyl radical and the stable methyl-benzoate:

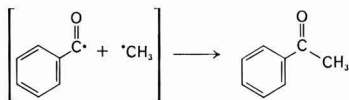


At 0°C no methyl benzoate could be detected by CIDNP, whilst the polarised signal of the recombination product (benzildimethylketal) could be seen.

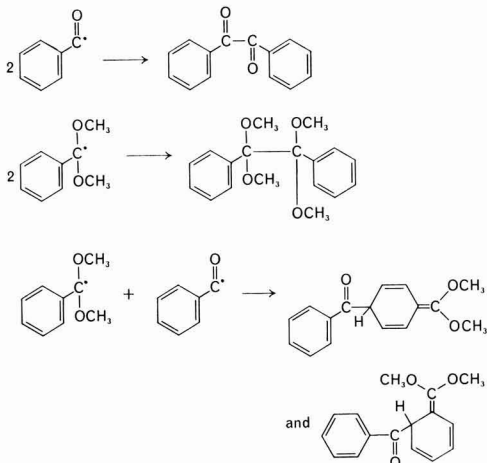
With increasing temperature the polarisation of the ester resonances increases and at room temperature methyl benzoate is the main product.

By CIDNP experiments with solutions of benzildimethylketal in benzene containing various amounts of monomers it could be shown that primary as well as secondary radicals formed during irradiation are effective in starting polymerisation. The future of the described radicals, besides starting polymerisation is expressed by the following schemes. Needless to say that these reactions might become important at low monomer concentrations or during light exposure of the finished article. All products mentioned have been detected by the CIDNP technique in benzene solution, as described above:

Acetophenone as a cage collapse product of the methyl- and benzoyl radical.

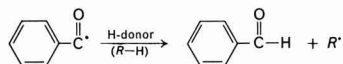


Two symmetrical dimers and two asymmetrical dimers of the benzoyl and substituted benzyl radical:

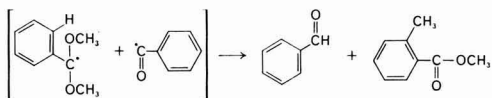


The formation of the asymmetrical dimers is a side reaction of the recombination reaction. The concentration of these products is very low.

The benzoyl radical additionally has the possibility of forming benzaldehyde by hydrogen abstraction as shown below:



The hydrogen donor (RH) may be the substrate, but it could be shown also that the substituted benzyl radical may function as a hydrogen donor. By the deuteration of the methyl groups and the solvent (benzene) it was found that the protons come from the aromatic ring:



Three different methyl substituted methyl benzoates could be detected by gas chromatography/mass spectroscopy (GC/MS) analysis of the reaction mixture. By CIDNP measurements it was shown that the orthomethyl compound is the main product.

The following section discusses the performance in various substrates of the photoinitiators (systems) described. Among the criteria which turned out to be of the greatest interest in the choice of a photoinitiator are cure efficiency and shelf-life. These properties in different UV-curing systems will be considered.

### Unsaturated polyester resins

Ref. 11

These vehicles were the first of commercial significance of all UV systems in the late 60's for wood coating<sup>11</sup>. Recently, they have obtained a new interesting outlet for the UV curing of GFR-articles.

The fact that styrene, or its homologues, are mainly used as cheap reactive thinners, determines the choice of the photoinitiator. Styrene is known to be a triplet quencher and this may explain why ketone/amine-combinations do not work with unsaturated polyester systems. The diffusion controlled photoreduction of the ketones may favour the energy transfer from the excited ketone to styrene.

Figure 1 shows how various photoinitiators perform in a high gloss unsaturated polyester resin containing a certain amount of allylic resin to overcome the air inhibition problem. Here the surface hardness as a measure of reactivity (pendu-

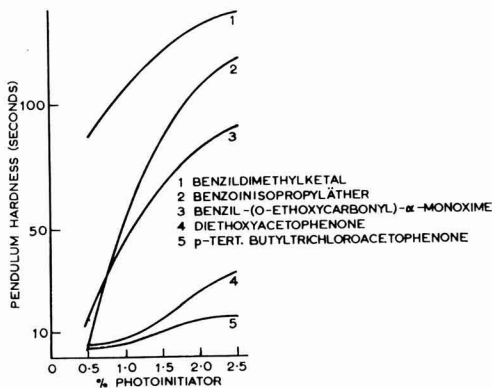


Fig. 1. Curing of unsaturated polyester resin

lum hardness method according to the German DIN standard 53.157) is plotted against the initiator concentration.

Obviously, benzildimethylketal shows the best performance of the photoinitiators used. Only this photoinitiator is able to cure the coating sufficiently at concentrations below 1 per cent in the cure cycle used.

The  $\alpha$ -acyloxime ester shows a similar performance to the benzoin ether, whereas the acetophenone derivatives slope away.

It is interesting to note that at high levels of photoinitiator, the hardness of the films even may decrease as shown in Figure 2.

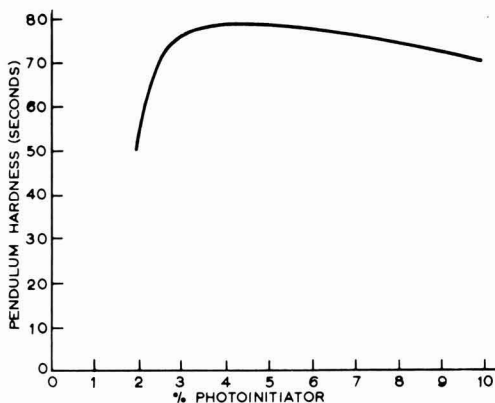


Fig. 2. Curing of an unsaturated polyester resin at various photoinitiator concentrations

Non-uniformity of light-absorption by the photoinitiator which leads to decreasing polymerisation rates going from the surface to the bottom of the film provides an explanation for this effect. Additionally, with increasing photoinitiator concentration primary radical termination increases leading to a shorter average chain length.

The efficiency of various photoinitiators in a particle board-filler based on 45 per cent of unsaturated polyester resins and 55 per cent of a mixture of  $\text{BaSO}_4/\text{talc}$  is given in Figure 3.

Obviously, the difference in reactivity between the benzoin ether and the  $\alpha$ -acyloxime ester is much more pronounced than in the clear system. The benzildimethylketal again provides, even at low concentrations, good hardness values, which favour the sandability of the particle board-filler after curing.

One of the most interesting aspects of UV technology is the possibility of manufacturing fully-formulated one-pack-systems, which are stable when kept in the absence of light.

Unfortunately, even this aspect turned out to be a limiting factor, since most of the photoinitiators used in the past decrease the shelf-life of the unsaturated polyester resin considerably. The following table shows that there are photoinitiators which do not show this difficulty. In a series of different polyester resins it was possible to confirm that the shelf-life of the resin is normally not affected by benzildimethylketal or 2,2-diethoxyacetophenone.

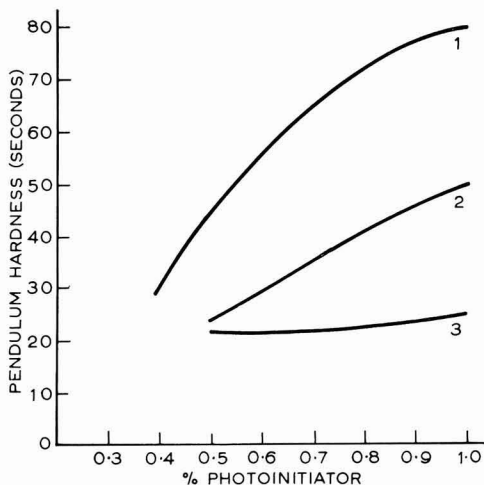


Fig. 3. Curing of a particle board filler using various photoinitiators:

1. Benzildimethylketal
2. Benzoinisopropylether
3. Benzil-(O-ethoxycarbonyl)- $\alpha$ -monoxime

Table 1

Shelf-life at 60° C of photoinitiators (2%) in an unsaturated polyester resin (60% in styrene)

Unsaturated polyester 60° C dark storage stability (days)	
benzildimethylketal	45
2,2-diethoxyacetophenone	45
<i>p</i> -tert.butyl-trichloroacetophenone	8
benzoinisopropylether	13
benzil-(o-ethoxycarbonyl)- $\alpha$ -monoxime	1

The reason why benzoin ethers affect the shelf-life, is found to be related to the activated hydrogen atom in the  $\alpha$ -position of the ether group. It is well-known that ethers of this structure easily react with oxygen, forming hydroperoxides. These intermediates may cause the thermal instability or some "cold curing" in the presence of transition metals which are usually present in filler materials. In earlier studies, it was found that the substitution pattern of the benzoin ethers also affects the shelf-life, short chain alkyl or non-branched ethers, such as the benzoinmethyl ether being the worst. A compromise between reactivity and shelf-life was, therefore, found to be benzoin isopropyl ether.

In principle, it is of course possible to improve the shelf-life of benzoin ethers by adding thermal stabilisers or radical scavengers such as hydroquinone, benzoquinone etc. The disadvantage of this procedure is that the cure efficiency may be also affected. Another possibility to improve the shelf-life of benzoin ethers is the substitution of the hydrogen atom by taking advantage of its acetic character.

#### Acrylates

The introduction of the acrylates has opened many new areas for the UV-technology. New coatings for metal, paper, plastics and wood with outstanding properties are now available. The main advantage over the unsaturated polyester resins, which made this development possible were higher cure rates and tailor-made resins from a variety of different monomers and prepolymers.

On the other hand, due to their higher cure rates, the difference between through cure and surface cure caused by oxygen inhibition in acrylate based formulations became substantial. In order to overcome this detrimental effect of oxygen the photoinitiator concentration and consequently the radical density had to be increased by a factor between 2-3, if the advantage of higher cure rate is not to be, at least partially, lost.

Two other possibilities for solving the air inhibition problem exist; elimination of oxygen by blanketing with inert gas and the use of co-initiators which accelerate the surface cure rate.

The inert blanketing technique has been limited to some extent by the cost of nitrogen which is used in a large quantity.

Amines as co-initiators, on the other hand, played an interesting role in the UV curing in an air atmosphere. The combination of benzophenone with tertiary aliphatic amines did not show the decrease in surface cure rate like other photoinitiators. Unfortunately, this combination showed some disadvantages in curing bulk coatings as shown in Table 2.

Table 2

Cure efficiency using 3 phr photoinitiator with 5 phr aliphatic amine

photoinitiator	pendulum hardness <sup>A</sup> (sec)	residual double by <sup>B</sup> (%)
benzildimethylketal	59	23
2,2-diethoxyacetophenone	31	40
benzophenone	20	64

Formulation A: based on polyester acrylate, 40 m/min/lamp

Formulation B: based on urethane acrylate, 50 m/min/lamp

Obviously, there is a good correlation between pendulum hardness and residual double bond content as determined by infrared spectroscopy. The lower cure efficiency using the benzophenone-amine combination results in a lower hardness of the film or a correspondingly higher content of residual double bonds.

The correlation between surface cure and body cure using various photoinitiators is demonstrated in a urethane-acrylate based formulation.

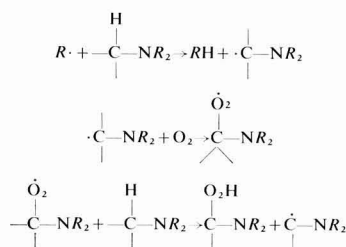
Table 3

Cure rates in a urethane-acrylate-based system

photoinitiator (5%)	surface cure rate in m/min/lamp	pendulum hardness in seconds at 10 m/min/lamp
benzildimethylketal	50	182
2,2-diethoxyacetophenone	30	170
<i>p</i> -tert. butyltrichloroacetophenone	30	166
benzoin isopropylether	20	181
benzil-(O-ethoxycarbonyl)- $\alpha$ -monoxime	30	155
benzophenone/ <i>N</i> -methyl-diethanol-amine	80	71

Obviously, the benzophenone/amine-combination performs best in curing the surface of the coating, whereas the body cure falls away. It should be mentioned that the use of aliphatic amines generally lowers the hardness of the film. Possibly the reason for this is a plasticising effect or their function as chain transfer agents. The way in which amines

are effective in suppressing oxygen inhibition is described by the following equations:



Thus, the role of amines could be understood as a reduction of dissolved oxygen by a radical chain process. In principle all photoinitiators can be combined with amines, but of practical interest so far is mainly the combination with aromatic ketones, benzilketals, and dialkoxyacetophenones. This is for the reason that the combination of amines with the other (photo-fragmentation) products results either in a poor shelf-life, as described later, or in a retarding effect which is found in the case of chlorinated acetophenone derivatives. As may be seen from Table 4, the addition of amine may accelerate or decrease the surface cure rate of acrylate based formulations considerably.

Table 4

Effect of amine addition on the surface cure rate in a urethane-acrylate based formulation

photoinitiator/coinitiator (3 phr) (5 phr)	surface cure rate (m/min/lamp)
benzildimethylketal	30
benzildimethylketal/amine	80
2,2-diethoxyacetophenone	20
2,2-diethoxyacetophenone/amine	50
<i>p</i> -tert. butyltrichloroacetophenone	20
<i>p</i> -tert. butyltrichloroacetophenone/amine	< 10

It should be mentioned that aliphatic amines may offer some problems in acrylate formulations due to emulsification of the vehicle in wet offset printing and exudation.

Therefore, a lot of effort was made to incorporate them by modifying resins with amines or ammonia, or by using amines with acrylate functionality.

Chlorinated aliphatic or aromatic compounds as co-initiators have been covered by several patents. It is well-known that these compounds are sensitive to UV irradiation and may also function as chain transfer agents. The use of these compounds is also somewhat limited by the risk of hydrochloric acid formation which may cause corrosion problems.

Table 5

Shelf-life of photoinitiators in an acrylate based coating

photoinitiator (2%)	60' shelf-life in days
benzildimethylketal	> 180
2,2-diethoxyacetophenone	> 180
<i>p</i> -tert. butyltrichloroacetophenone	27
benzoinisopropylether	2
benzil-(O-ethoxycarbonyl)- $\alpha$ -monoxime	1
benzophenone/ <i>N</i> -methyl-diethanolamine	1

Shelf-life is, of course, also one of the main criteria for photoinitiators in acrylate based formulations. Table 5



shows the shelf-life of a urethane-acrylate based coating containing different photoinitiators, which correlates well with the results obtained in unsaturated polyester resins.

The poor shelf-life of the benzophenone-amine combination is caused by the tertiary aliphatic amine used. It is well-known that amines may decrease the shelf-life of acrylate based formulations considerably, depending upon the level used. The poor shelf-life of the acyloxime ester is caused by thermal instability of the material. A combination of acyloxime ester with aliphatic amines leads to an exothermic decomposition-reaction which starts even at ambient temperature and causes early gelation of the acrylate system. Amines are also known to accelerate the decomposition of peroxides and, therefore, the pot-life of formulations containing benzoin ethers is further reduced in the presence of amines.

### Pigmented acrylate systems

Refs. 12, 13

UV curing of pigmented systems has always been a challenge to workers in this field. Pigments with different absorption characteristics in the near UV range may affect the cure rate to a large extent. In spite of this, even white and black inks are now UV cured.

Figure 4 shows the performance of the photoinitiator benzildimethylketal in a yellow and cyan offset printing ink, containing 15 and 20 per cent of organic pigment respectively. The inks were applied on a laboratory printing machine, UV cured and examined as described elsewhere<sup>12</sup>.

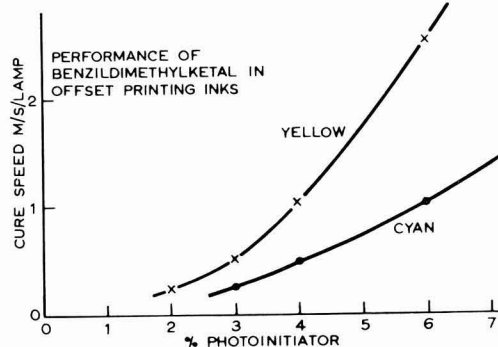


Fig. 4. Curing of an offset printing ink

The absorption of the pigments in the near UV range between 300 and 400 nm may explain the difference in reactivity. The performance of various photoinitiators as found using the same technique is given in Table 6.

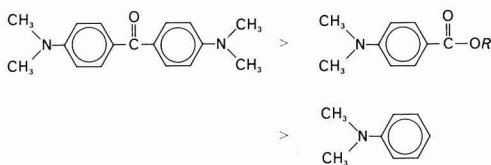
Table 6  
Performance of various photoinitiators in a cyan offset printing ink

photoinitiator (6%)	cure speed (m/s/lamp)
benzildimethylketal	1.0
2,2-diethoxyacetophenone	0.5
<i>p</i> -tert. butyl-trichloroacetophenone	0.5
benzil-( <i>o</i> -ethoxycarbonyl)- $\alpha$ -monoxime	0.5
benzoin isopropylether	0.25
benzophenone/Michler's ketone (4/2)	0.75
benzophenone/Michler's ketone (5/1)	1.0

With two strongly absorbing bands at about 250 and 370 nm ( $\epsilon$  values of about 15,000 and 30,000 respectively) Michler's

ketone has proved to be particularly effective in combination with benzophenone for the UV curing of printing inks. Of course, the colour formation which is observed using the system, restricts its use in clear and white pigmented coatings. For several reasons there is an interest in replacing Michler's ketone in printing inks. By tightening the molecule the efficiency of the corresponding amine in combination with benzophenone unfortunately becomes worse.

The following order of decreasing reactivity is observed:



In order to improve the performance of photoinitiators which undergo photo-fragmentation, combinations with more strongly absorbing aromatic ketones and aromatic amines were examined. Figure 5 shows an interesting result to be found by using a combination of benzildimethylketal, methylthioxanthone and the ethyl ester of the *p*-dimethylamino-benzoic acid.

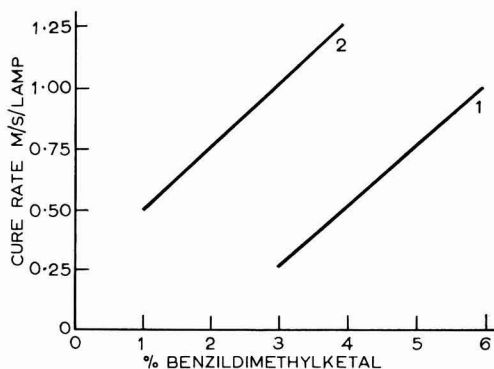


Fig. 5. Curing of an offset printing ink

1. benzildimethylketal
2. benzildimethylketal/methyl-thioxanthone (0.5%)/DMABE (2%)

By the addition of small amounts of the strongly absorbing methylthioxanthone, the efficiency of benzildimethylketal in the given (cyan) printing ink could be enhanced considerably. It should be mentioned that the combination of methylthioxanthone with DMABE (0.5/2) did not even work at 0.25 m/sec/lamp.

White pigmented inks have offered serious problems in UV curing due to the high levels of pigment needed in order to obtain good hiding power. Unfortunately, anatase type titanium dioxide, in spite of its better UV transmittance compared to rutile, has not become of commercial significance for this outlet due to rheological problems and its poorer hiding power<sup>13</sup>. The main class of photoinitiators used in this area so far are the thioxanthone/amine-combinations. Their good cure efficiency is demonstrated by Figure 6, showing the amount of volatiles (per cent) as a function of the exposure time (number of passes).

The efficiency of the thioxanthone/amine-combination is even further improved if iron iodide doped lamps are used, which is shown in Figure 7.

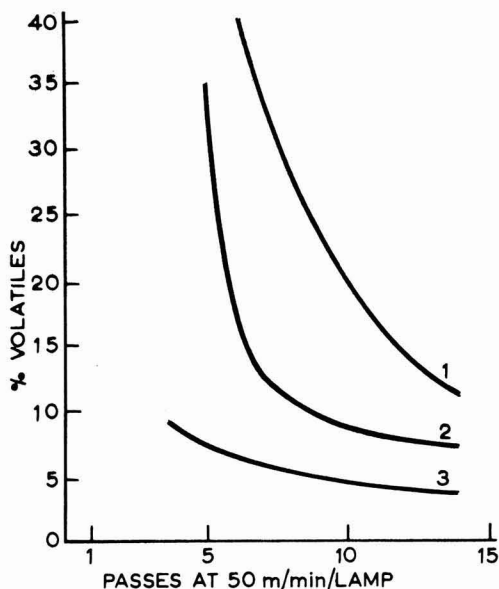


Fig. 6. Curing of a white basecoat containing 2% of aliphatic amine, with:

1. Ethyl-anthraquinone (4%)
2. Benzil (2%)
3. Chloro-thioxanthone (0.5%)

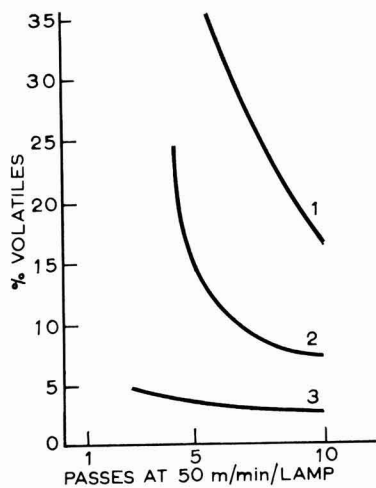


Fig. 7. See Fig. 6, but doped lamp used

Unfortunately thioxanthone/amine-combinations have a tendency to slight discolouration. Therefore, their usage has to be limited to low levels (below 0.5 per cent). Consequently, the surface cure rate is retarded.

By a combination with benzildimethylketal the surface cure rate of these systems can be enhanced as shown in Table 7.

Table 7

Combinations of benzildimethylketal with 2-chlorothioxanthone/amine in a white base coat for an exterior

benzildimethylketal (%)	2-chlorothioxanthone (%)	passes required for tack free surface
—	0.5	3
2	0.5	2
—	0.4	4
2	0.4	3
—	0.2	7
2	0.2	4

#### Light stabilisation in UV cured systems

Light stabilisers in UV cured systems seem to be a contradiction in terms, since the photoinitiator needs UV light for its performance and the object of the light stabiliser is to protect polymers against the negative influence of UV light. Consequently, using conventional UV absorbers it was found that the cure efficiency may be affected as indicated in Table 8.

Table 8

Cure efficiency in the presence of a UV absorber 300  $\mu$  films, benzotriazole type UV-absorber

% UV-absorber	exposure time (sec)	pendulum hardness	
		top side	under side
none	4.5	163	148
0.1	4.5	117	30
0.1	9.0	169	131

The films have been irradiated only on one side which resulted in big differences of the hardness values at the same cure cycle. On the other hand, by doubling the exposure time it was possible to obtain a well cured film on the reverse side.

In thicker films, like 2 mm unsaturated polyester plates, the difference in cure efficiency was even more pronounced.

Table 9

Cure efficiency in the presence of a UV-absorber (benzotriazole type)

% UV absorber	exposure time* (sec)	residual styrene content** (%)
none	6	0.9
0.1	18	8.2
0.1	24	3.2
0.2	48	3.3
—	cold cured	2.3

\* From both sides

\*\* According to DIN 16 945

The conclusion from these tests is that, depending on the film thickness used, it is possible to use conventional UV absorbers at low levels for the stabilisation of UV cured coatings. Retardation of cure rate and the risk of "overcuring" the surfaces has to be considered, which may provide weak links in the light stability of the binder.

UV curing in the authors' opinion opens a new interesting outlet for a new class of light stabilisers—the hindered amines. These light stabilisers do not function by the absorption of UV light as do conventional UV absorbers and consequently do not affect the UV curing.

The performance of these light stabilisers in stabilising a clear coating based on an aromatic urethane-acrylate prepolymer is presented in Figure 8.

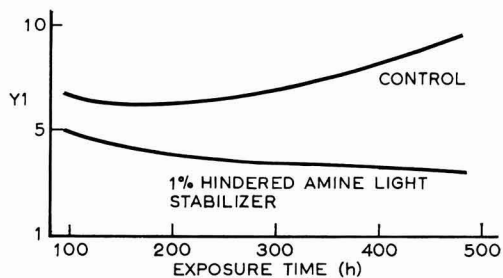


Fig. 8

The initial discolouration of the coating is due to a reversible yellowing caused by the photoinitiator used. This discolouration disappears gradually during further exposure. The sample which does not contain the light stabiliser shows a further increase in the yellowness index caused by the degradation of the polymer.

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# Present status of ultraviolet curable coatings technology in the United States\*

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## Summary

The different oligomers currently used in ultraviolet curable coatings and inks are emphasised as specially designed acrylated urethane oligomers. Acrylated monomers, both monofunctional and polyfunctional are discussed as a function of their respective disadvantages. Problems and advantages of several photoinitiators are

analysed and correlated with curing in air or in nitrogen blanketing. The toxicological aspects and recommendations for safe handling of UV curable materials are highlighted. Finally a review is given of different market segments where ultraviolet curing technology is applied successfully.

## Keywords

### Raw materials

raw materials used in manufacture or synthesis of ingredients for coatings

oligomers  
monomers

### solvents

diluent solvent

Properties, characteristics and conditions primarily associated with materials in general

cross linking  
photoinitiation

Processes and methods primarily associated with drying or curing of coatings

ultraviolet curing

Equipment primarily associated with drying or curing of coatings

ultraviolet curing

## L'état actuel aux Etats Unis de la technologie de revêtements durcissables par les rayons ultra-violetes

### Résumé

On mentionne les divers oligomères actuellement utilisés en revêtements et encres durcissables par les rayons ultra-violetes, et l'on souligne les oligomères uréthane/acrylés spécialement étudiés. On discute, en fonction de leurs inconvénients particuliers, les monomères acrylés et mono et polyfonctionnels. On examine les problèmes et les avantages de plusieurs photo-amorceurs et les met en rapport avec le durcissement à l'air ou dans un atmosphère d'azote. On met

relief les aspects technologiques des substances susceptibles à être durcies par les rayons ultra-violetes, ainsi que les précautions nécessaires à assurer la sécurité des opérations de manutention. Enfin, on donne une revue des divers secteurs du marché où l'application de la technologie de durcissement par les rayons ultra-violetes connaît un grand succès.

## Der gegenwärtige Stand der Ultraviolet-Härtungstechnologie für Anstrichmittel in den Vereinigten Staaten

### Zusammenfassung

Die zur Zeit laufend in durch Ultraviolet-Bestrahlung härtbaren Beschichtungsmittel und Druckfarben werden aufgeführt unter Betonung von speziell entwickelten Akrylester-Urethanoligomeren. Akrylestermonomere, sowohl monofunktionelle als auch polyfunktionelle werden hinsichtlich ihrer Nachteile besprochen. Probleme und Vorteile verschiedener Photoinitiatoren werden untersucht und mit Härtung an der Luft oder unter einem Stick-

stoffschleier in Beziehung gebracht. Die toxicologischen Gesichtspunkte werden betont, und insbesondere Vorschläge für vorsichtiges Arbeiten mit UV härtbaren Materialien gemacht. Schliesslich wird eine Übersicht über die verschiedenen Absatzgebiete gegeben, in welchen die Ultraviolet-Härtungstechnologie mit Erfolg angewandt wird.

## Introduction

The fact that the Newcastle Section of the Oil and Colour Chemists' Association justified the organisation within two years of a second Symposium on Ultraviolet Polymerisation in the Coatings Industry, indicates the growing worldwide interest of coatings and ink chemists in this relatively new technology.

Three to four years ago the industrial coatings industry greatly accelerated its programmes regarding air pollution, occupational safety and health hazards in response to legislative actions by key administrative agencies. At the same time, concern over energy shortages and energy cost in-

creases came to the forefront. The industry reacted to these problems by introducing new coating technologies, such as high solids, waterborne, powder and radiation curable coatings. Technology forecasts at that time predicted future market share figures for each of these new types of coatings for several industrial coating market segments. Most of these were over optimistic and some indicated that the conventional industrial coatings were doomed to disappear. The fact that these changes did not happen at the expected rate can be attributed to several factors. The raw material shortages at the end of 1973 and during 1974, combined with the severe economic recession during 1975 and part of 1976 is one factor. Another factor is that the technology was not advanced sufficiently to allow formulators and applicators to switch to

\*Paper presented at the Newcastle Section Symposium on "Ultraviolet curing" held at the University of Durham on 14-15 September 1977.

these new technologies without a sacrifice in product quality. Still another factor is related to human nature and resistance to change even though it may be for the best. Nevertheless, these new technologies are replacing, although at a much slower rate than initially predicted, conventional, organic solvent based coatings. All of the earlier mentioned new coatings technologies are in competition with each other to create their own share of specific market segments.

Radiation curable coatings, or more specifically, coatings curable by either ultraviolet or electron beam radiation, are establishing themselves extremely well in specific industrial coatings and ink market segments. Some of the major reasons for this change include the introduction of new, better designed raw materials and the improved technical skills of the formulators in overcoming most of the initial performance problems.

## Oligomers

Initially, especially in Europe but also in the USA, ultraviolet curing penetrated the industrial coatings market, and more specifically the particle board wood market, with polyester/styrene based fillers and finishes. Although this type of resin is used on a high tonnage basis, its use is still restricted to wood. The major shortcomings of the polyester/styrene system are its relatively slow cure speed, the high volatility of the styrene, its yellowing tendency and problems related to the sanding operation.

Over the last few years several other types of oligomers have made their appearance in the market place. Most of these contained an acrylate functionality, since this type of unsaturation provides the highest response to actinic light compared to methacrylate, vinyl or allyl functionalities. Within the group of acrylate modified oligomers distinct classes emerged:

- acrylated polyesters
- acrylated epoxies
- acrylated oils
- acrylated urethanes

All of these have several representatives in the marketplace and find uses in many market segments, due to specific structure related properties. For example, acrylated oil type oligomers, such as *Actomer X80*, (Union Carbide Corporation), are widely used in the lithographic printing area due to their excellent pigment wetting power, well balanced hydrophobic-hydrophilic behaviour, as required to assure processability on the press with regard to the fountain solution, and their relatively low toxicity. Combined with an acrylated epoxy resin, to provide improved hardness, smear resistance and reactivity, high quality lithographic and screenprinting inks have been developed.

Acrylated urethanes, as a class, provide the well known properties specific to conventional urethane coatings, such as high abrasion and chemical resistance together with an excellent balance of flexibility and hardness. However, as in the case of the conventional urethane coatings, the chemical design of the polymer will dictate the ultimate performance and balance of properties. Acrylated urethanes, as described in the US Patent 3,700,643 allow the design of such oligomers (see Table 1). Coatings based on these oligomers have been obtained with tensile strength over  $250 \text{ Kgcm}^{-2}$  and an elongation at break over 100 per cent combined with excellent abrasion resistance. This combination of properties has made

these types of acrylated urethanes the preferred candidates for a variety of applications such as high quality wood coatings, floor coatings and overprint varnishes for metal and paper.

Table 1  
Acrylated urethane oligomers  
U.S. Patent 3,700,643—Union Carbide Corp.

<i>Ucar Actomer X-116</i> —general use	
<i>Ucar Actomer X-117</i> —high abrasion resistance	
<i>Ucar Actomer X-118</i> —high flexibility	
<i>Ucar Actomer X-119</i> —balanced flexibility, toughness and abrasion resistance	
Unmodified <i>Ucar Actomer X-118</i> properties:	
Tensile strength, psi	3373
Elongation, percent	142
Sward hardness, sec.	12
Acetone resistance	139
Reverse impact, inch-lbs.	> 165

As new improved oligomers become readily available, it can be predicted that these types of acrylated urethane resins will allow UV coatings to penetrate a greater percentage of the industrial coating market. Ideally, these oligomers should have (1) minimum solution viscosities at increased molecular weight, so as to decrease the amount of diluent used; (2) decreased volume shrinkage during cure so as to minimise residual stress; (3) adequate functionality and related activity to decrease the residual unsaturation in "cured" films whilst increasing the degree of polymerisation during cure. These requirements, along with development of improved photoinitiators and curing equipment will help to meet the needs of many application areas.

## Diluents

As all currently available oligomers are too viscous to be applied on conventional coating equipment, most formulators now dilute the oligomers down to application viscosity (from 10,000 cps to 100 cps depending on the method of application). For this the formulator has a broad choice of reactive products at his disposal, although several have disadvantages which make their use limited. A review of several diluents allows the following conclusions:

1. *Butyl acrylate*: Although a good viscosity reducer, this material is very volatile. It finds some use in the wood coating area.
2. *Isobutyl acrylate*: This is also very volatile, but has found some use.
3. *2-Ethylhexyl acrylate*: Most widely used diluent at present; however its volatility is a drawback.
4. *Isodecyl acrylate*: This material is a good viscosity reducer, less volatile than 2-ethylhexyl acrylate, although still doubtful in some applications. It shows promise as a preferred diluent in many applications.
5. *Phenoxyethyl acrylate*: An excellent viscosity reducer and one of the least volatile monomers presently available for development.
6. *Tetrahydrofurfuryl acrylate*: Excellent viscosity reducer, but has a strong, lingering odour and reduces the shelf life of coating systems.
7. *Isobornyl acrylate*: Although it is used, it has a strong odour.

8. *2-Hydroxyethyl acrylate*: Although it is widely used at present, its high toxicity has forced people to formulate around this material.
9. *N-Vinyl pyrrolidone*: Excellent viscosity reducer, combined with reported low toxicity. Although it does not contain acrylate functionality it copolymerises with acrylates when used at the proper mole ratio. It also is reported to improve flexibility of the cured film. (See Table 2).

Table 2  
*N-Vinyl pyrrolidone*



<i>Ucar</i> Actomer X-117	90	90	90	—	—	—
<i>Ucar</i> Actomer X-119	—	—	—	95	95	95
1,6 Hexane diol diacrylate	10	—	—	5	—	—
Trimethylol propane triacrylate	—	10	—	—	5	—
<i>N-Vinyl pyrrolidone</i>	—	—	10	—	—	5
Sward hardness	10	20	16	22	26	26
% Elongation	50	30	87	37	29	53
Tensile strength, psi	3010	3900	3700	3890	3895	4400

## Crosslinkers

Crosslinkers are multifunctional acrylates which impart solvent resistance, stain resistance, and hardness to the cured film. The crosslinker type and the amount used has a definite influence on curing response and elongation of the resulting cured film. A large number of crosslinkers are available to the formulator. The following crosslinkers are widely used:

- 1,4-Butanediol diacrylate is widely used in the wood coating area.
- 1,6-Hexanediol diacrylate, with its lower volatility, is increasingly replacing neopentyl glycol diacrylate, although both are suspected skin irritation sensitizers. They are relatively good viscosity reducers.
- Pentaerythritol triacrylate is widely used in the printing area since it gives rapid cure response. However, the product is a severe eye irritant.
- Trimethylolpropane triacrylate has low volatility and is attractive in the ink area.

The decision to use one specific diluent and crosslinker over another should be considered carefully since this will influence the volatility, toxicity and viscosity of the wet coating and the physical properties of the cured film.

## Photoinitiators

Ref. 1

The commercial or development materials currently available can be divided into the following classes:

- Benzoin ethers
- Thioxanones and derivatives, such as 2-chlorothioxanone (2CTX)
- Benzophenone and derivatives, such as Michler's Ketone
- Derivatives of acetophenone, such as diethoxyacetophenone (DEAP).

Reviewing these classes of photoinitiators allows comment on some deficiencies encountered when using them. Benzoin ethers, when considered as a group, have low photoinitiator efficiency and they impart poor shelf life to most formulated coatings as compared to acetophenone derivative-type photoinitiators, such as DEAP and dimethoxyphenylacetophenone (benzil dimethylketal) (Table 3). Clearly, DEAP is more efficient than the benzoin ethers at significantly lower concentration<sup>1</sup>.

Table 3  
*Unreacted monomer remaining in film*

Initiator	(wt %)	Exposure, sec (fpm)	2.(150)	1.(300)
$\alpha$ , $\alpha$ -Diethoxyacetophenone:	(2%)	0%	2.0	—
	(1%)	0	2.2	—
	(0.5%)	1.9	15.5	—
Benzoin, isobutyl ether	(2%)	5.7	25.2	—
	(1%)	8.1	31.5	—
	(0.5%)	16.2	39.1	—
Films cured with inerted 100 Watts/inch mercury vapor lamps (flux 500 Watts/ft <sup>2</sup> )				
Coating: Oligomer		40	—	—
Monomer		60	—	—
Photoinitiator		as indicated	—	—

With only a few exceptions, all the commercial photoinitiators are solid and crystalline. This makes them difficult to incorporate into coating formulations, since generally the solubility of these photoinitiators in acrylate diluents and crosslinkers is low. Furthermore, these photoinitiators tend to crystallise from the wet coating during storage, which results in a reduction in curing efficiency and in film defects. Therefore a liquid photoinitiator, such as DEAP, is preferred.

Another problem encountered with several photoinitiators, such as with Michler's ketone and presently commercially available thioxanone types, is the fact that they impart extensive yellowing to the cured film. Although no photoinitiator is completely free from this shortcoming, most acetophenone types, and DEAP in particular, are best in this respect.

## Nitrogen blanketing

Refs. 2, 3

The polymerisation of acrylate monomers is highly inhibited by oxygen. Essentially, there are two means to overcome this problem. The first way is to alter the photoinitiator system. For instance, the addition of a tertiary alkanolamine, such as methyl-diethanolamine, to an arylketone type photoinitiator, such as benzophenone, helps overcome oxygen inhibition. (See Figure 1). Particularly interesting for air curing acrylate type systems is a mixture of two parts benzophenone and three parts *n*-methyl-diethanol amine<sup>2</sup>. The second way, and certainly the most efficient way, is to remove the oxygen from the curing environment by nitrogen blanketing. The Linde Division of Union Carbide has developed equipment which allows efficient nitrogen blanketing at extremely low nitrogen consumption. Calculations<sup>3</sup>, taking into account all involved factors, such as energy input cost, lamp service cost, water-cooling cost, nitrogen cost and photoinitiator cost, indicate that the real cost of running a production line with nitrogen blanketing, when using curing equipment such as manufactured by the Linde Division of Union Carbide Corp., represents a negligible additional cost factor to the operation. To apply one gallon of a floor tile coating, which sells at \$28 to \$33/gal., the additional cost to operate under nitrogen blanketing is 5 to 8 cents (less than 0.28 per cent). This insignificant

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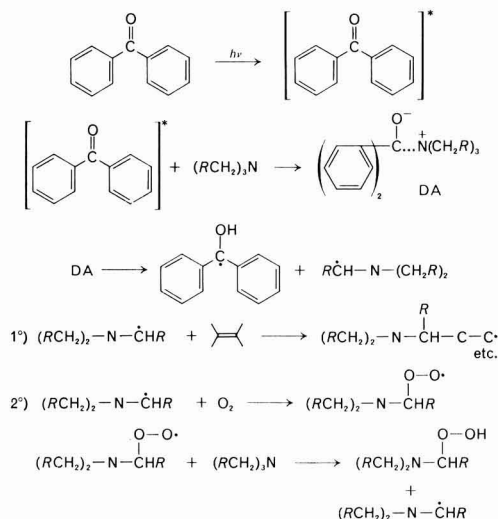
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**Fig. 1. Postulated reaction of the amine-derived radical functioning as oxygen trap**

By Dr. C. L. Osborn

Ref: U.S. Patent 3,759,807 to Union Carbide Corp.

cost increase is more than compensated by improved product performance such as hardness, gloss and abrasion resistance. It also increases the formulation latitude of the coating system. Therefore, in the US the use of nitrogen blanketing is widespread and still gaining in popularity.

## Curing equipment

Refs. 4, 5

Based on the mathematical derived equation that the exposure time per unit of volume cured is inversely proportional to the square root of the UV flux, the idea of process reciprocity has been developed<sup>4</sup>. The exposure time and flux may be reciprocally interchanged to maintain a constant volume cured. Furthermore, efficiency of the UV process (productivity of process divided by power requirement) has been defined to be inversely proportional to the square root of the flux. All these considerations have led to a new type of curing equipment using the "expanded flux" principle. This type of equipment is known as the "Linde PS 2800" Photocure system<sup>5</sup>. This concept provides more control of the process, highly efficient energy utilisation and low process temperatures. The latter is most significant, in that processing of very delicate, heat sensitive materials such as vinyl and other plastic films and sheets, can now be achieved without destructive alteration of the substrate.

## Toxicology and safety

A major concern of everyone involved with ultraviolet curable acrylate type coatings is how to avoid the hazards associated with skin absorption, inhalation, eye irritation and most of all sensitisation. It is the task of all responsible persons, at all levels, from raw material supplier to applicator, to inform and educate their subordinates and to take preventive measures in order to avoid accidents.

Toxicology studies on UV curable coatings and intermediates are performed in toxicology laboratories supported by Union Carbide Corp. at Carnegie Mellon Institute. Results have been published together with handling practice recommendations<sup>6</sup>.

As a result of a greater awareness of the associated hazards with some acrylates coupled with production experiences, several companies have made up a list of products which they do not tolerate in UV coatings, inks, and adhesives. Such products are generally selected on the basis of high overall toxicity rating or high volatility. Photocurable coatings, in liquid form based on acrylate chemistry, cause skin irritation, are toxic if ingested in sufficient quantities and are irritating to the eyes. Vapour forms of photocurable coatings will cause irritation to the respiratory system. These effects can be controlled by taking precautions related to good housekeeping and safe handling practices. For example:

- adequate labelling, showing the hazardous properties of the chemicals.
- adequate ventilation in the work area, with adequate face velocity at the hood combined with adequate air volume transport at the hood.
- wear proper protective clothing:
  - a. black neoprene gloves, such as Edmont Wilson black neoprene glove (290865)
  - b. chemical worker neoprene aprons
  - c. black rubber boots should be worn to clean up spills.
- apply barrier cream on the hands, after washing them with water and soap, but before putting on the gloves.
- use only isopropanol as cleaning solvent, avoid ketones, which penetrate the gloves.
- wear standard safety goggles or glasses at all times.
- clean up all spills immediately.

By following these common sense measures, the hazards in using acrylate-type photocurable coatings can be reduced to a level consistent with some conventional solvent-based industrial coatings.

## Markets for UV coatings

Because ultraviolet curing contributes to air pollution control, conserves energy, improves cure speed and throughput, and reduces capital costs by space saving and economical equipment purchase, it has become the preferred technology in many industrial coating, ink, and adhesives applications. Taking into account all these advantages, it is the author's opinion that two decisive factors have made ultraviolet curing a success in the USA: (a) energy conservation—regardless of energy cost and (b) the ability to coat heat sensitive substrates with less risk of substrate degradation. People have introduced ultraviolet curing units in their production lines and are still doing so, to meet market required production output with their allocated natural gas volume. Companies coating or printing heat sensitive substrates such as PVC flooring goods, PVC foil, paper and carton, polyester foil, etc. are those who most utilise UV technology.

Included in detailed analyses of market segments where ultraviolet curable coatings are applied commercially at present, the following can be listed:

- (1) PVC—asbestos tile, where added coating value is highly compensated by sales price, due to improved quality. (See Table 4).

Table 4  
Resilient flooring

Vinyl asbestos tile	
Historically:	not coated: waxing
	Retail selling price: \$0.30/ft <sup>2</sup>
U.V. curing:	Add-on value: \$0.08/ft <sup>2</sup>
	Retail selling price: \$0.80-1.20/ft <sup>2</sup>
	Sales argument: no wax-gloss
	Investment (coating-curing) \$200 m
	Output: 50 m ft <sup>2</sup> /day
Risk:	Consumer acceptance
Status:	7 lines U.V. converted out of 30 existing lines

- (2) PVC rollgoods for flooring, where improved quality is obtained at higher output by UV curing technology as compared to conventional coatings.
- (3) PVC wood veneer imitation foil: where UV coating technology allows reduction in foil thickness, with improved surface quality.
- (4) PVC wall covering.
- (5) Record sleeves: besides the printing operation, the replacement of the "Cellophane" or "Mylar" laminate by UV overprint lacquer has been successful due to production cost reductions.
- (6) Screen printing on paper and glass, where UV technology brought better quality and a decrease of overall production cost due to the possibility of using lower quality paper and decreased scrap rates.
- (7) Lithographic printing on paper and metal, where reduced space requirements made UV acceptable on reduced capital cost and energy consumption savings.
- (8) Bottle caps and closures and metal container overprint varnishes: UV technology has been favoured in these applications on energy saving considerations.

- (9) Printing Plates: UV has been innovated in this area due to cost consideration.

### Epoxy—UV Curing

Refs. 7, 8

This presentation on ultraviolet curing may be closed by mentioning a relatively new technology based on the cationic polymerisation of liquid cycloaliphatic epoxies, such as *ERL4221* and *ERL4289* from Union Carbide Corporation with catalysts such as substituted aryl diazonium salts described in the patent literature<sup>7</sup> or other types of catalysts known as "epoxy curatives"<sup>8</sup>. The catalyst, under influence of UV light, liberates a Lewis acid which polymerises the cycloaliphatic epoxy *via* oxirane ring opening.

This type of technology may find important applications in the general class of ultraviolet curable coatings and inks.

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# The design and construction of ultraviolet lamp systems for the curing of coatings and inks\*

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## Summary

There are now over 400 installations running using a U.V. drying process in Europe at present, and world wide the total figures must now run to several thousand.

The demand on the equipment manufacturer for higher efficiency, and an ever widening field of applications effecting the size and shape of the equipment, has tested the suppliers ability to meet the rapidly expanding and changing requirements.

There is a danger that new ideas are rapidly overtaken by events.

## Keywords

*Processes and methods primarily associated with manufacturing or synthesis*

ultraviolet curing

*Miscellaneous terms*

ultraviolet light

In this atmosphere the equipment manufacturer spends much of his time redesigning and modifying the mechanical features of existing equipment to allow them to be fitted into alternative site situations.

Lamp development, therefore, has largely been in the area of higher power from the same lamp size, as from a lamp point of view this is the simplest and most direct method of achieving higher efficiencies for a given equipment cost. However, there are problems of additional excessive heat and shortened lamp life and a balance has to be struck between these conflicting functions.

*Equipment primarily associated with drying or curing of coatings*

mercury lamp

xenon arc

ultraviolet curing

## La conception et la construction des appareils aux lampes ultra-violettes pour assurer le durcissement de revêtements et d'encre d'imprimerie

### Résumé

Il y a actuellement en Europe plus de 400 installations qui utilisent le processus de séchage par les rayons ultra-violettes, et dans le monde entier les chiffres doivent atteindre à plusieurs milles.

Les exigences pour un rendement plus élevé, et la gamme d'applications plus étendue qui exerce une influence sur les dimensions et la forme de l'installation, ont mis à l'épreuve l'aptitude des fournisseurs à répondre aux besoins en voie d'évolution et d'expansion rapides.

Les nouvelles idées risquent d'être dépassées rapidement par les événements, et par conséquent, le fabricant de matériel est obligé de

passer une grande partie de son temps en étudiant à nouveau et en modifiant les détails mécaniques du matériel déjà en service, afin que l'on puisse l'adapter à un autre emplacement.

Donc, l'évolution des lampes s'est située, en grande mesure, dans le domaine de la production d'une puissance plus élevée par les lampes dont la taille reste la même, puisqu'au point de vue de la lampe, il s'agit de la méthode la plus simple et directe d'assurer un rendement plus élevé à un prix ferme pour le matériel. Toutefois, il reste des problèmes tels que le dégagement d'un excédent de chaleur et la diminution de la vie utile des lampes, et l'on doit établir un équilibre entre ces deux fonctions opposées.

## Entwurf und Konstruktion von Ultraviolet Lampensystemen für die Härtung von Anstrichmitteln und Druckfarben

### Zusammenfassung

Bereits über 400 Anlagen sind in Europa in Betrieb, welche ein U.V. Trocknungsverfahren benutzen, und in der ganzen Welt dürfte die Zahl einige tausend betragen.

Die an die Ausrüstungserzeuger gestellten Anforderungen bezüglich höherer Leistungsfähigkeit, sowie ein sich stetig ausbreitendes, Grösse und Gestalt der Apparatur beeinflussendes Anwendungsgebiet spannte alle Nerven der Hersteller an, die sich rapide vergrößernden und wechselnden Anforderungen zu erfüllen.

Die Gefahr besteht, dass neue Ideen sehr schnell durch Ereignisse überholt werden.

In einer solchen Atmosphäre verbringt der Instrumentenbauer einen grossen Teil seiner Zeit mit Neuentwicklung und Modifikation der mechanischen Merkmale bestehender Apparatur, sodass diese unter alternativen Situationen eingebaut werden können.

Deshalb fand die Entwicklung von Lampen grossenteils auf dem Gebiete grösserer Stärke unter Beibehaltung der gleichen Lampengrösse statt, da dies vom Gesichtspunkte der Lampe aus die einfachste und direkteste Methode ist, um höhere Leistungen bei festliegenden Kosten für einen Ausrüstungsgegenstand zu erzielen. Es bestehen indessen Probleme hinsichtlich zusätzlicher überhoher Hitze und verkürzter Lebensdauer der Lampe, und deshalb muss ein Ausgleich dieser widerstreitenden Funktionen erwogen werden.

The lamp which has been almost universally accepted for this process is the medium pressure mercury arc lamp. The reasons for this choice are as follows:

1. The lamp produces a large amount of energy in the UV wave bands, approximately 15 per cent of the total output. The remainder consists of about 70 per cent infrared and the balance in white light.

2. The lamp has been used for many years in the photo-printing industry and is, therefore, well tried and tested.

3. The lamp produces a longitudinal arc and can be made in almost any length up to the mechanical and electrical limitations. The longest length so far manufactured is approximately 72 inches.

\*Paper presented at the Newcastle Section Symposium on "Ultraviolet curing" held at the University of Durham on 14-15 September 1977.

4. The lamp is almost entirely hand made at the present time, and therefore small batch production in a large variety of sizes can be accommodated.

The lamp consists of a quartz tube sealed at both ends and filled with a mixture of mercury and neon.

When a voltage is passed through the lamp the mercury slowly vapourises increasing the internal vapour pressure and drawing more current until a stable situation is reached.

Quartz lamps are relatively expensive because of the high cost of quartz and the hand manufacturing. Quartz is necessary as it is transparent to ultraviolet light and is extremely heat resistant. The surface temperature of these lamps, while running, is about 800°C. Most of the lamps currently being used in production are rated at 200 watts/inch, but other values are possible and levels as high as 800 watts/inch have been manufactured, and are being used for UV drying.

The factors affecting the choice of a specific rating are as follows:

1. Life of lamp is proportional to the deterioration in the transparency of the quartz jacket, which is in turn dependent upon the running temperature. Therefore, as the rating goes up, so the life of the lamp is shortened.
2. The size of the reflector system and the space into which it must be accommodated. Obviously a small reflector system will be much more affected by the heat output of the lamp, and this may seriously limit the possible rating of the whole lamp system.
3. The type and efficiency of the reflector cooling system.
4. The total price of the equipment. Obviously, it is cheaper to have one system at 400 watts/inch than two systems at 200 watts/inch, although the former may give more inflexibility as regards power selection.
5. The electrical limitations imposed on the lamp by the control requirements, and the current which the lamp can carry without terminal problems i.e. most lamps have a maximum current rating of about 8 amps, and it is desirable to limit the upper voltage of the lamp for practical and safety reasons. The practical lengths that can be made in these higher ratings are severely limited i.e. for 800 watts/inch, a lamp of about 6 inch length maximum is currently available.

To summarise the above points: The higher the design rating of the lamps the more efficient must be the cooling system. Also, it must be expected that a considerably shorter lamp life will result, and there are severe restrictions on the lengths that are available.

#### Additive lamps

Variations from these standard lamps are the use of minute quantities of metal halides, such as iodine or ferrous oxides, which are mixed with the mercury and have an effect of changing the spectral output of the lamp, depending on the type and quantity of the additive used.

However, additive lamps have many disadvantages at the present time:

1. The lamps tend to be more unstable electrically, and therefore the control system and the cooling has to be more sophisticated.
2. It has proved impossible to select an additive which is universally more efficient when used on all the enormous variety of coating and printing inks currently available.
3. Whilst the lamps can show an improvement in efficiency of up to 25 per cent when new, the deterioration rate of the lamp is often faster than with a conventional mercury arc lamp, so that the effective life at the higher efficiency level is often very much reduced.
4. Additive lamps are more difficult to make and, therefore, more expensive than the conventional lamp.

At present, therefore, although many of these lamps are being used experimentally, there are very few examples of their use in the production.

#### Pulsed lamps

This is another variation of the standard mercury arc lamp in which the electrical supply to the lamp is pulsed at every cycle i.e. normally 50-60 cycles/second.

The effect of this "pulsing" is to give the unit extremely high peak of energy instantaneously every 1/50 second with virtually nothing in between each peak, instead of the more even distribution radiated by the standard lamp, see Figure 1.

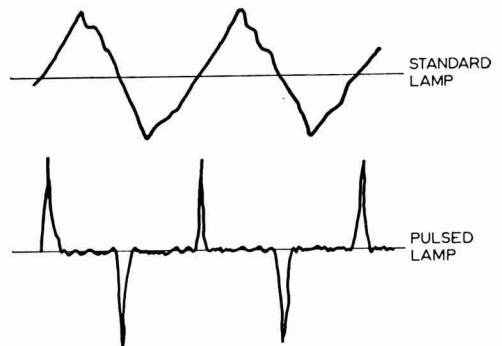


Fig. 1. The effect of pulsing

As it is fairly well established that curing rate of pigmented coatings is to some extent dependent on intensity, it can be argued that although the total output of the lamp has not changed, these high peaks of intensity give an improvement in curing efficiency.

However, although this has been found to be true in static curing situations, particularly with thick films, there are problems with moving substrates which, of course, constitutes the vast majority of curing applications. As this peak intensity only occurs every 1/50 of a second, even at modest speeds there is a considerable gap between one peak and the next causing alternate bands of high efficiency curing and very much lower efficiency curing. Attempts to even out these bands by the use of parabolic reflectors only serves to eliminate the high intensity levels received by the substrate, thus nullifying the original advantages.

Therefore, to summarise the current lamp position, it is true that most companies' manufacturing equipment are using the standard mercury arc lamp at about 200–250 watts/inch, and that current developments are moving towards higher powered lamps with more improved and efficient methods of cooling.

Much effort was put into the possibility of making a cold lamp to reduce the problems associated with the over-heating of the equipment, and possibly the substrate, with the large amount of infrared produced by these lamps. However, it is now generally accepted that a certain amount of infrared does improve the curing efficiency of the lamp to a significant extent, and that the removal of the infrared both at the source, or by the filtration through, for instance, a water jacket, does, in fact create a low efficiency system, so that in general these ideas have largely been abandoned.

### Reflector systems

In the curing of pigmented systems, it is now generally accepted the higher intensities provided by the use of the elliptical reflector are essential.

The actual shape of the ellipse depends on:

- the distance of the lamp from the substrate i.e. the focal length of the reflector, and
- the distance from the centre of the lamp to the back of the reflector (dimension "a" in the diagram). See Figure 2.

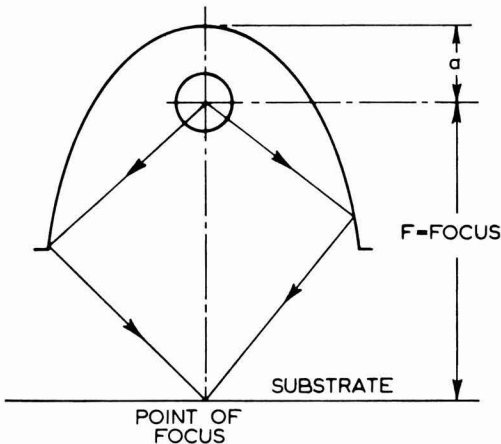


Fig. 2. A typical elliptical reflector arrangement

As the space available on the press or coater for the reflector system is often extremely limited, there is a tendency to keep both these dimensions as small as possible, therefore dimension "a" is normally between 1–1.5 inches and the focal length usually between 4–7 inches.

It is often not possible from a manufacturing point of view, or desirable from a geometrical point of view, to give a mathematically accurate shape, and therefore a vast range of reflector shapes are possible within the dimensions given, and it may well be that one shape is preferable for a certain sub-

strate or press configuration, whereas a reflector at the other end of the scale might be preferable, for a different set of circumstances. The author's Company, for instance, now manufactures five reflector shapes for different situations or applications.

It should be pointed out that although the theoretical maximum efficiency of an elliptical reflector is when the substrate is precisely at the focal point, in practice this is rarely achievable and the variation in curing efficiency is very small within  $\pm 0.5$  inch from that point. When more than 0.5 inch from the focal point, there is a discernible deterioration in the efficiency as the distance from the lamp increases.

With regard to clear coatings, there does not seem to be any significant advantage in the use of elliptical reflectors, and most suppliers are providing either parabolic or a compromise between the ellipse and the parabola, or in some cases a simple flat or angular reflector system. Again, the main criteria are the practical considerations in terms of space availability within the curing unit and the distance of the lamp from the substrate.

Except for very short lamps, this equipment cannot be switched on and off instantaneously. For a lamp of average length, it takes approximately 3–4 minutes to reach full power and if the lamp is switched off it may be necessary for it to cool down considerably before it becomes possible to restrike the arc. This might take as long as 8–10 minutes.

It is, therefore, undesirable to switch the lamp off for minor stoppages of the curing system or surrounding equipment. However, if any paper or equipment stops under the fully operational lamp it will be subjected to an extremely high temperature at the focal point. For this reason, many systems are fitted with some form of protective device that can take the form of either:

- shutters
- the removal of the lamp from the substrate
- the rotation of the reflector or system to direct the energy away from the substrate.

In this idling situation it is also normal for the lamp to be switched to half power.

Again, the practical considerations of space severely restrict the type of system used, and the author's Company uses at least three systems, depending upon the circumstances.

From the point of view of health and safety the hazards involved with these lamps are well known i.e.:

- the production of UV light which can be dangerous to the eyes and skin
- the production of ozone which is rated as a hazardous gas
- the normal electrical problems associated with high voltage equipment.

The various authorities and factory inspectorates have laid down the precise levels and conditions under which this equipment must be operated. These conditions are not difficult to achieve in the vast majority of applications. There are over 400 installations now operating in Europe alone, and the experience gained seems to have nullified the early fears

associated with the process. i.e. the safety conditions required are easily achievable, easily checked and maintained and are more readily understood and accepted by Unions, Management and the Factory Inspectorate than had been feared.

Finally, it must be said that there is no single lamp type, reflector shape or shutter system which, in combination, form an ideal system for all the wide variety of applications.

There are now available on the market an enormous range of inks, coatings, adhesives etc. The substrates to which they

can be applied consist of tin, paper, wood, cardboard, plastics and others. Application can be made on flat, round, and three-dimensional objects *etc.*, and the methods of applying these materials to the substrates vary with size, shape and complexity.

What is required, therefore, is a choice of equipment from which the user can select the configuration that most closely fulfils his particular requirements.

[Received 17 August 1977]

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*These papers were first presented at a Symposium of the Newcastle Section, entitled "Ultraviolet polymerisation", held at Durham University on 14 and 15 September 1977. Other papers from the Symposium will be published in the Journal during the next few months. The Association is preparing a limited number of reprints of these papers to be collated and bound as a separate volume and offered for sale. It is expected that the book will be available in the autumn of 1978, and further information regarding availability will be published in the Journal in due course.*

*April 1978*

## Next month's issue

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

### *OCCA 60th Anniversary*

**From gold to diamond, OCCA 1968—1978** by *S. H. Bell*

**The application of linear programming to paint and resin formulation** by *P. E. Kavanagh*

**Microanalysis of marine antifouling paints in the scanning electron microscope – its automation and application to less homogeneous paints** by *R. J. Bird and D. Park*

**The mathematical relationship between the viscosity of PVC plastisols including fillers and plasticiser number of fillers** by *J. Wypych and J. Walczak*

**A new method of determining the dispersibility of pigments and the optimal mill base formulation** by *J. Oyarzun*



# London

### The finishing of exterior timber

The third evening technical meeting of the 1977-78 session was held at the "Princess Alice", Romford Road, E7 on Thursday 19 January 1978. Dr E. R. Miller of the Princes Risborough Laboratory of the Building Research Establishment, presented a lecture entitled 'The finishing of exterior timber' to an audience of 36 members and guests.

In opening his talk, Dr Miller first mentioned that the general feeling that the present day resistance of wood and timber to exterior weather was not what it used to be, was due to a number of changing factors such as wood quality, architectural design, living standards etc. In his lecture Dr Miller examined and discussed some of these points.

Before amplifying the protection afforded by coatings Dr Miller described the chemical and morphological structure and properties of wood and showed how the differences between heartwood and sapwood and between hard and soft woods could effect the resistance of the wood to exterior exposure. The performance of surface coatings was also altered by the type of wood used.

Wood itself deteriorated on exterior exposure, either due to water, sunlight or fungal attack. Water uptake caused dimensional movement of the timber and led to grain raising, splitting and decay. For the latter to occur the moisture content of the wood had to be greater than about 20 per cent. Fungal attack caused disfigurement and staining of the timber. Sapwood was particularly vulnerable to this sort of attack and the increased use of such wood in recent years had led to greater incidences of fungal attack.

Sunlight (UV radiation) attacked the cellulose and lignin components in the outer layer of wood causing breakdown of the integral surface structure.

A combination of the three factors created more severe degradation of the timber. As an illustration of this Dr Miller exhibited slides of thin film veneers which had been exposed to sunlight under a variety of conditions. Those veneers exposed in wet atmospheres degraded very much more rapidly than those kept dry.

The change in the surface structure of the timber during exposure led to differences in the subsequent weatherability of such wood when overcoated compared with unweathered and coated wood. Regardless of the paint system used, the weathered timber did not perform as well as unweathered wood.

Dr Miller then went on to list the ideal requirements of an exterior wood finish and then compared the actual performance of pigmented and clear coatings, wood stains and water resistant coatings against this list.

In general, paint finishes had good application characteristics and gave good protection to UV radiation, but did not have particularly good film integrity over a number of years, control of moisture content, adequate extensibility or fungal resistance. Clear varnishes gave the same sort of properties as paints, and in addition, also permitted the passage of UV radiation. Exterior wood stains, containing fungicide and water repellants such as waxes or silicones, were shown to meet

all the requirements with the exception of film integrity (films need to be recoated every 2-3 years) and only reasonable water control. Such types were finding more favour with architects and were likely to be used more widely in the future.

In conclusion, Dr Miller said that previous developments in the treatment and preservation of wood had neither been as rapid nor as considerable as had really been necessary because timber was both complex in structure and at the same commonplace enough to be taken for granted. In recent years, however, the technical strides that had taken place were now beginning to reduce the problems associated with wood degradation.

After a very lively, extended question time, a vote of thanks to the lecturer was proposed by Mr R. Wallington to which the audience warmly responded.

A.J.N.

# Manchester

### Modern paint manufacturing techniques

Mr B. L. Lucas of Joseph Mason, Derby, delivered a lecture entitled "Modern paint manufacturing techniques" to 64 members and guests at The Manchester Literary and Philosophical Society on Friday 13 January 1978.

The lecturer was historical, parochial, provocative and predictive in his approach to the subject; he admitted that his company's interests were related to numerous small batches and that sandmills were not his speciality. The virtues of cavitation mixers with polythene block bottom liners were extolled and the irrelevance of rotor diameter in relation to container size stated. Future predictions had a sound basis and included vociferous reaction to increased legislation, raw materials now classified as hazardous and ultrasonics as the futuristic method of dispersion.

Question time, which included discussion of E.D. pigments and the increased use of aqueous pigment dispersions, was concluded by a vote of thanks proposed by Mr D. Clayton.

### Urethane media for heavy duty coatings

Mr C. Barker, a Manchester Section Member, employed by I.C.I. Ltd, Organics Division, delivered this lecture to 45 Students and Guests on Thursday 26 January 1978 at the Manchester Literary and Philosophical Society.

Beginning with the early history of various isocyanates, the lecturer described the toxicity hazards that influenced their eventual use. Reactive resins for isocyanates were discussed followed by the seven types of prepolymers and hydroxy acrylic resins available.

Key applications for urethane coatings illustrated with colour slides preceded a lively question period which covered such varied topics as urethane oils, blowlamp removal of urethane coatings, sensitisation and blocked isocyanates.

Mr G. T. Flood proposed the vote of thanks for the excellent lecture, which was received in the usual enthusiastic manner.

F.B.W.

## Thames Valley

The Section has held three Technical Meetings since the last report. These have been a talk by Dr D. Moody on "In-fologistics". This is a name, coined by Dr Moody (OCCAM Ltd), to describe the relationship of 'information' with Society, in the individual, group and global sense, with Government, industry, the professions - indeed, with the very survival of all forms of life. The word implies the practical result of moving, lodging and supplying information. The Lecture was both informative and amusing and the rage of questions showed the appreciation of the Members and visitors.

The December Lecture was not as advertised, and Mr G. Scott stepped into the breach at short notice. A long-time member of OCCA, he is widely travelled and has had a vast experience of paint manufacture in many countries. His talk on his career, from start to present time, was accompanied by slides and photos, many highly nostalgic showing blank spots

where once busy firms operated. It aroused many memories and was the start of a very animated discussion.

The January Lecture, by Mr G. Pollock of Osro Ltd was on 'Chemical colouring of metals'. This was a very interesting lecture on the 'blacking of metals', starting with the Great War, when the use of a highly polished rifle in the trenches usually meant sudden death and the Germans devised a method of dulling the metal to a black finish by means of chemicals. This has since developed into a fascinating industry of great scope and, judging by the lecture, of high ingenuity. Assisted by Mr Pollard of the same company, he kept the audience interested and full of questions. Although they do represent a competitive system to the use of paint, but not a protective system as such, all present gained something from the lecture. The company also specialises in 'de-burring' and the Section was treated to a résumé on this process—again, of much interest and more proof of the ingenious use of modern methods to solve ancient problems. A good discussion took place afterwards.

M.P.

## Information Received

### Increased production

Simplex-GE Manufacturing Ltd has increased their production following a change from solvent based paint to Drynamels' epoxy thermoset powder coating systems. Furthermore, the overall coating cost per component has been greatly reduced and the quality of the finishes has been increased with a marked reduction in the necessity of re-coating damaged sections.

### Electrostatic colour equipment

The finishing plant at Ford-Halewood will be equipped with a Multicolour-Esta by Behr-Industrieanlagen, Ingersheim, West Germany following ten years successful operation of two Esta devices for primer applications. All models of the Escort range including estates and a new convertible model, can be coated with the new system and a change of colour can be achieved in less than five seconds. The finish is a two coat system sprayed wet-on-wet using fourteen atomizers located on two roof machines and two side groups.

### Rationalised production

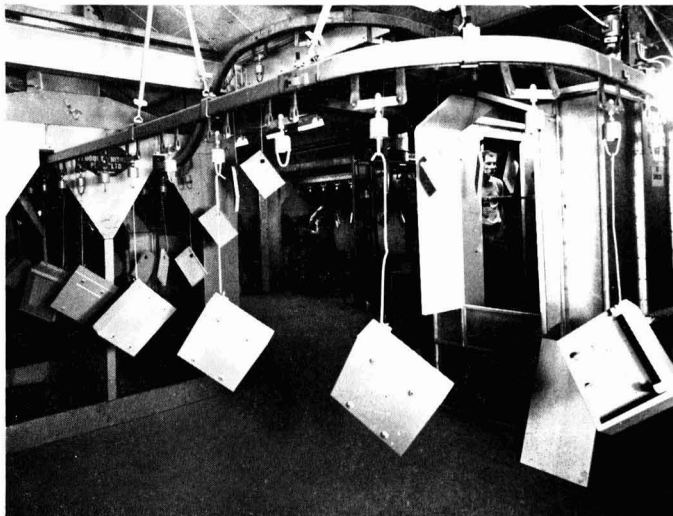
After an extensive study, Burrell Colours Ltd has concluded that production of pigments at the Cromford Colour Works, Derbyshire is no longer commercially viable and must cease after 1 July 1978. This decision is in line with the Company's long term planning and is allied to present and projected investment in new plant at the other U. K. locations, which is designed to give continued growth in total capacity. Steps are being taken to ensure continuity of supply of products from Matlock and most of the production will be transferred to other Burrell Colour units.

### Colour range changes

The Dulux Trade Group has expanded its BS4800 colour range so that all colours are available in both Dulux Trade Gloss and Trade Matt Emulsion.

### Specifications service

A new London Information service for users of American Specifications and Standards started on 1 January 1978. London Information is now in a position to advise



A view of the new powder coating system at Simplex-GE Manufacturing Ltd which has resulted in lower costs

customers who have bought standards from them, whether they have bought current documents and, if the documents have been superseded, London Information can supply the updated versions within a few days.

### Hot line

Harwell have established a hot line which will be able to advise customers on the analytical services available, as some customers had encountered difficulties in locating the department to help them.

### New products

#### New coating thickness gauge

Surfastest have available a new version of the well known Mikrotest coating thickness gauge from Elektro-Physik of Cologne. The instrument, which operates on the

principle of the attraction of a permanent magnet, is designed to measure all types of dry film applied to ferrous substrates. A variety of scales are available for non-magnetic or nickel coatings.

#### New UltrarR reagents

Hopkin & Williams have added four new chemicals to the considerable range of extremely high purity solvents and reagents. These are carbon tetrachloride, sodium sulphate, hydrobromic acid and iodine. Hopkin & Williams have also added several new items to their standard range of reagents and standard metal solutions for atomic absorption spectrophotometry.

#### Slip/peel tester

Imass, Inc., U.S.A. has announced a new instrument for measuring coefficients of



# Colour it by numbers...

PYE UNICAM COLOUR MEASUREMENT SYSTEMS WILL GIVE YOU THE RIGHT NUMBERS - RAPIDLY AND ECONOMICALLY.



The ability to specify a colour in an internationally agreed number convention is of immense benefit to industries where close control of finished product colour is vital. Numbers, which can be transmitted to and from suppliers by phone or telex, completely eliminate the vagaries of matching colour samples by eye.

Now, Pye Unicam can help solve colour specification problems at a realistic price with systems based on the SPB-100 scanning spectrophotometer. Materials may

be studied by transmitted light and, with a unique integrating spheroid attachment, by diffuse or total reflectance.

In its most sophisticated form the system has an on-line programmable calculator and a very versatile software package. Programmes are included for colour coordinates, colour difference and index of yellowness. Standard illuminants, observation angles and reporting conventions may be chosen by a simple question/answer routine.

1. plastics
2. paints, varnishes
3. glass, optical filters
4. paper, packaging
5. cosmetics
6. printing inks
7. pharmaceuticals
8. photographic products
9. food colourants
10. textiles
11. drinks and other liquids
12. ceramics, stone

TURN THE PAGE FOR FURTHER INFORMATION



## Pye Unicam

A SCIENTIFIC INSTRUMENT COMPANY OF PHILIPS

# Colour measurement at a realistic price with the Pye Unicam SP8-100.

The Pye Unicam SP8-100 is a new, high performance ultraviolet and visible spectrophotometer which has been designed from the outset with the requirements of colour measurement in mind. It is an excellent investment for laboratories with a wide range of analytical problems as it can be switched between colour measurement and normal spectrophotometric measurements in minutes, and without the need for any critical adjustments.

The SP8-100 has digital read-out of absorbance, transmission, reflectance and concentration.

Any of these may be plotted against time or wavelength on the integral chart recorder. The instrument has been designed to be extremely easy to use, even by new operators. For example, wavelength scanning is simply a matter of setting the start and stop wavelengths and pressing a button—the chart and monochromator are then aligned automatically by the unique Synchroscan feature. This ensures every recorder gridline is synchronised with a major wavelength for ease of interpretation.



## INTEGRATING SPHEROID

For the measurement of surface colour, an integrating spheroid (mean diameter 174mm) locates entirely within the sample compartment of the SP8-100. Samples as large as 160x170mm can be mounted without the need for cutting or folding. Small samples or varia-

tions across a larger surface may be examined using 3mm diameter apertures. Holders are also supplied for powder samples. Either total or diffuse reflectance may be studied by fitting the spheroid with the appropriate diffusing or absorbing caps opposite the sample.



## DATA OUTPUT AND COLOUR COMPUTATION



Colour measurements normally demand some form of data processing. The SP8-100 can output to a printer or teletype for off-line processing but, in many instances, an on-line programmable calculator will provide the most cost-effective answer.

Pye Unicam offer the powerful HP9815A calculator and a software package suitable for

most applications. The operator can select from a number of standard programs, or via a question and answer routine, establish a combination of parameters to suit particular needs. Three of these operator-defined programs can be stored and recalled at the touch of a button.

TO FIND OUT MORE ABOUT THE RANGE JUST PHONE, WRITE OR USE THE REPLY CARD



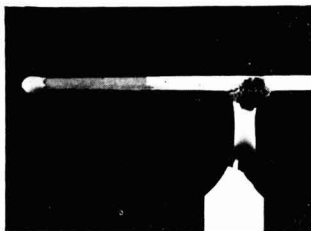
## Pye Unicam Ltd

York Street Cambridge CB1 2PX England  
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friction of surfaces and peel strengths of pressure-sensitive adhesives. The new slip/peel tester is designed for such measurements as slip of plastic films, smoothness of floor finishes, effects of surface finish on paper, rubber, metal or textiles and peel strengths of adhesives in various peel geometries. The instrument is synchronously driven and forces are measured electronically.

#### Flame retardant paint

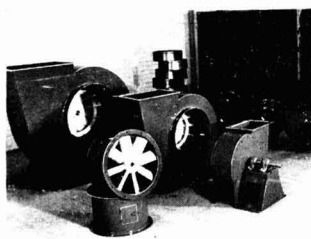
A new type of flame retardant paint has been launched by Blundell-Permoglaze Ltd. Called Fyrexo, it is the first intumescent paint to have been especially formulated for application to already painted combustible surfaces, both as a white decorative finish in its own right, and as a primer for other Blundell-Permoglaze paint systems in any colour from the BS4800 range. Fyrexo works by insulating the surface and stopping it becoming hot enough to ignite and continue the spread of flame. When flames attack the new paint it swells up to form a layer of rigid foam.



A demonstration of the flame retarding properties of the Fyrexo paint

#### Industrial fans

A new and comprehensive range of fans designed for industrial ventilation is available from Hivent Ltd. The range covers axial, backwardly inclined centrifugals and paddle-bladed centrifugals and is capable of air volumes ranging from 3400 to 50,000 m<sup>3</sup>/hr.



The new range of industrial fans from Hivent Ltd

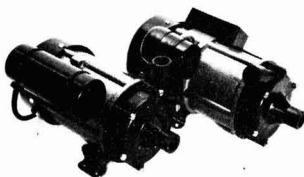
#### Body priming paint

A new primer surfacer coating, known as 'Min-Sand' has been developed by the Automotive Coatings Division of International Paint to meet the requirements of the motor industry. The paint is sprayed on to car bodies after the initial electrophoretic or conventionally dipped primer and, as the name implies, only minimal selective sanding is necessary before the colour coat is applied. Sand scratches do not show up and there is only the need for spot sanding

in local areas, which require rectification. The product has a high sheen which enables these defects to be identified.

#### Coupled pumps

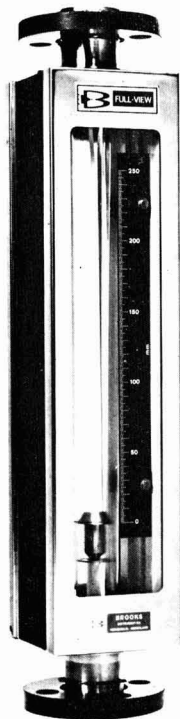
Totton Electrical Sales Ltd have announced two new models to their current range of pumps. Both pumps are driven by a ceramic magnet attached to the impeller which



The new coupled pumps from Totton Electrical Sales Ltd

eliminates the need for a shaft seal and stops leakage associated with seals. The pumps give 40 litres per minute maximum flow and have corrosion resistant coatings.

#### Full-view flowmeter



The Full-View flowmeter from Brooks Instruments in stainless steel which can be Teflon lined

Fully visible flow measurement of corrosive gases or liquids to a calibrated accuracy of  $\pm 1$  per cent full scale from 100 per cent to 10 per cent of scale reading is the performance rating achieved with the new series

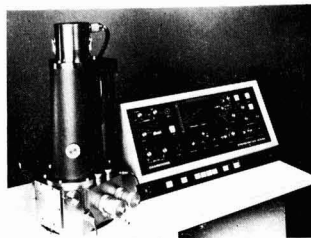
1120/21 Full-View Flowmeter from Brooks Instruments. The flow rate is indicated on the borosilicate glass metering tube in millimetres or as a percentage of maximum flow.

#### New infrared units

A new range of medium wave infrared lamps have been added to the range of equipment manufactured by Wallace Knight Ltd. The lamps are designed for use in any application in the paint and coatings industry where controllable heat forms an essential part of the manufacturing process. The lamps are available in a variety of sizes and have a peak emission between 2 and 3.5 microns.

#### New small SEM

Cambridge Instruments have introduced a small scanning microscope called Sterioscan 600M which incorporates the sophistication previously available only in their larger, more expensive, research-grade instruments. The new machine gives resolutions to 100Å with full upgradability from a wide accessory range and incorporates the same specimen handling capabilities as their top grade research machine.



The Cambridge Instruments Sterioscan 600M scanning electron microscope

#### New thixotrope from ICI

Thixomen, a new organic thixotrope from the Mond Division of ICI, is specially designed to produce high build properties in paints based on acrylics, chlorinated rubber, epoxies and vinyls. In conventional build paints it minimises pigment settlement. Thixomen also has applications in adhesives, mastics, sealants, printing and reprographic inks and elsewhere where anti-sag properties, coupled with good application properties and high film build, are required.

#### Courses, Literature, etc.

##### Heat transfer printing inks

A publication available from Eastman Chemical International AG, presents data showing how cellulose acetate propionate (CAP) can be successfully used as a replacement for ethyl cellulose in solvent-based heat transfer printing inks. This process offers brilliant colouring and sharp reproduction of intricate designs, and the publication compares grinding times, drying rates, dye-release properties in inks containing CAP and those based on ethyl cellulose.

##### Paint industry survey

DAFSA Analyse, a French company, have completed a comparative study of the European industry which compares seven French and seven English companies in depth as well as the paint industry structure of each major European country.



# OCCA-30 Exhibition

18-21 April 1978 at Alexandra Palace, London

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

## EXHIBITION PREVIEW

The Exhibition Committee is pleased to welcome to the OCCA-30 Exhibition 145 organisations, representing the United Kingdom and fifteen overseas countries:

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

It is particularly pleasing to note the return of many exhibitors from previous years, as well as new exhibitors, thus showing the strength of the support for this annual focal point for the surface coating industries.

### Dates and times

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

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

### Theme of the Exhibition

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

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

### Visits by principal officers of other societies

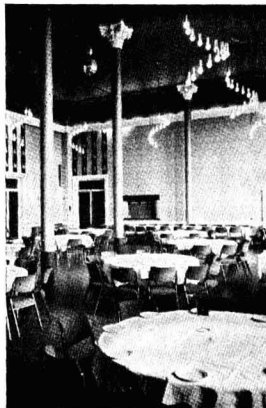
The Exhibition Committee has extended an invitation to the principal officers of many technical societies, research associations and government departments, to a private luncheon at Alexandra Palace on the opening day (Tuesday 18 April). The Committee will be conducting the party around the Exhibition and visiting the stands during the afternoon.

### Refreshments on stands

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

### Facilities at Alexandra Palace and travel arrangements

Visitors to OCCA-29 last year were impressed by the facilities available, which included two restaurants, two bars, a cafeteria and an exhibitors' bar. Other facilities this year include ample free car parking space, which is of considerable benefit especially to those using the M1 motorway which links with the North Circular Road.



The Edinburgh Room at Alexandra Palace, which will be open daily for luncheon.

The Association will once again organise a free bus schedule service to and from Turnpike Lane station on the London Underground, Piccadilly line. The journey from central London on the Piccadilly line, takes approximately eighteen minutes and connections to the Piccadilly line can be made easily from all main line stations.

Since the 1977 Exhibition, the extension

of the Piccadilly line to the Heathrow Central Terminal has been completed, which enables visitors arriving at the airport to board a train in the airport complex itself, which will take them directly to Turnpike Lane station. Heathrow Airport is the first main airport in the world to have been directly linked into the underground system of a major city.

The Association has arranged for the Exhibition to be held on its own at Alexandra Palace, which means the facilities are exclusively available for exhibitors and visitors to this important international meeting place. The Exhibition Committee, therefore, draws particular attention to this aspect of this annual Exhibition in providing an international focal point for the surface coatings industries.

### Official Guide

The Official Guide to the Exhibition has now been published and dispatched to all members of the Association and those requesting copies as a result of the Association's widespread publicity.

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

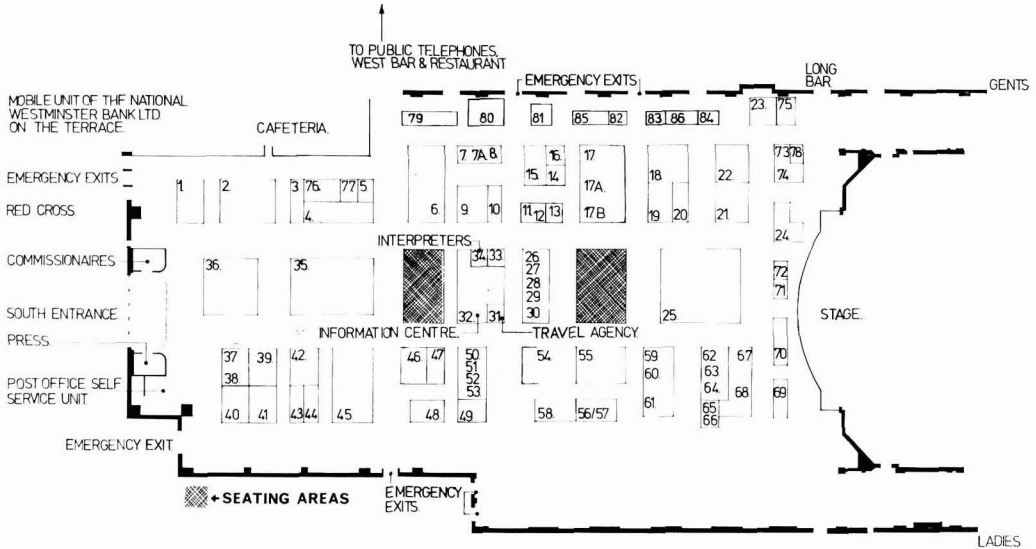
Copies of the Official Guide (including season admission ticket) are available at £2.00 (prepayment only) from the Association's offices and they will also be available for purchase at the entrance to the Exhibition Hall.

A charge is made for both the Official Guide and the season admission tickets to the Exhibition. The policy was introduced several years ago to deter casual visitors who otherwise collected large quantities of technical literature from exhibitors' stands; the policy has been welcomed by exhibitors and has in no way acted as a deterrent to bona fide visitors to the Exhibition.

### Information in foreign languages

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

# Plan of the Exhibition Hall at Alexandra Palace



Stand 87 Du Pont (UK) Ltd, situated between stands 81 and 85

## Exhibitors at OCCA-30

For position of Stand, see plan of Exhibition Hall above

For alphabetical list of Exhibitors, see Analysis of Exhibits table on pages 129, 130 and 131

### Stand

1	Ward Blenkinsop & Co. Ltd
2	Glen Creston Machinery Ltd
3	Roban Engineering Ltd
4	Jenag Equipment Ltd
5	Microscal Ltd
6	Tizamenti Vegyimuvek
7	G J Erlich Ltd
7A	Molteni (UK) Ltd
8	Ferchim Engineering SA
9	Sanyo Kokusaku Pulp Co. Ltd
10	Wheatland Journals Ltd
11	CIRP
12	Meca-Inox
13	Q-Panel Company
14	Polymers Paint & Colour Journal
15	Pilamec Ltd
16	Research Equipment (London) Ltd
17	KWR Chemicals Ltd
17A	Ludwig Schwerdtel GmbH
17B	Draiswerke GmbH
18	Allied Colloids Ltd
19	Mastermix Engineering Co. Ltd
20	A Gallenkamp & Co Ltd
21	Contraves Industrial Products Ltd
22	Cole Chemicals Ltd
23	Macbeth Division Kollmorgen (UK) Ltd
24	Instrumental Colour Systems Ltd
25	Montedison Group

### Stand

26	K & K Greeff Industrial Chemicals Ltd
27	Dow Chemical Company
28	Amoco Chemicals (UK) Ltd
29	Kirklees Chemicals Ltd
30	Barford Chemicals Ltd
33	Paintmakers Association of Great Britain Ltd
35	Lapointe Industries Ltd
36	Hercules Powder Company Ltd
37	Grilon (UK) Ltd
38	Emser Werke AG
39	Ciech—Import and Export of Chemicals Ltd
40	Millroom Accessories & Chemicals Ltd
41	Diffusion Systems Ltd
42	D.H. Industries Ltd
43	Baird & Tatlock (London) Ltd
44	Pye Unicam Ltd
45	Durham Raw Materials Ltd
46	Torrance & Sons Ltd
47	British Industrial Plastics Ltd
48	Dresser Europe SA
49	Silberline Ltd
50	Victor Blagden & Co. Ltd
51	Neville Cindu Chemie BV
52	BV Ashland-Sudchemie VH Necof
53	Chemetron Corporation
54	Compagnie Francaise Goodyear

**Stand**

55	Chemolimpex
56	Worsdall Chemical Company Ltd
57	Canadian Worsdall Chemical Co. Ltd
58	R.K. Print-Coat Instruments Ltd
59	Marchant Brothers Ltd
60	Veb Kombinat Nagema
61	Netzsch Feinmahntechnik GmbH
62	Capricorn Chemicals
63	Swada (London) Ltd
64	H. Haeffner & Co. Ltd
65	Floridienne SA
66	Micro Products Ltd
67	Croxton & Garry Ltd
68	Melbourne Chemicals Ltd
69	Fischer Instrumentation (GB) Ltd
70	John Godrich

**Stand**

71	Joyce Loebf Ltd
72	Industrial Dispersions Ltd
73	Elcometer Ltd
74	Erichsen GmbH
75	Eiger Engineering Ltd
76	Hooker Chemicals & Plastics Corp
77	Ferranti Ltd
78	MSE Scientific Instruments
79	Cornelius Group
80	Cordova Chemical Co
81	Red Devil Inc
82	Society of Dyers & Colourists
83	Lynchem Engineering Ltd
84	Flavel & Churchill (Liquid Handling) Ltd
85	Magmill France S.A.
86	Superfos Packaging (UK) Ltd
87	Du Pont (UK) Ltd

In addition to the Exhibitors listed above, reference is also made in the Official Guide to the following companies whose products are also on show :

**Stand**

42	A.B. Metall
25	ACNA
84	A.L. Process Equipment Co. Ltd
42	A.M.F. International Ltd
4	Allied Polymer Group
2	Bachofen, Willy A.
50	Blagden & Noakes Ltd
17	CdF Chimie
50	Campbell, Rex & Co Ltd
50	Chemical Supply Co. Ltd
67	Chemviron
21	Contraves AG
45	Curwen, M D Ltd
39	Daltrade Ltd
79	Daniel
64	Ecomax
79	Flare
73	Gardner Laboratory
7	Ge-Halin
78	Haake
79	Hilton-Davis
43	Hopkin & Williams Ltd
6, 16	ICI Ltd
65	Johnson, S C & Son Inc
79	Kemira Oy Vuorikemia
62	Langer, George M
67	Lucas Mejer
60	Maschinenfabrik Heidenau
79	Mearl

**Stand**

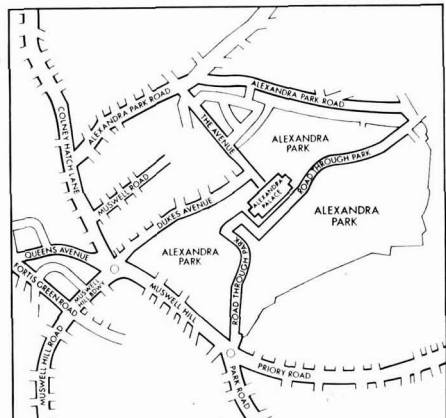
79	Methylon
65	Micro Powders Inc
45	Nuodex Ltd
7	O'Toole
42	Oliver & Battle
68	Omya
70	Original Hanau Quartzlampen GmbH
79	Oulu
42	Pamasol Willi Mader
84	Plastic Pumps Ltd
14	Portcullis Press Ltd
67	PQ International
7	Process Dipping Equipment
7	Queneuder
36	Radiant Colour
79	Roehm GmbH
1	Rondec
17	Societe Francaise D'Organo Synthese
42	Sussmeyer, Ateliers
73	Teledyne Taber
36	Ten Horn Pigments
73	UPA
7	Vibro-Mac
42	Vollrath, Paul
42	Vree, J De
64	Westerline Maskinfabric
25	Wm J MacNab & Sales Ltd

**Alexandra Park and locality**

There are adequate car parking facilities available at Alexandra Palace. Visitors travelling by road may consult the simplified diagram on page 136 showing the approach roads from central London and the Motorways, and the enlargement, right, of the area around Alexandra Park. The entrance through Dukes Avenue is for pedestrians only.

Visitors are reminded that the extension of the Piccadilly Underground line to the Heathrow Central Terminal at the airport has now been opened, so that those arriving at the airport can now board an Underground train within the passenger complex. Trains will be running at approximately four minute intervals during peak hours, and there is increased room in the compartments for travellers with luggage.

Those travelling to the Exhibition by the Underground system should leave the Piccadilly Line at Turnpike Lane station, from which point the Association will again be running a free bus shuttle service to and from the Exhibition.





### Stand telephone numbers

Many Exhibitors have applied to the Post Office for telephones to be connected to their Stand for use during the period of the Exhibition. A complete list (including late allocations made after the "Official Guide" went to press) is given below, but it is emphasised that these numbers are for use *during the opening hours of the Exhibition only.*

Stand	Exhibitor	Stand telephone number	Stand	Exhibitor	Stand telephone number
18	Allied Colloids Ltd	01-883 2545	4	Jenag Equipment Ltd	01-883 0745
28	Amoco Chemicals (UK) Ltd	01-883 1470	26	K & K Greeff Industrial Chemicals Ltd	01-883 1470
30	Barford Chemicals Ltd	01-883 1470	17	KWR Chemicals Ltd	01-883 3148
50	Blagden, Victor & Co. Ltd	01-883 0087	29	Kirklees Chemicals Ltd	01-883 1470
47	British Industrial Plastics Ltd	01-883 2699	35	Laporte Industries Ltd	01-883 8318 (3 lines)
62	Capricorn Chemicals	01-883 3335	23	Macbeth Division Kollmorgen (U.K.) Ltd	01-883 3775
53	Chemetron Corp	01-883 0087	59	Marchant Brothers Ltd	01-883 1363
39	Ciech Import & Export of Chemicals Ltd.	01-883 4022	31	Mark Allen Travel Ltd	01-883 1729
22	Cole Chemicals Ltd	01-883 1059	40	Millroom Accessories & Chemicals Ltd	01-388 2136
54	Compagnie Francaise Goodyear	01-883 3928	7A	Molteni (U.K.) Ltd	01-883 0815
21	Contraves Industrial Products Ltd	01-883 0680	60	Nagama, Veb Kombinat	01-883 1363
42	D.H. Industries Ltd	01-883 1531	(Mobile unit)	National Westminster Bank Ltd	01-883 2899
41	Diffusion Systems Ltd	01-883 2110	52	Necof, BV Ashland-Sudchemie V.H.	01-883 0087
27	Dow Chemical Company	01-883 1470	61	Netzsch Feinmahltechnik GmbH	01-883 1363
17B	Draiswerke GmbH	01-883 3148	51	Neville Cindu Chemie	01-883 0087
48	Dresser Europe SA	01-883 3999	32	OCCA Information Centre	01-883 2005
45	Durham Raw Materials Ltd	01-883 2399	14	Polymers Paint & Colour Journal	01-883 0120
38	Emser Werke A.G.	01-883 2548	58	R.K. Print-Coat Instruments Ltd	01-883 0458
7	Erlich, G. J. Ltd	01-883 0815	3	Roban Engineering Ltd	01-883 0240
8	Ferchim Engineering S.A.	01-883 0815	17A	Schwerdtel, Ludwig GmbH	01-883 3148
77	Ferranti Ltd	01-883 2788	49	Silberline Ltd	01-883 0087
2	Glen Creston Machinery Ltd	01-883 1161	63	Swada (London) Ltd	01-883 3335
37	Grilon (U.K.) Ltd	01-883 2548	46	Torrance & Sons Ltd	01-883 0932
64	H. Haeflner & Co. Ltd	01-883 3335	1	Ward Blenkinsop & Co. Ltd	01-883 3167
36	Hercules Powder Company Ltd	01-883 2240			
72	Industrial Dispersions Ltd	01-883 3536			
24	Instrumental Colour Systems Ltd	01-883 3585			

### Analysis of Exhibits

Stand	Exhibitor	Resins	Extenders, fillers, matting agents	Pigments	Additives, driers, surfactants etc.	Chemical intermediates	Manufacturing equipment, drums etc.	Laboratory apparatus and testing equipment	Miscellaneous
18	Allied Colloids Ltd	•			•				
28	Amoco Chemicals (UK) Ltd	•	•	•		•			
43	Baird & Tatlock Ltd					•		•	
30	Barford Chemicals Ltd	•	•		•				
50	Blagden, Victor & Co. Ltd	•		•					
47	British Industrial Plastics Ltd	•							
57	Canadian Worsdall Chemical Co. Ltd	•							
11	CIRP								Resin plants
62	Capricorn Chemicals		•		•				Insulation blankets
53	Chemetron Corporation	•		•					
55	Chemolimpex	•			•				
39	Ciech-Import & Export Chemicals Ltd			•		•			
22	Cole Chemicals Ltd	•	•			•			
21	Contraves Industrial Products Ltd							•	
67	Croxton & Garry Ltd	•	•	•	•				
80	Cordova Chemical Co.				•	•			



## Analysis of Exhibits—continued

Stand	Exhibitor	Resins	Extenders, fillers, matting agents	Pigments	Additives, driers, surfactants etc.	Chemical intermediates	Manufacturing equipment, drums etc.	Laboratory apparatus and testing equipment	Miscellaneous
23	Macbeth Color & Photometry Division of Kollmorgen (U.K.) Ltd ..							•	
85	Magmill France S.A. .. .. .						•		
59	Marchant Brothers Ltd .. .. .						•		
19	Mastermix Engineering Co. Ltd .. ..						•		
12	Meca-Inox .. .. .						•		
68	Melbourne Chemicals Ltd .. .. .		•						
66	Micro Products Co. .. .. .				•				
5	Microscal Ltd .. .. .							•	
40	Millroom Accessories & Chemicals Ltd ..						•	•	
7A	Molteni (U.K.) Ltd .. .. .						•	•	
25	Montedison Group .. .. .	•		•		•			
60	Nagama, Veb Kombinat .. .. .						•		
52	Necof, B. V. Ashland-Sudchemie VH ..	•		•	•				
61	Netsch Feinmahltechnik GmbH .. ..						•		
51	Neville Cindu Chemie BV .. .. .	•	•						
33	Paintmakers Association of Great Britain Ltd .. .. .								Technical education
15	Pilamec Ltd .. .. .						•		
14	Polymers Paint & Colour Journal .. ..								Technical journals and books
44	Pye Unicam Ltd .. .. .							•	
13	Q-Panel Co. .. .. .							•	
58	R.K. Print-Coat Instruments Ltd .. ..							•	
81	Red Devil Inc. .. .. .						•		
16	Research Equipment (London) Ltd ..							•	
3	Roban Engineering Ltd .. .. .						•		Bulk storage installations
9	Sanyo Kokusaku Pulp Co. Ltd .. .. .	•							
17A	Schwerdtel, Ludwig GmbH .. .. .						•		
49	Silberline Ltd .. .. .			•					
82	Society of Dyers & Colourists .. ..								Colour Index, etc.
86	Superfos Packaging (U.K.) Ltd .. ..								Packaging
63	Swada (London) Ltd .. .. .			•					
6	Tiszamenti Vegyimuvek .. .. .			•					
46	Torrance & Sons Ltd .. .. .						•		
1	Ward Blenkinsop & Co. Ltd .. .. .				•	•			UV curing agents
10	Wheatland Journals Ltd .. .. .								Technical journals
56	Worsdall Chemical Co. Ltd. . . . .	•							

## News of Exhibitors at OCCA-30

The OCCA Exhibition "Official Guide" is published many weeks before the dates of the Exhibition, so that visitors can obtain copies in advance and plan their itineraries. The "Official Guide" contains full descriptions of Exhibitors' Stands and much other useful information; copies of the "Official Guide" and season admission tickets may be purchased (at £2.00 each) either in advance from the Association's offices or at the entrance to the Exhibition, and additional copies of the "Official Guide" will be on sale at the Information Centre (Stand 32). Details of additional applicants for Stand space and news of Exhibitors issued since the publishing of the "Official Guide" are given below.

### Stand 82

#### Society of Dyers and Colourists

The Society will display details of its activities and publications, such as the *Colour Index*, and the *Journal of the Society of Dyers and Colourists*.

### Stand 87

#### Du Pont (UK) Ltd

##### Instrument Products Division

The exhibits on the *Du Pont* stand at OCCA 30 will consist of high performance liquid chromatographs and thermal analysis systems. Of interest to paint and polymer chemists will be the 830 *high temperature steric exclusion system*, which can rapidly measure molecular weight distribution. Columns compatible with both polar and non-polar solvents are available, and the latest infrared detector will beshown which affords both detection performance and analytical capacity. Also on show will be the new microprocessor based 850 *system* with methods storage and instant recall.

The 990 *Thermal Analysis System* allows development chemists to ensure that polymer behaviour is predictable during all stages of thermal processing. It is of modular design with a central temperature programmer/recorder, to which can be connected differential scanning calorimetry for measuring energy changes, thermogravimetric analysers for measuring weight changes, thermomechanical analysers for measuring dimensional changes and dynamic mechanical analysers for measuring modulus and damping behaviour. The latest addition to the thermal analysis line is the R90 which is a low cost microprocessor based controller and will allow routine applications to be performed with the minimum of operator involvement.

### Stand 43

#### Baird & Tatlock (London) Ltd

*Baird & Tatlock* will be showing their complete range of *Brookfield* viscometers including the *Rheolog* system (for recording viscosity as a function of time and/or temperature), the *Synchro-Lectric* viscometer on a *Helipath* stand which enables it to make measurements on non-flowing gels and pastes and the *Viscosel* Automatic Viscosity Control Unit which has applications wherever the viscosity of a flowing liquid needs to be kept within preset limits.

### Stand 85

#### Magmill France SA

*Magmill France SA* will be showing their range of multi-stage grinding machines for the surface coatings industries. During the grinding operation, the particles pass through three grinding zones, each designed to grind the particles to a finer size which results in improved consistency.

Further details of these late applicants for space and any further late allocations will be given in a separate leaflet distributed to visitors at the entrance to the Exhibition Hall.

### Hopkin & Williams

The largest range of *Dow Corning* silicones in the U.K. is set out in the new *Hopkin & Williams* catalogue of silicones and maintenance products. *Hopkin & Williams* range of reagents, stains and dyes will be well represented, as will the latest additions to their *AnalaR* and *UltraR* ranges of high purity chemicals

### Stand 22

#### Cole Chemicals Ltd

A comprehensive range of materials of particular interest to paint, ink and adhesives manufacturers will be shown on the *Cole Chemicals* stand, including:

**Additives.** A range of problem solving additives including non-mercurial fungicides and package preservatives.

**Aluminium powders and pastes; bronze powders.** A range distinguished by their fine lamination, enabling economies in use of 20-30 per cent when compared with competitive products.

**Fillers.** A range of clays, calcium carbonates, dolomites and marble powders.

**Grinding media.** Steatite and Stemalox granules, balls and bricks.

**Ion exchange resins.** For water treatment, effluent and process applications.

**Nonyl phenol and Di nonyl phenol.**

**PVA emulsions.** A range of homopolymer and copolymer emulsions.

**Plasticisers.** DBP, DIBP, DOP, DBM and liquid sulphonamide.

**Polyolefin powders.** For use in textured finishes.

**Resins.** A range of coumarone, hydrocarbon and terpene resins.

**Resins emulsions.**

**Urethanes.** For use in solvent and hot melt systems.

**Zinc oxides.**

### Stand 86

#### Superfos Packaging (UK) Ltd

*Superfos Packaging (UK) Ltd* will be exhibiting their comprehensive ranges of packaging materials and containers which have many applications in the surface coatings industries.

### Stand 21

#### Contraves Industrial Products Ltd

*Contraves Industrial Products Ltd* are showing their wide range of rotational viscometers and rheometers. Instruments on show include both industrial and laboratory models for determining the viscosity characteristics and flow behaviour of fluids or visco-elastic substances.

This wide ranging display includes multi-speed laboratory rheometers designed for product research and development, single speed instruments for batch quality control and industrial viscometers for in-line process control and coating applications. The *Rheomat 30*, for example, is a versatile 30 speed instrument for shear stress measurements over a wide range of shear rates. It may be used with either the *Rheoscan 20* or *30* programmer, plus an X-Y plotter to form a fully automatic rheogram plotting system.

Other models on the *Contraves* stand include the *Rheomat 15T, TV* and *STV* single 3 speed instruments, the *Covistat* industrial viscosity regulator and the *HV6* high pressure capillary viscometer.

### Stand 79

#### Cornelius Group

Exhibiting for the first time as a group, the *Cornelius* stand will present product areas of the constituent companies, e.g. *Cornelius & Co. Ltd, Cornelius Chemical Co. Ltd, Cornelius Produce Co. Ltd*.

Particular emphasis will be placed on the products of the *Surface Coatings Division*, both resins and pigments, including:

\*The range of acrylic resins from *Roehm GmbH*.

\**Fimtitan* titanium dioxide pigments from *Kemira Oy Vuorikemia*.

\**Flare* fluorescent pigments.

\*Pigments, dispersions and flushings from *Hilton-Davis*.

Methocel\* methyl cellulose ether does so much for your latex paints and not just as a thickener but as a protective colloid and a pigment suspension aid.

You can see it in the finish: excellent brushability. Less sagging. Increased stability in the initial drying stages.

It simply makes a good paint great. And as Methocel has good resistance to enzyme attack it helps give paint a long shelf life. Let us prove it to you. Write for more details now.

Dow Chemical Company Ltd.

a) Heathrow House, Bath Road, Hounslow TW5 9QY, Tel. 759 2600

b) Grove Chambers, Green Lane, Wilmslow, Cheshire SK9 1LN, Tel. 27 131

c) Swan Office Centre, 1508 Coventry Road, Yardley, Birmingham B25 8AD, Tel. 021-707 2525

-----  
Please attach this coupon to your letter-heading, stating your name and send to one of the addresses above for further details of METHOCCEL.

**METHOCCEL\*** 

**Adds body to your paint**

-----

# Paint with a great body.



\*Trademark of The Dow Chemical Company

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ask you the favour of getting acquainted with their  
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- MALEIC RESINS
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- ROSIN ESTERS
- ALKYD RESINS
- AMINE RESINS
- ACRYLIC RESINS
- DRIERS
- ORGANIC PEROXIDES
- NITROCELLULOSE

on Stand 55

*and*

- CHROMIUM OXIDE GREEN
- ZINC CHROMATE
- CHROME YELLOWS

on Stand 6

*at the*

## OCCA 1978

ALEXANDRA PALACE, LONDON—STANDS 6 and 55

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&  
CHLORINATED POLYETHYLENE



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THE FINEST COMPANIES IN THE WORLD**

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S=SUNSHINE  
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BOX, SPRAY RACK.  
Conventional Tests; any  
angle desired, new  
adjustment angle.

Services include—  
Instrumental colour and  
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performance testing of solar  
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transmittance spectro-  
photometry, radiometer  
calibration,  $I_3V_0$  of  
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We are a highly technically-  
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facility—ask your competitors  
about our service.

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AWPA, FSCT, ISES, ISO,  
OCCA, SAE



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*(List of representative clients given upon request)*

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\*Daniel speciality products.

\*Methylon modified phenolic resins.

\*Finresin specialised alkyds from Kemira.

\*Oulu tall oil fatty acids and derivatives.

\*Mearl range of pearl pigments.

## Stand 45

### Durham Raw Materials Ltd

Products manufactured by the *Durham Chemical Group Ltd* for use by the surface coatings industry being displayed this year include driers, biocides, dispersing agents and zinc dust.

*Nuodex*, *Nuosyn*, *Cargon* and *Celerate* driers have been quality standards of the industry for many years and the complete range of standard products will be displayed, including the new *Celerate Calcium 10%*.

*Nuodex* biocides are used as in-can and film preservatives for water and solvent based systems. The latest information on *Nuospt 95*, *Fungitrol 11* and *Nuodex 87* will also be featured.

Dispersion of pigments in organic media can still give problems in industry and the latest studies on the use of *Nuosperse 657* will be featured.

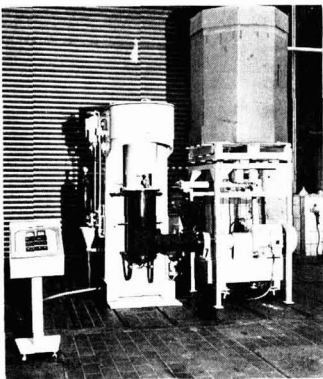
Corrosion of iron and steel is expensive and can lead to unsafe structures. Results obtained from exposure of zinc dust paints based on *Durham* zinc dusts and a wide range of organic and inorganic media will be presented.

## Stand 17B

### Draiswerke GmbH

*Draiswerke* will have photographs and diagrams of the new *PM40 STS* triple cooled mill used to feed the new *BASF* dry granulated pigment, dry resin etc. separately from the liquid phase to produce either liquid or oil inks.

Samples of the product can be seen on the stand together with results achieved on the



The Drais PM 40 STS triple cooled machine operating with direct feed for granulated pigment

*Drais STS Mill*, which is the only machine able to handle these new pigments.



Granulated pigment and resin which is fed dry to the Drais mill, without the necessity of pre-mixing with liquid

## Stand 73

### Elcometer Instruments Ltd

*Elcometer Instruments* have been appointed exclusive distributors in the U.K. for the extremely wide range of products manufactured by *Gardner Laboratories Inc.*, and this range will be introduced at OCCA-30.



A built-in micro computer coupled to Gardner's new optical system

In particular, instruments for the measurement of colour and such colour-related properties as gloss and haze will be featured.

*Elcometer* now provide the widest range of quality control surface coating instrumentation in Britain. This covers the measurement of surface profiles in cleanliness, coating adhesion, thickness and integrity. High-precision laboratory instrumentation for the measurement of colour and related properties as well as wear and abrasion testing systems are also now available.

## Stand 84

### Flavel & Churchill (Liquid Handling) Ltd

*Flavel & Churchill (Liquid Handling) Ltd* are *Turnkey* specialists for bulk handling installations for low-flash, highly flammable solvents, resins, oils etc. Also on show will be the *Flavell & Churchill* ranges of control panels and competitive ranges of tanks, pumps, flowmeters, valves, air eliminators and mobile dispensers.

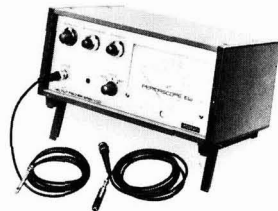
*Flavel & Churchill* mainly serve the paint and ink industries, specialising in site work and systems for bulk storage, effluent and water treatment etc.

Also on show on the stand will be the ranges of industrial flowmeters, valves, and dynamic self-checking level alarms from *A.L. Process Equipment Co. Ltd*, and a new highly efficient range of chemical and water treatment pumps from *Plastic Pumps Ltd*.

## Stand 69

### Fischer Instrumentation (GB) Ltd

*Fischer Instrumentation* will be exhibiting at OCCA for the first time after several years absence. During this time their range of coating/film thickness and porosity measuring gauges has been further developed using the latest electronic design technology.



The Permascope EW

The well known *Permascope type ES*, for coatings on ferrous materials, now has probes with specially coated, interchangeable tips, which enables measurements to be obtained from a near linear meter scale with much reduced variation due to substrate non-homogeneities. The instrument is also available in the type *ESD*, with fully LED digital readout and a BDC output for automatic printout of results, and in the *Deltascope* form, which is a compact lower priced instrument with single hardened contact probe and readout hold system.



The Deltascope

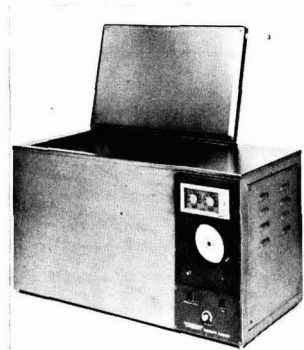
Also on display will be other *Permascope* instruments, including the *EW8* which is unsurpassed in eddy current technology. By using a new sapphire tipped miniature probe, surfaces with curvatures down to 2mm diameter can be probed with this instrument.

**Stand 70****John Godrich**

The *John Godrich* stand this year will show machinery covering the two fields of mixing and testing. Several new items will be on show.

*Chemcol-M* range of mixers and stands will be shown for the first time and up-dated versions of the standard *Chemcol* and *Rotostat* mixers will be running.

A new *Credit* humidity cabinet will be displayed showing the improvements carried out in the last year, and the *Suntest Light Fastness Tester*, together with the special flooding system will be shown. For the first time, the *Liebisch KS-300* sulphur dioxide cabinet for tests to DIN and B.S. will be available.



The Credit Humidity cabinet

Experts will be present to discuss the *Xenotest* light fastness and weathering machines and *Liebisch* salt spray cabinets, and other laboratory equipment and testing machines.

**Stand 2****Glen Creston Machinery Ltd**

This year *Glen Creston* will be showing the entire range of *Dyno Mills* from the giant 200 litre mill to the 0.3 litre laboratory machine. For the first time in the United Kingdom, it will be possible to be certain of seeing the machines which best suit customers' needs and verifying that they are production machines, not prototypes or designer's dreams.

Experience with *Dyno Mills* goes back some eleven years and includes nearly 200 installations in the United Kingdom alone; during this time there have been improvements, none of which have been introduced without extensive testing.

Of particular interest to United Kingdom and Eire customers is the policy of fitting British electrical equipment, where possible, which tends to reduce costs and simplifies complying with U.K. safety regulations.

Finally, the examples of grinding media, such as glass beads and zirconium oxide, will also be available for inspection on the stand.

**Stand 64****H. Haeffner & Company Ltd**

*H. Haeffner & Company Ltd* will be representing *Westerlins Maskinfabrik AB* of Sweden for their range of paint processing equipment. This range is comprehensive and takes in varying sizes of high speed mixers, laboratory mixers and paint sieves. This range of equipment is extensively used in Europe, but until now has not been actively sold in the U.K. market.

The Company will also be showing the range of mineral wool fibres produced by *Ecomax* of Sweden. These materials are produced as an alternative to asbestos fibre in the coatings industry and are available in fibre lengths to suit varying applications.

**Stand 72****Industrial Dispersions Ltd**

*Industrial Dispersions Ltd*, a company which specialises in powder/liquid technology, will be exhibiting for the first time at OCCA-30 with the theme of the 'Modern approach to dispersion'. As suppliers to such industries as paint, ink, plastics, rubber etc., standard ranges of products in plasticisers, oils, polyester, epoxy, glycol and universal media are offered.

In addition, the company tailor make to meet the more exacting requirements of specific industries or customers, and offers technical advice on all aspects of mixing and milling and will provide a colour matching service. The Company will be promoting pre-dispersed pigment pastes as the modern approach to dispersion and demonstrating that the savings in expensive dispersion equipment and costly skilled labour can be made their use economically viable.

Emphasis will also be placed on the positive contribution which the use of concentrated colour dispersions and master batches can make to the Health and Safety at Work Act.

**Stand 15****Pilamec Ltd****Megacol No. 1**

A vibrational ball mill adapted to paint function. The features include variable milling energy, no glands on the processing chambers which can be changed easily for colour changes. The total volume of the mill is less than 0.3m<sup>3</sup>, and it can be supplied fully engineered for paint production, with pump and tanks etc.

**Megapot vibratory pot mill**

A pot mill which is significantly faster than conventional pot mills. It can handle up to sixteen samples simultaneously or a pilot batch of approximately three litres. It is designed for wet or dry processing.

**Megapact vibratory ball mill**

This mill is designed for laboratory use, and is a tubular vibratory ball mill for continuous

or batch processing, both wet and dry. It can be supplied with pump and tank.

**Stand 4****Jenag Equipment Ltd**

A new air-driven *Jenag* strainer for the treatment of highly shear-sensitive products, including PVC pastes, plastisols and organosols, will be introduced by *Jenag Equipment* at OCCA-30. The machine, which has output capacity of 2000 litres per hour (depending on viscosity), will be particularly useful to the resins, food and wallpaper-making industries, and also in the manufacture of very heavy paints like ship antifouling.



The new Jenag air-driven strainer

**Stand 83****Lynchem Engineering Ltd**

*Lynchem Engineering Ltd* is the process engineering division of the *Lyndale Group* of fabricating and manufacturing companies.

Technical representatives and specialists in the design and installation of all types of synthetic resin plants will be in attendance to discuss equipment and advise on modern techniques of resin manufacture.

Plants ranging from the smallest pilot scale to production units in excess of 20 Tonne can be offered employing electric induction and indirect heat transfer heating systems.

**Stand 81****Red Devil Inc.**

Various models of machines for paint conditioning up to 25 litres capacity will be exhibited.

**Stand 3****Roban Engineering Ltd**

*Roban* will be showing a greatly increased product range as, in response to demands in the surface coatings industry, the *R300* solvent delivery, and *R400* road tanker offloading pumps, are now available with

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## LOW ENERGY CURE

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pneumatic motors. This type of drive yields significant savings in weight and size as well as being simpler in construction with greatly increased intrinsic safety.

These models supplement the established *R300/R400* pump range with single and three phase flameproof electric motors. The *R400* utilises the *Blackmer* rotary sliding vane pump as successfully used for many years by the U.S. road tanker industry.

Both pumps are self-priming positive displacement and suitable for use with vegetable oil and plasticisers as well as solvents. For emulsion, the *R500* is available with stainless steel rotary lobe or screw type pump mounted on the *R300* frame.

Also on show will be the *Roban* range of flowmeters with particular emphasis on the *807AT* low cost, totalising, and *R4PM5/VR* preset models which are suitable for a wide range of solvents and liquids.

Various accessories will be on show including preset control valves and linkage, microtrainer and explosion-proof micro-switch, enabling the *Roban R2* and *R4* totaliser flowmeters to be converted by the user to preset form if required.

*Roban* engineers will be present to advise on the storage of liquid raw materials and to explain the *Roban Turnkey* project service for the complete construction of above or below ground storage with distribution, pumping and metering schemes.

★ ★ ★ ★ ★ ★ ★ ★ ★ ★ ★ ★ ★ ★ ★ ★

# OCCA-31

## 3-6 April 1979

### Alexandra Palace, London

The Invitations to Exhibit at OCCA-31 will be despatched during the summer, for return by October 1978.

Any organisation which has not previously exhibited and wishes to receive a copy of the Invitation to Exhibit should contact the Director & Secretary at the address on the Contents page.

★ ★ ★ ★ ★ ★ ★ ★ ★ ★ ★ ★ ★ ★ ★ ★

## Report of Council Meeting

A meeting of Council took place at the Great Northern Hotel, London N1 on 22 February 1978 with the President, Mr A. McLean, in the chair. There were 30 members of Council present.

The Annual Report of the Council for 1977 was approved, together with the Agenda for the Annual General Meeting which will take place on 21 June 1978. Reports were received on the arrangements for the Sixtieth Anniversary celebrations, which will take place on the 11 and 12 May 1978.

Council was saddened to learn of the death of two distinguished members of the Association since its last meeting—Mr I. C. R. Bews, who had been both Honorary Secretary and Honorary Editor and Mr C. J. A. Taylor, who had been Editor-in-Chief of the Paint Technology Manuals. An obituary by Mr M. H. M. Arnold on Mr Bews appeared in the February issue of the *Journal* and it was hoped that an obituary notice on Mr Taylor would appear in the April issue. It was reported that the Association had been well-represented at the Memorial Service held to Mr Bews and at the Funeral Service for Mr Taylor.

It was unanimously agreed that any member whose subscription was three months in arrears would not receive the April issue of the *Journal* and would not be entitled to copies of the *Journal* published during the period of arrears.

It was reported that there were over 130 organisations from 16 countries on 84 stands participating at the forthcoming OCCA-30 Exhibition and that copies of the

"Official Guide" would be despatched to members early in March. Arrangements had been made to hold an informal luncheon on the opening day to which the principal officers of other societies and government departments, etc. had been invited and the response had been extremely good. The Manchester Section was once again organising a joint travel group to the Exhibition.

Reports were received on the meeting of the Publications Committee held in November and of the preliminary arrangements for the Conference which will take place at Stratford-upon-Avon, 20-23 June 1979, the title chosen by the Technical Committee at its meeting in November being "The challenge to coatings in a changing world".

Details of proposed Section Symposia were recorded and further details will appear in the *Journal* from time to time.

It was reported that at a meeting of the Professional Grade Committee held earlier in the day, one Fellow had been admitted, together with five Associates and three Licentiates who had transferred to Associateship grade. Council noted with extreme satisfaction that the Council of the Paintmakers Association of Great Britain Limited had passed the following resolution:—

"The Council of the Paintmakers Association of Great Britain recognises the need for maintaining scientific and professional standards within the Industry and recommends Members to encourage their scientific and technical staff to apply for admission to the Professional Grade of the Oil &

Colour Chemists' Association and to support the aims and activities of the Oil & Colour Chemists' Association."

Council felt that this support would be of great encouragement to the membership.

It was reported that Mr A. N. McKelvie had agreed to serve on the B.S.I. Committee PVC/21—Rusting and Preparation Grades, and Mr W. Phillips of the Manchester Section was appointed to replace Mr J. H. Sparrow, who had retired, on the B.S.I. Committee PVC/24—Water Thinned Wood Priming Paints.

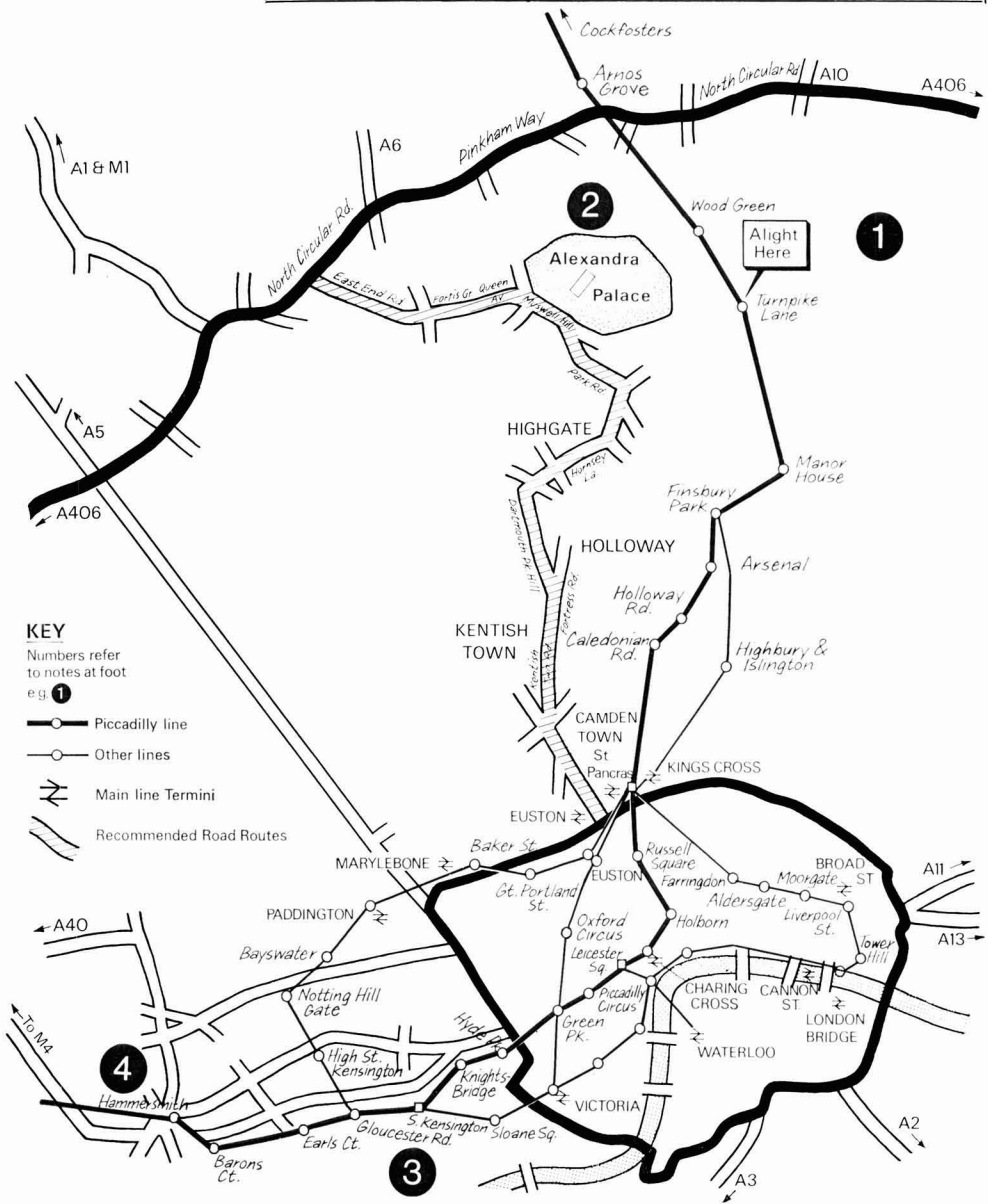
The Sections reported on other activities and authority to amend the Ontario and Newcastle Sections' Rules was given. Council was pleased to learn of the continued progress of the Divisions in New Zealand and South Africa.

Council noted with great pleasure that the Society of Dyers & Colourists had decided to confer upon the office of President of the Association honorary membership of the Society, this being only the third time in the Society's history that an office had been so honoured, as distinct from an individual. The presentation would be made to the President at the Society's annual dinner in Manchester on 28 April, in recognition of the Association's Sixtieth Anniversary and the close liaison which has been maintained between the two organisations for so many years.

There being no other business the President thanked members for their attendance and declared the meeting closed at 3.45 p.m.

# OCCA-30

For the benefit of intending visitors to the Exhibition, a map is reproduced below of the area around Alexandra Palace showing the mainline stations in central London, the Piccadilly, Circle and Victoria Lines of the underground system and also the road links with the North Circular Road and motorways. See also the enlarged map of Alexandra Palace, reproduced on page 128.



1. A free bus shuttle service will operate between Alexandra Palace and Turnpike Lane station on the Piccadilly Line (Underground), which is denoted by the thick coloured line. Destinations of trains may be marked as "Cockfosters" or "Arnos Grove" or "Wood Green".
2. Those travelling by road will find free car parking facilities at Alexandra Palace.
3. Visitors arriving at West London Air Terminal may board the Piccadilly Line trains at Gloucester Road station.
4. The Piccadilly Line has now been extended to the Heathrow Central Terminal at the airport, and visitors can now board a train at the airport which will take them directly to Turnpike Lane station, or to hotels in central London at which they are staying.
5. The map also shows the position of the mainline stations in relation to the Piccadilly Line.

## OCCA 60th Anniversary



### Celebrations

11-12 MAY, 1978

As members will be aware, the Association celebrates the 60th Anniversary of its foundation in May 1918, and it is felt that members would like some details of the proposed celebrations which will take place on 11 and 12 May 1978.

The actual date of the foundation of the Association was 16 May 1918, but it has been decided that the Thursday and Friday are more appropriate times to hold the celebrations.

The May 1978 issue of the Journal will contain an article by Dr S. H. Bell, OBE (President 1965-67) on main Association events since the 50th Anniversary in May 1968. Already many companies have reserved advertising space in this important issue in order to congratulate the Association on this achievement, and others wishing to do so can obtain full details from the Assistant Editor at the Association's offices at the address on the contents page.

#### Commemorative Lecture and Dinner

The Commemorative Foundation Lecture (instituted in 1963 in memory of the late H. A. Carwood, Esq., the first Honorary Secretary of the Association) will be given in the Court Room at the Painters' Hall, Little Trinity Lane, London EC4, on the



Sir John Methven

evening of Thursday 11 May by Sir John Methven, Director General of the Confederation of British Industry. The title he has chosen is "The place of business in our society". Admittance to the Lecture will be

by ticket only and will be followed by a short reception. Dinner will be taken in the Dining Hall of the Painter Stainers' Company at 8.00 p.m. Informal dress will be worn.

Immediately following the lecture, a special commemorative silver medal will be presented to Sir John Methven by the Master of the Worshipful Company of Painter-Stainers, Sir Ralph Perring Bt., to mark the occasion.

Tickets for the Lecture and Dinner will be available from the Association's offices at a cost of £12.00 plus VAT, and members will be sent an application form giving details in due course. The price of the ticket also includes the reception between the Lecture and Dinner, wines with the meal, and port or brandy after the meal. Past Presidents, Past Honorary Officers of the Association and Honorary Members will be invited as guests of the Association.

#### Commemorative Dinner Dance

The Association's Dinner Dance will be held at the Savoy Hotel, London WC2, on the evening of Friday 12 May, and Presidents of other societies, together with their ladies, will be invited to attend.

The reception will take place in the River Room at 7.00 p.m. and Dinner will commence at 7.30 p.m. in the Lancaster Room. Arrangements for a cabaret have been made and dancing to the Jay Langham Orchestra will continue until 1.00 a.m. Dinner Jacket will be worn.

Tickets, price £15.00 plus VAT will be available from the Association's offices, and members will again be sent full details.

Non-members who wish to receive application forms should write to the Director & Secretary at the Association's offices as soon as possible, particularly if they wish to make up parties (up to 12 per table) for the Dinner Dance.

Application forms for tickets for both functions will also be available at the Association's Information Centre (Stand 32) at the OCCA-30 Exhibition being held at Alexandra Palace, London N22, on 18-21 April.

## Scottish Section

#### Annual Dinner Dance

The Annual Dinner-Dance of the Scottish Section was held on Friday 13 January 1978 at the St. Andrews Suite in the Albany Hotel, Glasgow.

The function was well patronised by over 340 members and their guests, some travelling fairly long distances to attend.

After an enjoyable meal, the Chairman, Mr J. D. W. Davidson, welcomed the top table guests, and a toast was proposed for Ladies and guests. Mr A. McLean, President, replied for the Ladies and guests.

The dancing continued until the early hours of the morning, and it was obvious

that everyone enjoyed what was a thoroughly satisfactory evening.

G.J.L.

#### News of Members

Miss S. Gaetsky, an Ordinary Member attached to the London Section, has retired from her position as Director of Cornelius Chemical Co. Ltd.

Mr A. Hughes, an Ordinary Member attached to the Manchester Section, has been appointed to the position of Research Manager with Baxenden Chemical Co. Ltd.

Mr J. R. Green, an Associate Member attached to the Midlands Section, has been appointed Marketing Manager for Coating Resins at British Industrial Plastics Limited.



Mr J. R. Green

## Register of Members

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

### Ordinary Members

ADAMI, FERAZ, PO Box 4726, Taiz, Yeme. (*General Overseas*)

BARKER, LEONARD ARNOLD, 33 Clothierholme Road, Ripon, N. Yorks HG4 2DE. (*West Riding*)

DAVIDSON, JAMES ROBB, BSc, Shell Chemicals UK Ltd, 123 Bothwell Street, Glasgow. (*Scottish*)

GREENWOOD, TERENCE LAWSON, MSc, Amoco Chemicals Europe, 7 rue Veronnex, 1207 Geneva, Switzerland. (*General Overseas*)

HARRIS, BRINDLEY BEAUMONT, 61 Elm Tree Close, Nottorsthorne Lane, Liversedge, Yorkshire. (*West Riding*)

HOMER, JOHN, BSc, PhD, BTP Tioxide Ltd, Portrack Lane, Stockton-on-Tees, Cleveland. (*Newcastle*)

MOREL, EMMANUEL GUY, Hoechst SA Pty Ltd, PO Box 8692, Johannesburg 2000, South Africa. (*Transvaal*)

RENNIE, JAMES ALEXANDER, Gestetner Stirling Ltd, PO Box 19, Ladysneuk Road, Stirling. (*Scottish*)

SIMPSON, LESLIE AINSLEY, BSc, PhD, 22 Silverdale, Nunthorpe, Middlesbrough. (*Newcastle*)

### Registered Students

CULLEN, WILLIAM GERALD, 78 Castlefarm Estate, Swords, Co. Dublin. (*Irish*)

MCGUINNESS, WILLIAM JOHN, 21 Croydon Park Avenue, Marino, Dublin 3. (*Irish*)

SCALLY, TIERNAN JOSEPH, 8 Shanowen Drive, Santry, Dublin 9. (*Irish*)

VERONIQUE, ASHLEY FRANCIS, 28 Hastings Avenue, Ilford, Essex, IG6 1DU. (*London*)

WELLS, CHRISTOPHER JOHN, 32 Hall Drive, Harefield, Middlesex UB9 6LA. (*London*)

## Professional grade

At a meeting of the Professional Grade Committee held on 22 February, 1978 the following Ordinary Members of the Association were admitted to the categories shown. The Section to which each member is attached is shown in brackets.

### Fellow

SHEPHERD, Joseph (*Newcastle*)

### Associate

CLARKE, Raymond John (*London*)

COWIE, Edward Bruce (*General Overseas—Kenya*)

JAMES, Tudor Herbert (*General Overseas—U.S.A.*)

LEWIS, Geoffrey John (*Scottish*)

MCCALLUM, Ian Robert (*Scottish*)

### Transferred from Licentiate to Associate

CHAMBERS, Anthony (*London*)

CHURCHMAN, Anthony Edward (*London*)

FERNANDES, Larry Raphael Francis Joseph (*London*)

## Forthcoming Events

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

### April

#### Monday 3 April

*Hull Section:* Annual General Meeting to be held at the George Hotel, Land of Green Ginger, Hull at 6.30 p.m.

#### Tuesday 4 April

*West Riding Section:* Annual General Meeting to be held at The Mansion Hotel, Roundhay Park, Leeds 8, commencing at 7.30 p.m.

#### Thursday 6 April

*Newcastle Section:* Annual General Meeting to be held at St. Mary's College, University of Durham, Elvet Hill Road, Durham.

#### Friday 7 April

*Midlands Section—Trent Valley Branch:* Annual General Meeting to be held at the Crest Hotel, Pastures Hill, Littleover, Derby at 7.00 p.m.

#### Wednesday 12 April

*Scottish Section:* Annual General Meeting followed by "Demonstration of Hypnotism" by A. M. Hearne, MBE, at the Bellahouston Hotel, Glasgow at 7.30 p.m.

#### Thursday 13 April

*London Section:* Annual General Meeting at Rubens Hotel, Buckingham Palace Road, S.W.1.

#### Friday 14 April

*Bristol Section:* "Uses of solar energy" by Professor B. J. Brinkworth of University College, Cardiff, at the Royal Hotel, Bristol at 7.15 p.m.

*Manchester Section:* Annual General Meeting at The Woodcourt Hotel, Sale, Cheshire, commencing at 6.30 p.m.

#### Wednesday 19 April

*Ontario Section:* Annual General Meeting.

#### Thursday 20 April

*Thames Valley Section:* Annual General Meeting and talk on "Beekeeping" by Mr H. Aplin at the Beaconsfield Crest Motel (White Hart), Aylesbury End, Beaconsfield, Bucks at 6.30 for 7.00 p.m.

#### Friday 21 April

*Irish Section:* Annual General Meeting at the Clarence Hotel, Dublin 2, commencing at 8.00 p.m.

*Midlands Section:* Annual General Meeting at the Crown Hotel, Broad Street, Birmingham at 6.30 p.m.

#### Friday 28 April

*Bristol Section:* Annual General Meeting at the Royal Hotel, Bristol at 7.15 p.m.

### May

#### Thursday 11 May

*London Section:* Afternoon visit to the Paint Research Association, Waldegrave Road, Teddington, Middlesex TW11 8LD commencing at 2.30 p.m.





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Tel. 0532 584646.



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## UV

## Paint Technology Manuals Works Practice

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

**Price: £3.00**

## Convertible Coatings

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

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

## Biennial Conference PREPRINTS

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

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

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

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

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WEDNESDAY 19 APRIL	09.30 - 17.30
THURSDAY 20 APRIL	09.30 - 17.30
FRIDAY 21 APRIL	09.30 - 16.00

The Association will operate a free bus service to the Exhibition from (and return) Turnpike Lane Station (Piccadilly Line, London Underground).

Copies of the "Official Guide" including season admission tickets, at £2.00 each (including VAT) are obtainable in advance from the address below or at the entrance to the Exhibition.

Oil & Colour Chemists' Association,  
Priory House, 967 Harrow Road, Wembley, Middlesex,  
England, HA0 2SF  
Telephone: (01) 908 1086 Telex: 922670 (OCCA Wembley)

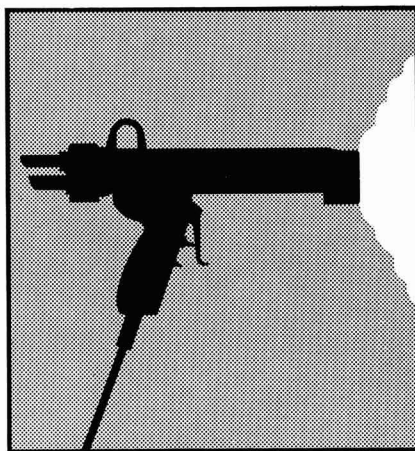
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CHEMISTS'

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TECHNICAL EXHIBITION

18-21 APRIL 1978

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

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