

Journal of the Oil and Colour Chemists' Association

# Report of 60th Anniversary Celebrations



Presidential Insignia

New developments in ultraviolet curable coatings technology

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Cure behaviour of photopolymer coatings

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Photoinitiator problems in clear coatings

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#### **Transactions and Communications**-

# New developments in ultraviolet curable coatings technology<sup>\*</sup>

#### By C. B. Rybny and J. A. Vona

Celanese Chemical Company, Marketing-Technical Development Laboratory, P.O. Box 1000, Summit, New Jersey 07901, USA

#### Summary

The broad potential of UV coating systems has been prevented from a variety of obvious applications because of certain problems inherent in the technology. Within the context of this paper, possible solutions are defined to two of the key problem areas, namely, photoinitiation in high build pigmented coatings and the

#### Keywords

Types and classes of coatings and allied products

pigmented coating

Raw materials for coatings binders (resins, etc)

> epoxy resin acrylic resin vinyl acetate polyester resin polyether resin

prime pigments and dyes

titanium dioxide

solvents

diluent solvent

reduction of vehicle viscosity. The successful commercial implementation of the findings reviewed should stimulate the development of new applications and thereby assist in the conservation of energy and reduction of air pollution.

Processes and methods primarily associated with manufacturing or synthesis

ultraviolet curing

drying or curing of coatings

radiation curing photoinitiation

Properties, characteristics and conditions primarily associated with

raw materials for coatings and allied products

viscosity

Miscellaneous

cost

#### Nouveaux développements dans le domaine de la technologie de revêtements durcissables par rayons ultra violets

#### Résumé

On ne saurait pas profiter des possibilités offertes par les systèmes durcissables par rayons ultra violets dans une gamme d'applications eventuelles qui s'imposent, à cause de certains problèmes inhérents à la technologie. Dans cet exposé on précise deux des problèmesclès, à savoir, la photo-initiation en les revêtements pigmentés du type "high-build" et également la reduction de la viscosité du véhicule. La réalisation avec succès sur le plan commercial des conclusions mentionées devrait encourager le développement de nouvelles applications et favoriser ainsi la conservation d'energie et la diminution de la pollution atmosphèrique.

#### Neuentwicklungen in der Technologie der durch UV Strahlung härtboren Beschichlungsmittel

#### Zusammenfassung

Der Grosseinsatz von UV Beschichtungsmittelsystemen wurde aus naheliegenden gründen von einer Anzabl von dafür geigneten Anwendungsgebieten ferngehalten, weil gewisse Probleme mit der Technologie eng verbunden sind.

Soweit zu dieser Abhandlung gehörig, werden zur möglichen Lösung zwei der Schlüsselproblemgebiete definiert und zwar

#### Introduction

The broad commercialisation of radiation technology has opened a gamut of new challenges to chemists and coating processors. As with any new concept, the crosslinking of polymers by means of radiation offers new opportunities. The incentives of lower overall cost, improvements in properties, energy savings, and pollution abatement are serving to stimulate continuous development throughout the industry. Photoinitiator in dickschichtigen, pigmentierten Anstrichmitteln, sowie die Reduzierung der Bindemittelviskosität. Die erfolgreide gewerbliche Auswertung der hier in Betracht gezogenen Feststellungen sollte die Entwicklung neuer Anwendungen anregen und dadurch zu der Einsparung vou Energie und der Verringerung der Pollution der Luft beitragen.

At present, ultraviolet light is the most widely used type of actinic radiation because of its relatively low cost, ease of maintenance, and low potential hazard to commercial users. Alternative radiation sources are also finding commercial acceptance in the present stage of industrial development. Sources, for example, which polymerise vehicles by electron emission are currently at a point of sophistication where exposure to X-rays is no longer a major concern. For many applications, the costs of shielding and higher capital costs

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

associated with these sources are more than compensated for by the increased throughput and improved coating properties. Although the concepts defined within the scope of this paper are directed primarily towards coating compositions curable by exposure to ultraviolet radiation, many of them can be translated to systems curable by alternative radiation sources.

Since the utility of any new technology can be measured only in terms of a finished product, the feasibility of a radiation curable coating system is directly related to its application and performance characteristics. The ability to resolve problems inherent in specific end uses is consequently of critical importance to the successful market development of UV coating systems. Two problems, common to many applications, which have required extensive research include photoinitiation in high build, highly pigmented coatings, and the reduction of vehicle viscosity. Potential solutions to each of these problem areas are offered in an effort further to enhance the development of this technology.

#### Photoinitiation

The preparation of pigmented UV coating systems has been a major challenge since the earliest stages of their development. For the most part, this has primarily been due to the reflection and absorption characteristics common to various colourants which prevent sufficient ultraviolet energy penetration efficiently to photoinitiate the vehicle systems. Package instability, prevalent with certain types of pigments, has also been a problem. The printing industry has, for the most part, circumvented the energy penetration problems by proper photoinitiator selection and its use at adequate concentrations. Because printing inks are applied as relatively thin films, the incident radiation is able to penetrate the full coating thickness resulting in good photo-response.

So far, the inability to cure efficiently high build pigmented coatings by exposure to UV radiation is still restricting this technology from many end uses. Elimination of the film thickness barrier from coating applications by utilising new initiator technology can, therefore, offer substantial diversification to potential markets. A viable approach to improve the photo-response of high build pigmented coatings is to incorporate a synergist for the conventional primary initiator system. One class of materials which functions as synergists is the perhalogenated alkanes. The use of such products not only improves photo-response, but also allows significant elimination of the higher cost primary photoinitiators. Although carbon tetrachloride is utilised in the examples which follow, it is not recommended for commercial systems because of potential health hazards which could accompany its use. Higher molecular weight perhalogenated analogues of lower toxicity, however, have been found to be equally useful.

Illustrated in Table 1 is a white UV curable coating system containing organically treated rutile titanium dioxide at a concentration of 25 per cent by weight. The system was prepared by thoroughly mixing the low viscosity vehicle with a pigment paste containing 2-chlorothioxanthone. The paste was ground on a three-roll mill after initially dispersing the titanium dioxide and initiator in trimethylolpropane triacrylate, a low viscosity (50-150 cps) multifunctional acrylate. Testing was conducted on coatings applied to Bonderite 1000 panels (Parker Test Panels) with a 1.5 mil Bird applicator and cured by exposure in air to a 200 watt per linear inch medium pressure mercury lamp having a four-inch elliptical reflector. As indicated by Tukon hardness measurements, the replacement of 50 per cent primary initiator with carbon tetrachloride significantly improves the cure response.

Table 1 White pigmented UV coating system

Weight %
40.0
2.5
7.5
25.0
25.0
phw
$A  \begin{cases} 3.0 \\ 2.0 \end{cases}$
B $\begin{cases} 1.5 \\ 1.5 \end{cases}$

Note: 1.5mil coatings cured in air by exposure to a 200watt/linear inch UV source for 3 seconds (3 passes at 20 feet per minute).

Photo- initiator package	Pigment grade*	Tukon hardness	Conical Mandrel (inches)	Reverse impact (inch- pounds)		
A	R-CR 3	9.5	Fail	0-4		
В	R-CR 3	13.9	Fail	0-4		
*Provided	by Tioxide	of America.	R-CR 3 =	triethylamine		

\*Provided by Troxide of America. R-CR 3 = triethylamine treated TiO<sub>2</sub>.

The broad function of the synergistic photoinitiator mixture is depicted in Table 2 where various grades of titanium dioxide differing in crystalline structure, particle size, and treatment are compared. A significant improvement in cure response is imparted by the carbon tetrachloride in every case regardless of the above factors. The current industry concerns of utilising exotic high cost white pigments, chalking anatase grades of titanium dioxide, or filler-type materials which contribute poor hiding and high viscosities, should consequently be obviated.

A more refined white coating system is shown in Table 3 where the primary formulation criteria are: improvements in initial paste viscosity; reduction of coating odour; and elimination of the volatile carbon tetrachloride. As illustrated, the incorporation of a wetting agent is effective in reducing the pigment paste viscosity sufficiently to allow ball milling without the use of any volatile monomeric diluents.

The second two areas of refinement are closely related since the primary initiator, which is frequently a major cause of discolouration in white systems, can be partially replaced with a perhalogenated hydrocarbon. Several of the problems associated with the incorporation of carbon tetrachloride are alleviated by the use of hexachloroethare, a solid, readily soluble substance. Reduction of the initiator concentration by replacement with hexachloroethane minimises the colour formation whilst maintaining the same high level of photoresponse common to the carbon tetrachloride-containing system.

In addition to good colour, significant gloss reduction can also be obtained in white UV coating systems. This can best be accomplished with the use of polyethylene powder as shown in example C of Table 3. In comparison with the majority of conventional flatting agents which reduce gloss at the expense of coating viscosity, the viscosity increase imparted by fine particle sized polyethylene is almost negligible. This, coupled with the UV transparency and chemically inert characteristics of polyethylene, are principal factors favouring its use.

Coating composition	Weight %
DRH-303 (Shell epoxy diacrylate) 2-Ethylbexyl acrylate	40.0
N-Vinyl pyrrolidone	7.5
Trimethylolpropane triacrylate	25.0
Titanium dioxide (defined below)	25.0
Photoinitiator mixture	phw
2-Chlorothioxanthone } Dimethylaminoethanol }	$\mathbf{A}  \left\{ \begin{array}{c} 3.0 \\ 2.0 \end{array} \right.$
2-Chlorothioxanthone Carbon tetrachloride Dimethylaminoethanol	B $\begin{cases} 1.5 \\ 1.5 \\ 2.0 \end{cases}$

 Table 2

 Comparison of varying titanium dioxide

*Note:* 1.5mil coatings cured in air by exposure to a 200 watt/linear inch UV source for 3 seconds (3 passes at 20 feet per minute).

Pigment grade*	TiO <sub>2</sub> Crystalline structure	Particle size, μ	Pigment treatment	Initiation system	Tukon hardness
RXL	Rutile	0.2-0.25	Inorganic	A B	15.8 19.0
R-FC 5	Rutile	~0.16	Inorganic	A B	9.3 17.9
R-FC 3	Rutile	~0.16	Organic	A B	12.9 20.0
A-LF-2	Anatase	0.2-0.25	Organic	A B	6.7 14.4
A-DM	Anatase	0.2-0.25	Inorganic	A B	6.2 16.9

\*Tioxide of America.

		Chemical resistance—6 nours exposure						
Pigment grade	Initiator System	Sulphuric acid, 10%	Acetic acid, 10%	Sodium hydroxide, 10%	Ethanol			
RXL	А	No effect	Very slight swelling	Etch	Blister			
	В	No effect	Very slight swelling	Slight etch	Blister			
R-FC 5	Α	No effect	Very slight blisters	Slight etch	Blister			
	В	No effect	No effect	Slight etch	Very slight blisters			
R-FC 3	Α	No effect	Slight	Slight etch	Very slight			
	В	No effect	Very slight blisters	Slight etch	Very slight swelling			
A-LF-2	A	Very slight swelling	Severe	Etch	Severe			
	в	No effect	Swelling	Etch	Swelling			
A-DM	Α	No effect	Slight	Etch	Swelling			
	В	No effect	Swelling	Slight etch	Swelling			

Chemical resistance-6 hours exposure

Note: All coatings above failed the conical mandrel bend and had reverse impacts of 0-4 inch-pounds.

Materials	Wt %	Wt %	wt %
Acrylated epoxy (DRH-303.1, Shell Chemical Co)	50.00	50.00	47.00
Rutile titanium dioxide (RTC-2, Tioxide of America)	25.47	25.47	23.13
Hexanediol diacrylate	10.19	10.19	10.29
Trimethylolpropane triacrylate		10.19	10.29
Pentaerythritol triacrylate	10.19	()	
Polyethylene powder (Polymist B-6, Allied Chemical Co)			5.14
Igepal CO-430 (GAF Corp)	0.13	0.13	0.13
2-Chlorothioxanthone (Sherwin-Williams)	0.02	0.02	0.02
Hexachloroethane (American Firstoline, Div of Instel Corp)	2.00	2.00	2.00
Dimethyalminoethanol (Pennwalt Corp)	2.00	2.00	2.00
Physical properties			
Tukon hardness	12.6	7.0	6.7
Reverse impact (pass/fail, in-lbs)	0-4	4-8	4-8
Conical bend (failure, inches)	1-1/8in	7/8in	1in
Gloss 60°	94.8	95.2	82.8
Gloss 20°	81.0	85.8	44.2

Table 3 Epoxy based white UV coatings

*Note:* 1.0mil coating cured in air by exposure to a 200watt/linear inch UV source for 3 seconds (3 passes at 20ft per minute).

The ability to cure coatings containing pigments of such high absorption and reflection characteristics in the UV region can, of course, be extended to most colours provided they do not interfere with the components of the coating system. Thus, a white basecoat can be prepared as a tint base for flushed pigments or as a dispersion medium for dry colourants.

#### Viscosity reduction

The preparation of low viscosity vehicles capable of being applied by conventional techniques has been a continuing challenge to every supplier of totally reactive "solventless coatings". A variety of techniques for achieving useful viscosities in radiation curable coating systems are available and should alleviate the majority of problems associated with this area of the technology. The options include: the selection of appropriate monomeric diluents; the use of suitable wetting agents in pigmented coating formulations; and the preparation of low viscosity resins. Each of these alternatives will be reviewed in some detail.

#### Diluents

The availability and proper use of monomeric diluents is critical in the development of radiation curable coating systems. As with any coating formulation, the ability to alter viscosity effectively is a key factor for the attainment of a broad range of applications. Currently, the principal governing requirements of monomer utility include: photo-reactivity; solvating efficiency; cost and toxicity. Failure to meet any of these requirements for a particular end use will frequently prevent the technological development of an entire market area.

The primary monomeric diluents used in conjunction with radiation curable vehicles presently include: 2-ethylhexyl acrylate; N-vinyl pyrrolidone and styrene. Even though all of these monomers are used commercially, each has limiting factors which restrict it to specific applications. Styrene, for example, is used primarily in particle board filler applications, because of its low cost and hardness characteristics. The relatively slow line speeds of particle board finishing operations makes practical the use of styrene-containing systems which react sluggishly and require exceptionally long exposure times. 2-Ethylhexyl acrylate, although several times more reactive than styrene, is still too unreactive to be important in high speed operations at higher concentrations. This monomer is frequently restricted to levels of less than 5 per cent in order to prevent residual monomer odour. N-Vinyl pyrrolidone is the most reactive of the monomers mentioned above. Its usage, however, is limited by the high cost and the tendency to impart package instability in a variety of formulations.

In general, certain basics have been established for the selection of suitable reactive monomeric diluents. Monomers containing electron-poor unsaturation, such as the acrylate esters, normally exhibit a favourable photo-response. Vinyl monomers, having electron-rich unsaturation, are generally polymerised very poorly by conventional photoinitiator systems, especially those functioning by electron transfer. They can, however, be utilised very effectively, if their reactivity ratios are such that they readily copolymerise due to propagating acrylic free radicals activated during UV exposure. Vinyl acetate is such a monomer, co-reacting very effectively with acrylic free radicals. Its extremely labile hydrogen atom, functional in chain transfer, contributes as well to its exceptional co-reactivity. The low skin and eye irritation characteristics, as well as its high solvating capabilities, introduce a new, broader formulating basis for extending the potential of UV technology.

The relative solvating efficiencies of several commonly used diluents are shown in Table 4. As indicated, vinyl acetate is four times more effective than comparable levels of 2ethylhexyl acrylate, the second most effective diluent monomer. The loss of coating weight during application and cure is also illustrated. This property, being a function of monomer volatility and reactivity, is an important consideration to every formulator, since most of the monomer volatilised in the curing chamber will polymerise on the lamp and reflector units causing undesirable shutdowns for cleaning. The minimal weight loss of the vinyl acetate-containing system during this time interval, together with the absence of residual odour after curing, are indicative of its exceptional reactivity. The coating hardness variations in each case represent the degrees of crosslinking, as well as the characteristic glass transition temperatures, of the polymerised diluent monomers.

#### Table 4

The evaluation of vinyl acetate vs conventional acrylates. Viscosity reduction, weight loss, and hardness

Coating composition			Weight %
Epoxy diacrylate (DRH	-303.1, Shell C	Chemical Co)	60.0
Trimethylolpropane tria	crylate		20.0
Monomer (illustrated be	elow)		15.0
Benzophenone			3.0
Dimethylaminoethanol			2.0
Monomer evaluated	Coating viscosity (cps)	Wt % of coating lost* (1.5mil film)	Tukon hardness
Trimethylolpropane triacrylate	>10,000	1.1	11.1
Vinyl acetate	275	1.2	10.7
N-Vinyl pyrrolidone	1,525	1.6	11.1
Cyclohexyl acrylate	2,015	2.5	10.0
2-Ethylhexyl acrylate	1,180	2.6	9.0

#### Curing schedule

After application and weight determination, set coating by exposure to source at 60ft per minute. Re-expose at 20ft per minute to obtain total cure and remove remaining residual monomer. Both exposures in air.

#### Source

200Watt/linear inch medium pressure mercury lamp with a fourinch elliptical reflector.

\*From time of application to cure.

The reaction rate and chain transfer capabilities of vinyl acetate in an acrylate system are emphasised in Table 5 where various levels of vinyl acetate are evaluated in a photoreactive system containing trimethylolpropane triacrylate. Vinvl acetate concentrations of up to 20 per cent by weight, result in minimal evaporation losses between coating application and cure indicating favourable copolymerisation rates. At levels of 30 per cent or greater, a marked increase in volatility occurs during the curing time interval. At every level, however, the cure rate of trimethylolpropane triacrylate is enhanced by the inclusion of vinyl acetate as indicated by Tukon hardness. Of course, since the reaction rates vary with the crosslinking monomers and other acrylated oligomers in a system, optimum diluent concentrations will vary with the coating composition. Typical UV formulations contain from 5-20 per cent monomeric diluent.

Various coating systems containing vinyl acetate as the diluent monomer are compared in Table 6 with comparable formulations containing 2-ethylhexyl acrylate, N-vinyl pyrrolidone, and cyclohexyl acrylate. As in a previous example, the only formulation variables are the monomer and epoxy concentrations. As shown in examples C and D, a substantially lower concentration of vinyl acetate can be utilised to obtain a viscosity comparable to that of the N-vinyl pyrrolidone system. This reflects a significant raw material cost reduction, even with a greater concentration of epoxy diacrylate.

The relative weight losses of uncured coating formulations containing vinyl acetate and 2-ethylhexyl acrylate versus a system without monofunctional monomer are depicted in Table 7. The evaluation was conducted in a hooded area vented with a 100 cubic foot/minute air flow. The periodic weight loss determinations indicate that the greatest volatility occurs in the vinyl acetate-containing system. Although this might initially be considered as a limitation of vinyl acetate, there are several comparatively volatile solvents commercially in use today which cause greater problems. Furthermore, many UV coating lines contain closed vehicle sumps which prevent the loss of volatiles.

The flammability hazard caused in vehicle systems by the inclusion of solvating materials is frequently a concern to manufacturers and end users. As in solvent systems, the flammability characteristics of most monomeric diluents are

Ta	ble	5
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The curing effects of vinyl acetate with trimethylolpropane triacrylate

	Composition (weight per cent)							
Components	Α	В	С	D	Е	F		
Trimethylolpropane triacrylate	100	90	80	70	60	50		
Vinyl acetate		10	20	30	40	50		

Notes:

(1) Add benzophenone and dimethylaminoethanol at 3 and 2 parts per hundred weight respectively. Apply composition to tared Bonderite 1000 panel with a 1.5mil Bird applicator and obtain gross weight. Cure by initial exposure at 20 feet per minute. Obtain final weight to determine weight loss.

(2) UV source: 200watt/linear inch medium pressure lamp with four-inch elliptical reflector.

Evaluations	А	В	С	D	Е	F
Weight loss (average %)	1.3	2.4	3.0	12.1	17.6	21.9
Tukon hardness	8.3	12.0	10.7	10.3	10.3	9.5

	Weight %						
Coating composition	Α	В	С	D	Е	F	
Epoxy diacrylate (DRH-303.1, Shell Chemical Co)	60	65	60	66	60	67	
Trimethylolpropane triacrylate	20	20	20	20	20	20	
2-Ethylhexyl acrylate	15						
N-Vinyl pyrrolidone			15				
Cyclohexyl acrylate					15		
Vinyl acetate		10		9		8	
Benzophenone	3	3	3	3	3	3	
Dimethylaminoethanol	2	2	2	2	2	2	
Viscosity, cps	1,200	1,180	1,525	1,300	2,015	1,90	

Table 6
Comparisons of UV coating systems formulated to comparable viscosities

Table 7					
Comparative	coating	system	evaporation ra	tes	

	,	Weight %	
Coating composition	Control A	В	C
Epoxy diacrylate (DRH-303.1, Shell Chemical Co.)	75	65	65
Trimethylolpropane triacrylate	25	20	20
2 Ethylhexyl acrylate		15	
Vinyl acetate	—		15

<b>T</b> ime 1	% coating composition lost through evaporation					
(hours)	System A	System B	System C			
0.5	I	0.2	2.3			
1.0		0.2	3.4			
1.5		0.2	3.8			
3.5		0.3	4.8			
5.5		0.3	5.1			
22.0	*	0.5	6.5			
24.0	< 0.1	0.6	6.9			

*Note:* Evaporation test conducted on a five gram vehicle sample placed in a two-inch diameter aluminium weighing dish and stored for the elapsed time in a hooded area vented with a 100ft per minute linear air flow.

significantly reduced when formulated into coating systems where the colligative properties of vapour pressure reduction are evident.

Table 8 shows the flash point variations of a UV coating system containing vinyl acetate, the most volatile monomer, at levels of up to 10 per cent. At the maximum vinyl acetate concentration, the flash point of the system, as measured by Tag Open Cup, is  $115^{\circ}$ F, significantly higher than that of neat monomer at  $23^{\circ}$ F.

#### Wetting agents

The use of wetting agents can be a significant factor in viscosity control for the formulation of pigmented coatings curable by exposure to radiation. Failure to incorporate such products into the diluent monomers prior to pigment addition will often result in preferential adsorption of the diluent by the pigment and consequent increases in the paste viscosities.

Table 9 gives the resultant viscosities of titanium dioxide pigment dispersions prepared using a variety of surfactants. In every case, the viscosities are sufficiently low to allow ball milling. The preparation of an identical dispersion in the absence of surfactant results in a product which requires milling on a three-roll mill. Each of the grinds exhibited thixotropic properties as indicated by the higher viscosities at lower shear rates.

Table 8UV coating flash point variations

		Weig	ht %	
Coating composition	Α	В	С	D
Epoxy diacrylate (RR-172, Cleanese Polymer Specialties Co)	72.5	70.0	65.0	
Trimethylolpropane triacrylate	25.0	25.0	25.0	
Vinyl acetate	2.5	5.0	10.0	100.0
Flash point, °F (Tag Open Cup)	>185	165	115	below 23

Pigment paste	Grams	Weight %		
Trimethylolpropane triacrylate	10.0	22.2		D' 1
Hexanediol diacrylate	10.0	22.2	n · 1	Dispersed
Wetting agent*	0.1	0.2	Premixed	in Cowles
TiO <sub>2</sub> pigment (RCR-3 from Tioxide)	25.0	55.4		Dissolver

 Table 9

 Evaluation of surfactants as wetting agents in UV curable pigment pastes

\*After pre-dispersing on Cowles mixer, the pastes were ball milled for approximately 45 hours.

	Wetting agent used				
	Witconal NP-40 (Witco Chem)	Witconal NP-100 (Witco Chem)	Igepal CO-430 (GAF Corp)	Triton X-100 (Rohm & (Haas Co)	
Grind	#8 on Hegman Grind Gauge (off scale)				
Brookfield viscosities					
Spindle #4 @ 12rpm (cps)	40,000	45,750	49,000	>50,000	
Spindle #4 @ 6rpm (cps)	75,000	81,000	89,000	91,000	
Resin blend down		Weig	ht %		
DRH-301.1 (Shell Chem Co Diacrylated epoxy resin) Pigment paste		50 50	and analyzing		
Brookfield viscosities					
Spindle #4 @ 30rpm (cps)	7,700	7,900	7,600	7,700	
Spindle #4 @ 60rpm (cps)	6,900	7,600	7,100	7,250	

#### Low viscosity resins

Although the majority of acrylic functional resins commercially available today are more than adequate from the standpoint of photoreactivity, their viscosities are often extremely high. This is primarily due to the urethane or epichlorohydrin-bisphenol A epoxy components most frequently present in such polymers. For coating applications where the performance properties do not require the use of such structures, acrylic functional alkyds, polyesters, or polyethers having lower viscosities, can be employed.

#### Acrylic functional polyesters

The preparation of reactive resins by the esterification of polyhydric esters with acrylic acid has been restricted from most commercial processes because of the low boiling point and thermal instability of the acrylic acid monomer. Esterification catalysts which function at temperatures of less than 100°C are also a principal requirement. The first topic details the parameters necessary to alleviate the problems associated with direct acrylic acid esterification. The polyester of Table 10 serves as the control resin for process development and optimisation. It is prepared by direct fusion of the intermediates under a nitrogen atmosphere.

The components necessary for the acrylating process include an efficient thermal free radical polymerisation inhibitor, an azeotropic solvent, and an appropriate low temperature esterification catalyst. Phenothiazine is particularly effective when used at 60 to 75 parts per million. This material is a more effective inhibitor in the absence of oxygen than the various ether derivatives of hydroquinone. Nitrobenzene, which is moderately volatile under these conditions, can also be included at concentrations of up to 25 parts per million to prevent polymerisation of the otherwise uninhibited acrylic

Table	10
Polyester of	control

Polyester composition	Molecular weight	Moles	Weight
Trimethylolpropane	134.2	1.0	134.2
Triethylene glycol	150.2	2.0	300.4
1,6-Hexanediol	118.2	1.0	118.4
Adipic acid	146.1	3.0	438.3
Dibutyl tin oxide		(0.08%)*	0.7

\*Based upon charge minus theoretical water of condensation.

Resin constants	Theoretical	Actual	
Molecular weight	883.3	841.2	
Equivalent weight	294.4	280.4	
Hydroxyl number	190.6	200.1	
Acid number		2.1	
Process time $=$ 9 hour Temperature, maximum	$m - 230^{\circ}C$		

acid that condenses overhead. This inhibitor is removed by vacuum stripping with an azeotropic solvent after completion of the synthesis.

The selection of a proper azeotropic system is vital to the success of the esterification and the usefulness of the final product. Various solvents are available which meet the criteria of condensate removal at temperatures below the boiling point of acrylic acid. Benzene, for instance, will efficiently remove water and maintain a suitable reaction temperature. Excess residual benzene, however, may be a problem and limit the application of the final product.

Using a solvent of high dielectric constant as a component of the azeotrope system should, theoretically, improve the efficiency of an acid catalyst. The combination of 1, 1-dichloroethane with benzene, however, gives no significant improvement as in Table 11, where percentage conversion measurements were determined by condensate removal at two-hour time intervals. Inexpensive low molecular weight hydrocarbon cuts\* containing a blend of aromatics and aliphatics have been used successfully, both in their neat states and in conjunction with other solvents such as benzene and toluene. In each of these cases, improved reaction conditions were achieved.

An efficient low temperature esterification catalyst system is as important to the process viability as the other components previously reviewed. Many of the more widely accepted conventional esterification catalysts, such as the organometallics, which function best in the temperature range of 150-250 °C are less than adequate at lower temperature esterification. The most active catalysts in the 80-100°C temperature range include the strong acids, namely: sulfuric acid; methanesulfonic acid; and benzenesulfonic acid. Such catalysts can be removed from the finished product with an anion exchange

\*Lactol spirits (Amsco) contains 10.2% aromatics, 56.1% saturated aliphatics ( $C_s-C_8$ ) and 33.7% cyclic aliphatics.

resin if necessary. Although efficient acid cation exchange resins such as Amberlyst 15 (Rohn & Haas) can also be used successfully in low temperature esterification, they must be included at much higher concentrations for acceptable efficiency (15-0 parts per hundred weight *versus* 0.5 parts per hundred weight of the other catalysts). These materials can, however, be regenerated for repeated use. The efficiencies of these catalysts are indicated in Table 12. The reactor temperature was maintained by a constant temperature bath at 105°C in every case.

An optimised system of low temperature esterification is illustrated in Table 13, in which acrylic acid is reacted directly with polyhydric ester control (Table 10) at stoichiometric equivalents of acrylic acid to hydroxyl. As indicated, the reaction proceeds to approximately 90 per cent conversion after ten hours at a maximum temperature of 95°C. The relatively low viscosity of the neat acrylic functional resin and satisfactory photo-response of its blend with trimethylolpropane triacrylate make it a potential candidate for numerous UV coating applications.

	Systems					
Components	F	1	В		С	D
Polyester	1.0 eq. 1.25 eq. 0.5 phw		1.0 eq.	1.	0 eq.	1.0 eq.
Acrylic acid			1.25 eq.	1.	25 eq.	1.0 eq.
Methanesulfonic acid			0.5 phw	0.	5 phw	0.5 phw
Phenothiazine	iazine 75 ppm 75 ppm 75 ppm izene 25 ppm 25 ppm 25 ppm	75 ppm	7:	5 ppm	75 ppm	
Nitrobenzene		opm 25 ppm 25 ppm		25 ppm		
Benzene	20	phw	14 phw	14	1 phw	and a second
1, 1-Dichloroethane			6 phw		-	
1, 2-Dichloroethane				6	phw	
Lactol sprits				-	-	20 phw
	%	G Conversi	on at hours	of reactio	n	
System	2	4	6	8	10	Max. reaction temp., °C
A	32.6	55.6	66.7	79.6	85.2	100
В	33.3	51.9	63.0	74.1	81.5	101
С	29.6	51.6	63.0	75.9	85.2	101
	250	(21	75 1	01 7	00 0	05

 Table 11

 The effects of azeotrope modification

Table 12

10.00		1.542	12 C. 11
Low	tomnoratura	catalyst	avaluations
LUW	<i>iemperuiure</i>	cararysi	evaluations

Catalyst	Concentration (phw)	Solvent (20 phw)	Ratio COOH/OH	Maximum reaction temp. °C
Amberlyst 15	15.0	Benzene	1.25/1	100
Sulfuric acid	0.5	Benzene	1.25/1	99
Methanesulfonic acid	0.5	Benzene	1.25/1	101
Benzenesulfonic acid	0.5	Benzene	1.25/1	101

	%0	Conversion	n at hour	s of react	ion
Catalyst	2	4	6	8	10
Amberlyst 15	15.3	34.5	46.0	61.3	72.8
Sulfuric acid	34.5	65.1	84.3	92.0	92.0
Methanesulfonic acid	32.6	55.6	66.7	79.6	85.2
Benzenesulfonic acid	34.1	56.8	72.0	79.5	84.3

UV	37	187
	_	

Polyester		1.0 equiv.		
Acrylic acid		1.0 equiv.		
Methanesulfonic a	cid	0.5 parts per	hundred weight	Based upon
Phenothiazine		75 parts per	million	theoretical
Nitrobenzene		25 parts per	million	product
Lactol sprits		20 parts per	hundred weight	) equiv. weight
% Conversi	on at hours	of reaction (n	nax. reaction tem	p. = 95°C)
2	4	6	8	10
35.9	62.1	75.1	81.7	88.2
Resin characteristic	cs after vacu	um strinning		
Viscosity	in the states	1660 cps		
Acid no.		39.0		
Acid equivalent		1438 g/eq.		
Gardner colour		<1		
Physical property e	valuation			
Blend down		Wt %	Cure schedule	
Acrylated ester		55.0	3 passes in air	under a 200
Trimethylolpropan	e triacrylate	30.0	watt per linea	r inch UV source
N-Vinyl Pyrrolidoi	ne	10.0	equipped with	a 4-inch elliptical
Benzopnenone	anal	3.0	reflector	
Dimethylaminoeth	anoi	2.0		
Physical properties				
Reverse impact		4-8 inch-po	ounds	
Conical mandrel		1-3/4 inch	failure	

Table 13 Acrylic functional polyester resin preparation

Acrylic functional polyethers

#### Refs. 1, 2

Polyethers make up another class of low viscosity resins of extreme utility in UV coating systems. The lower internal bonding typical of these resins as opposed to polyesters of similar molecular weights and structures results in even lower viscosities. Favourable cost factors also contribute to their viability. Many of the common polyethers which have been converted to acrylic functional forms include: ethoxylates and propoxylates of trimethylolpropane, or pentaerythritol, and polyethers of 1,4-butanediol.

Although direct esterification of polyhydric ethers with acrylic acid may be possible, many polythers will degrade under the acidic conditions of esterification. Trans-esterification, however, is quite useful and will not adversely effect the ether linkages. Of the catalysts evaluated, tetraisopropyl titanate was found to be the most effective. Ethyl acrylate also appears to be the most useful acrylate source based upon its boiling point and azeotropic characteristics. Efficient columns minimise the loss of ethyl acrylate relative to the ethyl acrylate/ ethanol azeotropic mixture and are consequently desirable for the procedure.

The reactor is initially charged with one hydroxyl equivalent of polyether and up to two and one-half ester equivalents of ethyl acrylate. The excess ethyl acrylate improves the transesterification reaction rate and functions as a component of the binary ethanol/ethyl acrylate azeotrope (B. P. 77.5) which contains 72-7 per cent ethanol and 27-3 per cent ethyl acrylate<sup>1</sup>. Some of the initial ethyl acrylate charge is also used to remove water contamination from the reactants in the form of an ethyl acrylate/water azeotrope  $(85/15 \text{ per cent respectively}, B.P. 81\cdot1^{\circ}C)^2$ . Water removal is imperative before charging the catalyst, since it reacts with the titanate.

Phenothiazine and nitrobenzene inhibitors must also be included prior to the initial heat-up at levels of approximately 50 ppm each. As little as 10 ppm of phenothiazine has proved effective in inhibiting polymerisation during some transesterifications. Up to 200 ppm of each have also been included with no apparent variation of photo-response.

The reaction can be monitored by infrared or gas chromatography analysis to determine the amount of ethanol being removed. Upon completion, the reactants are cooled to room temperature and subjected to reduced pressure (0.5-1.0mm Hg). The reactor is then slowly heated to  $100^{\circ}$ C and maintained for one hour to remove all remaining ethyl acrylate. The final product is well mixed with up to 1cc of water to destroy the catalyst, and then filtered to remove the spent catalyst (TiO<sub>2</sub>) which causes hazing of the resin.

Table 14 gives the composition required to convert a trimethylolpropane propoxylate into its acrylic functional form. Upon completion of the reaction, the polymer was vacuum stripped and purified as described above. The performance properties of the resultant polymer when formulated into a UV curable composition and photopolymerised at a one mil thickness on phosphate treated steel are also given.

As indicated, the use of a more highly functional crosslinking acrylate decreases the coating flexibility whilst increasing hardness and chemical resistance.

	Resin formulation	Eq. W	't.	Equiv.	Weight
	Pluracol® TP-340 polyether				
	(BASF Wyandotte)	101.1		1.0	101.1
	Èthyl acrylate	100.1		2.0	200.2
i,	Phenothiazine	$(\sim 60 \text{ p})$	om)		0.0176
-	Nitrobenzene	$(\sim 60 \text{ p})$	om)		0.0176
-	Tyzor® TPT (tetraisopropy)	100 - 200 - 0 <b>1</b> - <b>1</b> - 100 - 0			
	titanate, DuPont)	(1%) —			3.04
-		x-707			
	UV curable composition	Weight %			
	Acrylic functional polymer		80		
Mu Ber Dir	Multifunctional acrylate		15		
	Benzophenone		3		
	Dimethylaminoethanol		2		
			Polymer 4		Polymer
	Physical tests	HDODA	TMPTA		PETA
-					
	Pencil hardness	F-H	нв-н		нв-н
	Conical mandrel	7-1/2 5-1/2			5-1/2
	Reverse impact	0-4	0-4		0-4
	Chemical tests (23 Hours)				
-	Sodium hydroxide, 10%	No effect	No effect		No effect
	Sulfuric acid, 10%	No effect	No effect		No effect
	Acetic acid, 10%	Severe stain	Severe stain		Slight stain
	Methyl ethyl ketone	Blister	Blister (20 hr	s.)	Very slight etch
	Ethanol	Blister	Blister	.,	Very slight swel
	Manthialata	Vana diaka atak	Manualisht a	ter.	i ci j slight swei

Table 14
 Acrylated propylene oxide based polyether by ester interchange

#### Summary

The ability efficiently to alter the viscosity of a UV coating system, either by polymer design or diluent selection, is an important consideration for any vehicle manufacturer. The successful curing of high build pigmented coatings is equally important to the development of radiation coatings technology. It is hoped that the information described in this paper will provide the basis and incentives to open new markets for radiation technology.

#### Acknowledgment

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1. Riddle, E. H., "Monomeric Acrylic Esters", 1954, page 9. Reinhold Publishing Corporation, New York.

2. Ibid., page 9.

## Cure behaviour of photopolymer coatings\*

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#### Summary

The complexity of the principles underlying radiation curing of thin film surface coatings has necessitated a mainly empirical approach to the design of lamp assemblies. However, theoretical understanding may be useful as an aid to effective further development of the technology.

In this paper a model for the interaction of UV radiation with the active components of a simple photopolymer coating is developed. Expressions for the radiant absorption in the critical surface and bottom layers of the film are obtained. The origin of the spectral input data required by these equations and the validity of various

#### Keywords

Processes and methods primarily associated with drying or curing of coatings

photopolymerisation ultraviolet curing radiation curing photoinitiation approximations used in the approach are discussed.

In certain cases, predictions obtained from the equations are verified experimentally. A wave length dependence for surface and for through cure is demonstrated. The effect of photoinitiator concentration on cure rates is examined. It is shown that excessive amounts of photoinitiator can, in certain instances, be counterproductive. By comparing the calculated power required to cure the respective top and bottom layers of a coating, the effect of oxygen inhibition at the surface is revealed.

Properties, characteristics and conditions primarily associated with materials in general spectrum

dried or cured films light absorption

Miscellaneous terms energy requirement

#### Les caractéristiques de cuisson des revêtements photopolymères

#### Résumé

La complexité des principes fondamentaux du durcissement par irradiation des revêtements en feuils minces a exigé une tentative largement empirique à l'égard de l'étude des montages de lampes. Néanmoins, une compréhension de la théorie pourrait être utile en favorisant de nouveaux développements efficaces de la technologie.

Dans cet exposé on met au point un modèle pour décrire l'interaction de la radiation ultra violette et des constituants actifs d'un revêtement photopolymère peu compliqué. On obtient les formules de l'absorption de radiation par la couche critique à la surface et aussi par celle au fond du feuil. On discute à la fois les données de l'energie spectrale absorbée qu'exigent ces equations, et la validité des diverses approximations utilisées dans la tentative décrite.

#### Das Verhalten von photopolymeren Beschichtungsmitteln by der Härtung

#### Zusammenfassung

Die Kompliziertheit der Prinzipien, auf denen die Härtung dünner Beschichtungen durch Bestrahlung basiert, machte eine vorwiegend empirische Konstruktion der Lampenarrangements notwendig. Theoretisches Verständnis dürfte jedoch zuwirkungsvoller Weiterentwicklung der Technologie verhelfen.

In dieser Abhandlung wird ein Modell für Reaktion von UVStrahlung mit den aktiven Komponenten eines einfachen Photopolymeren Beschichtungsmittels entwickelt "Ausdrücke" werden geschaffen für die Strahlungsabsorption in den kritischen Ober- und Unterschichten des Films. Der Ursprung der von diesen Gleichungen geforderten Werte für die spektrale Eingangsenergie, sowie die

#### Introduction

#### Refs. 1, 2, 3

The growth of interest in photochemically curable surface coating systems has stimulated development programmes by both the manufacturers of radiation curing equipment and the suppliers of raw materials with the result that the converter and his customers are confronted with an enormous range of Dans certains cas, on a obtenu une vérification expérimentale des prédictions dérivées des équations. On démontre qu'il existe un rapport entre la longueur d'onde et à la fois le durcissement superficiel et complet du feuil. On étudie l'influence qu'exerce la concentration de photo-amorceur sur la vitesse de durcissement. On démontre que, dans certains cas, la présence des quantités excessives de photo-amorceur peut exercer une influence négative. Au moyen d'une comparaison des quantités d'energie calculées à être requises pour durcir les couches supérieures et inférieures d'un revêtement, on saurait mettre en évidence l'effet de l'action inhibitive provoquée à la surface par l'oxygène.

Gültigkeit verschiedener in der Behandlung benutzten Näherungswerte werden besprochen.

In gewissen Fällen werden aus den Gleichungen erhaltene Voraussagen experimentell bestäligt. Es wird gezeigt, dass für oberflächliche, sowie Durchhärtung Abhängigkeit von der Wellenlänge besteht. Der Einfluss der Photoinitiatorenkonzentration auf die Härtungsgeschwindigkeit wird geprüft. Es wirdgezeigt, dass Überschuss von Photoinitiator ingewissen Fällen contraproduktiv sein kann. Die Wirkung der Sauerstoffinhibierung auf die Oberfläche wird durch Vergleich der zur Härtung von Oberund Unterschichten eines Films berechneten nötigen Energie dargelegt.

variables from which they have to design specific curing systems. A desirable end product is one which provides the required surface properties at the maximum possible throughput rate and with a minimum expenditure of energy. The number of potential raw material combinations and the complexity of the photopolymerisation process, together with commercial pressures which may limit the duration of development programmes, neccessitate a mainly empirical

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

approach to product formulation, and also to the design of curing assemblies. However, several investigations of a more fundamental nature have been undertaken. Such studies are justifiable in so far as the information they yield may provide guidance for the chemist engaged in the development of novel systems, assist in the efficient screening of alternative raw materials and elucidate the interpretation of any test procedures carried out on the cured samples. Some emphasis has been placed on the establishment of methods for the assessment of the degree of cure attained by the various coating systems and the various curing equipment.

Thus, McGinniss and Dusek1 have devised a technique for the evaluation of UV induced polymerisation rates in bulk samples for a variety of acrylate monomers. It is questionable as to how far their results may be extrapolated to behaviour in thin films. A division into tack free, thumb twist free, or scratch free surface has been used by Bassemire, et al<sup>2</sup> to gauge the extent of cure for films of UV curable inks. In a detailed study of the effects of various photosensitisers and differing radiation sources, Krajewski et al3 have used postcure weight loss, on baking, as a measure of the degree of polymerisation in thin film samples. The quantitative results obtainable by this technique make it attractive. It does not, however, allow a distinction to be drawn between cure at the film surface and the total through-cure. In the investigations reported in this paper, the importance of the two types of cure are emphasised. To this end, results are examined in terms of finger tackiness, which measures surface cure, and either the number of MEK rubs or the degree of lateral finger pressure required to move the film, which provides an indication of through-cure.

The Inmont Corporation has undertaken a number of studies in the more fundamental areas of radiation film curing. The findings reported in this paper are based on work that has been undertaken by Dr H. Rubin at the Central Research Laboratories of Inmont. Initial work involved the design and construction of instrumentation to monitor various parameters relevant to the photopolymerisation concept. A quartz light probe, terminating in a photosensor, which in turn was connected to a photometer, was used to probe the irradiance of a typical UV curing assembly. For such equipment, elliptical reflectors are commonly placed behind tubular UV lamps. Consequently, the irradiance, that is the rate at which light energy passes through any specified unit area in front of the lamp, is a summation of divergent radiation from the lamp and radiation returned from the reflector. Under these conditions, absolute measurements of irradiance are difficult. Further, the significant output of infrared radiation from most UV sources precludes direct probing of regions close to the lamp with photosensors. A device allowing variable, but precisely controlled, periods of specimen exposure to an ultraviolet source was built. A facility permitting changes in the lamp to sample distances was incorporated. Additionally, as the specimen was mounted on a revolving drum, simulation of multilamp exposure was possible. Also, by mounting neutral filters, constructed from precision perforated steel sheets, close to the lamp, the irradiance intensity could be varied. With such filters in place, the apparatus could be used as a sensitometer to determine the cure response at various light intensities.

The need to distinguish between surface and through-cure is emphasised in the derivation of a theoretical analysis for radiation curing of thin films. Within the limitation of certain simplifying approximations, some predictions have been calculated from this theory and where appropriate these have been verified experimentally.

#### Theory of radiation curing of thin photopolymer films

#### Ref. 4

A model for the interaction of radiation with a semitransparent film is given in Figure 1. Assuming different refractive indicies for each medium, partial reflection will occur at each interface.



Fig. 1. Model for light absorption by a simple coating film

The reflectances of the air/film and the film/substrate interfaces may be defined as  $\rho_S$  and  $\rho_B$  respectively. The radiant energy (in this work the approximation is made that the irradiation is directly proportional to  $I_0$ , the radiance of the source) is attenuated by the air/film reflection and through absorption by photo-active components as it passes through the film. The transmission T across a film of thickness *l*cm with a single active component present in a concentration of *c* moles/litre and having an extinction coefficient  $\epsilon$  can be calculated from Beer-Lambert's Law:

$$T = 10^{-\epsilon cl}$$

Energy reaching the film/substrate boundary is again partially transmitted and partially reflected. This process, in theory, occurs indefinitely, but in practice light attenuation is so rapid that only the first few traverses across the film are of significance.

For surface cure to occur, it is necessary for at least a certain quantity of energy to be absorbed by a top layer of the film, the thickness of which may be defined as  $I_S$ . Likewise, through-cure will occur if sufficient energy to effect cure reaches the bottom layer of film  $I_B$ .

For convenience, after putting  $I_S = I_B$  ( $I_0$  in Figure 1 and hereafter) we can define these layers as fractions of the total thickness,  $f_S = I_0/l$  and  $f_B = 1 - I_0/l$ .

The absorptions of radiant power which occur in each passage can be summed to obtain an infinite geometric series.

$$I_{film} = I_0(1 - \rho_S) (1 - T) (1 + T\rho_B) \sum_{n=0}^{\infty} (T^2 \rho_S \rho_B)^n \dots (1)$$

Since T is defined to lie between 0 and 1, the series will converge and thus:

$$\sum_{n=0}^{\infty} (T^2 \rho_S \rho_B)^n = (1 - T^2 \rho_S \rho_B)^{-1} \dots \dots \dots (2)$$

An expression for energy absorption by the partial layers  $(l_0)$  can be obtained by a process of subtraction. Thus:

$$I_{l0} = I_0(1 - \rho s) (1 - 10^{-\epsilon cl}) \dots (3)$$

the forward passage and

$$I_{l0} = I_0(1 - \rho_S) T \rho_B[(1 - 10^{-\varepsilon cl}) - (1 - 10^{-\varepsilon cl(l-lo)})]..(4)$$

for the return passage. By using the fraction  $f_S = l_0/l$  to simplify equations (3) and (4) and substituting into equation (1) (the expression for  $I_{film}$  can be expanded to sum of two components, one representing radiation absorbed in a surface to base direction, and the other in a vice versa direction) an expression for radiant absorption by the surface layer is obtained:

$$\mathbf{S}_{1} = \frac{I_{O}(1-\boldsymbol{\rho}_{S})}{1-T^{2}\boldsymbol{\rho}_{S}\boldsymbol{\rho}_{B}} \left[1-T^{f}_{s}+T\boldsymbol{\rho}_{B}\left[(T^{1-f}_{s}-T)\right]\dots\dots(5)\right]$$

Similar treatment results in an expression for the power absorbed in the bottom layer.

$$B_1 = \frac{I_O(1-\rho_S)}{1-T^2\rho_S\rho_B} \left[T^f_B - T + T\rho_B(1-T^{1-f_s})\right]....(6)$$

These equations prevail for a single photo active component. A model UV curable surface coating will contain a photoinitiator and an oligomer. Equations (5) and (6) can be extended to handle this situation by making the substitution  $T = T_1T_2$  and multiplying by an inner filter factor<sup>4</sup>:

$$\frac{\varepsilon_1 C_1}{\varepsilon_1 C_1 + \varepsilon_2 C_2}$$

This fraction defines the amount of the total energy, that is absorbed by a photoiniator present in concentration  $C_1$  and having an extinction coefficient  $\varepsilon_1$ . Energy absorbed by the oligomer (concentration  $C_2$  and extinction coefficient  $\varepsilon_2$ ) will result in only a small amount of polymerisation and is neglected in this approach.

The equations for the effective radiant power absorbed in the surface and bottom layers respectively thus become:

$$S_{2} = \frac{\varepsilon_{1} C_{1}}{\varepsilon_{1} C_{1} + \varepsilon_{2} C} \cdot \frac{I_{0} (1 - \rho_{s})}{1 - (T_{1}T_{2})^{2} \rho_{s} \rho_{B}} \cdot [1 - (T_{1}T_{2})^{f_{s}} + T_{1}T_{2} \rho_{B} (T_{1}T_{2})^{1 - f_{s}} - (T_{1}T_{2})^{2} \rho_{B}] \dots (7)$$

$$B_{2} = \frac{\varepsilon_{1} C_{1}}{\varepsilon_{1} C_{1} + \varepsilon_{2} C_{2}} \cdot \frac{I_{0} (1 - \rho_{s})}{1 - (T_{1}T_{2})^{2} \rho_{s} \rho_{B}} \cdot [(T_{1}T_{2})^{f_{B}} - T_{1}T_{2} + T_{1}T_{2} \rho_{B} - T_{1}T_{2} \rho_{B} (T_{1}T_{2})^{1 - f_{B}}] \dots (8)$$

As  $I_O$ ,  $\varepsilon$ ,  $\varphi_B \varphi_B$  and T are functions of wavelengths, it follows that equations (7) and (8) are wavelength dependent.

It is important to remember that the basic assumption made in this approach is that properties of the cured coating are strongly dependent on the radiant power absorbed by the photoinitiator at zero time.

#### Results

#### Refs. 3, 5, 6, 7, 8

The results of studies on the irradiance profiles of a typical UV lamp system have been reported fully elsewhere, as also have the results of cure speed investigations and experiments with sensitometer equipment.<sup>5,6</sup> Only certain principal findings need reiteration here.

The dependence of through-cure on the lamp to coating distance, and hence the irradiance, was found to be essentially constant, whilst surface cure increased rapidly with increasing density of radiant power. This was interpreted in the following manner. Intense irradiance will generate a proportionally higher concentration of free radicals than will diffuse irradiance. At the surface, this leads to an enhancement of the polymerisation reaction over the competing oxygen scavenging. In the bulk film, where oxygen concentration is presumed to be low, the polymerisation rate is more dependent on the total available energy than on its distribution. Sensitometer experiments indicated that cure was more strongly influenced by the irradiance value than by the exposure time. Practically, this implies that for a given degree of cure the throughput rate may be more efficiently increased by raising the power than by increasing the number of lamps used. In this work, thin, unpigmented film coatings were used. Different considerations prevail with thick and pigmented systems. In particular, attention is drawn to the extended exposure time which may increase the contribution of the infrared component of the source to the curing process.

Values for the variable in equations (7) and (8) were variously computed or estimated. Literature values<sup>7</sup> for the radiant power emissions over the spectrum of the UV-normal lamp were averaged over wavelength increments of five nanometers, such that problems from the spiked spectral bands were avoided. This data (Table 1) provided suitable values for  $I_0$  in equations (7) and (8), hence derived values of  $S_2$  and  $B_2$  are for five nanometer bandwidths.

 
 Table 1

 Sum of continuum and line spectral radiant power in 5 nm bands for the UV-normal lamp

Wavelength band	Watts	Wavelength band	Watts	
200-205	0	315-320	0.269	
205-210	0.008	320-325	0.250	
210-215	0.060	325-330	0.230	
215-220	0.165	330-335	0.756	
220-225	0.263	335-340	0.184	
225-230	0.310	340-345	0.165	
230-235	0.465	345-350	0.145	
235-240	0.948	350-355	0.125	
240-245	0.303	355-360	0.105	
245-250	1.153	360-365	0.088	
250-255	4.817	365-370	7.60	
255-260	2.660	370-375	0.061	
260-265	0.790	375-380	0.054	
265-270	2.47	380-385	0.005	
270-275	0.255	385-390	0.004	
275-280	0.523	390-395	0.064	
280-285	0.980	395-400	0.004	
285-290	0.645	400-405	3.12	
290-295	0.323	405-410	0.47	
295-300	1.408	410-415	0.004	
300-305	2.445	415-420	0.004	
305-310	0.295	420-425	0.003	
310-315	5.336	425-430	0.003	

Spectrophotometer experiments were carried out to obtain the UV absorption spectra for the photoinitiator, benzoin butyl ether (BEB) and the monomer, a commercial acrylate resin. From the spectra, coefficients  $\varepsilon_1$  and  $\varepsilon_2$  were calculated. Values at the end points of each 5nm band were averaged to give the data shown in Table 2. In Figure 2, semilog

Table 2
 Spectral absorption coefficients, benzoin butyl ether, and acrylate
 oligomer, averaged over 5nm bandwidths

$\Delta\lambda$	ε1	ε2	$\Delta\lambda$	ε <sub>1</sub>	ε2
215-220		13,920	305-310	390	79
220-225	1930	12,575	310-315	326	71
225-230	1498	12.021	315-320	295	71
230-235	1677	10,914	320-325	274	79
235-240	2573	7.592	325-330	274	79
240-245	3923	3,693	330-335	253	79
245-250	4894	1.827	335-340	243	79
250-255	5284	1.273	340-345	221	79
255-260	4820	1,210	345-350	190	79
260-265	3744	1 400	350-355	179	63
265-270	2552	1.764	355-360	148	63
270-275	2099	1 795	360-365	137	55
275-280	939	2 539	365-370	116	55
280-285	548	2 547	370-375	95	55
285-290	569	1 859	375-380	74	63
290-295	738	791	380-385	63	63
295-300	654	198	385-390	42	71
300-305	506	95	390-395	42	71
500 505	500	,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	395-400	32	71



Fig. 2. Absorption spectra of (a) benzoin n-butyl ether and (b) the acrylate oligomer

plots of  $\varepsilon_1$  and  $\varepsilon_2$  against wavelength allow the regions of photochemical interest to be discerned. The peak 330nm,  $\varepsilon_1$  max  $\simeq 250$  is postulated as the  $n - \pi^*$  transition for electrons in the carbonyl function of the photoinitiator. At shorter wavelength, 250nm,  $\varepsilon_1$ , max  $1.3 \times 10^4$ , a  $\pi \pi^*$ transition is postulated for the carbonyl electrons. Although not itself a photo-chemically reactive state, rapid decay to the significant  $n - \pi^*$  state occurs. The absorptions, 280nm,  $\varepsilon_2$  max  $\simeq 4 \times 10^3$  and 230nm,  $\varepsilon_2$  max  $\simeq 2 \times 10^4$  in the acrylate spectrum are identified as arising from substituted benzene rings and are photochemically inactive. In the region of 205nm is an intense peak arising from the acrylate group which is of photochemical relevance. However, it may be seen from Table 2 that little radiant energy is available at this wavelength. Inspection of UV absorption curves for the coating system together with the data from Table 1 reveals only three bands in which significant photochemical activity is to be expected. These are the 250-255nm, 310-315 nm and 365-370nm bands indicated as *A*, *B* and *C* respectively on Figure 2.

To calculate the inner filter factor, molar concentration of components were first computed from equation (9).

$$C_x = \frac{1000 \cdot W_x \cdot d}{MW_x} \quad \dots \dots \dots \dots \dots (9)$$

where MW is the molecular weight, W is the component weight fraction and d is the solution density. An estimate for the surface reflectance was taken as  $\rho_S = 0.04$ , which is a typical value for an air to 'glossy' polymer interface. The bottom reflectance coefficient  $\rho_B$  was arbitrarily assigned the value 0.5 in most calculations but, as will be shown later, it was allowed to vary from the maximum to minimum values (ie  $0 \rightarrow 1$ ) in one instance to investigate its influence on cure behaviour. Finally, a value of one micron was chosen as a suitable thickness which would adequately represent the local properties in the partial film layers. In calculations of  $S_2$  and  $B_2$  total thicknesses, l, in range  $1\mu$  to approximately 1000 $\mu$  were examined.

Calculation of  $S_2$  and  $B_2$  were carried out over the 250–255 nm and 365–370nm band widths, each of which falls in the vicinity of a strong absorption by the coating and embraces very intense mercury emissions. More accurately, the contribution under the whole absorption band should be summed. In Figure 3, accurate computations of  $B_2$  (solid line) are



Fig. 3. Radiant power absorption in the bottom one micron layer of film coating vs total film thickness

compared with results obtained using the 5nm band 365–370 nm, which includes an intense emission (encircled data points). For the system under study, it may be seen that the approximation provides acceptable values. Figures 4 and 5 display the results of calculations of the radiant power







Fig. 5. Radiant power absorbed in the surface one micron layer vs total film thickness: 265-260nm band

absorbed in the upper layer of coating as a function of the total film thickness at varying photoinitiator concentrations and for the respective important bandwidth. The variation in the coefficient  $\varepsilon_1$  at 250nm and 365nm is shown in the amount of light returning to the top surface. This is seen to be negligible for  $l > 2\mu$  at 250–255nm band, but is of significance for thicknesses up to  $64\mu$  at the longer wavelengths. Radiant power absorbed at the shorter wavelength is about ten times as great as that in 365–370nm band and it is reasonable to predict that the former absorption predominates in surface cure.

Some insight into the wavelength dependence of throughcure is suggested by Figure 3, where dramatic differences are found in the radiant power absorbed for the different wavebands in the bottom  $1\mu$  layer. If  $E_{\lambda}$  is the energy required just to cure the bottom layer; and hence give a through-cure and if t is the exposure time for through-cure; it follows that  $E_{\lambda} = Bt$ . When samples are submitted to a UV source in the controlled exposure device, referred to previously, the duration of exposure is inversely proportional to the conveyor velocity V.

Hence:

$$E_{\lambda} = \frac{B_{\lambda}}{V}$$
 ....(10)

A series of experiments was carried out in which the BEB—acrylate system was coated on to board at varying film weights and the velocities for just obtaining through-cure were determined using the controlled exposure equipment referred to previously. Required values of  $B_2$  were obtained from graphs, such as Figure 6, by taking the *B* values for the appropriate photoinitiator concentration and constructing *B* vs thickness plots. The results are summarised in Table 3, and clearly support the view that the photoinitiator absorption band centred at 330nm dominates in the provision of energy for through-cure.



Fig. 6. Radiant power absorbed vs weight fraction of photoinitiator: 250-255nm band

Table 3
 Dependence of energy for the just through-cure condition on wavelength

Film thickness	Conveyor velocit	v	
( <i>l</i> µ)	( <i>V</i> . fpm)	E (250–255nm)	E (365–370nm)
5.5	1100	$5.3 \times 10^{-6}$	$6.4 \times 10^{-5}$
11.0	850	$2.4 \times 10^{-8}$	$7.4 \times 10^{-5}$
20.0	596	$6.7 \times 10^{-15}$	$5.8 \times 10^{-5}$

Examination of Figures 4 and 5 show that the light absorbed in the surface  $1\mu$  in the 250–255nm band is fairly independent of the total film thickness and increases with increasing photoinitiator concentration up to the experimental limit of 32 per cent. Therefore, larger amounts of photoinitiator point to more efficient surface cure or, in practical terms, a faster throughput rate for the same energy consumption. Of course, the levels of photoinitiators are limited in reality by such considerations as cost and by its effect on the physico-chemical properties of the cured coating. Further, from an inspection of the  $B_2$  absorption curves for the 365–370nm region, Figure 7, a strong dependence on the total thickness, as well as on the BEB concentration



Fig. 7. Radiant power absorbed vs weight fraction of photoinitiator; 365-370nm band

is evident. A shallow maximum exists in the power absorption vs photoinitiator concentration plots, and this shifts to lower values of initiator concentration as the overall film thickness increases. Consequently, in thick coatings excessive amounts of photoinitiator, whilst allowing efficient curing of the film to a tack free state, may be counterproductive in so far as the overall film curing is concerned. Indeed, Krajewski and Packer<sup>3</sup> have reported experiments for which the overall degree of polymerisation in coating samples which had been subjected to an equivalent curing procedure was found to fall as the concentration of initiator was increased. They suggest that this behaviour could be interpreted in terms of ideas put forward by Hutchison and Ledwith<sup>8</sup>, who, in a conventional photochemical study, observed a maximum in the plot of polymerisation rate vs photoinitiator concentration. Hutchison and Ledwith postulated that at low photoinitiator concentrations, radical fragments were generated fairly evenly throughout the reaction system. As the concentration was increased more fragments would be formed and hence an increase in polymerisation would be predicted. However, at proportionally higher initiator concentrations, more and more radicals would be generated in the regions closer to the UV source and this non-uniform distribution would result in an overall lowering of the polymerisation rate. In the case of a film coating, this would correspond to less photochemical activity in the critical bottom layer and consequently a slower rate of through-cure.

Literature values for the reflectance coefficients in the UV of typical coating substrates were not available. It is, nevertheless, possible to explore, within the limits of the theoretical model, the effect of substrate  $\rho$  values of through-cure. As very little radiation is returned to the surface layer of all but abnormally thin films, top cure is essentially independent of the substrate reflectance. From the previously discussed conditions for through-cure, it will be realised that the energy which must be absorbed just to cure the bottom layer of film is independent of both substrate reflectance and coating thickness. Energy absorptions in the lower 1 $\mu$  layer can be calculated by substituting selected values of  $\rho_B$  into equation (8). These reflectance, to 1, for total reflection. Now from equation (10)

putting

The relative conveyor speeds for the just through-cure condition at different substrate reflectances can thus be obtained. Selecting typical values for the variables in equation (8), a calculation was carried out to probe the influence of substrate reflectance on through-cure in the important wavelength band range 365–370nm. The results, summarised in Table 4, suggest that substrate reflectance will be a significant factor in determining throughput rates in so far as through-cure is concerned.

 Table 4

 Calculated influence of substrate reflectance on through-cure

 (l = 15.75, W = 0.01, f = 0.9375) 

Substrate reflectance coefficients	Relative B (365–370nm)	Relative cure speeds (conveyor velocities)		
0	0.0053 Watts	69 fpm		
0.1	0.0058	73		
0.5	0.0079	100*		
0.9	0.0115	145		
1.0	0.0129 "	165 ,,		

\*Arbitrary reference value

Equation (10) can be used to give insight into the relative energy requirements for top and bottom cure. In a typical experiment, an approximately 12.5 $\mu$  thick film coating of acrylate monomer containing about 10 per cent BEB required conveyor exposure speeds of 1420ft per min and 989ft per min for surface and through-cure respectively. The calculated ratio of absorbed energy between the surface 1 $\mu$  layer and the bottom 1 $\mu$  layer was found to be approximately 20:1. The problem of local competition between singlet state oxygen and photoinitiator fragments for the oligomer, at the surface, has been raised before. It is proposed that the extra energy required for surface cure is needed to produce a sufficient excess of radicals to overcome the oxygen inhibition.

The theoretical treatments given here have assumed a simple two component surface coating. In practice, other radiation absorbing, but not necessarily photo-active, components may be present. Generally, these will not act in such a way as to increase the effective light pathway. It thus seems reasonable to propose that the cure behaviour of thin but complex film coatings will approximate to that predicted for comparatively thicker samples of the model system.

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## Photoinitiation problems in clear coatings\*

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#### Summary

The object of the work is to determine the general properties of photoinitiators and photoinitiating systems in UV-curable compositions in order to obtain the best adaptation to end-uses defined by coating thickness. Gel permeation chromatography, infrared

#### Keywords

Raw materials—paint additives

catalysts, accelerators, inhibitors

photoinitiation

Processes and methods primarily associated with manufacturing or synthesis

photo chemistry

drying or curing of coatings

photochemical reaction ultraviolet curing photoinitiation spectroscopy and mechanical properties of free films are used to help in determining the best combination of photoactive compounds for thin (4 to 6 microns) and thick (60 microns) clear coatings.

Properties, characteristics and conditions primarily associated with materials in general

tack

raw materials for coatings and allied products

molecular weight distribution

#### Problemes relatifs aux photoïnitiateurs en matiere de revetements non pigmentes

#### Résumé

Le but de la présente étude consiste à déterminer les propriétés générales de photoînitiateurs ou de systèmes photoînitiateurs dans des compositions capables de sécher sous irradiation UV et à les adapter à des revêtements définis par leur épaisseur. La chromatographie de perméation sur gel, la spectroscopie infrarouge et la

#### Photoinitiationsprobleme mit Klarlackfilmen

#### Zusammenfassung

Das Ziel dieser Arbeit ist die Bestimmung der allgemeinen Eigenschaften von Photoinititiatoren und photoinitierenden, Systemen in UV-härtbaren Systemen zwecks bestmoglicher Adaptation für den Endgebrauch, definiert durch Filmdicke. Um die beste Kombination

#### Introduction

#### Refs. 1, 2

Photocrosslinking of polymer systems is now a well-known method of changing rapidly the properties of a film without any contribution by thermal energy. Such systems, based for example on cinnamate or azide chemistry, were readily applied to imaging systems with considerable success. Selective insolubilisation of organic matter or the reverse effect has been applied in many areas, such as printing plate making, electronic circuitry and silk screen fabrication. However, high sensitivity could be obtained only by using high molecular weight, therefore solid, prepolymers, which could be deposited in film form only from a volatile solvent.

The reason for this is that a limited number of crosslinks is enough to transform the coating into a reticulated network.<sup>1</sup>

In this study, the concern is mainly with the rapid transformation of a solventless liquid composition into a non-tacky solid coating, by means of ultraviolet radiation. détermination des propriétés mécaniques de films libres sont mises à contribution pour trouver la meilleure combinaison de composés photoactifs en films minces (4 à 6 microns) ou épais (60 microns) en absence de pigments.

photoaktiver Verbindungen für dünne (4 bis 6 Mikrons) und dicke (60 Mikrons) Klarlacke bestimmen zu konnen, werden Gelpermeabilitäts-Chromatographie, Infrarot-, Spektroskopie, sowie die mechanischen Eigenschaften freier Filme benutzt.

UV-cured styrene and polyester-based formulas were first used in the wood industry in 1968<sup>2</sup>. The photodissociation of benzoin ether derivatives initiated the co-polymerisation of styrene with the fumarate or maleate double bonds of the polyester, resulting in a very hard and durable coating. Surface polymerisation, however, was inhibited by air. A partial solution to this problem was found by adding wax, which would retard oxygen diffusion by floating to the surface during the first stage of hardening. Glossy and scratch-resistant varnishes could then be obtained only by sanding and polishing the surface.

The next important step occurred at that time in the printing industry. The need for inks capable of drying fast enough to suit new high-speed presses was becoming more and more urgent. After a period of development by trial and error, UV-curable offset printing inks capable of drying at a speed of 500 metres per minute (30 km/h) were used industrially with a quality much superior to that obtained with convential inks.

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

An entirely new approach had to be used to develop binders and photoinitiators suited to offset printing:

Coating thickness is so thin that a means had to be found to overcome the inhibiting effect of air on polymerisation.

Very reactive polymerisation unsaturated monomers had to be used.

Photoinitiators had to be adapted to the spectra given by commercially available UV lamps and pigments.

Ink rheology and offset lipophilic/hydrophilic balance had to be adapted to existing presses.

These problems were very soon solved and UV-drying of inks became a commercial reality.

However, the advantages of curing with UV are such that replacements for epoxy, melamine or urethanne-based coatings are now actively sought. The first results which were obtained from the type of binders and photoinitiators which had been developed for other purposes were at first disappointing: the films were brittle and highly discoloured, adhesion to a given substrate was often poor and so on. Therefore, a study of the influence of binders and photoinitiators on the mechanical properties of coatings had to be initiated in order to determine the best combination of ingredients for each specific application. The object of this work was to approach the problem of the photoinitiating system in a general way, without taking the substrate into account.

## General characteristics of the unsaturated components

#### Introduction

Refs. 3, 4, 5

Few unsaturated compounds are capable of polymerising fast enough to suit the requirements of an UV-cured composition.

Most monomers and prepolymers used today are based on the acrylic unsaturation. They may sometimes be blended with acrylamide or methacrylic acid derivatives; sometimes N-vinylpyrrolidone is used as a reactive diluent.

UV-curable compositions have also been based on other principles, for example the photoinitiated chain addition of mercaptans to allylic compounds<sup>3</sup>, cationic initiation of epoxy compounds<sup>4</sup>, photoliberation of acid catalysts in the presence of aminoplasts<sup>5</sup>.

The development of most of these systems has been impeded by problems of cost, low reactivity or of potlife.

#### Binders

Ref. 6

Most acrylic binders have relatively low molecular weights, most often between 500 and 3,000. Higher molecular weights are rarely encountered, if only because of the difficulty of bringing the viscosity down to practicable values by means of reactive diluents.

Binders usually belong to one of the following categories:

the reaction product of an hydroxylated acrylic monomer, such as hydroxyethyl acrylate, with an equimolecular quantity of a di-isocyanate. The resulting monoisocyanate is then condensed with a hydroxy-functional polyester or polyether;

the direct esterification of acrylic acid with a hydroxyfunctional acrylic copolymer, a polyester or an epoxy compound.

One example of an acrylic terminated polyester is the co-esterification product of adipic acid, hexane diol, trimethylolpropane and acrylic acid (Fig. 1).



This structure is in fact idealised. In reality, it more or less agrees with the mean composition of the reaction product described in a published procedure<sup>6</sup>.

#### Photoinitiators

Refs. 7-13

Some very active polymerisation photoinitiators are now available commercially.

The best-known of them is benzophenone. This cannot be used in the presence of styrene, because of triplet quenching  $(E_T(BZO) = 68.5 \text{ kcal}.M^{-1} E_T(STY) = 61 \text{ kcal}.M^{-1})$ ; photoexcited benzophenone and its analogs readily initiate the polymerisation of acrylic derivatives.

Photoexcited benzophenone is a well-known hydrogen abstracter, especially in the presence of alcohols and ethers. Hydrogen transfer in the  $\alpha$  position to the oxygen results in the formation of two radicals; at least one of them rapidly reacts with an acrylic monomer, thus initiating polymerisation. It is thought that the semibenzopinacol acts as a terminator by chain transfer<sup>7</sup>.

Tertiary aliphatic amines have the interesting possibility of increasing the photoreactivity ("photoactivation") of benzophenone or its analogues to the extent that thin coatings can be cured at high speed in air to a non-tacky surface. This effect has been attributed to the formation of a charge-transfer complex at diffusional rates followed by proton transfer<sup>8</sup>. The resulting electron-rich  $\alpha$ -amino radicals can then add rapidly to electron-poor acrylic unsaturations. Complex formation with amino compounds efficiently competes with energy transfer to oxygen, and this greatly contributes to improved surface cure (Fig. 2).

Examples of tertiary aliphatic amines which are actually used in practice are the ethanolamines, which can only be used at low concentration because of lack of stability in solution, and aminoacrylates, which have the advantage of being copolymerisable (Fig. 3).



Fig. 2. The benzophenone-amine combination: An example of a bimolecular dissociating photoinitiating system



Other amino compounds, such as Michler's ketone, behave in a more complex manner. They are usually characterised by an intense charge transfer absorption band (Fig. 4), often located in the vicinity of the intense 366 nm emission band of a mercury vapour lamp. It is postulated that the lowest excited state in the triplet manifold,  $n-\pi^*$  in character in non-polar solvents, sensitises benzophenone through a triplet exciplex, where hydrogen transfer from one of the methylamino groups occurs<sup>9</sup>.



Fig. 4. Charge transfer absorption of aromatic carbonyl-amines

Other compounds are capable of forming radicals through a monomolecular process (Fig. 5). The benzoin ether group has been thoroughly studied; it has been established that radical formation occurs with bond scission  $\alpha$  to the benzoyl group<sup>10</sup>. A recent addition to this family is benzil dimethyl keta<sup>11</sup>. The auto-oxidisable benzilic hydrogen atom of benzoin ethers is replaced with a methoxy group. The result is improved stability in the presence of monomer.



Fig. 5. Monomolecular dissociating photoinitiators Benzoin derivatives group

 $\alpha$ -chlorinated acetophenones (Fig. 6) are known to liberate chlorine radicals upon photolysis<sup>10</sup>. In the initial stage of curing, most of them react with acrylic unsaturated monomers, therefore initiating polymerisation. In the last stage, when monomer concentration is low, hydrogen abstraction with formation of hydrochloric acid is favoured. This may be used to promote a subsequent acid-catalysed condensation<sup>12</sup>.



O-acylated oximinoketones (Fig. 7) as a class were discovered recently. They readily fragment upon photolysis into two reactive radicals and a nitrile<sup>13</sup>.



Fig. 7. Monomolecular dissociating photoinitiators o-acylated oximinoketones group

#### Effect of conditions of cure on polymerisation

#### Introduction

Ref. 14

Chong has studied from a theoretical point of view the effect of oxygen on the photoinduced polymerisation of a printing plate model<sup>14</sup>.

It was concluded that because of the reaction of oxygen with propagating radicals, producing unreactive peroxy radicals, oxygen concentration is the highest at the coating/air interface. There is a competition between the propagating reaction of polymerisation and the reaction of free radicals with oxygen. The ratio of these rates is dependent on the reactivity of the monemers, the oxygen diffusion rate, the photoinitiator concentration and the light intensity.

It is, therefore, important that all factors which define light intensity at different levels through the film should be considered in formulating an UV-curable composition. It can be expected that:

the degree of surface curing will affect surface tack and gloss;

the degree of below-surface curing will affect scratch resistance and surface hardness;

the degree of interface curing will generally affect adhesion to the substrate.

Thus it can be concluded that the most difficult thing to achieve is good surface curing. This problem has been solved to a certain extent by using high-intensity focussed medium pressure mercury vapour lamps and high photoinitiator concentrations. It is expected that the high photon dose-rate combined with increased film optical density produce such a high concentration of radicals that the oxygen inhibiting effect is swamped.

#### Molecular weight distribution

In what manner will such a procedure influence film properties? A simple experiment can be performed in order to give a partial answer to this problem (Fig. 8).



Fig. 8. Gel permeation chromatography of a 5 per cent solution of benzil dimethyl ketal in 2-ethyl hexyl acrylate photolysed in the presence of air (0.5mm thickness, 80 W/cm lamp; HQ = hydroquinone is added to stabilise the solvent)

A 5 per cent solution of a very efficient photoinitiator, benzil dimethyl ketal, in 2-ethyl hexyl acrylate, a monofunctional monomer with low volatility, is poured in a shallow aluminium cup to a 0.5 mm depth. This concentration of photoinitiator is chosen so that all UV-emission bands between 254 nm and 405 nm are absorbed, although calculation shows that 254 nm and 315 nm radiation will be absorbed near the surface, whereas 366 and 405 nm radiation will be capable of penetrating in depth.

The cup is passed a certain number of times under the focussed 80 W/cm lamp at constant speed.

Gel permeation chromatography of the product obtained after two passes at 0.13 m.s.<sup>-1</sup> (unbroken line) under the lamp shows the presence of a low molecular weight polymer with a maximum at a molecular weight of approximately 6400 and about 35 per cent residual 2-ethyl hexyl acrylate.

The total number of Einsteins absorbed per litre of solution can be estimated at about 1.7.10<sup>-2</sup>. The corresponding amount of consumed monomer is about 3.3 Moles.1-1. Therefore, to each photon corresponds 190 molecules of monomer. Considering that poly (ethyl hexyl acrylate) with a degree of polymerisation (DP) of 190 would have a MW of 35,000, such a polymer would appear at 17 GPC counts. This, in fact, is the highest molecular weight obtained (see graph). One may perhaps speculate that peroxy radicals formed by the addition of oxygen to propagating radicals are capable of abstracting hydrogen atoms: the polymerisation would then initiate again from the site of abstraction. This would result in high quantum yields, but low molecular weights. Supporting this hypothesis is the presence of a small quantity of gel observed in the diluted solution of irradiated material.

Preliminary experiments indicate that molecular weight is increased by irradiating in the absence of air through a transparent Mylar film, whilst the presence of gel in the diluted solution is no longer apparent. After irradiating the film at ten times the previous dose (0.17 Einstein/litre), the monomer is converted mostly to low molcular weight polymer.

It would be expected that most polymers produced in the conditions which have just been described will have poor mechanical properties. Linear homopolymers of low molecular weight monomers are usually viscous liquids.

The only way to produce hard and non-tacky UV coatings consists in introducing polyfunctional binders and monomers.

Examples of polyfunctional monomers are polyol acrylates, such as pentaerythritol triacrylate or hexane diol diacrylate.

#### Surface tack and double bond conversion

Refs. 15-17

Surface tack is usually evaluated by rubbing talcum powder on to the surface of an irradiated thin coating of varnish. The surface is considered to be dry if the talcum powder can be wiped off without leaving a mark.

A series of different photoinitiators can be compared in a standard solution of binders in a bifunctional monomer, diethylene glycol diacrylate, in the presence or in the absence of an amino acrylate (Fig. 9).

Ebecryl 592 is one of UCB's range of UV-curable resins.

*MHD 19* is a polyester acrylate which has been described earlier<sup>15</sup>.

Unshaded columns refer to unactivated formulae, whereas the shaded columns refer to amine-activated formulae.

DEAP is diethoxyacetophenone from Union Carbide. Irgacure 651 is benzil dimethylketal from Ciba. PPBO is 1-phenyl-1, 3-propanedione-2-0-benzoyloxime<sup>13</sup>, and Trigonal P1 is 2,2,2-trichloro-4-t-butyl acetophenone from Akzo. Sandoray 1000 was supplied by Sandoz.

These results show that in general, *Ebecryl 592* is much more reactive than *MHD 19*. Only in the case of a single photoinitiator is the former more reactive. The best results were obtained with the three last photoinitiators.

A dramatic increase of surface reactivity is observed in the presence of an amino acrylate activator. Benzophenone, which has poor reactivity when alone, becomes one of the most efficient photoinitiators. The improvement is especially dramatic with polyester acrylate *MHD19*; the reactivities become comparable.

An objective way of determining the extent of polymerisation in an UV-curable film is to measure the degree of conversion of the double bonds by an infrared method.

A 4 micron coating is applied to a sheet of reflective metal, such as tin plate. The disappearance of the  $804 \text{ cm}^{-1} \text{ CH}$ bond resonance of the acrylic group can be followed as a function of the dose of UV incident on the film (Fig. 10).

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Fig. 9. Effect of photoinitiator and added amino acrylate on surface cure (30 per cent prepolymer in DEGDA with 5 per cent photoinitiator)



Fig 10 Relationship between double bond conversion and dose photoinitiating system; benzophenone + Uvecryl P104 (coating thickness: 4 microns)



Fig. 11. Relationship between double bond conversion and benzophenone concentration (coating thickness: 4 microns)

This set of graphs was obtained by increasing the concentration of benzophenone in a solution of polyester acrylate in a difunctional monomer, hexane diol diacrylate, and in the presence of an amino acrylate activator, *Uvecryl P 104*.

In the absence of benzophenone, polymerisation proceeds to some extent, but soon stabilises at a plateau value of 20 per cent double bond conversion. Initiation, in this case, may be due to abstraction of amino compound hydrogen by photoexcited monomer, as it has been suggested in the case of the amine-sensitised polymerisation of methyl methacrylate<sup>16</sup>.

With increasing concentration of benzophenone, the initial rates and plateau values increase until a maximum effect is reached in the vicinity of a 4 to 9 per cent concentration.

The effect of concentration is seen more clearly in Fig. 11, where the degree of conversion is shown as a function of benzophenone concentration. Each line joins the points which were obtained by irradiating with the same dose of ultraviolet light.

Degree of conversion is put as a function of the square root of benzophenone concentration in order to magnify the effect of low photoinitiator concentration and also because of the general relationship between the square root of photoinitiator concentration and the rate of photoinitiated polymerisation (at least under stationary conditions).

It can be readily seen that the optimum concentration of benzophenone lies between 5 and 9 per cent.

The unity of dose used in this study is purely relative; it relates to a single pass at a speed of  $1 \text{ m.s}^{-1}$  under a focussed 80 W/cm mercury vapour lamp. Correlation with the actual energy absorbed by the film is possible only if one takes into account the emission spectrum of the lamp and the absorption spectrum of the irradiated coating.

In fact, it can be concluded from these results that up to a certain concentration, the main use of the photoinitiator is to absorb the incident light.

A 4 micron coating containing 9 per cent benzophenone absorbs 56 per cent of 257 nm incident light. At wavelengths of 300 and 313 nm, absorption values are negligible. It can perhaps be speculated that the higher the concentration of photoinitiator, the more of the radicals produced by a given quantity of photons are concentrated near the surface. Cage recombination of radicals becomes more favourable, therefore lowering the overall efficiency. Still another effect may be simply the dilution effect of non-polymerisable material. Surface and mixing effects have been studied elsewhere<sup>17</sup>.

This diluting effect could be reduced to some extent by using a copolymerisable photoinitiator. An example is *Uvecryl P 36*, an acrylated benzophenone derivative (Fig. 12).

On a purely weight to weight basis, Uvecryl P 36 is less reactive than benzophenone. A Uvecryl P 36/benzophenone ratio of 2.5 is usually recommended in order to obtain the same degree of surface reactivity, as determined by the talcum powder method. This roughly corresponds to the actual concentration of the benzophenone group in the product.



Fig. 12. Relationship between double bond conversion and dose photoinitiating system: Uvecryl P36 + Uvecryl P104 (coating thickness: 4 microns)

It is noteworthy that even a high concentration of photoinitiator does not tend to yield constant conversion plateau values as in the case of benzophenone.

The optimum concentration of photoinitiator which results in the highest possible degree of conversion is between 10 and 20 per cent (Fig. 13). This is just double the optimum concentration of benzophenone previously determined.

It can, therefore, be concluded from these results that up to approximately 16 per cent concentration, the diluting effect of *Uvecryl P 36* is negligible, at least from the point of view of double bond conversion.

The main factor to be considered is how to avoid using needlessly large quantities of photoinitiator, although this is rapidly determined by the formulator by subjective testing, such as the determination of surface tack.

Unactivated benzophenone, as has been said earlier, is capable of initiating polymerisation with the aid of light in thin film conditions, although a non-tacky surface is difficult to obtain.

This is easily explained by the lowest curve in Fig. 14. It can be seen from the results of two distinct experiments that the conversion plateau value is in the vicinity of 50 per cent.

However, traces of an aminoacrylate compound significantly alters this picture. The plateau value increases considerably, up to 75 per cent. Surprisingly, when the dose of UV is less than about 1 psm<sup>-1</sup>, the amine acts as an inhibitor. This inhibiting effect is progressively reduced with increasing concentration of *Uvecryl P 104* until an equivalency is reached in the vicinity of 5 per cent concentration. The highest initial rates and conversion plateau values are obtained for a concentration of amine of 25 per cent, and then decreases when the concentration is increased even more. These results suggest a complete modification of initiation mechanism.


Fig. 13. Relationship between double bond conversion and Uvecryl P36 concentration (coating thickness: 4 microns)



Fig. 14. Relationship between double bond conversion and Uvecryl P104 concentration

Photoinitiator: Benzophenone (coating thickness: 4 microns)

#### Mechanical properties of UV-cured free films

Only very thin coatings in the 4 to 6 micron range have been considered so far. They are, to a certain extent, representative of varnishes which are used today to lacquer paper, cardboard or metal. In other applications, where high gloss or hardness is required, thicker coatings become necessary. The degree of surface tack is no longer the most important degree of cure criterion. General hardness must also be evaluated. It becomes more difficult to achieve a good degree of flexibility because of the highly crosslinked structures which are obtained. A minimum degree of cure at the coating/ substrate interface is necessary in order to obtain good adhesion.

Flexibility, hardness and so on are difficult to evaluate, because they are often affected by the nature of the substrate. One way of avoiding this is to prepare free films of minimum thickness and to measure their mechanical properties with a dynamometer.

60 microns thick by 1 cm samples of films prepared from irradiated solutions of *MHD* 19 polyester acrylate in a monofunctional monomer, phenoxy ethyl acrylate (*Ebecryl* 110), are pulled at a constant rate of 5 cm/min with an *Instron* dynamometer. Elongation and stress at break are measured, as well as the modulus of elasticity, Young's Modulus. Most of the films were prepared from a hard, reactive polyester acrylate, which tends to give hard and brittle films. This effect is lessened with the use of a monofunctional monomer. Very flexible binders were not available at the time of the study. Elongations at break are usually in the 5-16 per cent range. Stress at break varies from 15 to 150 kg/cm<sup>2</sup>.

The general mechanical properties can best be evaluated by plotting Young's Modulus E as a function of dose (Fig. 15).

The use of a monoacrylate as a monomer instead of hexane diol or diethyleneglycol diacrylate results in much lower reactivity. A minimum of 5 psm<sup>-1</sup> is necessary in order to detach the films from the substrate on which they were drawn in liquid form.

In this graph two monomolecular dissociating photoinitiators are compared at two different concentrations.

It will be observed that Young's Modulus increases substantially with the dose. By increasing the concentration



Fig. 15. Effect of monomolecular dissociating photoinitiators on Young's modulus (free film thickness: 60 microns. Solution of 48 parts MHD 19 in 50 parts Ebecryl 110)

to 5 per cent, no changes in the properties are evident with *Sandoray 1000*, but with *Irgacure 651* higher moduli are obtained.

These results can be readily explained by comparing the absorption spectra of the two photoinitiators. Differences in the 200-280 nm region are not important, but in the 298-313 nm region, where intense mercury emission lines are present, the ratio of molecular absorption coefficients is around 50, in favour of Sandoray 1000. One of the reasons why nonamine activated Sandoray 1000 is often more reactive with respect to surface curing than Irgacure 651 may be greater efficiency in absorbing UV. It can easily be calculated that 90 per cent of incident 313 nm light is absorbed within the first third of the 60 micron thick film in the case of Sandoray 1000 even with 2 per cent of photoinitiator. With 5 per cent Irgacure 651, approximately 0.5mm of film would be necessary to absorb 90 per cent of the light. It would be expected that mechanical properties of films cured with Sandoray 1000 will start to decrease only with much lower photoinitiator concentrations.

Thick films tend to be cured more uniformly with *Irgacure* 651. This is why it is important to consider the degree of absorption of photoinitiators in the 300 nm region when thick films are to be cured more or less homogeneously.

Benzophenone is quite similar to *Irgacure 651* from the absorption spectrum point of view. Therefore, the observation that an increase in photoinitiator concentration decreases the value of Young's Modulus is quite surprising (Fig. 16).

Therefore, causes other than the shape of absorption must be sought. However, results obtained with *Irgacure 651* 



Fig. 16. Effect of a bimolecular dissociating photoinitiator on Young's modulus (free film thickness: 60 microns)

were better in all cases, and with all other factors being equal it may be expected that it will yield harder films than with other photoinitiators. This has often been confirmed in actual practice.

The effect of Uvecryl P 104 is shown in Fig. 17.



Fig. 17. Effect of amino acrylate on Young's modulus (free film thickness: 60 microns)

Non-amine activated benzophenone is not very reactive. Only 2 per cent of amine is enough to halve the minimum dose of UV necessary to obtain minimal mechanical properties.

It was observed that in thin film curing, the optimum concentration of *Uvecryl P 104* was between 10 and 20 per cent. Increasing the amine concentration increases Young's Modulus at low dosage, but decreases it at high dose. The optimum concentration of amine is, therefore, much lower in thick films. This result is interpreted as a plasticising effect of the mono acrylate *Uvecryl P104* by tertiary alkylamines.

This contributes to decreasing mechanical properties without affecting double bond conversion.

#### Conclusions

The results discussed show that free film mechanical properties can be used to characterise the effect of the photoinitiating system, when infrared measurements of double bond conversion become useless. As a rule, Young's Modulus, which is often used to characterise a coating's general mechanical properties, is seen to be closely related to the dose of UV absorbed by the film. It is suggested that when coatings are evaluated, a single formulation should be tried at a range of UV doses which might be encountered in actual practice. Lamp manufacturers' specifications vary so much that different wavelength distributions, intensity fall-off at the lamp ends, secondary infrared effects and so on may be encountered by different users. UV-curable formulations then should have a built-in capacity to adapt themselves to different curing situations.

#### Acknowledgments

The author is grateful to Dr A. Vrancken for his constant help and encouragement. Assistance from Mr M. Demartin in the synthesis of prepolymers and Mr J. H. M. Hoebeke in the determination of the mechanical properties of free films is also acknowledged. Discussions with Dr W. Demarteau, have been helpful in determining the final form of this work.

#### Trade names

Uvecryl and Ebecryl are registered trade names for UCB's range of UV and EBC photoactive compounds, prepolymers and monomers.

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### Erratum

#### From gold to diamond. OCCA 1968-1978

It is regretted that the following paragraph which should have appeared after the first paragraph under the heading "The Divisions, Sections and Branches" on page 143 of the May issue of the *Journal* was inadvertently omitted: "In April 1976 our youngest Section, the *Ontario Section*, was inaugurated with due celebration after a brief period as a Branch of the London Section, whose Chairman and comnittee colleagues had energetically stimulated its formation."

# Next month's issue

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

The UV curing of acrylate materials with high intensity flash by R. Phillips

Parameters in UV curable materials which influence cure speed by A. van Neerbos

The UV curing behaviour of some photoinitiators and photoactivators by M. J. Davis, J. Doherty, A. A. Godfrey, P. N. Green, J. R. A. Young and M. A. Parrish

The use of differential scanning calorimetry in photocuring studies by A. J. Evans, C. Armstrong and R. J. Tolman

The place of business in society by Sir John Methven

# Section Proceedings Hull

#### Exterior wood finishes

The Fifth Ordinary Meeting of the 1977–78 session was held at the George Hotel, Land of Green Ginger, Hull on Monday 6 February. A lecture entitled, "Exterior wood finishes" was presented by Mr P. Whiteley of the Building Research Establishment, Garston.

The work of the Building Research Establishment over many years, on the protection of exterior joinery was described. It was estimated that approximately 25 per cent of timber in houses today is affected by rot which can be traced to the high sap content of new wood and high humidity. Dimensional changes in wood can be as high as 10 per cent; normally the changes are of the order of 2–3 per cent. Paint defects can be traced to poor quality primers, undercoats of inferior flexibility and hard inflexible top coats. Mr Whiteley commented that emulsion polymer primers can be formulated which perform as efficiently as solvent based primers. However, care must be taken to keep the water soluble content and the pigmentation as low as possible commensurate with an acceptable primer quality; British Standard 5082 is the culmination of the B.S.I. primer studies.

With respect to wood preservatives the lecturer observed that preservatives are criticised for causing loss of drying of top coats and for colour staining. The fault is often the poor drying of the preservative; again, care in formulation is of paramount importance.

Wood staining has a shorter protective life than paint; approximately 3 years; colour fading and dirt retention are the main disadvantages. However, compared with a poor painting system it has its recommendations.

Practical painting tests on window frames, doors etc. are the best methods for assessing performance. In this way, paint failure can often be traced to a basic design fault, e.g. sharp edges, bad joints, vertical cracks in putty etc. The Building Research Establishment have a painting test building constructed with wood cladding, window frames and cement block sections. These are repeated on both the North and South face. Different painting systems have been applied and are under observation.

Finally, Mr Whiteley outlined some pointers towards methods of improving the performance of exterior wood finishing.

These were:

Factory application of primer coat, Well formulated preservatives, Good quality/flexible undercoats, Top coat of good durability essential.

Mr P. Nunn proposed the vote of thanks after a lively dissussion period. Fifteen members and visitors attended.

# Manchester

#### Student works visit

Donald Macpherson & Co. Ltd, Radcliffe, Lancashire, was the 204

venue for this visit by 23 Student Members on Wednesday 8 March 1978.

Situated on the banks of the industrial River Irwell, this former Prisoner of War Camp has been transformed into a large modern paint factory and is the main production unit for the fourth largest U.K. paint manufacturer.

Messrs. Ford, Hughes and Lord were hosts to the members who were entertained during the afternoon by an introductory talk and subsequent visits to the various sections of the factory. These included the Decorative, Industrial, Resin Plant and Packaging areas. Particularly impressive was the latter where three quality checks were performed on products destined for those branded stores that are a household name.

The Manchester Section committee are indebted to the Directors of Donald Macpherson for enabling this interesting works visit to take place.

#### Lung cancer mortality of workers in chromate pigment manufacture : An epidemiological survey

The final lecture of the present session was presented by Dr Joan M. Davies, PhD, of the Institute of Cancer Research and Mr M. Hobbs, BA, of I.C.I. Ltd, Organics Division. The venue was the University of Manchester Institute of Science and Technology, on Monday 13 March 1978. A Section record attendance or more than 150 members and visitors was recorded.

Dr Davies delivered the epidemiological part of the lecture and initially made reference to the three surveys that have been conducted in the U.S.A., U.K. and W. Germany respectively. She then described her work in the U.K. based on surveys in three factories referenced A, B and C. Classification was based on type of exposure, length of service and starting date; further influencing factors were smoking habit/area mortality rates and type(s) of chromates manufactured.

Mr Hobbs then described, with the aid of colour slides, the improved handling techniques developed for use with chromate pigments. In his opinion, the chromate pigments, especially the lead types, would enjoy increased usage in strictly controlled manufacturing, packaging and end-user operations.

The 75 minute lecture was followed by a lively and varied question time of 30 minute duration. A general conclusion reached was the apparent increased toxicity of zinc chromate when compared with lead chromates. Mr I. Ford proposed a vote of thanks for this excellent and topical lecture.

# Scottish

F.B.W.

#### Eastern Branch

FDR

#### Printing inks for paper

A meeting of the Eastern Branch of the Scottish Section was held at the King Malcolm Hotel, Dunfermline on 18 January, when Mr G. H. Hutchinson of Croda Inks Ltd gave a lecture entitled 'Printing inks for paper – present and future developments'.

The paper covered a review of developments in printing inks for papers and boards with reference to the major printing processes, offset lithography, letterpress, flexography and

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We recommend standard oil-resinous products as well as such sophisticated ones as : vinyl, chlorinated rubber, epoxy, epoxy-bituminous, polyurethane, epoxy-ester, solvent free paints.



#### 1978 (6) SECTION PROCEEDINGS

gravure, drawing attention to any ink and paper relationships that had a bearing on ink drying and printability. Developments were discussed in relation to the four main methods of printing ink drying, namely, penetration, oxidation, precipitation and evaporation; this was followed by an outline of more recent progresss in radiation drying technology including ultraviolet curing inks, electron beam curing inks and the latest trends in the development of offset lithographic printing inks which dried by infrared radiation.

Factors influencing progress in printing ink technology had been the continuing demand for higher production rates on faster running printing presses and the need for inks to print on an increasing variety of new papers, boards, plastic films and foils. Also, there have been other influential factors, such as the problems of raw materials shortages, attention to environmental pollution problems, the Health and Safety at Work *etc* Act 1974 and all these factors had stimulated research into new types of water-based flexographic and gravure inks, low odour and low temperature drying heatset web offset inks, and ultraviolet curing inks. Molecular models constructed from pipe-cleaners were used to demonstrate the chemistry and the drying properties of these products. More recent topics of research included powder-set inks and the non-impact process of ink jet printing.

Some University research was contributing to progress and the fundamental work at the University of Bath on ink drying by hot air jets and the de-inking of paper project at the University of Surrey was mentioned.

Finally, it was suggested that there should be more cooperation between the ink and paper manufacturers so as to obtain a better understanding of the performance of printing ink on paper. More fundamental knowledge was needed of the factors controlling smoothness and absorbency of paper which greatly influenced the printability and drying performance of printing inks.

#### T.M.

# Transvaal

#### Corrosion of metals

A meeting of the Transvaal Section was held on 23 February, 1978 at the South African Bureau of Standards, Pretoria.

The Chairman, after announcing the election of Mr P. A. J. Gate as South African Vice President in succession to Mr D. J. Pienaar introduced the speaker for the evening, Dr B. G. Callaghan of the National Building Research Institute.

Dr Callaghan delivered a paper entitled "The corrosion of metals in the building and construction industries."

The high cost of corrosion to South Africa was highlighted, it being estimated that this could amount to R300 million per annum. After dealing with the mechanism of corrosion, Dr Callaghan elaborated on the importance of correct design to avoid the jointing of two metals which are capable of forming a bimetallic cell. On the other hand, the electrochemical nature of the corrosion process can be put to good advantage by, for example, covering steel with a thin layer of zinc as in the case of galvanised steel.

The effect of the environment on corrosion was dealt with in some detail and a comprehensive survey was given on corrosion rates for various metals and metal coatings at sites in Pretoria, Durban, Cape Town and Walvis Bay. The sites included marine, industrial and rural environments. A survey was given of the metals used in the building industry, namely conventional steel, low-alloy steels, copper and copper alloys, aluminium and its alloys, stainless steel, lead and zinc.

The importance of adequate surface preparation before application of paint systems was highlighted.

The lecture was illustrated with alarge number of slides including a number of electronmicrographs showing in particular the devastating effect of entrapped concentrations of corrosive salts.

After a lively question time, a vote of thanks was proposed by Mr G. Munroe in which he expressed the view that the level of this lecture was such as to enhance the reputation of South African technology.

In closing the meeting, the Chairman thanked the South African Bureau of Standards for sponsoring the meeting.

# West Riding

P.A.J.G.

#### Fundamentals of modern emulsion paint formulation

A meeting of the West Riding Section was held on Tuesday 7 February 1978 at the Mansion Hotel, Leeds. Mr J. Clark of B.T.P. Tioxide Ltd presented the lecture which dealt with emulsion paint formulation.

Mr Clark aimed his paper at the more junior members of the Section. He talked about the components in a modern emulsion paint and discussed the factors affecting the choice of emulsion, pigment, extender and dispersant. The ways in which the properties of the various raw materials affect the finished product were discussed in a fundamental but detailed manner.

The lecture attracted a very large audience and was very well received. Due to the interest generated, the lecture may be published in due course as a Student Review. Mr C. Butler thanked Mr Clark for his excellent lecture and B.T.P. Tioxide Ltd for the hospitality provided.

#### Anticorrosive primers based on inert pigments and ferrites

On Tuesday 7 March, a meeting of the West Riding Section was held at the Mansion Hotel. Mr Schleusser of Bayer A.G. presented a paper dealing with anticorrosive primers and ferrites.

Mr Schleusser pointed out for a primer to achieve the desired anticorrosive effect several compromises must be made. It is thought that corrosion is an effect of the permeability of water and oxygen. For this reason paint systems are needed to reduce permeability. The important parameter of a paint system is its pigment volume concentration (P.V.C.). At the pigment/binder interface, if no water forms then the anti-corrosive property of the pigment increases up to the critical P.V.C. level.

Slides were then shown demonstrating how high pigmentation gives capillary action which leads to corrosion. It was shown that the effect occurs with both active and non-active pigment, and the higher the P.V.C. the worse is the corrosive effect. Active pigments are thought to prevent corrosion less than inactive pigment. However, with a low P.V.C. and low hiding power, degradation due to ultraviolet light may occur. The influence of the binder is said to be often greater than that of the pigment, regardless of whether the pigment is active or inactive. Long oil alkyd resins with low viscosity have been found best for anticorrosion. Slides illustrating the effect of various binders on anticorrosion (salt spray tests) were shown. The role of an inactive pigment or extender is to improve the anticorrosive properties of the system particularly when combined with a good active pigment. Mr Schleusser stated that for the pigment to produce its best effect it must be well dispersed in the system. If large pigment agglomerates are present, problems with permeability will occur and this is where corrosion will start.

Mr Schleusser went on to introduce ferrite compounds. These are the newest development in the anticorrosive field. They are active pigments. There are two types, calcium and zinc ferrites and they both act as active anticorrosives by forming metal salts at the metal/binder interface.

#### INFORMATION RECEIVED JOCCA

These new pigments exhibit their maximum properties near their critial P.V.C. They may be combined with inactive pigment and even small additions of the active pigment were shown greatly to improve corrosion protection. Ratios of 70:30 and 50:50 were regarded as the optimum but even with only 10 per cent ferrite, good results may be achieved. The lecturer suggested that ferrites do not give as high an anti-corrosive effect as zinc chromate, but zinc chromate is very bad for pollution.

Mr Schleusser concluded that active pigments function by a different mechanism from inactive ones. This does not mean that they are necessarily better especially if they are not tested at the same P.V.C.

A lively question time followed the lecture.

R.A.C.C.

## Information Received

#### Miox Ltd

Kaerntner Montanindustrie GmbH, Austria, has announced that Miox Micaceous Iron Oxide pigment will be distributed in the U.K. by Miox Ltd of 162 High Street, Tonbridge, Kent.

#### Polypropylene in paint

The use of polypropylene as a reinforcement in paints of various kinds is being studied by Plasticisers Ltd, the largest U.K. producer of polypropylene fibres and filaments. The study is part of a major investigation by the company's Research and Development department into new applications for polypropylene in a broad range of products. Polypropylene can be admixed with paint in the form of very short (0.5mm) chopped lengths, in deniers as fine as 30 microns diameter or in larger particles depending on the effect required. Such mixtures can result in new textures and threedimensional effects, good acoustic and thermal insulation properties, improved weatherability and good non-slip properties.

#### **Testing agreement**

The Yarsley Technical Centre of Redhill, Surrey and U.S. Testing Company Inc., of New Jersey, U.S.A. have agreed to collaborate to enable American amd British companies to test materials and products for compliance with each other's national standards. Both companies have comprehensive laboratory facilities for the testing and analysis of products and materials.

#### New phosphate plant without effluents

In August 1977 Otto Dürr Anlagenbau GmbH handed over to Bauknecht Gmbh in West Germany, a paintshop which is the first in the world to have a complete water recycling system integrated. With the new system, approximately 90 per cent of the fresh water volume required is recycled and 60 to 80 per cent of the chemical products necessary for cleaning within the pre-treatment plant are retrievable.

#### Dow Badische takeover

The Dow Chemical Company has reached an agreement in principle for BASF to acquire full ownership of their 50/50 joint venture, Dow Badische Company, a manufacturer of chemicals and man made fibres



The chemical aeresol mixing/blending plant which D.H. Industries have supplied to the National Oil & Chemical Marketing Co. Ltd in Nigeria

in America. With the purchase of Dow's interest, BASF is positioned to broaden its chemical business and strengthen its earnings potential in North America. Most of Dow Badische's technology is based on BASF processes and know-how.

#### New test house

A new £250,000 test house is being built by Simon-Rosedowns Ltd of Hull to expand and modernise its research, testing and training facilities. The company designs and equips plants for the extraction and processing of vegetable oils and fats. The building will house a new laboratory and mechanical and chemical test bays, thereby providing a training and familiarisation area for use by customers' and the company's personnel.

#### **Reduced installation problems**

A chemical aerosol mixing/blending plant has been supplied by D.H. Industries Ltd to National Oil and Chemical Marketing Co. Ltd, a company jointly owned by the Federal Government of Nigeria and the Royal Dutch Shell Group.

To minimise installation problems onsite, D.H. Industries designed the chemical plant as a modular packaged unit which would be shipped to Nigeria exactly as if it were a standard I.S.O. container. The chemical plant was assembled and fully tested before being shipped in its fully assembled form on the top level of a container ship. It is thought that this approach to small scale chemical plant construction will be of immense benefit in cases where project schedules demand a minimisation of on-site installation problems.

#### Morris Ashby Ltd

Billiton (UK) Ltd has announced that its offers for the whole of the share capital of Morris Ashby Ltd became fully unconditional on 12 April. 100 per cent acceptances have been received in respect of both the Ordinary and Preference Shares. The Billington International Group of Companies, part of the Royal/Dutch Shell Group, are active in all phases of the non-ferrous metal industry. Morris Ashby is a private company and is a leading producer of zinc oxide and zinc dust. Morris Ashby's activities are complementary to Billiton's and its acquisition will enable Billiton to expand and strengthen its U.K. operations.

#### 1978 (6) INFORMATION RECEIVED

#### Butanediol plant of GHC taken on steam

GAF-Huls Chemie GmbH a joint venture of GAF Corp., U.S.A. and Chemische Werke Huls AG, has recently started production and delivery of 1,4-Butanediol which is used in the manufacture of special polyurethanes, rubbers, polyesters and plasticisers.

#### U.K. agents

Alfa Chemicals Ltd of Staines, Middlesex have announced that agreement has been reached with SCM Glidden for Alfa to act as sole U.K. and Irish distributors for the Silcron line fine particle silicas. Silcron products are used for matting agents for a a wide range of surface coatings, but particularly for industrial and wood finishes. Alfa Chemicals Ltd have also been appointed as sole U.K. and Irish distributors for a range of polyurethane resins produced by Iroquois Chemicals of West Germany. The resins are used in adhesives, textile coatings and plastic industries.

#### Pigment company expansion

Foscolor, the specialist pigment supply company, a member of the Foseco Minsep Group, well known for their pigment dispersions for printing ink manufacture, are planning a major expansion. A large extension to the existing building at the Foscolor, Bickershaw Lane factory at Wigan is to be commissioned.

#### New products

#### Lightweight mask

A lightweight air-fed mask suitable for refinishers using isocyanate cured topcoats has been introduced by Safety Products Ltd. Named the Clearways Air Fed Vizor, it offers a simple and effective answer to safety for personnel spraying two-pack gaints. The mask comprises a clear vizor mounted on an adjustable headband. Air is continuously admitted through a diffuser inside the base of the mask, escaping through the seals and the small gaps where the mask does not conform exactly to facial contours.



#### The Safety Products Ltd lightweight mask which satisfies Section 6 of the Health and Safety at Work Act

#### Ultra pure water

Stanmore Scientific Systems are now marketing the new Vaponics Type I System

for producing ultra pure water, at points of use in the laboratory, from already purified water supplied from a central deminaraliser or still. This compact unit brings the control and simple measurement of purity to each user of water from a central supply source. The system is capable of removing impurities which may be introduced in feed pipes or normal water storage tanks.

#### Line marking by airless spray

A line marking unit designed for use with any Wagner Airless paint sprayers has been introduced in the U.K. by Gray-Campling Ltd. The Wagner Line marker facilitates the quick and easy spray painting of clearly defined and even lines for the marking of, for example, parking bays and safety zones. The unit may be detached from the sprayer enabling the sprayer to be used for other painting jobs.



#### The line marking unit designed for use with a Wagner airless paint sprayer

#### New methacrylate resins

Degussa has developed two methacrylate resins for the production of powder lacquers that are marketed under the provisional designations VP-LM 36/41 and VP-LM 35/11.

VP-LM 36/41 is a resin with epoxide groups which can be cross-linked with polycarbonic acid. The powder lacquers produced with this resin can be pigmented to deep shades even without the use of special additives and give improved weathering resistance.

VP-LM 35/11 is a resin containing carboxyl groups that can be cross-linked with solid epoxide resins of the bisphenol-A/ epichlorohydrin type.

#### Expanded dye range

Atlantic Chemical Corporation has expanded the Atlasol (solvent soluble dyes) line to include Atlasol spirit dyes, which have varied special uses for the colouring of wood, paper, leather, film, aluminium foil and varnishes. Improved lightfastness, more brilliance of shade and deeper shades which are not obtainable with regular oil soluble dyes are claimed.

#### In-line filter

A general purpose, in-line filter unit available in several different versions to meet the specialised filtration requirements of a wide range of liquids is available from British Filters Ltd. The FB.799 is designed for permanent mounting directly in the pipeline, and element changes can be carried out easily and quickly simply by removing the cover, without disturbing pipework connections.

#### **Refinishing range**

A wide range of colours are available in the new International Acrylic Base/Clear refinishing materials for motor vehicle repairs which has been launched by International Paint – Vehicle Finishes Division. Designed for the repair of vehicles originally finished in Base/Clear metallic, it is equally suitable for panel or spot repairs.

#### Filling machines

Morgan Fairest Ltd have available the Albro vacuum measure filling machines which range from simple manually operated units, to fully automatic, multi-head installations and which are designed to fill exact quantities of still, free flowing liquids into containers. This range of machinery is particularly appropriate when accurate filling is required to comply with Weights and Measures Legislation.



# The Albro vacuum measure filling machine from Morgan Fairest Ltd.

#### New flow sensor

A significant advance in liquid flow control is claimed for a simple and inexpensive flow sensor manufactured by Elmic Ltd. The device can be fitted at any point in a pipework system to detect loss of flow, to allow action to be taken to avoid consequential damage. It comprises a reed switch in a flexible rubber mounting with a contact base, extending from a moulded plastic housing. As soon as the flow rate drops below a given level the reed switch completes a circuit and sounds the alarm.

#### New pump

Charles S. Madan & Co. Ltd have introduced a new Double Differential Diaphragm pump to their range of air powered pumps which satisfied many application needs of a medium volume portable powered unit. Available in two forms, general duty and chemical, they are capable of deliveries in excess of 20 G.P.M., are light and easy to handle.

#### Airless spray unit

A new extra-high pressure, high volume airless spray unit which handles and atomises all currently available heavy-viscosity spray coating materials, is being marketed by Graco. Designated the 60:1 King, the new unit meets a demand from sections of the heavy coating industry for a spray system with the power to overcome losses due to high material viscosity, low air line pressure or use with extra-long lines.

#### New smoke detector

OCLI Europe have available a new silicon planar photodetector for smoke detection applications. Mounted on a TO-5 header with a clear epoxy dome lens it works on the principle of detecting scattered light from smoke particles.

#### New drum handling attachments

A range of mechanical drum handling attachments is now available through Hercules Hydraulic Ltd, who have been appointed sole U.K. distributors of Verto-matic drum handlers. Available for either fork or carriage mounting, the attachments are quickly fitted to any type of lift truck for handling 25 gallon and 45 gallon capacity drums.

#### Automatic colouring system

A new product recently launched by Foscolor Ltd means that small thermoplastic moulding and extrusion shops can now change from solid pigment to liquid colour-

#### Notes and News-

ing feed. Compact and durable, the new product is a complete system of automatic colouring at the machine and incorporates a unique colour injector used in conjunction with high quality liquid masterbatch.

#### Valve removal

BUL Systems have developed a version of their membrane valve range which overcomes the problems of cross contamination and clean-down in powder handling systems. The System allows removal of the valve by merely releasing a toggle catch and swinging aside a spring clip, the actuating cylinders remaining *in situ* and the connections are not disturbed. Where necessary, a stand-by valve can be slipped into place immediately.

#### New ketone solvent

Eastman has announced the commercially available methyl n-propyl ketone (MPK), a highly active medium evaporating straightchain ketone solvent. MPK is a solvent for many conventional film formers, including vinyl, nitrocellulose, cellulose acetate butyrate, acrylic, epoxy, urethane and polyester. Because it is a highly active solvent, MPK is particularly suited for use in high solids, low energy coating formulations to help lower curing temperatures or shorten baking cycles, and is exempt from the U.S. "Rule 66".

#### Airmix 804

Kremlin Spray Painting Equipment has introduced a new version of the unique Airmix system designated the Airmix 804 unit, to meet the requirements of the lower volume paint user. The new unit has an adjustable fan width, provides high quality finishes and is easy to maintain. It is easy to clean and colour changes are possible in a short time.

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#### INFORMATION RECEIVED JOCCA

#### Conferences, courses etc.

#### Coatings conference

Kent State University, Ohio, is organising a conference to be held on 24-28 July entitled 'Coatings and polymer characterisation'.

#### Methylamines

ICI Ltd, Petrochemicals Division has published a booklet dealing with safety aspects of handling, storage and physical properties of their range of methylamines.

#### **Oilseeds conference**

The Oil Technologists' Association of India is holding a conference on Oilseeds and Oils to be held in New Delhi from 9–13 February 1979.

#### **Electrophoretic painting**

R. H. Chandler Ltd has published a book entitled 'Advances in electrophoretic painting 1975-77'. The book reviews the changes that have taken place in this rapidly expanding field and contains author and patent indexes.

#### **Industrial floors**

Schering Chemicals Ltd has issued a booklet which is complementary to a film they have produced describing the problems of flooring industrial areas, entitled 'It all depends on the hardener'. The booklet describes the use of new bonding agents and procedural techniques based on epoxy resin systems, which largely overcome many problems in this context.

#### Industrial finishing chart

Cray Valley Products Ltd has introduced a wall chart as a general guide to the selection of the most suitable resin for a given application according to the type of cure required.

### OCCA 60th Anniversary Commemorative Celebrations

The Association was founded on 16 May 1918 at a meeting of fourteen Founder Members, the last of whom, Mr H. R. Wood, died in July 1977 at the age of 90.

In 1968, the Association celebrated its Fiftieth (Golden Jubilee) Anniversary by two days of special functions and it was felt that the Diamond Jubilee should also be marked this year by two functions on successive days close to the foundation date.

Accordingly, on May 11 the Association held a well attended Commemorative Foundation Lecture, Reception and Dinner at the Painters' Hall, Little Trinity Lane, London EC4, to which were invited as guests of the Association all surviving Past Presidents and Past Honorary Officers of the Association.

At 6.30 p.m., the company, which had assembled in the Court Room, welcomed the President, Mr Angus McLean, accompanied by the Commemorative Foundation Lecturer, Sir John Methven, and the Master of the Worshipful Company of Painter Stainers, Sir Ralph Perring Bt. Introducing the lecturer, the President mentioned that he had been Deputy Chairman of ICI's Mond Division before becoming first Director-General of Fair Trading and then moving to his present position of Director-General of the Confederation of British Industry, where he had made a substantial contribution to the better understanding of the working of British industry. He then invited Sir John to deliver the Commenorative Foundation Lecture, the topic which he had chosen being "The place of business in our society". The lecture was extremely well presented and received, and Sir John has kindly agreed to allow its publication in a forthcoming issue of the *Journal*.



Shown in the Painted Chamber at the Painters Hall are (*left to right*) Sir John Methven (Director-General CBI, Commemorative Lecturer), Mr A. McLean (President), Sir Ralph Perring Bt. (Master, Worshipful Company of Painter Stainers) and Mr R. H. Hamblin (Director & Sceretary)

#### 1978 (6) NOTES AND NEWS

Following the Lecture, an inscribed silver medal was presented to Sir John Methven by the Master of the Worshipful Company of Painter Stainers, Sir Ralph Perring Bt., a former Lord Mayor of London. The



#### The inscribed silver medal which was presented to Sir John Methven on behalf of the Worshipful Company of Painter Stainers

medal was one of those specially commissioned by the Worshipful Company of Painter Stainers from a design by Jocelyn Burton to commemorate the Silver Jubilee of Her Majesty Queen Elizabeth II in 1977, and was inscribed for the presentation as follows: "Presented to Sir John Methven, Foundation Lecturer, Sixtieth Anniversary Celebrations, Oil and Colour Chemists' Association, May 1978".

Following the presentation, a reception was held in the Painted Chamber for the Past Presidents and in the Court Room for the remainder of the company, before dinner was served in the Dining Hall.

After Dinner the President proposed the Loyal Toast and in his Address of Welcome mentioned each of the Past Presidents and Past Honorary Officers present:

#### Presidents

- Dr H. W. Keenan (1944-47)
- Mr H. Gosling (1953-55)
- Mr N. A. Bennett (1957-59)
- Dr S. H. Bell OBE (1965-67)
- Mr F. Sowerbutts (1967-69)
- Mr A. S. Fraser (1969-71)
- Mr L. H. Silver (1973-75)
- Mr A. T. S. Rudram (1975-77)

#### Past Honorary Officers

- Mr M. H. M. Arnold (Hon. Editor 1955-58)
- Mr D. S. Newton (Hon. Editor 1962-65 and Hon. Secretary 1969-74)
- Mr F. Cooper (Hon. Treasurer 1970-76)

He paid tribute also to the work done on behalf of the Association over the period of sixty years both at Council and at Section level and to the spirit of comradeship which he had experienced at first hand during his visits to Sections during his first year of office. He extended a welcome to the other Association guests, Sir Ralph Perring Bt., Mr C. Sweett, a Past Master of the Worshipful Company, who was Chairman of its Education Committee, and the Clerk of the Company Mr H. M. Pearce. He felt that the Association would wish to record its thanks to its Director and Secretary, Robert Hamblin, and his staff on this occasion, both for the organisation of the Celebrations and for all their work on behalf of the Association. Finally, he asked the members of the Association to join him in a toast to their guests.

In reply, Sir Ralph Perring made reference to the work of the Painter Stainers in the field of education and the way in which the Association, as present day technologists, could be regarded as fulfilling many of the same functions as the old craft guilds in educational matters. The Company nowadays made annual presentations of prizes to encourage achievement in the fields of painting and decorating and he looked forward to even closer liaison with the Association in this aspect of their activities. Sir Ralph's speech was of great interest to the audience and, in concluding the proceedings, the President thanked him on behalf of the Association.

The formal proceedings being completed, many of those present took the opportunity of renewing old acquaintanceships and the function terminated at 10.15 p.m.

On the following evening the Commemorative Dinner and Dance attended by 300 members and guests was held at the Savoy Hotel, London W.C.2. The following principal officers of other organisations were present as guests of the Association:

- Mr A. J. Hughes (President, Paintmakers Association of Great Britain Limited) and Mrs Hughes.
- Sir Ralph Perring Bt. (Master, The Worshipful Company of Painter-Stainers) and Lady Perring.
- Mr J. H. Leach (President, The Society of Dyers & Colourists, which Society had in April conferred the rare distinction of admitting the holder of the office of President of the Association to Honorary Membership) and Mrs Leach.
- Professor R. O. C. Norman (President, Royal Institute of Chemistry).
- Mr H. Slack (Chairman, The British Colour Makers' Association) and Mrs Slack.
- Mr B. Boocock (Chairman, The British Resin Manufacturers' Association) and Mrs Boocock.
- Mr R. H. Pinner (President, Institute of Metal Finishing) and Mrs Pinner.
  - Mr M. J. Levete (Director, Paintmakers Association of Great Britain Ltd) and Mrs Levete.
  - Mr N. K. Bridge (Director, PIRA) and Mrs Bridge.
  - Mr E. Sangster (Director, National Federation of Painting & Decorating Contractors) and Mrs Sangster.

At 7 p.m. a Reception was held in the River Room and dinner was taken in the Lancaster Room at 7.30 p.m. Many favourable comments were heard on the setting and atmosphere provided by the Lancaster Room and by the trio who played throughout the dinner.

After dinner, the President proposed the Loyal Toast and an Address of Welcome in which he included not only principal officers of other societies who had honoured the Association by their presence, but also those from overseas, particularly the Chairman of the Ontario Section, Mr Walter Fibiger and Mrs Fibiger, Mr and Mrs L. Larson and Mr and Mrs J. Rooney (both of the FSCT who had also attended the 50th Anniversary Celebrations). The President had a special welcome for the ladies and he concluded by asking all members of the Association to join him in a toast to the guests, coupled with the name of the Guest of Honour, Mr A. J. Hughes.



Shown at the Commemorative Dinner Dance are (left to right) Mr A. McLean (President), Mrs McLean, Mrs Hughes, Mr A. J. Hughes (President, Paintmakers Association of Great Britain Ltd)

In response to the toast, Mr Hughes paid tribute to the work and achievement of the Association over the sixty years and offered the best wishes of all the guests for its continued well being. He drew attention to the many areas of common interest between the Paintmakers Association and OCCA and welcomed a closer liaison and cooperation between the bodies in the future.

Dancing to the Jay Langham Orchestra followed the speeches, and since this was a special occasion, the evening was divided into sections by two cabaret acts: the "Voyagers" who first entertained the company with singing and, after an interval during which tea and cakes were served, Alan Shaxon and Anne mystified everyone with some ingenious and imaginative illusions. The party continued until 1 a.m., when the President concluded the 60th Anniversary Celebrations by wishing everyone a safe journey to their homes.

The two days of Celebrations had been well attended and, judging by the comments of the participants, most enjoyable and successful.

A special eight page commemorative booklet designed by the Director & Secretary was given to each guest attending either of the functions. It includes a full colour reproduction of the Presidential Insignia, details of the two functions, photographs etc. There are a few spare copies and any member wishing to receive one should write to the Director & Secretary as quickly as possible; dispatch will be made strictly on application while stock lasts.

# **The Perkin Connection**

Members of the Association will have noted with pleasure in the Report of the Council Meeting in the April issue of the Journal (page 135) that, in recognition of the valu-able contributions made by the Association to the development of colour application technology and the close liaison which has existed between the Association and the Society of Dryers & Colourists, the Society had decided to mark the Sixtieth Anniver-sary of the foundation of the Association by conferring Honorary Membership upon the office of President of our Association. The Society was founded in 1884 and this will be only the third time that Honorary Membership of the Society has been so conferred, the the first occasion being to the Prime Warden of the Dyers Company (1886) and the sec-ond to the President of AATCC (1971). The ceremony of presentation of the Illuminated Address by Mr J. Atkinson (President of the Society) to the President of the Association (Mr A. McLean) took place at the Annual Dinner of the Society at Manchester on 28 April, and, in his reply of thanks, Mr McLean touched upon the very close ties between the two organisations. It is felt that Members of the Association will be interested in what might be termed the "Perkin *Connection*". The Society's headquarters in Bradford are known as Perkin House and the Society bestows an award (the Perkin Medal), both of them in recognition of the work of Sir William Perkin, who, at the age of 18 in 1856, discovered the first synthetic dye. Priory House, the Association's own offices, stand a quarter of a mile from the Chestnuts where Sir William Perkin lived for 50 years and where today there is a residential area known as Perkin Close, a brick obelisk in the churchyard at the entrance to the Chestnuts, which was placed there by the



The President, Mr A. McLean receiving the illuminated address from Mr J. Atkinson, President of the Society of Dyers and Colourists in April, bestowing Honorary membership of that Society upon the holder of the office of President of the Association

Wembley History Society, not only to commemorate Sir William's scientific achievements, but also the fact that he was instrumental in founding the first church built on the site and – for those who might find the walk from the station to the Association's offices a little long, there is a park bench on the Green opposite, with a plaque which also commemorates Sir William Perkin. If one leaves the Association's offices and proceeds in the other direction, one is once more reminded of Perkin, since, just over half a mile away is Greenford Green, where Perkin, together with his father and brother Tom, both from the building trade, set up the first synthetic dye factory beside the Greenford Canal in 1857. The little factory was ill-equipped and, since all three were unskilled in this type of work, the local inhabitants of the village are reported as having been concerned, if not terrified, by minor explosions. Within a few years. Sir William handed over the management of the factory to his father and brother and set up a research laboratory in a house opposite the factory which still stands in Oldfield Lane. The old bell, which tolled the workers to the factory, could still be seen until recently from the Lane. The factory was sold in 1873 and it is believed that the sale took place in the house of Sir William's nephew, which is now known as the Cottage, and is owned today by Glaxo Holdings Ltd. Sir William Perkin was well known as a local benefactor and, as well as the church on the Harrow Road, he helped to found a cottage hospital and organised various social clubs. He was President of the Society of Dyers & Colourists when he died in 1907, and was buried in Roxeth Churchvard just over the Harrow Hill beside the Association's offices.

Sir William had three sons – the eldest was a Professor, first at Manchester and then at Oxford, and the second, Dr F, M. Perkin was Head of Chemistry at the Borough Polytechnic and the first President of the Oil & Colour Chemist' Association, whilst the youngest son was Professor of Colour Chemistry at Leeds University, where one of his students was George Campbell, well known to many members of the Society, who is the senior surviving Past President of the Association. R H H.

# **Scottish Section**

#### Eastern Branch

#### **Burns Supper**

A Burns Supper was held on 10 February in the Commodore Hotel, Edinburgh, which was well attended by members and friends. After a Scottish style dinner the guests were entertained in the traditional manner, the highlight of the evening being the immortal memory of Robert Burns.

It was obvious at the end of the evening that a good time was had by all. Thanks are due to the many sponsors for their donations of raffle prizes.

#### Ladies' Evening

After the A.G.M. on 22 March, the members joined their wives to enjoy a talk given by John Haig & Sons, Whisky Distillers, about the manufacture and marketing of Scotch Whisky.

The talk was illustrated with a film and various blends of whisky were available to be sampled.

The thoroughly enjoyable talk was followed by a "spirited" question period. A vote of thanks was proposed by Mr I.

McCallum to which the company showed their appreciation in the usual manner.

ΤM

# Manchester Section

#### Informal Buffet Dance

The Royal Air Force Association Club, Sale, Cheshire was the venue, for the third successive year, of this event held on Friday 10 March 1978.

Dancing commenced at 8.00p.m. to the musical strains of the Edelburgers and the 188 members and guests gyrated and occasionally danced until the excellent buffet provided a gastronomic break. The sex discrimination act was involved later in the evening when 37 prizes were awarded, in true Bingoistic Jargon, to female recipients only. Before the dancing finished at 12.30 a.m. the R.A.F. Wings Appeal was supported to the sum of £21.55 by the bizarre method of coin rolling to centrally located target prize of liquor that was to be won 'afore we went'.

Absence of R.A.F. uniforms was the only real degree of informality observed at this excellent function once again organised by Mr A. E. Thornhill, to whom we are all again grateful.

#### **Annual General Meeting**

The 54th A.G.M. of the Section was held at the Lancashire County Cricket Club, Old Trafford, on Friday 14 April 1978 at 6.30 p.m.

Approximately 80 members and 2 visitors attended the meeting which initially dealt with Reports, Election of Officers and a ballot to decide the two successful candidates of three who were standing for the two vacancies on Committee. This meeting marked the retirement, after two years' service, of a very popular Chairman, Jack Mitchell; he is succeeded by Tony McWilliam, a former Publications Officer of the Section.

After an excellent meal, those members who were not playing Liar Dice were privileged to witness Phase III of the infamous "Jack Mitchell Quiz". The Quizmaster this year was Bill McDonnell, who claimed to hail from Knotty Ash and the two teams could be identified only as those who supported facial fungus versus the daily shavers. Once again the latter team won, though the ambidexterity of the Quizmaster must remain in doubt for many months.











































# EXHIBITION REVIEW

INTERNATIONAL INVOLVEMENT ENORMOUS AT OCCA-30

Exhibitors from 16 countries and visitors from more than 50

OCCA-30, the Thirtieth Annual Technical Exhibition of the Oil and Colour Chemists' Association was held at Alexandra Palace, London N22, from 18 – 21 April 1978, and was without doubt an inspiration to the troubled chemical industry, bringing together technical personnel of the supplying and consuming industries from 53 countries.



One of the corridors at the Exhibition, crowded with throngs of visitors

Amongst the 147 organisations represented at this unique international forum for the surface coatings industries, were direct exhibitors from the following 16 countries:

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

A healthy feature of the Exhibition this year was the high proportion of new companies represented for the first time, as well as many exhibitors from previous years, which meant that the number of stands at the exhibition was considerably higher than it had been for several years.

#### The international focal point for the surface coatings industries

The motif for the Exhibition, designed by Robert Hamblin, used the symbol of a moving indicator on a calendar to emphasise the importance of the continuing dialogue, year by year, between suppliers and manufacturers, and the inward pointing letters demonstrated the many points from which visitors converge to this annual international forum. The exhibition is unique in its endeavour to bring together the technical personnel from all aspects of the industries to meet in an informal atmosphere, allowing the free interchange of ideas and the rapid dissemination of knowledge of new products and new developments on existing products.

#### The crowd puller!

Many exhibitors have expressed themselves as amply rewarded for their participation at OCCA-30, and indeed, some stated that if they had not been present on the last three days, the contacts that they made on the first day alone would have more than justified their presence! Admissions to the Exhibition were by season ticket, and the numbers recorded at the turnstiles at the entrance were in excess of 10,000. The visitors are known to have come from at least 53 countries, from all over Europe, the Americas, the Middle East, the Far East, Africa and Australasia.

The Exhibition was open for four days, and the advantages of the venue at Alexandra Palace are well known: the ample free car parking space, restaurant facilities, a cafeteria, licensed bars and the pleasant surroundings – all at the sole disposal of the Association. Since the Exhibition last year, travel facilities had been greatly enhanced for overseas visitors by the opening



Visitors to the Exhibition this year came from fifty-three overseas countries

of the Heathrow Central Terminal of the Piccadilly Underground line at the airport giving a direct link to Turnpike Lane Station, from which point the Association once again ran a free bus shuttle service to and from the Exhibition.

This year, following the experiment at OCCA-29, exhibitors were once again allowed to serve alcoholic refreshments to their visitors on the stands. This is an aspect which is appreciated especially by the smaller exhibitors, since it allows their staff to remain on the stands throughout the period of the Exhibition.



A view of the Association's Information Centre at the Exhibition where staff and interpreters were available to assist visitors, with one of the seating areas situated in the foregound

#### **Exhibition luncheon**

Following the successful innovation at OCCA-29 last year, an Exhibition luncheon was again organised for the opening day. Invitations were extended to principal officers of other societies, Members of Parliament, and other special guests, and in the afternoon the party was conducted around the Exhibition by the Committee.

#### Information Centre

Enormous interest was aroused at the Association's Information Centre where OCCA publications, badges, etc. could be purchased and where information was available on the optional Professional Grade, membership of the Association, subscription to the *Journal* and advance details of the Association's next Conference to be held in Stratford, 20-23 June 1979. As usual, interpreters were available at the Centre for the many overseas exhibitors and visitors.

#### **Technician Education Council**

The Paintmakers Association of Great Britain Ltd organised a display on behalf of the Technician Education Council at the Information Centre, as a successor to the Association Technical Education displays, and information on the courses available in the UK under the scheme was available. Courses of study at many Technical Colleges directly relevant to the surface coatings industry are now available.

As a result of participation in this year's Exhibition, some exhibitors have already asked for larger sites to be reserved for them at the OCCA-31 Exhibition next year, and a number of companies which were not able to be present this year have also made requests to be sent full information as soon as it becomes available.

#### The cost effective Exhibition

Arrangements have been made for OCCA-31, which will be held at Alexandra Palace from 3-6 April 1979. Invitations to Exhibit will be despatched at the end of June and the allocation of space will take place in October 1978. Any company wishing to receive details of OCCA-31 should write to the Director & Secretary of the Association, Mr R. H. Hamblin, Priory House, 967 Harrow Road, Wembley, Middlesex HA0 2SF, England.



The motif for OCCA-31, designed by Robert Hamblin, emphasises the unique aspect of the OCCA Exhibitions, which annually provide an international focal point for the surface coatings industries. In 1978, exhibitors were drawn from sixteen countries and visitors from over fifty countries.



The Official Guide to the Exhibition is prepared and dispatched several weeks in advance of the dates of the exhibition in order to allow visitors the maximum opportunity of planning their visits to the stands. Information concerning late applications for stand space is published in the *Journal* in the issues preceding the Exhibition together with information concerning products to be shown, which was not available at the time of the printing of the Official Guide.

This year, for the first time, the Official Guide contained a chart classifying the exhibits into product types which was a great benefit to visitors at the Exhibition. Accordingly, in the same fashion, the Technical Report of the Exhibition on the following pages is also classified into product types and contains much information which was not available before the Exhibition. Anyone having difficulties in contacting the firms who were present at the Exhibition should write to the Association's offices, and their enquiries will be forwarded to the relevant company.

The exhibits are classified into the following categories:

Additives, driers, surfactants and hardening agents

#### Chemical intermediates

Extenders, fillers and matting agents

Laboratory apparatus and testing equipment—optical equipment, viscometers, dispersing equipment, testing equipment and general equipment

Manufacturing equipment and drums etc---dispersing equipment, pumps and valves, screens and filters, flowmeters, filling equipment, other equipment

Oils and fatty acids

Pigments-inorganic, metallic and pearl, dispersions and pastes, organic, fluorescent

Resins—general, acrylic, alkyd, amino, emulsion and water based, epoxy, polyester, polyurethane

Solvents and plasticisers

Technical journals and services.

#### The aim of the Exhibition

The aim of the Exhibition is the presentation of technical advances in those industries supplying the paint, varnish, printing ink, colour, linoleum, and other allied industries. The technical advances may relate to: (i) new products, (ii) new knowledge relating to existing products and their uses, or, (iii) in suitable cases, existing knowledge which is not generally available in the consuming industries.

Whilst the Exhibition Committee naturally encourages the showing of new products, it does not stipulate that a new product has to be shown each year, since it fully appreciates that there are occasions when this is not possible. Accordingly, the Committee draws attention to the fact that new technical data on existing products are regarded as acceptable subject matter.

# **Exhibition Report 1978**

# Additives, driers, surfactants and hardening agents

#### ALLIED COLLOIDS LTD

Additives for emulsion paints, particularly the glossy types: *Dispex* dispersing agents, *Viscalex* "V" acrylic thickening agents, *Glascol* "G" acrylic solution polymers for improvement of gloss and flow. *Surcol* 1460—see under *Resins acrylic*.

#### CAPRICORN CHEMICALS

UK agents for the G. M. Langer range of additives. Lanco waxes, micronised polyethylene, polypropylene and amide waxes to impart scuff and slip resistance. The Lanco Glidd wax dispersions of low molecular weight polyethylene to give slip and provide mar, block and water resistance.

Antibubble compounds, silicon-free additives for solvent based systems. Anti-float compounds, various powdered and liquid compounds. Aqueous thickeners/suspending. agents. *Hectorite* and *Macaloid* montmorillonite clays.

#### CHEMOLIMPEX

Driers, single and mixed versatates. Organic peroxides of methyl ethyl ketone, cyclohexane and benzoyl and mixed peroxides.

#### COLE CHEMICALS LTD

Biocides. A range of preservatives, including non-mercurial fungicides.

#### CORDOVA CHEMICAL CO.

Polyfunctional aziridines as cross linking additives, modifiers or reactive intermediates, which are highly reactive with materials containing active hydrogen atoms, particularly with carboxylated polymers to give rapid low temperature curing. The resulting coatings have improved solvent resistance, reduced water sensitivity, excellent adhesion, hardness and abrasion resistance. The products are: Xama 2, Xama 7, N-hydroxy ethyl ethyleneimine, and iminol methylacrylate.

#### CORNELIUS GROUP

The Daniel range of speciality products.

#### **CROXTON & GARRY LTD**

Colorol range of Lucas Meyer's additives for pigment wetting.

Forbest additives for printing inks and paints to prevent blocking and to give abrasion resistance.

Shiraishi Homosol range of calcium carbonate thioxotropic additives.

*Elotex WS 45* to provide a water barrier effect in cementitious products.

Tektamer 38 a new Chemviron environmentally acceptable in-can preservative.

#### DOW CHEMICAL COMPANY

Dowicil 75 a very active, low toxicity, biodegradable, water

soluble preservative for emulsion paints, adhesives latices and metal working fluids.

Methocel cellulose ethers for thickening water based paints which also impart improved flexibility and storage stability.

*XD 8062.00*, a new generation hardener for applying epoxyphenolic chemistry to powder coatings, especially developed for the sole purpose of crosslinking epoxy powder coatings.

XD 7080, a curing agent combining acrylic and epoxy technology in a water emulsifiable coating system. It gives air-drying coatings with improved weathering and the cure properties of solvent based epoxies.

*XD 556-01*, a low viscosity epoxidised bisphenol F containing liquid epoxy system, compatible with common epoxies, which can be blended to give specific properties.

#### DURHAM RAW MATERIALS LTD

Nuodex, Nuosyn, Curgon and Celerate ranges of driers, which have been quality standards for many years, and the new Celerate Calcium 10%.

Information on *Nuodex* biocides, *Nuosept 95*, *Fungitrol 11*, and *Nuodex 87* for use as in-can and film preservatives for water and solvent based systems.

Nuosperse 657, for use in the dispersion of pigments in organic media.

#### EMSER WERKE AG

See Grilon (U.K.) Ltd.

#### S. A. FLORIDIENNE NV

See Micro Products Ltd.

#### GRILON (U.K.) LTD

Grilonit curing agents for water dispersible and solvent free epoxy coatings.

#### HERCULES POWDER CO. LTD

Water soluble polymers of the cellulosic type and also *Xanthan* gum as thickening agents. See also under *Resins*, *general*.

#### K & K GREEFF INDUSTRIAL CHEMICALS LTD

A range of *Veba Chemie AG* polyethylene waxes for use as matting agents and lubricants in plastics, inks, paints and polishes.

#### KWR CHEMICALS LTD

The CdF Chemie range of curing agents.

#### LAPORTE INDUSTRIES LTD

The range of *Laponite* inorganic thickening agents, which can be used in emulsion paints.

#### MICRO PRODUCTS CO.

The *Micro Powders Inc.* range of synthetic and polyethylene waxes for improving rub and scratch resistance, including the *Polyfluos* range of high slip additives for use in can and coil coating, metal decoration and UV and IR cured inks.

#### WARD BLENKINSOP & CO. LTD

The Quantacure range of UV initiators covering a wide

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range of products.

Aduvex ultraviolet light absorbers.

Additives, catalysts, fungicides, antioxidants sunscreen agents and cationic reagents.

The *Rondec* screen printing machine equipped with a UV curing system, using WB photoinitiators, was demonstrated showing a three dimensional object being printed, cured and packed in a few seconds.

Exhibits shown included: UV resin coated wall panels from *Syma Ltd* using resins supplied by *Wellstor*; UV coated record sleeves from *Robor Ltd* using inks supplied by *Ault* & *Wiborg Ltd*; a unique application by *Pilkington PE Ltd* to UV coated optic fibres.

#### WORSDALL CHEMICAL CO. LTD

*Estoflow*, a new acrylic based flow improver for paints, coil coatings, inks and powder coatings.

#### Extenders, fillers and matting agents

#### CAPRICORN CHEMICALS

Hectorite and Macaloid montmorillonite clays.

#### COLE CHEMICALS LTD

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

#### CROXTON & CARRY LTD

*Q-Cel* a light weight spherical filler from *PQ International* for automotive polyester filling compounds.

The Syloid range of amorphous silica flatting agents.

*Hakuenka* ultra fine calcium carbonate for use in high gloss offset inks.

Shiraishi Homocol calcium carbonate additives.

The Burgess range of thermo-optic white calcined clays.

The *Elotex* range of redispersible homopolymer and copolymer powders for crack fillers, groutings, tile adhesives, wall paper adhesives and powder paints.

#### S. V. FLORIDIENNE NV

Special grades of waxes to produce matting with most coatings.

#### H. HAEFFNER & CO. LTD

A new type of fibre from *Rockwool AB*, Sweden, to replace asbestos fibre, available in fibres to suit various processes and purposes.

#### MELBOURN CHEMICALS LTD

The *Omya* and *Britomya* ranges of whiting and calcite extenders and *Granicalcium* white calcite chips for textured wall finishes.

Albarex secondary pigment for anticorrosion paints which is highly cost effective.

 $Hydrocarb \ L$  to replace blanc fixe, giving economic advantages in printing inks, alkyd paints, stoving enamels and powder coatings.

# NEVILLE CINDU CHEMIE B.V. Chemical intermediates

#### AMOCO CHEMICALS (U.K.) LTD Further information on the use of isophthalic acid and

trimellitic anhydride, as well as Resin 18 (polymethyl styrene) in alkyd resins.

TMA 123 describes low cost solvent borne industrial and can coatings

IP 61 Medium oil length alkyds for high durability road markings.

TMA 124 Water soluble, air drying vinyl toluene/methacrylate alkyds giving improved gloss and hardness.

Amoco-Fina Indopol polybutenes for roofing compounds, pressure sensitive adhesives and exterior textured coatings.

#### COLE CHEMICALS LTD

Vinyl acetate monomer. Nonyl and dinonyl phenol.

#### CORDOVA CHEMICAL CO.

Polyfunctional aziridines for use as reactive diluents. See under additives

#### EMSER WERKE AG

See Grilon (U.K.) Ltd.

#### GRILON (U.K.) LTD

A range of glycidyl ether reactive diluents, including Grilonit RV 18004 which has exceptionally low vapour pressure and toxicity.

#### LAPORTE INDUSTRIES LTD

Caprolactone, an internal ester of  $\varepsilon$  hydroxy caproic acid is an excellent reactive solvent for introducing flexibility into synthetic resins, such as thermosetting acrylics, polyesters, epoxies and PVCl combinations.

#### MONTEDISON GROUP

Urea, trimethylol propane, pentaerythritol, Formurea 80 and maleic anhydride.

#### WARD BLENKINSOP & CO. LTD

A substantial range of pharmaceutical chemicals and intermediates is available, based on the extensive range of organic routes available.

#### Laboratory apparatus and testing equipment

#### Optical instruments and associated equipment

#### DIFFUSION SYSTEMS LTD

A selection of optical test equipment for the paper, ink, paint and plastics industries, including instruments previously made by EEL, which are now available with digital read-out.

#### INDUSTRIAL COLOUR SYSTEMS LTD

Industrial Colour systems for the determination of the







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#### MICROFINE MINERALS AND CHEMICALS LTD

Mineral fillers and extenders including mica of all type, Suzorite whiting, talc, barytes, dolomite and china clay.

optimum cost combination of pigments to produce any required shade and also for initial formulation and batch to batch control. Numerical tolerance values obtained by a colour difference meter allow shade continuity to be ensured and at the same time allow increased production.

#### MACBETH COLOR & PHOTOMETRY DIVISION OF KOLLMORGEN (U.K.) LTD

Munsell Colour Order System for visual evaluation and the Macbeth Daylighting Cabinet. The Macbeth MS2000 abridged spectrophotometer has a pulsed xenon light source, laser tuned optical selector and microprocessor, giving a choice of four illuminants and three colour difference programmes. The unit has a cathode ray viewing screen and data entry keyboard. It will detect metamerism and measure fluorescence, compute colour differences etc., without an external computer.

The *Macbeth MC 1010* colorimeter is based on a similar technology and offers high speed computation from a microprocessor. There are no moving parts in the unit which has excellent correlation with the spectrophotometers.

#### PYE UNICAM LTD

A versatile colour measuring system based on the SP8-100 UV/visible spectrophotometer, giving accurate and reprodicible results without incurring the high cost of a fully computer controlled instrument. Several systems are available with different data handling facilities, including an on-line programmable calculator version. The system enables materials to be studied in transmission, under diffuse or total reflection conditions, with a choice of standard illuminants and observation angles.

The instrument has a large integrating spheroid with a cell complying with CIE requirements, which is unique in having no direct path for specularly reflected light to the photomultiplier. The software enables laboratory values to be presented in the Anlab (40) or the CIE (1976) conventions.

#### Viscometers

#### BAIRD & TATLOCK (LONDON) LTD

The Brookfield range of multispeed rotational viscometers. The Brookfield Rheolog Recording Viscometer gives a continuous record of viscosity with time which can be fed into a variety of recording equipment. The Helipath Stand Accessory enables the measurement of non-flowing materials previously considered unsuitable for non-flowing measurement. The Viscocel Model VTA 120 has been designed for the automatic and continuous recording of the viscosity of inks whose viscosity has to be maintained at a constant value by controlled addition of solvents to compensate for evaporation and other losses.

#### CONTRAVES INDUSTRIAL PRODUCTS LTD

A complete range of rotational viscometers and rheometers including multispeed laboratory rheometers for research and development and single speed instruments for quality control.

*Rheomat 30* is a 30 speed instrument embracing a very complete range of shear rates of interchangeable measuring systems, including cone and plate, cylindrical, very high shear, high pressure and high temperature types. It can be







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used with the Rheoscan 20 or 30 programmers and an automatic X-Y recorder.

Rheomat 15T is a semi-automatic viscometer for general purpose laboratory and development work. TV and STV are single and three speed instruments for relative and absolute measurements. HV6 is a high pressure capillary viscometer for measuring the flow of non-Newtonian materials at very high shear rates.

Covistat and DC series see under Manufacturing equipment.

#### FERRANTI LTD

Ferranti Portable Viscometer, a coaxial cylinder viscometer in which the outer cylinder is motor driven and the inner cylinder is free to rotate against a calibrated scale. The cylinders are immersed in the liquid to be measured and the viscosity is given by the scale reading. Sets of cylinders are available to cover the ranges 0-0.168/0-3.2 in 15 ranges, 0-0.9/0-40 in 9 ranges, 0-33/0-1460 with additional cylinders and 0-297/0-19684 poises. The scales are calibrated in 0-100 and converted to poise by constants provided.

Ferranti Shirley Viscometer. A cone and plate instrument, in which the dynamic flow characteristics are automatically plotted as a graph. Behaviour data may be plotted as shear rate vs shear stress and time vs shear stress. Temperature, cycle time and shear rates are monitored under strict control.

#### MSE SCIENTIFIC INSTRUMENTS

A complete range of Haake Rotational Viscometers. The Rotovisco systems are of the highest quality and cover extreme ranges of viscosity, shear rate and shear stress. A system may be developed on a building brick principle to suit the requirement of users, by selection of sensor systems; fixed, variable or programmable speed ranges; torque measuring head and data ouput. The key factors of the new model RV 12 include digital torque indication and small cycle torque measurements. The new Haake Rotovisco PG 142 programmer may be used with all Rotovisco systems and enables curves to be plotted by an X-Y recorder covering a wide range of programmes. The VT181/4 Viscotesters are two speed rotational viscometers for measurement under closely defined conditions or rapid routine tests and can be adapted to an optional recorded output. VT01/02 are battery operated, hand held, single speed viscometers for Newtonian liquids. A new Haake cone and plate sensor system was displayed.

#### RESEARCH EQUIPMENT LTD

The new ICI Low Shear Rate Viscometer designed to measure the flow properties of paint films at low shear rates, equivalent to those encountered in flow-out of brush marks, sags or spray droplets. The shear decreases during the test period (30 seconds) and the viscosity is measured during the initial-final parts of the test period. The plate deflection may also be measured. Viscosity and deflection data may be stored and assessed, and a period of conditioning (1, 4 or 16 secs) may be introduced before the viscosity measurement is made. The ICI Plate and Cone Viscometers were also shown

#### **Dispersing equipment**

#### D. H. INDUSTRIES LTD

The Supermill is available in a 1.5 litre size and the Sussmeyer Sand Mill also in laboratory size. See under Manufacturing equipment.



FERRANTI LIU



#### DRAISEWERKE GmbH

The PM 250 TEX roll mill is available in 1.3 litre size. See under Manufacturing equipment.

#### EIGER ENGINEERING LTD

The *Motormill* is produced in a 1 litre size. See under *Manufacturing equipment*.

#### G. J. ERLICH LTD

Laboratory sizes of the *Molteni* Planetary mixers and the *TM Series* high speed dispersers can be obtained. See under *Manufacturing equipment*.

#### GLEN CRESTON LTD

Two versions of the laboratory *Dyno Mill* of 0.6 and 1.4 litre chamber capacity. The 1.4 1 model is suitable for pilot scale and small batches. See under *Manufacturing equipment*.

#### JOHN GODRICH

A new *Rotostat Type 05* mixer for processing high viscosity fluids and having no suspended basket assembly was demonstrated on the laboratory scale.

#### MARCHANT BROTHERS LTD

The latest version *TRA* 4 of the  $3'' \times 6''$  laboratory triple roll mill was shown. See under *Manufacturing equipment*.

#### MASTERMIX ENGINEERING CO. LTD

A new 1 litre *Mastermill* horizontal bead mill. See under *Manufacturing equipment*.

#### MILLROOM ACCESSORIES & CHEMICALS LTD

A laboratory model of the *Rotamix*, which is a down scaled version of the production model and the *Whirl*. See under *Manufacturing equipment*.

#### PILAMEC LTD

A Megapot vibratory ball mill intended for continuous or batch laboratory work. See under Manufacturing equipment.

#### TORRENCE & SONS LTD

Laboratory cavitation dispersers, triple roll mills, the *IS* Attritor and the 3.5" Micro-flow mill.

#### Testing and special equipment, supplies

#### BAIRD & TATLOCK (LONDON) LTD

Hopkins & Williams laboratory chemicals in the AnalaR, UltraR and G.P.R. ranges.

#### DUPONT (U.K.) LTD

High performance liquid chromatographs (HPLC) and thermal analysis systems were shown. The 830 high temperature steric exclusion (HTSEC) system enables rapid measurement of the molecular distribution of polymers and this is related to the melt, flow and mechanical properties of polymers. The columns can be used with either polar or non-polar solvents. The latest infrared detector was shown. Both HPLC and HTSEC can be used in conjunction with the 850 system microprocessor.

The 990 thermal analysis system is of modular design with

a control/temperature programmer/recorder, to which can be connected apparatus for differential scanning calorimetry, thermogravimetric analysis, thermochemical analysis, measuring dimensional changes, viscosity measurements in the temperature range  $-160^{\circ}$ C to  $1200^{\circ}$ C and and dynamic mechanical analysis for measuring modulus damping behaviour. The latest addition to this system is the *R* 90 microprocessor based controller.

#### ELCOMETER INSTRUMENTS LTD

Physical testing instruments, including the *Elcometer* range for measurement of surface profile and quality, coating integrity, adhesion and thickness. Testing equipment in the *Gardner, Erichsen* and *Taber* ranges and *UPA* provide specialised forms of thickness measurement, including Beta back scatter techniques.

#### FISCHER INSTRUMENTATION (GB) LTD

*Permascope* range of coating/film thickness and porosity measuring instruments. *Permascope ES* for coatings on ferrous metals having greatly reduced variation due to substrate non-homogeneity and access to an uncoated surface for calibration is unnecessary.

ESD is a fully LED Digital instrument fitted with BCD output for automatic print-out.

The *Deltascope* is a compact, lower price instrument with a reading hold until another measurement is made. *Permascope EW8* is for use on non-ferrous materials and is insensitive to substrate conductivity thus reducing calibration procedures, using eddy current technology.

*Poroscopes* of various types to DIN and RAL specifications for the measurement of paint film porosity.

#### GALLENKAMP, A. & CO. LTD

*Mettler* electronic balances in a wide range of capacities, some models are fitted with BCD output to enable ancilliary equipment to be connected.

The Nelson Computer Services Checkweigh System was shown in which up to 3 Mettler balances can be connected. The system controls the weighing and records the actual weight taken, so that a daily stock record may be produced.

#### JOHN GODRICH

The Liebisch K 300  $SO_2$  cabinet and the Suntest Lightfastness Tester, the Credit Humidity Cabinet, the Liebisch Salt Spray cabinet and the Xenotest machines.

#### JOYCE-LOEBL LTD

Disc Centrifuge Micron Particle Size Analyser, which enables the determination of particle size/weight distribution in emulsions, pigment suspensions, inks, etc. to be determined. Application reports were available for many typical uses. The Luzex 450 Particle analyser is a television based instrument for rapid particle size analysis, which counts and sizes particles and measures the area occupied by particles. Results are obtained rapidly with good reproducibility.

#### MICROSCAL LTD

The flow microcalorimeter enables the measurement of heat effects when competitive reactions and displacements take place on the surfaces of powders and solids. This provides a better understanding of the reactions themselves





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and of the surfaces on which they occur. The Mark 2V vacuum model facilitates heat of immersion and degassing measurements, in addition to the preferential adsorption studies possible with the earlier Mark II model. The range of lightfastness testers has been extended. The Mark IV instrument provides a higher fading rate, whilst maintaining the correlation with daylight fading tests. The Mark III instrument uses UV sources and is intended for use with materials of very high lightfastness. Instruments for particle size analysis and the random sampling of powders remain available.

#### THE Q-PANEL COMPANY

The QCT Condensation testers which are more rapid than humidity tests and more representative than salt spray. Moisture in the form of rain or dew is simulated, the advantages being simpler control, reduced maintenance and lower cost. The QUV Accelerated Weathering Tester simulates rain and dew and has eight flourescent UV lampsto produce the effect of sunlight. Excellent correlation with outdoor performance has been reported. Q-Panels are standardised steel and aluminium panels for many forms of test and recording and are available in various types and sizes.

#### RESEARCH EQUIPMENT LTD

The new ICI Low Shear rate Viscometer is described under Viscometers<sup>2</sup>. Other equipment developed by ICI include the Cone and Plate viscometer, drying time apparatus, the pneumatic micro-indentation apparatus and the pressure weight per gallon cup. REL equipment includes power operated scratch test apparatus, salt spray cabinets, impact and abrasion testers, metering pumps and fineness of grind gauges. A four track applicator was shown.

#### R.K. PRINT-COAT INSTRUMENTS LTD

The motorised Mark III Printing Proofer was demonstrated printing by gravure and flexo processes and for the first time on rigid substrates, the instrument features a removable flexo-stereo roller, accurate over-printing in register and self sharpening doctor blades. The K Hot-melt Applicator is a motorised rotary unit with accurate temperature control which can apply hot melts at varying viscosities and layers on to materials used in the packaging field. K-Lox roller applies thin films on absorbent substrates —i.e. flexographic inks. K Control Coater with Vacuum Bed, an additional model of the K-Coater with a vacuum bed for applying films as little as 3–4 microns thick or very heavy coatings over 500 microns thick.

#### Manufacturing equipment, drums etc

#### **Dispersing equipment**

#### COLE CHEMICALS LTD

Steatite and Stemalox granules, balls and bricks for use as grinding media.

#### D. H. INDUSTRIES LTD

Sussmeyer/Oliver & Batlle. Dispermix high speed cavitation mixer/dissolver. Viscomill—a new horizontal closed continuous mill for normal and high viscosity paints and inks which will disperse materials as heavy as litho and web offset inks. Supermill with unique seal, screen, cooling and washing features with shell capacities from 5 to 200 litres. The Sussmeyer range of Sand Mills from laboratory to

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120 gals shell capacity. The *Centrimill* batch bead type mill with centrifugal discharge in 50, 100 and 250 litre capacities. From *Paul Vollrath*, twin and variable speed mixers with from 0.3 to 200 hp motors in electric and hydraulic construction, the range also includes the *Exentrik* and dual shaft dissolvers.

#### DRAISWERKE GmbH

Perl mills of various types including PM 250 TEX which is specially designed for pigment dispersion. This widely used machine is available in sizes from 1.3 to 1000 litres capacity and can be applied to horizontal or vertical operation. PM 25 STS triple cooled mill it is reliable, efficient, simple in design and easy to manipulate. The Drais Disperser with a S.T.A.R. head enables for the first time the manufacture of offset inks and paints to a fineness below 5 microns without the necessity for further milling. The Drais Planetary Mixer Type F 300 are the most efficient machines available on account of the new redesigned planetary gear system. It was shown in conjunction with the Schwerdtel Press, Type S2-M. The direct feed Perl mill for dispersing BASF granular pigment was illustrated. This is a process which is now being widely studied for eliminating dusting and general handling problems associated with powder pigments.

#### EIGER ENGINEERING LTD

Motormills which employ a new concept in agitated bead/ sand mills. The milling chamber is fitted directly on an extended motor shaft carrying several impellor blades. The mills can operate in horizontal or vertical positions, they are less costly than are conventional lay-outs, but incorporate all the features expected in a modern mill. Pressurised water/solvent washing facilities are provided and cooling is by a spiral, baffled water jacket. Sizes available are from 1 to 150 litres, they are virtually silent, vibration free and have low maintenance costs.

#### G. J. ERLICH LTD

Molteni Planetary Mixers from laboratory size to 300 litres with a variable mechanical drive and from 500 to 1000 litres capacity with a fully enclosed hydraulic circuit drive which gives a much wider range starting from 0 at full torque. Also from Molteni, High Speed Mixer-Dispersers of the TM series with rotating vessels of up to 1000 litres, the Butterfly Mixers with hydraulic drives and also dual shafts. Hydraulic Turbo-Mixers which operate through a floor and have capacities up to 3000 litres. Triple roll mills from laboratory size to the S.100, the Z.3H has an automatic hydraulic adjustment of the rollers. Microsphera is a grinder-disperser for pastes up to the highest viscosity.

#### GLEN CRESTON LTD

The Dynomills, the first horizontal bead mill designed specifically for milling and dispersing in every application and available in laboratory, pilot and full scale sizes. Emphasis was placed on safety including personnel, over pressure and over temperature. The 15 1 model was shown with a safety cut-out to prevent accidental starting without the cylinder in position. Examples of grinding media were shown including lead-free glass beads and zirconium oxide beads which have a useful life eight times that of glass.

#### JOHN GODRICH

Chemcol mixers MSO, MS1, and MS3 with MS2 and MS0 (air driven) shown in operation. These machines have no



bearing in the immersed portion, permitting easy cleaning and a full flow mixing head. Variations in the flow patterns can be obtained by drawing material from the top and bottom of the vessel or from the top or the bottom only. Several designs of stator are available and a variety of speeds can be chosen to provide a range of mixing capacities. The new *Chemcol-Mirap* range of mixers were shown which offer a greater versatility. New *Rotostat* machines were shown. *Type 05* is a laboratory model and the larger *Models* 10 and 30 are designed to process high viscosity fluids and have the advantage of a suspender basket assembly allowing better flow and easier cleaning.

#### H. HAEFFNER & CO. LTD

Information was available on the Westerlins Maskinfabrik range of machinery for the paint and ink industries, now represented in this country by H. Haeffner & Co. Ltd.

#### MAGMILL FRANCE SA

A multistage grinder based on the basic facts that as grinding proceeds the number of particles present increases and their size decreases and that after the first crushing stage the grinding media must be given more energy to provide sufficient shear to reduce the primary crystals in size. The machine has three working zones, each increasing in volume as the grinding proceeds and each charged with balls of increasing number and decreasing size. The blades are fitted to a single shaft and the zones are separated by screens to prevent the balls shifting from one zone to another.

#### MARCHANT BROTHERS LTD

The Nagema Production Triple roll mills up to  $16'' \times 40''$ and the new TRA4  $3'' \times 6''$  laboratory version which incorporates the best features of the earlier model and provides for the up-to-date needs of the industry. The Netzsch Bead Agitator Mills provide high production rates in both horizontal and vertical types using the 'Molinex' and 'John' systems for grinding. Finished high viscosity paste inks and concentrates eliminate the need for chip use.

#### MASTERMIX ENGINEERING CO. LTD

The new Mark 5 Underdriven Mastermill P.M.D. is available in sizes from 1350 to 6750 litres (working capacity). The slow speed drive under the vessel to a completely new type of agitator not only increases the mixing/dispersing efficiency, but also gives improved head room for loading facilities. The new Autoheight device has been developed for automatically raising or lowering the high speed shaft. The height of the high speed blade is continuously monitored and adjusted to obtain optimum performance during the loading and dispersing cycles. New refinements to the Mastermill range include a pressure limiting switch with an adjustable pre-set to stop the machine should undue pressure arise in the mixing chamber. The H.V.S. range of High Speed, High Torque and Twin Shaft mixers remain available.

#### MILLROOM ACCESSORIES & CHEMICALS LTD

The Rotamix disperser for the paint, printing ink and food industries, in which the rotor draws material from the top and bottom of the vessel and forces it outwards through the stator grill. The Rotamix SM 4 laboratory model and the Whirl were shown. Other machines shown were the Biotomix twin headed Disperser mixer and a Powder mixer of the cone type. Grinding media from Steatize balls to Porcelain balls to steel balls and glass beads.

#### MOLTENI (U.K.) LTD

See under G. J. Erlich Ltd.

# VEB KOMBINAT NAGEMA TRANSPORTMASCHINEN EXPORT-IMPORT

Heidenau triple roll mills Horizontal type 3KFI/600 (280 × 540mm), Inclined type 8/3/I (322 × 800mm) and 9/3/I (400 × 1000mm). The 9/3/I has been introduced and provides four point hydraulic roll pressure control as on the 8/3/I machine. All rolls are fitted with a two speed drive, applied through a clutch and gear box. See also under Marchant Brothers Ltd.

#### NETZSCH FEINMAHLETECHNIK GmbH

The range of machines available are suitable for low, medium and high viscosities, employing the *Molinex Eccentric ring* and *John systems* and also micro-beads. The *John system* 576 can deal with high viscosity pastes normally requiring a triple roll mill. The central rotor and surrounding container are water cooled. The *LM* 506 is suitable for medium viscosity material. The *LM* mill can use beads as small as 0.5mm to achieve a very high degree of dispersion. The *Netzsch Ringslot Separator* ensures separation of the grinding media from the charge. The *LM* mill is capable of obtaining NC and polyamide based inks having the gloss and transparency of chip materials at temperatures of approximately 45-48°C.

#### PILAMEC LTD

A new mill without glands was featured, it is based on the *Megamil 75*, vibrating energy is employed which can be adjusted to suit the particular formulation. The *Megapot* vibratory pot mill for continuous or batch work in the laboratory. The *Megablenda* is an oscillatory ball blender and was also exhibited.

#### **RED DEVIL INC**

Various models of paint reconditioning machines capable of handling containers of up to 25 litres capacity.

#### TORRANCE & SONS LTD

The Torrance Roto Mill (horizontal bead mill) was shown for the first time. The grinding chamber has 250 litres capacity and is jacketed for maximum cooling or heating. Emphasis has been placed on ease of access to the chamber and on the use of water resistant hardened steel alloys for the agitator discs. The machine is designed for continuous operation and can operate under pressure or in an inert atmosphere. The *Pilot Model 1Q Attritor* has established the advantages of the *Torrance Circulatory System* process which can run on a 24 hour basis without risk or attention, as it is fitted with a "fail safe" device. New additions to the range are a 10hp high speed cavitation disperser and a new 10hp paste mixer. The high speed batch bead mill is a very economical equivalent of existing systems of this type.

#### Screens and filters

#### D. H. INDUSTRIES LTD

The Cuno filters and ancilliary equipment, the AB Metall Preba filter housings and Filbag bag type strainers.

#### G. J. ERLICH LTD

Screens from Molteni.





#### JENAG EQUIPMENT LTD

Air driven strainer for the treatment of highly shear sensitive products, such as PVCl pastes, plastisols and organosols, having a nominal capacity of 2000 litres per hour. The new model 1223/9/SB 1.5 is suitable for the resin and wall paper industries and for very heavy paints. The use of the air driven Jenag strainer was demonstrated.

#### **ROBAN ENGINEERING LTD**

The new RIFM line strainer specially designed for use in conjunction with flow meters.

#### **Filling equipment**

#### D. H. INDUSTRIES LTD

The J. De Vree V.1 weight operated filling machine, the V.154 pneumatically operated volumetric filling machine and the V.183 automatic plastic bucket feeder. The Pamasol range of aerosol filling plant from laboratory to 150c.p.m.

#### G. J. ERLICH LTD

The Ge-Halin filling machines for cartridges, cans etc., the H series is semi-automatic. The rotary fully automatic KVF-1 filling machines, both have dosing equipment for heavy pastes.

#### LUDWIG SCHWERDTEL GmbH

S2-M Press/Can destacker/conveyor and vacuum closing unit were shown. The press is fitted with a dosing head to allow filling within  $\pm 0.5$  per cent over a range of 50cc to 6 litres. Colour changes can be made in five minutes.

#### Pumps and valves

#### FLAVEL & CHURCHILL (LIQUID HANDLING) LTD

Pumps and valves for handling all liquid materials. including the A. L. Process Equipment Co. Ltd valves and dynamic self checking level alarms and a range of pumps from Plastic Pumps Ltd.

#### MECA-INOX

Ball valves, gate valves and other valves in stainless steel for the chemical industry and also stainless steel fittings unions etc.

#### **ROBAN ENGINEERING LTD**

A new air driven version of the R 300 series positive displacement solvent pumps, covering a range of 4.5 to 135 litres per minute. A new Mark 2 version of the R 400 tanker off-loading pump, using the Blackmer rotary sliding valve pump.

#### LUDWIG SCHWERDTEL GmbH

A comprehensive range of hydraulic pumps.

#### Flowmeters and viscometers

#### CONTRAVES INDUSTRIAL PRODUCTS LTD

The Covistat industrial regulator for control in open tank and coating applications. The measuring head may be completely submerged and remote control and read-out is available. The DC series of single or multispeed control viscometers for continuous in-line viscosity measurement, they are suitable for high temperature and high pressure applications.









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#### DRESSERWAYNE LTD

The DresserWayne range of Line Meters, which includes the 2PM/4 Micrometer, accurate to  $\pm 0.125$  per cent over a range of 540 to 5400 litres/hour. A pre-set repeating counter is available and is suitable for pressures up to 3.5 Kg/cm<sup>2</sup> and a temperature of 49°C. A wide range of accessories are available for most applications.

#### FLAVEL & CHURCHILL (LIQUID HANDLING) LTD

Industrial flowmeters by the A.L. Process Equipment Co. Ltd.

#### ROBAN ENGINEERING LTD

The 807 ATS solvent flow meter was shown. The Pepperl-Fuchs intrinsically safe batch controller for use in hazardous areas was also shown.

#### Other manufacturing equipment

#### CAPRICORN CHEMICALS

Allplas thermal insulation blankets. Allplas balls are blow moulded from polypropylene, diameters 20, 45 and 150mm, and are unaffected by most industrial chemicals. Each ball has a circumferential rim which causes them to interlock and prevents rotation, in order to reduce exposure of the liquid to the atmosphere. They are used to reduce odour and evaporation losses, retard splashes and can reduce tank heating costs by up to 70 per cent. They can be used on tanks of any shape and are unaffected by changes in the liquid level.

#### CIRP

*CIRP* specialise in the engineering and production of synthetic resin plants. A one unit model of their *Turnkey* chemical system was shown on the stand. Conical reactors which can be produced in sizes from 200 l to 5000 l have been developed, and the 200 l vessel can cope with charges as small as 20 l and still obtain good mixing. A range of valves and fittings from *Meca Inox* were shown in stainless steel, carbon steel, or alloys such as Hastalloy, which are especially suitable for use with paints and resins.

#### D. H. INDUSTRIES LTD

Sussmeyer resin plant and solvent recovery systems, and the AIVD "Clean Can" pan cleaning plant.

#### G. J. ERLICH LTD

The *Ferchim* feeding plant and pneumatic drum emptying plant. Fully automatic vessel and drum cleaning plant, also by *Ferchim*.

#### FERCHIM OF SWITZERLAND

See under G. J. Erlich Ltd.

#### FISCHER INSTRUMENTATION (GB) LTD

Coating process control via coating data obtained from thickness measurements monitored during the process and regulation of the quantity applied, using open or closed loop systems.

#### FLAVEL & CHURCHILL (LIQUID HANDLING) LTD

Pumps, control units, tanks and all ancilliary equipment required for liquid handling, especially for the *Turnkey* system.

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#### HAVAN PLASTICS LTD

Autotex Special Hand Cleaner for removing cellulose, primers, fillers, synthetic paints and enamels, which has been approved by the Royal Institute of Health and Hygiene.

#### LYNCHEM ENGINEERING LTD

Information and photographs of synthetic resin plant with particular reference to electric induction and oil heated types.

#### MARCHANT BROTHERS LTD

The Service and Spares Division provides machinery maintenance, overhaul of roll mills, mixer and general equipment, including camber roll grinding. Comprehensive factory plant removal and installation service is offered to the industry.

#### ROBAN ENGINEERING LTD

The *Turnkey* project, construction service for liquid raw materials, bulk storage and distribution schemes could be discussed with their engineers.

#### SUPERFOS PACKAGING (UK) LTD

Containers produced in high density polyethylene and polypropylene in sizes ranging from 2.5 to 25 litres for paints and many other products. *Superfos* offer complete packaging systems including the *Packline* machine for automatically filling and lidding plastic packs. In addition, a full printing service is available, either screen or offset litho printing in up to four colours.

#### Oils and fatty acids

#### B.V. ASHLAND-SUDCHEMIE VH NECOF

Necomar marine oil cyclopentadiene copolymers. A strongly penetrating, hydrophobic liquid, permeable to water vapour and very resistant to alkalies and water. It is very suitable for wood impregnation, wood stains, transparent varnishes, priming coats and as an additive to improve the adhesion of rust inhibiting primers.

#### CORNELIUS GROUP

Oulu tall oil fatty acids and derivatives.

#### Pigments

#### Inorganic

#### VICTOR BLAGDEN & CO. LTD

The *Chemetron* range of transparent iron oxides for automotive paints and inks.

#### CHEMETRON PIGMENTS DIVISION

Transparent red and yellow oxides for stable colourants and UV absorbers for wood stains and bronze automotive finishes in conjunction with aluminium flake.

#### CIECH-IMPORT AND EXPORT OF CHEMICALS LTD

Zinc chromate, zinc tetroxy chromate, chrome yellow, basic lead sulphate and ultramarine.

#### COLE CHEMICALS LTD

A range of zinc oxides.







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#### CORNELIUS GROUP

The Kemira Oy Vuorikemia range of Finntitan titanium dioxide pigments.

#### **CROXTON & GARRY LTD**

The Promisda natural red oxides of iron.

#### H. HAEFFNER & CO. LTD

Natural and synthetic iron oxides including several new grades and a new grade of micaceous iron oxide.

#### HOOKER CHEMICALS & PLASTICS CORP.

*Ferrophos*, an anticorrosive pigment to enhance the performance of zinc-rich coatings, with which it acts synergistically. It provides improved welding performance, reduces zinc fume and gives welds of no, or very low, porosity at significantly reduced cost.

#### LAPORTE INDUSTRIES LTD

The continuing progress of the chloride process technology was featured. The pigments displayed were: Runa RH 472 High durability and ease of use. Runa RO 676 Highest durability in long life coatings. Runa RE 372 Medium surface treated grade combining good colour and opacity in top quality medium PVC emulsion paints. Runa RE 376 Highly surface treated, maximum opacity and stability and film integrity in medium—high PVC emulsion paints. Runa RH 52 A sulphate process pigment which combines gloss potential and easy dispersibility in critical industrial systems and gives high durability not usually found in this type of pigment.

#### MELBOURN CHEMICALS LTD

Albarex, a highly effective secondary pigment for use in anticorrosion paints.

#### MONTEDISON GROUP

A wide range of titanium dioxides in both rutile and anatase types for all purposes ranging from paints and inks to rubber, plastics and flooring materials. **RS** is a new rutile grade, specifically developed for plastics subjected to outdoor exposure.

#### TISZAMENTI VEGYIMUVEK

A wide range of chromium based pigments, including zinc chromate, chrome yellows, chromium oxide green, etc. The zinc chromate is basic chromate for primers but can also be used as a brilliant greenish yellow in either self shades or in admixture with blue. The lead chromes are based on the *ICI* processes and are available in eight shades from primrose to middle chrome.

#### Metallic and pearl pigments

#### **REX CAMPBELL & CO. LTD**

The Silberline Manufacturing Inc and Silberline Scotland ranges of non-leafing aluminium pastes and Sparkle Silver of exceptional brilliance for a number of industrial, automotive and general paints and for printing inks.

#### COLE CHEMICALS LTD

Aluminium powders and pastes and bronze powders notable for their fine lamination which enables 20/30 per cent economy in use.

#### CORNELIUS GROUP

The Mearl range of pearl pigments, including  $TiO_2$  coated mica, etc.

#### **CROXTON & GARRY LTD**

Metallic and Polyester Jewels, sparkling flake pigments for decorative effects in 68 colours for metallic industrial finishes and textured wall paints. The *Resist* range of coated, tarnish resistant bronze powders which can be sprayed electrostatically.

#### DURHAM RAW MATERIALS LTD

Recent developments in the use of *Durham* zinc dusts in anticorrosive paints were illustrated.

#### SILBERLINE LTD

A new Sparkle Silver 3334 AR, intermediate between the coarse 3000 AR and the medium 5000 AR. The new grade has the brilliance of the coarse grade, but gives freedom from seeding and the gloss given by the fine grades. The use of Sparkle Silver non-acid resistant pastes, where special quality non-leafing products can be used to advantage, such as: PVCl decorative leather cloth, vinyl paper coatings, hammer finishes and gold effects in non-leafing inks for packaging.

#### **Dispersions and pastes**

#### B. V. ASHLAND-SUDCHEMIE VH NECOF

The *Necolor* range of pigment/pastes for application in wood stains and similar materials.

#### VICTOR BLAGDEN & CO. LTD

Chemetron dry and flushed colours for paints, inks and plastics.

#### CHEMETRON PIGMENTS DIVISION

Sangatuck red is available in the dispersed form. The *Optimix* system whose components can be mixed without generating incompatibility embraces a system of 3 vehicles, 6 pre-dispersed pastes and 3 compounds.

#### CORNELIUS GROUP

The Hilton Davis range of dispersed and flushed pigments.

#### H. HAEFFNER & CO. LTD

The Colorblend Dispersion Service embracing pigment dispersions into aqueous media to impart freeze-thaw stability, thixotropy, etc.; dispersion of powdered materials to eliminate dust and aid metering; blending dispersions where additives and pigments are used as mixtures and pigment dispersion for both flexible and rigid PVCI.

#### INDUSTRIAL DISPERSIONS LTD

Industrial dispersions for a variety of industries. The predispersed pigment pastes were illustrated for many applications using paste concentrates. A range of *Standard Dispersions* is offered in plasticisers, oils, polyester and epoxy resins, glycols and "*Universal*" media.

#### MONTEDISON GROUP

A new range of *Sintosol N* aqueous pastes of the azo and phthalocyanine types for decorative emulsion paints and

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water based inks, which offer considerably impr strength and stability.

#### SWADA (LONDON) LTD

The *Z0 Extra Concentrates* for inks have been upgraded to improved printability and a new shade *Luna yellow* has been added. A water based pigment paste for paper coating in the A, D or E types is available. See also under *Fluorescent pigments*.

#### Organic

#### VICTOR BLAGDEN & CO. LTD

The *Chemetron Inc.* range of dry and flushed colours for paints, inks and plastics.

#### CHEMETRON PIGMENTS DIVISION

A new dry alkali blue, easily wetted by oil ink vehicles in the preliminary mixing process, subsequent to grinding carbon black will also disperse this blue. *Sangatuck red* is a clean bright colour ranging from *Red lake C* to the orange area. It can be used to replace molybdate orange in gravure and flexographic inks. These reds are available in the dry or dispersed form. *Azalean B*, a blue hue magenta, now in the dry form may be used as an economic alternative for more expensive pigments.

#### CORNELIUS GROUP

The Hilton-Davis range of pigments.

#### HERCULES POWDER COMPANY LTD

The latest advances in pigment technology were described.

#### MONTEDISON GROUP

A wide range of ACNA organic pigments, including mono azo derivatives, bis azo derivatives, aniline condensates and phthalocyanines for all purposes in the paint, printing ink and plastics industries. The *T pigments* have been developed to replace chrome yellows in a variety of applications.

#### SWADA (LONDON) LTD

A new limited range of non-flourescent basic dye toners (*PMA*, *PTMA*, *SMA* and *CF*) and four azo colours.

#### Fluorescent

#### CORNELIUS GROUP

The Flare series of fluorescent pigments.

#### HERCULES POWDER COMPANY LTD

The Radiant Colour range of fluorescent pigments was exhibited.

#### SWADA (LONDON) LTD

The Fiesta Daylight Fluorescent Pigments. ZO Extra concentrates for letterpress and litho inks now include the new Luna yellow (See Organic pigments). The E-series for paper coating also includes Luna yellow. The Z-series for plastics is now replaced by the ZN-series, which are stable up to 290°C and contain no formaldehyde. For paints the A and D types continue to be used, but the new E-series gives certain advantages. The GT pigments are based on the same resin matrix as the T-series, and are now largely used in urethanes, and are suitable for skate board wheels.

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#### Resins

#### General

#### AMOCO CHEMICALS (U.K.) LTD

The Amoco-Fina Indopol Polybutenes for roofing compounds or hot-melt applied pressure sensitive adhesives, sealers and exterior textured finishes. Resin 18 is a poly  $\alpha$  methyl styrene for use in alkyd paint formulations.

#### B.V. ASHLAND-SUDCHEMIE VH NECOF

Necomar marine oil/cyclopentadiene copolymer which is strongly penetrating, hydrophobic and permeable to water vapour, it is very resistant to alkalines and water and is suitable for wood impregnation, woodstains, transparent varnishes and as an additive to improve the adhesion of rust inhibiting primers.

#### VICTOR BLAGDEN & CO. LTD

The Ashland-Sudchemie range of cyclopentadiene modified oils. See above. The Neville Cindu range of hydrocarbon resins. See below.

#### CHEMETRON PIGMENTS DIVISION

*Tagset* liquids and gels providing a new series of ink vehicles suitable for rapid drying under IR radiation.

#### CHEMOLIMPEX

Colophony based resins, resin esters, and maleic resins. Colophony modified phenolic resins for printing inks.

#### COLE CHEMICALS LTD

A range of coumarone, speciality hydrocarbon and terpene resins.

#### CORNELIUS GROUP

The *Methylon* modified phenolic resins. Nitro cotton in various viscosity ranges and damping alternatives and clear or pigmented plasticised chips.

#### **CROXTON & GARRY LTD**

Quram, a quarternary ammonium silicate binder for water based zinc-rich paints.

#### COMPAGNIE FRANCAISE GOODYEAR

*Pliolite* range of synthetic rubber copolymer solution resins for interior and exterior masonry finishes, including copolymerswith styrene, vinyl toluene, butadiene and acrylics; they have excellent adhesion, water and alkali resistance, chemical inertness and heat stability, together with reliable drying.

#### HERCULES POWDER COMPANY LTD

Water white resins and higher melting reactive phenolic modified resins. An increased range of *Dresinol* resin based emulsions which are completely solvent free.

#### KWR CHEMICALS LTD

The Orsol phenolic resins for paints, can coating, drum lining and electrodeposited primers. Modified Orsol resins for can coating from Societ's Francaise D'Organo Synthese.

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#### MONTEDISON GROUP

Phenolic resins (Resiafen and Resurfene).

#### NEVILLE CINDU CHEMIE B.V.

Necires LF and LX petroleum derived hydrocarbon resins and Nevchem. Coumarone—indene resins Necires ALX-100 and Necires RF. A range of low viscosity epoxy resin extenders Necires EPX-L and coal tar compounds having good compatibility with epoxies and polyurethanes.

#### SANYO-KOKUSAKU PULP CO. LTD

A range of Superchlon chlorinated polyolefins, including polypropylene (CPP), polyethylene (CPE) and chlorinated rubber (CR). CPP and CPE are chlorinated rubber like resins which have been in use for over 10 years, easily soluble in many aromatic solvents giving rather low viscosity solutions. Plasticised and pigmented films have excellent acid and alkali resistance, good exterior durability and low water permeability. They are specially suitable for high build marine and maintenance paints and also for printing inks. CPP is compatible with chlorinated rubber. CPE does not require additives which might affect the final coating properties and is suitable for airless spray finishes.

# WORSDALL CHEMICAL COMPANY LTD and CANADIAN WORSDALL CHEMICAL CO. LTD

The use of *Surcoprene* rubber hydrocarbon resin in paints and inks is well known and improvements in quality were shown. The melting point has been raised, the viscosity increased, and compatibility with other varnish like raw materials has been greatly improved. Results have shown that with the *Estokyds* tested, compatibility was universal.

#### Acrylic resins

#### ALLIED COLLOIDS LTD

The Surcol range of acrylic polymers. The new Surcol 1460 is a reactive polymer which cross links with UF and MF resins and with isocyanates giving excellent adhesion to metals and plastics; the uses include aircraft and vehicle finishes and printing inks for plastic films. Other Surcols include polymers for continuous coating and the reprographic industry. The Glascol G acrylic solutions.

#### CORNELIUS GROUP

The range of acrylic resins from Roehm GmbH.

#### S. A. FLORIDIENNE NV

The S. C. Johnson & Son Inc. full range of Joncryl acrylic polymers which provides acrylic resins for almost every use in the coating and printing ink industries. Water based acrylics, see under *Emulsion & water based*.

#### COMPAGNIE FRANCAISE GOODYEAR

The range of *Pliolite* resins (*Resins-general*) includes acrylic co-polymers in various viscosity grades and various rheological properties.

#### KWR CHEMICALS LTD

Orsol acrylic resins for water based inks and for water/ alcohol flexo and gravure inks from Societé Francaise D'Organo Synthese.

#### MONTEDISON GROUP

Acrylic and styrene-acrylic dispersions (Crilat) and acrylic resin solutions (Resiacril).

#### Alkyd resins

#### BARFORD CHEMICALS LTD

A range of drying and non-drying alkyd resins, including an economic alkyd based on a modified coconut oil fraction, and water dispersible alkyd resins.

#### BRITISH INDUSTRIAL PLASTICS LTD

Beetle resin BA549, a short oil semi-drying alkyd designed primarily for use in low bake systems. It may be used in combination with many Beetle melamine resins, including BE687, BE664, BE683 and BE681. Such systems will cure well at around  $90-95^{\circ}$ C and then continue to post cure well at room temperatures for several weeks. All these systems have excellent shelf stability.

#### CHEMOLIMPEX

Over thirty alkyd resins for numerous applications.

#### CIECH-IMPORT & EXPORT OF CHEMICALS LTD

A range of phthalate resins is available.

#### CORNELIUS GROUP

The specialised *Finntitan* alkyd resins for specific purposes from *Kemira Oy*.

#### MONTEDISON GROUP

A range of Resial and Restrol alkyd resins.

# WORSDALL CHEMICAL COMPANY LTD & CANADIAN WORSDALL CHEMICAL CO. LTD

A new alkyd, *Estokyd A20* for use in inks and quickset ink vehicles *Estoset 605* and *Estoset 620*.

#### Amino resins

#### BRITISH INDUSTRIAL PLASTICS LTD

*BE683*, a new n-butylated melamine formaldehyde resin supplied at relatively high solids and at a viscosity suitable for tanker delivery. It has a wide range of compatibility, high reactivity, yet good flow and a high tolerance for white spirit. *BE683* is useful in the formulation of "metal dec", automotive and high class general industrial finishes, and in paper coatings. It is particularly recommended for use with oil-free alkyds such as *Beetle BA500* and a wide range of thermosetting acrylic resins.

#### CHEMOLIMPEX

A range of amino resins.

#### EMSER WERKE AG

See under Grilon.

#### GRILON (U.K.) LTD

The Grilamid Nylon 12 for powder coatings was shown.

#### MONTEDISON GROUP

The Resiplast, Resfurin and Resmelin ranges of amino resins.

#### Emulsion and water based resins

#### ALLIED COLLOIDS LTD

The *Glascol* range of water based resins including a number suitable for water based inks, temporary coatings, can coatings and artist's colours.

#### BARFORD CHEMICALS LTD

See under alkyd resins.

#### EMSER WERKE AG

See under Grilon.

#### S. A. FLORIDIENNE NV

A number of S. C. Johnson & Sons Inc. acrylic resins for water, alcohol or glycol based inks, water dilutable inks, a high solids acrylic emulsion for water based inks and coatings. A colloidal suspension of acrylic tetrapolymer for wood finishes, paper coating, inks and metal decoration and an improved styrenated acrylic dispersion for fast drying water flexo and roto inks. Versacryls 763, 765 and 766 are water based coatings as alternatives to hot wax coatings and polyethylene film laminates in the packaging industry.

#### GRILON (U.K.) LTD

Grilonit epoxy resins and curing agents for water dispersible epoxy coatings.

#### HERCULES POWDER COMPANY LTD

An increased range of *Dresinol* resin based emulsions which are completely free from solvent. Water soluble polymers from both European and USA sources.

#### KIRKLEES CHEMICALS LTD

Vinyl acetate copolymer emulsions emphasising economy and quality. A new emulsion is now offered for high PVC paints, in addition to those already manufactured for textured silk and interior/exterior paints.

#### KWR CHEMICALS LTD

The Societé Francaise D'Organo Synthese acrylic vehicles for water based inks and coatings and for water/alcohol based flexo and gravure inks.

#### MONTEDISON GROUP

Homopolymer and copolymer vinyl acetate dispersions and acrylic and styrene-acrylic dispersions (*Crilat*).

#### **Epoxy** resins

#### CHEMOLIMPEX

A range of epoxy resins is available.

#### DOW CHEMICAL COMPANY

The experimental epoxy resin *XD5567.01* is a very low viscosity bisphenol F containing liquid epoxy resin. See under *Additives*.

#### EMSER WERKE AG

See under Grilon.

#### GRILON (U.K.) LTD

The *Grilonit* epoxy resin and *Grilonit* curing agent developments for water dispersible, solvent free epoxy coatings were described. *Grilonit* powder coatings based on epoxy and epoxy-polyester combinations were also displayed.

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All photographs in the Exhibition Report and report of the 60th Anniversary Commemorative Celebrations were taken by the Exhibition Official Photographer, RON LUCAS PHOTO-GRAPHY LTD.

#### Acknowledgment

The Exhibition Committee is indebted to the Honorary Editor, Mr S. R. Finn, for his work in the compilation of this report.

#### FLAVEL & CHURCHILL (LIQUID HANDLING) LTD

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The comprehensive Colorblend Dispersion Service was shown.

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# PAINTMAKERS ASSOCIATION OF GREAT BRITAIN LTD

A display on behalf of the *Technician Education Council*. The *TEC* which is completely changing the pattern of technician training was described and details of the aspects relevant to the paint and allied industries were available.

#### POLYMERS PAINT & COLOUR JOURNAL

Copies of *PPCJ* were displayed, in addition to the paint and allied industries; the journal now publishes a special supplement on adhesives. The 1978 edition of the *PPCJ Year Book* and the "*Technology of Powder Coatings*" by S. T. Harris published by the *Portcullis Press Ltd* were shown.

#### ROBAN ENGINEERING LTD

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#### SOCIETY OF DYERS & COLOURISTS

The Society's various publications were displayed, including the *Colour Index*, a selection of literature, together with "Careers in colour", and staff were available to discuss the activities of the Society in the field of colouration.

#### WHEATLAND JOURNALS

Copies of *Paint Manufacture*, which covers all aspects of paint production, were shown. *Finishing Industries* and the 1978 edition of the *Finishing Industries Manual* were also available.

# **Report of Council Meeting**

A Meeting of Council took place at the Great Northern Hotel, London NI on 5 April, 1978 with the President, Mr A. McLean, in the Chair. There were twenty-four members of Council present.

Reports were received on the final arrangements for the 1978 Exhibition, and the lease for the hire of Alexandra Palace for the holding of OCCA-31 (3-6 April, 1979) was signed.

The Annual Accounts of the Association were adopted for inclusion in the Annual Report of the Council to be circulated in May. The arrangements for the Luncheon Lecture arranged by the London Section to precede the Annual General Meeting on 21 June were complete.

It was resolved that the names of any members still in arrears with 1978 subscriptions at the end of June should be removed from the Register.

The Honorary Editor reported that the Review of OCCA-30 would appear in the June issue of the *Journal*.

Details concerning the papers offered for presentation at the Association's Conference at Stratford-on-Avon (20–23 June, 1979) were given.

Reports were received on the arrangements for the 60th Anniversary Commemorative Lecture and Dinner to be held on 11 May and the Dinner Dance on 12 May.

Section Chairman and Representatives reported on the activities of their Sections and Branches at home and overseas, including the Divisions in New Zealand and South Africa.

This being the last Council Meeting before the Annual General Meeting, the President thanked those members who would be retiring from Council for their service and all members of the Council for their support throughout his first year of office, before declaring the Meeting closed.

#### News of Members



During February, Dr Marianne Ellinger, a long standing member of the Association, visited Australia and is shown sporting her OCCA tie even in Australia's hottest ever summer.

Mr F. W. Cole, an Ordinary Member attached to the London Section and an Associate in the Professional Grade, has taken up a position with Morris Ashby Ltd.

Mr S. W. H. Naqvi, an Ordinary Memebr attached to the General Overseas Section, formerly attached to the Thames Valley Section, has been appointed Technical Manager for Jenson & Nicholson of Pakistan Ltd.

# 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.

#### June

June

Irish Section: Golf outing-details and date to be announced.

#### Friday 2 June

Scottish Section—Eastern Branch: Car rally and barbeque—details to be announced

#### Wednesday 21 June

#### Association Annual General Meeting

The Annual General Meeting for 1978 will be held on Wednesday 21 June at the Piccadilly Hotel, London W1, commencing at 12.30 p.m. with a reception in the Edward Room. This will be followed at 1.00 p.m. by Juncheon.

At 2.15 p.m., in the Margaret Room, Dr D. Davies, Chief Scientist of the Department of Industry, will deliver a lecture entitled "Resource problems in the downstream chemical industries".

At 3.00 p.m., or as soon thereafter as the lecture shall have terminated, the Annual General Meeting will be held in the Margaret

Room. Members not wishing to attend the luncheon and lecture will, of course, be able to participate in the Annual General meeting.

Tickets for the luncheon/lecture are priced at £6.48 each, inclusive of wine and VAT, and are available from the Association's offices at the address on the Contents page.

#### Symposia

#### New Zealand Convention

The New Zealand Division is organising its 16th Annual Convention to be held in Rotorua from 27th to 30th July 1978. Details can be obtained from Mr P. F. Sharp, P.O. Box 22-122, Otahuhu, Auckland, New Zealand.

#### **Coatings 78**

The South African Division of the Association is organising a Conference to be held on 2-5 November 1978 in Cape Town with the title 'Coatings 78 – Economy, ecology, performance'. Those wishing to receive further details should contact Mr P. J. Quorn, c/o Inmont SA (Pty) Ltd, Kinghall Avenue, Epping No. 3, 7460 South Africa.

# **Professional Grade-**

The following Ordinary Members have been admitted to the Professional grade.

The Section to which each Member is attached is shown in brackets.

#### Associate

Christopher Peter Rycroft (London)

#### Licentiate

Randal Peter Rowntree (*Manchester*) Anoop Kumar Saggar (*General Overseas—Kenya*)

## **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**

Bowd, GRAHAM LAWRENCE, BSc, 20 Kamara Road, Glen Eden, Auckland 7, New Zealand. (Auckland)

BULL, ANTHONY, 31 Hawksmoor Close, Whitchurch, Bristol. (Bristol)

CAVENDISH, JAMES, Manchem Ltd, Ashton New Road, Manchester. (Manchester)

- CROZIER, PETER HUGH, BSc, 51 Rutland Road, Mount Wellington, Auckland 6, New Zealand. (Auckland)
- GIFFNEY, JOHN KENNETH, 61 Victoria Street, P.O. Box 13-231, Onehunga, New Zealand. (Auckland)
- GILMORE, ROBERT ANDREW, 10 Ramsdale Road, Bramhall, Stockport. (Manchester)
- HOUGHTON, CHARLES ARCHIBALD, BSc, P.O. Box 727, Cape Town 8000, South Africa. (Cape)
- REECE, PATRICK WILLIAM, 21 Chipperfield Drive, Kingswood, Bristol. (Bristol)
- SMALLWOOD, PAUL RAYMOND, BSc, 43 Warwick Drive, Hazel Grove, Stockport SK7 5LQ. (Manchester)

TURNER, ROY, GradRIC, 10 Highbarn Road, Alkrington, Middleton, Manchester M24 1DT. (Manchester)

Tye, Geoffrey Charles, Lusteroid Paints, Box 22122, Otahuju, New Zealand. (Auckland)

#### **Associate Members**

- FASTIER, JOHN DUNCAN, HENRY H. YORK & Co. Ltd, Box 407, Auckland, New Zealand. (Auckland)
- LAW, MICHAEL DOUGLAS, 99 Chelsea View Drive, Birkenhead, Auckland, New Zealand. (Auckland) ROYDES, JAMES TREVOR, 80 Hollin Avenue, Middleton, Manchester,

Lancs. (Manchester)

#### **Registered Students**

- BRODERICK, THOMAS PATRICK, 466 Southend Road, Elm Park, Hornchurch, Essex. (London)
- SHAH, PANKAJ, 354/55 Raviwarpeth, Krishnanivas, Poona 411002, Maharastra, India. (General Overseas)


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(Reference Audit Bureau of Circulations Reviews. Jan-Dec 1976)

For full details of advertising in this, and other Association publications, contact D. M. Sanders, Assistant Editor

#### Journal of the Oil and Colour Chemists' Association (JOCCA)

Priory House, 967 Harrow Road, Wembley, Middx. HA0 2SF, England Telephone: 01-908 1086 Telex: 922670 (OCCA Wembley)



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## 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.

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