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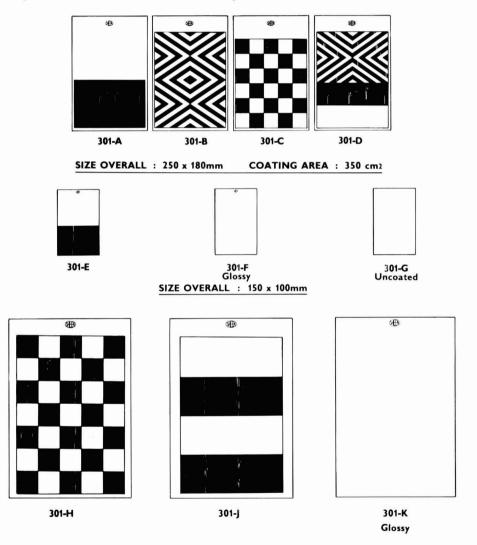


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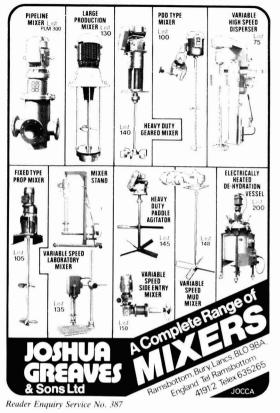
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# JOURNAL of the OIL AND COLOUR CHEMISTS' ASSOCIATION

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# Performance of a novel corrosion inhibiting pigment of low toxicity, in ferrous metal primers, applied to marginally prepared surfaces

# P. A. Draper

Patrick A. Draper, Coating and Construction Resin Consultants, P.O. Box 325, Hillcrest 3650, Natal, South Africa

#### Summary

The new ferro-organic pigment shows promise as a non-toxic replacement for conventional anti-corrosive pigments. Details of laboratory evaluation by means of accelerated testing of the pigment are presented. The protection afforded to abrasive blast cleaned steel is compared to that of wire brushed, previously rusted steel. In addition the results of a limited amount of outdoor exposure are also discussed. The pigment has been useful in both epoxy and alkyd binders and the paper deals with the experimental work carried out to produce starting formulations of primers in these mediums which have performance at least equal to those based on pigments such as red lead, zinc chromate and zinc phosphate. A tentative theory for the corrosion inhibiting action of the pigment is advanced.

# Introduction

Since the impact of ecological studies and modern methods of classifying potentially poisonous materials, the paint industry had never been the same. Gone are the days, when paint chemists nonchalantly used the first material which came to hand to solve a particular problem. Now, first of all there is a soul searching, to decide whether the material is likely to fall foul of legislation and secondly the chemist is so brainwashed, if he uses a suspect material, he examines himself critically daily for untoward bumps, lumps or wheezing. Small wonder then, that when a new product becomes available, which is proclaimed free of all these problems, it is latched upon with glee and soon becomes a household word in the industry. Sometimes such products are of doubtful value, and often so expensive that their use becomes prohibitive. This is the background against which the development described in the paper was commissioned.

A new pigment for the passivation of steel corrosion was first produced some years ago and a limited laboratory evaluation of its properties was carried out. At this stage it showed some promise but as with many new substances, yields, even on a laboratory basis were so low, that it was totally uneconomic to produce commercially. Research was then concentrated on improving the yield. After some years a commercially acceptable yield was achieved. This is the subject of a patent application. Because the yields were such that pigment now appeared economic, an extensive evaluation was undertaken. which it was hoped would answer the following questions:

- Is the material useful as an anti-corrosive pigment?
- Will primers produced from the pigment, be competitive?
- 1985(10)

• Is the pigment toxic?

This paper sets out to attempt to answer these questions.

#### Experimental

# Description of the pigment

The pigment is a near black amorphous powder. It is described chemically, as an organo-ferrous compound. It is marginally soluble in water with a specific gravity of 1.64. During all the development work the pigment had no trade name and became known merely as "AC pigment". This provides an easy non-commercial means of referring to the pigment and it is referred to as such in this paper.

Figures 1 and 2 are the chemical characterisation of the new pigment. Figure 1 is an IR trace on the dry pigment and shows the molecule to be an hydroxyl rich polyphenolic material. Figure 2 is a scanning electron microscope elementary analysis of AC pigment. It can be seen that the major inorganic component is iron and there is a complete absence of heavy metals.

# **Evaluation of corrosion resistance**

It was decided to choose common binders used in anticorrosive primers and to prepare primers from them using simple, classical formulations. Controls using common anticorrosive pigments, were to be prepared at the same time and finally a number of well known proprietory primers of the same type were to be included to compare the overall standard of the program. Epoxy resin primers are common shop primers worldwide and it was decided to choose epoxy as one binder. In maintenance situations, alkyd based systems are much more common, especially when good surface preparation is not possible and an alkyd was chosen for the other binder.

# Test Method

Much has been written on the unreliability of the various sodium chloride salt spray tests in predicting primer and paint durability<sup>1,2,3</sup>. However there is some consensus<sup>2,4</sup> that salt spray testing with a mixture of ammonium chloride and sodium chloride provides more realistic performance data. Accordingly it was decided to use the "Dr Harrison mixture" of 3.25 per cent ammonium sulphate and 0.25 per cent sodium chloride. Prohesion testing<sup>5</sup> which is claimed to give superior prediction, in addition calls for intermittent exposure. However, led to a modification of the cycle. It was suggested that the reason for the different and more

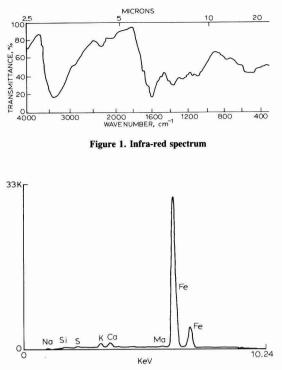


Figure 2. Elementary analysis by scanning electron microscope

realistic mode of failure was alternate swelling and shrinking of the paint film during wet and dry cycles. It is unlikely that a paint film will fully absorb water in one hour and completely dry in one hour. It was thus decided to run a 12 hour wet, 12 hour dry cycle. The exposure time recorded in the results is the total time, i.e. wet and dry cycles combined. In addition to the accelerated corrosion tests described above, certain of the better formulations were also exposed in the Durban Bayhead area. This is an industrial/marine environment and Durban is one of the most corrosive areas in the country. Recent results published by the Council for Scientific and Industrial Research<sup>6</sup> show a corrosion rate for mild steel in the Bayhead area of 55.9 micrometers per year for the first two years. It will be noted that this is extremely high and is caused by the year round high humidity (usually between 70 per cent and 95 per cent) and the high average ambient temperature which is seldom out of the range of 20-30°C.

# **Panel preparation**

Two substrates were used throughout the tests. In the first case, abrasive blast cleaned steel to Swedish standard SA3, was used while in the second, a sheet of naturally rusted cold rolled steel was selected and cut into panels. These were wire brushed to Swedish standard C St2 and then rinsed with distilled water and dried before use.

A nominal 30 micrometres of the primer was applied followed by 30 micrometers of the relevant enamel. Due to the difficulty of using precision methods of paint application, because of distortion of the panels caused by surface preparation, spray application was used. This entailed dry film thickness checking and where necessary, recoating panels where the film thickness was out of the 25-35 micrometre range for either coat. In the case of primers, the paint film was not cross scribed but the top coated panels were.

#### **Evaluation of exposed coatings**

After exposure the primers were rated according to Swedish standard SIS 18 51 11 using the photographic standards. At each inspection the panels were compared and rated on a 10-1 scale of paint protection efficiency, 10 being rust free and 1 being completely rusted. In addition, blister and creep at the scribe was recorded.

# **Epoxy primers**

A published Shell formulation<sup>7</sup> was chosen as the starting point for the test products. The main binder was Epikote 1001 cured with Versamid 115. However, the acrylic intercoat adhesion modifier was changed to polyvinyl butyrate. In this paper all quantities in the formulations are volumes of pigment, as pigment volume concentration (PVC), for facility of comparision.

# Establishing controls

The most common pigments used in epoxy based primers are zinc chromate and zinc phosphate. In order to establish controls, a range of primers was prepared with increasing PVC and submitted to preliminary corrosion testing as described above. The primers which performed the best, based on these pigments were selected for use as controls in the later tests on the new pigment. Figure 3 is a graphical summary of the results. Reference to Figure 3 shows that a PVC of the active pigment of 4.3 per cent gave somewhat better results, especially on the wire brushed surface.

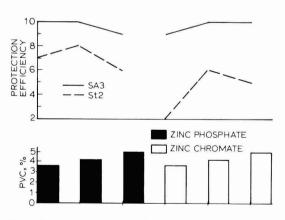


Figure 3. Corrosion resistance of epoxy primers on conventional pigments

#### Test series of epoxy primers with AC pigment

A range of primers was prepared with increasing AC pigment quantities and applied to panels as described

previously. Later in the paper there is some discussion on the mechanism of the corrosion inhibiting action of the pigmet. It was suspected that metallic pigments may interfere with the theorised action and to test this a duplicate series included red iron oxide. The total PVC was balanced to 30 per cent by varying the talc quantity. The two control formulations on zinc phosphate and zinc chromate are included for completeness and the exposure series also included two proprietory primers, one zinc chromate and one zinc phosphate of unknown PVC. Table 1 lists the formulations used.

# Results of corrosion tests on AC pigment epoxy primers

The results of the corrosion tests are represented graphically in Figure 4 and apply to a salt spray exposure period of 1000 hours for the primer. These are the results shown in the graphs.

A number of observations may be made from these results:

- Red oxide, at the levels used in the formulations, has a deleterious effect on the salt spray resistance of the primers containing the new pigment.
- 2. A marked peak occurs at about the 10 per cent AC Pigment PVC level and performance drops off on either side of the peak.
- 3. The wire brushed panels performed at least as well as the blast cleaned panels and on these results even a little better.

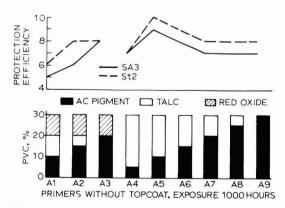


Figure 4. Corrosion resistance of epoxy primers containing different levels of AC pigment

# Comparative results on all the epoxy primers

The best formulation from the previous series (A5) is compared with the two controls (A10 and A11). Figure 5 presents the results graphically. The results for the topcoated panels are also included. Exposure time was 1000 hours for the primers and 2000 hours for the top coated panels.

Table 1. Pigment combinations used in epoxy test series

Reference	AC pigment	Talc	Red Oxide	Zinc Phosphate	Zinc Chromate
A1	10.00	10.00	10.00		
A2	15.00	5.00	10.00		
A3	20.00	0.00	10.00		
A4	5.00	25.00			
A5	10.00	20.00			
A6	15.00	15.00			
A7	20.00	10.00			
A8	25.00	5.00			
A9	30.00	0.00			
A10		6.50	19.20	4.30	
A11		6.50	19.20		4.30

Comparing the primers without a top coat, the AC based primer was better than the controls, except for the commercial control based on zinc phosphate. It also performed better than the proprietory zinc chormate which had a salt spray resistance about equal to the control zinc chromate. The control zinc phosphate performed considerably worse than the proprietory product. This is not surprising as one expects a commercial product to have been optimised. On the other hand, the new pigment has upgraded the basic primer to the extent that it is virtually equal to the best commercial product and better than like formulations on conventional pigments. It is interesting to note that the proprietory zinc chromate primer performed very well when overcoated, with respect to rust creep from the scribe on the blasted panel. On the wire brushed panel it was the least successful. The AC gave similar results (very little creep) on the blasted panel but was also equal to the best on the wire brushed panel.

### **Alkyd primers**

A standard commercial alkyd resin was used for the binder in this series. The manufacturer of the resin modified it with the necessary driers, anti-skinning agents etc. The resin has the following properties:

Trade name: Beetle 30-2033 Solids (%): 68-72 Solvent: White spirit Colour (PRS Scale): 2 maximum Viscosity (Gardener Holdt): Z4-Z6 (25°C) Phthalic Anhydride (% min): 25% Type of oil: Mixed vegetable

#### Establishing controls

As in the case of the epoxy, an optimum level of zinc chromate and zinc phosphate was sought by preparing primers on increasing levels of active pigment. Total PVC was 37 per cent. None of the primers, except the red leads, performed well when exposed to salt spray without topcoats, and failure was so rapid that it was impossible to discriminate between the different formulations. The topcoated panels performed much better and a pattern soon developed as shown in Figure 6.

It can be seen that the primers applied to blast-cleaned

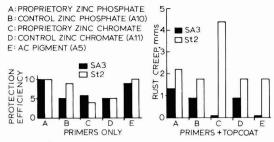


Figure 5. Corrosion resistance of controls and best AC formulation

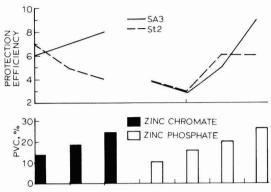


Figure 6. Corrosion resistance of alkyd control formulations

panels improved as the pigment increased. On wirebrushed panels, the zinc chromate primers got worse as the pigment increased however. The highest levels were chosen for use in the later testing and these approximate the levels laid down in the national specifications for these materials. The exposure time was 2000 hours.

# Test series for alkyd primers

As with the epoxies, a range of primers was prepared and applied to panels as described previously. In this case too, a comparison with primers containing AC pigment and including red oxide was undertaken. The total PVC was balanced to 37 per cent with talc. Table 2 gives the breakdown of the pigment loadings.

# Results of corrosion tests on alkyd primers with AC pigment

Blistering occurred on some of the panels and graphical representation of the results is difficult. They are presented in Table 3.

The following observations can be made from these results:

- 1. As with epoxy, red oxide has reduced the performance of the primers when used in conjunction with AC pigment.
- 2. The quantity related peak is again strongly evident in this case. Later work suggests that about 10 per cent

 Table 2.

 Formulations of alkyd primers with varying AC pigment

Reference	AC pigment	Talc	Mica	Red Oxide	Zinc Chromate	Zinc Phosphate
B1	7.40	28.00	7.00	2.00		
<b>B</b> 2	14.10	13.90	7.00	2.00		
<b>B</b> 3	20.50	7.50	7.00	2.00		
<b>B</b> 4	26.30	1.70	7.00	2.00		
B5	29.60	0.00	5.40	2.00		
<b>B</b> 6	5.00	30.00	7.00	0.00		
<b>B</b> 7	8.90	30.00	7.00	0.00		
<b>B</b> 8	17.00	13.00	7.00	0.00		
<b>B</b> 9	24.20	5.80	7.00	0.00		
<b>B</b> 10	31.00	0.00	6.00	0.00		
B11	0.00	3.20	7.00	2.00	24.80	
B12	0.00	1.30	7.00	2.00		26.70

 Table 3.

 Corrosion resistance of alkyd primers based on AC pigment

1	AC	Wire brushed		В	last cleaned
Reference	pigment	Rust	Blistering	Rust	Blistering
B1	7.40	6	6F on X	6	7M on X
<b>B</b> 2	14.10	5	nil	8	nil
<b>B</b> 3	20.50	6	6M on X	6	8M on X
<b>B</b> 4	26.30	5	6M on X	8	8F on X
<b>B</b> 5	29.60	5	6F general	7	Threads on X
<b>B6</b>	5.00	7	nil	7	6F on X
<b>B</b> 7	8.90	9	nil	8	6F on X
<b>B</b> 8	17.00	8	nil	7	nil
<b>B</b> 9	24.20	7	8M on X	7	8F on X
<b>B</b> 10	31.00	6	8M on X	7	6F on X

Reference numbers B1-B4 contained 2% PVC red oxide. Blistering rated in accordance with ASTM scale.

PVC is the optimum amount of AC pigment to use in an alkyd primer.

3. On the 8.9 per cent AC pigment formulation, general rusting on the panel is unaffected by the method of surface preparation. The defects associated with the scribe were less than on the blast cleaned panels.

# Comparison with controls and standard formulations

The control formulations have been discussed previously. In addition, some of the controls used were commercial products produced under the S.A. Bureau of Standards mark<sup>8,9</sup>. The published details of these products are shown in Table 4.

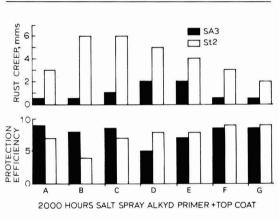
The results of the corrosion tests have been summarised graphically and are presented in Figure 7. The corrosion resistance of the alkyd primer based on AC pigment has once again proved equal to the best, as far as protection efficiency is concerned. Resistance to rust creep is also very good and taking overall properties, the AC pigment

Table 4.		
Compositions of SABS products use	ed as	controls

Primer type	Binder type
Zinc chromate	Long oil alkyd (20% phthalic anhydride)
Red lead Type II Grades 1 & 2	50% Alkyd (30% phthalic anhydride) 50% Raw linseed oil
Red lead Type III	Alkyd resin (30% phthalic anhydride)

Pigment composition (% mass/mass)	Zinc chromate		Red lead Grade 2	
Zinc chromate (min)	50			
Zinc oxide (min)	10			
Talc	15 (min)	14.7 (max)	14.7 (max)	
Mica	15 (max)	4-6	4-6	
Red oxide etc	remainder	15.0 (max)	35.0 (max)	
Red lead		65.0 (min)		

The pigment content of the non volatile portion of the red lead primers is approximately 75% by mass whereas the zinc chromate primer is specified at a PVC of 35-40%.



A: ZINC PHOSPHATE CONTROL (B12) B: ZINC CHROMATE CONTROL (B11) C: ZINC CHROMATE, SABS 769, TYPE 1 D: RED LEAD SABS 312, TYPE II E: RED LEAD SABS 312, TYPE 2, GRADE I F: RED LEAD SABS 312, TYPE 2, GRADE II G: AC PIGMENT IN ALKYD, 8.9% PVC (B7)

# Figure 7. Corrosion resistance of AC pigments primers and controls

compares favourably. The red lead systems, except for Type III did not perform very well in the tests but experience has shown that this is usually the case when red lead paints are exposed to accelerated testing. The mode of failure, (winkling around the scribe) is not the usual appearance of a natural corrosion failure of a red lead system and as such, should be regarded as suspect. The salt spray exposure time was 2000 hours and the results apply to the full system of primer and top coat.

# **Exterior exposure results**

As mentioned previously, at the time of compiling the

paper, the exposure panels are only a few months old. However, they do include a much wider range of commercial paints than was used in the accelerated tests.

Summarising the results in the case of the epoxies, no failure is yet evident on any of the overcoated panels. The primer only panels show, in many of the commercial primers, considerable rust spotting. On the wire brushed surfaces, the AC pigment is among the best at this stage and on the SA3 surface, the AC pigment is second only to the commercial zinc phosphate primer which performed so well in the salt spray testing. The performance peak exhibited by the AC pigment at the 10% level at this stage is confirmed by natural exposure.

The alkyds, without topcoat, show the two linseed oil red leads to be performing best at this stage, followed by the AC pigment and finally the SABS zinc chromate. This confirms the accelerated testing. However, alkyd primers are seldom exposed for long periods without the full system, and this is probably not a serious drawback. No defects are yet visible on the full system of primer and top coat.

# Comparison of primer costs with different pigments

The cost of a coating is best compared by calculating the volatile free raw material cost by volume. This effectively compares the cost of the dried film of the product. The following costs per litre, were calculated using current local raw material prices and an estimated cost for AC pigment of £2.25. Table 5 lists these costs in sterling which, due to local variations in raw materials and exchange rate fluctuations should be regarded as comparative rather than absolute.

Table 5. Comparative costs of primers

Epoxy zinc chromate control	£2.60
Epoxy zinc phosphate control	£2.69
Epoxy AC pigment	£2.10
Alkyd zinc chromate control	£2.15
Alkyd zinc chromate, SABS 769	£2.03
Alkyd zinc phosphate control	£1.65
Red lead SABS 312 Type 2, Grade I	£1.70
Red lead SABS 312 Type 2, Grade II	£1.51
Red lead SABS Type 3	£1.88
Alkyd AC pigment	£1.02

#### Theory for the inhibiting effect of AC pigment

No paper, on a new material is complete without some explanation of how the materials works. With some trepidation, the following theory is advanced. At this stage it should be regarded as very tentative.

It has been postulated<sup>10</sup> that rust formation beneath a paint film is a cyclic process as shown in Figure 8. It is reasonable to assume that if the cycle were broken, then the corrosion process would be retarted. Reference to the infra-red trace in Figure 1 shows the presence of hydroxyl groups. These groups are complexed with iron in the AC pigment manufacturing process and under neutral

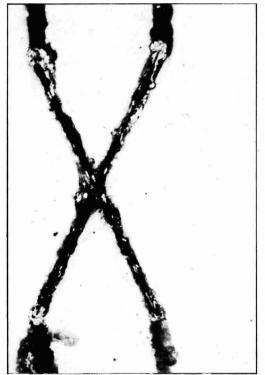


Plate 1: 2000 hours salt spray. Formulation B7 (8.9% AC pigment). Wire brushed surface.



Plate 3: 2000 hours salt spray. Formulation B12 (zinc phosphate), Wire brushed surface.

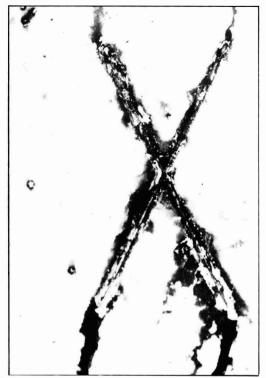


Plate 2: As for plate 1 except that surface blast cleaned to SA3.

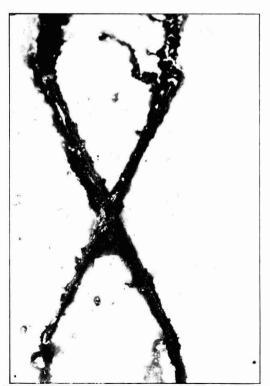


Plate 4: As for 3 except that surface blast cleaned to SA3.



Plate 5: As for plate 1 except that a high level of AC pigment  $(B10\!=\!31\%)$  has been used.



Plate 7: 2000 hours salt spray. SABS zinc chromate on SA3 surface.



Plate 6: As for plate 1 except that the formulation contains red oxide. (B1)



Plate 8: 2000 hours salt spray. SABS red lead, Type 2, grade I on SA3 surface.

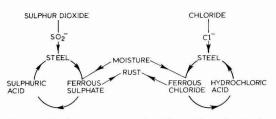


Figure 8. Cyclic process of accelerated rust formation in the presence of moisture and atmospheric pollutants

conditions, a stable complex is formed. This is AC pigment. However, if the pigment encounters acid conditions as would happen in the cycle shown in Figure 8, then by le Chateliers principle, the reaction could shift, and more ferrous ions could be complexed. The ferrous ions formed in the corrosion cycle would be available to be complexed and would be insolublised, and so the cycle would be broken.

It is possible that this is also the reaction of tannin based, so called rust converters. It is doubtful that much reaction occurs with stable iron compounds such as  $Fe_2O_3$ , but possible that some reaction occurs with soluble ferrous ions. Unfortunately, because such products are water soluble, if left beneath a paint film, they cause blistering in wet conditions. The result is, most manufacturers recommend the removal of the product by washing off before painting, so there can be no residual effect.

In the case of a pigment which is virtually insoluble and an integral part of the primer, these disadvantages fall away. Essentially the pigment remains inert until corrosion products begin to form, and only then does it begin to work i.e. when needed.

#### **Toxicological properties**

The scanning electron microscope analysis shown in Figure 3 shows that no heavy metals are present in the pigment. It is well known that these are the usual cause of toxicity in pigments. Pigments containing lead and chrome for example are extremely dangerous to health. Chemically, the organic portion of the molecule is known to be safe. However, a series of toxicological tests on rats at the subacute level (1g per Kg of body weight per day) is at present well advanced and to date no ill effects have been observed. In the interim, it appears that the pigment is relatively safe, especially when compared with most of the anti-corrosive pigments in common use.

# Conclusions

The new pigment is a fascinating product which is likely to expand the range of paint raw materials available to the formulator.

The three questions, which are asked in the introduction, can be answered as follows:

If the promise shown by accelerated corrosion testing is confirmed by outdoor exposure results, then the new material is a useful anti-corrosive pigment.

Reference to Table 5 shows that primers based on the new pigment should be competitively priced based on an estimated production cost of  $\pounds 2.25$  per Kg.

All indications to date are that the pigment is completely safe to handle and use.

# **Acknowledgements**

The evaluation of the pigment which led to this paper was commissioned by NCS Plastics (Pty) Ltd, P.O. Box 392, Pinetown, 3600, South Africa, who developed the product. The author wishes to thank them both for their encouragement and for their permission to publish the results.

[Received 8 March 1985

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# next month' issue

The Honorary Editor has accepted the following papers for publication in the November issue:

Internal stress and film formation in emulsion paints by D. Y. Perera The adhesion of chlorinated rubber to mild steel using a blister technique by A. Elbasir, J. D. Scantlebury and L. M. Callow Infrared studies of surface species on insoluble inorganic oxide particles immersed in liquids by C. H. Rochester

# **Colouration of radiation curing systems**

# V. Kaden and P. Eckert

Ciba-Geigy A. G., Basel, Switzerland

# 1. Introduction

When colouring radiation-curing binders the user very often encounters a reaction which, in its most varied aspects, differs from that found using conventional systems. Often the blame for this is directly attributed to the colorants used, without taking into account the complexity of the whole system with its many-sided, internal interactions. The remarks which follow are therefore intended to emphasize such interrelations from the point of view of the pigment user. This paper aims further to demonstrate the possibilities which currently exist with regard to UV-curing colouring systems, to point out peculiarities and, if possible, to explain these, to facilitate the selection of various system partners and also to encourage further work in this field.

The examples selected are based on practical tests and concern, primarily, the broad field of the application of printing inks and transparent coatings on various substrates. The printing process in particular, itself a complex process which is dependent on many parameters, supports this way of looking at such multi-component systems. Thus, according to the rules governing colour mixing, three primary colours ought to be sufficient to cover the whole colour spectrum. Why, then, are there so many yellow, rubine and blue pigments in the pattern cards of pigment producers?

The answer to this question shows clearly the problem: it is not just the colour, the impression of colour on the senses, which the system gives through the addition of colorants — it is rather the need to satisfy the numerous other requirements of an application at the same time. Suffice it to mention only dispersibility, economy, flow behaviour, stability, fastness properties etc., etc.

If we consider, for a moment, the gravure printing process. Here we have an example of a complex system which in spite of initial difficulties in its development and the fairly stringent requirements put upon the binder, solvents, flow properties, etc., now occupies an extremely important place in the printing industry. Similarly in the case of radiation-curing systems, which is a much more recent application than that of gravure printing. Although UV-curing has now been in use industrially for about 15 years<sup>1</sup> and has in recent years received a considerable boost<sup>2,3</sup>, one still comes across lists of disadvantages in the literature which can put off many a potential user. Here too, such 'disadvantages' should be seen as opportunities, in a similar way as was the case with other processes. The search for possible solutions or the best conditions is considerably eased, especially in the field of UV-curing, if in so doing more account is taken of the totality of the interactions than the individual factors.

# 2. UV-Curing

Before going on to discuss colorants and other additives

and their effect on the actual system, the basic characteristics of this relatively new technology are listed briefly, in spite of the fact that it has been described many. times in the literature.

The main difference is in the film formation mechanism. In the case of conventional inks or paints, where preformed macromolecules are already present the solvent tends not to be present in the final film whereas for UV films the "solvent" which involves mainly oligomers takes part in the cross-linking process.

#### 2.1 The Polymerisation Mechanism

In the case of prepolymers, oligomers and monomers listed for UV-curing olefinic unsaturated compounds are involved. These are most often acrylates where they have been specially modified chemically in each case according to their intended use (see section 2.3). All these materials are capable of radical polymerisation whereby the reactive radicals in the case of UV-curing are initially formed with the assistance of so-called initiators. The individual steps of this reaction can be outlined as follows:

#### 1. Initiation

 Breakdown of the initiator into radicals (following light absorption and transition to an excited state).

$$J-J \longrightarrow 2J^*$$

b. Addition to compounds capable of polymerisation.

$$J^* + M \longrightarrow J - M^*$$

2. Propagation

Addition of further molecules.

$$J-M^* + nM \longrightarrow J-(M)^*$$

- 3. Termination
- a. Recombination

$$2J-(M)^*_{n+1} \longrightarrow J-(M) \xrightarrow{2n+2} J$$

b. Transfer reaction

$$J-(M)^*_{n+1} + HX \longrightarrow J-(M) \xrightarrow[n+1]{} H + X^F$$

- x\* Can trigger off an independent reaction or when resonance stabilised bring polymerisation to an end.
- c. Addition of  $0_2$ :

$$J-(M)^* + O_2 \longrightarrow J-(M)-O-O^*$$
 stable.

# Table 1.

# Brief description of various coatings

	Conventional Printing Inks/Paints	UV-Curing Coatings
Binders	Polymers, drying oils, chemical reactive components	Reactive prepolymers
Solvents/ Dilutents	Organic solvents, Hydrocarbons with a high boiling point	Reactive monomers Reactive oligomers
Additives	Colorants, Dryers, Auxiliary Materials, Waxes etc., etc.	Colorants, Photoinitiators, Stabilisers, Waxes etc., etc
Film Formation Mechanism	Chemical or physical	Radical polymerisation

### 2.2 Photoinitiators

The selection of suitable photoinitiators is dependent upon various factors<sup>9</sup> such as:

- Spectral absorption,
- Effectiveness, tendency to break down into radicals,
- Stability of the radicals,
- Compatibility with and solubility in the reaction medium,
- Inherent colour,
- Darkroom stability.

It also depends on the particular intended use of the system. The following two diagrams illustrate the way in which the nature of the initiators and their concentration influence the technical properties when in use.

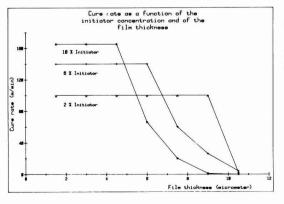


Figure 1. Cure rate as a function of the initiator concentration and the film thickness. (Source: G. Hencken, *Farbe und Lack*, 10, 1975, 922)

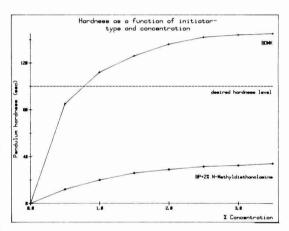


Figure 2. Hardness as a function of initiator type and concentration. (Source: Journal Rad. Curing, 7, 1983, 17)

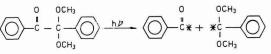
From the large number of photoinitiators available today some typical types are (for example):

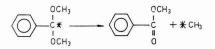
Benzoine ether	(IPBE)
Benzophenone	(BP/MK)
Thioxanthone	(ATX)
Ketale	(BDMK, <sup>(R)</sup> IRGACURE 651)
Acetophenone	

Mechanism of radical formation

a) Homolytic Breakdown

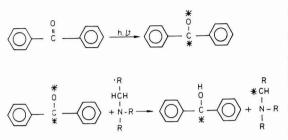
e.g. BDMK, (R)IRGACURE 651





# b) Synergy with H Donors

e.g. Benzophenone and Amine (Michlers Ketone); high reactivity and high absorption and therefore especially suitable for thin films.



# 2.3 UV-Binders

As has been mentioned, there are many acrylic-based examples included in those compounds which are capable of polymerisation. At the same time they represent the most widely varying types<sup>11-14</sup>; in various modified forms as monomers, oligomers and prepolymers.

Polyolacrylates Polyesteracrylates Urethaneacrylates Alkydacrylates Polyetheracrylates etc.

This great variety allows the user to formulate quite specificially and according to his own particular needs. As well as paying attention to the basic chemical structure, one must always, above all, pay attention to the properties of the prepolymers which form the main constituent of the binder and consequently influence the final properties quite considerably. The admixing of suitable monomers and/or oligomers likewise adversely affects the final properties. However, in many instances it helps in controlling its behaviour during processing (viscosity, rheology, stability, solubility, dispersibility, reaction rate, economy<sup>15</sup>).

# 2.4 Colorants

Colorants, i.e. pigments and dyes, can, due to their chemical structure or due to the presence of impurities such as salts and surfactants, influence the polymerisation process in different ways. In the case of reduced storage stability (see below) they have the effect of starting off the reaction. On the other hand, if there are pigments present which bear quinoid groups, nitro or amine substituents, a radical polymerisation can be partially or temporarily inhibited.

The part played in the reaction represented by the absorption of the dyes in the UV region in which the initiator is effective is also of significance.

The dye absorbs a certain amount of the irradiated energy which reduces the light absorption of the

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photoinitiator and hence the radical concentration. The consequence of this is a slowing down of the cure rate for the coloured system. This influence can be countered through careful selection of the initiators and the pigments with regard to their UV absorption, but also by the use of the appropriate radiation sources.

In addition, it is worth mentioning specific pigment/system combinations which, after a short time, lead to deeper shades and a modified reactivity. Pigment Orange 34 can be quoted as an example of this. In combination with acrylate prepolymers and certain amines, which often act together in the initiator system, this pigment exhibits a marked change in shade. It is still unclear as to whether in a similar way to the amine conditioning of ethyl acetoacetate yellow pigments the keto group in the colour molecule changes partly with the amine to a red-coloured Schiff's base.

# 2.5 Summary

- The concentration of the initiator radicals is proportional to the effective amount of irradiated UV light and determines the initiation rate.
- The concentration as well as the reactivity of the initiator radicals influences the average molecular weight of the subsequent polymers and consequently their technical properties when in use (brittleness, shrinkage, adhesion etc).
- The polymerisation process can be inhibited, retarded or terminated by means of oxygen.
- The reaction rate of the unsaturated compound is dependent upon the number of C-C double bonds per molecule, on the polarity of this double bond as well as the spatial configuration of the double bond<sup>17</sup> (steric hindrance, viscosity).

#### 3. The use of organic pigments in powder form

# **3.1 Dispersion**

It has already been pointed out in earlier papers<sup>18</sup> that there are peculiarities which can have a decisive influence in the selection of the pigment for use in UV-curing acrylate systems. It was noticeable that with regard to colour strength and colour strength development that some pigments which in conventional solvent systems or oil alkyd binders yield only moderate results, come off really well in radiation curable acrylics. Organic pigments, on the other hand, which are surface-treated and optimised so that they can be used specifically in conventional systems, often achieve not quite so good results in radiation-curing acrylate systems. The reason for this differing behaviour is shown by the connections between the physico-chemical characteristics of the pigments and the binders, as the following examples make clear (where the influence of the binder and the pigment are examined).

### 3.1.1. Characteristics of binders used

A conventional oil alkyd resin (Binder 1) is compared with a polyfunctional prepolymer acrylic resin (Binder 2) and an acrylic modified soya alkyd resin (Binder 3), whose properties are between Binders 1 and 2. The binder characteristics are shown in the following table.

# Table 2.

# **Binder Characteristics**

Binde	r Chem. Type		Wetting Tension in contact with water (mN/m)
1.	oil alkyd quick set	14	43
2.	polyfunctional acrylic prepolymer	7	66
3.	acrylic-modified soya alkyd resin	60	54

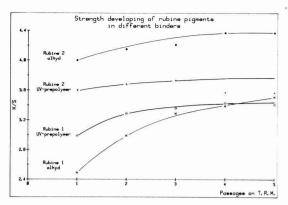


Figure 3. Colour strength development of Rubine Pigments 1 and 2 in various binders

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Pigment	Chem. Type	C.I.	Dispersiblity in oil inks	Resin required g.resin/100g pigment Resin type 1 2 3	Wetting tension in contact with water (mN/m)	Polarity of chem. type
Rubine 1	Ca-4B Toner	P.R. 57:1	normal	86/61/71	49	high
Rubine 2	Ca-4B Toner	P.R. 57:1	easy	74/76/80	31	high
Blue 1	Phthalocyanine	Р.В. 15:3	normal	69/64/65	56	low
Blue 2	Phthalocyanine	P.B. 15.3	easy	63/60/70	30	low

Pigment	characteristics <sup>19-21</sup>

# 3.1.2 Characteristics of pigments used

Each time a Rubine and a Phthalocyanine Blue Pigment which were rated as being so-called "normally dispersible" from the <sup>(R)</sup>IRGALITE series were compared with their so-called "easily dispersible" versions, from the <sup>(R)</sup>IRGALITE L series. They are characterised as shown in Table 3.

# 3.1.3. Measurements

The pigments were dispersed in five passages on a triple roller mill (Buhler, SDH 200) at 25 per cent pigmentation. After each passage samples were taken and they were mixed with white pastes of similar lightening power based on the appropriate binders. The K/S values were taken as the standard for the colour strength according to DIN 53234.

# 3.1.4 Results

The comparision between the Rubine Pigments in the two binder systems is shown in Figure 3, where the extremely good dispersion behaviour of the 'easily dispersible' Rubine 2 in the oil alkyd system is clearly seen. It has been specially developed for this purpose.

Rubine 1 not only produces a lower colour strength in the oil alkyd system but also develops it more slowly than Rubine 2. On the other hand the two pigments approximate to each other in their ease of dispersion in the case of the acrylic resin: Rubine 1 now becomes equally easily dispersible; the colour strength level of Rubine 2 falls compared to the alkyd system.

Figure 4, shows the differences for all three binders, taking Blue 1 as an example. High colour strength with the UV acrylic resin, one which is clearly lower in the oil alkyd system and a colour strength for the acrylic modified oil alkyd resin which lies between these two values. Differing ratios are found in the case of Blue 2.

It is easily dispersible in the oil alkyd showing a good colour strength, and shows a similar behaviour in the UV acrylic resin.

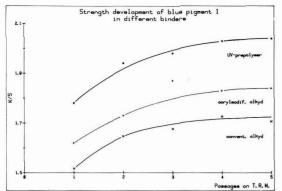


Figure 4. Colour strength development of Blue 1 in three different binders

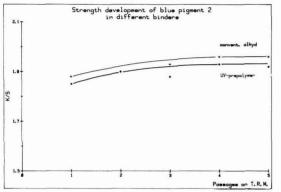


Figure 5. Colour strength development of Blue 2 in binders 1 and 2

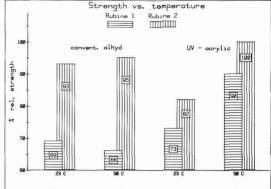


Figure 6. Colour strength for Rubine Pigments 1 and 2 as a function of the dispersion temperature

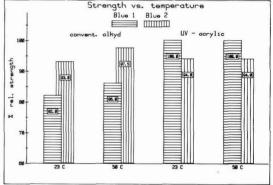


Figure 7. Colour strength of Blue Pigments 1 and 2 as a function of the dispersion temperature

#### 3.1.5 Influence of the dispersion temperature

The results for two dispersion temperatures, 23°C and 50°C are shown in Figures 6 and 7 which follow. In this connection the highest K/S value obtained for each pigment pair is taken as 100 per cent irrespective of the binder type. As can be seen the dispersion temperature influences the result in differnt ways.

Rubine 1, a Ca-4B toner which is a relatively polar pigment, is colouristically stronger in the more polar acrylic resin due to better wetting at a higher temperature than at room temperature. The fact that it has a lower level in alkyd resin anyway and that it falls further at higher temperatures could be explained by a lower degree of interaction, polar pigment — slightly polar resin.

By contrast the already high colour strength for Rubine 2, which has been specially developed for oil inks, can even be increased by a rise in temperature. In the case of the acrylic resin this increase in colour strength is even more pronounced. Rubine 2 behaves like an untreated toner at higher temperatures. This leads to the assumption that

there is a loosening of its original rosin and perhaps even an exchange of the resins at the pigment surface. The results for the blue pigments are given in Figure 7.

Both pigments, because they are less polar that 4B toners, increase their colour strength with an increase in temperature only in the oil alkyd binder. Blue 1, as a non-resinated pigment, does not reach the high colour strength of the resinated Blue 2 in this type of binder, but exceeds these for both temperatures in the acrylic resin. However, the fact that at higher temperatures Blue 1 does not show additional colour strength indicates that the pigment is obviously already well wetted at room temperature in this acrylic system.

On the other hand Blue 2 demonstrates the good alkyd compatibility of its resination as there is no increase in colour strength in going either from the conventional alkyd to the UV system or by increasing the temperature of the acrylic.

# 3.1.6 Summary: Colour strength — Wetting tension

If one compares the relative colour strengths of the

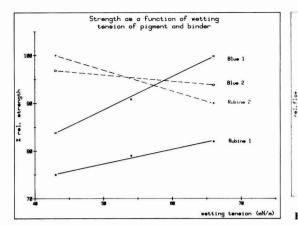


Figure 8. Colour strength as a function of the wetting tension of pigment and binder

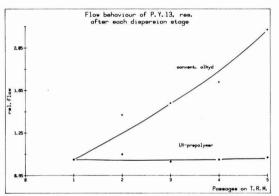


Figure 9. Relative flow of resinated PY.13 after each dispersion stage

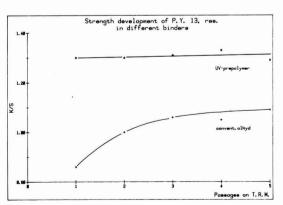


Figure 10. Colour strength development of resinated PY.13 in binders 1 and 2

individual pigments with the wetting tension of the binders tested, then the following picture emerges. (See Figure 8).

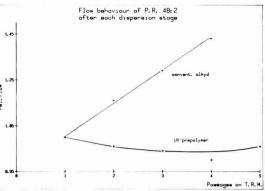


Figure 11. Relative flow of PR. 48:2 after each dispersion stage

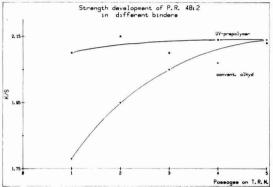


Figure 12. Colour strength development of PR. 48:2 in binders 1 and 2

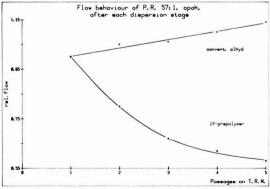


Figure 13. Relative flow of opaque PR. 57:1 after each dispersion stage

Pigments Rubine 1 and Blue 1, which have a higher wetting tension against water (see Table 3), than their resinated versions, develop higher colour strengths with increased wetting tension of the binders, while, conversely,

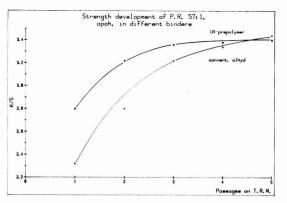


Figure 14. Colour strength development of opaque PR. 57:1 in binders 1 and 2

both pigment 2's produce their best results in the binder with the lowest wetting tension.

# 3.1.7 Viscosity Changes During Dispersion

Three pigments are taken as examples, of which only the Rubine belongs to the group which was discussed earlier. Figures 9 to 14 show the viscosity changes during dispersion as well as the related colour strength development curves for two binders (oil alkyd and acrylic resin). The details with regard to viscosity are relative, whereby the measured falling time for dispersion stage one (a falling bar viscometer was used) was set as being equal to 1.

The different trend for both types of binder is obvious here.

The conventional alkyd resin shows the generally wellknown picture, whereby increasing viscosity is linked to increasing colour strength, i.e. increasing particle fineness<sup>22</sup>. The picture for acrylic resin is clearly different from this where, in spite of an increase in colour strength, there is no increase in viscosity noted and with regard to pigments P.R. 48:2 and P.R. 57:1 there is even a noticeable decrease in viscosity. A plausible explanation for this behaviour is still to be found. Acrylates would appear better wetters and more firmly adsorbed at the pigment surface that alkyd resins. Consequently, they permit a higher pigment concentration during dispersion and retain their capacity for co-polymerisation with other acrylic resins<sup>23</sup> used with the final formulation is subsequently adjusted.

# 3.2 The storage stability of coloured systems

# 3.2.1 General

Any use of UV-reactive systems, in order to be economically justifiable, today requires a minimum of six months storage stability at room temperature. In general, the manufacturers of binders fulfil this requirement by the use of appropriate stabilising additives. It is, however, wellknown that some colorants have a negative effect on the storage capability. This becomes noticeable as a gradual gelling process which starts at the bottom of the container. In this connection other mechanisms play a part not only those initiated by radicals<sup>24</sup>, with the result that, apart from the pigments, other substances which are also present can initiate the reaction.

Often it only requires impurities from the synthesis, residues from the raw materials and intermediate products, or the presence of salts or dissociating substances. The causes of this have in no way been explained. Since colorants must be considered bulk manufactured products and hence not analytically pure, the best the user has to go on is the practical test which has been matched to his sytems. The pigment manufacturer himself must, on the other hand, carry out the most widely ranging tests possible, if he wants to be successful in offering his products for sale. Therefore, the statements which follow with regard to colorants are based on experience involving several typical binder systems, unless otherwise stated.

The binders tested were of the following types:

- Polyurethane acrylate
- Prepolymer polyolacrylate
- Epoxidacrylate
- Soya epoxidacrylate

# 3.2.2 The Storage Test

The material to be tested is stored sealed at room temperature and/or at 45°C in the absence of light. In order to exclude false results due to the introduction of oxygen from the atmosphere, it is necessary to carry out the testing while totally excluding oxygen, and best of all in a nitrogen atmosphere.

By carrying out the storage at a higher temperature the test times are substantially reduced. As many experiments have shown the minimum shelf life demanded by industry is sufficiently assured, insofar as the coloured binders remain stable for 30 days at 45°C.

# 3.2.3 Practical tests and results

# 3.2.3.1 Influence of the pigments

From the large number of <sup>(R)</sup>IRGALITE pigments tested some representative examples have been taken and are shown in the following synopsis (Table 4). This makes clear the influence they have on storage stability. They were triple roll milled at 15 per cent pigment with 5 per cent photoinitiator. (<sup>(R)</sup>IRGACURE 651).

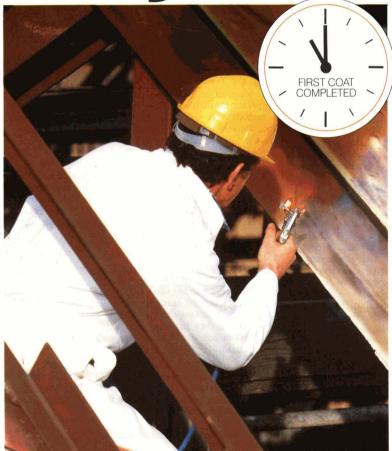
For phthalocyanine blue pigments all the versions tested were storage stable. Yellow pigments could show an influence of the resination and for the red pigments the cation could influence the shelf life.

# 3.2.3.2 Influence of the photo-initiators

Photo-initiators, which are added to UV-curing systems as radical formers, can, due to their reactivity and concentration, have a considerably adverse effect on the storage stability, from time to time, as the summary shown in Table 5, which includes some examples, shows.

It is well-known that storage stability times can be improved by the use of additives, so-called inhibitors or

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The tried and tested method of coating steel with industrial paint is, let's face it, very practical.

But not very quick.

However, when the paint contains new waterbased 'Haloflex', drying time can be measured in minutes instead of hours.

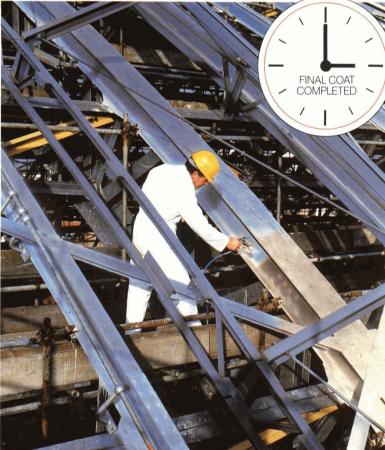
Paint systems, based on 'Haloflex', a resin supplied by ICI Mond Division, dry in under an hour at room temperature. So on the average job, you can start applying the second coat as soon as you have finished the first.

What's more, for big jobs, scaffolding only has to be erected once for each section, instead of having to be laboriously repositioned and then re-erected for additional coats.

'Haloflex' has been widely used in Europe and

# Haloflex, pleted a 3 day than a day.





America, and has proved itself time and time again to give a resistance to the environment comparable to that of other industrial paint systems, but without any toxic drawbacks.

For more information send your card to us at the address below. We'll have details to you faster than the time it takes for a coat of conventional paint to dry.



ICI MOND DIVISION, DEPT P, PO BOX 13, THE HEATH, RUNCORN, CHESHIRE, WA7 4QF, ENGLAND.

# Table 4.

# Average storage stability at 45°C

Pigment C.I.	Characteristic	Stability in days
Pigment Yellow 12	opaque un-resinated	XXXXXXXXXX * (31)
	transparent resinated	XXXXXXXXXX *(31)
Pigment Yellow 13	opaque un-resinated	XXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXX
	transparent resinated	XXXXXXX (21)
Pigment Red		
48:3 48:4,	Sr Salt Mn Salt	XXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXX
Pigment Red 57:1	opaque, slightly resinated	X (3)
57:1	transparent, resinated	XXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXX
Pigment Blue 15	all types	XXXXXXXXXX *(31)

\*Test stopped; inks are still unchanged.

# Table 5.

Influence on the average stability: a) As a function of the initiator concentration; b) As a function of the kind of initiator.

_	Pigment	Initiator	Conc.	Stability in days at 45°C
0	Pigment Yellow 13	BP + MK 1:1		XXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXX
a 	Pigment Red 48:2			XXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXX
	Pigment Yellow 13	BP + MK IRGACURE 651	3%	XXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXX
b	Pigment Red 222	BP + MK IRAGACURE 651		XXXXXX (20) XXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXX

(System: Soya epoxide acrylate and Polyolacrylat-Monomer (4:1), 15% Pigmentation, 3 Passes Triple Roll Mill, 23°C.) stabilisers. Nitro compounds, tertiary aromatic amines or quinoid structures are often used for this purpose<sup>25</sup> However, these by their nature adversely affect the subsequent reaction rate during hardening as well and, in this connection bring other unknowns into play with regard to the remaining properties. This is equally relevant with regard to the introduction of air during or after colouring<sup>26,27</sup>. It is, therefore, more advantageous to dispense with such additives by the careful selection and optimisation of the concentration of photoinitiators.

# 4. Use of pigment preparations

#### 4.1 General

In addition to powder pigments, solid pigment preparations have long been used in many solvent-based paint and printing ink systems<sup>27</sup>. The best known are the various types of chips, as well as solid preparations in powder or granular forms.

These are colorants consisting of an organic or inorganic pigment of extremely fine particle size which are dispersed and incorporated in a resinous carrier.

Which carrier resin is used necessarily depends on the requirements of the intended area of application. The primary prerequisites for the use of pigment preparations are:

- Solubility of the carrier resin in the application medium (paint, binder, solvent).
- Compatibility of the carrier resin with the binder of the application medium.

In addition, if the carrier resin is not identical with the application binder, it should not have a negative effect on the film properties.

Because of these requirements, a large number of the pigment preparations commercially available today (CAB and NC chips, preparations based on ethyl cellulose and vinyl copolymers) are not suitable for use in radiationcuring binders.

Limited application is only possible in exceptional cases, for example when used in combination with a certain amount of conventional volatile solvent such as ester or ketone.

#### 4.2 Acrylate-based pigment preparations

The development of aqueous acrylic resin binders, as used today particularly in gravure and flexographic printing inks, called for suitable pigment preparations<sup>28</sup>. These acrylate resin preparations have, in certain circumstances, also proved suitable for use in UV-curing acrylate systems.

The carrier resin of the acrylate resin preparations suitable for use in these systems — marketed under the trade name <sup>(R)</sup>MICROLITH-WA — demonstrates good compatibility and solubility in a number of monomers and prepolymers (Table 6).

By the appropriate choice of reactive solvents and

Acrylic resin-based pigment preparations — solubility of carrier resin

Solvent	Solubility
Water, pH 7	-
Water/ethanol, pH 8.5	+
Ethanol	+
Isopropanol	+
NVP	+
TMPTA	+
HDDA	+
DDA	_
TMPTA/DDA 1:1	+
HDDA/DDA 1:1	+

Table 7.

Formulation example for a UV-curing binder — coloured with a pigment preparation

Parts by Weight	Components
5	Pigment preparation
20	N-Vinyl-2-pyrrolidone
40	1,6-Hexanediol diacrylate
30	Urethaneacrylate
5	Photoinitiator

diluents from the monomers and oligomers currently available and by the appropriate choice of prepolymers, many formulations can be found in which pigment preparations of the type described can easily be processed into printing inks and other coatings.

N-vinyl-2-pyrrolidone (NVP) has proved to be a very effective solvent in this context and has the added advantage of having a low toxicity rating<sup>29</sup>. NVP combines excellent dissolving power with high reactivity and can be copolymerized with a large number of partners.

From the many formulations that are possible, Table 7 gives a representative recipe.

#### 5. Use of dyes

#### 5.1 General

One possible method for colouring radiation-curing binders is of course the use of dyes.

In contrast to pigment preparations, dyes present fundamentally fewer compatibility problems. Dyes can, however, have a greater effect on the stability and the curing of binders. It should thus be determined in each case whether the following criteria are fulfilled:

- Solubility in binder and/or in individual components,
- Stability of solutions and reactive systems which have been coloured,
- Influence on curing,
- Fulfilment of fastness requirements.

# 5.2 Solubility of Metal Complex Dyes

In view of the requirements described, the most suitable dyes are the metal complex types, such as for example <sup>(R)</sup>ORASOL dyes. Their solubility<sup>31</sup> in a large number of organic solvents is a good basis for their adequate solubility in monomers.

As with pigment preparations, so too with metal complex dyes the use of N-vinyl-2-pyrrolidone enables good results to be obtained. Solubility in other monomers and oligomers varies considerably and is shown by examples of three dyes which differ both chemically and in terms of shade (figures 15, 16, 17).

The appropriate choice of dyes or solvents and diluents often provides the answer to solubility problems.

# **5.3 Colouristic Peculiarities of Dyes**

From time to time, surprising changes in shade are noted when dyes are used. This manifests itself by the fact that either the expected shade is not obtained or a colour change is observed when materials which have been conventionally coloured or printed with dyes are then over-lacquered with a radiation-curing clear lacquer.

To date, however, no conclusive evidence has been found for a direct connection between these effects and radiation curing.

It is much more likely that this is the result of a binderspecific effect on the  $\pi$ -electron resonance system of the dyes. However, as can be shown, this does not apply specifically to dyes in radiation-curing systems but can also be found in conventional binder systems whose drying is purely physical (figures 18, 19, 20).

# **5.4 Resistance and Fastness**

Radiation-curing systems, however, do not only present new problems for the use of dyes, they also offer new opportunities.

The binder, the dye/binder ratio and the solids content of a conventional solvent system always affect to a greater or lesser extent the degree of fastness and resistance that can be achieved. The higher the binder content, the greater the resistance with dyes particularly to migration and to chemicals.

As a result of their high solids content radiation-curing binders would be expected to offer good chances for the

	Solubility of Solvent Yellow 88 (g/liter)
Solvent	
MASCHE	
BDMA	
ТМРТА	
DDA	
HDDA	
NVP	
Acetone	
Ethanol	
Ethylaostate	

Figure 15. Solubility of Solvent Yellow 88

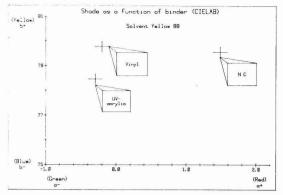
	Solubility of Solvent Orange 59 (g/liter)	
Solvent		
MASCHE		
BDMA		
ТИРТА		
DDA		
HDDA		
NVP		
Acetone		
Ethanol		
Ethylacetate		

Figure 16. Solubility of Solvent Orange 59

	Solubility of Solvent Brown 42 (g/liter)
Solvent	
MASCHE	
BDHA	
THPTA	
DDA	
HDDA	
NVP	
Acetone	
Ethanol	
Ethylacetate	

Figure 17. Solubility of Solvent Brown 42

incorporation of good protection of dyes thus providing good levels of fastness and resistance. This has been demonstrated with a number of dyes subjected to comparative testing in UV-curing acrylate printing inks and conventional polyamide printing inks (figures 21 and 22).





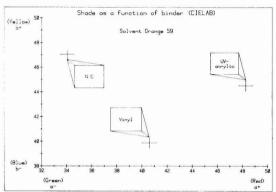


Figure 19. Shade of Solvent Orange 59 as a function of binder

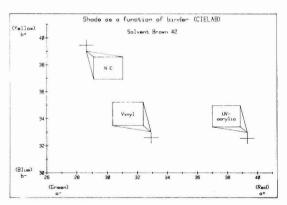


Figure 20. Shade of Solvent Brown 42 as a function of binder

Results for lightfastness have also been attained that are superior to those for nitrocellulose binders and that are, in certain cases, as good as those for vinyl chloride/vinyl acetate copolymers, which are well known for their high fastness ratings. (Table 8).

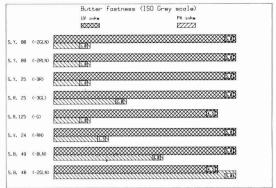


Figure 21. Butter fastness in a conventional ink system (Polyamide) and in a UV-cured ink

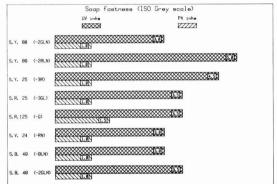


Figure 22. Soap fastness in a conventional ink system (Polyamide) and in a UV-cured ink

# Table 8.

Lightfastness of metal complex dyes in various binders

Dye (0.36 g/m <sup>2</sup> on polyester film)	Lightfastness (Blue Scale)	
(0.00 g m on polyester mm)	(Blue Seale)	
Solvent Yellow 88		
- Vinyl resin binder	7	
- Nitrocellulose binder	6	
- UV-acrylate binder	7	
Solvent Orange 59		
- Vinyl resin binder	5	
- Nitrocellulose binder	2	
- UV-acrylate binder	5	
Solvent Brown 42		
- Vinyl resin binder	6	
- Nitrocellulose binder	3	
- UV-acrylate binder	4	

# **Summary and Prognosis**

The pigmentation and colouration of radiation-curing binders is a technology that in many respects requires the development of new know-how.

The choice of colorant must meet the specific requirements of radiation-curing systems, which differ in various respectes from conventional binder systems.

The following points should be noted in particular:

- The differing wetting properties which lead to different dispersion behaviour of pigments,
- The influence on storage stability and/or hardening behaviour by the colorants,
- The solubility and compatibility which is inadequate in the case of the majority of commercially available pigment preparations,
- The solubility properties of dyes in monomers and oligomers which in most cases are still unknown.

In summary it can be said that radiation-curing systems still present a wide and substantially unresearched area of the application technology of pigments, pigment preparations and dyes.

This leaves considerable scope for creative research into possible applications and, as radiation curing continues to become more widespread, also paves the way for more intensive use and product development.

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# corrigendum

# Cationic electrodeposition primers over zinc coated steels. Part 1: Effect of voltage and substrates on coating quality by C. K. Schoff and H. J. Chen (JOCCA, 1985, 68 (8), 185).

On page 190, column 1, paragraph 2, the eighth line should read:

Hydrogen from water electrolysis and other processes that occur during deposition could be trapped in the coating and, possibly, in the zinc and/or zinc phosphates layers to produce a number of defects.

# Acrylic and polyurethane dispersions in industrial coatings for plastics

# **R. Arnoldus**

Polyvinyl Chemie Holland B.V., Sluisweg 10, P. O. Box 123, 5140 AC, Waalwijk, Holland

# Introduction

Conventional materials in use are being increasingly substituted by plastics that are no longer considered to be just alternatives for other materials but are preferred by engineers for their specific properties such as low specific weight, a wide variety of mechanical properties and also plastics are often more economic.

Most people think of plastics as a postwar phenomenon, but in fact plastics were discoverd over one hundred years ago. Since that period of time the growth of plastics' use has been dramatic and the role of plastics in modern society is getting increasingly important. When we look around us, we can find plastics everywhere, sometimes even very well camouflaged.

In the future it is to be expected that the use of plastics will become more common not only in furniture, domestic appliances, and packaging industries but predominantly in big potential applications such as automotive, business machine, and audio/video industries.

Nowadays in Western Europe approximately 15 million tons of plastics are used and it is expected that this will be 18-19 million tons in 1990. Table 1 gives a rough division of the plastics found in W-Europe.

# Table 1.

Plastics in W-Europe (1984)

Plastic	Total (%)	Automotive (%)
PE	37	
PVC	24	16
PP	13	23
PS	8	2
PU	7	24
ABS	3	11
PA	2	6
PMMA	1	3
PC	0.6	1
PETP/PBTP	0.6	1
PPO	0.3	2
UP		7
Rest	3.5	4

The so-called engineering plastics and composites especially show a strong growth and new plastics are "born" regularly. Reinforcement and polymer-blending, mixing and modifying are keywords for present developments in plastics. All this will undoubtedly mean that coatings for plastics will follow the same trend as the growth in the consumption of plastics dictates a growth in the consumption of coatings.

The automotive industry is a typical example where the penetration of plastics has been very drastic. An average of 100-150kgs of plastics material is used in our European cars today and it could be expected that this figure would double in the 90's, the main reasons being further aim at weight reduction, assembly rationalization, improved corrosion resistance, compliance with safety regulations and styling or design. As in many cases plastics have to be coated, the coatings industry will have to find answers to the need for specific coatings onto specific plastics for specific applications. This means that the coating industry (and we as their suppliers) must be able to respond in a flexible manner in order to have the right coating system available at the right time.

# **Plastics need coatings**

From a decorative standpoint plastics are coated for the following purposes:

- To allow for a wide variety of colours (even in exact shades).
- To provide effects like metallics, woodgrain etc.
- To change degree of gloss.
- To cover surface defects (moulding imperfections, pores etc.).
- Lower dirt pick up.

More important often are the functional reasons for coating plastics such as:

- Protection against aggressive chemicals and solvents.
- Weather protection (yellowing, loss of mechanical strength).
- Mechanical protection (abrasion, marring).
- Making plastics conductive, antistatic or flame retardent.
- Protection against plasticizer migration.

In many cases a combination of these points will make people decide to paint a plastic part. Stylists and designers also have an enormous impact on such a decision, e.g. in automotive appearance the trend is "fully body coloured" vehicles, or at least matching coloured parts instead of contrast colours.

#### **Problems when painting plastics**

Paint systems for plastics have to be suitable for use on those particular plastics, otherwise problems and damages can occur.

- It is necessary to know the exact type of plastic and its function as even within the same basic group plastics can be very different from each other.
- Plastics are in many cases more or less solvent sensitive which can lead to surface etching or swelling.
- Adhesion on plastics is sometimes very difficult to achieve. In all cases thorough cleaning and/or pretreatment is necessary.
- Heat resistance of plastics is variable and mostly limited. This means that mostly paints systems have to be used that develop properties at forced drying or ambient temperature (physical drying and two-pack systems).
- Mechanical performance will be changed by a coating. Impact resistance in a plastic substrate is usually lessened when coated with a solvent type material. This reduction in impact strength is most probably related to the mouldstressed areas of the part being attacked and consequently revealed by the solvents. The solvents of the paint formulations therefore have to be "mild" to minimize the resultant impact reduction.
- Furthermore the flexibility of the coating system needs to be high enough to prevent paint cracking and subsequent propagation in the plastic. This is specially important where plastic is used specifically for its impact strength, such as protective headgear and bumpers of polycarbonate.
- Owing to destruction of cellular structure by solvents from the paint gassing of foams can cause blistering and other surface defects.

It is clear that plastic coating systems have to be carefully developed and formulated to specific requirements, and only good cooperation between plastic producer, paint producer and enduser can help to prevent that problems arise.

# Water-borne coatings for plastics

Nowadays there are many different types of coatings that can be and are used on plastics. Physical drying thermoplastic acrylic based coatings and two-pack polyurethane systems are extremely popular for applications such as business machines, hi-fi and tv equipment, automotive interior and exterior parts etc.

The use of these type of coatings seems somewhat clouded due to increasing governmental environmental legislations, concern over toxicity and working conditions and the advances in the development of water-based acrylic and urethanes.

The growth of water-based coatings for plastics in the USA is estimated at about 17 per cent annually and the same trend is starting to show in Europe.

Water-based dispersions are particularly suitable as raw

# Table 2. Adhesion of water-based dispersions to various plastics

Plastic	Surface	Acrylic	Acrylic	Acrylic	Urethane
	Tension in mN/m	ĩ	2	3	1
PVC	39	0	0	0	0
PS	33	0	0	5	5
ABS	36	0	0	0	0
PP	30	5	5	5	5
PPO	34	0	3	5	5
PPO-foam	36	0	0	0	0
PUR compact	32	0	3	5	3
PC mod.	34	0	0	5	0
PC	34	0	0	0	0
RIM	38	1	4	5	5
PA	38	0	1	5	5

0 = excellent

5 = bad

# Table 3

# Critical surface tension values for various materials

Substrate	Critical surface tension Dynes/cm		
Iron	45		
Aluminium	45		
Nylon 6.6	43		
Polysulfone	40		
Cellulose Acetate	39		
PVC	39		
PMMA	39		
ABS	34-38		
Polystyrene	33		
Polyethylene	31		
Polyurethane	29		
Silicone Rubber	22		
Polytetrafluoroethylene	18		

materials for plastic coatings as they dry relatively fast and do not require high temperatures to develop properties.

Advantageous for water-based coatings is their ability to estimate or greatly reduce some of the problems associated with solvent-borne coatings:

- No solvent attack to plastic (less stresscracking).
- Less degradation of mechanical properties.

On the other hand, water-borne coatings are not trouble free either as:

• Mostly forced drying conditions (50-90°C) are required

# Table 4.

# Table 5.

# Critical surface tension of a substrate coated with various release agents

# Surface tension of various solvents

Release agent	Critical surface tension	Solvents	Surface tension Dynes/cr	
-	Dynes/cm		-	
		Alcohols	21-24	
		Glycols	35-47	
Polymethylsiloxane	24	Glycol ethers	28-35	
Fatty acid monolayer	24	Esters	23-32	
Polytetrafluoroethylene	18	Ketones	23-31	
Flurocarbon-acid		Aromatic hydrocarbons	28-29	
mono-layer	15	Aliphatic hydrocarbons	18-23	
Perfluorolauric acid		Water	72	
mono-layer	6		4.00 <sup></sup>	

# Table 6.

Surface tension of various glycol ethers compared to their surface tension mixed with water

Glycol ether	Surface tension Dynes/cm at 25°C	Surface tension of 20% solution in water at 25°C, Dynes/cm	
Cellosolve	29.4	47.1	
Butyl cellosolve	28.6	28.9	
Carbitol	35.2	49.6	
<b>Butyl Carbitol</b>	31.0	33.2	
Hexyl cellosolve	30.3	28.5A	
Propasol M	28.3	46.8	
Propasol DM	29.9	43.3	
Propasol P	27.0	30.4	
Propasol B	27.4	32.4B	

for proper water evaporation and resistance development.

- Adhesion cannot be adjusted in the same way by solvents.
- There exist some restrictions in appearance.

With newly developed acrylic and urethane dispersions however, many problems like wetting, adhesion, mechanical and chemical properties could be or will be solved. We will discuss some essential properties more detailed.

### Adhesion of water-borne coatings on plastics

Plastic surfaces can be classified with respect to surface energy as "low energetic", which seems to point to the fact that surface wetting by a coating is not easy, especially not when this is a water-borne coating. Many people feel that

1985(10)

solvent-borne coatings have adhesion to plastics because the solvents used in the coatings "bite" into the plastic. Inherently it is believed that water-borne coatings have poor adhesion to plastics due to lack of large amounts of solvents.

Although in a few cases, the addition of aggressive solvents to a coating can enhance its adhesion to plastics, nowadays water-borne coatings are available which have the same excellent adhesion.

Table 2 gives an example where three different acrylic/styrene copolymers and one urethane dispersion have been tested on various plastic substrates.

An important factor to consider when designing a waterborne coating for plastic is *wetting*. A majority of the adhesion failures of coatings on plastics is due to the inability of the coating to sufficiently wet the substrate. It is therefore useful to take the critical surface tension necessary to wet a plastic into account. The lower the critical surface tension for a substrate, the more difficult it becomes to wet and gain adhesion. Table 3 shows typical examples of surface tensions of plastics and we know how hard it is to adhere to some of them.

In many cases plastics are more difficult to adhere to than other substrates owing to surface contaminants such as processing aids, lubricants, or moldreleases that drastically change the surface tension of a substrate (Table 4).

Solvents play an important role on the surface tension of a coating and a wide range of surface tensions is available (Table 5).

For water-based coatings the glycol ethers are the most commonly used. When glycol ethers are mixed with water, the surface tension of the combined system can be quite different.

Table 6 compares the surface tensions of various glycol ethers versus a 20 per cent solution of each in water. It is interesting to note that a few of the glycol ethers do not change appreciably when water is mixed with them.

Predicting the adhesion of water-based coatings by measuring surface tensions of both substrate and coating or even by measuring the contact angle of a coating droplet on the substrate was found to be inaccurate and not always correlating with practical experience. It is more probable that the key to adhesion is found in the composition of the dispersion itself. And as the composition of a dispersion is exclusively known to its producer, the formulator will have no other leads than adhesion information from the producer and the results of his own practical investigations.

A great deal of research time has been spent by the authors investigating the phenomenon of wetting and adhesion; and products are now available which have the ability to wet and adhere to a wide variety of plastics.

Even water-based dispersions with the ability to adhere to polypropylene will become reality in the coming years and with that very universal coating systems.

The adhesion of water-borne coatings on plastics can in some cases be upgraded through the use of certain additives. One of them is our polyfunctional aziridene crosslinker that functions by reacting with both functional groups in the resin and functional groups in the plastic. In many cases however adhesion is decreased by the crosslinker addition, especially when functional groups in the polymer are responsible for adhesion to the plastic.

Adhesion of water-based coatings on plastics is also influenced by the used drying temperature. Under forced drying conditions adhesion development is quicker and often better than at ambient temperatures. Although adhesion is a basic property that a coating must have, other properties such as hardness, water resistance, chemical resistance etc. are needed to meet various end use demands.

# Hardness

Hardness is a composite property combining concepts of resistance to penetration, deformation, scratching and

marring. Water-borne acrylic emulsions can be "tailormade" to give almost any hardness by varying the Tg, the molecular weight and by using special polymerization techniques. Water-borne urethanes can also be made very hard or very soft.

In many cases, the hardness of a water-based coating is greatly affected by:

- The amount and type of pigments.
- The amount and type of coalescing solvents.
- The amount and type of plasticizers.

Coalescing agents and plasticizers therefore should be carefully evaluated for their effects on hardness.

The use of polyurethane dispersions in combination with hard acrylic emulsions has opened a new way of formulating with minimum amounts of coalescing agents or plasticizers.

# Water resistance

In the past few years the water resistance of water-based emulsions has been greatly improved due to advance in polymerization techniques, new monomers and surfactants. It is safe to say that there are water-borne resins available now which will meet or exceed the water resistance specifications of coatings on plastics for many end uses.

Various formulation ingredients however can have a profound affect on the water resistance of a coating and again careful selection is needed.

# Chemical and solvent resistances

One of the most significant features of acrylic emulsions and urethane dispersions is their ability to be polymerized and used at much higher molecular weights than their solution counterparts due to viscosity considerations. This particular factor allows dispersions to develop good chemical and solvent resistances just by physical drying. A further step in resistances can be made by crosslinking water-based systems with polyfunctional aziridines. As this crosslinking system has disadvantages such as mixing before application and handling of reactive materials, our research efforts are directed towards resistant systems without crosslinking. New generations of acrylic emulsions and urethane dispersions will therefore be introduced to the market.

# Selection of products for water-based plastic coatings

Acrylic emulsions and urethane dispersions and blends of both are being used commercialy on plastics for areas such as furniture, vinyl upholstery, vinyl flooring, interior aircraft parts, interior and exterior automotive parts, business machines etc. Main problem in formulating these products is the right emulsion choice. In some cases just one emulsion will be sufficient to reach the required performance, in other cases combinations of acrylics and/or urethanes will be needed when many different performance properties are required at the same time.

Table 7 illustrates the various possibilities and demonstrates that often a combination of products *must* be used.

 Table 7.

 Main products for water-based plastic coatings

Code	PS/ABS	Adhesion PC/Mod.PC	PPO/PA	Hardness	Flexib. Impact	Solvent r Alcohol	esistance Petrol	Water Humidity	Chem. Resist.	Outdoor Durability
Al	+	++	+	0	+	0	0	++	0	++
A2	++	++	++	0	0	_	_	_	-	-
A3	-	0		++	-	++	+	0	+	+
A4	0	+		0	+	0	0	++	+	+
A5	0	+	0	0	+	0	0	++	+	++
EA6	0	0	0	++		+	+	0	++	+
EA7	++	++	++	+	0	++	_	0	0	0
A8	++	++	+	+	-	-	0	+	-	++
U1	_	+	_	+	++	_	++	0	+	0
U2	_	_	-	+	++	-	++	0	0	0
EU3	-	++		0	++	+	++	++	÷	++
EU4		++		+	++	++	++	++	++	++

Key

A = Acrylic, U = Urethane, E = Experimental

Referred is to non crosslinked products

++ = excellent

+ = good 0 = fair

0 = 1an

- = bad

With these combinations following properties important for plastic coatings can be reached:

- Good adhesion on many plastics.
- Flexibility can be adapted to the substrate.
- Excellent mechanical resistances (also at low temperatures).
- Good chemical resistances.
- Yellowing resistance and excellent outdoor durability.
- No high stoving temperatures needed.
- No solvent attack to the substrate.

With the help of some practical examples, how selections can be made, is illustrated.

# **Example 1**

Business machine coatings

Substrate: ABS, PS, PPO, PC, fosfated steel.

Requirements: - Adhesion, hardness, impact

- Taber abrasion
- Solventspot tests (toluene, alcohol, chl. solvents)
- Chemical spots (Coke, coffee, nailpolish)
  Cold check
- Cold check - Humidity resistance
- Practical solution: Combination of A1 and A3. Combination of U1 and A3.

The business machine industry is known as a fast growing, high quality demanding industry that uses enormous quantities of different plastic types mostly in the form of injection moulded structural foams. In practically all cases these plastics have to be coated, often in the well known textured patterns.

Apart from the already mentioned environmental and hygienic reasons, this industry uses water-based coatings as requirements include applicability on all types of plastic and metal at the same time, and water is acceptable for all these substrates.

Water-borne business machine coatings are still more common in the USA than in Europe, but a clear trend towards water is noticeable now in UK, France, Germany and Benelux.

# Example 2

Interior Automotive Plastic Coatings

Substrate: ABS, PVC, PPO, Fiberboard.

- Requirements: Adhesion, Scratch resistance
  - Cold check
  - Humidity resistance
  - Rub resistance dry and with cleaning
    - solvents (alcohol, alifatics, detergents)

Practical solution: - Acrylic A4, EA6 or EA7 as sole binders - Combination A8/U1 with crosslinker

Water-based coatings for interior automotive plastics are very widely used in the USA, although in several cases in Europe also dispersions based systems are used. The variety of parts, often made of different plastics and with different coating requirements makes it difficult to just give one single solution. The rub-resistance tests with solvents are especially decisive when chosing the polymer.

# **Example 3**

Coatings for Audio/Video Equipment

Substrate: ABS, PS, fosfated and galvanised steel

Requirements: - Adhesion, scratch resistance

- Rub resistances dry and with ethanol/dry cleaning naphta
  - Perspiration resistance
  - Hand-cream resistance
  - Climatic tests
  - Metallic colours

Proposed solution: - Combination of A1/A3

- Combination of A1/EA6
- Combination of EA7/A3
- Combination of EA7/EA6

In several Western European countries this subject is relevant as for environmental reasons a water-based alternative is wanted for the used TPA based coatings.

The introduction of aluminium hydro pastes enables us to formulate water-borne metallics. These hydro pastes contain a stabilizer to protect the metal flake and or surfactant to aid in dispersing the pigment in water. The aluminium/water reaction however is complex and more investigations will be needed to optimilize these systems.

Hand cream resistance is one of the most critical requirements here, (tested at  $70^{\circ}$ C), especially in combination with good adhesion.

# **Example 4**

Barrier coating for automotive bumpers

Substrate: Modified polycarbonate

**Requirements: - Adhesion** 

- Overcoatable with two pack solid colours or base/clear metallics
- Remaining good impact resistance even when paint is applied on stressed substrate
- Humidity resistance of total system

Proposed solution: - Combination A1/A3/U2 - Combination A4/U2 - Combination A5/EU3

In the USA especially this is an actual problem, as automotive appearance is "fully body coloured". For several plastics a "barrier" is required under the solvent based topcoat system that has to match the exact body colours. Although water-borne urethanes seem to be made for this job, the fact that solvent based topcoats will not adhere to them, makes is necessary to combine acrylics and urethanes in this case.

# Conclusions

- The use of plastics will increase and so will the use of plastic coatings.
- Water-based coating systems for plastics are a reality now and will continue to find greater acceptance.
- New products will be introduced to meet high quality level demands.
- Only close co-operation between plastic user, plastic producer, paint producer and binder producer can solve the problems that the constantly changing demands create.
- That is why the author is willing to have close contact with those paint producers, that are not only interested, but that are really prepared to invest in developing coatings for plastics.

# Acknowledgements

I would like to thank my USA-colleague John F. Fitzwater for his valuable contribution to this paper.

[Received 18 April 1985

# Accelerated weathering in UVCON: Fast and cheap.



Reader Enquiry Service No. 290

# **Natal Section**

# Trademarks and copywright

A meeting of the Natal Section was held at the Westville Hotel on Wednesday 12 June 1985.

A lecture entitled "Trademarks and Copywright" was given by Mr Ian Morrison, a Patent Attorney. This was the follow up from Mr Morrison's previous lecture of March 1984. The speaker initially outlined the laws controlling trademarks and copywright. A trademark is a registered item and the law is divided between trademarks and trade names. A trademark must be distinctive of the goods and the speaker gave the derivation of certain well known marks, e.g. Kodak, Coca-Cola, 4711.

To register a trademark, an extensive search must be made before an application is accepted. Once the registrar is satisfied the application is open for objection for two months. Once approved, a trademark is renewable after ten years. For up to seven years a mark may be expunged if reasonable objections are made. However, after this period a mark can only be expunged if non use is proven for a period of five years.

Abbreviations and numbers can only be registered as trademarks after proving extensive common usage. To prevent trademarks becoming generic terms as in the past, e.g. nylon, duco, recent applications are extremely specific in respect of the exact detail of the article concerned.

Mr Morrison concluded his lecture by explaining how copyright, which traditionally covered the expression of the idea in an artistic medium, i.e. music, literature, films, has recently been used in the industrial field whereby companies retain the copywright of employees articles or formulae whilst in their employ.

A vote of thanks was given by Alan Mackenzie and the audience responded with a generous round of applause.

R. H. Philbrick

# conference dircurrionr

Discussion is encouraged at OCCA Conferences following the presentation of papers. Those who put the questions were asked to fill in discussion slips with the details of their questions. Edited versions of the discussions that followed two of the papers published in the **August 1985** issue appear below: questions in ordinary roman type, answers in italic. Only those questions for which discussion slips were received are published.

# Cationic electrodeposition primers over zinc coated steels Part 1: Effect of voltage and substrates on coating quality <sup>C. Schoff</sup>

G. C. FETTIS: In addition to the substrate related pits, have you seen other causes such as separation of clear resin at the bottom of the pits or the effect of impurities in the tank such as silicones?

C. SCHOFF: The pits and craters that I described in the paper were caused by electrochemical processes, but we are quite familiar with the surface tension driven craters that you mention. Certainly, silicones, mineral oils, resin gels and other impurities in the electrodeposition bath or on the substrate can cause such craters.

D. SCANTLEBURY: It is well known that the corrosion rate of zinc is dependent on the impurity levels, specifically iron, present as an iron/zinc intermetallic. This is thought to be due to the increased kinetics of the hydrogen evolution reaction on the iron/zinc intermetallic compared with pure zinc. Have you thought about this as a possible explanation?

C. SCHOFF: We are not aware of the influence of iron/zinc intermetallics on the corrosion rate of zinc. This could be involved in the formation of defects, but at this point we are

not certain as to the importance of low voltage, low field strength processes such as corrosion to electrochemical cratering. Cationic electrodeposition is a high voltage (250-400V) phenomenon involving high field strengths. Conventional thinking is that corrosion, intermetallic couples, etc., would be swamped by the electrodeposition process. However, our depositions begin with the voltage off and recent experiments show that cratering is found after only a second of deposition. Therefore, the cratering in our lab bath probably begins at relatively low voltages and field strengths. Automobile electrodeposition baths are live on entry of the car bodies, but the point of entry is a considerable distance from working anodes. Conditions could be such that intermetallic couples would influence behaviour. More work needs to be done to sort this out.

W. J. VAN OOIJ: Has the author studied the effect of phosphating the substrate on its cratering sensitivity?

C. SCHOFF: Our results and those of others indicate that phosphating the substrate has little or no effect on its cratering sensitivity. The zinc phosphate appears to be transparent to the electrochemical processes involved in the formation of the defects.

# conference dircurrions

W. J. VAN OOIJ: I have observed that craters on galvanised steel are predominantly formed in areas rich in A1203 at the surface. This could suggest that the distribution of anodic and cathodic sites at the surface of certain metals and alloys is an important factor, e.g. on zinc, zinc-iron, etc. It would explain why pure zinc sheet shows a better performance. At the anodic sites, zinc is dissolved (at high pH) at low voltages which results in "pockets" of high ionic conductivity and hence, discharge phenomena. Could you comment on this?

C. SCHOFF: Your comments are very interesting. We had accepted the contention of steel companies and others that alkaline cleaners and phosphating would remove the aluminium oxide from the surface of galvanized steel. It appears that we should analyze the surfaces of our substrates to determine the exact materials on them. I agree that zinc dissolution at anodes could result in regions of high ion coincentration and, therefore, high conductivity which would be the "hot spots" giving electrical discharge.

# Oxo-acetates-new solvents for high solid paint formulations

# M. A. Taylor

G. C. FETTIS: Does the new solvent range enable the European appearance with low solids automotive metallic paints to be achieved with high solids VOC compliant systems?

M. A. TAYLOR: Full evaluation programmes of these new Exxate grades are running in co-operation with several European resin and coating manufacturers. We understand that meeting the appearance standards as obtained with low solids metallic automotive paints while using high solids metallic automotive paints with VOC compliance systems is one of their targets still to be achieved. While awaiting the results of these evaluation programmes. we expect them to confirm our opinion that required appearance standards can be achieved with Exxate solvents.

# new/

### **Casa Chemicals to distribute Dow** Latex DL-420 E

Casa Chemicals Ltd of Consett, Co Durham, associate of specialities manufacturer Thomas Swan, has been appointed distributor for DL-420 E, a carboxylated styrene acrylate polymer manufactured by the Dow Chemical Company.

Principal application of DL-420 E is a binding agent in building industry products including stucco coatings, water bourne paints and ready mixed tile adhesives. Principal advantages claimed for the product in these applications include excellent resistance to the outdoor environment, a high pigment tolerance and the provision of a highly rigid "body" for stucco coatings. These benefits help to provide end products of high elasticity and good "crack covering" properties that are easy to apply and that will provide acceptable results with relatively thin coatings. Formulated products also have very good shelf life.

Typical properties of DL-420 E include: solids content, 48-50 per cent; Brookfield Viscosity (RVT 20 rpm), 500 mPa.s (max); styrene content, 47 per cent, particle size, 0.14 um; tensile strength (dry/wet), 900/700 N/cm<sup>2</sup>, elongation (dry/wet) 450/350 per cent, nature or deposited film, clear and continuous. Stability to light Cray Valley Products Limited have

exposure, mechanical stability and compatibility with fillers are all said to be excellent.

Reader Enquiry Service No. 29

#### Paint and DIY products acquisition

Reed International Plc has acquired, through Reed Holdings Inc the share capital of Deer-O Paints and Chemicals Ltd, of Phoenix, Arizona for approximately \$2.6 million in cash.

Deer-O, which operates a paint factory and three stores in Phoenix, together with another store in Tucson, earned \$0.5 million before tax on turnover of \$8.2 million in its last financial year.

This purchase continues the expansion of Reed International's Paint and DIY interests in North America. It will be integrated with Frazee Industries of San Diego, California which was acquired in July 1984, and whose operations include two stores in Phoenix.

Reader Enquiry Service No. 30

# **Cray Valley Products Iberica SA**

acquired from Croda International PLC their shareholding in Croda Espanola SA. As a result of this acquisition the Spanish company will be renamed Cray Valley Products Iberica SA and will in due course add to its range of Alkvds, polvamides, amine resins and vinyl emulsions many CVP resins. The company will continue to be managed by Carlos and Xavier Ochoa who retain their shareholding in the joint venture. Products will still be manufactured at Mollet del Valles near Barcelona, while in addition CVP resins currently imported into Spain for the coating industry will be sold by the Spanish company. Mario Calcagno the current agents for CVP will remain as sales agents for dimer and fatty acids.

Reader Enquiry Service No. 31

# **Essochem Solvents plans Antwerp** expansion

Essochem Belgium is developing a multimillion dollar investment to increase the capacity and flexibility of its solvents plant at Antwerp. The new facilities are planned for start-up in mid-1986.

The Antwerp plant is already Europe's largest hydrocarbon solvents plant with a capacity of 360,000 tons per year. The

#### expansion will increase it to 550,000 tons per year. A significant portion of the new investment will be spent on offsite facilities, and in concert with the recent expenditures of over seven million dollars in this area will give this plant added flexibility to respond to specific customer needs and to ensure consistently high quality products. It produces Essochem's full range of hydrocarbon solvents, including Varsol, Exxsol D, Isopar, Escaid and Solvesso Speciality Solvents. These products are used in a broad spectrum of end uses including coatings, adhesives, inks, oil seed extraction and polymerization.

Reader Enquiry Service No. 32



### Luminescent glazes and decorating colours for the ceramics industry

The Chemicals Research Division of Degussa AG, Frankfurt am Main, has developed phosphorescent and fluorescent glazes and decorating colours for the ceramics industry. The new products, said to be suitable for firing in the temperature range from 780°C to 1,100°C, are available from the company's Ceramic Colours Division under the name "KeraLum".

The impression of colour in conventional glazes and decorating colours is created by absorption of a certain part of the light which falls upon them, whereas the remainder of the spectrum is reflected. The newly-developed KeraLum products, however, are based on a completely different principle. They absorb radiated energy, which is given off again when the electrons are raised to a higher state of excitation by converting them to a lower state of energy, as a result of which visible light of precisely defined wavelength is emitted.

KeraLum phosphorescent decorating colours and glazes possess the property of retaining excitation energy and consequently of emitting it again over a long period. Fluorescent KeraLum products, on the other hand, emit the energy again very shortly after excitation. In both cases, excitation may be carried out by light from the visible range of the spectrum or by ultraviolet A light (366 nm, disco light), ultraviolet C light (254 nm) and X-rays.

Potential areas of application for the new KeraLum decorating colours and glazes are luminous wall and floor panels for tunnels, emergency exits and underground rail stations, decorative ceramics for discotheques and bars, ceramic advertising panels and company logos.

# new/

#### **Glasurit puts Pearl fizz in Cheryl Baker's boat**

A 1974 32ft Fairline Phantom owned by Cheryl Baker of Bucks Fizz is the first boat in the UK to be refinished in Glasurit "Pearl Effect" acrylic paint by Kaymar Marine of Brighton Marina. The paintwork was completed in time for exhibition at the Brighton Boat Show.

The refinish, which was part of a refit by Kaymar, was carried out in conjunction with Glasurit Valentine demonstrators and involved removing fittings, scraping and cleaning the hull, drilling out and repairing flaws such as star cracks, sanding down and masking out.

White Pearl Effect was used on the

cabin, deck and hull, with the superstructure sprayed in blue Pearl Effect. White and metallic blue stripes were then applied with Glasurit 21-line and 54-line paints respectively.

Glasurit was the first company to introduce "Pearl Effect" which was originally introduced for the automotive industry but is an excitingly new finish ideal for boats. It contains white mica particles that refract light. The colour therefore changes (flips) as the subject, viewer or light source moves, an effect similar to that of Mother of Pearl.

Reader Enquiry Service No. 34



Cheryl Baker and her Pearl finished Fairline Phantom

Special KeraLum decorating colours and glazes are formulated to react only to hard X-rays, which means that wall and floor panels of this material can be used to alert personnel working in X-ray rooms if harmful, uncontrolled radiation is escaping.

Reader Enquiry Service No. 33

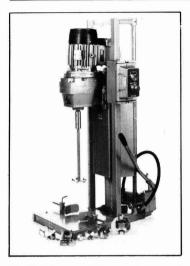
# Credit laboratory high speed disperser/mixer

John Godrich have recently introduced Credit laboratory high speed disperser/ mixer. It is claimed to have been designed with ruggedness in mind, flexibility of operation and economic production.

Supplied as standard with an EX d flame-proof motor with variable speed

drive, it will mix between half a litre and twenty-five litres of material using standard dissolver discs. It has, however, been designed to carry out a multiple of different jobs using a variety of different types of mixing head.

Apart from the dissolver disc style of mixer, it can also be fitted with the well known and tried Rotostat X1 mixer/emulsifier head or the Rotostat T05 high shear. mixing head. These units are of the rotor/stator design with a free flowing stator thus giving lower mixing times and capable of mixing, dispersing and emulsifying heavier viscosity materials than normal. The Credit HSD can also take the Berthou propeller for efficient agitation and other sizes of dissolver discs. The stand that it is mounted on is built with a hand operated hydraulic lifting device and can



Credit laboratory high speed disperser/ mixer

lift to a height of 420 mm. The hydraulic lift can be motorised, if required. It is supplied as standard with adjustable vessel clamps and has a detachable floor level. The variable speed drive of between 700 and 3500 rpm gives flexibility of oepration using the many and easily changeable mixing heads available for this machine.

Reader Enquiry Service No. 35

# Speedcoat 3, a quick dry P/U finish

Speedcoat 3, PPG's commercial vehicle polyurethane reinforced enamel—the third member of the Speedcoat trio of commercial transport finishes, all derived from one mixing system—has been given a boost and is to be re-launched as a "Quick Dry P/U Finish'.

Retaining all the properties of chemical resistance of a polyurethane combined with the glossy looks of a conventional alkyd, new Speedcoat 3 is now said to be faster drying.

Recoatable overnight with tapping times that compare favourably with most P/U systems on the market, new Speedcoat 3 has already been in use by selected customers for around twelve months.

Speedcoat 3 Quick Drying P/U Finish is especially recommended for spray application for large trucks and buses.

Reader Enquiry Service No. 36

# New Elecometer magnetic coating thickness gauge

Elecometer Instruments have launched a new magnetic coating thickness gauge, the Elecometer 211.

The new gauge is claimed to incorporate all of the features of their Elecometer 111 'Inspector' gauge and many other features which have been developed to make it the most complete gauge of its kind in the world. An increased accuracy of ±5 per cent and a range of 0-1000 um or 40 thou/mils on one scale are key improvements, making the 211 a very capable and highly cost effective instrument. It is rugged and portable and needs no power supply, especially designed for those more rigorous and hazardous on-site painting environments. Factory calibrated, the gauge measures all non-magnetic coatings on a ferromagnetic base.

Reader Enquiry Service No. 37

day Symposium, which will discuss the latest developments in automotive coatings, and will debate whether the presentday sophisticated paints and painting systems have created additional problems for the refinishers.

Reader Enquiry Service No. 38

### Wood finishing

Verniciatura del Legno (Wood finishing) will organize the II Technical Conference on finishing of wood in exterior.

The main subject will be: Biologicial protection of wood; Organic painting of wood; Water borne paints for wood finishing; Vacuum treatments; and Standards.

The conference will take place in Milan on 22 November 1985. For further information contact Verniciatura del Legno, Via Imbriani 10, 20158 Milano, Italy.



The Elcometer 211



#### Vehicle Finishing and Refinishing

A two day Symposium on Finishing and Refinishing of Vehicles takes place on 4-5 December 1985 at the West Hotel, London.

There have been significant developments in the protection of automobiles during the last decade. Better design, better paints and better application methods have resulted in greatly improved mass-produced cars.

The Paint RA, in association with the Motor Industry RA, is organising this two



John Richmond joins Polymer Laboratories as technical representative. John has a comprehensive and very practical background, including teaching instrument techniques to post graduates at Surrey University. Previously a Perkin Elmer Sales Specialist, John will promote PL's DMTA, Minimat, etc., in the southern UK region, Switzerland, Italy, Germany and other selected overseas territories.



J. C. Richmond

# occa new/

# Manchester Section

# 1985 Northern Section Golf Tournament

OCCA members and their guests from all Sections were invited to the 1985 event held on Tuesday 28 May 1985 at Pannal Golf Club, Harrogate, Yorkshire. The weather was excellent, the course in fine fettle and after a light and liquid lunch 33 members and their guests did battle in groups of three according to Dr Stableford's Rules of Golf (scoring).

It was all over bar the usual golf frustrations of not achieving one's best on the day and the evening was enhanced by an excellent meal according to the usual high standard of "Yorkshire Grub".

Then it was time for the prizes, the main award being the Tony McWilliam Trophy—based on the submission of the best 3 individual scores from each OCCA Section.

Once again the Manchester Section won the Trophy with a combined score of 99 points accorded to Brian Lamb (36), John Sinclair (34) and Ron Ashton (29).

The Host Section, West Riding, were second with a commendable 80 points and the team was composed of Phillip Jones (37), Terry Wright (22), M. Baker (21).



Manchester Section winning team with Section Chairman Mr B. Windsor shown on the left, and Mr J. Jackson, Chairman Golf Committee, in the background.

The guests were led by M. Wright (38) and P. Rowan (38) with Phillip Jones being the highest scoring OCCA member. It was pleasant to welcome OCCA members from the London, Midlands Sections and for the first time in this competition a lady competed as a guest of her husband. Admiration of the state of matrimonial harmony forbids mention of the respective spouse scores. An excellent golf tournament, many thanks to the many companies and individuals who donated prizes and the Manchester Section Golf Committee for their organisation, hopefully to be repeated on Tuesday 27th May 1986.

F. B. Windsor

# South African Division

It had long been the custom for Council to ask the Sections in South Africa and New Zealand each to nominate a prominent member resident in those countries as Vice Presidents.

Thus, when the Divisions were formed in these two countries by a grouping of Sections, the Vice Presidents in both cases also became the Chairmen of the Divisions.

To mark their service as Chairmen of Divisions, Council has authorised the production of special lapel badges for the Vice Presidents who have also held office as Chairmen of Divisions. The Past Chairmen's badges for the South African Division were presented at an ordinary meeting of the Transvaal Section on 24 July 1985 by the present South African Division Chairman and Vice President, Ralf Johannsen.



(From left to right) Bob Rouse (Chairman 1982-84), Helen Gaynor (Chairman, Transvaal Section), Peter Gate (Chairman 1978-80), Ralf Johannsen (Chairman, South African Division and Vice President), Dirk Pienaar (Chairman 1976-78). (Note: Roly Eglington—Chairman 1980-82—received his badge at the Natal Section meeting on 14 August 1985).

# **Newcastle Section**

# The British Titan Cup

The 24th annual tournament for the British Titan Cup was played for over the High Plains Bishop Auckland golf course on Friday 7 June 1985.

The competition, a Stableford, was won by Mr J. G. Bell of Crown Paints, Haltwhistle. This is the sixth time that John has won this trophy since its inception in 1962, "congratulations John". The best performance by an invited guest was returned by Mr W. W. Jack of Tioxide (UK) Ltd.

Although the weather has been better on previous occasions, the 17 competitors enjoyed a very pleasant afternoon.

H. Fuller



Mr R. G. Carr (left), Chairman of Newcastle Section, presenting the British Titan Cup to Mr J. G. Bell.

# **Transvaal Section**

# The thirty sixth AGM

The 36th AGM of the Transvaal Section was held at the Escom Club, Megawatt Park, on 22 March 1985. The meeting was kindly sponsored by NCP Chemical Marketing, who provided a potable spirit nosing contest for arriving guests, plus a most able and amusing after-dinner speaker in the person of Mr John Counihan.

The Chairperson, Mrs Helen Gaynor, tabled her report for the past year, during which ten general meetings and one field trip were held.

The following members were elected for the forthcoming year:

Chairman: Helen Gaynor Hon. Secretary: Ron Cromarty Hon. Treasurer: Peter Quorn Hon. Publications Secretary: Bryan Bailey Committee: Dirk Pienaar, Peter Gate, Jan de Jong

B. Bailey



At the meeting of the Professional Grade Committee held on 14 August 1985 the following admissions were made:

#### Transferred from Associateship to Fellowship

Morpeth, Frederick Johnson (Manchester) Windsor, Frederick Barry (Manchester)

# Admitted to Associateship

Drury, James Roland (Transvaal) Mehta, Ramesh Mulshanker (London) Ronson, David James (Auckland) Say, Terence Edwin (Natal) Sheath, Alan (Auckland)

# Admitted to Licentiateship

Wilkinson, Stephen James (Manchester)

# Manchester Section, Oil & Colour Chemists' Association 60th anniversary diamond jubilee brochure

Manchester Section of the Oil & Colour Chemists' Association celebrates its 60th Anniversary on 6 November, 1985. To commemorate this event Manchester Section has published a Diamond Jubilee Souvenir Brochure which is being circulated to OCCA members, both in the UK (with the September issue) and overseas. Of special interest is an article on the history and development of the surface coating and allied industries, with particular reference to the North West. The article is illustrated with many interesting historical photographs, maps and drawings. There is also an article outlining the history of the Manchester Section from 1925 to the present day.

A limited number of copies are available for **non OCCA members** at a price of £2.00 (cash with order) to cover cost of post and packaging from: The Hon. Secretary, Mr S. G. Hayes (c/o Mrs Linda Jenkins), Tioxide UK Ltd., Didsbury House, 748/754 Wilmslow Road, Didsbury, Manchester M20 0DW.

The brochure is available free of charge to General Overseas Members of the Association. Please quote your OCCA reference number as shown on your 1986 subscription reminder.



# **Ordinary Members**

Booth, D. I. (General Oversees-Yemen) Colley, K. S. (Wellington)
Dobson, S. E. (Irish)
Graham, D. L. (London)
Maguire, B. F. (Transvaal)
Mogilnicki, W. S. (Transvaal)
Obade, A. S. (General Overseas-Yemen)
Scott, J. F. (Transvaal)
Sheibani, S. M. (General Overseas-Yemen)
Smith, G. F. (London)
Stuart, E. I. (Wellington)
Teo Yan Huay, L. (General Overseas-Singapore)
Wischeart, S. P. (Wellington)

# **Associate Members**

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Reprints of articles and papers in **JOCCA** are now available

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The data collected by Tracker is analysed against the recommended curing schedule by the notebook computer. Using the unique DATAPAQ VALUE routine (where the temperature profile is presented as an index of curing achieved), optimising changes in the process conditions can be made that can increase productivity by up to 10%.

Tracker is simple and easy to use. No skilled interpretation or guesstimates of the data are required - Tracker does it all. In addition the mini-printer produces a hard copy of both routine quality control data and an accurate 4 colour graph.

This previously unobtainable information allows a very high level of control of the production line resulting in more consistent quality finishes, optimum energy usage and the elimination of colour drift from overcure or costly reprocessing caused by undercure.

The complete Tracker system consists of a 64k NEC notebook computer, 4 colour mini-printer, black box', computer interface, 4 temperature probes, thermal jacket and all necessary leads and cables. The system fits neatly into a tough carrying case.

For further information and a site demonstration of Tracker please ring:-John Bates 0223 862141



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# **Sun Chemical Corporation**

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