

# JOURNAL OF THE OIL AND COLOUR CHEMISTS' ASSOCIATION



✓ Vol. 51 No. 12

December 1968

Reflow thermosetting acrylic resins—some aspects  
of their performance and usage

*J. R. Taylor and H. Foster*

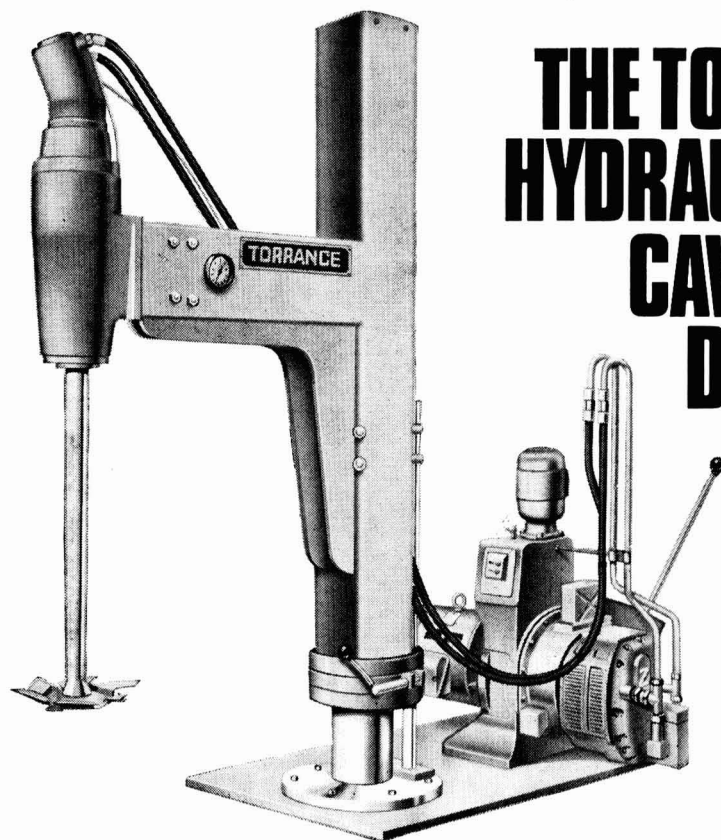
The rheological shear-rate-limit method as an aid in  
paint production

*K. M. Oesterle*

**INDEX—1968**

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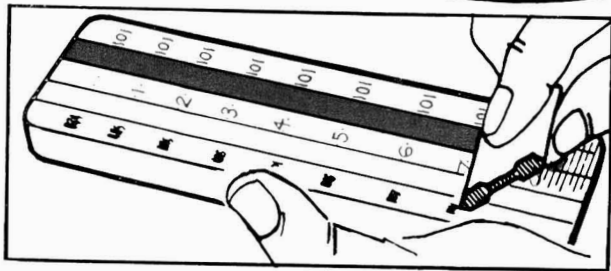
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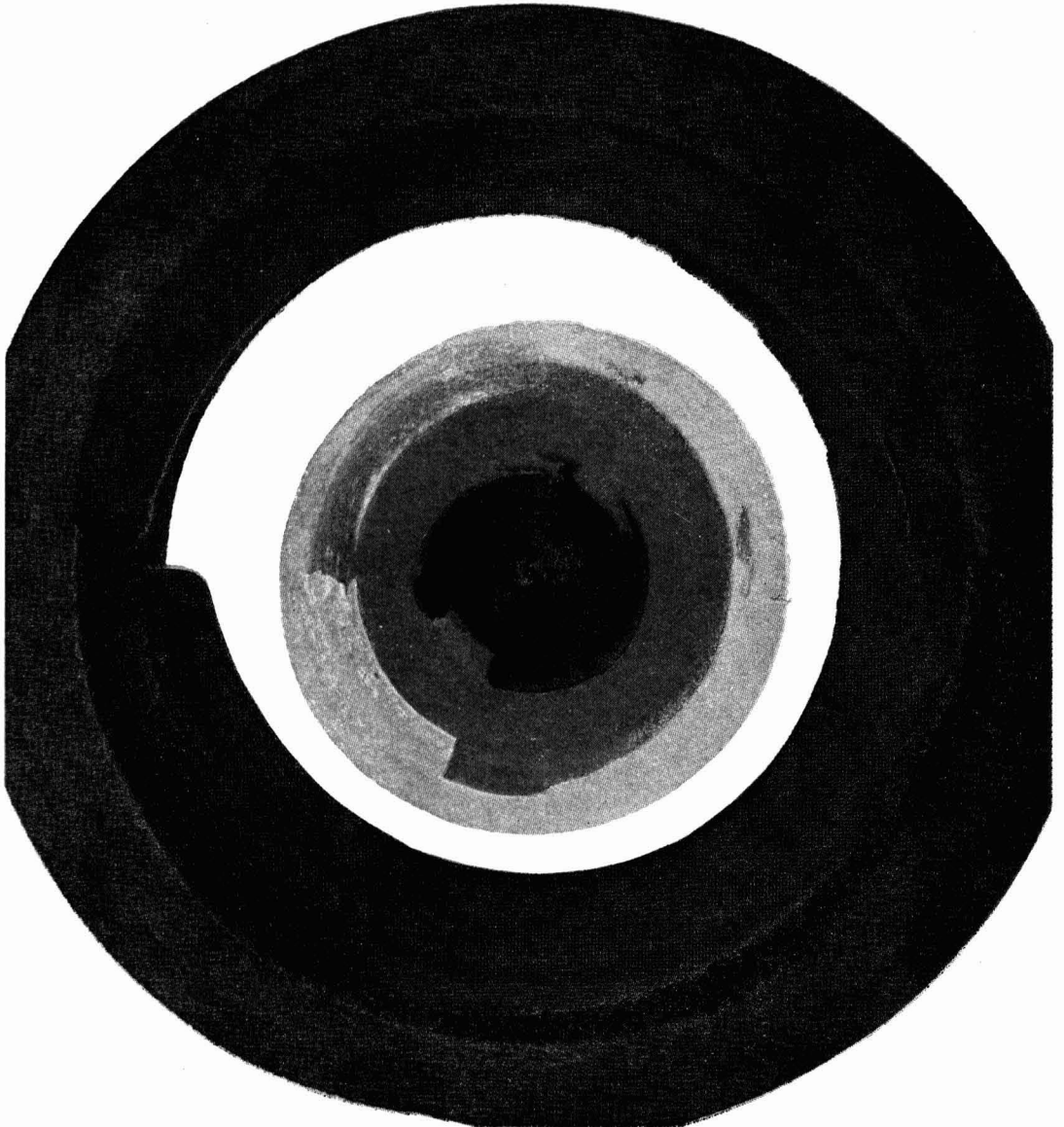


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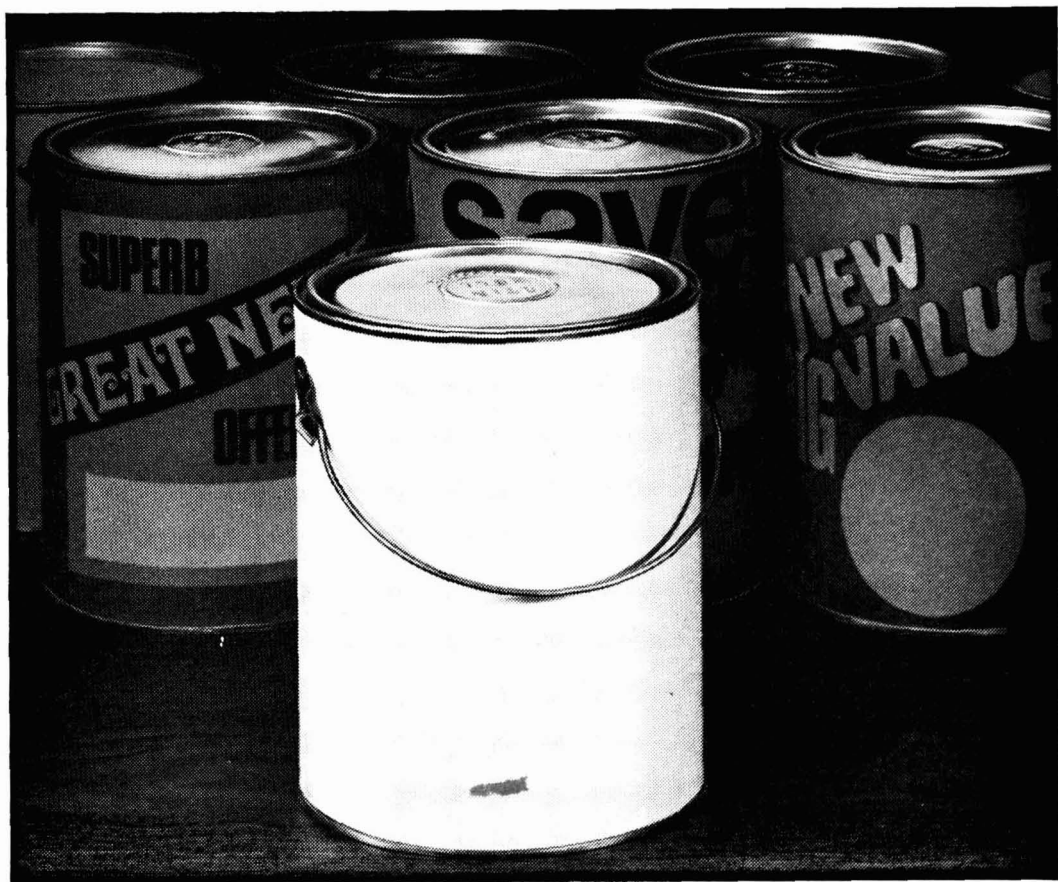
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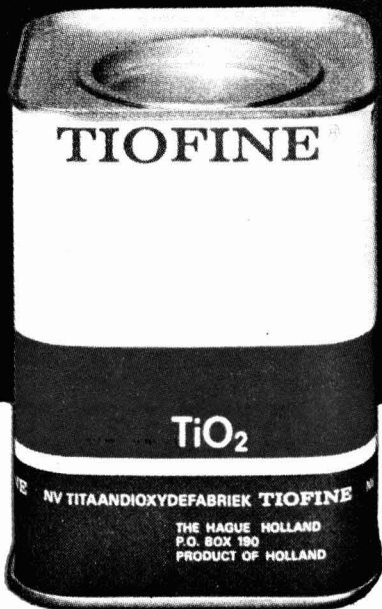
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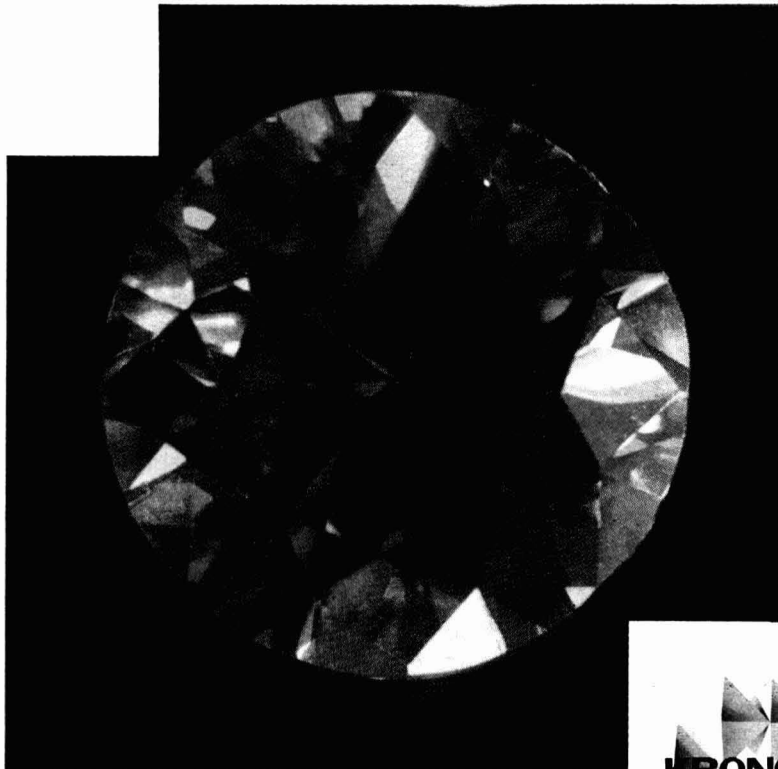
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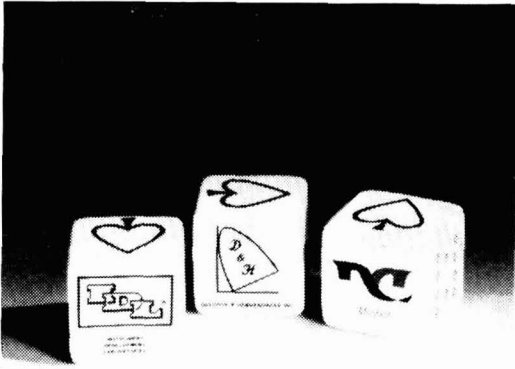
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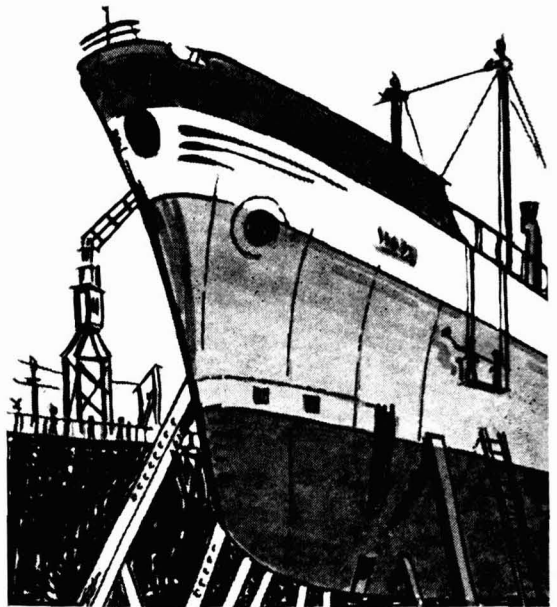
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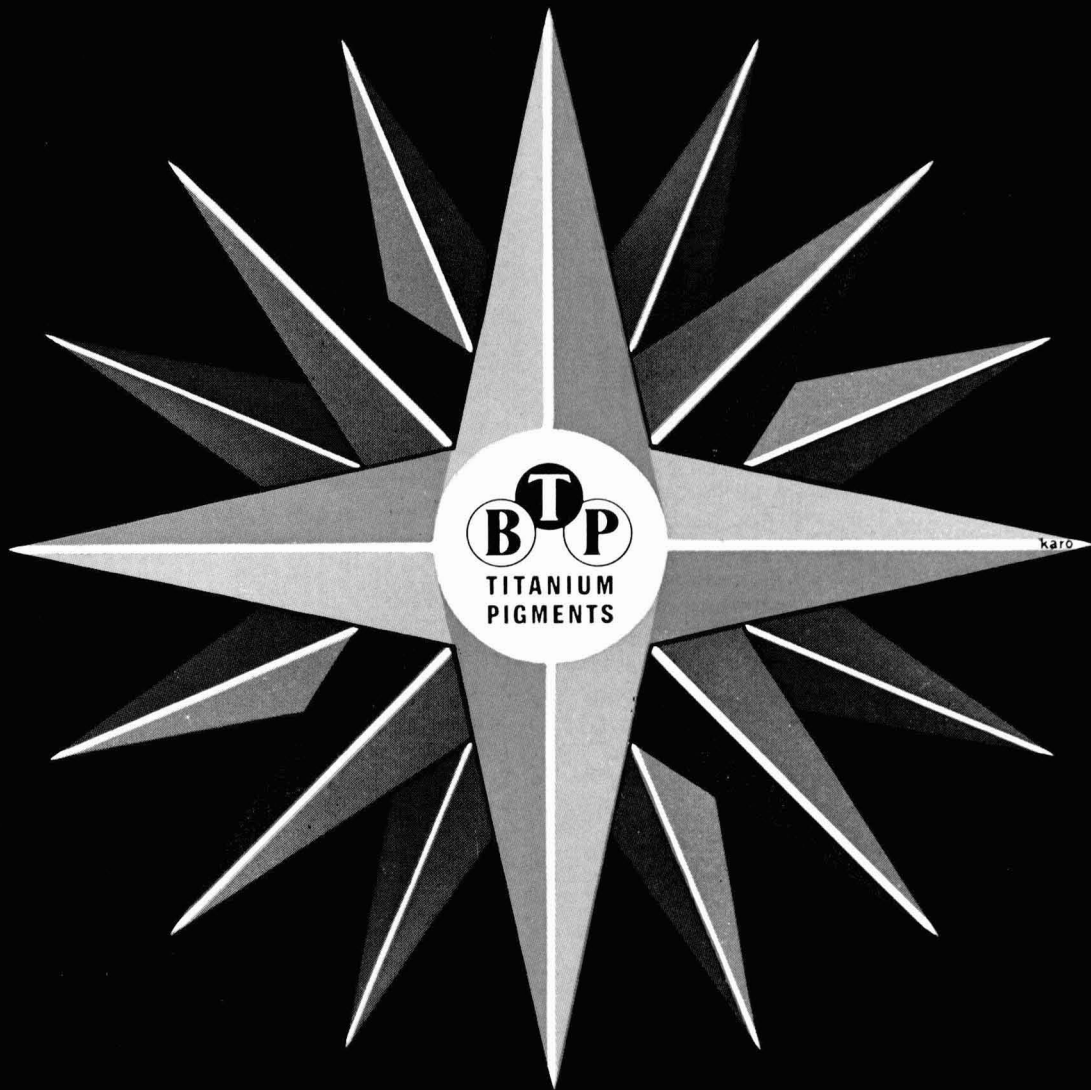
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## ***Transactions and Communications***

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# **Reflow thermosetting acrylic resins—some aspects of their performance and uses\***

**By J. R. Taylor and H. Foster**

BP Chemicals (UK) Ltd., Sully, Penarth, Glamorgan

### *Summary*

An account is given of the practical and theoretical considerations of the use of thermosetting acrylic resins containing hydroxy acrylates and crosslinked with melamine resins in the automobile industry.

The factors such as choice of resin, pigmentation, curing rates, which affect sanding properties, flow and overspray acceptance in reflow systems are described. An attempt is made to correlate the suitability of a film to give good reflow properties by measurement of film hardness, gloss, and surface profile.

The colour and distribution of pigments such as titanium dioxide and aluminium powders in the paint films before and after sanding and reflow are investigated by examination of film sections using the optical and electron microscopes. The performance of these new acrylic resins in the cold check resistance test for thick films incorporating final coats of the enamel, acid catalysed and stoved at low temperature are shown. The weathering properties of metallic and solid colour enamels are also compared with those based on alkyd/amino resin systems.

Reference is made to the possible restrictions in formulation of thermosetting acrylic enamels by the introduction of the Los Angeles Rule 66 against atmospheric pollution by certain organic solvents.

### **Resinés acryliques thermodurcissables destinées aux revêtements refluidisables**

#### *Résumé*

On considère, au point de vue de leur utilisation par l'industrie automobile, les aspects pratiques ou théoriques des résines acryliques thermodurcissables, contenant d'hydroxyacrylates, reticulées par des résines mélamines.

Les facteurs telles que, type de résine, pigmentation, vitesse de durcissement, qui influencent l'aptitude à ponçage, l'écoulement et la tolérance de relaquage dans des systèmes refluidisables sont décrites. On essaie de déterminer l'aptitude d'un feuillet à rendre de bonnes propriétés de refluidisation par le mesurage de dureté de feuillet, de brillant et de profil superficiel.

La couleur et répartition des pigments tels que le dioxyde de titane et des poudres d'aluminium dans le feuillet de peinture avant et après le ponçage et également la refluidisation sont étudiées par les examens microscopiques optiques ou électroniques des sections de feuillet. On démontre le rendement de ces nouvelles résines acryliques pendant l'essai de résistance à "cold check" des films épais comprenant les couches supérieures de l'émail, catalysée par acide et cuite au four à faible température. On fait une comparaison des caractéristiques de vieillissement des émaux à poudre métallique ou de teinte solide avec celles des systèmes basés sur des résines alkydes/amines.

On fait référence aux prescriptions possibles à l'égard de la formulation des émaux à résine acrylique thermodurcissable suivant l'introduction de l'arrêté municipal No. 66 de la ville de Los Angeles concernant la contamination de l'atmosphère par certains solvants organiques.

---

\*Presented to the West Riding Section on 13 February 1968.

## **Aufschwitzbare, Wärmehärtbare Akrylharze**

### *Zusammenfassung*

Die praktischen und theoretischen Gesichtspunkte, welche bei der Anwendung wärmehärtbarer Akrylharze, die Hydroxyakrylsäureester enthalten und bei Anwendung in der Autompbilindustrie mit Melaminharz vernetzt werden, in Frage kommen, werden daegelegt.

Es werden solche Faktoren, wie die Wahl des Harzes, der Pigmente, die Härtungsdauer, welche die Sandelung, den Verlauf, die Spritzstaubaufnahme im Aufschwitzsystem beeinflussen, beschrieben. Auch wird durch Messungen versucht, die Eignung eines Films zum Aufschwitzen mit der Filmhärte, dem Glanze und dem Oberflächenprofil in Beziehung zu bringen.

Unter Zuhilfenahme des optischen und des Elektronenmikroskopes werden Filmschnitte auf Farbe, Pigmentverteilung z.B. von Titanweiss und Aluminiumpulver in Lackfilmen vor und nach Sandelung und Aufschwitzen untersucht. Das Verhalten dieser neuen Akrylharze beim Cold Check Test für dicke Filmsysteme, welche die Filme der Überzugsemailen einschliessen, wird aufgezeigt, und zwar wenn mit Säure katalysiert oder bei niedrigen Temperaturen im Ofen getrocknet. Ebenfalls wird die Wetterbeständigkeit metallischer und einfarbiger Emailen mit solchen auf Alkyd-Aminharzbasis Systemen verglichen.

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### *Резюме*

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Описываются такие факторы как выбор смолы, пигментация, скорость сушки, влияющая на качества очистки, поток и переопрыскивание в перетекающих системах. Делается попытка уточнить соотношение применимости пленки дающей хорошие качества перетекания, путем измерения твердости пленки, лоска и поверхностного профиля.

Обсуждаются цвет и распределение таких пигментов как например двуокись титана и алюминиевые порошки в красочных пленках до и после очистки песком и перетекания, путем анализа срезов пленки, применяя оптические и электронные микроскопы. Указываются рабочие характеристики этих новых акриловых смол в холодном контрольном испытании сопротивления плотных пленок включающих конечные покрытия эмали, катализированных и осушенных при низкой температуре. Характеристики выветривания металлических и сплошных цветных эмалей сравниваются с свойствами алкидно-аминовых смольных систем.

Делается ссылка на возможные ограничения в формулировке термореактивных акриловых эмалей введением правила 'Лос Анжелес 66' относительно атмосферного загрязнения некоторыми органическими растворителями.

## **Introduction**

The general chemistry of thermosetting acrylic resins based on methylol acrylamide and hydroxylated acrylic monomers has been described previously in several papers<sup>1-5</sup> and therefore no detailed reference will be made to this in the present paper. Typical monomers which may be present in this type of resin are given in Table I. The acrylic resins discussed in this paper are mainly those containing pendant hydroxyl groups which may be crosslinked with etherified amino resins. Such acrylic resins are suitable for use in many types of industrial finishes, but have become established particularly in the automotive

Table 1

*Typical monomers which may be present in a thermosetting acrylic resin*

Monomer	Contribution
Methyl Methacrylate Styrene Vinyl Toluene Acrylonitrile	Hardness
Ethyl } Acrylates Butyl } and 2-Ethyl Hexyl } Methacrylates Butyl Maleate }	Flexibility
Acrylamide Butoxymethyl-Acrylamide Hydroxy Alkyl Acrylates Glycidyl Acrylates Acrylic Acid	Crosslinkages
Acrylic Acid Methacrylic Acid Maleic Anhydride	Cure acceleration

industry in USA, Great Britain and Europe. They are especially suitable for the formulation of metallic finishes, since the gloss, polishing and weathering properties are usually of a very high order. It is usually possible to re-establish the original gloss of an exposed thermosetting acrylic enamel by burnishing with a mop, and in this respect these finishes behave in a similar manner to nitrocellulose and thermoplastic acrylic lacquers. The thermosetting acrylic resins which form the subject of much of this paper may be thought of as a second generation which make it possible to formulate paints having reflow properties similar to those based on thermoplastic resins. Some of the problems associated with these resins and paints are discussed, since little information is at present available on this subject.

### **Curing mechanisms of hydroxylated acrylic resins**

The thermosetting acrylic resins now being used in automotive finishes are cured by the addition of a suitable melamine resin and stoved at temperatures between 120 and 150°C for approximately 30 minutes, or at lower temperatures (80-90°C) after the addition of an acid catalyst such as phthalic acid or butyl hydrogen maleate. It is believed that for optimum properties, including excellent outside durability, the crosslinked film of such an acrylic/melamine system is obtained when only a proportion of the total number of crosslinking sites have reacted. A study has been made of the rate of reaction of hydroxylated thermosetting acrylic resins stoved with butylated melamine resins, in order to ascertain the degree of crosslinking which occurs in reflow systems during:

(a) the initial low bake (70-100°C), and

(b) on final baking at normal stoving temperatures (120-150°C)

The degree of cure was followed by the determination of the amount of material extracted from the cured film by methyl ethyl ketone in a Soxhlet apparatus.

Typical results for a conventional hydroxy acrylic and a reflow hydroxy acrylic resin are given in Fig. 1. Resin B (see Table II) is typical of reflow resins specifically intended for use in systems where the initial bake may vary from 17 minutes at 70°C to 22 minutes at 100°C (oven temperature). It will be seen from the curing curves that the reflow resin B cured more slowly at 100°C than the conventional thermosetting acrylic resin. However, on stoving for 30 minutes the reflow resin gave satisfactory crosslinked systems with 80 and 95 per cent unextracted at 130° and 140°C respectively.

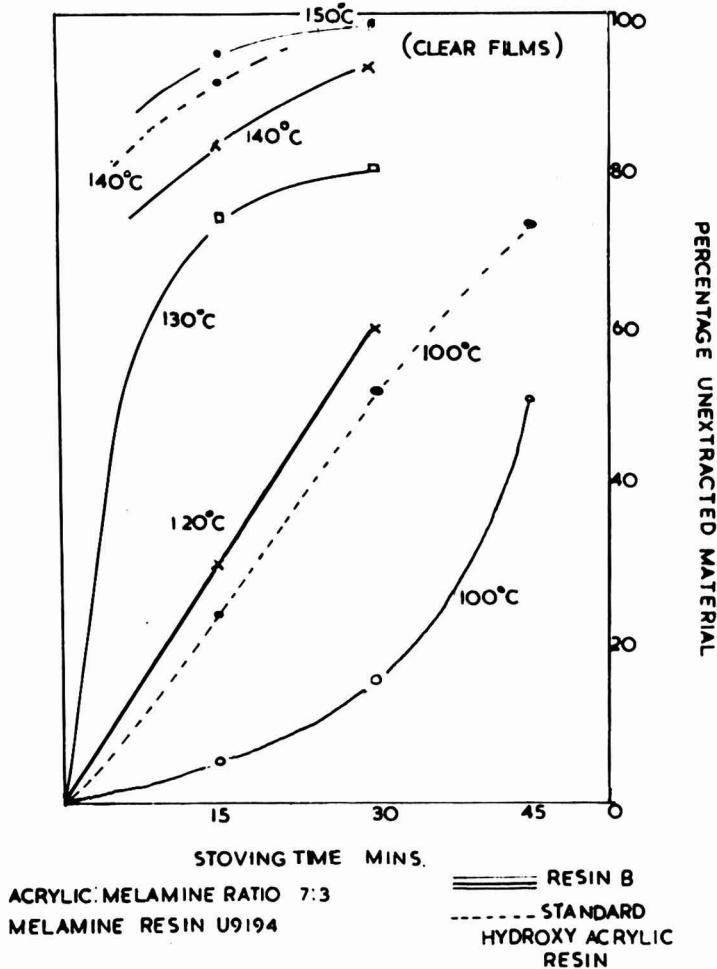


Fig. 1. Curing rates by solvent extraction technique—low and high temperature bakes

### Repair-in-process

Thermoplastic acrylic resins have been established for some time as suitable media in surface coating enamels in the automotive industry. One of their advantages has been the ability to reflow during the so-called repair-in-process techniques. Until recently it was assumed that thermosetting acrylic resins, because of their chemical nature, were not capable of being used in

such processes, since during the initial stoving period flow would be prevented by excessive crosslinking of the reacting acrylic and melamine resins. It is, however, interesting to note that certain British, American and European car manufacturers have been interested in the possibility of using thermosetting acrylic resins in this process, in order to reduce the number of cars that have to be returned to the finishing line because of surface defects.

Two repair-in-process systems have been evaluated by car manufacturers *viz.* (1) the bake-sand-bake and (2) the bake-sand-respray-bake techniques. In both of these the enamel coat is sprayed and given an initial bake at a low temperature in the range of 70-100°C, at which stage solvent evaporation has taken place with very little crosslinking so that the coating remains thermoplastic. The paint film must then be hard enough to permit defects to be sanded out with abrasive paper without excessive clogging. Subsequently the latter film is tack wiped and, in the bake-sand-bake process, immediately restoved at 140°C. During this final stoving the paint film must soften sufficiently to allow reflow to occur, thus obliterating the sanding marks. In the bake-sand-respray-bake process the sanded areas are tack wiped and resprayed with a second coat of the enamel without any masking of the remainder of the car body. The paint is then given a final stoving at 140°C for 12-15 minutes. During the final stoving period all dry spray from the respray operation must be completely accepted by the first coat of enamel so that both coats are virtually indistinguishable.

The requirements of a reflow resin are summarized schematically in Fig. 2

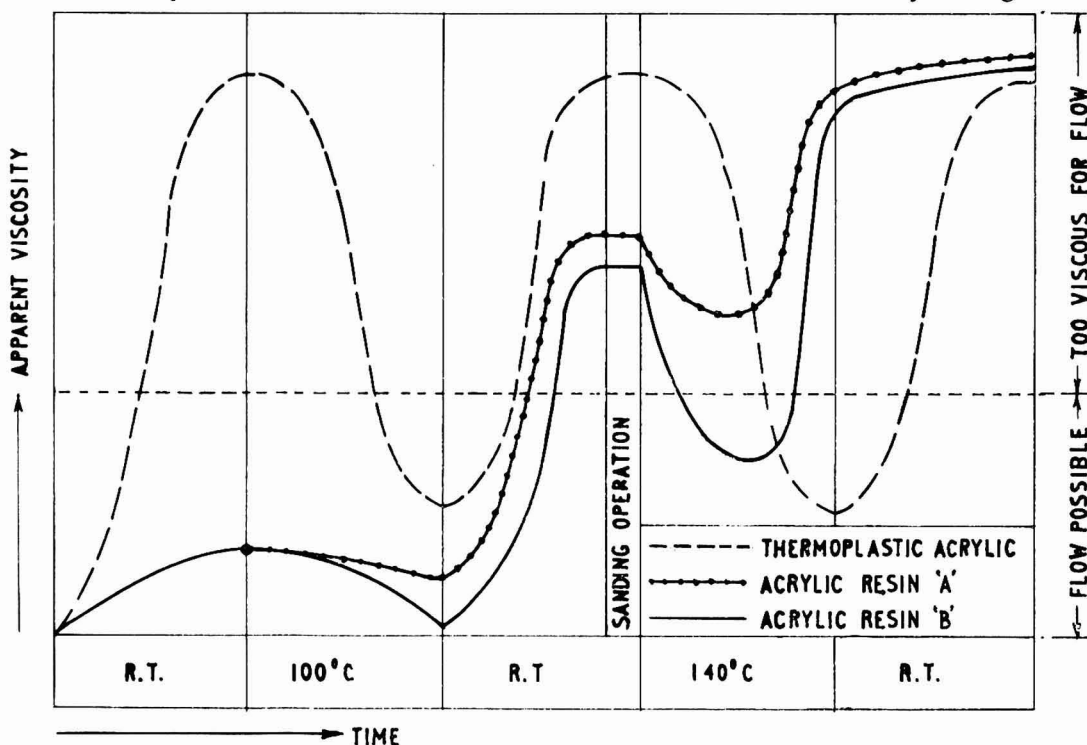


Fig. 2. Schematic diagram illustrating reflow of three types of acrylic resins

which illustrates the anticipated flow properties of three hypothetical resin systems:

- (a) A typical thermoplastic acrylic resin (TPA)
- (b) a typical thermosetting acrylic resin system (TSA)
- (c) a reflow thermosetting hydroxylated acrylic resin system (RTSA)

*Stage 1: Evaporation of solvent*

During this stage the TPA resin, because of its high molecular weight, should reach its maximum hardness, whereas the TSA and RTSA resins will still be comparatively soft at room temperature.

*Stage 2: Low temperature bake*

The TPA resin viscosity drops rapidly as the panel temperature increases.

Both TSA and RTSA are thermoplastic at this stage, but as the panel temperature increases, the crosslinking reaction commences, and the actual viscosity change is dependent upon both of these two properties.

RTSA resins are known to be slower curing at low temperatures than conventional TSA resins and therefore it is likely that the curves for these resins diverge as shown in the diagram.

*Stage 3: Removal from low temperature oven*

As the panels cool down to room temperature they become hard enough to sand. The TPA recovers its original solvent-free hardness.

*Stage 4: Final high temperature bake*

The initial stage of the final bake is critical. Both the TSA and the RTSA resins will flow to different extents, but in the case of the TSA resin the crosslinking reaction will take place too rapidly for reflow to occur, whereas the RTSA resin will have sufficient time to reflow before it becomes crosslinked.

*Stage 5: Removal from high temperature oven*

All three types of resin should attain a similar "viscosity" (hardness) after cooling to room temperature.

It will be seen that the important reduction in viscosity is that which occurs on the rebake after the sanding period, and attempts have been made to record viscosity changes in this region using the Weissenberg Rheogoniometer. Further work is now in progress on this subject.

**Method of test for reflow properties**

Fig. 3 outlines a test which was developed to assess sandability, reflow and overspray acceptance using a single panel. A 12in x 6in mild steel panel is given one coat of reflow enamel and then stoved at the specified initial low temperature bake, after which the right hand side of the panel is sanded with 400 grade silicon carbide paper. The bottom three inches of the panel are then masked off and the lower exposed area of the panel is given a second coat of reflow enamel such that the overspray from this second coat forms a strip about half-way up the exposed area. The mask is then removed and the panel is given a final bake in the region of 15 minutes at 140°C. Examination of the panel



can then be made visually but instrumental methods have been found to be generally more satisfactory.

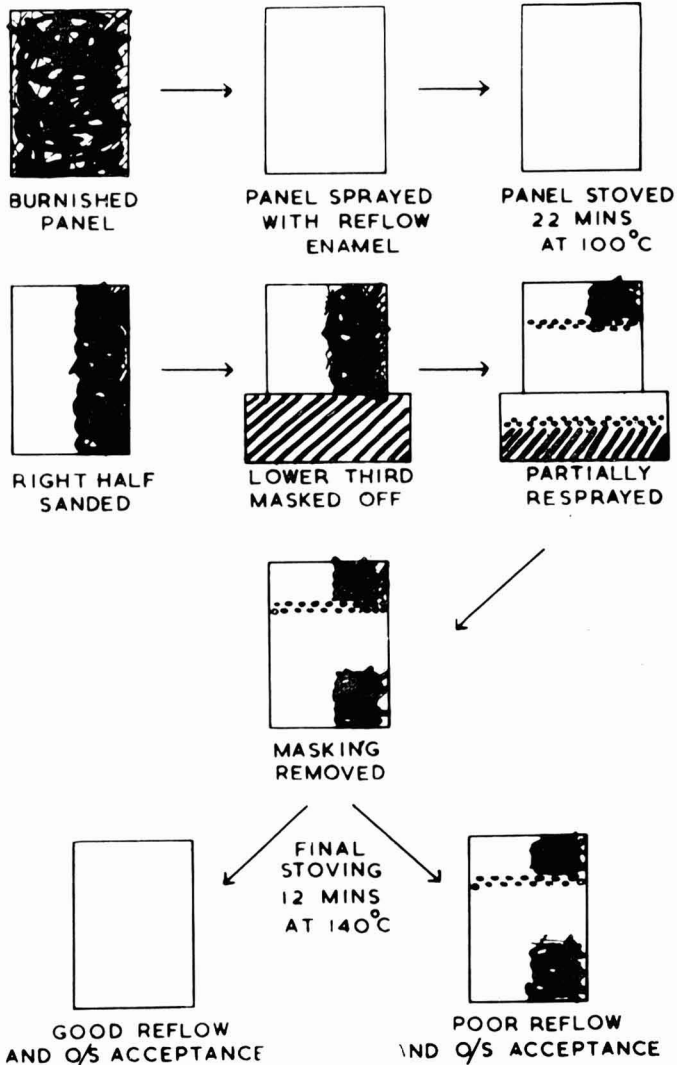


Fig. 3. BP method of test for reflow acrylic resins

**Practical automotive specifications for reflow thermosetting acrylic resins**

The Ford specification ESB-M32J-102A was released in July 1967 and for the first time a standard of reflow performance was indicated to the resin and paint manufacturer. Initial low bake schedule was fixed at 15 minutes at temperatures ranging from 71-99°C (160-210°F) and in practice this meant that the enamel must be hard enough to sand after stoving for 15 minutes at 71°C but sufficiently thermoplastic to reflow and accept overspray after stoving for 15 minutes at 99°C. In addition the enamel was required to perform equally well after allowing 72 hours to elapse between the initial bake and the final sanding operation. Table 2 shows the performance limits required of reflow thermosetting acrylic

Table 2  
Physical constants of two reflow acrylic resins

	Type of resin	Solids content %	Viscosity at 25°C poises	Acid value	Solvents
Resin 'A'	Hydroxylated thermosetting acrylic	60%	approx. 15	approx. 12	1:1 Xylol: n.Butanol
Resin 'B'	„	„	22-28	2.5-4.0	„

	Performance limits					
	limits of low bake	final high temp. bake	sanding lubricant	grade of sandpaper	sanding performance	reflow and o/spray acceptance
Resin 'A'	15 min at 80-90°C	30 min at 120°C	none	600	easy dry sanding after initial bake of 15min at 80°C	good after 15min at 90°C
Resin 'B'	15min at 70-100°C	12min at 140°C	mineral seal oil	400	easy wet sanding after initial bake of 15min at 70°C	good after 15min at 100°C

resins and the physical constants of two such resins A and B. Type A represents a typical hydroxylated acrylic resin which when crosslinked with a melamine resin will only reflow after a comparatively low initial bake, and resin B represents a reflow thermosetting acrylic resin which was designed to pass the Ford Reflow Specification ESB-M32J-102A. Both resins have fairly high solids contents of about 60 per cent and viscosities of 15-30 poise at 25°C.

A white enamel based on Resin A reflowed satisfactorily after an initial low temperature bake of 15-30 minutes at 80°C or 5-10 minutes at 90°C (oven temperatures) but was too highly crosslinked to reflow after an initial bake of 15 minutes at 100°C (panel temperature).

Resin B reflowed satisfactorily after stoving for 15 minutes at 100°C (panel temperature) as a clear film and when pigmented, although it was found that both acrylic/melamine ratio and the pigmentation had a profound effect on its reflow properties.

### Sandability

Assessment of dry sandability of a film after the initial low bake presented some difficulties, especially in a series of enamels, since the results could not be duplicated with the degree of accuracy required, especially when comparing results between two operators. An attempt was therefore made to correlate sandability with film hardness as measured by (a) the pencil method, and (b) the Vickers Diamond Indentation apparatus. Fig. 4 shows the relationship obtained with both thin and thick paint films of 0.001 and 0.002in dry film thickness respectively.

CORRELATION OF DRY SANDABILITY WITH HARDNESS AND FILM THICKNESS AFTER INITIAL LOW BAKE—RESIN A

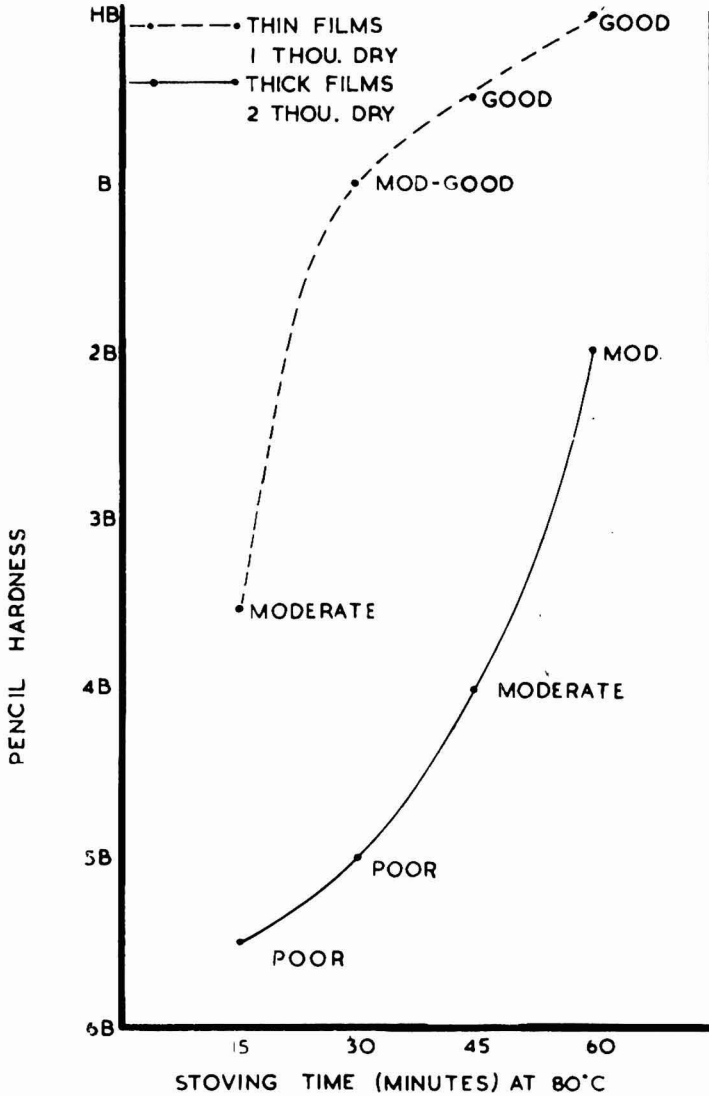


Fig. 4. Correlation between dry sandability, pencil hardness and film thickness of resin A

The remarks at each point on the diagram refer to the dry sandability which was the mean of three assessments, each given by four operators. It was tentatively concluded that a minimum pencil hardness of B was required to give acceptable dry sanding properties. A similar type of graph was obtained with the Vickers Diamond Indentation Hardness figures replacing the pencil hardness

data, but these were rejected since the films were so soft that the depth of indentation obtained with the lowest possible load of 15gm was of the same order as the dry film thickness. In such cases the hardness of the substrate would be expected to have a direct influence on the results obtained. Van Laar<sup>6</sup> has suggested that the depth of penetration of the diamond should not exceed one-tenth of the total film thickness. The wet sanding technique using a mineral seal oil required by the new Ford specification reduced the problem of sandability, even at low pigment loadings.

### Reflow and overspray acceptance

The reflow and overspray acceptance properties deteriorated with an increase in the initial low temperature baking schedule as would be expected, and normally good reflow was accompanied by good overspray acceptance and vice versa. A visual method of assessing reflow and overspray was used but found to be unacceptable for accurate assessment of trends in a series of experimental enamels. A number of other methods were used, such as gloss and surface texture measurements. The surface texture or roughness was assessed by means of a Talysurf instrument. Various types were available, the two most popular being the comprehensive model which draws a replica of the surface under test, and a simpler type of instrument which gives a numerical value of the roughness of the film in terms of the Centre Line Average Height (C.L.A. height). This type of measurement is illustrated in Fig. 5.

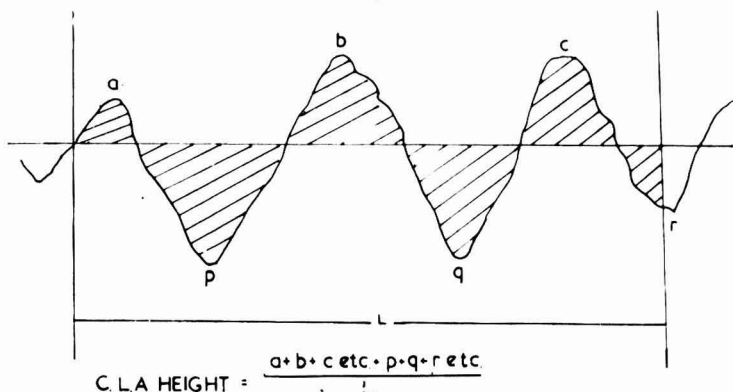


Fig. 5. Definition of C.L.A. height measurements by Talysurf

The C.L.A. height is defined as the sum of the areas above and below a centre line drawn through the surface profile, divided by the sampling length. This was recorded in micro-inches and it can be seen from the equation that a smooth surface gave a low C.L.A. height reading whereas a rough surface gave a high reading.

The instrument had an electrical integrating component which produced a direct reading in terms of C.L.A. height.

Figs. 6 and 7 are reproductions of surface textures at various stages of a bake-sand-respray-bake process, obtained from a comprehensive model Talysurf instrument.

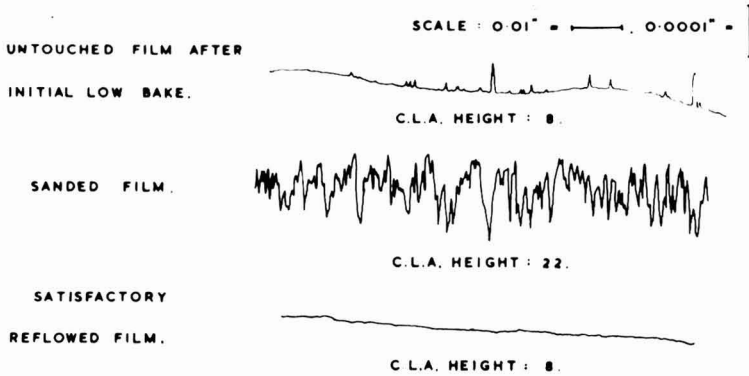


Fig. 6

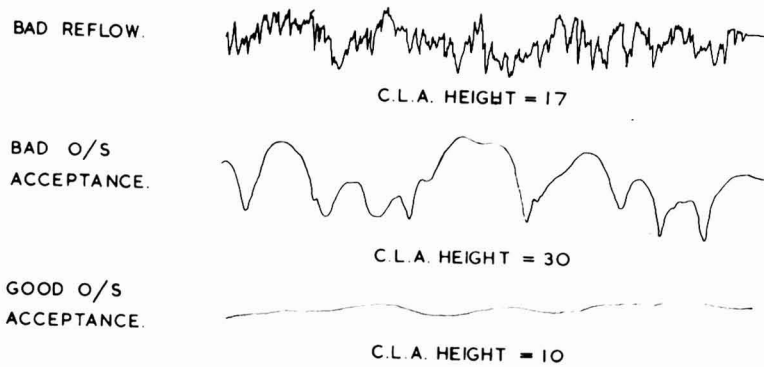


Fig. 7

Fig. 6 and Fig. 7. Talysurf traces of surface texture during a bake-sand-respray-bake process

The untouched film was one which contained a number of small surface defects, as shown in the diagram. After sanding and subsequent reflow these defects were virtually eliminated in one case, although the C.L.A. height returned to its original value of 8 micro-inches. In the other case illustrated, very little reflow occurred and the C.L.A. height of the sanded film only fell from 32 to 17 micro-inches.

Good and bad overspray acceptance could also be assessed by means of C.L.A. height measurements, as shown. An approximate method of predicting reflow in certain cases was by measurement of film hardness prior to reflow. Relationship between hardness, texture and gloss of the film during a reflow process is shown in Fig. 8, which indicates that a loss of gloss due to restricted reflow occurred in the particular case quoted when the stoving time of 22 minutes at 100°C was exceeded.

Three properties of reflow enamel films are illustrated by the graph:—

- (1) The variation in C.L.A. height of the fully cured surface of the films after different initial low temperature baking schedules varying from 10 to 60 minutes at 100°C followed by sanding with 400 grade silicon

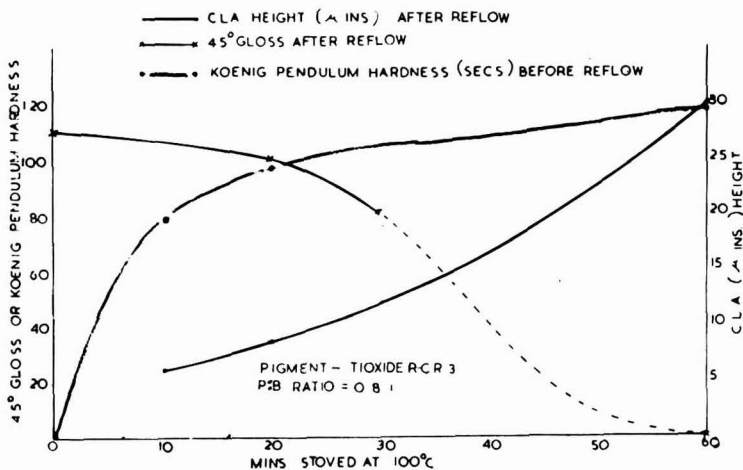


Fig. 8. Relationship between gloss, C.L.A. heights and hardness

carbide paper. The C.L.A. height increased steadily with stoving time until a value of 31 micro-inches was reached after initially stoving for 60 minutes at 100°C. This was of the same order as that of a sanded paint film.

- (2) The corresponding gloss level of the fully cured film was also dependent upon the initial low temperature bake prior to sanding. As expected, the gloss of the reflowed area dropped rapidly as the initial low bake schedule was increased, confirming little or no flow of sanding marks after an initial bake of 60 minutes at 100°C. This is confirmed by the apparent degree of crosslinking obtained in Fig. 1 when a reflow resin (B) was stoved at 100°C—after 45 minutes, 52 per cent of the film was unextracted by MEK.
- (3) Koenig pendulum hardness values of films after the initial low temperature bake increased with increasing stoving time at 100°C, but levelled out after a stoving schedule of approximately 20 minutes at 100°C was exceeded. For this reason it was difficult to affix a “critical” hardness figure for predicting satisfactory flow during the final high temperature bake.

### Choice of acrylic resin

The type of thermosetting acrylic resin suitable for reflow is an important factor. Most of the original hydroxylated acrylic resins tended to give cross-linked systems too rapidly at the temperature required for initial stoving and consequently did not permit reflow or over-spray acceptance after the initial low temperature bake of 17 minutes at 100°C. The photomicrographs shown in Fig. 9 indicate the importance of choosing a suitable acrylic resin which will permit acceptance of overspray from the second coat.

Overspray particles of yellow paint over a partially cured standard hydroxylated acrylic enamel are seen to remain on the surface of the film after the second high temperature bake, whereas similar overspray particles over a reflow hydroxylated acrylic enamel have been accepted by the still thermoplastic first

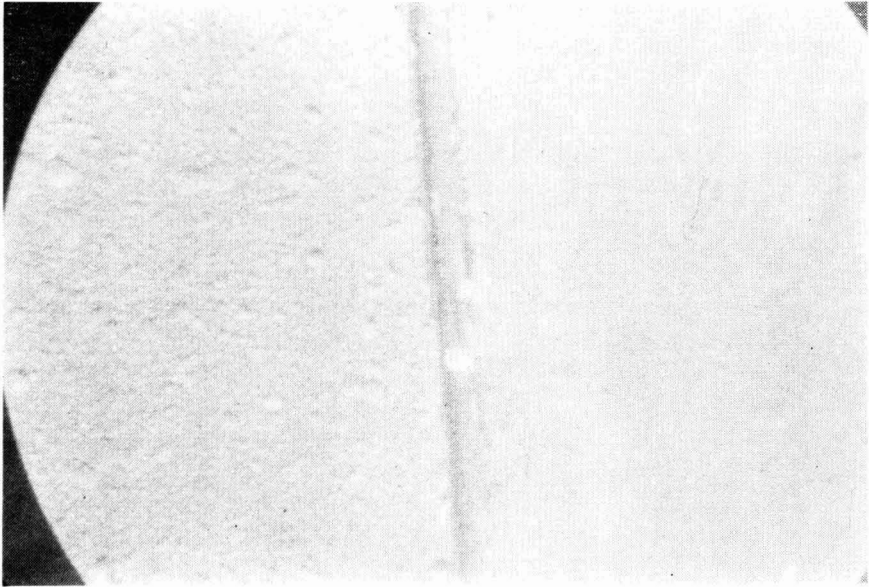


Fig. 9. Photomicrograph of two resprayed areas (yellow on blue)

coat, leaving a smooth surface. In practice, of course, both first and second coats would be of the same colour.

Fig. 10 shows a practical way of producing overspray similar to that obtained when the side of a car is resprayed and overspray falls on to the roof section; good and poor overspray acceptance are seen by the blurred images of the fluorescent strip light reflected by the horizontal parts of the test panels after the vertical faces were given a second coat of enamel.

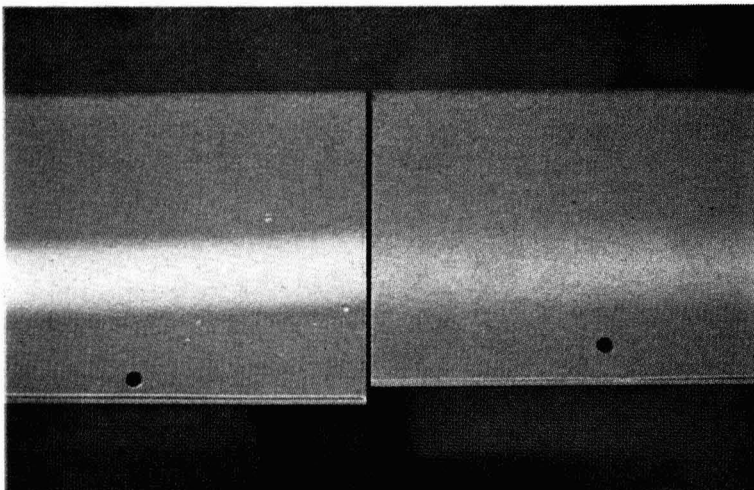


Fig. 10. Practical method of producing overspray (bent panel method)

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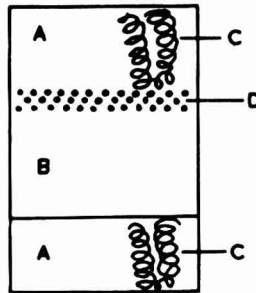
**Effect of type of melamine resin on reflow properties**

During the development work of the reflow thermosetting acrylic resins a large number of melamine resins were screened and only two were finally selected as being suitable for incorporation into reflow enamels.

Table 3 illustrates the importance of selecting the right melamine resin for crosslinking. The surface textures after reflow are given in terms of Centre Line

*Table 3*  
*Effect of melamine resin on reflow properties*

Area of Test	CLA heights of surfaces		
	Ac:Mel 1 7:3	Ac:Mel 2 7:3	Ac:Mel 3 7:3
Original film (A)	4.0	4.0	4.5
Resprayed area (B)	2.5	3.5	4.0
Reflowed area (C)	9.0	6.0	7.5
Area of overspray (D)	36	5.0	10
Sandability after initial bake	Excellent	Excellent	V. Good



Average Height readings obtained from a Talysurf Instrument. It can be seen from the table that melamine resin 2, a butylated specially modified melamine resin, gave the lowest C.L.A. height values over the areas C & D, indicating good reflow and overspray acceptance.

Melamine resin 3 gave moderately good results in this test but the resulting films were more difficult to sand.

**Acrylic : melamine ratio**

The recommended acrylic:melamine ratio for hydroxylated acrylic resins is often 70:30 based on solid resins. This gives the optimum balance between hardness, flexibility and chemical resistance. The effect of varying this ratio was investigated and the results are seen in Table 4. The initial bake was at 90°C for 15 minutes and this was chosen because it approached the limit of resin A under test. The results indicate that reflow and overspray acceptance were



Table 4

*Effect of A:M ratio on reflow of resin A*

Initial low bake of 15min at 90°C

P:B ratio=8:10

A:M ratio	O/S acceptance	Reflow	Sandability
6:4	Very good	Very good	Very good
7:3	Very good	Very good	Excellent
8:2	Moderate-good	Good	Excellent

favoured by a low acrylic:melamine ratio and that sandability was favoured by a high acrylic:melamine ratio. It was usually possible to obtain satisfactory sanding properties without clogging the sandpaper during the rubbing down process.

A comparison was made between a conventional hydroxy acrylic resin such as Epok D2102 and the reflow resin B. The acrylic:melamine ratios were varied between 60:40 and 80:20 as seen in Figs. 11 and 12. The initial bake in all cases was 22 minutes at 100°C (oven temperature). From the curing curves obtained with the reflow resin B, initial thermoplasticity, i.e. slow curing rate, was obtained with the low acrylic:melamine ratio, while the high acrylic:melamine ratio gave a much faster initial cure. The hardest ultimate film was obtained at the recommended ratio of 70:30.

Similar observations may be made in the case of Epok D2102, but it was obvious that more crosslinking had taken place in each case after the initial bake of 22 minutes at 100°C. There was no great difference in film hardness between resin B and D2102 at the 70:30 ratio after a final bake of, e.g., 20 minutes at 140°C.

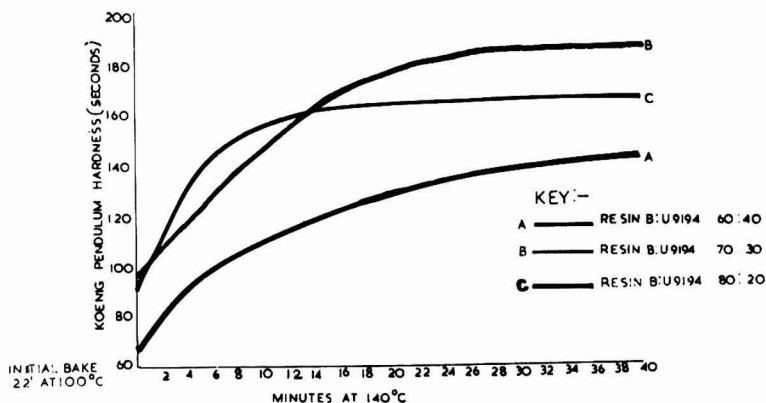


Fig. 11. Effect on pendulum hardness of varying the A:M ratio with resin B

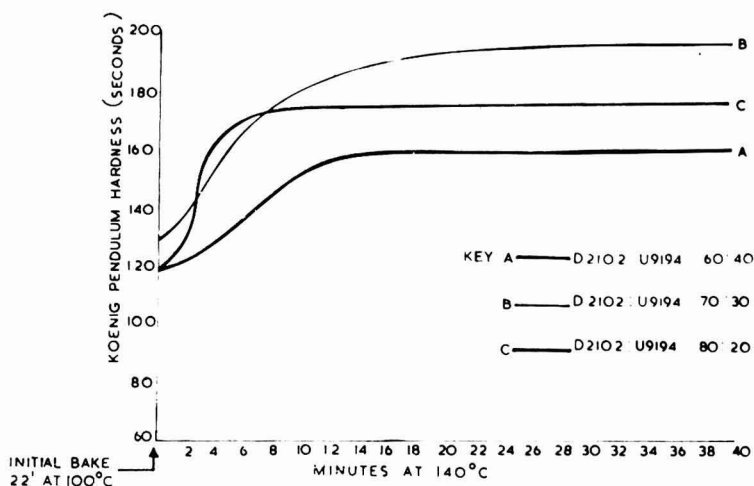


Fig. 12. Effect on pendulum hardness of varying the A:M ratio with D2102

### Effect of final stoving temperature

The reflow and overspray acceptance properties of resin A were examined when the temperature of the final bake was varied. The difference in reflow and overspray acceptance was only slight when the temperature of final stoving was raised from 120 to 150°C (See Table 5). The result indicated that, contrary to what might at first be expected, the reflow properties were favoured by a higher final stoving temperature, suggesting that the partly stoved resin remains thermoplastic during the initial stages of the high temperature bake, and that the initial rate of the crosslinking mechanism was comparatively slow.

Table 5

Resin A. Effect of final stoving temperature on reflow properties

Bake		O/S acceptance	Reflow	Sandability
1. 15min at 80°C + 30min at 120°C	..	Very Good	Excellent	Good
2. 15min at 80°C + 30min at 150°C	..	Excellent	Excellent	Good
3. 15min at 90°C + 30min at 120°C	..	Very Good	Very Good	Excellent
4. 15min at 90°C + 30min at 150°C	..	Very Good	Excellent	Excellent

Pigment—Tioxide R-C R 3  
 Pigment:Binder Ratio—8:10

### Factors affecting reflow properties

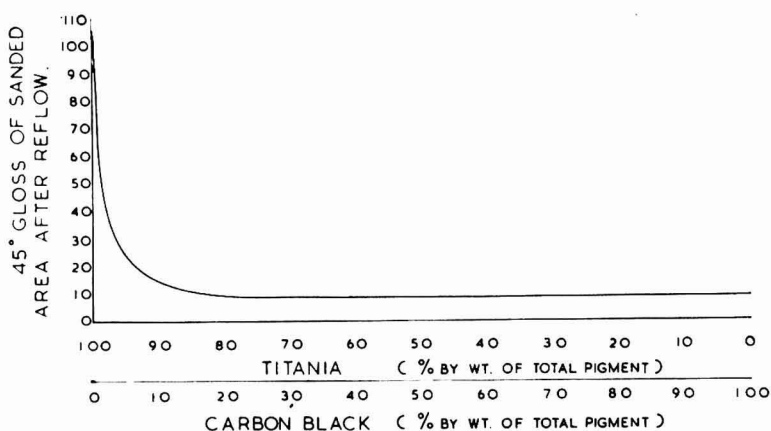
#### Pigment wetting

The reflow characteristics of a resin can be affected not only by its molecular weight and reactivity, but by its wetting properties with respect to a particular pigment. Miranda<sup>7</sup> has stated that Levis & Spencer in unpublished work found that no unusual problems are associated with pigment dispersion in systems based on acrylic resins containing acid and hydroxyl groups, but, in the case of a

reflow process, pigment wetting can, in fact, play an important role in the performance of the paint film.

### *Choice of pigment*

It was found that pigments which were difficult to disperse, such as carbon blacks, some phthalocyanine blues, and even certain grades of titanium dioxide, had an adverse effect on the reflow properties of enamels containing these pigments. Seven grades of titanium dioxide pigments were examined, and of these only four gave satisfactory reflow properties, when used at a pigment: binder ratio of 0.8:1. The grades which allowed good reflow and gave satisfactory uniform films were those recommended by the manufacturers as easily dispersible types, and these gave higher gloss readings than those which did not give good reflow properties. The white enamels which gave satisfactory flow were then tinted pale grey with a carbon black tinter based on Degussa FW200. This carbon black which, it will be understood, is not normally recommended as a tinting pigment, had given very satisfactory results in jet black acrylic enamels. Test results showed that the presence of this carbon black predispersed in resin B had an adverse effect on the reflow properties of the grey enamels. An indication of the level of this particular carbon black which could be incorporated in grey enamels based on satisfactory rutile titanium dioxide without impairing the reflow properties is given in Fig. 13.



**Fig. 13. Effect of carbon black tinter on reflow of enamel based on  $\text{TiO}_2$**

The graph indicates that reflow is impaired by the addition of a very small proportion of carbon black. The actual grade of carbon black used in this work was one of the oxidised high colour channel blacks, normally used in deep shades. In practice a coarser grade would normally be used for tinting white enamels. In the case of this particular carbon black, as little as 3 per cent replacement of titanium dioxide resulted in serious loss of reflow, as seen by the gloss of the sanded area of the films after the reflow bake.

Fig. 14 shows the effect on reflow properties of the grade of titanium dioxide used in grey enamels, each tinted with identical quantities of carbon black.

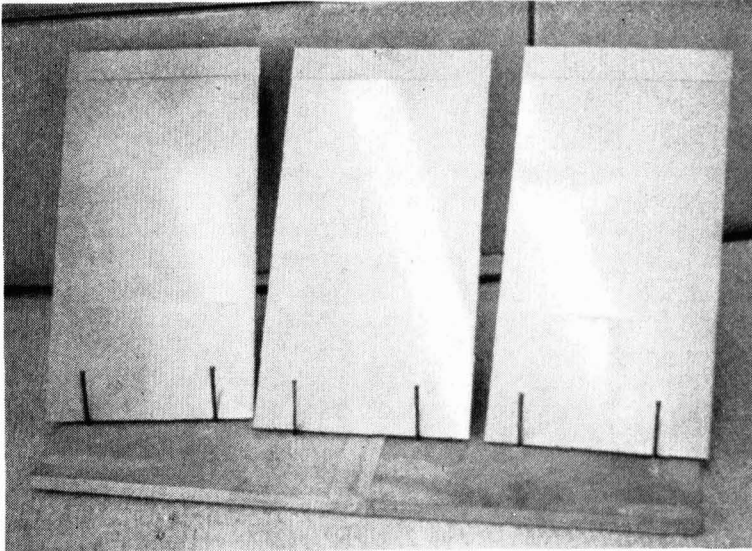


Fig. 14. Effect on reflow properties of altering the grade of titanium dioxide in grey enamels

The three enamels shown were based on (from l. to r.) Tioxide R-CR, R-CR2 and R-CR3, and were each tested according to the procedure outlined in Fig. 3. The reflections of the fluorescent strip lighting indicate the degree of reflow and overspray acceptance obtained.

#### *Pigment to binder ratio*

Variations in the pigment to binder ratio of white enamels based on titanium dioxide were examined with resin A between 0.6:1 and 1:1 (Table 6). It will be seen that at the lower pigment to binder ratios overspray acceptance is enhanced but sandability is reduced. This relationship was later confirmed by other reflow resins over a wider range of pigment to binder ratios including clear films which reflowed so well that it was almost impossible to identify the reflowed and oversprayed areas.

Table 6

*Resin A. Effect of P:B ratio on reflow properties*

Initial low bake of 15min at 90°C A:M ratio 7:3			
P:B ratio	O/S acceptance	Reflow	Sandability
6:10	Excellent	Excellent	Good
8:10	Very Good	Very Good	Excellent
10:10	Mod.-Good	Very Good	Excellent

Fig. 15 shows the results obtained using clear resins.

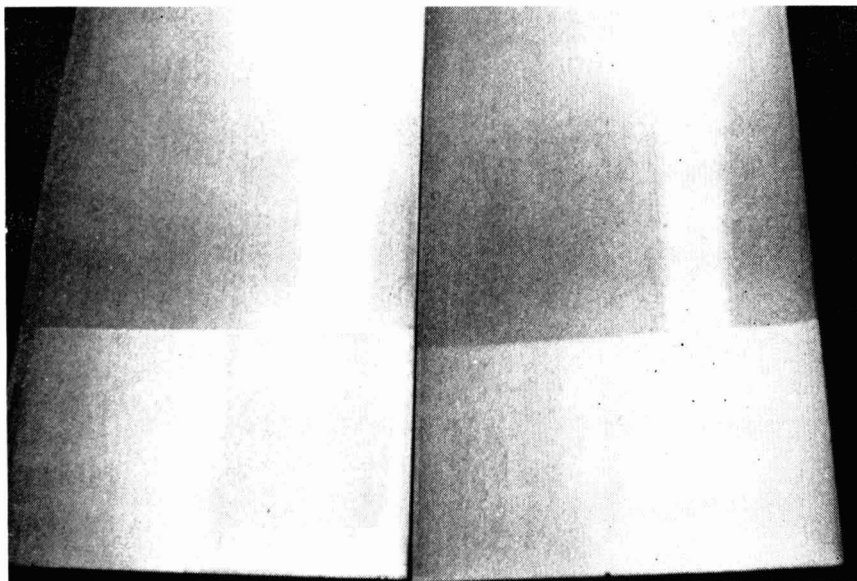


Fig. 15. Clear films of Epok D2105 and resin B

The left-hand panel was coated with a standard hydroxylated acrylic resin system and little or no reflow of the sanded area was obtained. The right-hand panel was coated with a resin B system which reflowed satisfactorily under the same curing conditions. The apparent differences in colour of the masked and unmasked areas was due to the different thicknesses of the clear films.

### *Pigmentation*

It has been seen that certain pigments can restrict the flow properties of a reflow resin. In the case of colours formed from a mixture of various pigments, a colour change might be expected after sanding and reflow in cases where one or more of these pigments were prone to flooding or where flotation occurs. Blue tints from white enamels based on Tioxide RCR6 produced slightly darker reflowed areas than the untouched areas, suggesting that flooding of the white pigment had occurred.

Another well known phenomenon associated with pigmented systems is that of flocculation. Two pastel enamels based on rutile titanium dioxide with (a) a flocculation resistant phthalocyanine blue Chromophtal 4G and (b) a standard grade Chromophtal GF were both given a low temperature bake and then sanded and reflowed. The latter pigment gave some darkening of colour after sanding and subsequent reflow. It would be expected that the shear forces acting during reflow were too small to affect the flocculated state of pigment in the film, and therefore the darker colour of the reflowed area was attributed to the removal of white flood during sanding. It is assumed that the colour change would be reversed if flooding of the blue pigment were to occur. It is interesting to note that the reflow of the unflocculated film was better than

that of the flocculated material, and less colour change was also observed. Photomicrographs of these films before and after sanding and after reflow are shown in Figs. 16-21.

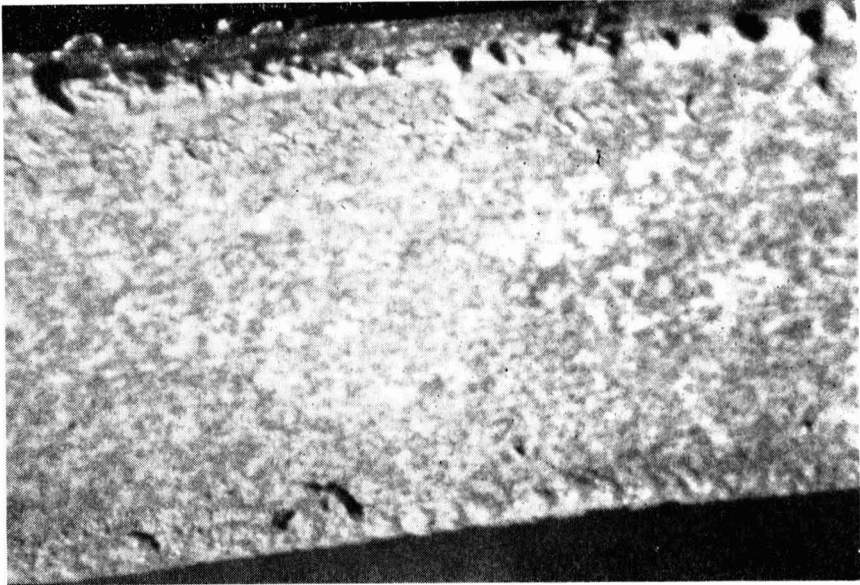


Fig. 16

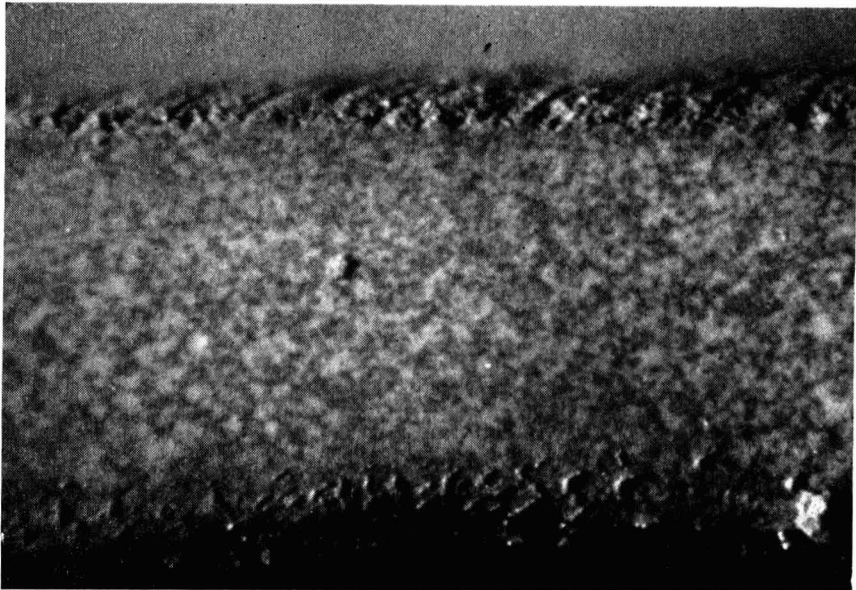


Fig. 17

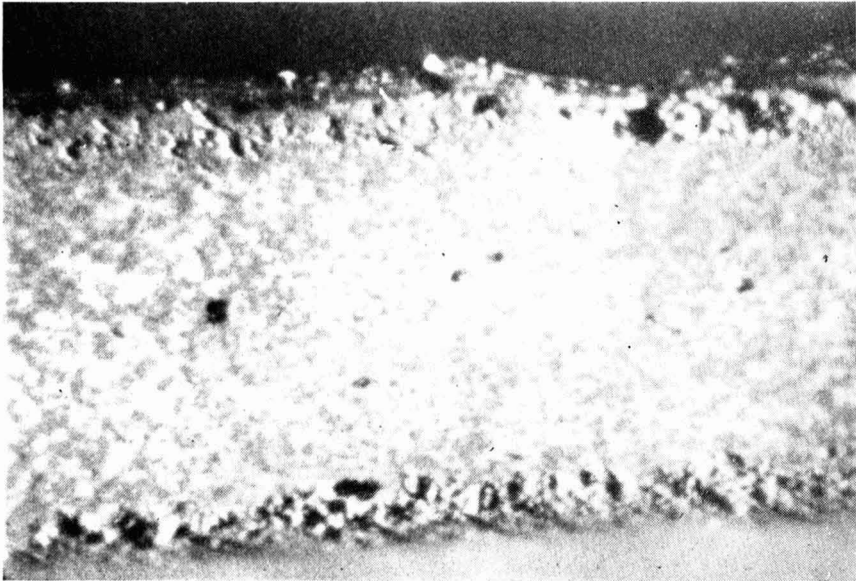


Fig. 18

Fig. 16, Fig. 17 and Fig. 18. Micrographs of cross sections of blue enamel films. (Pigment flocculated)

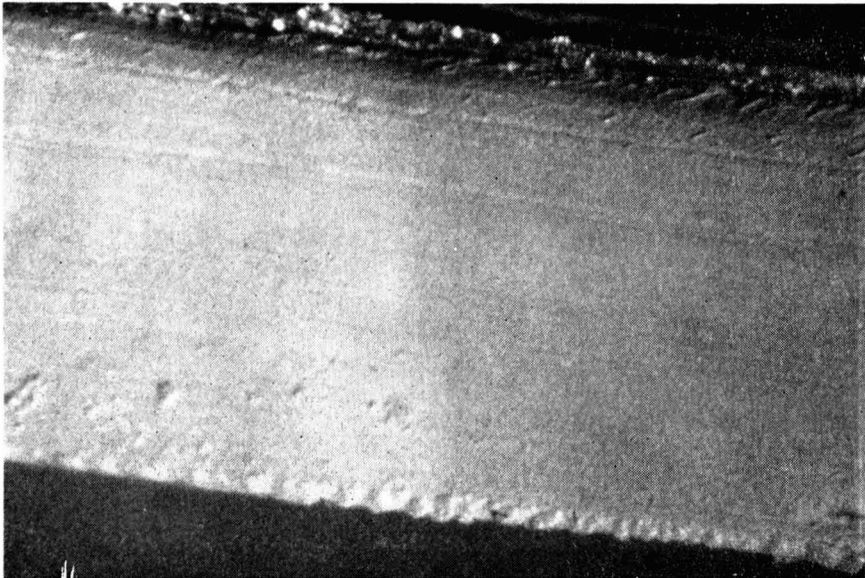


Fig. 19

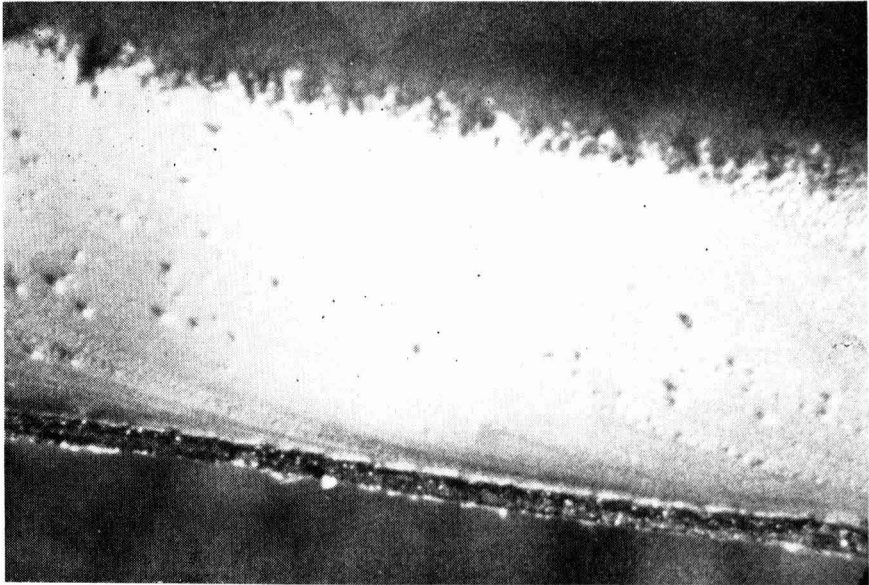


Fig. 20

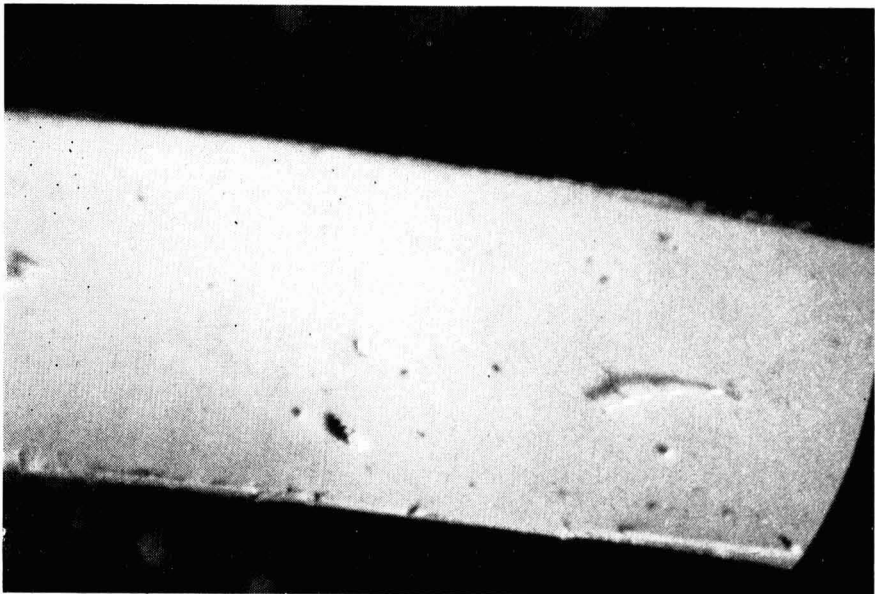


Fig. 21

Fig. 19, Fig. 20 and Fig. 21. Micrographs of cross sections of blue enamel films. (Pigment unflocculated)



Figs. 16, 17 and 18 are cross-sectional micro photographs of flocculated pale blue enamels after initial bake, after sanding, and after final bake, respectively. Figs. 19, 20 and 21 are similar photographs obtained with a non-flocculated finish. Comparing Figs. 18 and 21, it can be seen that the sanding marks from the flocculated system remained in the film after the final bake, whereas they flowed out and were no longer visible in the unflocculated system.

It has been shown that certain grades of titanium dioxide had an adverse effect on the reflow properties of enamels. Therefore it was necessary to investigate methods of improving reflow by the addition of various additives. For example, 2 per cent of triethanolamine (TEA) based on the pigment considerably improved the reflow properties of an unsatisfactory enamel. Three possible reasons for the improvement are suggested.

- (1) The addition alters the dispersed state of the pigment and thus improves the flow of the enamel.
- (2) It acts as a plasticising solvent (boiling point 360°C).
- (3) A salt is formed with carboxyl groups from the acrylic resin thus retarding the cure.

Table 7 indicates the effect of three additives on the reflow properties of a system which initially gave poor reflow.

Table 7

*Effect of certain additives on reflow properties of the enamel based on a flocculated pigment*

Additive	B.Pt. of additive	Koenig hardness (secs)		45° gloss of reflowed area after final bake	CLA height of reflowed area after final bake	Visual assessment of reflow
		After initial bake	After final bake			
None	—	107	142	25	16	Very poor
TEA	360°C	98	146	94	8	Good
DBP	340°C	98	148	46	15	Poor-mod
Pine oil	198-222°C	97	142	37	13	Poor

The reflow properties of an enamel based on a flocculated pigment were greatly improved by the addition of 2 per cent triethanolamine (based on the pigment) to the finished enamel. The use of an equivalent quantity of plasticiser having approximately the same boiling point (dibutyl phthalate) had some effect, but it appeared from the results that the specific pigment wetting properties of the triethanolamine played a significant part in the increased flow obtained. It was interesting to note that the films were not softened by the use of these additives.

### **Metallic finishes**

The increasing use in automotive enamels of metallic finishes is well known and is possibly the reason for the widespread acceptance of hydroxylated acrylic finishes which technically provide an ideal carrier for non-leaving grades of aluminium pigments. Metallic enamels based on acrylic resins possess excellent gloss, colour retention, resistance to acid spotting, and good durability. In these respects acrylic:melamine systems are usually more acceptable than alkyd:melamine in metallic finishes.

### **Reflow metallic enamels**

The effect of addition of aluminium pigments to a reflow acrylic resin system was examined. Particular note was made of the position of the aluminium flakes in the film before and after reflow. It was quite evident that if the pigment particles were not uniformly dispersed throughout the thickness of the film colour changes would take place during reflow. Electron photomicrographs were taken of cross sections of these metallic films in various stages of a bake-sand-bake process as shown in Figs. 22-24.



**Fig. 22**



Fig. 23

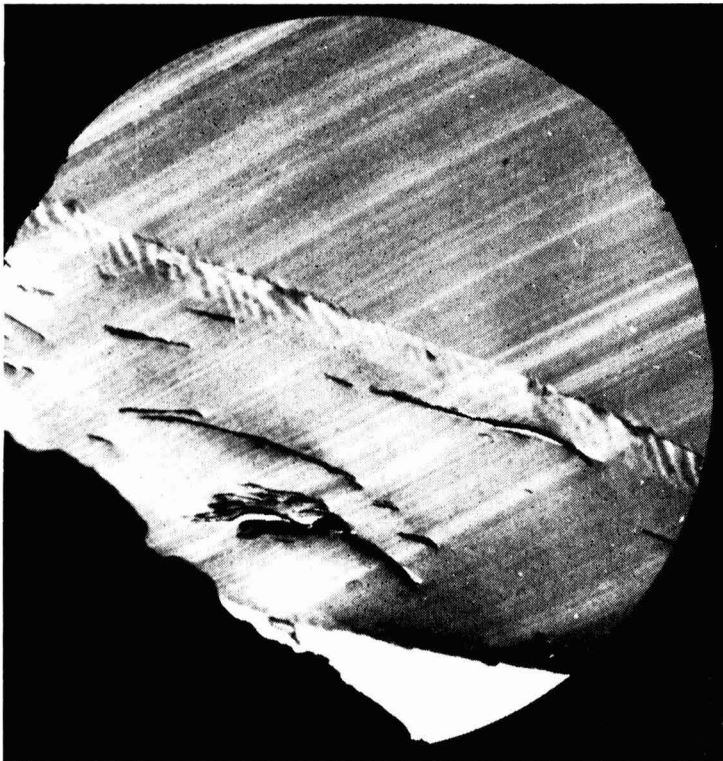


Fig. 24

Fig. 22, Fig. 23. and Fig. 24. Electron micrographs of cross sections of metallic film

### Factors affecting cold check resistance

It has been found in practice that cracking can occur in thick films of both alkyd/amino and acrylic/amino motor body enamels when large variations in temperature and humidity are experienced. It is believed that repair-in-process will significantly reduce the number of defects in the finish of the body shell during application of the paint, but in the case of damage occurring during the fitting of the trim and engine it is necessary to have the means of repairing this by the application of the same paint suitably catalysed with an acid in order to stove by infra-red at a reduced temperature, e.g. 80°C. It was thought possible that the change in resin composition necessary to obtain adequate repair-in-process properties might have a deleterious effect on the acid catalysed system. During this type of repair, films of a total thickness of between 0.007 and 0.01in were occasionally found in localised areas. It was found that the degree of crosslinking of the resin systems had a profound effect on the performance of thick films of enamels subjected to cycles of humidity in extreme variations of temperature. The Ford Motor Co. have included a cold check requirement in some of their specifications, and certain parameters affecting performance of acrylic enamels during this test were therefore examined. The enamels used in this work contained titanium dioxide at a pigment:binder ratio of 0.8:1 and were based on hydroxylated acrylic and butylated amino resins as vehicles. The thick films having an acid catalysed final coat were subjected to cycles as shown in Fig. 25.

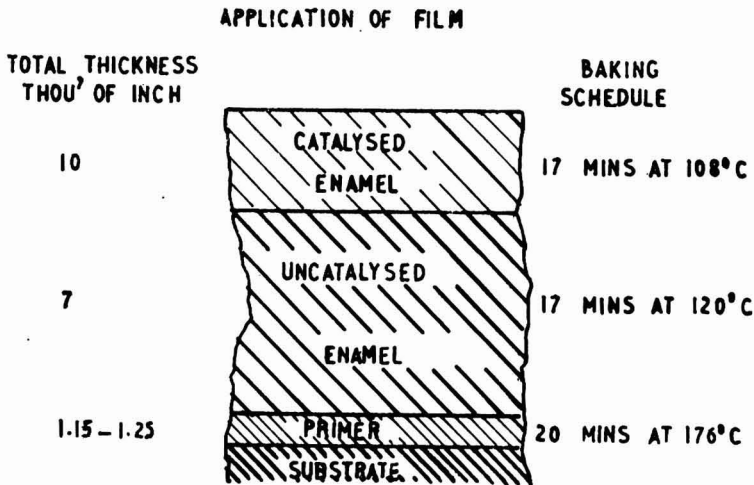


Fig. 25. Ford cold check test

The following conclusions were drawn from these tests.

- (1) The substrate played a part in the resistance to cold checking, owing probably to such factors as adhesion, coefficient of expansion of both substrate and film, and to differences in cohesion and coefficient of expansion of different layers in the paint film. This conclusion seems to be confirmed by the fact that the free enamel film and a film on an aluminium substrate gave satisfactory performance after 17 cycles whilst a

film applied directly to plate glass checked badly after one cycle. Application of the enamels to phosphated steel panels without the use of a primer gave breakdown after four cycles, and blistering occurred during the humidity cycle.

- (2) The number of coats of enamels required to achieve the specified 0.007in film thickness of the fully stoved film did not significantly affect the performance in this test.
- (3) It was significant that the replacement of the acid catalysed coats by standard material with corresponding increase in stoving temperature reduced the resistance to cracking of the stoved film, contrary to what might be expected. In addition, it was noted that slightly undercured films failed the test more rapidly than films which were given a full bake after each coat. Contrary to expectations the film allowed to partially dry out in the laboratory after the humidity cycle failed more rapidly than one which was transferred immediately from the humidity cabinet to the refrigerator as specified in the cold check test.

The results show that this procedure produced failure in eight cycles. Fig. 26 illustrates this failure and that of another film on plate glass.

- (4) The change in hardness of the films during these tests was interesting since results indicated that films increased in hardness, as measured by the Vickers Diamond Pyramid Hardness tester (Fig. 27), during the cycling.

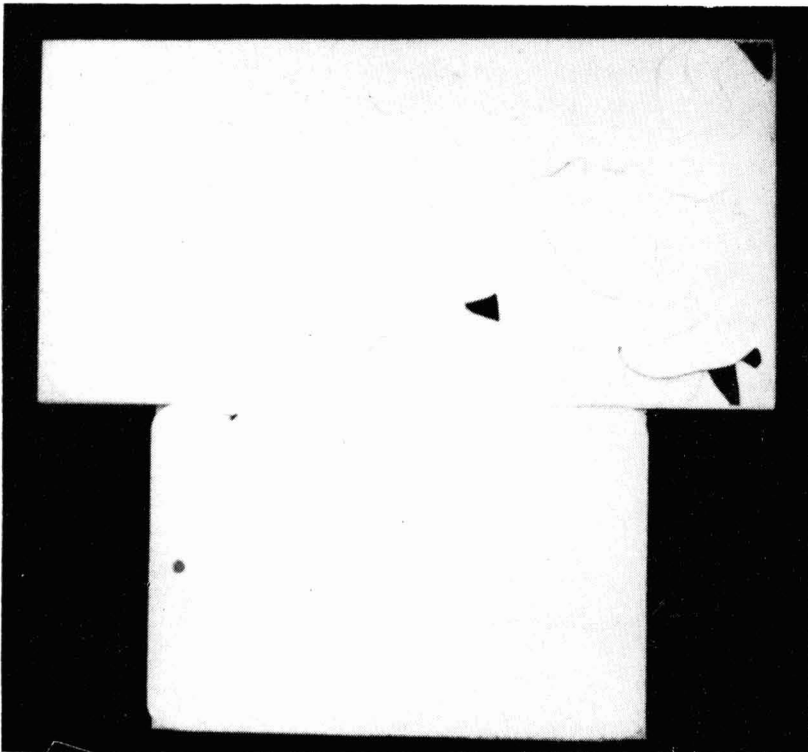


Fig. 26. Photograph of cold check cracks (a) on M/S (b) on plate glass

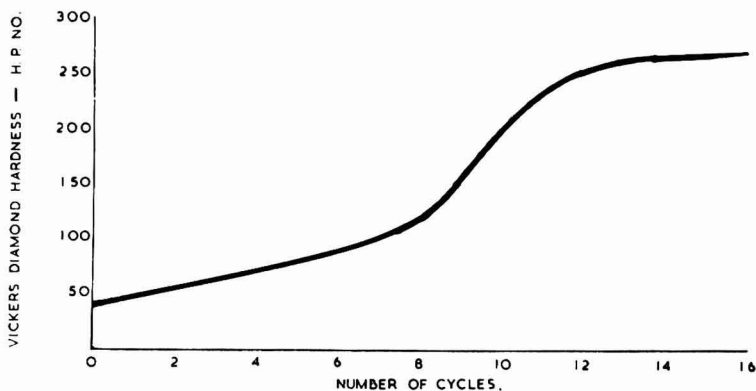


Fig. 27. Vickers Diamond Hardness during cold check cycling

## Weathering of thermosetting acrylic resins

### Three-component thermosetting acrylic systems

It is well known that acrylamide acrylic resins normally require temperatures in excess of  $150^{\circ}\text{C}$  to obtain a fully cured film, usually in conjunction with an epoxy resin. In order to reduce this temperature to that required in the automotive industry, three-component systems were introduced which incorporated acrylamide acrylic resins combined with alkyd and melamine resins. These enamels have given excellent gloss, weathering and pigment wetting properties, and were used for some time on a large scale in the automotive industry. Because of difficulties experienced in some systems the two-component types comprising hydroxylated acrylic resins crosslinked with melamine resins have now replaced this type of system. A comparison between Florida weathering of a 5:4:1 blend of acrylamide acrylic:alkyd:melamine resin system and 70:30 hydroxylated acrylic:melamine resin combination is given in Fig. 28. It can be seen that in

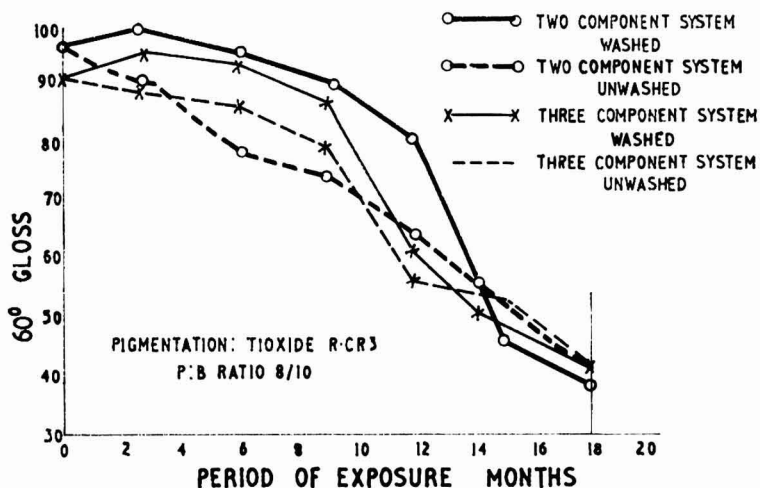


Fig. 28. Florida weathering of 5:4:1 acrylamide acrylic:alkyd:melamine enamel versus 7:3 hydroxy acrylic:melamine system

general the hydroxylated acrylic resin system is slightly superior to the three-component system, but both give fairly satisfactory results. Later work indicated that the three-component system gave better weathering properties in metallic finishes than straight alkyd/amino enamels, but did not perform as satisfactorily as two-component hydroxy acrylic/melamine metallic systems. The introduction of a large number of metallic enamels into the colour range of car finishes has therefore dictated the use of hydroxylated acrylic resins.

#### Two-component hydroxy thermosetting acrylic resin systems

In order to illustrate the outstanding durability of hydroxylated acrylic resins in metallic enamels, a comparison was made over a period of 18 months in Florida of metallic enamels based on high quality hydroxy acrylic:melamine and alkyd:melamine systems. The results are shown in Fig. 29.

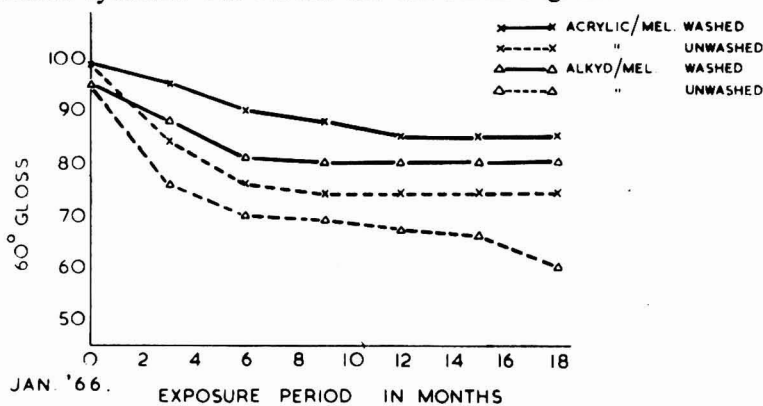


Fig. 29. Florida weathering of hydroxylated acrylic versus alkyd in metallics

One important advantage of hydroxylated acrylic resins over alkyds in the automotive industry is their ability to polish up more readily to a high gloss after a period of weathering. This property is demonstrated in Table 8 which gives the C.L.A. height readings taken from polished alkyd and acrylic panels

Table 8

CLA height measurements of different areas of panels exposed for 18 months in Florida

Area tested	CLA height ( $\mu\text{in}$ )	
	Acrylic/melamine	Alkyd/melamine
Unexposed area .. .. .	2.5	1.8
Exposed area .. .. .	6.9	2.5
Polished exposed area .. .. .	1.6	6.5
Repolished exposed area .. .. .	Not necessary	3.5
60° gloss of exposed area (polished) ..	100	77

Pigment Ti oxide R-CR 3  
Acrylic resin Epok D2101

P:B ratio 0.8:1  
Alkyd resin Epok A6004/70

Melamine resin Epok U9192

A:M ratio 7:3

after 18 months' exposure in Florida. Photographs of these panels after polishing are reproduced in Fig. 30.

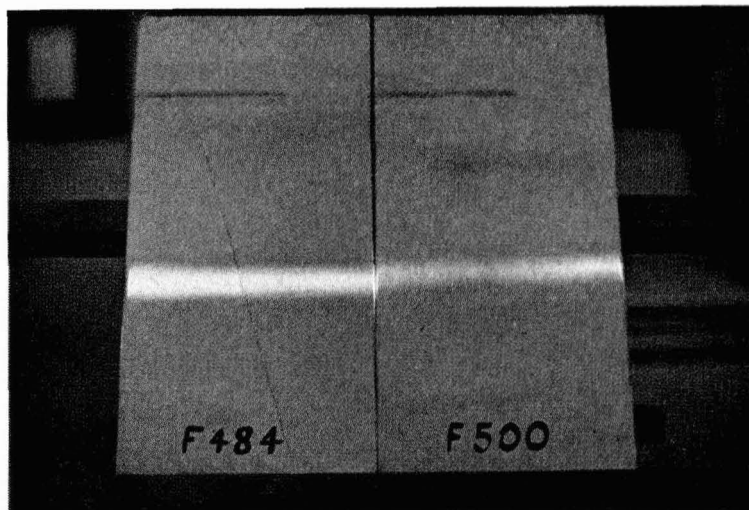


Fig. 30. Polished Florida panels. Acrylic and alkyd

### Future trends in thermosetting acrylic resins

#### *Solvent composition of the resins*

The pollution of the atmosphere by industrial and domestic fumes in large cities in America has resulted in legislation restricting the type of solvent used in surface coating applications, since other methods of removal of the harmful volatile materials, for example by condensation or combustion, are not always completely effective. In America the Los Angeles Rule 66 which was enacted in July 1966 to control the emission of certain organic solvents in the atmosphere has had direct impact on automobile enamel specifications. Thermosetting acrylics thus required considerable modification with regard to solvent composition when this rule was applied. So far no similar statute has been passed in the UK, but American-owned automobile companies have written these requirements into some of their new specifications.

Rule 66 deals with the photochemical reactivity of certain organic substances which are present as solvents in paint and resin formulations. Such solvents can be photochemically oxidised to aldehydes under suitable atmospheric conditions causing lung and eye irritations. These include some aromatics, olefins, and branched chain ketones. E. C. Larson and H. E. Sipple<sup>8</sup> have reviewed the position in the USA with regard to exempted solvents. Poy<sup>9</sup> discussed the maximum allowable concentration of restricted solvent by volume of the total solvent. Table 9<sup>9</sup> shows the amounts which are allowed.



Table 9

Maximum allowable concentration (%) of restricted solvent per volume of total solvent

Solvent	Rule 66	Regulation 3 industrial coating	Non-industrial coating
<i>1. Aromatic H/C</i>			
A. Polysubstituted .. .. .	8	20	8*
B. Monosubstituted:			
(i) Toluene .. .. .	20	20	20
(ii) Ethylbenzene .. .. .	20	20	20
(iii) Other .. .. .	8	20	20
<i>2. Aliphatic H/C .. .. .</i>			
	U	U	U
<i>3. Olefins .. .. .</i>			
	5	20	8*
<i>4. Chlorinated solvents</i>			
A. Trichloroethylene .. .. .	20	U	U
B. Perchloroethylene .. .. .	U	U	U
C. Saturated .. .. .	U	U	U
<i>5. Oxygenated solvents</i>			
A. Olefin-containing .. .. .	5	20	8*
B. Aldehydes .. .. .	U	20	8*
C. Branched ketones .. .. .	20	U	U
D. Other .. .. .	U	U	U

U=Unlimited

\*An additional 12% of volume of nonsubstituted hydrocarbon is permitted.

### Conclusions

The performance of hydroxylated acrylic/melamine resins in reflow enamels is dependent upon several inter-related factors including the chemical constitution of the resins, type and level of pigmentation, and the ratio of acrylic-melamine resins in the paint. It has been indicated that it is possible to formulate a resin having the required speed of cure to give satisfactory sanding, reflow and over-spray acceptance properties for repair-in-process operations, and at the same time to be resistant to cold checking under severe conditions in thick films. In view of the possible legislation regarding air pollution, modification of existing acrylic polymers and continued resin development appears to be necessary. The weathering properties of thermosetting acrylic resins under severe conditions, especially when used in metallic enamels, is shown to be extremely satisfactory.

### Acknowledgments

The authors wish to thank the Directors of BP Chemicals (UK) Limited for permission to publish this paper and also like to thank their colleagues P. J. Fry, G. A. Cannon and K. V. Green for helping in practical details and T. I. Price and F. M. Couchman for their co-operation in obtaining data for this paper.

[Received 12 July 1968]

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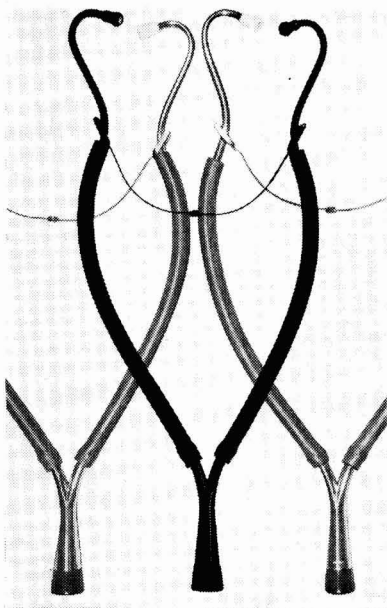
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# The rheological shear-rate-limit method as an aid in paint production

By K. M. Oesterle

Kusnacht-Zurich and K. W. Mader A.G. Lachfabrik, Kilwangen, Switzerland

## *Summary*

The development of the method, which has been reported elsewhere, together with its underlying principles, is briefly reviewed. A new interpretation of the rheological equilibrium curves and their value in paint technology are discussed.

Examples are given of the application of the method to the control of production and raw material quality, to the development of dispersion processes and to the investigation of pigment-vehicle interaction.

It is suggested that the method can provide insight into the mechanism of surface and interfacial phenomena without the need of other more costly and time-consuming techniques.

## La méthode de la limite de vitesse de cisaillement rhéologique comme aide à la production des peintures

### *Résumé*

Une courte introduction fait passer de revue les méthodes préliminaires qui donnaient la base pour l'établissement de la courbe rhéologique d'équilibre et — en vue des problèmes de l'industrie des peintures — de la nouvelle manière d'interprétation, comme succession des valeurs limites.

Des exemples nous donnent une impression de la possibilité d'appliquer la méthode pour contrôle des matières premières, pour la disposition de la production des peintures, ainsi que pour pénétrer dans les problèmes de rhéologie et des effets de surface et des interfaces.

Il paraît que cette méthode nous donne des connaissances des effets de surface par une voie simple, rapide et assez bon marché, au moins suffisante en grande partie aux besoins de l'industrie des peintures. D'autres méthodes donnent sûrement des résultats bien plus détaillés, mais elles sont normalement beaucoup plus chères et demandent plus de temps.

## Die rheologische Grenzgeschwindigkeit—Gefälle-Methode als Hilfe in der Anstrichfarben—Produktion

### *Zusammenfassung*

Es wird ein kurzer geschichtlicher Ueberblick über die Vorläufer der Methode gegeben und anschliessend folgen Hinweise über die Aufnahme von Gleichgewichtskurven. Deren rheologische Deutung als Ausdruck der Folge rheologischer, eingegabelter Grenzzustände dürfte für die Lackindustrie von weitreichender Bedeutung sein.

Anschliessend folgen Anwendungsbeispiele aus den Gebieten der Rohmaterialkontrolle, der Produktionskontrolle, der Produktionsführung, sowie Studien über Rheopexie und Pigment/Bindemittel-Wechselwirkung.

Es scheint dass die Methode in der Lage ist, in Grenzflächenvorgänge Einblicke zu gewähren, die andere Methoden nur auf teurere, zeitraubendere, dafür aber auch detailliertere Weise zu erbringen vermögen.

## Метод реологического предела скорости сдвига как пособие в производстве красок

### *Резюме*

Кратко рассматриваются разработка этого метода, о котором доложено отдельно, и его основные принципы. Обсуждаются интерпретация кривых реологического равновесия и их значение в технологии красок.

Прилагаются примеры применения этого метода в контроле производства и качества сырья, в развитии дисперсных процессов и в анализе взаимодействия между пигментом и растворителем.

Высказывается мнение что этот метод поможет объяснить механизм поверхностных и поверхностно—раздельных явлений, без необходимости других более дорогих и более длительных технических приемов.

### **Historical aspects**

The measurement of flow-curves in the study of rheological problems has been known for a long time<sup>1</sup>. Buchdahl<sup>2</sup>, Thimm<sup>3</sup>, Lower<sup>4</sup>, Walker<sup>5</sup>, Zettlemoyer<sup>6</sup>, and especially Pawlowski<sup>7</sup> and Wapler<sup>8</sup> working with a coaxial cylinder viscometer, considered the way in which the stress reaches an equilibrium value at constant shear rate to be significant for a thixotropic system. Williamson<sup>9</sup>, and later Peter<sup>10</sup>, studied the nature of the flow curve resulting from a plurality of such equilibrium values.

In 1963, 1964 and 1966<sup>11</sup> the present writer showed how this equilibrium curve can be obtained in a rapid and easy way, and that the parameters of the curve are of great significance in paint technology. By this method, called by the author the "shear rate limit (SRL) method," information is obtained about the flow behaviour of the paint (i.e. whether it is Newtonian, Binghamian, thixotropic or rheopectic) and about pigment-vehicle interaction, which provides guidance in the development of processing techniques and the establishment of criteria of quality.

### **Abbreviated theory of the principles of the SRL-method**

The ideal in measuring the characteristics of a paint would be to do it in such a way that the forces employed in carrying out the measurement did not produce changes in the nature of the paint. From such ideal measurements one would be able to deduce exactly how the paint ought to behave in any given method of application.

Similarly in rheological measurements it would be advantageous if one could reproduce results in a succession of tests, independent of the hydrodynamic history of the sample provided that no chemical change had occurred between measurements.

This is a rather theoretical ideal, unattainable by the usual method, but approached in the SRL-method.

The SRL-method produces curves which permit:

- determination of thixotropy in a reproducible way irrespective of rheological history,
- numerical expression of the distinction between thixotropy and plasticity,
- a test of deviation from standard production,
- prediction of the behaviour of a new formulation in a given dispersing machine,
- evaluation of pigment-vehicle interaction.

### Principle

If a paint possesses a thixotropic gel-structure produced by secondary forces, this structure can be destroyed by mechanical forces, such as the shear applied to the paint in rotational viscometers.

The more highly developed the gel-structure, the greater will be the gel-micelles and the greater will be the possibility of their touching each other when moving in the paint. A small movement will then cause micelles to rub against one another, resulting in a reduction in viscosity even at low shear rates. The gel-forming forces and the mechanical gel-destroying forces are counteracting each other; consequently, for each degree of gel-formation there must exist a certain definite shear rate, just sufficient to prevent the growth of the micelles. This corresponds to the equilibrium or limiting relationship attained asymptotically when shearing stress is plotted against time at constant shear rate. By carrying out the experiment at a randomly selected series of shear rates, equilibrium values of shearing stress may be plotted against the

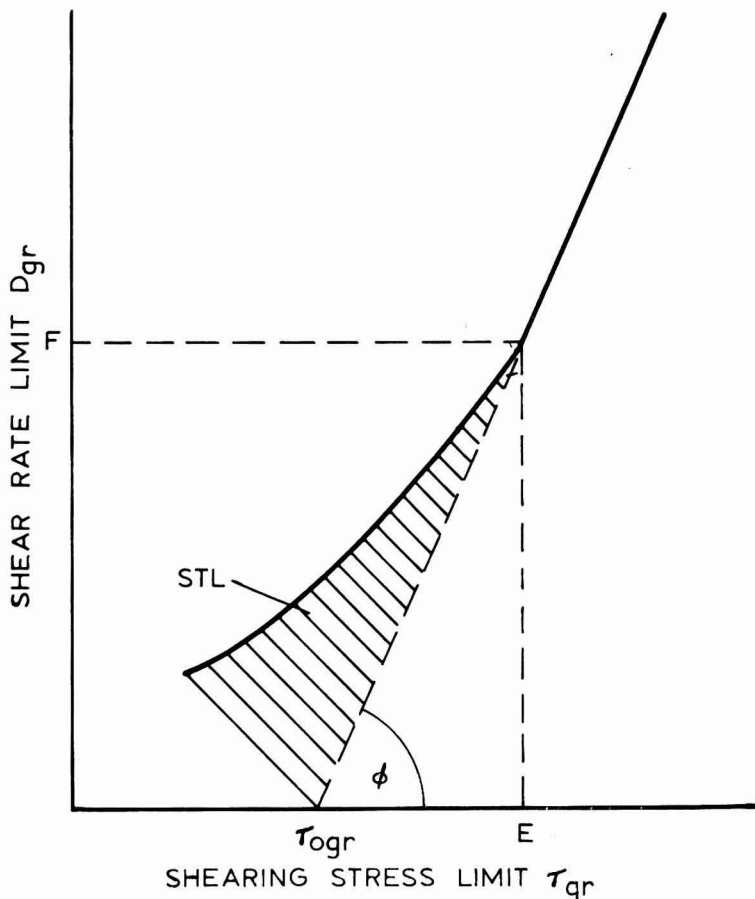


Fig. 1. Viscosity limit curve

corresponding values of shear rate to give the equilibrium or "viscosity limit" curve (Grenz-Viscositäts Kurve) of Fig. 1. It is the author's experience that such curves are best obtained by using a concentric cylinder viscometer with a driven bob and a gap of 2-4mm. The author has employed the Contraves Rheomat 15. The gap of this instrument is not so small as to show wall effects or to destroy structure completely by lack of space and high shear rate. Furthermore the driven bob reduces the danger of plug-flow.

The viscosity limit curve yields four of the five parameters which, in the author's theory, characterise the rheological behaviour of the sample. (Note that the subscript gr denotes an equilibrium value  $gr = \text{Grenz-}$ .)

$\tau_{o\ gr}$  = the yield value limit (Grenzfließgrenze), significant in assessing particle-particle interaction, as time-independent structure due to vehicle adhering firmly to pigment. Units, dyne  $\text{cm}^{-2}$ .

$\gamma_{s\ gr}$  = limiting coefficient of apparent plasticity given by  $\gamma_{s\ gr} = \cot \varphi$ , significant when a more mobile time-independent structure envelops the pigment particle, and an indication of time invariant viscosity (micelle-formation alone or round the pigment). Units, dyne sec  $\text{cm}^{-2}$ .

STL = area significant for specific thixotropic power (Scheinbare Thixotrope Leistungsfläche) i.e. power per unit volume due to thixotropic forces and structure. Units, dyne  $\text{cm}^{-2} \text{sec}^{-1}$ .

$D_{grw}$  = ultimate shear rate limit i.e. the shear rate at which all thixotropic structure is destroyed. This corresponds to a shearing stress value of  $\tau_{grw}$  and their relationship determines the speed of gel-recovery GRV (Gelrückbildungsgeschwindigkeit) as shown by Doherty & Hurd<sup>12</sup>.

$$\text{GRV} \simeq D_{grw} \cdot \tau_{grw} \quad \text{dyne cm}^{-2} \text{sec}^{-1}$$

As shear rate is increased, the viscosity limit curve becomes linear at  $D_{grw}$  (point EF of Fig. 1.) but linearity is again lost at very high values of D. In practice this latter is not normally achieved. Of course there is a danger that at high values of D heat will be generated and affect the material chemically. This can be checked by repeating the initial measurements on the same sample.

#### *Bingham behaviour vs thixotropy*

A distinction between these phenomena provides the fifth parameter characterising the material. The viscosity limit curves are very similar in practice but the distinction is made clear by a consideration of the  $\tau$ -t (Pawlowski) curves used in their construction. Typical Pawlowski curves are shown in Fig. 2. Similar considerations apply to the distinction of dilatancy from rheopexy as systems with very brittle and fine structure round the pigment.



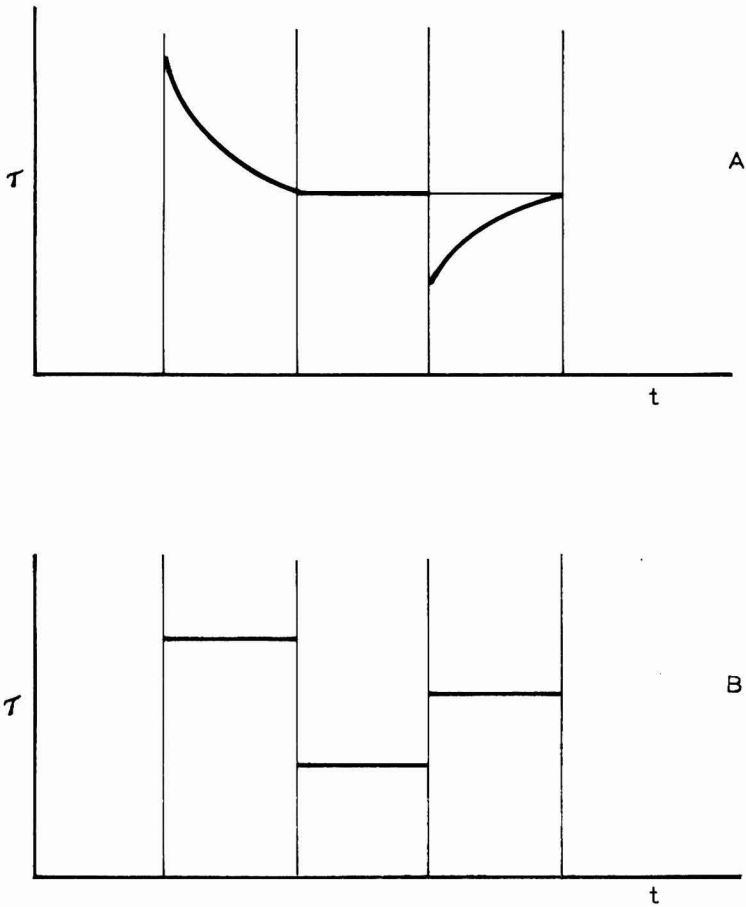


Fig. 2. Typical Pawlowski curves for thixotropic (A) and Bingham (B) behaviour

**Reproducibility of the technique**

A sample of a primer known to have good storage stability was tested on the Rheomat 15 using System C at 20°C. Five days later a further series of readings were taken. The co-ordinates of the resultant SRL curves which are given below indicate the good reproducibility of the technique. Note that  $\tau_{gr}$  is expressed as viscometer scale units.

Data obtained on 17 February 1966.

$\tau_{gr}$ scale units	..	..	6.5	19.0	23.5	42.0	71.0
$D_{gr}$ sec <sup>-1</sup>	..	..	16.5	31.0	36.0	56.0	83.0

Data obtained on 22 February 1966

$\tau_{gr}$ scale units	..	..	6.8	19.0	23.0	42.0	70.5
$D_{gr}$ sec <sup>-1</sup>	..	..	16.0	31.5	36.0	56.5	83.0

## Suggested interpretation of rheograms

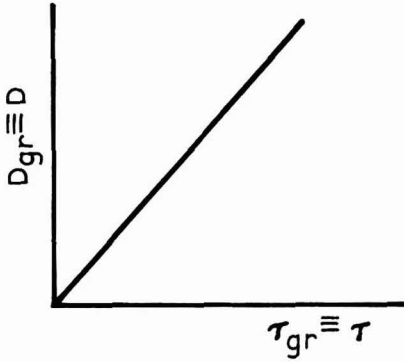


Fig. 3. Newtonian: Independent movement of molecules of the fluid and the suspended particles.

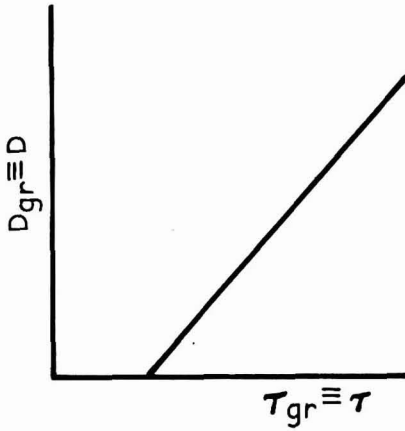


Fig. 4. Simple plasticity. Suspended particles are of such a size as to create a yield value. This implies that a definite energy barrier must be overcome to initiate flow but once in motion the particles move freely.

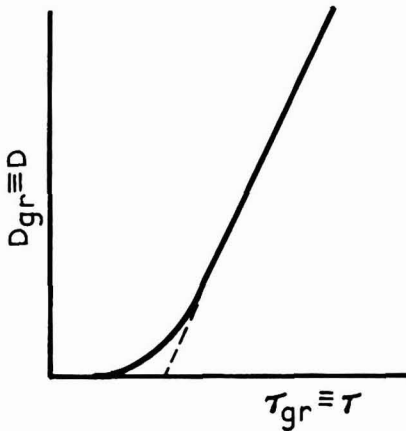


Fig. 5. Bingham body (Pseudoplastic). Particles are surrounded by a thin, more or less rigid, time-invariant structure. At high shear rates simple plasticity is exhibited. Viscometer readings are not asymptotic.

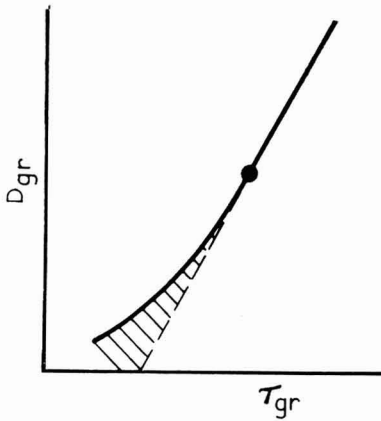


Fig. 6. Thixotropic body: Interaction between particles forms a time-variant structure. Formation and destruction of this structure corresponds to an isothermal reversible sol-gel transformation<sup>13</sup>. Viscometer readings are asymptotic.

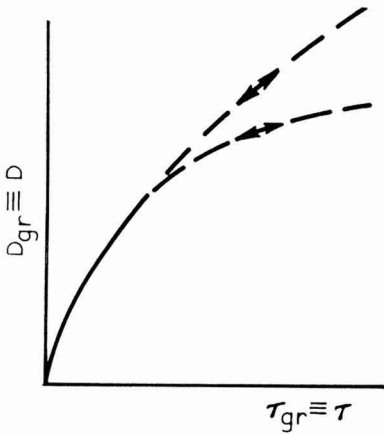


Fig. 7. Dilatant body: Particles seem to be covered by a thin, brittle envelope. The envelope is destroyed or damaged at high shear rates so that the particles come into direct contact. In the grinding process the particles collide inelastically. Grinding is easy but re-agglomeration readily occurs. At low shear rates the viscometer readings resemble those of a Bingham body but tend to oscillate at high shear rates due to instability associated with rupture of the envelope.

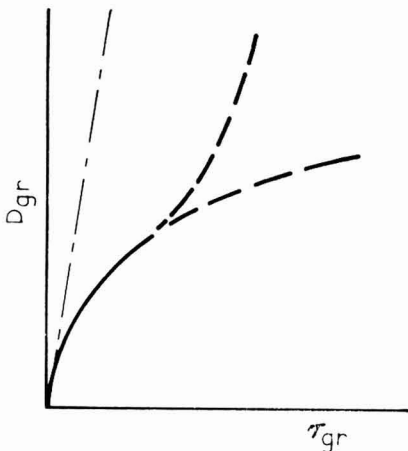
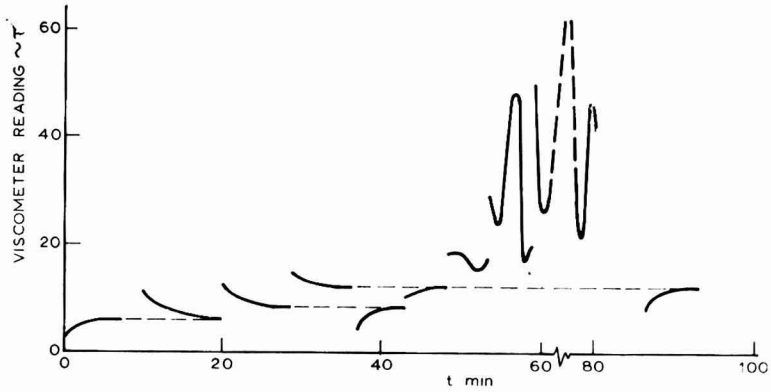
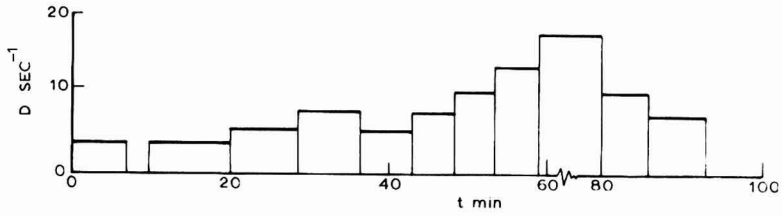
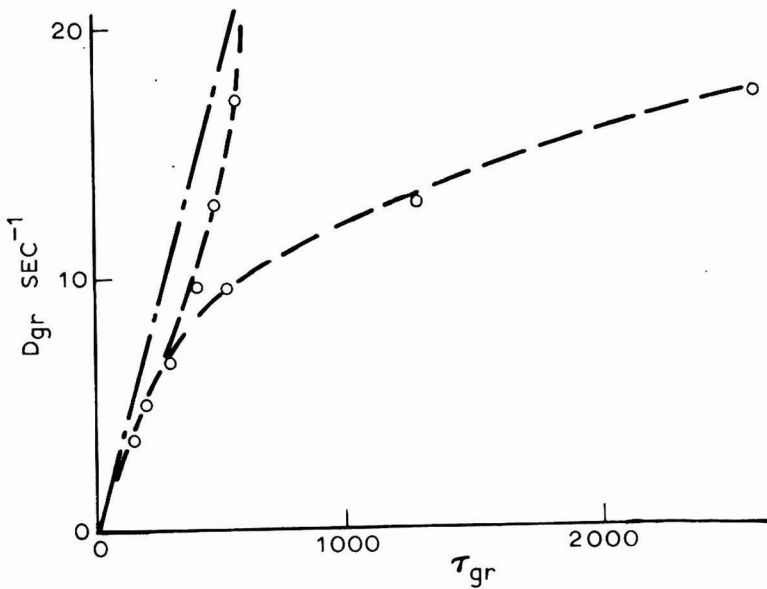


Fig. 8. Rheopectic body: Resembles the dilatant body but deformation and recovery of the envelope is time-variant. Below a critical shear rate the behaviour is apparently thixotropic; above it the system becomes unstable and viscometer readings oscillate. See also Fig. 9.



(a)



(b)

Fig. 9. Rheopectic body (71 per cent zinc chromate suspension): (a) Viscometric data obtained on Rheomat 15, System C, Clearance 3.2mm, temp 20°C; (b) Derived viscosity limit curve

### Survey of rheological behaviour

Determination of the viscosity limit curve is one of the best methods for surveying the rheological behaviour of a substance. Mathematical methods may be used to determine the ultimate shear rate  $D_{grw}$  and the area of specific thixotropic power STL but they are rather complicated and in practical paint making it is usually sufficient to consider the general shape of the curve. This may be said to summarise the rheological behaviour of the sample.

It may be noted in passing that the hysteresis loop method of estimating thixotropy is influenced by the programming of the successive shear rates applied. The equilibrium (limit) values in the author's method eliminates such bias.

#### Suggested interpretation of results on various thixotropic paints.

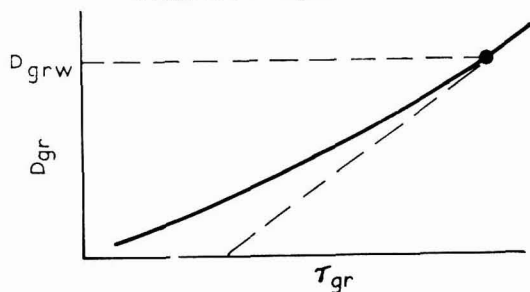


Fig. 10. Strong interaction between pigment particles and vehicle. High yield value due to large particles formed by agglomeration and/or to presence of time invariant envelope (adsorbed vehicle). Large STL, high  $D_{grw}$  due to rapid gel regain.

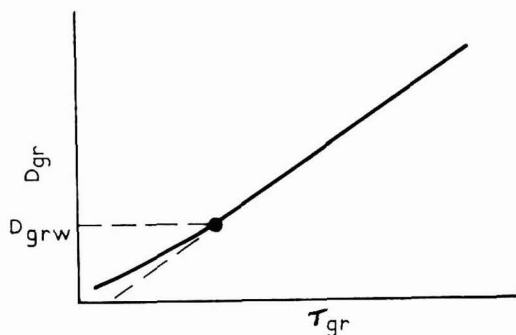


Fig. 11. Little interaction between pigment and vehicle. Small particles possibly covered with a slippery layer. Slow gel regain. Time-variant thixotropic structure.

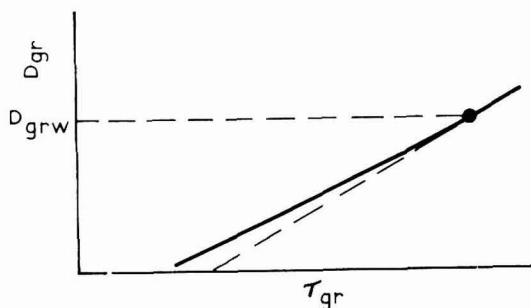


Fig. 12. Large micelles present, relatively time invariant. Low Time-variant thixotropic structure with rapid regain.

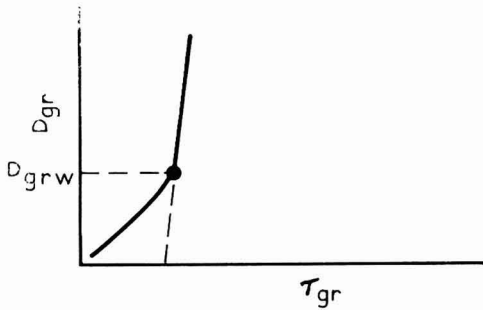


Fig. 13. Vehicle contains small micelles. High thixotropic structure. Slow gel regain.

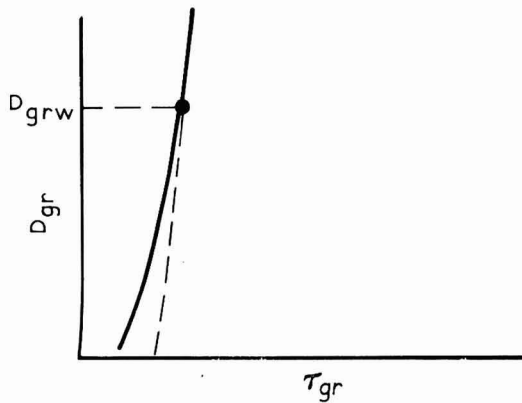


Fig. 14. Resembles the paint of Fig. 13 but has high  $D_{grw}$  and hence rapid gel regain.

### Application to production control

The reproducibility of the SRL curves means that, for a given formulation, it may be used to control the effectiveness of the dispersion process. In such production control it will usually be sufficient to determine only one or two points on the curve which should indicate whether the behaviour of the batch is within the required tolerance. Clearly, materials which "mature" after manufacture, or are rheologically unstable with respect to time, must be sampled and tested to a carefully and reproducibly timed schedule. Such, unfortunately, is the rule rather than the exception with paints so that it is worth spending some time and effort on establishing a timed sampling and testing drill whereby a two-point test becomes meaningful.

Measurements made during the process of maturing have only a transitory value; the most meaningful results are obtained on the fully matured paint.

Fig. 15 shows some typical results obtained in the author's factory.

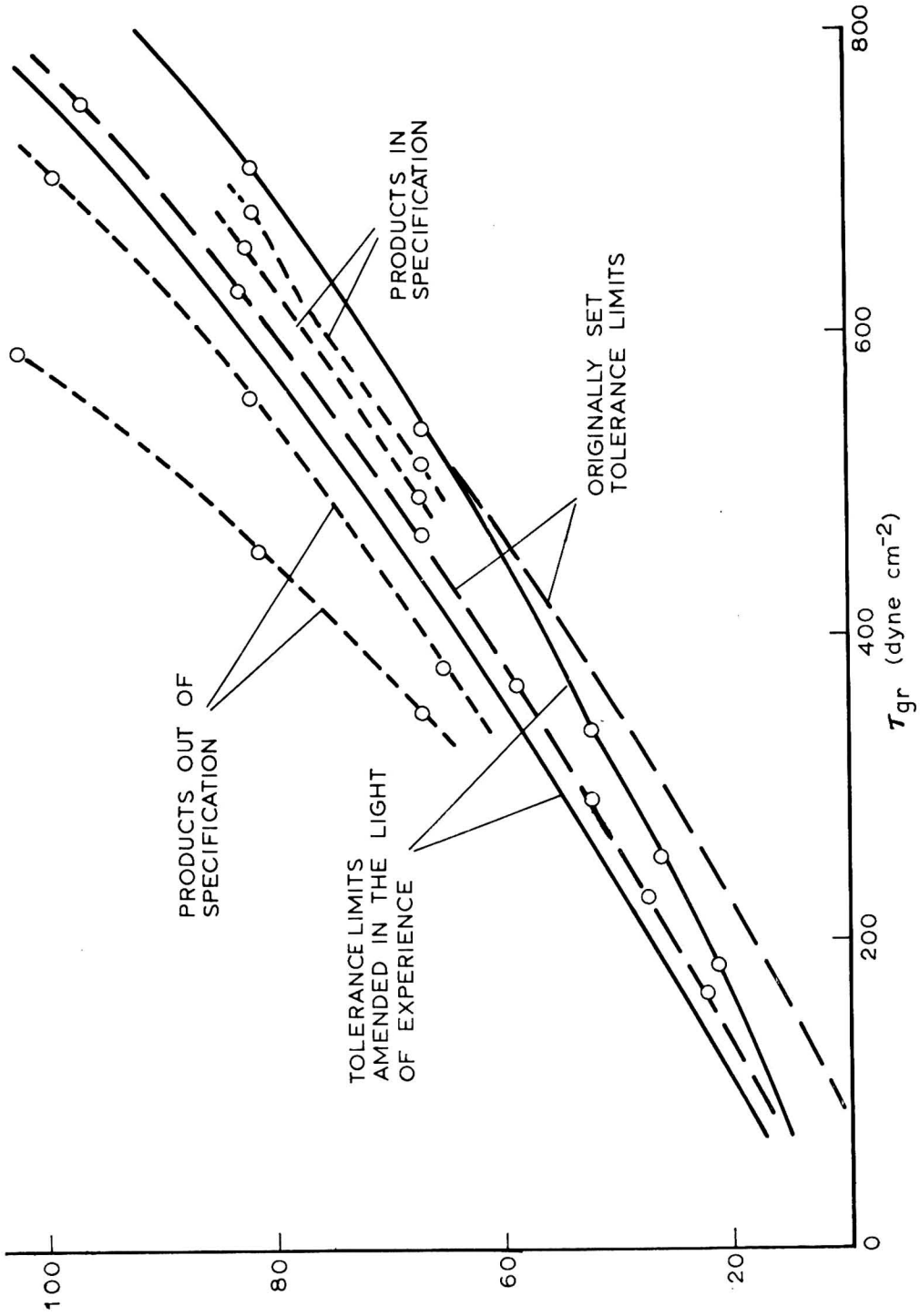


Fig. 15. Production control of red-brown primer-surfacer SRL method using Contraves Rheomat 15

### Application to raw-material control

The quality of paint products depends as much on the interactions between their components as on the nature of the components themselves. These interactions are largely instrumental in determining rheological behaviour patterns. Hence, the influence of a particular raw material on the rheological behaviour of a standard mix provides a useful index of raw material quality. Given a standard vehicle, for example, it is a simple procedure to evaluate a pigment against a predetermined standard by means of the SRL method. The converse, using a standard pigment, is equally true, permitting the rapid evaluation of vehicles in rheological terms.

It will be clearly apparent to the reader that such evaluations require, as an essential prerequisite, precisely standardised procedures for dispersion and testing and careful attention to the timing of all operations. Results of the form shown in Fig. 19 could obviously correspond as well to raw material testing as to production control.

### Development of production methods

The nature and timing of the schedule of dispersion operations is of great importance in determining paint quality. The SRL method provides a means of predicting the result of applying schedules proposed by the development laboratory to large scale manufacture.

Suppose that one starts with a simple formulation comprising carbon black, alkyd resin, wetting agent and solvent, and that the sequence of operations is mix, rest, Cowles, sand-mill. Each operation may be carried out for a short time or for a long time and possible combinations of schedules would be those shown as I to VIII in Fig. 16. Strikingly different products may be obtained

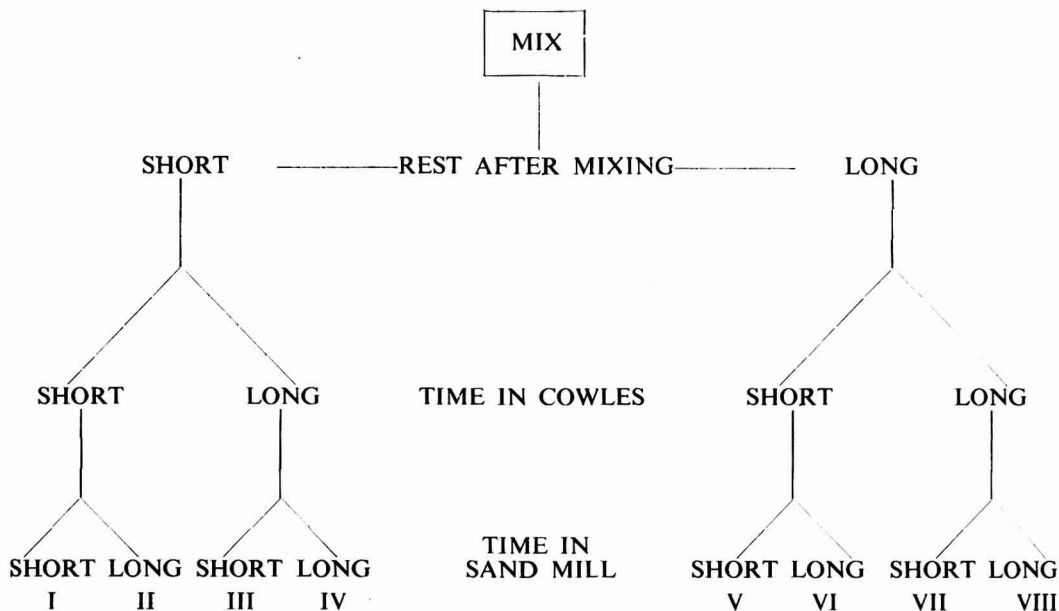
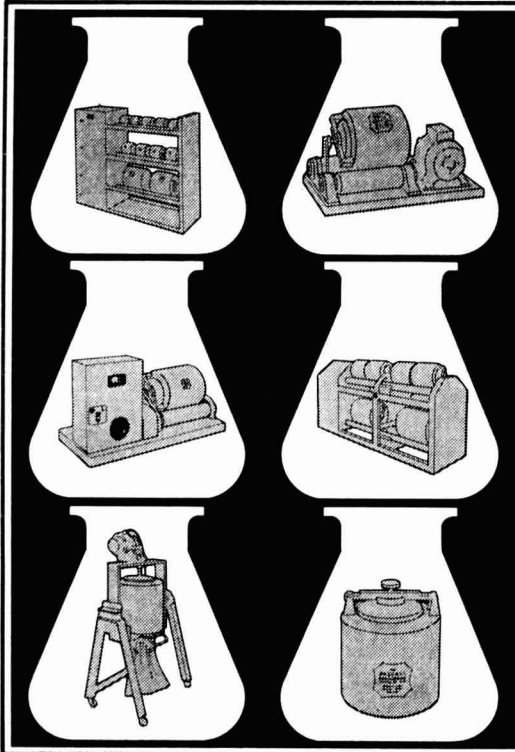


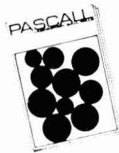
Fig. 16. Some possible production schedules





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from the same combination of raw materials when such differing manufacturing schedules are employed. The relevant test results, illustrated in Fig. 17, may be interpreted as follows:

- (a) Short rest-time after mixing      Pigment-vehicle interaction is slight. Sand mill just reduces particle size and lowers viscosity. Prolonged time in Cowles raises viscosity and increases STL but subsequent effect of sand mill is unchanged.
- (b) Long rest-time after mixing      Pigment-vehicle interaction accentuated. After short time in Cowles, the sand mill reduces particle size in an optimal way but in such a way as to increase viscosity. Prolongation of Cowles-time leads to viscoelastic behaviour and very high STL.

### Evaluation of pigment-vehicle interaction

The excellent reproducibility of the SRL measurements permits the investigation of the effects of special pigments and additives on thixotropy and of the influence of resins, solvents and adsorbed layers, effects considered as "pigment-vehicle interaction."

Figs. 18 and 19 show the effect of inducing thixotropy in an alkyd-titanium dioxide system by means of additions of filler and thixotropic resin respectively. Fig. 18 shows the increase of STL, yield value limit and limiting coefficient of apparent viscosity which results from increased pigmentation. The final shear rate limit  $D_{grw}$  does not rise so sharply and the indications are that it will attain a maximum value at some high level of pigmentation. A plausible explanation of these results lies in the fact that, beyond a certain pigment concentration, insufficient vehicle is present in interstices to permit the development of thixotropic structure. The characteristic feature of the STL, yield value and viscosity curves is their curvature away from the abscissa as pigmentation is increased.

Fig. 19 illustrates the effect of replacing increasing amounts of alkyd by thixotropic medium at constant pigmentation level. The STL and yield value limit plots curve towards the abscissa whilst that of limiting viscosity is of a complex form. These are broadly explicable in that both STL and yield value may be expected to attain maximum values when all the alkyd has been replaced by thixotropic medium.

The contrast between Figs. 18 and 19 provides a basis for deciding whether in any given instance it will be preferable to induce thixotropy by the one means or by the other. In general it would seem that the use of thixotropic media is less critical than the use of pigment or filler for this purpose.

The use of the method to demonstrate clear differences in pigment-vehicle interaction due to change in vehicle acid value is illustrated in Fig. 20. Curves A and B differ only in the value of the coefficient of apparent plasticity. They

are interpreted as an indication of little or no interaction—a kind of “pseudo-wetting.” In contrast, curves C and D demonstrate the marked change in rheological behaviour which ensues from strong interaction (effective wetting) between the high acid value medium and the rutile—Syloid pigment.<sup>11</sup>

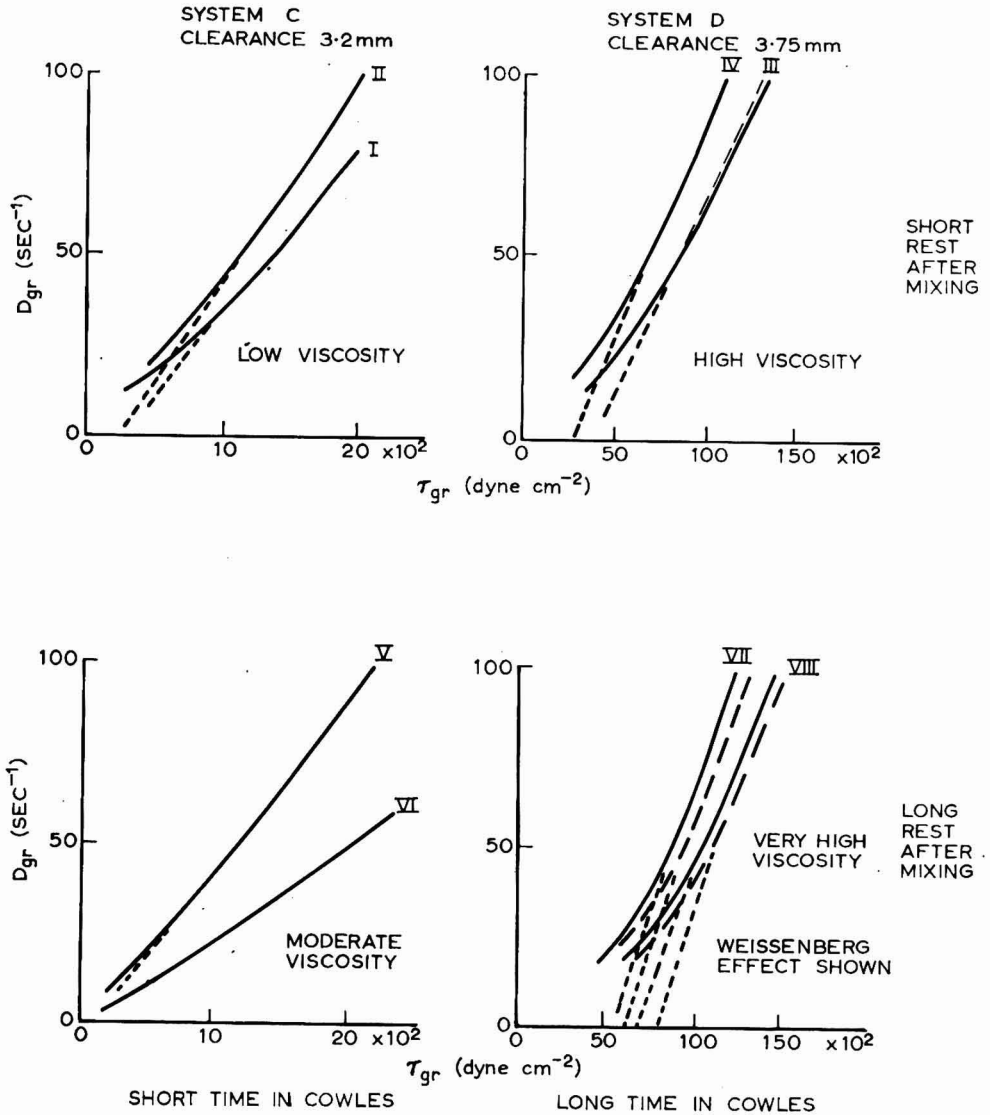


Fig. 17. Rheograms of carbon black dispersions SRL method. Rheomat 15 at 20°C

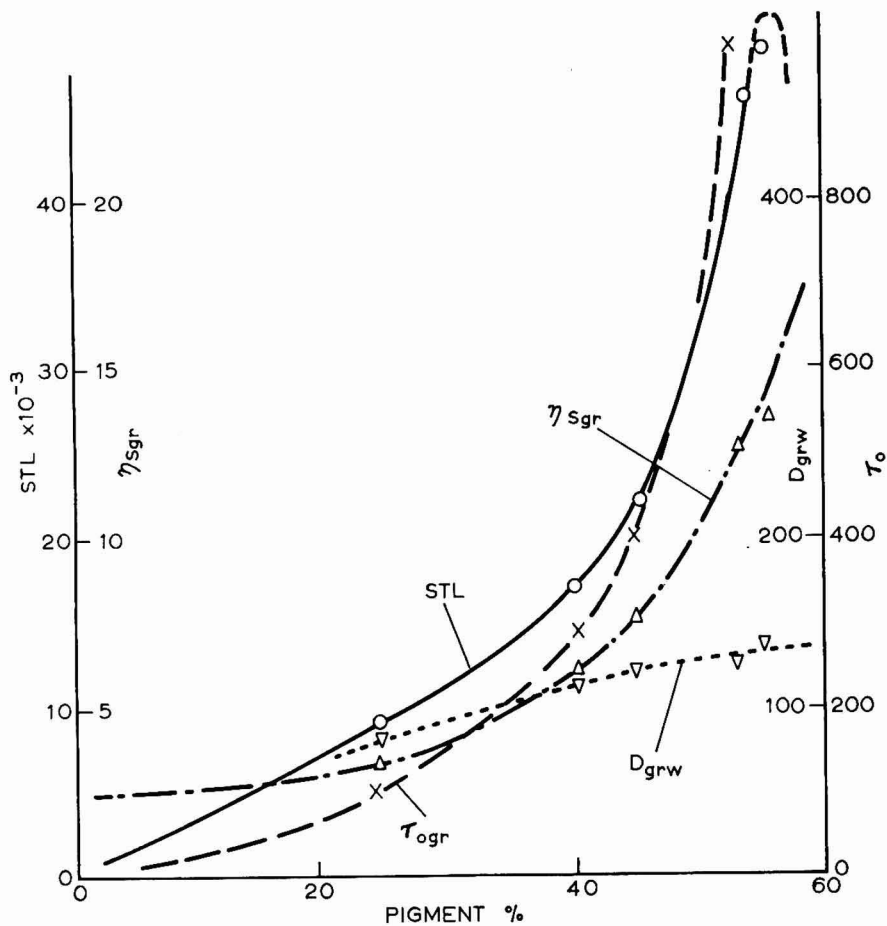
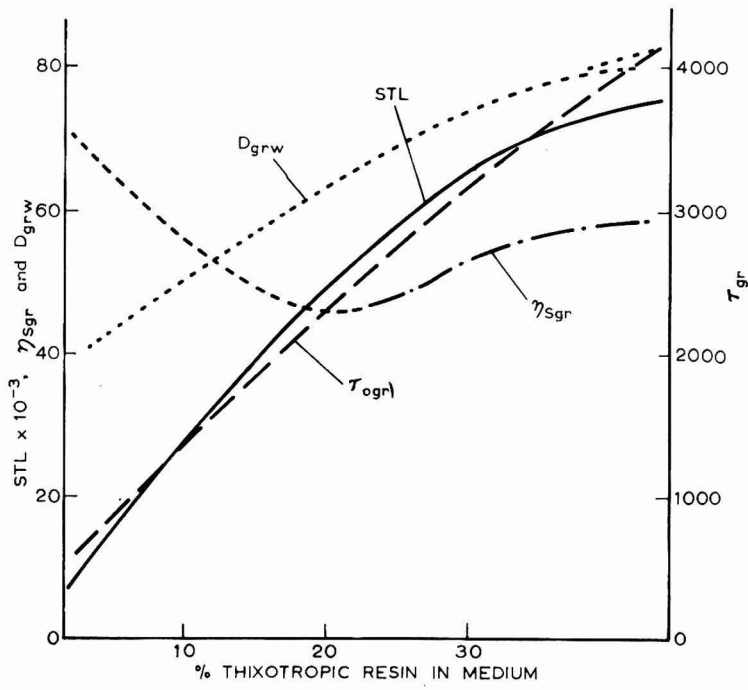


Fig. 18. Thixotropy induced by pigmentation



**Fig. 19. Thixotropy induced by thixotropy medium**

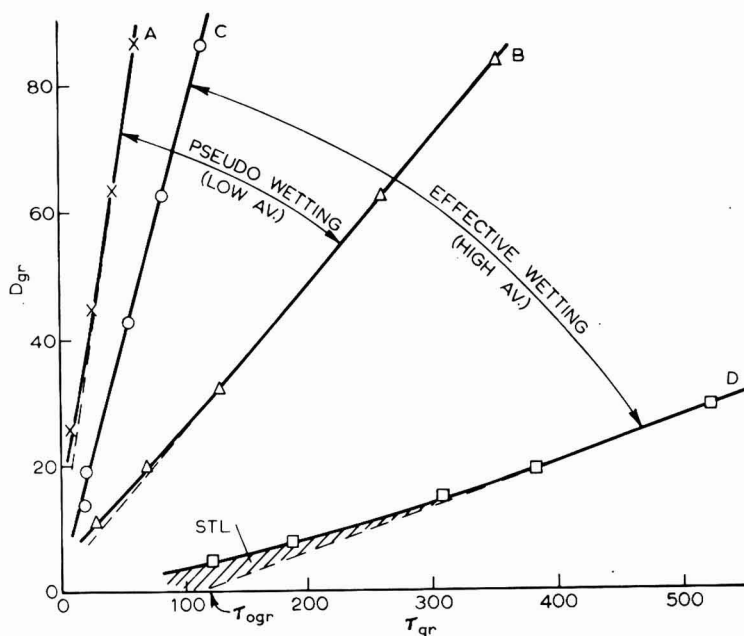


Fig. 20. Pigment-vehicle interaction

Curve	A	B	C	D
Non-reactive alkyd 60% ..	147	147	—	—
Reactive alkyd 60% .. ..	—	—	147	147
Solvent .. .. .	73.5	73.5	73.5	73.5
Rutile and Syloid .. ..	—	272	—	273

See ref. 11 for further details

As a study of the influence of solvents on pigment-vehicle interaction by the SRL method the data of Fig. 21 may be considered. Curve A is that of a simple alkyd solution in xylol and the effect of pigmenting it with Syloid given by curve A' is seen to involve little change in STL. Replacing part of the xylol by ethylene diamine (curve B) or *n*-butanol (curve C) leads to substantial viscosity reduction. Pigmentation of the *n*-butanol-xylol solution to the same level as before gives curve C' which shows a high STL. This effect is attributable to the provision of hydrogen bonding centres by the *n*-butanol, and exemplifies the creation of thixotropic behaviour by effects due essentially to the solvent.

Finally, concerning the influence of adsorbed layers, it may be noted that Zettlemoyer et al<sup>15</sup> showed after extensive investigations that there exist on rutile two kinds of adsorbed water: chemically adsorbed water forming hydroxyl groups on the surface (strongly bonded), and physically adsorbed water which

can be eliminated by heating at 120°C. Zettlemoyer has suggested that the physically adsorbed water may be attached to the surface of the pigment by hydrogen bonds.

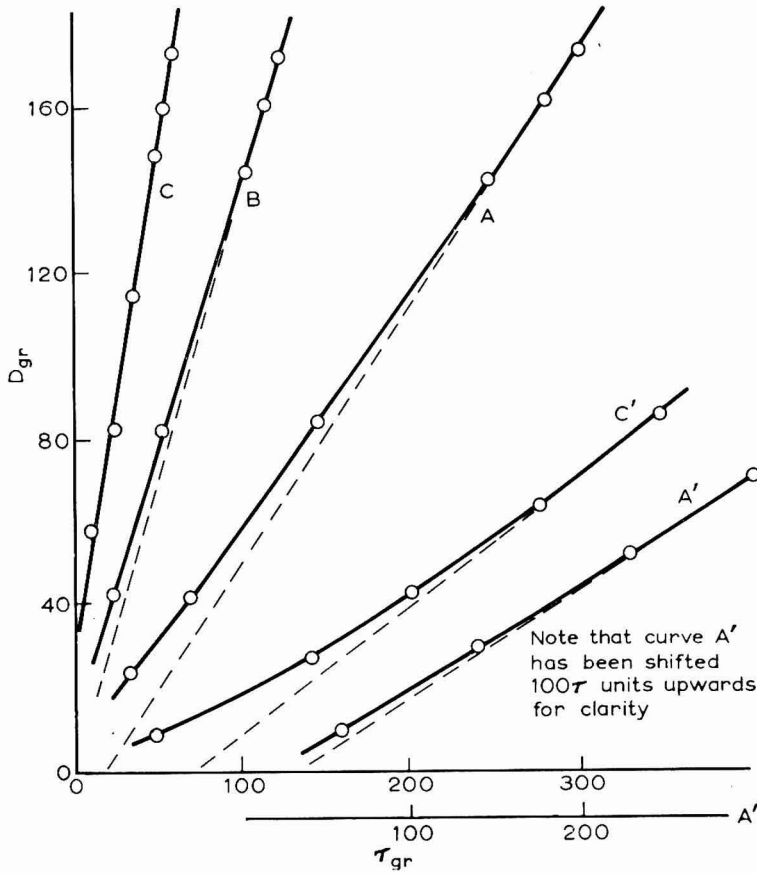


Fig. 21. Effect of solvent on pigment-vehicle interaction

Curve	A	A'	B	C	C'
Alkyd .. ..	147	147	147	147	147
Xylol .. ..	73.5	73.5	34.5	34.5	34.5
Ethylene diamine ..	—	—	39	—	—
<i>n</i> -Butanol .. ..	—	—	—	39	39
Syloid .. ..	—	57	—	—	57

The present writer applied the SRL method to investigate the situation on the surface of anatase. His results are summarised in Fig. 22.



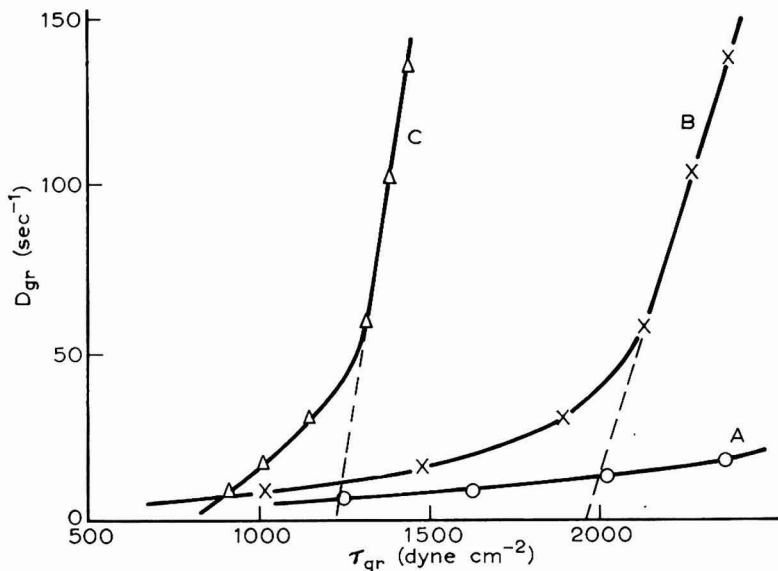


Fig. 22. Effect of adsorbed layers on pigment-vehicle interaction: Curve A Dry anatase in water; B Moisture-saturated anatase in water; C Dry anatase in 96 per cent *n*-butanol plus 4 per cent toluene

The strong interaction between distilled water and anatase which has been heated to eliminate physically adsorbed water is shown by curve A. This yields a good suspension ("macro-homogeneous") with a very high coefficient of apparent viscosity. Anatase previously physically saturated in terms of adsorbed moisture was difficult to suspend in distilled water and the suspension gave the very different curve B. This suspension was characterised by large particle size, relatively small coefficient of apparent viscosity and high STL. Curve C shows the effect of suspending the dried pigment in *n*-butanol to which some toluene had been added to reduce association via H-bonds.

It would seem that the SRL-method is capable of yielding useful information about the effects of adsorbed layers without the need to resort to i.r. spectrophotometry or thermodynamic measurements. Fundamental interpretation may admittedly be more difficult and equivocal but the method has the merit of being applicable by any paint technologist equipped with a suitable viscometer.

### Acknowledgments

The author's thanks are due to Mäderlack AG, Killwangen, Switzerland, for permission to publish; to Miss H. Schupfnger, Mrs J. Macekovic and Mr Fr. Schneider who made the measurements during the development and confirmation of the method; and to Dr A. Giesekus, Dr Pawlowski, Dr Meskat (Leverkusen) and Professor S. Peter (Hannover) for helpful and stimulating discussions.

[Received 27 March 1968]

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## Next month's issue

The Honorary Editor has accepted the following papers for publication and these are expected to appear in the January issue:

**"A new mechanism—syresis—by which pigment particles can migrate,"**  
by *H. H. McEwan and S. J. Gill.*

**Hull Section Co-operative Research Programme: "Acceptability of colour matches,"** by *F. D. Robinson.*

**"A laboratory d.c. supply unit for electrodeposition,"** by *D. Grime.*

## Errata

It is regretted that in the paper by Dr E. Wagener in the October issue, the key to Fig. 4, page 887, was incorrect.

It should read:

- Standard grade
- Special grade at equal volume
- Special grade at equal weight

# Review

## THE PRINCIPLES OF HARMONY AND CONTRAST OF COLOURS

By M. E. CHEVREUL, with introduction and notes by Faber Birren: New York: Reinhold Publishing Corporation, 1967. Pp. 255, price £11 14s. 0d.

Chevreul's book, a classic, is most attractively presented, the value and interest of the original work being greatly enhanced by the introduction and explanatory notes by Faber Birren, the well-known American colour consultant, and by some fine colour illustrations.

Born in 1786, M. E. Chevreul was recognised internationally as one of France's most eminent scientists and became the greatest living authority on animal fats when still a relatively young man. He lived to the age of 103, his long life being one of continuous intellectual enterprise and accomplishment. At the age of 44 he turned his attention to colour contrast, prompted by an investigation into complaints about the quality of colours produced in the Gobelins tapestry workshops. As Director for the Royal Manufactures at the Gobelins, he found it was contrast with other colours that affected the appearance of the products in question, so giving the impression they were of poor quality. From exhaustive observations he made of such visual phenomena, Chevreul ultimately formulated his "Laws of Simultaneous Contrast of Colours," the principles he evolved being generally accepted today as a natural part of colour theory.

Reprinted in this volume, after being out of print for many years, is the first of two English translations—that by Charles Martell, which was published in London in 1854. Beautifully printed in Japan and lavishly illustrated, the volume contains 28 colour plates, 15 of them adapted from the original French edition. The introduction describes Chevreul's life, achievements and background, setting the scene admirably for the account of his colour studies and conclusions which follows. Birren's notes appear alongside each page of the original work to provide an invaluable running commentary. There is, too, a section devoted to Chevreul's influence on the French Schools of Impressionism and Neo Impressionism with excellent colour reproductions of paintings by Seurat, Turner, Delacroix, Pissarro, Monet, Renoir, Van Gogh and Cezanne.

Faber Birren makes an outstanding contribution to this publication, the clear conciseness of his notes forming, appropriately enough, a striking contrast to the prolixity of Chevreul's writing. The conscientious reader will need patience and persistence to find the way through Chevreul's wordy labyrinth and will find it irksome to be taken along the same path several times. Birren offsets this most effectively, interpreting the terminology used and explaining what Chevreul is driving at without wasting a word, then putting it into proper perspective by qualifying his assumptions in the light of more recently acquired knowledge.

It could be said that a copy of this volume should be on every bookshelf were it not for the fact that few shelves could conveniently accommodate

its cumbersome bulk (it measures 13in x 11½in x 1½in thick). However, much of its appeal lies in the unusual format, and the large colour reproductions of the Impressionist paintings the size of the volume makes possible.

This publication is of most direct value to designers and painters. Indeed, this was Chevreul's intention, as he showed when he wrote about his observations of his ". . . hope that many classes of artists, particularly dressmakers, decorators of all kinds, designers of patterns for textile fabrics, paper-hangings etc., will derive some benefit from consulting them." But colour chemists would also derive no little benefit from the fuller understanding this book makes possible of factors affecting the end results obtainable with the products they develop. It should certainly be read by all concerned with colour, whether it be in their work, in their hobbies or in their general interest in the subject.

S. A. WOOD

# Information Received

(In case of difficulty regarding addresses, members and subscribers to the JOURNAL should apply for details to Director & Secretary of the Association at the address shown on the front cover.)

The Association was pleased to be represented recently at the Press showing of a new electrical heating system developed by the **Electricity Council Research Centre** in conjunction with the **East Midlands Electricity Board** and **Joseph Mason & Co.** of Derby.

The system operates on the thermal storage system, which, as it uses electricity at the cheaper night rate to charge the storage unit, gives a great saving in cost.

The plant consists of a 500 gallon stainless steel process kettle, which can hold up to 350 gallons of charge and is heated externally by hot air which is ducted by fan from the storage unit. The heating jacket is divided into four horizontal sections so that the kettle can be partially heated when it is not fully loaded.

The air is blown through the storage heater core, which is constructed of 3in × 5in × 24in in cast iron bricks, at 5,000 cu ft/min, and produces an average recirculating temperature of 280°C from a core temperature of about 700°C.

Joseph Mason claim that the running cost of this plant is half that of the previous gas-fired plant.

**Columbian International (Great Britain) Limited** recently celebrated the opening of new offices at Annabelle House, Hounslow, Middlesex, and the coming on stream of a new tread black plant at their Four Ashes site, with a reception in the new offices.

The new plant is said to double Columbian's UK production of carbon black. It will produce blacks for the tyre industry, but colour grade blacks will continue to be produced at Four Ashes.

Three new hydrophobic silicas are to be marketed by **Bush Beach & Segner Bayley Limited**, it has recently been announced. The silicas have hydrophobic surfaces which cannot be wetted with water, and are claimed to be dispersible in all organic liquids and mixtures of water and water miscible organic liquids.

Among applications claimed are: addition of small quantities to powders or fine crystalline materials to improve their free-running properties, and use in water thinnable paints, where an addition of up to 2 per cent of hydrophobic silica is said to improve salt spray resistance.

**Hardman and Holden Limited**, a member of the R.T.Z. group, and **Chemische Werke Munchen Otto Baerlocher GmbH** have recently announced the completion of a cross-licensing agreement for the manufacture of a number of chemicals from each other's product series. The companies have complimentary interests in the supply of chemicals to a wide range of industries, and the agreement is to enable both to provide improved service by broadening their respective product ranges.

The availability of a new system for accurate colour matching by computer has recently been announced by **Redifon-Astrodata Limited**.

Called *Redi-Colour*, the system, which is said to have been fully tested in practice prior to marketing, consists of two sections, a specially designed differential photometer, and a computer which incorporates automatic data transfer and conversion.

Reflectance values measured by the photometer are automatically transferred and stored in the computer, and converted for use in colour recipe computation, where

they are used in conjunction with dye boxes. These units store the colour properties of both dyestuff and substrate, and can be standardised using up to seven different concentrations. The recipe section of the computer calculates what dyestuffs and concentrations will provide an accurate match for the job in hand.

A tristimulus difference calculator allows the operator to match colours for six different types of illuminant, and the cost of the proposed recipe is automatically displayed.

The system is claimed to be adaptable to paint formulation.

**BXL Limited** have recently issued a new four page folder on the *BXL Bexol* adhesives service. The leaflet, publication No. 6809, is available from BXL.

It has recently been announced that **A. P. V. Holdings Ltd.** has acquired the whole of the issued share capital of **D. J. Osborne & Co. Ltd.**, one of the leading specialist manufacturers of industrial mixers. The existing *APV-Kestner* range of mixers is largely complementary to that of D. J. Osborne, and the merger is said to enable the A. P. V. Group to offer an extremely comprehensive range and improved service to customers.

A new company, **Skilbeck Industrial Paints Limited**, has recently been formed by the Skilbeck Group.

Modern paint production and quality control facilities have been established at Kempston, Bedfordshire, where the laboratory and technical service department is already located. The company aims to offer specialised high quality industrial finishes of advanced specification, tailor-made to customer's requirements.

**The Tintometer Limited** has recently announced a new and improved version of the *Lovibond Colour Scale*. This scale of red, yellow and blue glass filters was drawn up by Joseph Lovibond in the 1880's and in the last few years the original specifications were found to have slight discrepancies in linearity. The *Renotated Lovibond Scale*, which has been made possible by advanced techniques of measurement and glass making, is now completely additive in all values. The Renotated scale is already incorporated in the *Lovibond Schofield Tintometer*, and will be used in all new *Tintometer* models. A simple conversion table from old to new scales is being issued free of charge for all users of existing *Tintometers*.

**Thomas Industrial Automation Limited** has recently announced an addition to their range of level indicators and controllers. The *CS 100|I/D/H| Leveltron*, as well as the usual TIA features, offers the facility of high and low level control using only one electrode and one controller.

The extent of the differential depends on the dielectric constant of the material being measured, but may be as great as 90 per cent of the total electrode length in some cases. The low and high level points are independently adjustable by means of controls fitted on the front of the instrument.

A new range of process inks for the offset quality printing market had recently been announced by **Fishburn Printing Ink Company Ltd.** The *Vivaking* range is claimed to produce very high gloss results on the majority of coated stocks, with freedom from set-off and drying problems. Emulsification and scumming are said to be minimised, and high fidelity print quality obtained.

**Spelthorne Metals Ltd.** has recently announced that, following redevelopment of its Rickmansworth factory, its address is now Church Street, Rickmansworth, Herts.

During the past two years the factory and warehouse have been rebuilt and equipped with new processing plant and handling equipment. The final stage has been the construction of offices and laboratories into which the Berkeley Street staff have now moved.

It has recently been announced that, in the interests of greater efficiency and better service to their customers, the headquarters, management and administration of **Colthurst & Harding Limited** and **Gaymel Paints Ltd.**, both members of the **Pinchin Johnson Paints Group**, will be combined and located at Bath Road, Bristol. Integration will be complete by 1 January 1969.

On 9 October 1968, a number of paint manufacturers and members of the Press visited the factory of **Joseph Mason Limited**, Derby, to view a new method of paint storage using tanks manufactured by **Porter Lancastrian Limited**.

The tanks, which are available in a variety of sizes from 36 to 180 gallons, comprise an outer shell of spun aluminium with a disposable liner made of polythene.

The liner is inserted in its rolled state through a hole at the bottom of the tank. This is closed by a door which seals into the spout of the liner and carries the outlet connection.

The paint is removed from the tank by introducing compressed air at about 10-20psi to the outer shell. This squeezes the liner and so removes the paint.

The advantages claimed for this system are: paints can be stored without the formation of skins since air can be excluded from the liner; virtually all of the paint can be removed from the liner in a useable condition, at the most half pint of paint is lost per batch; no container cleaning is necessary, since the liner is disposable and cheap to replace.

Joseph Mason Limited claim that the saving in paint with this method is about 5 per cent.

The main disadvantage of the system appears to be that since the system is totally enclosed there is no means of mixing the paint once inside the liner. Therefore, paints with even the slightest tendency to settle cannot be stored by this method.

# **Bristol**

### **Radiation curing of paint films**

The 163rd meeting of the Bristol Section took place at the Royal Hotel, Bristol, on Friday 27 September 1968, under the chairmanship of Mr D. S. Newton, when a paper entitled "Radiation curing of paint films" was given by Dr F. L. Dalton of the United Kingdom Atomic Energy Authority. Dr Dalton commenced by describing how the techniques of electron beam curing could overcome some of the disadvantages of conventional coating methods, in terms of speed, costs and efficiency.

He described how the electron beam was produced by a thermoionic gun operating in vacuum and how it was projected into normal atmosphere through a metal window which must be thin enough for beam penetration, but also sufficiently thick to withstand the vacuum pressures. In practice the metal window consisted of titanium or magnesium/aluminium alloy of approximately 0.001in thickness, and the beam was "manipulated" to cover the workpiece by means of a scanning device, or a linear emitter.

In the curing of coatings of average paint film thicknesses voltages of approximately 300kV were required and these could be obtained with conventional transformer/rectifier equipment. However, the curing of thicker coatings required higher energy, in the order of 500-600kV and this required the use of specially developed and more expensive transformers.

Using present techniques some loss of efficiency was encountered in curing very thin films of under 0.001in. For instance a coating of 25 micron would only require a voltage of 40kV and this was much below the minimum energy of 100-150kV necessary to emit the beam into normal atmosphere.

In discussing production techniques, Dr Dalton said that it was possible to obtain line speeds in the order of 15ft per minute, which had been found suitable for wood and furniture finishing. One of the advantages of radiation curing techniques was the rapid hardening of the coatings and consequently dust contamination and stacking difficulties were practically eliminated. Further, the treatment of coatings on heat sensitive substrates raised no problems since the amount of energy could be governed to treat only the surface coating and not affect the substrate.

Dr Dalton went on to describe a typical layout of an electron beam unit for line production, in which he indicated that certain safety precautions should be taken, since X-rays were produced when the electrons were reflected and adequate protection for the operators must be provided.

Radiation curing techniques were suitable for systems capable of polymerisation by free radical reactions. Unsaturated polyester resins of the type used for wood finishing were wholly suitable and much of the initial work had been applied to this particular field; as no catalyst and promoter were required, one-pack polyester finishes were used primarily.

Acrylic systems had also been investigated and Ford of Detroit were cited as having published patents covering acrylic based finishes. Normally, solvent free systems were required, but where this was not possible the solvent present should be capable of reaction.

Epoxy-polyurethane systems which cured by condensation reactions were not wholly suitable for radiation cure in air owing to oxidation side effects when high voltages were involved.



Dr Dalton finalised the discussion by mentioning the possibility of electron beam curing in vacuum, which would overcome many of the problems associated with projecting the electron beam into air. Epoxy phenolic systems had been cured by this means and speeds of up to 600ft per minute were a possibility.

Dr Dalton's fluid presentation aroused considerable interest and in the discussion opened by Mr H. Foster he answered many questions relating to the details of the process and its practical applications.

A vote of thanks was proposed by Mr G. R. Duckett and this was supported by all those present.

F.E.R.

## Hull

### Joint meeting

The first ordinary meeting of the Hull Section was held on 7 October at the College of Technology, Hull. It took the form of a discussion between the Association, headed by the Section Chairman, Mr E. A. Brown, and the local branch of the National Federation of Master Painters, headed by its chairman, Mr F. A. Wells.

A wide selection of questions was put forward for discussion, including: the possibility of introducing quicker drying finishes for use in winter conditions without any sacrifice in quality; the proportion of the total cost of a job due to preparation, application, overheads and paint cost; why pigmented polyurethane finishes were being introduced when they were considered unsuitable as clear varnishes for timber under outside conditions; the sale of paints complying with the new Building Regulations requiring Class 1 Fire Protection.

Many members of both the Association and the Federation took part in the discussion, which finally had to be closed by the Chairman.

Twenty-one OCCA Members, six Federation Members and four visitors were present.

S.R.F.

## Midlands

### Trends and developments in automobile finishes

The first technical meeting of the current session was held at Birmingham Chamber of Commerce on Friday 20 September. The chairman, Mr D. J. Morris, introduced Mr B. J. Nowlan who was to speak on "Trends and developments in automobile finishes."

Commencing with pretreatment, Mr Nowlan said that this was a very specialised subject on which he would not dwell. Developments accompanying the introduction of electrophoretic primers had included the use of chelating agents like tannic acid and  $\alpha$ -nitroso- $\beta$ -naphthol. Attempts to incorporate chelates in paints had not so far met with success.

In the field of primers, solvent based alkyds, applied by slipper-dip, were being increasingly supplanted by electrophoretic materials. There were two methods of tank control, namely the amine deficient system and the membrane control system. Hitherto the amine deficient system had involved high viscosity materials which were difficult to handle, but low solids, low viscosity, material had been introduced and used successfully in America. The present trend was to use no supplementary electrodes and often to use two-stage voltage systems. Earlier the media had been maleinised oils and water soluble alkyds. More recent developments included styrenated alkyds, epoxy esters and resinous polyols.

There was interest in developing high build electrophoretic primers to eliminate the need for a primer-surfacer. The cheaper system would seem to be a low build primer, followed by a primer-surfacer, but figures were not available. Whilst conventional primers tended to hide defects in the panel, electrophoretic materials followed the contours.

There had been little interest in water-borne primer-surfacers, and conventional materials had better hold-out for use under acrylics. Developments had followed the sequence: alkyds, styrenated alkyds, epoxy esters and styrenated epoxy esters. There was some interest in resinous polyols based on styrene and allyl alcohol but sinkage problems had not been resolved. The banning of lead and chromate pigments had stimulated interest in other anti-corrosive pigments.

In the field of enamels there had been a progressive change-over from alkyd/amino systems to acrylics. Thermoplastic acrylics had been developed with a narrower spread of molecular weights and having better gloss. Thermosetting acrylics had been improved with respect to petrol resistance and improved melamine-formaldehyde resins had been developed for acrylic systems. Great interest had been shown in the bake-sand-bake process, where reflow was able to absorb overspray, and this had led to the development of enamels for "repair in process." "Repair in process" enamels would eliminate the need for masking and removal of the car from the main line, but the process would entail considerable modification of existing lines. The development of acrylic organosols, with higher solids contents than present thermoplastic acrylics, would be very suitable for "repair in process" but there were stability problems to be overcome. The growing demand for metallic finishes was likely to stimulate further developments in acrylics.

There was interest in electrophoretic enamels for use as single-coat systems on components such as wheels.

For refinishing enamels the cellulose-synthetic types were being supplemented by air-drying acrylics and acrylic modified synthetic systems. There was a trend away from small spray shops towards the use of catalysed low-bake curing, but a growing interest in air-assisted electrostatic spraying was unlikely to bring about the demise of the spray gun, which was versatile and adaptable to change.

At present a body required the assembly of 20 panels but a basic unit could be made from two plastic pressings and be much lighter. There would no longer be corrosion problems but painting would be required for protection against u.v. and to give a colour range. Since plastics distorted at 180°F, paints requiring conventional stoving schedules would be unsuitable. Force-dry acrylics would be satisfactory.

Radiation curing could basically alter the approach to car finishes. It could be used with plastics and give integral coats by graft polymerisation. Initially there were likely to be application problems because the monomers were unpleasant and the prepolymers tacky and awkward to handle.

Car finishes could also be affected by external influences like safety considerations and government legislation on air-pollution could restrict the materials permitted in paints, but the conditions in Los Angeles, which had led to the introduction of rule 66 in the United States, were exceptional.

The interest which Mr Nowlan's talk had aroused was reflected in the large number of questions asked during the discussion period. Mr J. R. Green expressed the appreciation of the meeting in a vote of thanks.

L.R.S.

## **Trent Valley Branch**

### **Electrostatic spraying**

In an attempt to create wider interest in the branch, and also to increase membership, Trent Valley branch has this year organised technical meetings in other centres as well as Derby.

The first technical meeting of this session was therefore held at Loughborough University on Thursday 26 September. The facilities provided at this venue in the Edward Herbert building were excellent. Thirteen members and 16 guests were present to listen to a talk on, and practical demonstration of "Electrostatic spraying," by Mr R. C. Leivers of Volstatic Limited.

Mr Leivers first explained the general principles of electrostatic spray painting, which depended on the fact that electronically charged particles of unlike sign attract each other. To apply this principle to practical production line finishing, finely atomised paint particles were charged to a high negative potential with respect to the object to be coated.

In consequence the particles were moved by electrostatic forces to envelop the surfaces of the object.

This type of spraying had the advantage that all surfaces of that article being sprayed, were completely covered, including the rear and sides, also that paint losses were very low compared with other paint spraying systems since there was no overspray.

Pure electrostatic spraying had one major drawback, the inability to spray coat deep recesses due to the "Faraday cage" effect.

The speaker explained how this drawback had been overcome by surrounding the sprayhead with a low velocity air cap which forced the paint particles forward. By using various types of sprayheads, therefore, it was now possible to spray electrostatically a much wider variety of articles than before. It was explained that in electrostatic spraying the size of the atomised paint particle was important, the smallest possible particles being the most efficient. Recent improvements in this direction had been achieved with airless electrostatic and also hot airless electrostatic equipment. Such equipment was being used commercially to apply paint to structural steel, such as girders, with a controlled uniform film thickness, at an economic cost due to savings in labour and materials.

After the talk one of Mr Leivers' colleagues adequately demonstrated the principles discussed.

Much of the discussion afterwards by members and guests concerned the types of articles which could be coated by various electrostatic means.

J.R.B.

## Thames Valley

### **Pretreatment for electropainting**

The opening meeting of this winter session was held at the Royal White Hart Hotel, Beaconsfield on 26 September. A large audience heard Mr D. James, of Pyrene, speak on "Pretreatment for electropainting."

He began by saying that during the early development of electropaints, workers in the field thought it might be possible to dispense with the pretreatment, but it was soon recognised that some form of pretreatment was essential in order to retain adequate corrosion resistance. A zinc phosphate treatment was generally preferred for steel, but the selection of the appropriate type for a particular set of circumstances was a matter of some complexity.

Various attempts had been made to produce the ideal process for all electropaints, with singular lack of success. One technique of some promise was to apply a conventional zinc phosphate coating at 250mg per square foot and then with the work piece cathodic and still immersed in the phosphate bath to deposit metallic zinc. In spite of the good corrosion resistance, commercial exploitation had been prevented by failure of the zinc to "throw" in recessed areas and by failure of some electropaints to be compatible with the zinc rich surface.

Mr James then went on to discuss the phenomena of dissolution of part of the zinc phosphate coating. His company had noted dissolutions varying between 10 and 80 per cent of the original coating weight. The exact mechanism of the removal of the coating into the paint film (only traces appear in the paint bath) was not fully understood, and it was fortunate that neither corrosion resistance nor physical properties were affected adversely. Workers in Germany had used radioactive phosphorus  $P_{32}$  to study the problem and some typical data were quoted:

Original zinc phosphate coating	..	..	..	100.0%
Retained on steel after painting and stripping	..	..	..	86.02%
Detected in paint coating	..	..	..	10.41%
Found in paint bath	..	..	..	1.20%

The salt spray resistance of electroprimers was improved by an increase in the zinc phosphate coating weight, though not linearly. Of course factors other than corrosion resistance needed to be considered, for example, visual imperfections appeared, and the mechanical properties of a system might deteriorate if too high a coating weight was attempted for a given paint, and again, a higher coating weight was more expensive. Of more importance was the need to try to maintain a uniform coating weight over the total surface. It might be concluded that for optimum results in a given situation the pretreatment must not be considered in isolation but rather in relation to the electroprimer that was to be used and to the degree of protection that was necessary for an adequate job.

Mr James also mentioned some work reported by the British Aluminium Company Limited at the IMF Brighton Conference on the electropainting of aluminium and its alloys, in which chromate/oxide coatings from a fluoride/phosphate/chromate process yielded excellent defect-free paint films based on alkyd, alkyd/melamine and phenolic resins.

He ended with some references to studies of poor chromate rinsing. If chromate ions were allowed to contaminate the paint bath the consequences could be most unfortunate. Furthermore the chromate rinse should be most carefully selected and the demineralised rinse should not follow immediately, otherwise its benefit could be lost. If hexavalent and trivalent chromium ions were used, however, the gain in corrosion resistance could be as great as 25 per cent even after an immediate demineralised rinse.

The talk concluded with some excellent slides of large plant installations both in Europe and Britain.

Questions covered the whole subject and were put by Messrs. Turnhill, Skinner, Inshaw, Evans, Moore, Simpson, Cooke, Armitage. In answer to one questioner a colleague of Mr James explained his view of what happened during dissolution.

The vote of thanks was proposed by Mr J. A. Harris.

R.E.G.

## West Riding

### Accelerated weathering of paints

The first Technical Meeting of the 1968/9 session was held in the Griffin Hotel, Leeds, on Tuesday 10 September 1968. Mr L. H. Silver, the Section Chairman, introduced the speaker for the evening, Mr M. Hipwood of the Paints Department of the Chemical Inspectorate, who gave a paper on the "Accelerated weathering of paints."

Mr Hipwood explained that he would review a number of investigations carried out by an Inter-Services Research and Development Committee, which had been appointed to look into the subject of accelerated weathering—this investigation

leading to the publication of British Standard 3900: F3—and then to continue with a review of further work carried out using this new cycle in comparison with some of the cycles developed in the USA and UK. Service equipment should of course function satisfactorily in extremes of environment, so naturally the Services were interested in methods of test to evaluate the durability of coating materials etc. Practical exposure trials were considered essential for such evaluation and a site producing the most degrading conditions was selected, the coating exposed for some specified period and then evaluated. Florida exposure was accepted by the automobile manufacturers, but this was not only expensive but also not very standard as a method in that one year's climate was not necessarily the same as the next, and further, with modern coatings, one year might not be a sufficiently long period. For these reasons there had been a great deal of work carried out by several groups of workers, in an effort to find a method of carrying out this exposure in a shorter period of time.

The first results were reflected in Ministry of Defence Specification DEF 1053 Method 26, familiar to most Members of the Association, and this was most valuable for checking paints of known composition, enabling one to detect changes in raw materials after a testing cycle of about six weeks. This cycle had many limitations, especially where the coatings were of unknown composition, and the continuous water-spray could produce severe blistering with multi-coat systems.

Because of these shortcomings a further committee was established in 1958 to find a more reliable method, and their first recommendation was to keep the same basic testing equipment but to modify it somewhat—in this way saving a great deal of expense for the great number of paint manufacturers who had already invested in the original equipment. The first part of this work was reported in the February 1964 issue of the *Journal*, and for the benefit of those present unfamiliar with this work Mr Hipwood gave a brief resume. The committee then extended its field to cover contemporary coating techniques; epoxy, polyurethane, etc., and, using the newly found modifications to the DEF 1053 Method 26 Spec. i.e. the 2/10 cycle, the 4/4 cycle, the xenotest cycle etc, a programme was arranged to compare these with actual exposure tests at various sites in the UK and abroad, the latter being at Singapore, Cloncurry in the Australian desert and Innisfail—a tropical site in Queensland, Australia.

The conclusions reached from this programme were that if a paint was a bad paint then the failure would be reproduced at all sites, except that the severity of failure varied with site, and overseas sites were in most cases at least three times as severe as the Glascoed site in Wales. The 2/10 cycle appeared to be a reliable cycle for the prediction of performance of coatings exposed in this country, although it was rather light with some of the strong coloured finishes—but it was certainly a significant advance over DEF 1053 Method 26 and the cycle was recommended to the BSI who subsequently adopted it as Standard 3900 part F 3. The Standard was then subjected to comparative tests by the member countries of the ISO with each contributing body's own preferred test: when the collated results were examined recently it was found that the new Standard was eminently acceptable to all concerned and it was now being written up prior to adoption as an ISO Standard.

To round off the paper Mr Hipwood reviewed the cycles used in other areas, starting with the carbon arc cycle then developing into the more sophisticated systems. These were the ASTM method, including the dew cycle modification, the xenon arc, mercury discharge lamp, fluorescent lamps—marketed as Climatest by De La Rue Frigistors, and biased more towards use by the Plastics Industry—and finally the EMM-Aqua system.

Giving his conclusions, Mr Hipwood said that the carbon arc lamp was more successful with paints based on rutile  $TiO_2$ , perhaps because the main emission band was 385 nanometers and this was extremely important with paints containing  $TiO_2$ . The ASTM was more realistic for the US contributors because natural conditions of

hot continental climate related more closely to the artificial accelerated test, but the British Standard method was to be preferred for exposures relating to temperate climates. The dew cycle had been disappointing in that it had not discriminated in the correct order of performance, and Mr Hipwood's own opinion was that the 4X Filters should not be removed. The mercury arc, because of its line type emission, was perhaps too selective in breakdown of certain types of polymer, the xenon arc was moving in the right direction but there was still the fall off in spectral energy in the uv region and this was why it was not a very repeatable test, but the fluorescent lamp idea was going to be a useful one for testing plastics.

The Joint Services Committee was still very active, although the research so far had been of a very empirical nature and they had been making assessments of breakdown, not measurement, and the next step would be to define methods of measuring film breakdown quantitatively. The techniques being investigated so far were the ICI micro-indentation technique for measuring the mechanical properties of a film, which was very useful in that it could be carried out on the test panel, unlike the tensile strength method where the film had to be stripped from the panel with a possible relaxation of stresses between the panel and film, and other unknown effects, weight-loss measurements, and infra-red techniques to record the chemical changes occurring in the film during exposure to weathering.

Mr Hipwood acknowledged the assistance of various members of the Inter-Services Committee and his indebtedness to the Chief Scientist, Ministry of Defence (Army), for permission to give the talk.

The paper was profusely illustrated by slides and in the discussion that followed the following members and visitors contributed—Dr Watkinson, Mr Gray, Miss V. M. Whitehead, Mr Drew, Mr McKean. The vote of thanks was proposed by Mr N. Cochrane.

J. N. MCK.

### **Printing on plastics**

At the second technical meeting of the West Riding Section, on 8 October, held jointly with the local branch of the Institute of Printing, a paper on the subject "Printing on plastics" was presented by Mr J. Hastings-Long. A large display of examples of production printing on all forms of plastic film, sheet, and mouldings, together with illustrations of roller and dip coating techniques, was available for examination by the audience before and after the lecture, and these greatly contributed to the interest of the subject and stimulated discussion at the question time which followed.

Mr Hastings-Long opened his talk by suggesting that the title of the paper should not be taken too literally but that the scope of the paper should include the decoration of plastics. Decoration was not confined solely to printing but also to spray, roller and dip-coating techniques and various in-the-mould methods of decoration, and many of these were carried out in conjunction with more conventional printing methods.

He outlined the various classes of plastic with which one came in contact in daily life and emphasised that when one talked of printing on plastics one was concerned basically with the major printing processes; letterpress, lithographic, dry-offset or letterpress-offset, flexographic, gravure and screen process printing. The plastics substrates on which these processes were applied were defined broadly as film, sheet and mouldings, and Mr Hastings-Long sub-divided the mouldings class into blow-mouldings and extrusions, injection mouldings, and rotational castings.

The main printing processes used on film were flexography and gravure printing, using a solvent/resin ink, and the machines could be roll or sheet fed, the latter being more common in gravure than in flexo. The only major advance in flexography had been the development from very simple solvent/resin systems to more complex bi-component systems to overcome problems such as ink-adhesion under conditions

of freeze-thaw in refrigerated foodstuffs packaging, or ability to withstand "boil-in-the-pack" techniques. With gravure printing one was again dealing with a relatively simple solvent/resin system, of rather different character to the flexo inks, but one which was capable of producing exceptionally high quality results. The major innovation here was the adaptation to printing an already moulded article, and as examples Mr Hastings-Long cited the printing of woodgrain patterns on an injection-moulded kitchen-knife handle, or a vacuum-moulded TV cabinet where the presses had been adapted to print on the pre-shaped material.

Printing on sheet or heavier gauge films would be expected to be by normal letterpress and lithographic processes, but unfortunately this was where the ink-maker and printer usually came unstuck because such substrates were usually more difficult to handle and also presented adhesion problems. One of the few successful applications of litho-printing on clear plastic sheet was in the medical education field where various organs of the human body were printed on clear sheet, and thus could be superimposed, one on the other, to present a three-dimensional representation to the student.

Printing by letterpress or litho on flexible plastic sheet failed mainly because of the problem of plasticiser migration to the surface of the sheet, thus an initially satisfactory print was found on storage to develop rubbing problems, etc. However, there were certain materials on the market where satisfactory prints could be obtained by letterpress and litho, and Mr Hastings-Long showed the new polyolefin sheet from Bakelite-Xylonite Ltd. which was almost indistinguishable from art paper.

The screen printing process has undergone a tremendous revival of late, due to the difficulty of printing plastics by other methods, and this was accelerated by the advent of the blow-moulded container. The major factor was the adaptability of the process to enable an ink to be produced from virtually any combination of the raw materials available today, thus enabling one to print on vinyl with a vinyl ink, on acrylic with an acrylic ink, etc. It was not easy to envisage printing letterpress with an acrylic ink on acrylic sheet, but with screen process this was perfectly possible, and there was practically no limit to the size or shape of the article to be printed, as could be seen from some of the articles on display. Screen printing on blow-mouldings had now largely superseded the earlier method of dry- or letterpress-offset printing, whose main disadvantage was the slowness of production due mainly to lengthy drying times. Modern screen printing produced work at the rate of 5,500 units per hour, in four colours in perfect register with intermediate drying stages between each print, and resulting, in some cases, in a printed container which could be filled at the terminal stage of the printing run.

Two developing techniques in the screen-printing field were concerned with the production of a printed image which was diffused into the surface of the plastic, thus producing a print which was to all practical purposes permanent until the surface was physically abraded to the depth to which the image had penetrated. One technique was developed from the textile printing of polyester fibres and this had now been adapted to the printing of sheet plastics produced from polyester, polyacetal, certain polyamides and grades of cellulose acetate butyrate. The system was complicated by the fact that one was working with dyes rather than with pigments, therefore there could be no opaque prints and a white was impossible, thus making it important to choose with care the background on which one was going to print. A bright transparent red on a green background was certainly not going to be possible. However there was a choice of application available—screen, dry-offset, or spray-printing. After each application the material must be stoved at a temperature appropriate to the substrate, and the image was produced by dye transfer from ink medium to substrate with the aid of the vehicle, which was a vital part of the system. The only disadvantage at present was that after processing there was an excess of ink, which must be washed off with water. A major use at present was in the marking of nylon gear-wheels with

a manufacturer's code number without mechanically affecting the performance of the item.

The second technique was the one specifically for use on untreated polypropylene, which had the advantage over its predecessors of retaining a sharp image after up to two years storage, whereas earlier efforts had resulted in a continuing migration of the printed image downwards and sideways giving a progressively hazy and ill defined print. This technique was only evolved after testing a wide range of potentially suitable dyes and selecting those with the necessary non-migratory properties—resulting in a very limited shade range.

Mr Hastings-Long then went on to describe in detail the problems of screen-printing as applied particularly to the production of signs, and described techniques of sandwich printing with acrylic monomer inks, producing the effect of opal perspex by an over-lacquer printing on glass reinforced polyester material, etc.

Coming to the non-printing aspects of decoration of plastics, Mr Hastings-Long showed examples of spray-painting, namely a turbo-fan heater case in high-impact polystyrene spray-painted in part and printed in part; a TV cabinet masked and sprayed and then over-printed with metallic foil by hot-blocking technique; a doll in untreated polypropylene to which had been applied a special primer followed by a two-component spray paint system, the dolls head being sprayed with a pvc finish; a hammer finish on rigid pvc; a reinforced polyester safety helmet spray painted with a two-component system; spray-painted rear-light assemblies for autos; moulded ABS components, spray-painted with special primer prior to vacuum metallising; and several others. Dip and roller coating techniques were also briefly touched upon and examples shown of marble effects, produced by the latter method, on chip-board substrate.

In the discussion that followed Messrs. Groom, Khan, Kay and Jones contributed and Dr L. J. Watkinson, a former colleague of Mr Hastings-Long, proposed the vote of thanks.

J.N.MCK.



# **President leaves on world cruise**



**From left to right: Mr F. Sowerbutts (President), Mrs Sowerbutts, Mr I. C. R. Bews (Hon. Secretary) and Mr R. H. Hamblin (Director & Secretary)**

On 28 October the President, Mr F. Sowerbutts, and Mrs Sowerbutts left Southampton on the "Northern Star" for a private sea cruise, which had been arranged before Mr Sowerbutts became President. During the course of the cruise Mr and Mrs Sowerbutts will leave the "Northern Star" in order to visit the Sections in South Africa and New Zealand and also the Oil and Colour Chemists' Association Australia.

Mr Sowerbutts is taking with him the

Commemorative Messages received by the Association at its Fiftieth Anniversary Celebrations in May, and the Sections in both South Africa and New Zealand as well as OCCA Australia have arranged functions to coincide with his visit.

Previous occasions on which OCCA Presidents in office have visited Australia and New Zealand were Mr P. J. Gay in 1960 and Mr L. O. Kekwick who also visited South Africa during the course of a business tour in 1952.



Seen at London Airport: (left to right), The President, Mrs Sowerbutts, Mrs Penfold, Mr A. R. Penfold

### Mr A. R. Penfold returns to Australia

On Monday 7 October, Mr A. R. Penfold (Honorary Member both of the Association and of the Oil & Colour Chemists' Association Australia) and Mrs Penfold left London Airport to return to their home in Sydney. Mr and Mrs Penfold have been in the United Kingdom since early May when they attended the Jubilee

Celebrations on behalf of OCCA Australia.

They were seen off at the Airport by the President, Mr F. Sowerbutts, and Mrs Sowerbutts, who, later in the month, left on their private sea cruise.

Also present at the Airport was the Director & Secretary, Mr R. H. Hamblin, who took the photograph.

## Jordan Award

The Committee appointed by Council to consider the applications received for the Jordan Award met in February and, after long and careful consideration, decided that it was not possible to make the Award on this occasion.

It is intended, therefore, to invite further applications to be received in 1968, and the Committee will consider these early in 1969, with a view to making the Award at the Eastbourne Conference, which will be held 17-21 June 1969. The rules of the Award are appended below for the benefit of new members.

1. The Award (**which on this occasion will be £100**) will be made for the best contribution to the science or technology of surface coating by a member of any nationality working in either the academic or industrial

field who is under the age of 35 at the date of application.

2. The final date for submission of applications will on this occasion be 31 January 1969.
3. The selection of the recipient of the Award will be made by a Committee under the Chairmanship of the Association's Hon. Research and Development Officer.
4. There will be two methods of application. First, by the submission of a paper describing original work by the candidate which is offered for publication in the *Journal* or has been so published during the two years prior to the closing date for application. The alternative method will be by recommendation by a superior for work which for reasons

of commercial secrecy cannot be published; in this case the candidate will be expected to submit a dissertation on a topic relating to his work and demonstrating his superior knowledge of the principles thereof. The Award is for *individual* merit

and clear evidence of the candidate's own contribution will be required if a paper is offered under joint authorship.

5. Applications should be addressed to the Director & Secretary at the address shown on the front cover.



## TECHNICAL EXHIBITION

Since the publication of the first list of exhibitors for 21-OCCA in the October issue of the *Journal*, alterations have taken place as follows:—

British Oxygen Chemicals Limited, Bakelite- Xylonite Limited and A. Revai & Co. (Chemicals) Limited should be deleted and Kollmorgen (UK) Ltd., Prüfbau, Styrene Co-Polymers Limited and S.P.C.M. Matieres Colorantes de Mulhouse should be added to the list.

21-OCCA, the Association's Twenty-First Technical Exhibition, will be held from 24-28 March 1969 at Alexandra Palace, London N22, and will be the largest ever organised by this Association, with 107 Stands and exhibitors from ten overseas countries.

#### SIX-LANGUAGE CARDS

In order to help overseas exhibitors and visitors cards in six languages (English, French, German, Italian, Russian and Spanish) have been prepared, showing the route to Alexandra Palace.

These will be sent to many thousands of addresses on the Continent, and anyone wishing to send one of these to an associate overseas may obtain one without charge from the Association's offices.

#### OFFICIAL GUIDE

Copies of the *Official Guide* are now being prepared and will be despatched to members early in the New Year. Non-members may obtain a copy free of charge from the Association's offices.

#### EUROPEAN LIAISON LECTURE

The London Section of the Association will be holding its European Liaison Lecture on Thursday 27 March 1969 at 18.00hr in the Alexandra Room, Alexandra Palace, London N22. The title of the lecture will be the "Progress of electrodeposition in Europe" and the lecturer will be Dr Karl-Heinz Frangen. A licensed bar will be available for members after the lecture and an applica-

tion form will be enclosed in each copy of the *Official Guide*.

#### EXHIBITION LUNCHEON

The details concerning the Exhibition Luncheon, which is to be held at the Savoy Hotel, London WC2, on Monday 24 March, with Lord Kings Norton as

Guest of Honour, will also be enclosed in each copy of the *Official Guide*. It is stressed that attendance at the Luncheon is not restricted to exhibitors only, and an application form for tickets, price £3 each, will be enclosed in each copy of the *Official Guide*.

## Eastbourne Conference

### “Film formation and curing”

At the meeting of the Council on 23 October, Council decided that the registration fees for the Eastbourne Conference (17-21 June 1969) shall be:

Members:	£10
Wives:	£5
Non-Members:	£15
Student Members:	£5

Special arrangements will be made for members of the London and Thames Valley Sections only wishing to attend

on particular days.

Full details of the lecture programme with summaries of the lecture and biographies of the authors will appear in the January 1969 issue of the *Journal* and registration form, (giving full details of all functions arranged and fees) will be sent to all members early in January also.

Non-members wishing to receive a copy should write to the Association office at the address shown on the front cover.

## Scottish Section

### Dispersion Symposium

The Scottish Section is planning a symposium on “Dispersion in theory and practice,” to take place in 1970. The chosen dates are 21-23 May and the

venue will be the new town of East Kilbride (near Glasgow), where excellent modern facilities are available. Speakers of international reputation will be invited and particular emphasis will be placed on discussion.

## Obituary

### Mr G. Johnson

It is with regret we record the death, on 25 September, of Mr G. Johnson, an Ordinary Member attached to the Manchester Section, and Member of the

Section Committee in the early years after its formation.

Mr Johnson, who was 80, was Technical Director of Isaac Bentley and Co. Ltd. from 1941 until his retirement in 1955.

### News of Members

Dr H. Rechmann, an Ordinary Member attached to the General Overseas Section, has been elected Chairman of the Paint

and Pigments Group of the German Chemical Society. He took office on 1 October 1968, and will hold the position for a period of two years.

### Federation of Societies for Paint Technology

The Federation of Societies for Paint Technology has recently issued a "State of the art review" entitled "Recent

developments in architectural and maintenance painting."

Copies are available from the Federation's office in Philadelphia, at a price of \$1.25 each, or \$1 each if 100 or more are ordered.

## Association Notices

### Applications for membership

It is felt that the members would like to be reminded of the standard of competence for the election of candidates to Ordinary Membership of the Association, as laid down by the Council, when they are sponsoring candidates for election. The qualifications for the granting of Ordinary Membership at the present time are:

1. A degree in a scientific subject or any generally accepted equivalent qualification; or an approved technological qualification in a subject covered by the Association.

2. Or where there is adequate evidence of the technical competence of the candidate other than the obtaining of the qualifications mentioned above, the qualifying period of practice in the industries covered by the Association shall be normally not less than seven years.

Associate Membership is open to those employed in the industries who do not qualify for Ordinary Membership.

The Council has further resolved that Student Membership should be open without restriction to the age of 21 years and may be extended to 25 years of age, where candidates are following courses of technical study to the satisfaction of their employers or technical college lecturers.

### Retired members

Council also wishes it to be widely known that in 1962 it introduced a reduced membership subscription rate for members who have retired from business. This applies to a member who has completed 20 years as an Ordinary or Associate Member and has retired

from business, and normally has reached the age of 60; he may apply for his name to be retained on the Register of Members at an annual subscription rate of £1 1s and he will retain the same rights of membership as the class of membership to which he was attached upon retirement.

Members wishing to avail themselves of this concession should write, in confidence, to the Director & Secretary at the address shown on the front cover of this *Journal*, giving the relevant information under the four headings: (a) name, address and Section, (b) date of election, (c) date of retirement, (d) age.

### Change of address

Members changing their address are urged to inform the Association's office immediately so as to avoid any misdirection of mail. This is particularly important as far as the *Journal* is concerned.

Will members please note that since membership of the Association is entirely on an individual basis, if notification of the change of an address for a company is sent to the Association's office this will not necessarily guarantee the change of address in the Association's records of the member concerned unless the name of the member is stated on the communication.

### Binding of the "Journal"

Members will be pleased to know that W. Heffer & Sons Ltd., Hills Road, Cambridge, will undertake the binding of back volumes of the Association's

*Journal* sent in by individual members at a cost of £1 10s per volume.

Members wishing to avail themselves of this facility should send the parts direct to W. Heffer & Sons Ltd., enclosing a remittance of £1 10s and ensuring that notes bearing their names and addresses are enclosed with the parcels.

### 1969 members' subscriptions

Members are reminded that 1969 Membership subscriptions to the Association are payable on 1 January 1969. Forms are despatched to all members in October and November, depending upon address.

New members may like to know that

the Commissioners of Inland Revenue had approved of the Association for the purpose of the Finance Act, 1958, Section 16, so that a member subject to United Kingdom income tax is entitled to a deduction from the amount of his emoluments assessable to income tax under Schedule E for the whole of his annual subscription to the Association, provided the subscription is defrayed out of the emoluments of his office or employment and that the interests covered by the objects of the Association are relevant to such office or employment.

Claims for adjustment of PAYE code numbers should be made on form P.358, copies of which may be obtained from local tax offices.

## Register of Members

The following elections to membership have been approved by Council. The Sections to which the new members are attached are given in italics.

### Ordinary Members

BARRON, JOHN RICHARD, BSc, Geigy (UK) Ltd., Hawkhead Road, Paisley. (*Scottish*)

BARTRAM, ERIC, BSc, Flat 1, 369 Wilmslow Road, Fallowfield, Manchester 14.

(*Manchester*)

BOWLER-REED, JOHN, PhD, DIC, ARCS, BSc, FICorrT, The Bee Hive, Beehive Road, Binfield, Berks.

(*Thames Valley*)

GILES, PETER WILLIAM, BSc, 49 Esmond Grove, Dial Hill, Clevedon, Somerset.

(*London*)

GRAHAM, GERALD, BSc, 511A, Gale Street, Dagenham, Essex.

(*London*)

GRUNDY, KENNETH HENRY, BSc, MSc, PhD, ARIC, Development Department, Fibreglass Ltd., St. Helens, Lancs.

(*Manchester*)

KEELEY, JOHN WALTER, 92 Scribes Lane, Hall Green, Birmingham, 28.

(*Midlands*)

MACLEOD, ALISTAIR REID, 8 Struma Drive, Clarkston, Glasgow.

(*Scottish*)

MCILWAINE, PETER, Ault & Wiborg (Ireland) Ltd., J. F. Kennedy Drive, Bluebell, Inchicorb, Dublin.

(*Irish*)

MILLS, WINTON GORDON BOYCE, BSc, CEng, FRIC, MIChemE 28 Dunstall Road, Wimbledon, SW20.

(*London*)

POTTER, BRIAN, AMIME, K. W. Chemicals Ltd., 55/57 High Holborn, WC1.

(*London*)

PROSSER, JOHN LAWRENCE, BSc, 18 Dunleary Close, Heathside, Hounslow, Middlesex.

(*London*)

RAVEN, PETER WILLIAM, 76, Jubilee Avenue, Romford, Essex.

(*London*)

SHEPHERD, TOM, ARIC, 4 Gordon Crescent, Newton Mearns, Glasgow

(*Scottish*)

SIMON, JOHN, Ault and Wiborg (Ireland) Ltd., J. F. Kennedy Drive, Bluebell, Inchicorb, Dublin.

(*Irish*)

THOMSON, IAN A., 26 Laverockhall, Lanark, Scotland. (*Scottish*)

TURNER, DEREK FREDERICK, 19 Marmora Road, East Dulwich, London, SE22.  
(*London*)

WHEATLEY, KENNETH VALENTINE, BSc, Ault and Wiborg (Ireland) Ltd., John F. Kennedy Drive, Bluebell, Dublin 12. (*Irish*)

### Associate Members

KNAPE, WILLIAM, 14 Melrose Road, Island Bay, Wellington, New Zealand. (*Overseas*)

KOLB, WALTER, British Department, Dyes Division, CIBA Limited, Basle, Switzerland.  
(*Overseas*)

TEMPLETON, DONALD GEORGE, 8378 Shaughnessy Street, Vancouver 14, British Columbia, Canada. (*Overseas*)

### Student Members

BOATE, BRIAN WATSON, Ault and Wiborg (Ireland) Ltd., J. F. Kennedy Drive, Bluebell, Dublin. (*Irish*)

CARROLL, STEPHEN, Ault and Wiborg (Ireland) Ltd., J. F. Kennedy Drive, Bluebell, Dublin, 12. (*Irish*)

CLARK, IAN FERGUSON, 6 Belmont Road, Paisley, Renfrewshire. (*Scottish*)

COUGHLAN, DEREK CHRISTOPHER, Riverside Cottage, Killinghall Bridge, Harrogate, Yorkshire. (*West Riding*)

JONES, JENNIFER, 15, Manor Crescent, Knaresborough, Yorkshire. (*West Riding*)

LOKKERBOL, DAVID HENRY, 52 Windsor Street, Uxbridge, Middlesex. (*London*)

REYNARD, PAUL ANTHONY, 3, Station Square, Pateley Bridge, Near Harrogate, Yorkshire. (*West Riding*)

THOMSON, DONALD, 26 Glasgow Road, Paisley, Renfrewshire, Scotland. (*Scottish*)

VIVIAN, SIMON EDWARD, 135 South Park Crescent, Catford, London, SE6. (*London*)

## Forthcoming Events

*Details are given of meetings in the United Kingdom up to the end of the month following publication, and in South Africa and the Commonwealth up to the end of the second month.*

### Monday 2 December

**Hull Section.** "Surface Preparation," by Mr T. R. Bullett of the Paint Research Station, to be held at the Hull College of Technology (Bullock Lecture Theatre) at 7.00 p.m.

### Thursday 5 December

**Newcastle Section.** Joint Meeting with the Society for Analytical Chemistry, to be held at the Royal Turks Head Hotel, Grey Street, Newcastle upon Tyne, at 6.30 p.m.

### Tuesday 10 December

**West Riding Section.** "Exports in the Paint Industry," by Mr L. H. Silver of Silver Paint and Lacquer Co. Ltd., to be held at the Griffin Hotel, Boar Lane, Leeds 1, at 7.30 p.m.

### Wednesday 11 December

**London Section.** "Gel Permeation Chromatography," by Mrs S. M. Rybicka of the Paint Research Station, to be held at East Ham Technical College, High Street South, London E6, at 6.30 p.m.

*Thursday 12 December*

**Scottish Section.** "Chemistry in the Caribbean," by Prof. R. A. Raphael of Glasgow University, to be held at the Whitehall Restaurant, Renfield Street, Glasgow, at 6.00 p.m.

*Friday 13 December*

**Manchester Section.** "Modern Inks for the Packaging Industry," by Dr H. G. Smith of Fishburn Printing Ink Co. Ltd., to be held at the Liverpool Building & Design Centre Ltd., Hope Street, Liverpool 1, at 6.30 p.m.

**Irish Section.** "Crime Prevention," by Inspector Peter McGing, to be held at the Clarence Hotel, Wellington Quay, Dublin, at 8.00 p.m.

**Bristol Section.** "Some Factors Affecting the Opacity and Brightness of Emulsion Paint Films," by Mr J. Fairless of English Clays Lovering and Pochin Co. Ltd., at the Royal Hotel, Bristol, at 7.15 p.m.

*Saturday 14 December*

**Scottish Section—Student Group.** "Computers," by a speaker from I.B.M. (UK) Limited, to be held at the Lorne Hotel, Sauchiehall Street, Glasgow, at 10.00 a.m.

*Wednesday 18 December*

**Scottish Section—Eastern Branch.** "Crime Prevention," by Chief Superintendent Brown of the Edinburgh City Police, to be held at the Wee Windaes Restaurant, 142 High Street, Edinburgh, at 7.30 p.m.

*Thursday 2 January*

**Newcastle Section.** "Wallpaper—its Development, Manufacture and Design," by Mr R. E. Grime of Wallpaper Manufacturers Ltd., to be held at the Royal Turks Head Hotel, Grey Street, Newcastle upon Tyne, at 6.30 p.m.

*Friday 3 January*

**Bristol Section.** "Solvents: some further topics," by Mr L. A. Tysall of Shell Research Ltd., to be held at the Royal Hotel, Bristol, at 7.15 p.m.

*Monday 6 January*

**Hull Section.** "Developments in Emulsion Polymers," by Mr K. A. Safe of Vinyl Products Ltd., to be held at the Hull College of Technology (Bullock Lecture Theatre) at 7.00 p.m.

*Friday 10 January*

**Manchester Section.** "Emulsion Paints: Instrumental Aids to Binder Selection," by Dr C. Bondy of Revertex Ltd., to be held at the Swan Hotel, Churchgate, Bolton, at 6.30 p.m.

*Tuesday 14 January*

**West Riding Section.** A paper yet to be named, dealing with Solubility Parameters and the effects of Los Angeles Rule 66; by a speaker from Shell Research Limited, to be held at the Griffin Hotel, Boar Lane, Leeds 1, at 7.30 p.m.

*Wednesday 15 January*

**London Section.** "Aspects of the use of Cellulose Ethers in Emulsion Systems," by Dr Sven Lindenfors of Mo och Domsjo AB, Sweden, to be held at East Ham Technical College, High Street South, London E6, at 6.30 p.m.

**Manchester Section—Student Lecture:** "Currently Available Methods of Applying Surface Coatings," by Mr D. Stewart of Styrene Co-polymers Ltd., to be held at the Manchester Literary and Philosophical Society, at 4.30 p.m.

*Thursday 16 January*

**Irish Section.** Annual Dinner Dance at the Dublin Intercontinental Hotel.

**Scottish Section.** "Investigations of the Rheological Behaviour of Emulsion Paints," by Dr F. Wagener of Titan-gesellschaft mbH, to be held at the Whitehall Restaurant, Renfield Street, Glasgow, at 6.00 p.m.



**Thames Valley Section.** "Some Developments in Marine Painting," by Mr A. McIntosh of Shell Research Laboratories, to be held at the Royal White Hart Hotel, Beaconsfield, Bucks, at 7.00 p.m.

*Friday 17 January*

**Midlands Section.** "High Speed Coating Processes—Latest Developments," by Dr T. Williams of BISRA, to be held at 8.00 p.m. at the Winston Restaurant, Balsall Heath Road, preceded by dinner at 6.30 p.m.

**Scottish Section.** Annual Dinner Dance at the Central Hotel, Glasgow, at 6.30 p.m.

*Saturday 18 January*

**Scottish Section—Student Group.** "Colour Television," by Mr Alexander J N. Hope of Barr & Stroud Limited,

to be held at the Lorne Hotel, Sauchiehall Street, Glasgow, at 10.00 a.m.

*Wednesday 22 January*

**Scottish Section—Eastern Branch.** "Layout of Machines and Machine Types in the Paint and Ink Industries," by Mr D. P. Sullivan of D. H. Industries Ltd., to be held at The Wee Windaes Restaurant, 142 High Street, Edinburgh, at 7.30 p.m.

*Friday 31 January*

**Bristol Section.** "The Physical Testing of Printing Inks," by Mr C. C. Mill of P.I.R.A., to be held at the Royal Hotel, Bristol, at 7.15 p.m.

**Irish Section.** "Phthalocyanine Pigments," by Dr D. A. Plant of ICI Ltd., to be held at the Clarence Hotel, Wellington Quay, Dublin, at 8.00 p.m.

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- (C)—Correspondence  
 (D)—Discussion  
 (E)—Editorial and Comment  
 (N)—Notes and News  
 (R)—Reviews  
 (S)—Section Proceedings  
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Journal of OCCA (Bound) 1936-1967 (inclusive).  
Official Digest (Bound) 1947-1966 (inclusive).  
Reports on the Progress of Applied Chemistry 1940-1966 (inclusive).  
Chemical Society Annual Reports 1941-1953 (inclusive).

For further information write to Box No. 297.

### SITUATIONS WANTED

Ex-Managing Director of Public Company seeks position (full-time or part-time) on Board of Company wanting to modernise, expand or diversify. Wide industrial contacts; technical and legal qualifications. Box No. 294.

### AGENCY OFFERED

Dutch company, well established as supplier to paint and printing ink industries in Belgium and Holland, offers agency to British producers wishing to extend their markets in those countries. Box No. 295.

**21-OCCA TECHNICAL EXHIBITION** ● See page 1043

## SITUATIONS VACANT

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**WESTERN INK CO. LTD.** require an ink chemist aged 21 to 25 to join an expanding technical team.

Although having concentrated primarily on liquid inks in the past, we are developing in the oil-based ink industry as well and have a guaranteed future in this field. So if you are in our required age range, have final qualifications in ink technology, experience in letterpress/litho inks and can offer vision, energy and professional self-discipline, we in turn will make you a worthwhile offer. This could include renting a Company house or advantageous mortgage facilities.

Our Works are situated in Somerset countryside only 11 miles from the centres of Bristol and Bath.

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**The General Manager,  
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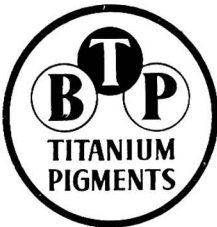
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The ideal applicant will be a graduate Chemist, with the enthusiasm and proven ability to sell to a consumer industry. An opportunity for acquiring the necessary technical know-how will be provided during the initial period in the Company's Technical Service Laboratories at Stockton-on-Tees, after which the man selected will operate from one of the major cities of the UK.

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● Nopco\* 1407 - an efficient, low cost paste anti-foam, compatible with most latex systems. Two to five pounds per 100 gallons of paint does the job. And it's FDA approved.

Nopco\* DD-72 - A new 99% active liquid defoamer requiring low levels of addition for

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