

JOURNAL

OF THE

OIL AND COLOUR CHEMISTS' ASSOCIATION



Vol. 49 No. 3

March 1966

Two papers from the 7th Australian Convention

Some studies in heterogeneous polymerisation
Electrodeposition of one coat paints containing titanium dioxide

The influence of additives and calcination on the pigment properties of titanium dioxide: part II

The microelectrophoresis of pigment particles

OCCA 18 14 - 18 March

for latest information and details turn to page 241

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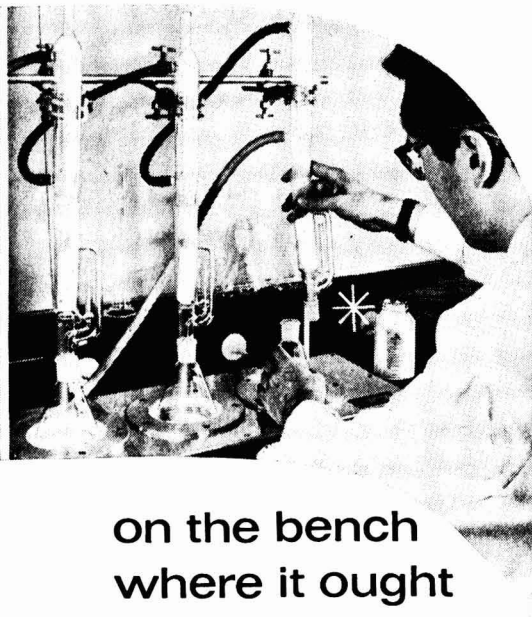
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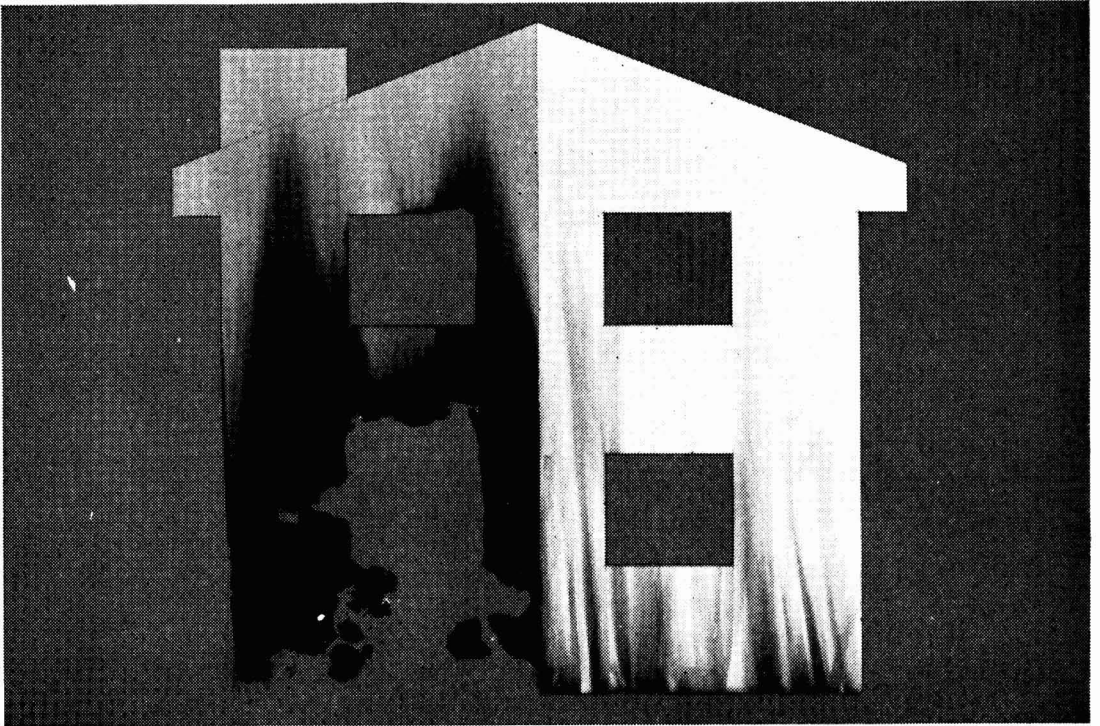
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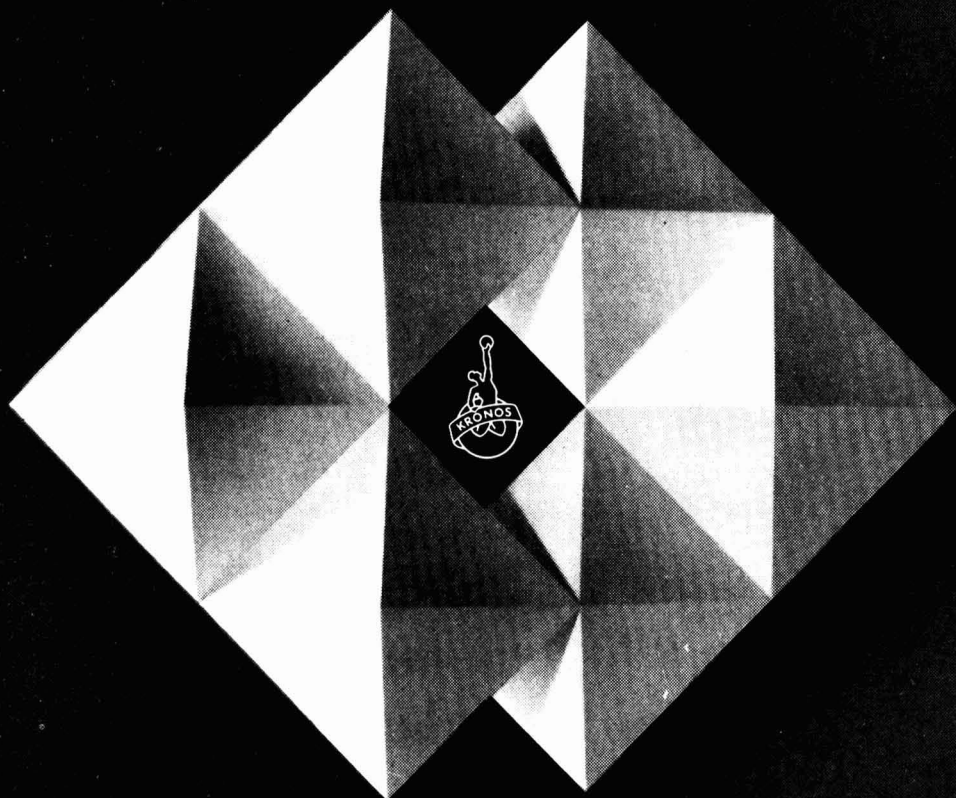


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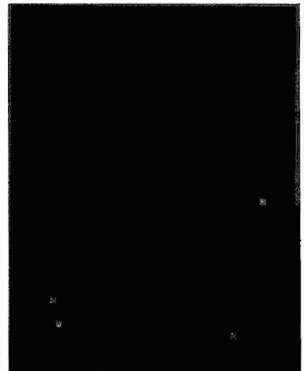
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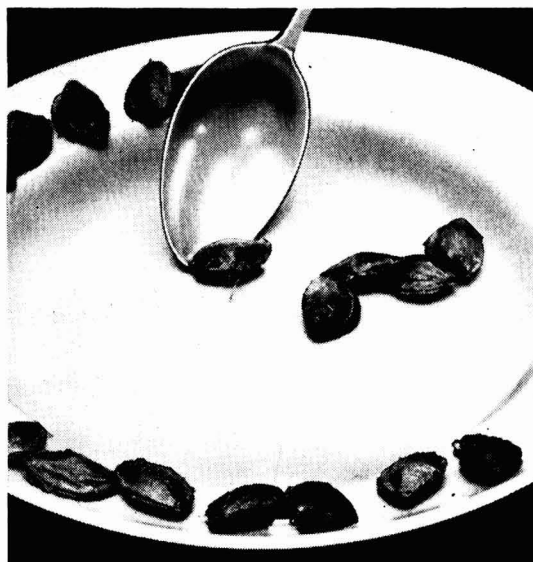


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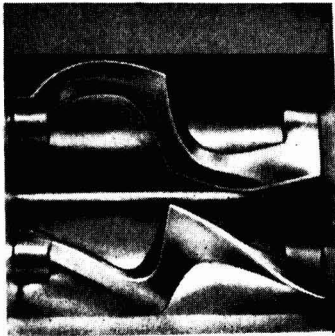
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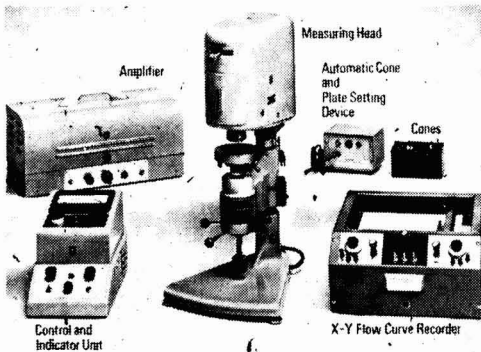
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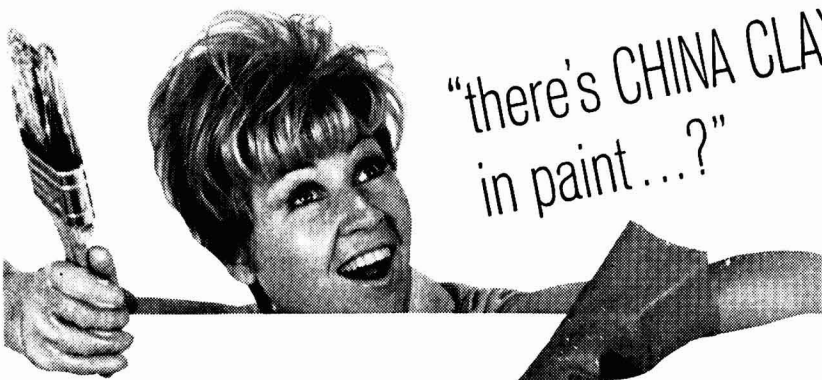
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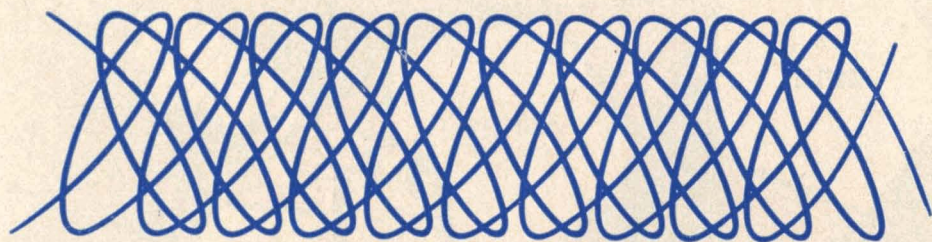
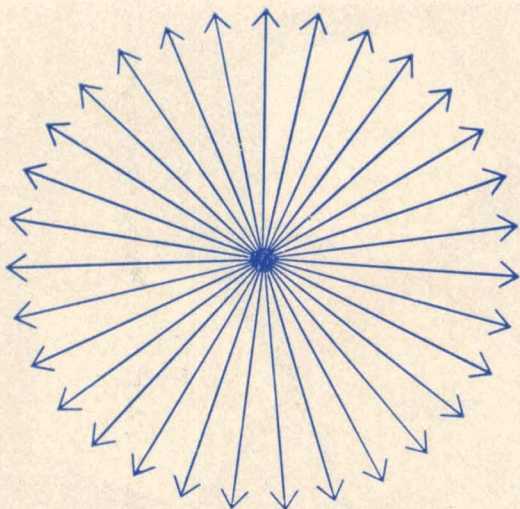
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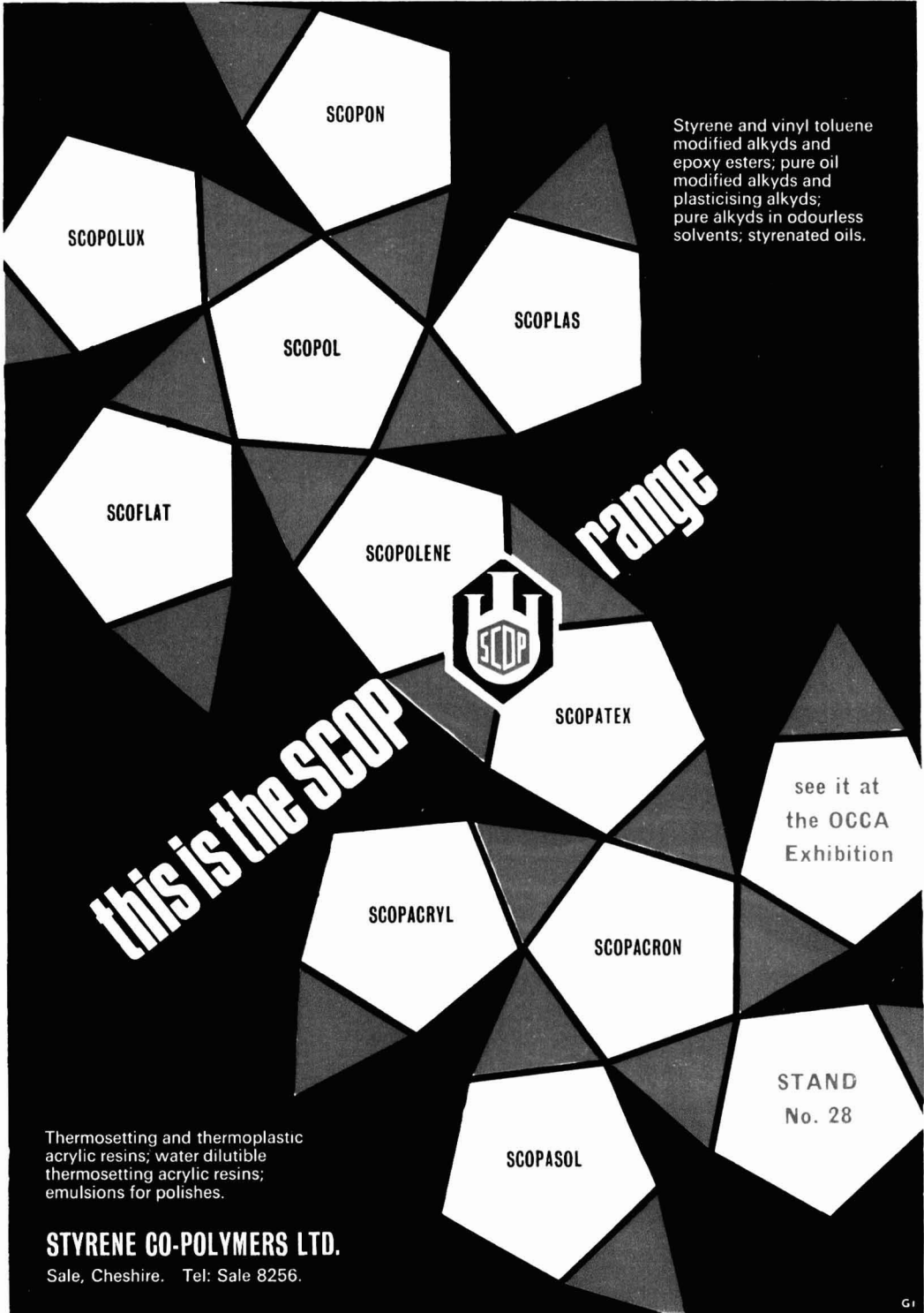
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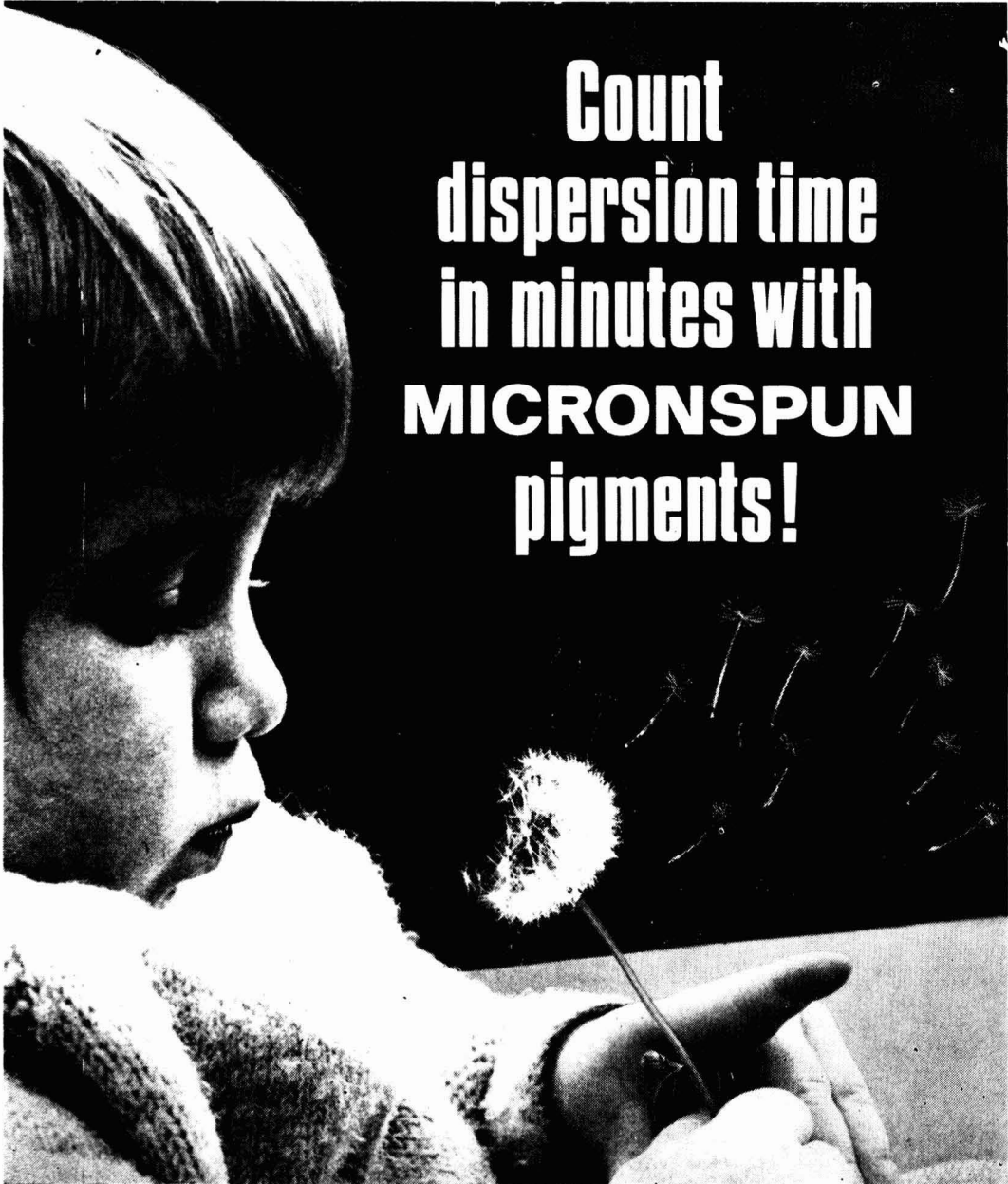
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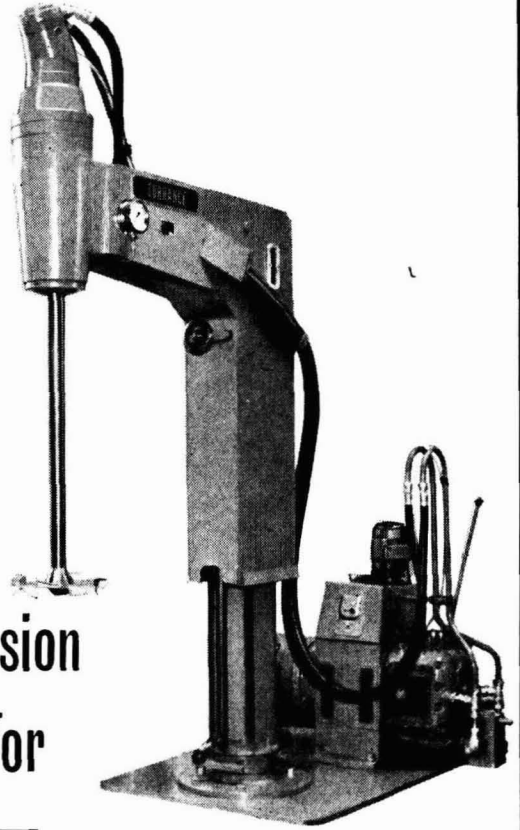
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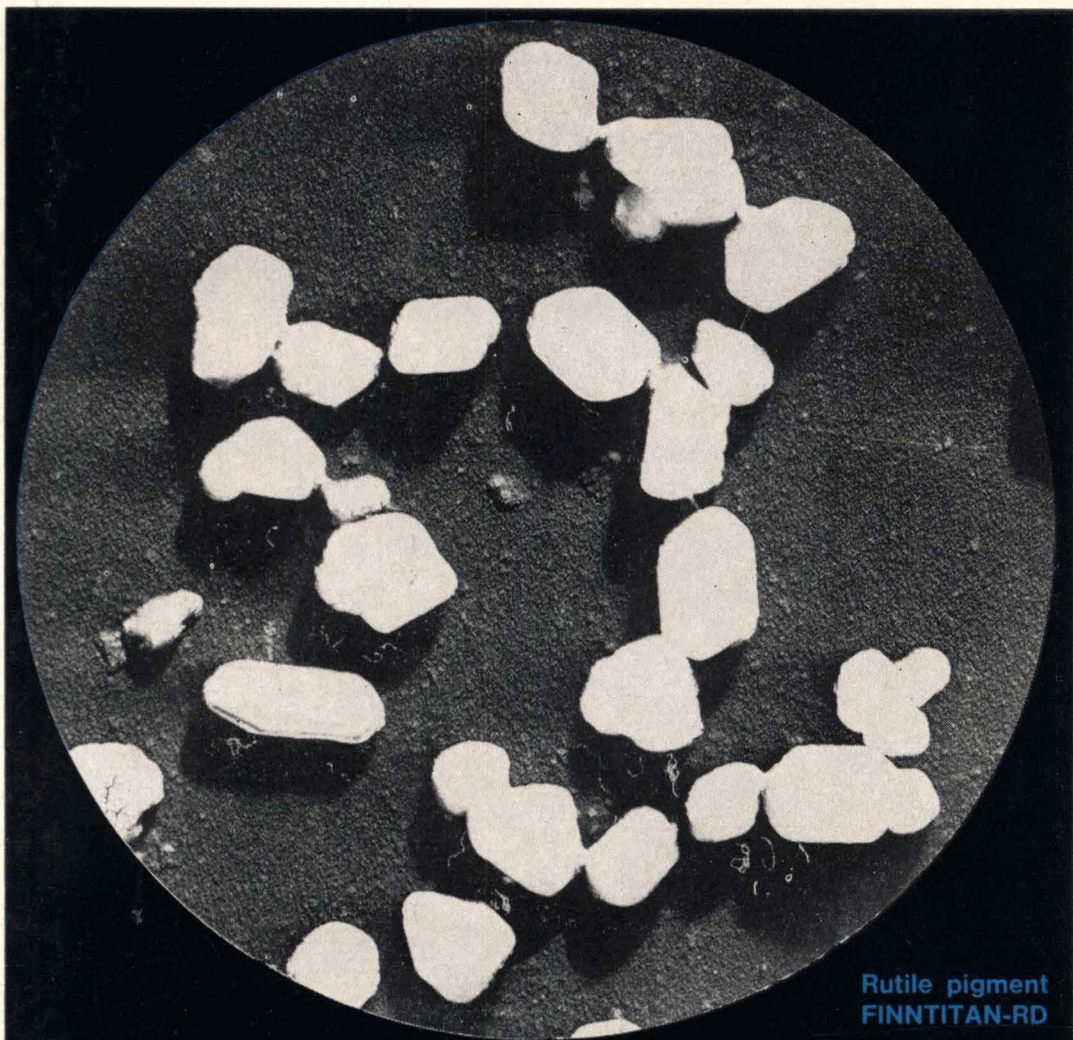
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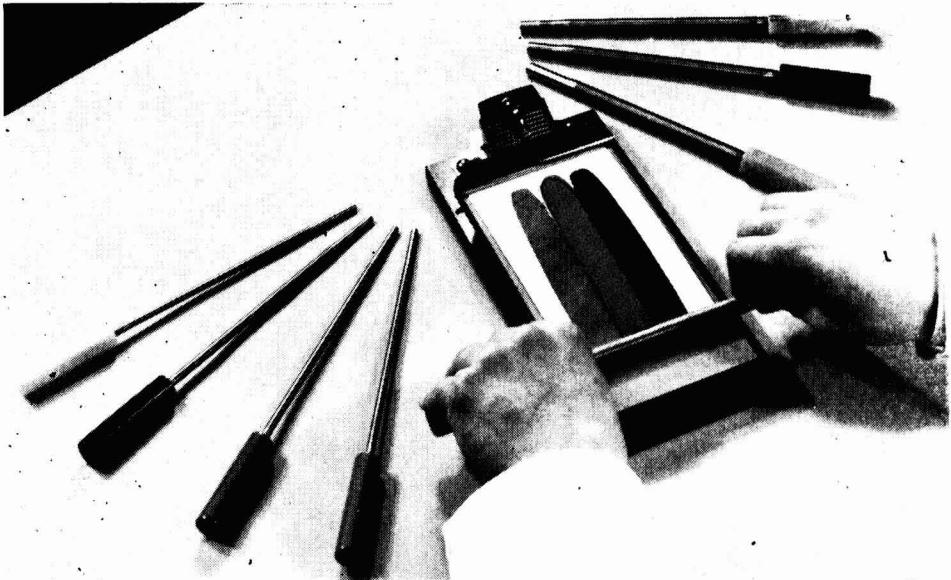
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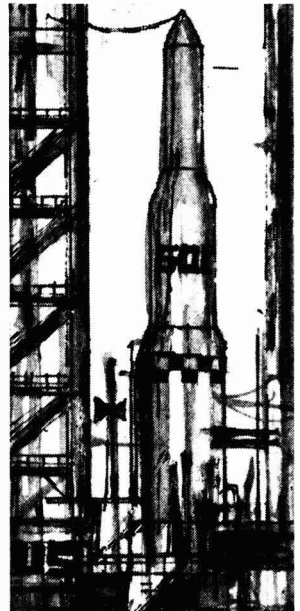
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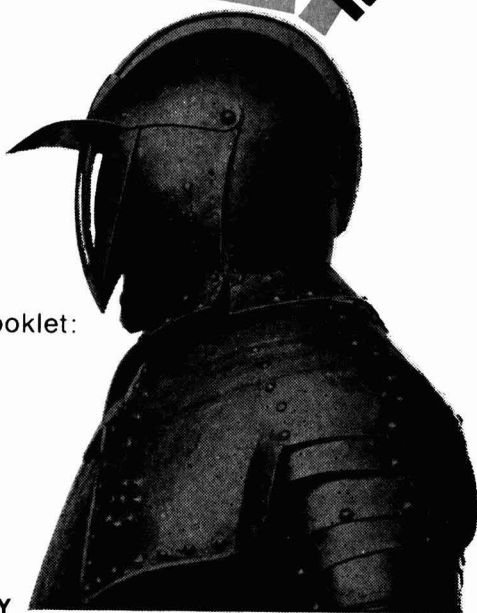
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JOURNAL OF THE OIL & COLOUR CHEMISTS' ASSOCIATION

Vol. 49 No. 3 March 1966



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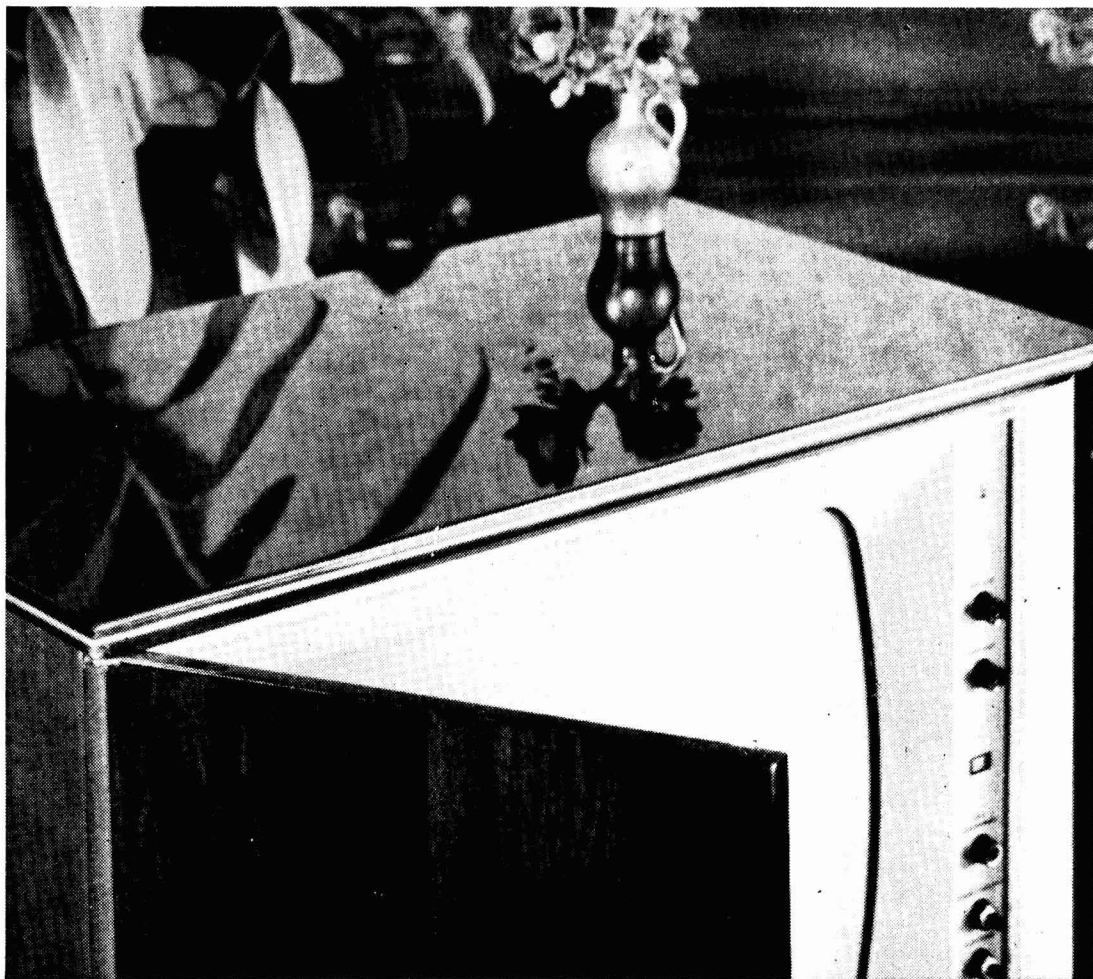
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Some studies in heterogeneous polymerisation*

By A. E. Alexander

The University of Sydney, Sydney, Australia

Summary

Although polymerisation in heterogeneous systems is important technically very little is known of the molecular processes involved. The paper surveys the problems found by a group working on simple aqueous systems and proposes some tentative conclusions.

Two groups of polymers have been studied (A) the insoluble in water type and (B) the water soluble type. Also investigation has been focussed on the role of the surfactants.

Quelques études de polymérisation hétérogène

Résumé

Bien que polymérisation effectuée aux systèmes hétérogènes est d'une importance industrielle, on ne reconnaît pas bien les processus moléculaires de la réaction. L'article rend compte des problèmes mis au jour par une équipe qui s'occupe des systèmes simples en milieu aqueux et à la fois propose certaines conclusions tentatives.

On a étudié deux types de polymères qui étaient respectivement (A) insoluble à l'eau et (B) soluble à l'eau. Une investigation fut mise au point également sur le rôle des surfactifs.

Einige Untersuchungen Heterogener Polymerisation

Zusammenfassung

Man weiss trotz ihrer technischen Wichtigkeit sehr wenig über die bei der Polymerisation heterogener Systeme stattfindenden molekularen Vorgänge. Diese Abhandlung gibt einen Überblick über die damit verbundenen Probleme, wie sie von einer Arbeitsgruppe bei einfachen wässrigen Systemen festgestellt werden und versucht einige Schlüsse zu ziehen.

Es werden zwei Gruppen von Mischpolymeren untersucht : (A) die wasserunlösliche Gattung und (B) die wasserlösliche Gattung. Die Untersuchungen konzentrierten sich auch auf die Rolle, welche die oberflächenaktiven Stoffe spielen.

Некоторые анализы гетерогенной полимеризации.

Резюме

Хотя полимеризация в гетерогенных системах технически важна, очень мало известно о сопровождающих молекулярных процессах. В статье дается обзор проблем найденных группой изыскателей работающих с простыми водяными системами и предложены некоторые предварительные выводы.

Изучались две группы полимеров: а) тип нерастворимый в воде, и б) тип растворимый в воде. Также принимается во внимание роль поверхностно-активных веществ.

* Presented to the 7th Australian Convention, Terrigal, New South Wales, on 18 June 1965.

Introduction

Despite the enormous technical importance of polymerisations carried out in a heterogeneous system, emulsion polymerisation being perhaps the best known example, a study of the published literature reveals how little is known of the detailed molecular processes involved. In an attempt to elucidate some of the basic physico-chemical principles a small research group in the University of Sydney, under the general direction of Dr. A. G. Parts and the author, has for some years been looking at heterogeneous polymerisation in relatively simple aqueous systems. The paper will survey the problems which have been examined and consider some of the tentative conclusions which have been reached.

Various monomers have been studied, and for the present purpose they can be grouped as follows :

A. Polymer insoluble in water (the usual case),

- (i) Monomer soluble in its polymer, e.g., vinyl acetate, methyl acrylate, vinyl stearate ;
- (ii) Monomer insoluble in its polymer, e.g., acrylonitrile. |

B. Polymer soluble in water, e.g., acrylamide.

In all studies referred to the initiator has been potassium peroxydisulphate, $K_2S_2O_8$. For obvious reasons a good deal of attention has been focussed on the role of the surface active agent (surfactant) which normally has to be present to maintain the resultant polymer latex in a stable colloidal form.

The principal measurements have been concerned with :

- (i) Rate of polymerisation, particularly as influenced by the nature and concentration of surfactant,
- (ii) Particle size of resultant latex, particularly as influenced by the nature and concentration of the surfactant,
- (iii) Determination of particle size of latex,
- (iv) Molecular weight of resultant polymer, in the absence and presence of surfactant,
- (v) End-groups on polymer arising from incorporation of initiator free radicals,
- (vi) Partition of monomer between the aqueous and polymer phases,
- (vii) Concentration of free radicals in polymerising systems,
- (viii) Penetration of $\cdot SO_4^-$ radicals into surfactant micelles,
- (ix) Effect of dissolved oxygen,
- (x) Effect of monomer on decomposition rate of $K_2S_2O_8$,
- (xi) Colloid stability of resultant latex.

For the sake of conciseness vinyl acetate and methyl acrylate will be considered together ; the behaviour of vinyl stearate was reported earlier¹.

Vinyl acetate and methyl acrylate

The first system examined was vinyl acetate in the absence and in the presence of low concentrations of anionic, non-ionic and cationic surfactants (below the

critical micellar concentration (cmc), i.e., in the absence of micelles).^{2,3} The initial monomer concentration was below the saturation solubility, thus avoiding the complication of emulsion droplets.

The rate of polymerisation (determined by means of a simple dilatometric technique) was found to vary considerably with the nature of the surfactant. Anionic surfactants increased the rate, with the C₁₆ sodium sulphate being more effective than the C₁₂ member. A non-ionic (Triton X-100) surfactant had no effect, whereas cationic surfactants decreased the rate, the C₁₆ quaternary ammonium bromide having the same effect as the C₁₂ member. Electron micrographs of the final latex showed that the anionic surfactant reduced the particle size, the cationic increased it, and the non-ionic had no effect. These rather striking observations were interpreted in the following way⁴.

Each $\cdot\text{SO}_4^-$ radical produced by the thermal decomposition of the initiator is captured by a monomer molecule in the aqueous phase and then begins to propagate.

The growing radical $\cdot\text{M}(\text{M})_n\text{SO}_4^-$ will tend to coil up and to absorb monomer, since vinyl acetate and its polymer are completely miscible. Furthermore when n has increased sufficiently (probably to about 30) this composite radical will become insoluble and, having only a small charge, will readily coalesce with other similar radicals. Coalescence will become progressively more difficult with the build-up of surface charge density arising from the $\cdot\text{SO}_4^-$ groups on the end of the polymer chains, until eventually the *zeta* potential of the particle exceeds the "critical" value necessary to prevent further aggregation with other particles of similar size. Further growth of these stable particles can then occur only by absorption of monomer or of the small radicals. As the number of stable particles increases a stage will be reached at which effectively all new radicals will be absorbed into the stable particles before they can grow appreciably; once this happens no more new particles will be formed.

The effect of soaps on the polymerisation rate stems from the interaction of the soap molecules with the small polymer aggregates which interaction tends to reduce or to promote their coalescence.

By virtue of the negative charge carried by the polymer particles, strong adsorption of the cationic soaps would be expected, thus lowering the *zeta* potential and so promoting coalescence. This, in its turn, will permit mutual termination between growing radicals from different particles, so that the smaller number of bigger particles and the reduced rate of polymerisation found experimentally are readily explicable.

Adsorption in this case being primarily determined by the coulombic attraction between opposite charges, the absence of any noticeable difference between the C₁₂ and C₁₆ members is not surprising.

In the presence of anionic soaps, the polymer particle will adsorb the organic anion of the soap, leading to an increase in *zeta* potential and the attainment of the "critical" value at a much smaller particle size. The subsequent coalescence of these small aggregates, which normally occurs in the absence of soap, is thus prevented. Mutual termination of growing radicals will be reduced and so the rate of polymerisation is enhanced compared with that in the absence of soaps.

In this case, since the coulombic forces are repulsive, adsorption of the organic anion is governed primarily by the length of the paraffin chain, so that the cetyl sulphate ion would be expected to be more effective than the dodecyl homologue.

The absence of any appreciable effect of the non-ionic soap on the polymerisation rate adds weight to the belief that the effect of soaps at concentrations below the cmc is determined in general by the charge carried by the adsorbing ion.

In order to test the general applicability of the above theory, methyl acrylate was chosen, being isomeric with vinyl acetate, having a similar water solubility, and being soluble in its polymer. The results obtained were basically similar to those with vinyl acetate, although certain differences were apparent in that the latex formed in the absence of surfactant was much less stable and the curve of (*percentage conversion*)¹ versus *time* was not linear⁵.

Acrylonitrile

Acrylonitrile differs from the two previous systems in that the monomer is insoluble in the polymer. (Vinyl chloride is another important member of this group, but is less amenable to experimental study).

When polymerised in aqueous solution under similar conditions to those above, acrylonitrile showed some marked differences in behaviour. Instead of the marked sigmoidal curves obtained with vinyl acetate and methyl acrylate the rate versus time was almost linear over much of the reaction, and the resulting latex was much more difficult to stabilise by surfactants⁶. In such a system polymerisation in the latex particles is clearly impossible but there remains, apart from the true aqueous reaction, the possibility of some polymerisation on the surface of the particles.

Some preliminary studies⁷ have been made of the polymerisation of acrylonitrile at concentrations above the saturation solubility (i.e., in true emulsion systems) but their understanding depends on further clarification of the simpler systems.

Acrylamide

In this case both the monomer and polymer are water soluble, so that no phase separation occurs. From the above theory it would be expected that surfactants would be without effect upon the rate of polymerisation. Experimental studies using the monomer at 0.26 molal, with 10 mM $K_2S_2O_8$, (temperature 50°C), have shown that the rate is unaffected by the addition of a non-ionic (Renex 690) and an anionic surfactant ($C_{12}H_{25}SO_4Na$) but is reduced by the addition of a cationic surfactant ($C_{16}H_{33} > C_{14}H_{29} > C_{12}H_{25}N(CH_3)_3Br$, surfactant concentration $5 \times 10^{-2}M$ in all cases).⁸

The action of the cationic surfactant may arise from interaction with the $S_2O_8^{2-}$ anion forming a rather insoluble precipitate which can be colloiddally dispersed when the surfactant is in excess.

Determination of particle size in latices

The accurate measurement of average particle size, particularly in fine latices, appears to be much less simple than it would appear from the literature. Five

techniques are currently being studied namely electron microscopy, light scattering⁹, analytical ultracentrifuge¹⁰, spreading and counting¹¹, and soap titration¹². Cross-checking using the same latex is being carried out where feasible.

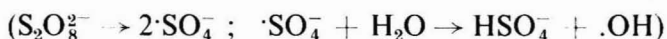
Molecular weight of formed polymer

Only preliminary measurements have so far been carried out. Using the simple aqueous solution of methyl acrylate (0.36M), with 6mM $K_2S_2O_8$ ($T = 40^\circ C$), the integral average molecular weight passes through a maximum at about 10 per cent conversion. In the presence of 10^{-4} M sodium hexadecyl sulphate a similar curve is obtained but with the maximum at about 25 per cent conversion⁵.

This rather unexpected behaviour has been tentatively ascribed to the initial growing polymer chain having few monomer molecules associated with it.

End-groups on polymer chains

The simple picture of chain initiation and termination given above would lead to a sulphate group at each end of the polymer chain. These groups can be assayed by a dye partition technique or by radiochemical means (using ^{35}S in the $K_2S_2O_8$ initiator)¹³. Published data shows considerable discrepancies, part of which could arise from experimental difficulties (particularly the counting technique used in the radiochemical method), from initiation by $\cdot OH$ radicals,



or by hydrolysis of the sulphate groups during the working-up procedures.

Each of these is being carefully examined, the second by the use of tritiated water¹⁴. The most recent results indicate a value close to two for the terminal sulphate groups on each chain.

Partition of monomer between aqueous and polymer phases

If, as is generally assumed, the main locus of polymerisation is in the latex particles, then the rate of reaction should be given by² :

$$-\frac{d[M]}{dt} = k_p C_M \bar{n} N_C$$

where k_p is the propagation constant,

C_M is the concentration of monomer in the particles,

\bar{n} is the number of free radicals per particle,

N_C is the number of particles per unit volume,

$[M]$ is the overall concentration of monomer.

For the systems discussed above the overall monomer concentration falls during polymerisation, so that C_M will also change. Partition coefficient data enable an estimate to be made of the value of C_M at any stage of the reaction.

Such data can be obtained very conveniently by using latices polymerised to completion, adding a known quantity of monomer, coagulating the latex by

a trace of cationic surfactant, and analysis of the aqueous phase (e.g., by interferometer)^{6,10}.

Concentration of free radicals in polymerising systems

Some preliminary attempts have been made, so far without success, to detect the presence of free radicals in an aqueous polymerising system, using a Varian ESR spectrometer¹¹. This lack of success is ascribed to the presence of the water reducing the sensitivity of the equipment.

Penetration of $\cdot\text{SO}_4^-$ radicals into soap micelles

According to the Harkin theory of emulsion polymerisation the first step consists in the penetration of the free radical (e.g., $\cdot\text{SO}_4^-$ from $\text{K}_2\text{S}_2\text{O}_8$) into a surfactant micelle, where it attacks solubilised monomer and so initiates polymerisation. The very low concentration of $\cdot\text{SO}_4^-$ radicals makes it difficult to study this proposed step directly, but a very closely related process, namely the penetration of MnO_4^- ions into surfactant micelles containing reactive organic compounds (e.g., vinyl stearate, oleyl alcohol) can be followed readily¹⁵.

Results obtained so far suggest that the penetration of MnO_4^- into anionic micelles is energetically highly unfavourable, suggesting that even with relatively insoluble monomers such as styrene, the first step occurs in the aqueous phase rather than in the soap micelles.

Effect of monomer on decomposition rate of $\text{K}_2\text{S}_2\text{O}_8$

The question of the influence of monomer and various surfactants on the rate of decomposition of $\text{K}_2\text{S}_2\text{O}_8$ is clearly a very pertinent one. Experiments using $\text{K}_2\text{S}_2\text{O}_8$ concentrations of about 0.1M (i.e., much higher than in normal polymerisations) indicate that the rate is increased⁵. On the other hand using radiotracer techniques and concentrations of $\text{K}_2\text{S}_2\text{O}_8$ approximating to that used in polymerisation has shown no increase in the rate¹⁶. Further work is clearly required.

Colloid stability of resultant latex

In the course of the various studies outlined above a number of interesting qualitative observations of colloid stability have emerged. For example the latex from vinyl acetate (prepared in the absence of surfactant) was more stable than that from methyl acrylate. The latices from acrylonitrile were much less stable still and tended to give gels even at about 3 per cent solids⁶.

With methyl acrylate the exclusion of oxygen resulted in a reduced colloid stability of the latex, suggesting that the oxygen-monomer compound postulated as being formed has some surface activity.⁵

These observations are clearly very relevant to the rheological properties of latices, and warrant further investigation.

[Received 11 August 1965]

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Discussion

MR. K. QUEEN said that the difference in effect between anionic and cationic surfactants had been demonstrated and compared in turn with no surfactant. While the anionic had given a much faster rate of conversion, all types ultimately reached a peak at approximately 80 per cent. One would normally expect that the polymerisation proceeding most rapidly would have a higher final percentage conversion. Was there a reason why they all levelled off at the same point?

PROF. A. E. ALEXANDER said that all did finally reach 100 per cent but because of the large lapse of time involved, the conversion was only followed to about 80 per cent. In these systems, there was a fixed amount of monomer and as the reaction moved towards completion the rate became slower due to monomer depletion and to the polymer particles causing an increase in viscosity.

MR. L. WILLIAMSON said he was very interested in the oxygen structures mentioned, as he had worked in the field of non-aqueous polymerisation. One of the interesting problems was the energy of the oxygen-oxygen bond which had been found to vary immensely with various structures. Could Prof. Alexander give an approximation of the calculated bond energies for the structures indicated, and how stable they would be?

PROF. ALEXANDER said there had been a large amount of discussion about oxygen effects in polymerisation and there seemed to be considerable differences of opinion in the literature. He had pointed out that the suggested structures were based on some rather simple considerations and he therefore would not like to go into more detail at this stage. It was worth noting, however, that the likelihood of a type of copolymer existing between oxygen and the various monomers could prove to be of technical importance, since, apart from its effect on colloid stability there was also the possibility that, having once been formed, it might subsequently break down.

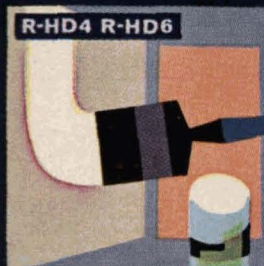
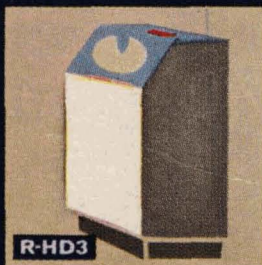
MR. H. STEPHEN asked if there would be any tendency for acrylamide particles to release persulphate ions in the final stage of conversion to give a large concentration of these ions.

PROF. ALEXANDER said he thought not, because the cationic surfactant, unless attacked by the persulphate, would be consumed in the course of polymerisation. The actual amount of persulphate which had decomposed into free radicals and thereby initiated the polymerisation was normally small and that, over a period of about an hour, the amount of persulphate actually lost was effectively nil.

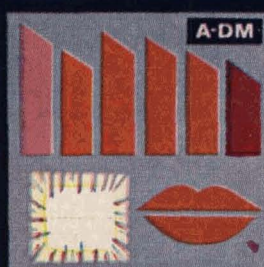
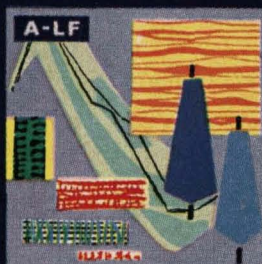
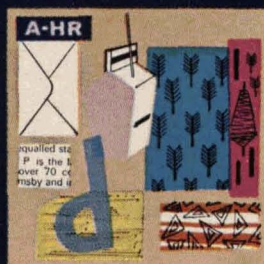
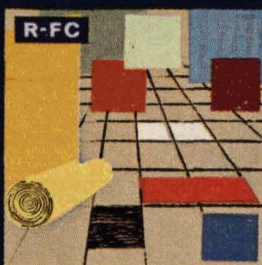
MR. L. BORSODY asked if Prof. Alexander found that the oxygen content of the components in the polymerisation would induce polymerisation or reduce the rate and was it correct to say that if the persulphate concentration was increased, the stability of the latices decreased?

PROF. ALEXANDER said that oxygen itself was an inhibitor in these systems and the curves indicated covered the reaction after the inhibition period had ended. The inhibition period was quite reproducible in any given system even if one did not take any great precautions to remove oxygen. In the work on methyl acrylate, the normal inhibition period was about 90 minutes, after de-aeration it was reduced to approximately eight minutes. Once reaction starts the actual kinetic curve seems to be little affected, although as I said, de-aeration does affect the colloid stability of the final latex.

To the second question Prof. Alexander said that Mr. Borsody had surmised correctly. He hoped to do more work on colloid stability since there had been, particularly in the last ten or 15 years, a resurgence of interest in this field and these latices provided almost ideal systems for quantitative study. From preliminary work, there was no doubt that as the salt concentration was increased the stability was reduced and the different salts fell into the expected pattern.



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The influence of additives and calcination on the pigment properties of titanium dioxide : Part II

The influence of potassium and sodium salts

By V. Blechta and M. Lavička

Inorganic Chemistry Research Institute, Ústí nad Labem, Czechoslovakia

Summary

The influence of sodium and potassium chloride and of sodium and potassium sulphate additions to hydrated TiO_2 on the pigment properties of the product, obtained by calcination at temperatures of 850 to 1000°C has been studied. Sodium salts act as positive, potassium salts as negative rutilisation catalysts. From the point of view of tinting strength, the optimum shifts toward lower temperatures when impregnation is carried out with sodium salts, optimum values being lower than those for potassium salts.

L'influence des additifs sur la calcination et les propriétés pigmentaires du dioxyde de titane : 2ième Partie

L'influence exercée par des sels de potassium et de sodium

Résumé

On a étudié l'influence des chlorures et des sulfates de sodium et de potassium—ajoutés au TiO_2 hydraté—sur les propriétés pigmentaires du produit de calcination à des températures de 850° à 1000°C. Les sels de sodium s'agissent de catalyseurs de rutilisation, tandis que ceux de potassium se comportent en qualité d'anti-catalyseurs. Lorsqu'on emploie les sels de sodium, le pouvoir colorant optimum du pigment se trouve vers des températures moins élevées et c'est plus faible que l'optimum rendu par les sels de potassium.

Der Einfluss von Additiven und Kalzinierung auf die Pigmenteigenschaften von Titandioxyd : Teil 2

Der Einfluss von Kalium- und Natriumsalzen

Zusammenfassung

Der Einfluss von Natrium—und Kaliumchlorid und von Natrium—und Kaliumsulfat Zusätzen zu Titandioxydhydrat auf die Eigenschaften des durch Kalzinierung bei 850 bis 1000°C erhaltenen Produktes wurde untersucht.

Natriumsalze verhalten sich als positive, Kaliumsalze als negative Katalysatoren, wenn die Rutilform erzeugt werden soll. Sofern die Behandlung mit Natriumsalzen erfolgt, erhält man durch Verschiebung nach den Bestwerten niedrigerer Temperaturen insofern bessere Resultate als das Optimum dieser Temperaturen niedriger liegt, als das für Kaliumsalze.

Влияние примесей и кальцинации на свойства грунтовых красок изготовленных на базе двуокиси титана. Часть 2.

Влияние солей калия и натрия.

Резюме

Изучалось влияние хлористого натрия и калия и примесей серноокислого натрия и калия в гидратизированной двуокиси титана на грунтовые красочные свойства продукта полученного кальцинацией при температурах от 850° до 1000° С. Соли натрия действуют в качестве положительных, а соли калия в качестве отрицательных рутиловых катализаторов. Что касается способности придавать тон, то оптимальное значение движется к нижним температурам когда пропитывание осуществляется солями натрия, и оно ниже чем значения для солей калия.

Introduction

In their preceding work¹ the authors have studied the influence of ammonia and sulphuric acid additions to hydrated TiO₂ on the pigment properties of the calcinate. In the present work the influence of some impregnation additives (sodium and potassium salts) is studied on the pigment properties of the calcinate.

Experimental

The initial material was hydrated titanium dioxide of anatase structure, containing 0.0913 g H₂SO₄/g TiO₂, obtained by thermal hydrolysis of sulphate solutions from ilmenite on an industrial scale. The experimental methods, i.e. the method of preparing the sample for calcinating, the calcination in a grate-less fluidisation furnace, the method of determining the tinting strength of the calcinated samples by means of an objective method and the determination of rutile in the final product have been described earlier¹. In order to allow a unified manner of expressing the amount of impregnation additives, they are given in "gram cations per 100 g TiO₂." This quantity denotes the gram-atoms of the cation in the compound used to impregnate 100 g TiO₂.

Results and discussion

An analysis of literature data shows that sodium and potassium salts are among the most important impregnation agents which are added to hydrated titanium dioxide before calcination. Therefore it was thought worth-while to determine the influence of the four most important salts, i.e. potassium and sodium sulphate, and sodium and potassium chloride, on the pigment properties of the calcinate. The influence of these compounds has been followed at three concentrations ; the results being shown in Figs. 1, 2 and 3.

From Fig. 1 it may be seen that sodium salts are positive, potassium salts are negative rutilisation catalysts and optimum tinting strength is shifted toward lower temperatures in the case of sodium salts, achieving lower values.

A similar conclusion follows from Figs. 2 and 3, where the amount of impregnation salts has been increased. An interesting effect is observed from curves which illustrate the rutilisation of the calcinate in cases where impregnation was done with KCl. (Fig. 2, curve 8, and Fig. 3, curve 12). With the increasing calcination temperature, retaining the same calcination time, the

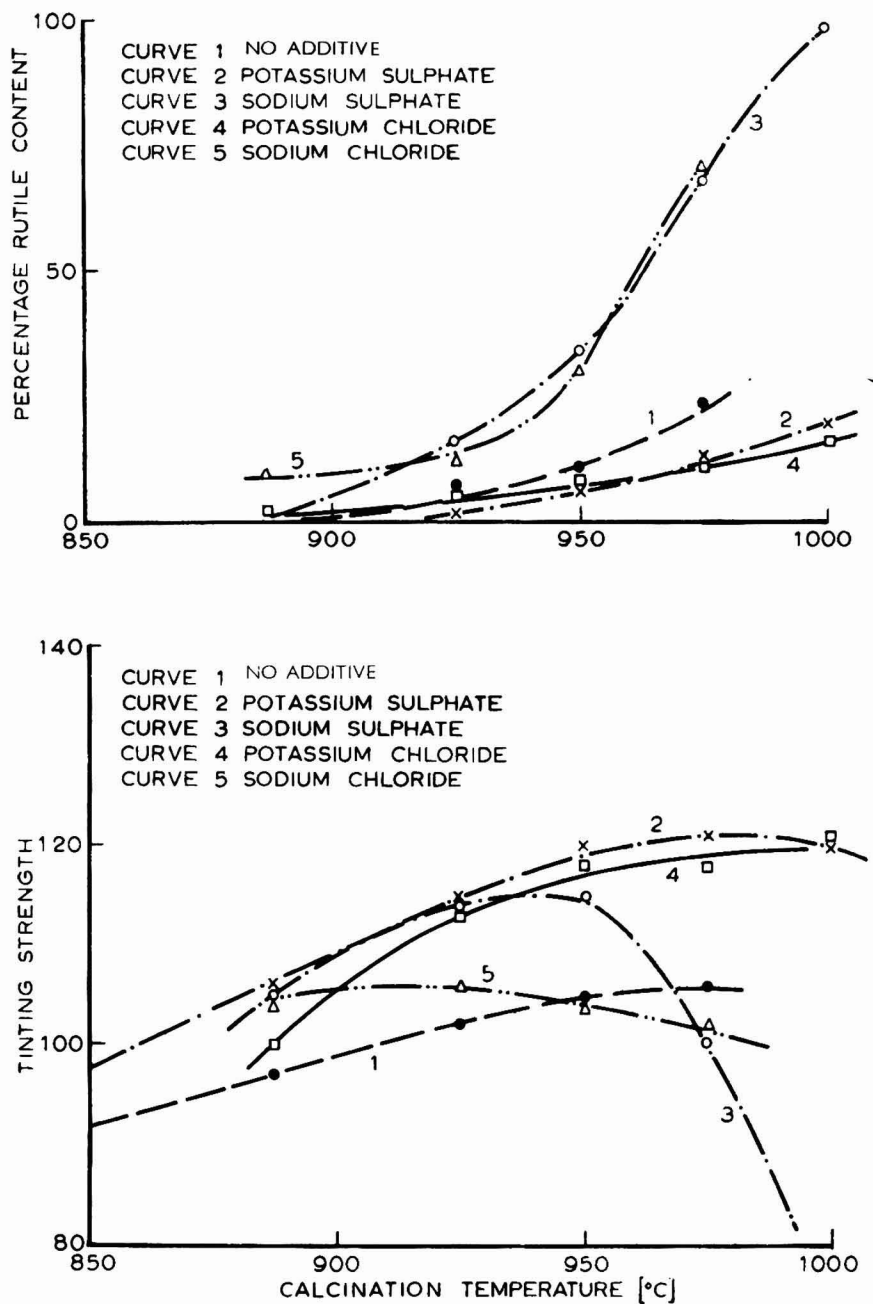


Fig. 1. Influence of alkaline salts on the pigment properties of the calcinate (calcination time 30 mins.). Impregnation— 0.574×10^{-2} gm. Cat/100gm. TiO_2

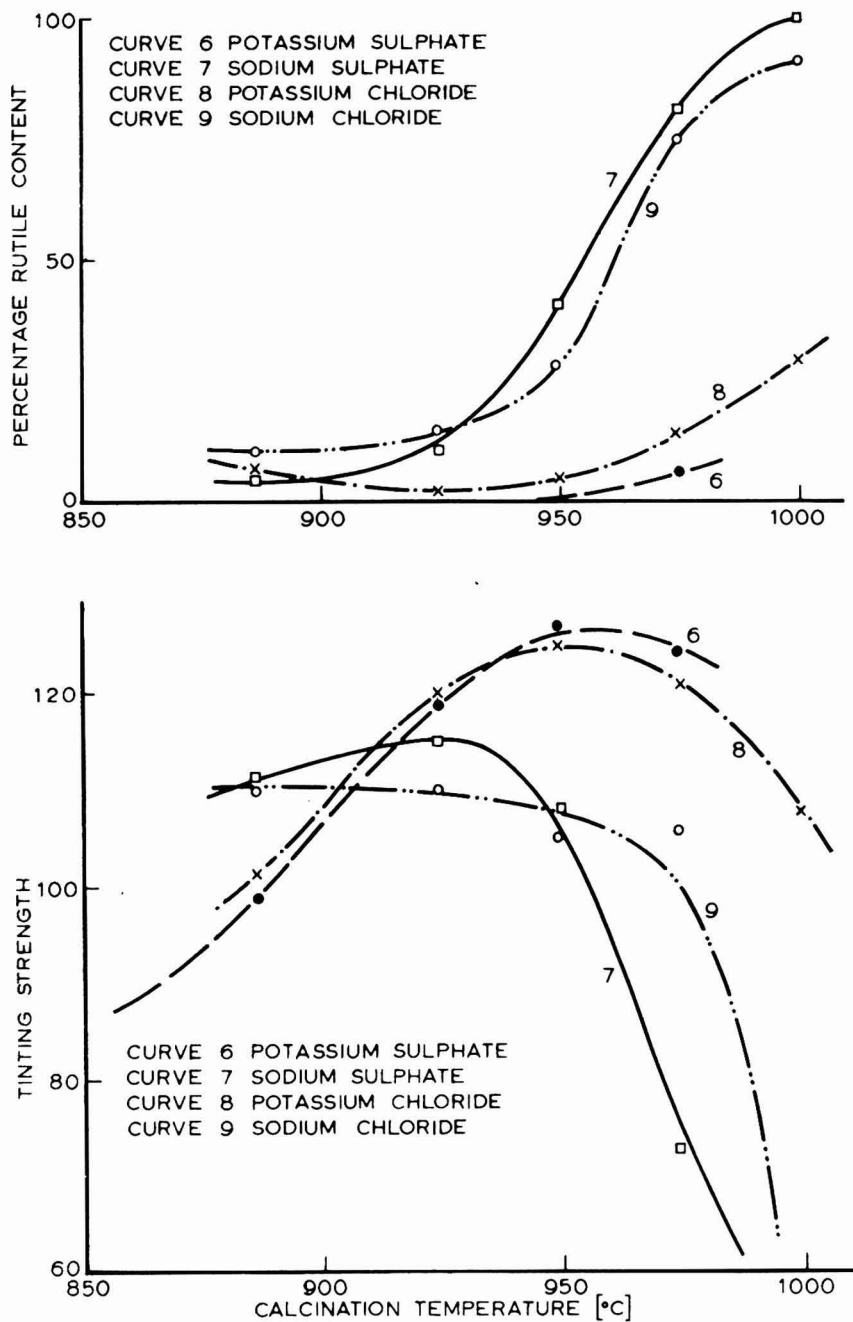


Fig. 2. Influence of alkaline salts on the pigment properties of the calcinate (calcination time 30 mins.). Impregnation— 1.147×10^{-2} gm. Cat/100gm. TiO_2

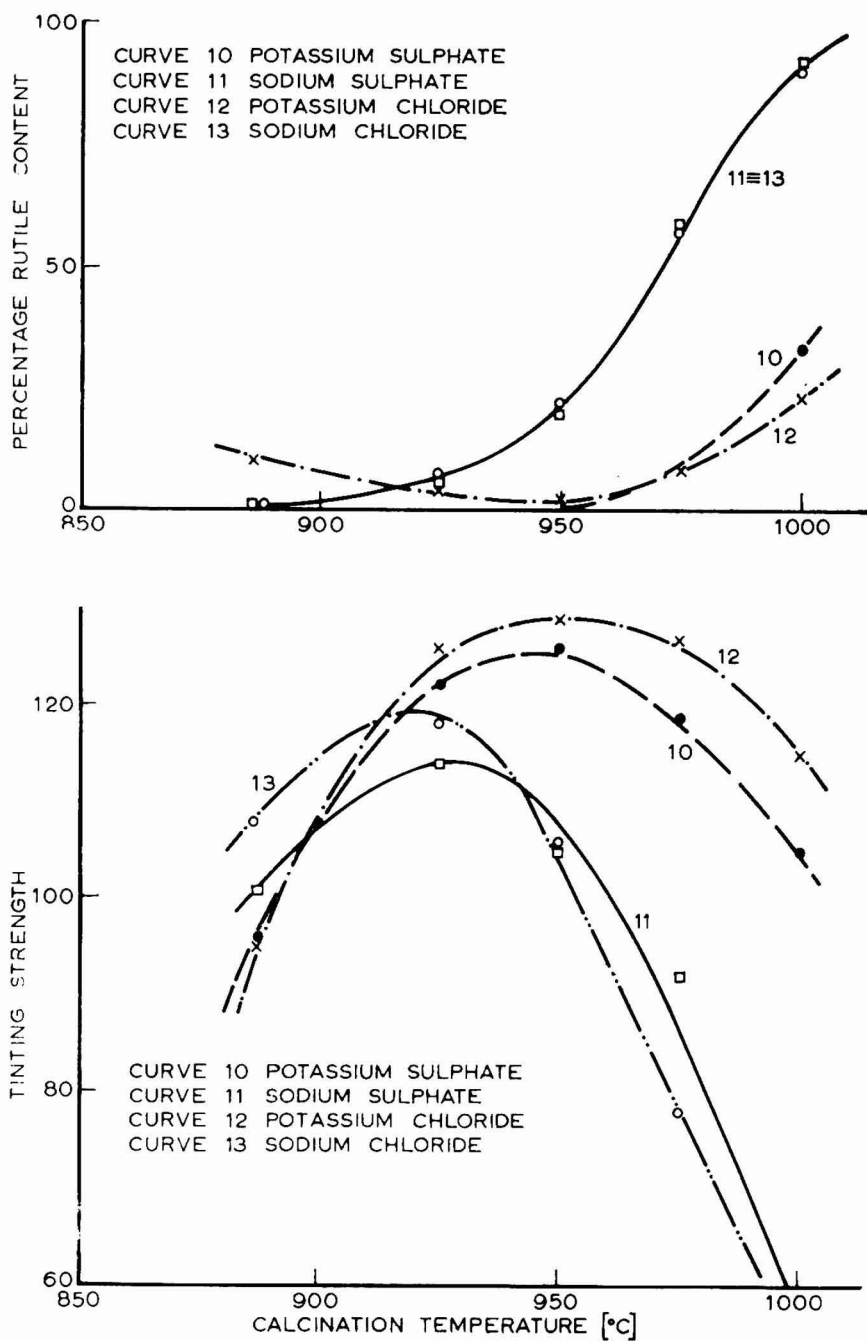


Fig. 3. Influence of alkaline salts on the pigment properties of the calcinate (calcination time 30 mins.). Impregnation— 1.722×10^{-2} gm. Cat/100 gm. TiO_2

rutile content decreases and only later increases. The experiments have all given the same result. Up to now no explanation can be put forward for this effect. Furthermore, Figs. 1, 2 and 3 show that curves obtained for calcinates impregnated with potassium salts, tend to run close to one another. A similar conclusion holds for calcinates impregnated with sodium salts. However, there is a large difference between calcinates impregnated with compounds having different cations.

The influence of sodium and potassium salts on the tinting strength of the calcinate is evident from Fig. 4, which is a plot of the tinting strength of the calcinate at the optimal calcination temperature, as a function of the amount of impregnation salts. From the plot it follows that, in the given impregnation range, potassium salts result in a calcinate of higher tinting strength than sodium salts. Especially interesting is the anomalous behaviour of sodium chloride.

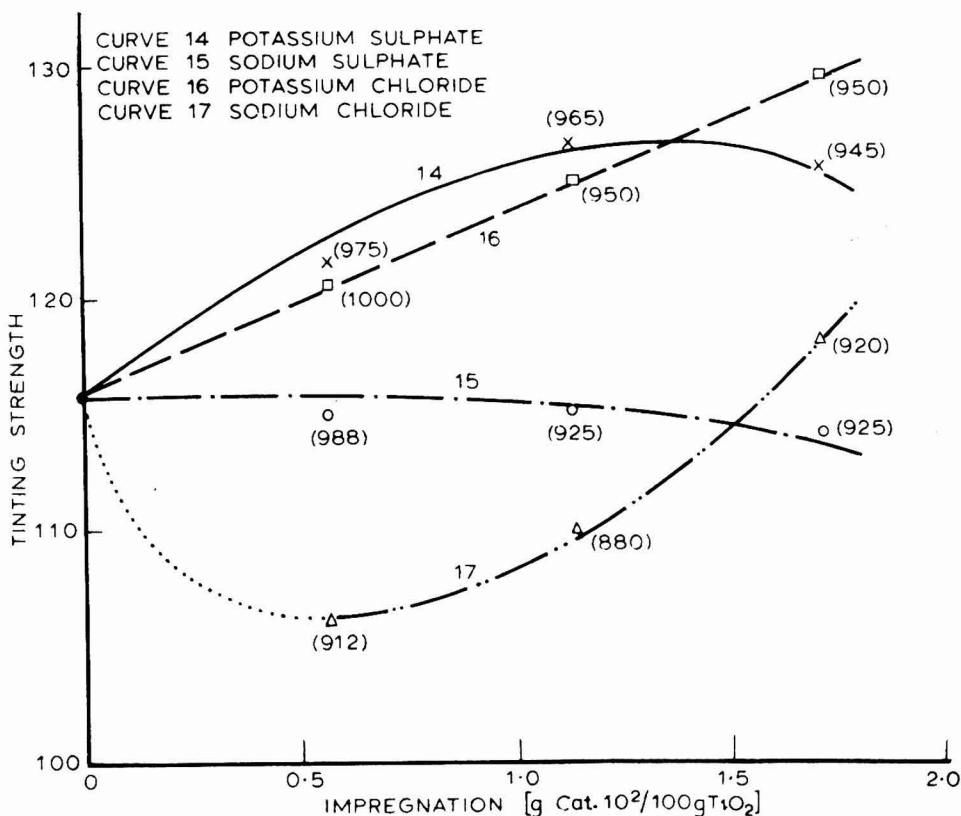


Fig. 4. Influence of alkaline salts and of their amount on the tinting strength of the calcinate at the optimum calcination temperature (calcination time 30 mins.)

Conclusion

The influence of sodium and potassium salt additions to hydrated TiO₂ before calcination was studied. The following conclusions may be drawn from the observations made :

1. The cation of the impregnation agent has the main, and the anion the secondary, influence on the quality of the calcinate.
2. Potassium chloride and sulphate are negative rutilisation catalysts.
3. Sodium chloride and sulphate are positive rutilisation catalysts.
4. The temperature optimum of the tinting strength is shifted toward lower temperatures in the case of sodium salts, achieving lower values, than in the case of potassium salts.

[Received 11 August 1965]

References

1. V. Blechta, M. Lavicka, *JOCCA* 1965, **48**, 455.

Electrodeposition of one-coat paints containing titanium dioxide*

By G. Landon and I. H. Ashton

British Titan Products Co. Ltd., Billingham, Co. Durham

Summary

Titanium dioxide pigments can be used to formulate paints which will give single-coat white and pastel-tinted glossy finishes applied by electrodeposition. Only resins giving a good colour, such as the water-soluble acrylics, have been studied. Zinc-plated steel is used as a substrate. Factors affecting gloss are described, such as pH, pigment/binder quotient, the use of surfactants and the choice of pigment. The super-opaque rutile pigments give the best results. Treatment of the paint film with gaseous ammonia before stoving gives a gloss comparable with that of sprayed coatings. Preliminary results for durability (Marr weatherometer) are quoted.

Electrodéposition de peintures monocouches contenant du dioxyde de titane

Résumé

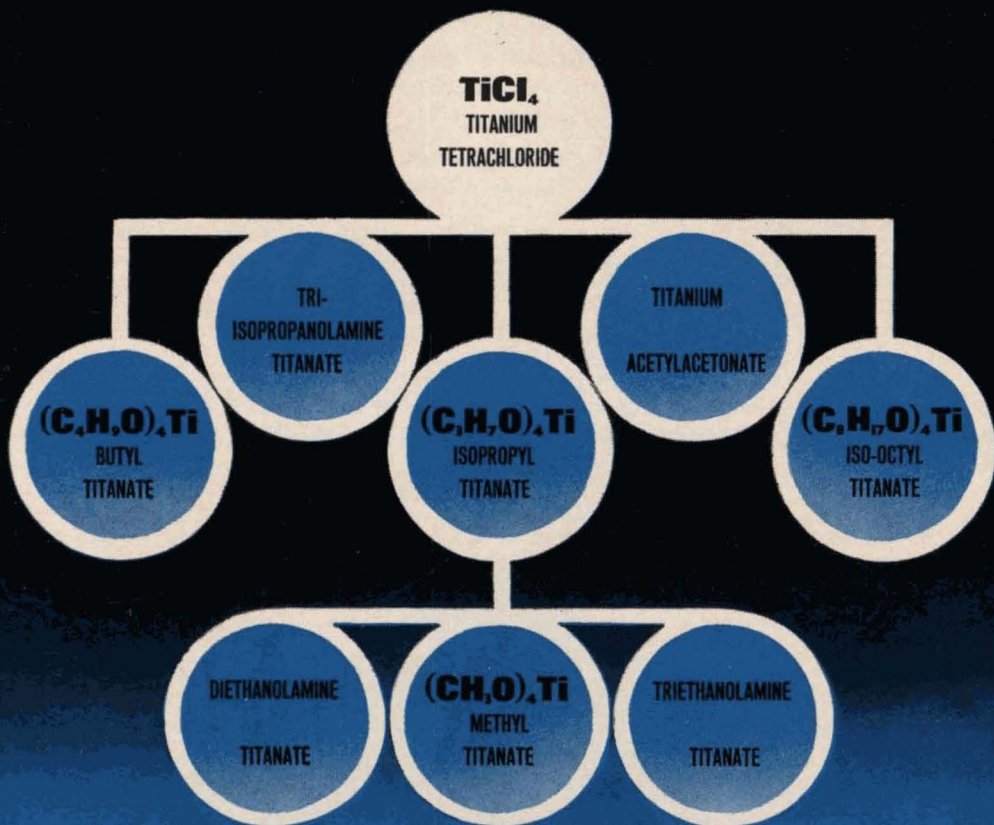
Les pigments de dioxyde de titane peuvent s'employer pour la mise au point des formules de peintures brillantes à finition, monocouches, blanches ou de teintes pastel. On a étudié seulement des résines donnant une bonne couleur, telles que les acryliques solubles dans l'eau. Comme support on se sert de l'acier plaqué de zinc. On décrit les facteurs ayant un effet sur le brillant tels que le pH, le rapport pigment/liant, l'emploi des surfactifs, et le type du pigment. Les pigments de rutile superopaques donnent les meilleurs résultats. Traitement du feuil de peinture par le gaz ammoniac avant séchage au four donne un brillant comparable à celui des revêtements appliqués par pistolet. On mentionne des résultats préliminaires de durabilité obtenus au weatheromètre de Marr.

Elektrophorese Einschichtiger, Titandioxyd enthaltender, Anstrichfarben*

Zusammenfassung

Es ist durchaus möglich Titandioxyd—Pigmente enthaltende Anstrichfarben zu formulieren, die sich als einschichtige, weisse oder pastellfarbige, glänzende Überzugslacke auftragen lassen. Es werden lediglich Harze, z.B. wasserlösliche Akrylharze, die hellgefärbte Bindemittel, ergeben, untersucht. Als Substrat wurde verzinktes Eisen angewandt. Beschrieben werden die Faktoren, die den Glanz beeinflussen, wie z.B. pH, Pigment/Bindemittel Quotient, der Einsatz oberflächenaktiver Stoffe und die Wahl geeigneter Pigmente. Die besten Ergebnisse erhält man mit hyperopaken Pigmenten. Behandlung des Anstrichfilms vor der Ofentrocknung mit Ammoniakgas ergibt einen Glanzgrad, der mit dem gespritzter Filme vergleichbar ist. Vorläufige Dauerhaftigkeits-ergebnisse (Marr Weatherometer) werden aufgezeigt.

*Presented to the 7th Australian Convention on 19th June, 1965.



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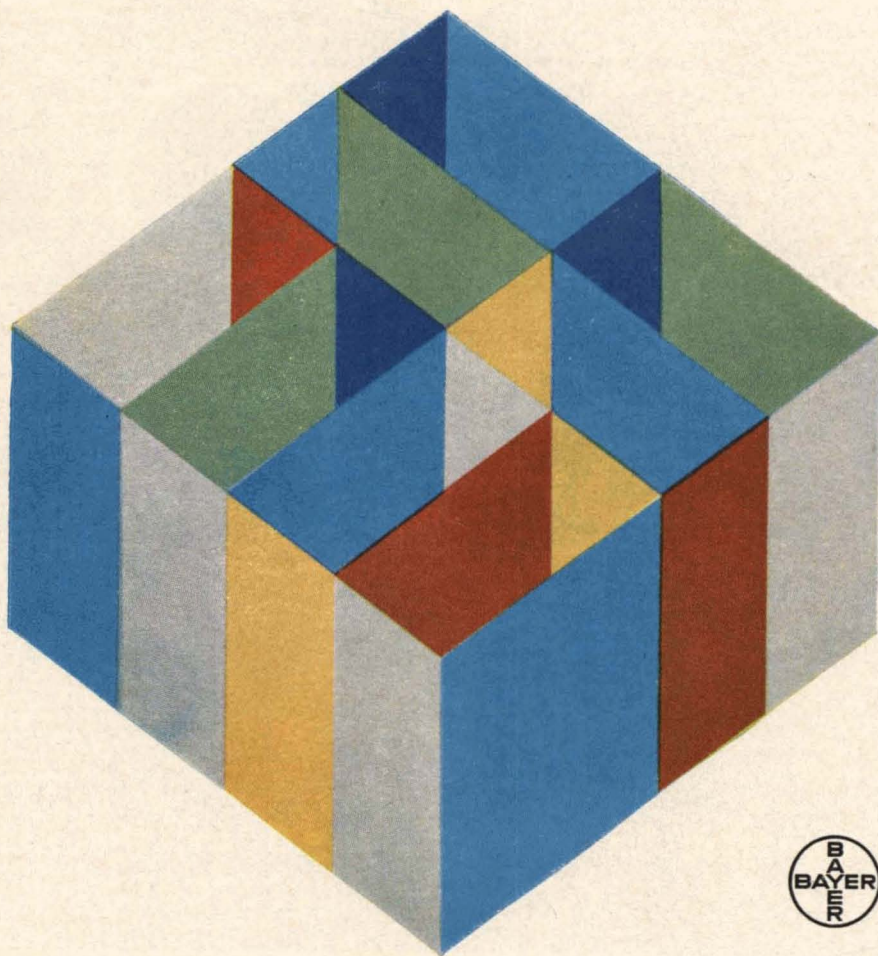
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Электро-осаждение однослойных красок содержащих двуокись титана

Резюме

Грунтовые краски содержащие двуокись титана можно применять для формулировки красок дающих однослойные белые и пастильного оттенка блестящие отделки приложенные посредством электро-осаждения. Изучались только смолы дающие хороший цвет, как, например, акриловые растворимые в воде. В качестве подслоя применяется сталь с покрытием цинка. Описываются факторы влияющие на блеск, как, например, значение pH, отношение грунтовой краски к связующей среде, применение поверхностно-активных веществ и подбор грунтовой краски. Рутиловые грунтовые краски обладающие сверх-непрозрачностью дают наилучшие результаты. Обработка красочной пленки аммиаком перед обжиганием сообщает блеск сравнимый с распылительными покрытиями. Даются предварительные результаты опытов на выносливость (Измеритель атмосферных воздействий Марра).

Introduction

Since the original work of Burden and Guy¹, much has been published on the method of electrodeposition for applying paint films to metallic substrates. Whereas the advantages of the method are therefore well known, the range of finishes for which it can be used is, at present, very limited.

One aspiration of the industry is to be able to produce by electrodeposition white or pastel tinted finishes in a single coat having a high gloss. It is the purpose of the present work to show that this can, in fact, be done, though the practical difficulties of applying the method to a production line remain to be tackled.

In general, paints for electrodeposition are made from resins which are rendered soluble in water by reaction with ammonia or amines. The choice of suitable pigments is large. The paints are diluted with water to a low solids content, usually in the range 10 to 20 per cent by weight, and an alcohol is sometimes added to counter any tendency of the resin to precipitate. The pH of the paint is maintained at a suitable value, usually around 8. The article to be coated is immersed in the paint and made anodic, while a cathode is similarly immersed. On a plant scale a constant DC voltage is generally used, in the range 20 to 300 volts, depending upon the formulation of the paint and the dimensions of the tank. Current densities range from 1 to 10 milliamps per cm² and the deposition of a paint film may take between 0.5 and ten minutes.

Electrodeposition is most widely used for red primers, for which suitable resins include the water-soluble phenolics and oxidising alkyds. The dark colour of the phenolics, however, renders them quite unsuitable for light coloured decorative finishes. The oxidising alkyds give better pastel tints (though these are somewhat dull), but are not suitable for whites. The poor colour is due partly to the resin, but, as noted by Finn and Mell² and by Berry³, is due also to the presence in the paint film of ferrous ions, from the steel substrate, which becomes oxidised.

Another difficulty with all decorative paints applied by electrodeposition (with the exception of certain dark colours and blacks) is the poor gloss which is invariably caused by gross flocculation of the pigment in the deposited film. The pigment floccules sometimes reach macroscopic proportions and contribute, incidentally, to the poor colour by lowering the hiding power of the pigment.

In the present work, two steps were taken to avoid the problem of discoloration. The first was to choose a water-soluble acrylic resin which would give little or no yellowing on stoving; the second was to use zinc-plated steel as a substrate. In the course of some preliminary experiments a number of resins was examined. Some alkyd-amino systems, which gave good colour, showed evidence of disproportionation during deposition. One or two acrylics, on the other hand, were unsuitable because they possessed electrical properties typical of an emulsion rather than of a true solution. The resin finally chosen for further study was Synocryl 840S, a water soluble acrylic/melamine resin manufactured by Cray Valley Products Ltd.

The choice of zinc-plated steel as a substrate was justified on the grounds of simplicity, and because the complete absence of soluble ferrous ions at the surface of the metal leaves the paint film quite free from iron discoloration. Other methods for eliminating discoloration remain to be studied; meanwhile it is to be hoped that the results for zinc-plated steel will be of interest because the additional resistance to corrosion given by the zinc will be required in certain applications.

The solids content of the paint should be low, in order to avoid excessive waste due to the loss of the dip coat which is removed, after electrodeposition, by washing. On the other hand, too low a solids content leads to excessive power consumption owing to the high resistance of the bath, and also increases the risk of instability of the paint because there is a limit to which a resin may be diluted without its precipitating from solution. From preliminary experiments a solids content of 20 per cent by weight was chosen as suitable, though lower values would also be satisfactory. All the results given below were obtained with paints at 20 per cent solids.

It became clear at an early stage that it was more convenient in the laboratory to deposit paint films at constant current than at constant voltage. As noted by Berry³, the quality of the paint film then became less dependent upon the geometry of the system, and the number of coulombs passed was conveniently proportional to the duration of deposition. Constant current was therefore used in most experiments. Stepped increases in voltage would approximate to this arrangement on a plant, but further experiments would be called for if operation at a uniform constant voltage were contemplated.

Once it had been established that the choice of resin and substrate gave paint films which were free from discoloration, it remained to find means of achieving good gloss. A range of titanium dioxide pigments was evaluated. Some experiments were carried out on paints having a pigment/binder quotient (P/B) of 0.25 by weight, since moderately good gloss could be obtained simply by keeping the pigment content low. Very thick films were necessary to give opacity, however, and when advantage of using superopaque pigments was discovered, further experiments were carried out with thinner films at a P/B of 0.5. It was at this level of pigmentation that the effect of adding coloured tinters was first studied.

Finally, it was found that the only way to produce really good gloss comparable with that given by spraying was to neutralise the acidity of the

deposited film by means of an alkaline after-treatment, preferably consisting of the exposure of the deposited film to an atmosphere of gaseous ammonia.

Two important properties have not been touched on in the present work. These are throwing power and the behaviour of the paint during long-term trials. It is believed that paints made from Synocryl 840S and titanium dioxide pigments should have reasonably good throwing power, but the likely behaviour of a paint during exhaustion and subsequent replenishment of the bath cannot be guessed. Further experiments would therefore have to be done before such paints could be used on a plant scale.

Materials

Rutile pigments of the following types were among those investigated :

Code	Pigment type
1a	High durability
1b	High durability, high opacity
1c	High durability, fast dispersing
2a	Uncoated rutile
3a	Good durability
3b	Good durability, high opacity
3c	Good durability, emulsion paint grade
3d	Good durability, high opacity, zinc-free
4a	Fine particle
5a	Chloride process, high opacity
6a	Silicone treated
7a	Phosphate treated

The pigments which gave the best gloss were pigments *1b* and *3b*, which were, respectively, Tioxide R-CR3 and Tioxide R-HD3.

In the production of pastel-tinted finishes, the following coloured pigments, manufactured by ICI Dyestuffs Division, were used :

Monolite Fast Red YS (Dibromoanthranthone)
 Monolite Fast Yellow 10GS (4-chloro-2-nitroaniline-o-chloroacetoacetanilide)
 Monastral Fast Blue LBXS (Copper phthalocyanine (α form))
 Monastral Fast Green GNS (Polychloro copper phthalocyanine)

Experimental method

The cell used in most of the experiments was a glass tank 15 cm internal diameter and 7.5 cm deep into which was placed 1 litre of paint. The paint was continuously stirred with a magnetic stirrer. Three electrodes were arranged

in the tank so that the central anode was parallel to, and equidistant from, a cathode placed on either side of it. The electrodes were clamped to a moveable superstructure which could be placed over the tank in a reproducible position, so that the spatial arrangement of the system was always the same. Each electrode was 10 cm long and 5 cm wide and each was placed in a vertical position so that only the lower half (5×5 cm) was immersed in the paint. The distance between the anode and each cathode was fixed at 5.5 cm in all experiments, after it had been established that film properties were largely independent of this distance.

The cathodes were made from 18 swg (0.122 cm thick) pure titanium metal sheet. In most of the experiments the anode was made from a proprietary brand of zinc plated mild steel (18 swg) which had the appearance of having been hot rolled prior to plating. The thickness of the zinc plating was approximately 30 microns (μm). In some experiments the anodes were made from a similar steel which was plated in the laboratory; in other experiments a high quality cold rolled steel was similarly plated.

The electrical power for the cell was taken from a full-wave rectifier and smoothing unit delivering a maximum of 120 volts DC. This unit was fed from the mains through a Variac transformer so that a DC potential, continuously variable from 0 to 120 volts, was available. The maximum current available was 3 amp.

The voltage across the cell and the current passing through it were indicated on two Model-8 Avometers. Either the voltage or the current could also be plotted continuously as a function of time with a Bryans 20180 X/Y plotter, with the X-axis running as a time base.

For constant voltage operation, some adjustment of the setting of the Variac transformer was required during deposition to maintain the potential at the chosen value, since the regulation of the power pack was poor.

For operation at constant current the transformer was continuously adjusted to maintain the current at the value required. Greater convenience and precision were achieved during later experiments when the power pack was replaced by a Constant Current Power Supply, type KTI 1010, manufactured by KSM Electronics Ltd. This unit supplies constant current free from ripple over the range 0.030 to 10 amp at voltages ranging up to 120 volts.

Prior to deposition each anode was weighed. Deposition of the paint was then carried out and the anode removed from the bath. The excess liquid (dip coat) was washed off with running, cold water and the painted anode was dried for one hour at room temperature. The dried film was stoved for 30 minutes at 150°C , after which the anode was weighed again. The area of the paint film was measured accurately, allowance being made for the thickness of the edge, and the weight of paint per cm^2 was calculated.

The pH of the paint in the cell was measured, before and after deposition, with a pH meter. The conductance of the paint was measured with a Wayne Kerr universal AC bridge, and the specific conductance of the paint could be calculated from the measured conductance of the cell when filled with

M/100 potassium chloride solution. The pH of paint films was measured immediately after deposition with BDH universal indicator.

Measurement of the P/B in the cured paint film was effected by scraping a small quantity of the film from the metal and weighing it in a silica crucible. A second weighing was made after the film had been ashed by heating for three hours at 700°C.

Measurements of film thickness were made with a sensitive dial gauge, giving a random error of $\pm 1 \mu\text{m}$ for the mean of four readings, i.e. ± 4 per cent on a film 25 μm thick. The readings were taken by allowing the anvil of the gauge to fall from the continuous paint film on to the underlying metal, over a step which was made by carefully removing a small area of the film. Systematic errors were possibly greater than ± 4 per cent, however, and it was generally preferred to calculate the film thickness from weight measurements and the calculated density of the film.

Colorimetric comparison of white panels was made by measurement with a Harrison colourmeter. The colour index (CI) is given by :

$$100 \times \frac{(\text{reflectance in red light}) - (\text{reflectance in blue light})}{(\text{reflectance in green light})}$$

Increasing positive values of the CI indicate increasing yellowness ; negative values indicate blueness. The reflectance in green light is taken to give a measure of brightness.

Measurements of gloss were made at 20° or at 45°. The gloss values reported are the percentage reflectances at these angles, the reflectance of black glass being taken as 100 per cent.

The effect of temperature variation was not studied, but all operations, including the air-drying of the painted panels, were carried out in a laboratory maintained at a constant temperature of 20°C and at a constant relative humidity of 65 per cent.

All of the paints used were prepared by ball milling in accordance with the methods developed in the Technical Service Department of British Titan Products Co. Ltd. (see Appendix 1).

Appendix 2 gives the method used when steel panels were plated with zinc in the laboratory.

Results

Preliminary

Paints were prepared from pigment *Ia* at various values of P/B and at a solids content of 20 per cent. These paints were deposited at constant current. It was found that there was a limit of about 90 to 100 volts below which films of good appearance were obtained, and above which evidence of gas evolution during deposition could be seen in the paint films. To be satisfactory, a film had to be deposited before this voltage limit was reached. The curves of voltage as a function of time showed increasing slope with increasing P/B (Fig. 1). A satisfactory film thickness could not, therefore, be built up at P/B=1.0 or at P/B=0.75, because the number of coulombs, and hence the film thickness, was limited by the voltage rise.

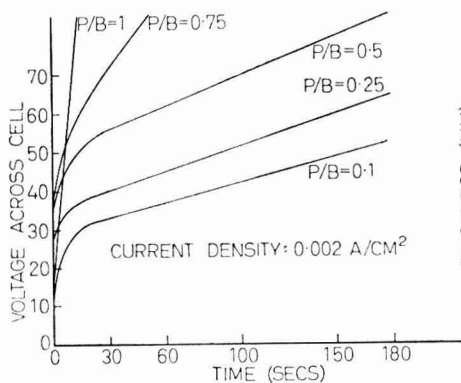


Fig. 1. Variation of voltage with time for various values of P/B

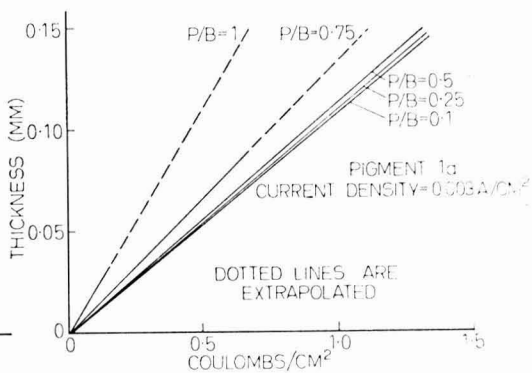


Fig. 2. Effect of P/B on the rate of increase of film thickness

It was found also that there was a slight reduction in coulomb yield as the P/B was reduced (Fig. 2). Slight gloss was noted at $P/B=0.5$. At $P/B=0.25$, 45° gloss was 60 per cent and 20° gloss was 15 per cent. Above $P/B=0.5$ the films produced were matt, and it was found possible to rub pigment from the surface of cured films deposited from paints of $P/B=1.0$ and 0.75 .

Variation of P/B in deposited film

The above results led to an investigation of the relationship between the P/B in the paint and in the deposited film. Twelve paints were prepared with pigment *1a* at a P/B of 0.25. Depositions were carried out at constant current for periods ranging from 30 seconds to six minutes. The P/B for each paint film was measured, and was taken to represent a mean value for the whole thickness of the film. It was found that the P/B invariably increased with the duration of deposition, and hence with the thickness of the film.

For convenience, the mean pigment concentration, \bar{p} , was calculated from the P/B . The film thickness was calculated from the weight of the film and from the P/B , assuming densities of 4.0 for the pigment and 1.0 for the resin. When \bar{p} is plotted against film thickness (Fig. 3) the curve shows the growth of the mean pigment concentration with increasing film thickness. To determine

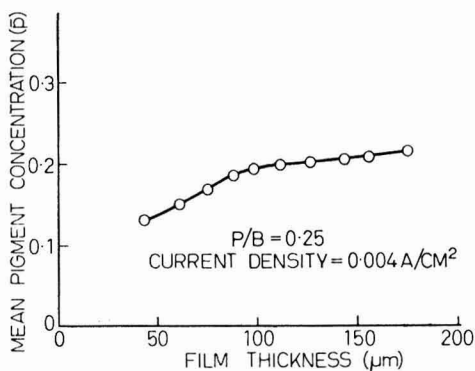


Fig. 3. Variation of the mean pigment concentration with the thickness of the deposited film

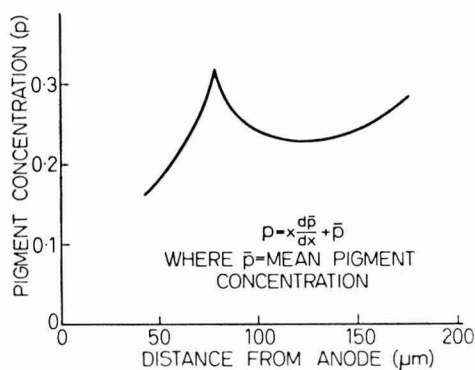


Fig. 4. Variation of the local pigment concentration through the thickness of the deposited film

the way in which the pigment concentration itself varies through the film, it is necessary to differentiate this curve. At a distance x from the metal/paint interface, it can be shown that the pigment concentration is given by :

$$p = p + x \frac{dp}{dx}$$

Values of p calculated from Fig. 3 are plotted in Fig. 4. Qualitatively, the examination of electron micrographs of a section from an electrodeposited film confirmed the presence, shown in Fig. 4, of a region adjacent to the substrate which was deficient in pigment.

Since the P/B of the paint in the bath is 0.25, the P/B in the paint film should ideally be the same. This would correspond to a value of :

$$p = p = \frac{0.25}{1.25} = 0.20$$

Fig. 4 shows that, in the early stages of deposition, p is less than 20 per cent and, thereafter, is greater than this value. Thick films, in particular, have a high concentration of pigment at the surface.

The effect of pH

The effect of the pH of the paint on the gloss of the deposited film was investigated. For this experiment, a paint at P/B=0.1 and 20 per cent solids was used since it was found that gloss could be measured more accurately at this value of P/B. The pH of the paint was changed by thoroughly stirring either N acetic acid or N ammonia into the paint. Changes were made in the range 8.1 to 8.8. The natural pH of the paint was found to be 8.3 and adjustments were made towards pH 8.8 with ammonia, and then back towards pH 8.1 with acetic acid. The final adjustment was made back to 8.3 with ammonia. Additions of either alkali or acid increased the specific conductance of the paint, but the gloss of the paint films produced did not appear to be a function of the specific conductance, but, rather, a function of the pH of the paint (Fig. 5). The paint appeared to have a fairly strong buffering action at about pH 8.3. Experiments with paints of P/B other than 0.1 gave a similar optimum for a pH of 8.3.

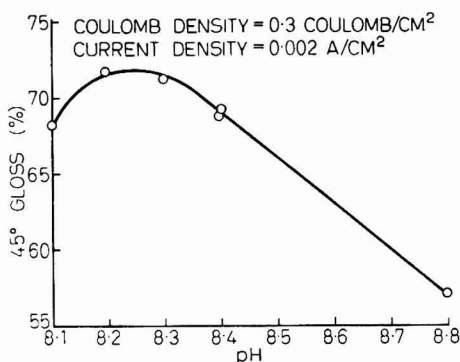


Fig. 5. The effect of the pH of the liquid paint upon the gloss of the deposited film

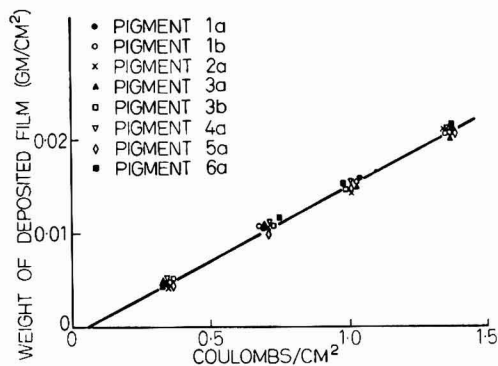


Fig. 6. The coulomb yield for various pigments at P/B=0.25

Measurements of pH were made on a number of paint films, immediately after deposition, with BDH universal indicator solution. Values were obtained for a number of different resins, both pigmented and unpigmented. In every case, the pH in the film was in the region of 5.0 to 5.5.

The choice of pigment

The gloss attainable with different grades of rutile titanium dioxide was investigated, first in a series of paints prepared at $P/B=0.25$. A second series of paints was prepared at $P/B=0.5$. Gloss values for each series were measured at 45° . In the results given in Table 1, the omission of a result implies that the pigment was not investigated.

Table 1
Gloss at 45° given by various pigments

Pigment	Gloss ($P/B=0.25$)	Gloss ($P/B=0.5$)
1a	60	49
1b	66	73
1c	—	57
2a	49	—
3a	64	—
3b	70	78
3c	—	44
3d	—	69
4a	64	43
5a	66	72
6a	54	—
7a	—	52

Graphs of weight per cm^2 against coulombs per cm^2 showed that, in each series, the choice of pigment has little effect on the coulomb yield. This result is illustrated for the first series ($P/B=0.25$) in Fig. 6, in which the best straight line drawn through all the points gives a mean yield of 0.015 grams of paint per coulomb. The coulomb yield for paints at $P/B=0.5$ is similarly calculated to be 0.023 grams per coulomb.

The above values for gloss, together with the visual appearance of the panels, lead to the conclusion that high opacity pigments tend to give better gloss at the higher values of P/B , and that pigments *1b* and *3b* give the best gloss.

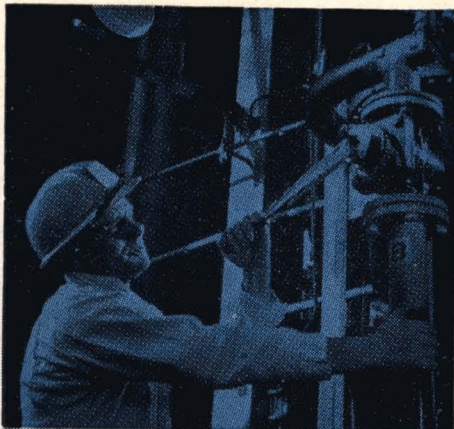
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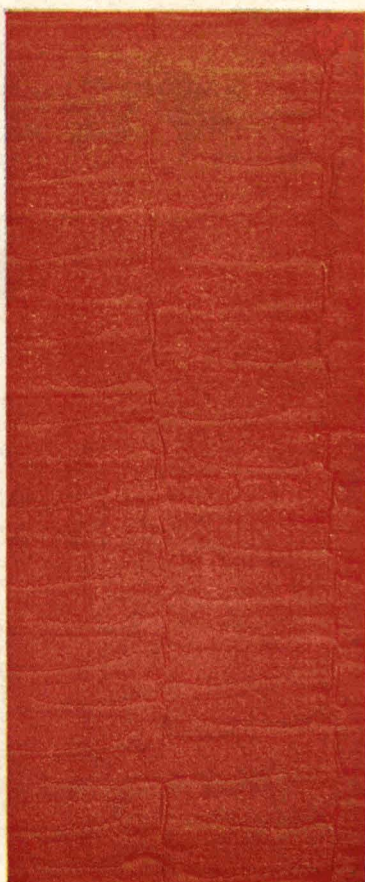


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The effect of surface-active agents

Earlier work suggested that the coagulation of the paint to form a film on the anode might be too severe and that gloss might thus be reduced. It was thought that the addition of an anionic surfactant might alter this situation. Paints were prepared using pigments *1a* and *1b* at $P/B=0.5$ and 20 per cent solids, and a 5 per cent solution of sodium hexametaphosphate (hereafter referred to as NaHMP) was added to them to give a range of addition from 0 to 0.3 per cent NaHMP on the weight of the pigment. Graphs of 45° gloss as a function of per cent NaHMP on the weight of the pigment were prepared (Fig. 7). A further experiment was carried out using the same two pigments at $P/B=0.25$, with the same range of NaHMP additions.

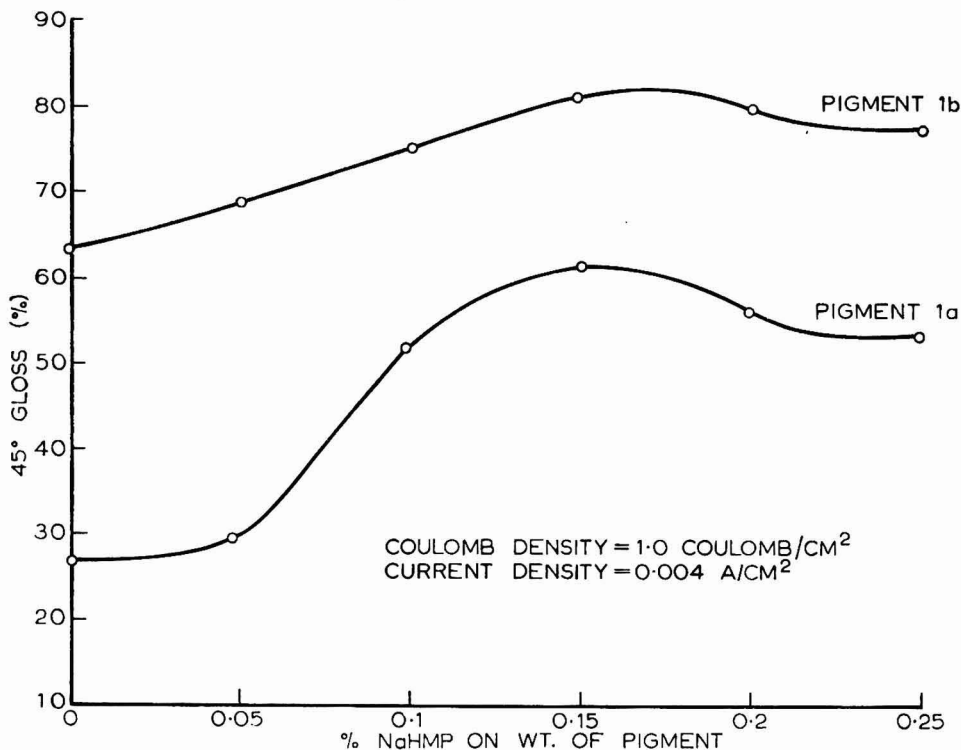


Fig. 7. The effect of additions of sodium hexametaphosphate upon gloss

These experiments were repeated with the NaHMP additions being made to the millbase, and the results were compared with those given by the NaHMP additions to the finished paint. The results showed that the optimum addition to each pigment is 0.15 per cent NaHMP on the weight of the pigment. Similar results were obtained by adding NaHMP to the millbase or to the finished paint. The most interesting result obtained from the graphs is that at $P/B=0.5$, with pigment *1a*, the gloss increased from 27 to 60 per cent. Smaller increases were noted with pigment *1b*. At $P/B=0.25$, the increases in gloss were slight. The addition of NaHMP had little effect on the coulomb yield, or on the slope of the curve of voltage against time at constant current.

The additions did not effect the pigment distribution in the deposited film, nor did they appear to have much effect on the electrical conductance of the paints.

Further experiments, with non-ionic surfactants, produced no increase in gloss. Experiments with amino-hydroxy compounds, which tended to increase the pH of the paints, served only to reduce the coulomb yield. No increase in gloss was observed, whether or not the pH was adjusted to its original value.

An interesting effect was observed with additions of ammonium polyacrylate at a level of 1 per cent on the weight of the resin. Two polyacrylates were used, for which the estimated molecular weights were 2,000 and 20,000 respectively. The two materials had a similar effect. No significant change in the gloss or in the coulomb yield was observed, but the gradient of the voltage/time curve was reduced. Additions of ammonium polyacrylate would therefore permit the passage of a greater number of coulombs below the limiting voltage at which gassing occurs. This would make it possible, for instance, to deposit films of adequate thickness at high values of P/B (see Fig. 1).

The effect of organic solvents

Experiments were conducted to study the effect of plasticising the film temporarily, in order to give more fluidity during drying, in the hope that gloss would be increased. Paints were prepared with pigment 1a, at P/B=0.5 and solids content of 20 per cent. The mill base was diluted to 20 per cent solids by the addition of a water/butanol solution, so as to give a range of butanol additions from 0 to 50 per cent of butanol on the weight of the solid resin. It was found that the addition of more than 20 per cent butanol on the solid resin produced films that were disrupted or removed when the dip coat was washed off. A reduction in 45° gloss was noted with the addition of 10 per cent butanol, and there was no improvement in gloss with 15 per cent butanol. At 20 per cent butanol, an improvement in gloss was noted, together with increased smoothness of the deposited film (Fig. 8). The addition of butanol had little effect on the coulomb yield, for additions up to 15 per cent. With 20 per cent butanol, the coulomb yield was slightly reduced. Some reduction was noted in the slope of voltage as a function of time at constant current.

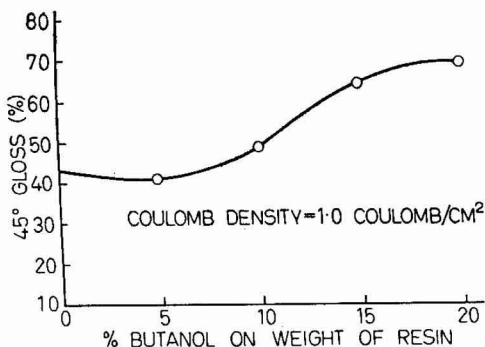


Fig. 8. The effect of additions of butanol upon gloss

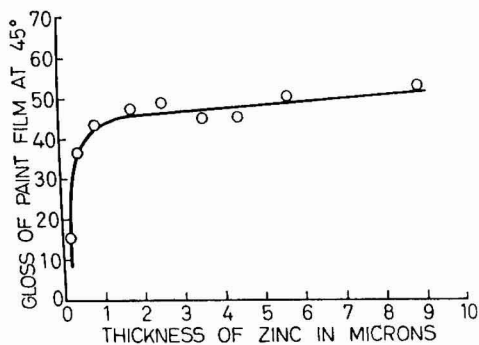


Fig. 9. The effect of the thickness of the zinc plating upon the gloss of the deposited paint film

The effect of the thickness of the zinc plating

The mean thickness of the zinc on the plated steel which was obtained commercially was about 30 μm . To investigate the possible effect upon gloss of the thickness of the zinc, a series of steel panels was plated in the laboratory with zinc at various thicknesses. The panels were coated with paint made from pigment 1b at P/B=0.5, the application density being kept constant at 0.019 g/cm². The values of gloss for the paint films, measured at 45°, are plotted in Fig. 9, which reveals a lowering of the gloss when the thickness of the zinc is less than about 2 μm .

Production of pastel tints

Paints were made from pigment 1b at P/B=0.5, total solids=20 per cent, and were tinted with the red, yellow, blue and green pigments previously referred to. These were each added at levels of 1 per cent and 2 per cent on the weight of the titanium dioxide (see Appendix 1).

Deposition at 4 milliamps/cm² gave good paint films and the presence of the coloured pigment appeared to have a negligible effect upon the shape of the volts/time curve and upon the coulomb yield.

From each paint, 12 successive panels were coated, thus removing approximately 8 per cent of the total solids from the bath. The tone of every panel was clean, and the colours were comparable to those normally given by sprayed finishes. With each paint, the difference in chromaticity between the first and last panel was either imperceptible or almost so. Visual assessment was born out by Beckman spectrophotometer curves which, only in the case of the yellow tints, indicated some fluctuation in reflectance at the blue end of the spectrum, and in no case established a trend with the order in which the panels were prepared.

The colour of the panels appeared to be independent of film thickness, but removal of the surface always revealed a lighter tint underneath. This phenomenon suggests either flooding during stoving, or possibly a difference in the degree of dispersion at the surface of the film, since chemical analysis of the blue paint films showed that the ratio of TiO₂ to phthalocyanine was constant, and equal to that in the bath, within limits of ± 2 per cent.

All the coloured panels gave better visual gloss than white panels which were prepared under the same conditions. This observation was substantiated by measurement, as follows :

Addition	45° Gloss
None	77
1% red	88
2% red	88
1% yellow	85
2% yellow	83
1% blue	90
2% blue	88
1% green	89
2% green	86

These values were maintained during successive depositions from the blue and the green paints, but the last panels to be coated with the red and the yellow paints had gloss values reduced to about 55.

Improvement of gloss with ammonia

Measurements of the pH of freshly deposited paint films had always given low values, in the region 5 to 5.5. It therefore seemed certain that the pigment in any deposited film must be flocculated, and this was confirmed by electron micrographs which showed that, even though NaHMP improves gloss, it did not improve the state of dispersion of the pigment to the degree reached in a paint film applied by conventional means. Furthermore, the clear layer which is present on the surface of a normal paint film, according to electron micrographs published, for instance, by Hibberd⁴, was absent from an electro-deposited film.

To permit redispersion of the pigment in an electrodeposited film, it seemed logical to replace the alkali which was removed during deposition. The following methods were tried, with paints made from pigment *1b* at a P/B of 0.5, deposited at 0.004 amp/cm² :

Treatment with gaseous ammonia : A quantity of .880 ammonia was placed in the bottom of an empty desiccator. Panels were coated at 0.025 g/cm², washed in the usual way, and placed immediately in the desiccator in a vertical position for periods of 3, 10 and 30 minutes. The panels were then dried overnight before stoving. All the panels treated with ammonia showed blemishes caused by the formation of small bubbles (0.1 to 0.3 mm diameter) during stoving. An attempt to eliminate bubbling by heating panels to 50°C for 30 minutes before stoving was unsuccessful. Finally, the bubbles were eliminated by reducing the deposition time and hence reducing the application density to 0.019 gm/cm². The visual gloss was greatly improved by the treatment with ammonia, and there was no visual difference between the three treated panels. Gloss values measured at 20° and 45° were as follows :

	Gloss	
	20°	45°
No ammonia	11	70
3 min. in ammonia ..	45	77
10 min. in ammonia ..	32	79
30 min. in ammonia ..	47	82

In a second experiment, the washed panels were allowed to dry for three hours at room temperature before being placed in the ammonia atmosphere. Gloss values were slightly lower than in the preceding experiment and an exposure of 30 minutes was necessary to give the full improvement in visual appearance.

From the point of view of gloss, the appearance of panels exposed to ammonia for three minutes (wet) or 30 minutes (dry) was closely similar to

that of a paint film applied by spraying on to an identical substrate. However, the measured gloss values achieved by spraying were higher: 91 at 45° and 61 at 20°. The discrepancy was attributable to two factors. First, the surface finish of the substrate (commercially obtained zinc-plated steel) was poor, and the surface irregularities appeared to be followed more closely by the electro-deposited film than by the sprayed film, giving a slightly deeper "orange peel" effect. Secondly, the ammonia treatment necessitated the use of a low density of application, and some irregularity in film thickness resulted from this. The irregularity recurred over the whole of the surface with a relatively high modulus of approximately 1 or 2 mm and was not entirely removed by the viscous flow which the ammonia treatment permitted.

Panels of cold-rolled steel having a good surface finish were plated with zinc in the laboratory. Paint films deposited on these panels were greatly superior in appearance, but still exhibited surface irregularities of high modulus. Although the visual gloss was now possibly superior to anything that could be achieved by spraying, the measured values were still low.

Photomicrographs of polished sections through paint films electrodeposited on to the two types of substrate are reproduced in Fig. 10. They illustrate the way in which the surface of an electrodeposited paint follows the irregularities of the substrate which, in the case of the commercially obtained material, are apparently due in part to gross variation of the thickness of the zinc plating. The irregularities of high modulus in the paint films are not clearly shown.

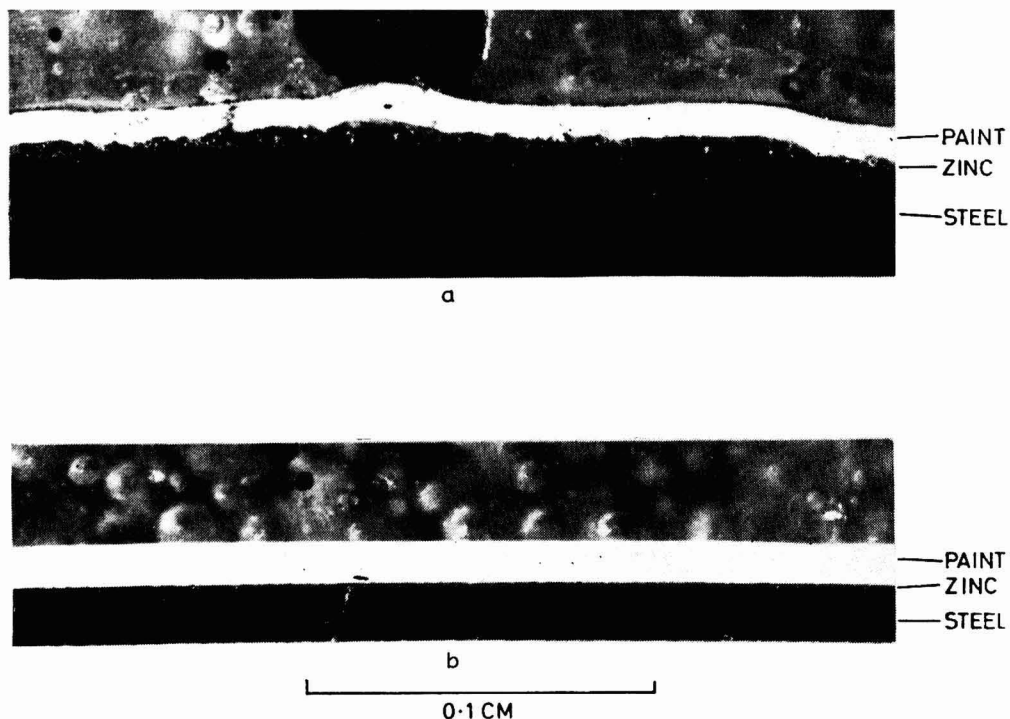


Fig. 10. Sections of electrodeposited paint films on two substrates showing (a) a proprietary zinc-plated steel and (b) a cold rolled steel plated with zinc in the laboratory

Treatment by dipping : Panels coated at an application density of 0.019 g/cm^2 were washed in the usual way and then dipped in various aqueous solutions before being allowed to dry at room temperature. The duration of immersion in each solution was about five seconds. Solutions of urea were included on the supposition that thermal decomposition could liberate ammonia in the paint film during stoving.

The following solutions gave a moderate improvement in gloss, but in no case did the improvement match that given by the gaseous treatment :

N/10 ammonia
5 per cent urea
5 per cent ethylene diamine

Solutions more dilute than the above gave little or no improvement. Solutions of ammonia stronger than N/10 caused the paint film to drip from the panel.

Colour and opacity of white deposited films

Visual comparison showed clearly that electrodeposited films gave increased opacity when treated with gaseous ammonia, owing to the improvement in dispersion thereby achieved. The opacity of an electrodeposited film, after treatment with ammonia, appeared to be comparable with that of a sprayed film, but, because of practical difficulties, the opacities were not measured.

A paint made from pigment *1b* at P/B=0.5 was electrodeposited thickly (0.025 g/cm^2) on to zinc-plated steel and the colour was compared with that given by a similar paint applied in a single coat, sprayed on to white tinplate. Values for the colour index and brightness were as follows :

	CI	Brightness
Electrodeposited	3.8	92.1
Sprayed	4.2	89.0

The colour indices were thus similar, each paint giving a slightly cream tone, and the electrodeposited paint was perceptibly brighter.

To study the effect of the ammonia treatment upon colour, a series of paint films was deposited at a lower application density (0.019 g/cm^2). Half of the panels were treated with gaseous ammonia before stoving. The measured results were as follows :

	CI	Brightness
Treated	5.2	90.5
Untreated	3.7	90.0

The treated panels were thus slightly more cream in tone than the untreated panels.

Artificial weathering trials

A detailed study of durability remains to be carried out, but a preliminary comparison of sprayed and electrodeposited coatings has been made with a Marr Weatherometer.

Each method of coating was used on a number of substrates, detailed below. Pigment *1b* was used throughout at a P/B of 0.5. The application density for the sprayed coatings was 0.020 g/cm² and for the electrodeposited coatings 0.025 g/cm². A larger cell was used for the electrodeposition so that panels measuring 15 × 5 cm could be used. These were painted on one side only and a narrow strip at one end was left uncoated. After painting, the edges of each panel were filed at right angles to the face, so as to expose the metal.

The panels were assessed qualitatively after 650 hours in the weatherometer, by which time rust had spread inwards from the edges, and rust patches had appeared on the face of each panel sufficient in number to render quantitative comparison of colour and gloss unreliable. Results are shown in Table 2.

Table 2

	Substrate	Gloss	Colour	Rusting
Sprayed coatings	Mild steel	Good	White	Severe
	Phosphated steel	Good	White	Severe
	Primed (iron oxide) steel	None	White	Slight
	Zinc-coated steel	Poor	White	Moderate
Electrodeposited coatings	Mild steel	None	Yellow	Severe
	Phosphated steel	None	Yellow	Severe
	Zinc-coated steel	None	Yellow	Severe

The yellowing of the electrodeposited panels was gross and suggestive of iron contamination. It was fairly uniform and could perhaps have come from the water spray itself rather than from the edges of the panels. The spray water used in the weatherometer was de-ionised steam condensate and did, in fact, contain about one part per million of colloidal iron. It seems likely that an affinity exists between colloidal iron and the resin in an electrodeposited paint film. The discoloration existed only at the surface of the film; scraping with a knife showed that the paint underneath had remained white.

Discussion

Whereas it is a common experience that, in certain resin systems, different titanium dioxide pigments give widely differing performance during electrodeposition, it has been shown in the present work that with Synocryl 840S the choice of pigment has little effect upon the electrical properties of the paint. The only property of the paint film which shows marked dependence upon the choice of pigment is gloss. The deposited paint film is invariably acid, as noted by Burden and Guy¹, and the pigment is invariably flocculated

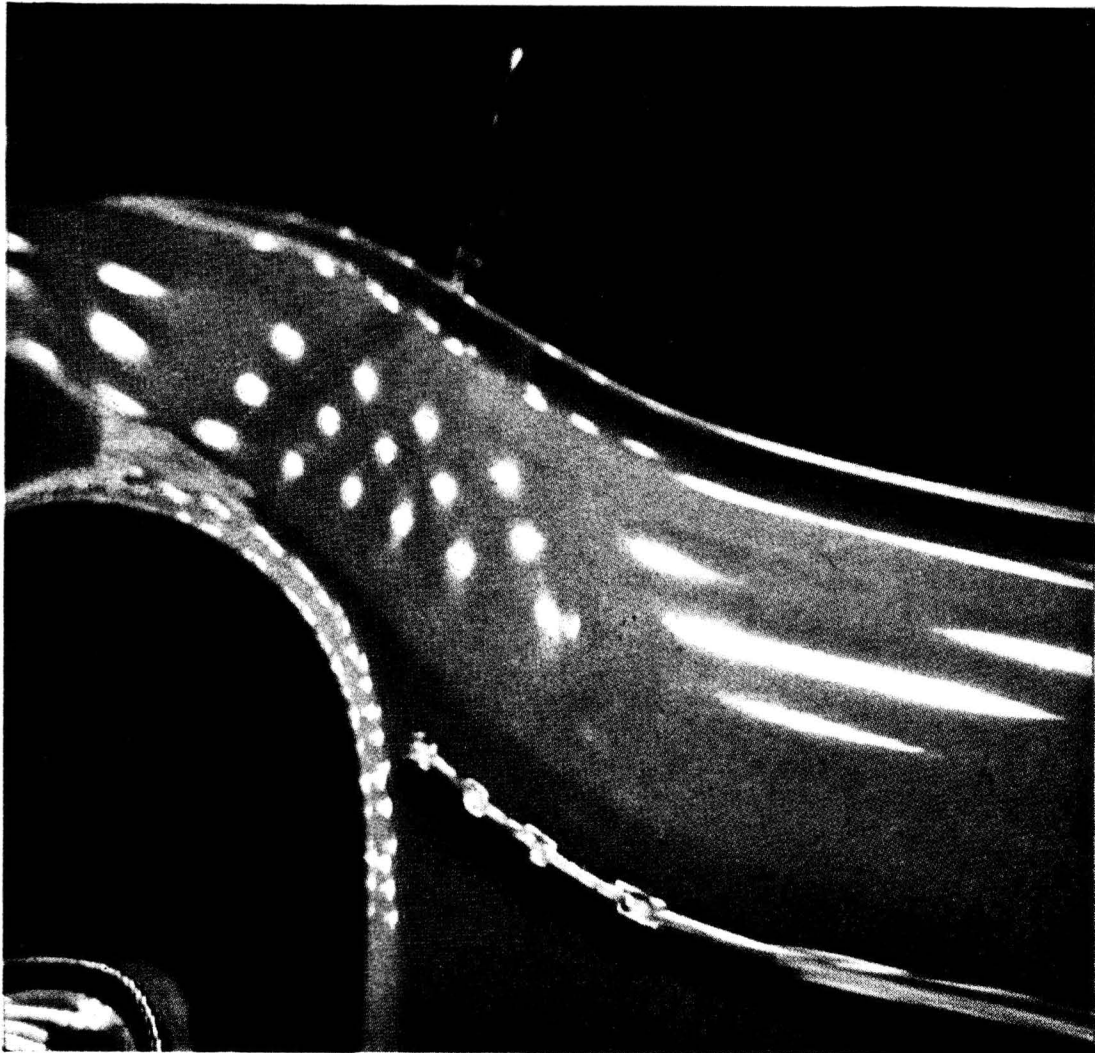
in the film. Nevertheless, the best gloss is given by the superopaque pigments such as Tioxide R-CR3 and R-HD3. Although comparative measurements have not been made, it is likely that these pigments will also give the best values for other desirable properties such as hiding power, tint reduction, etc.

It has been shown that gloss can be improved by additions to the paint of surface-active agents and organic solvents, but that, when the superopaque pigments are used, the improvement is only marginal. In view of the probable difficulties of formulation control during exhaustion of the bath, it is doubtless better to avoid such additions if possible. Since no additive has been found which gives better gloss with an ordinary pigment than that which is achieved with a superopaque pigment alone, there seems little point in using any of the additives which have been studied. The only exception is ammonium polyacrylate, which gives some control over the shape of the volts/time curve, and hence over the thickness of the deposited film.

The production of pastel tints, in so far as this has been studied, seems to be an uncomplicated procedure. No difficulties due to pigment separation have been encountered, and the appearance of the deposited paint films has been excellent. The falling off of gloss with successive depositions from the red and yellow paints is possibly connected with the presence of surfactant on the surface of the tinting pigments. The blue and the green paints did not exhibit this defect, and doubtless many other coloured pigments will be found to give consistent results. The superopaque pigment used in these coloured paints, Tioxide R-CR3, gave exceptionally clean tints fully up to the standard achieved by traditional methods of application.

The pH of every deposited film which has been examined has been found to lie in the region 5.0 to 5.5, and there is every reason to believe that a similar result would obtain with any resin which is deposited anodically. Not only is the rutile pigment flocculated, but the resin is coagulated to give a film in which flow is impossible; indeed, this is the reason why a coherent film is formed in the first place. Despite the above limitations, the gloss given by the superopaque pigments is quite good and is certainly adequate for many types of appliance finish.

In order to produce an electrodeposited paint film in which the pigment is properly dispersed, as in a film applied by conventional methods, it is essential to raise the pH. This can be done logically by replacing the ammonia (or amine) which has been removed from the resin and transferred to the cathode during deposition. The advantage of using ammonia is that it can be applied to the film in excess, the excess being automatically removed by subsequent evaporation. It has been found convenient to apply the ammonia from the gas phase immediately after the freshly deposited film has been washed. Such a film, though tough, is porous, and it appears to absorb some of the film of water on its surface, swelling in the process. Clearly, the application of an ammonia atmosphere at this stage will lead to very rapid solution of the gas throughout the depth of the paint film. The coagulated resin becomes miscible once more with water, limited flow is possible within the film, and the pigment is again able to form a stable dispersion. The mechanism by which this dispersion is achieved is not obvious and invites speculation.



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If the paint film is too thick, the application of gaseous ammonia causes bubbling during subsequent stoving. On the other hand, since the pigment is properly dispersed by this treatment, the thickness of the film can be reduced without loss of opacity. This eliminates bubbling and simultaneously reduces the cost of the process.

By visual assessment, the gloss developed with the ammonia process is equal to that achieved by spraying. There remains, however, an undulation at the surface of the film, having a modulus of one or two millimetres, which interferes with instrumental measurements of gloss and which detracts to some extent from the appearance of the finish. It seems to be due to localised variations in the application density during deposition of a thin paint film. It is a minor problem, but one which calls for further study.

Another type of defect which lowers the gloss is the reproduction by the paint film of the surface texture of the substrate. This seems to be a characteristic of the electrodeposition process which can only be overcome by using a smooth substrate, and it forms a problem which will have to be faced if high quality finishes are to be produced by a single-coat process.

With regard to colour and brightness, the quality of electrodeposited whites is equal to the standard set by sprayed finishes. Treatment with ammonia results in a perceptible yellowing of the film, to an extent which would seldom be considered undesirable.

Preliminary results for durability of electrodeposited films are disappointing, but these apply only to acid films which have not been neutralised with ammonia. It is possible that the ammonia treatment may give better resistance to staining and better retention of gloss, but experimental evidence for this is required.

If zinc plated steel is used as a substrate, the quality of the zinc plating is important. The fact that gloss was reduced when the thickness of the zinc was less than 2 μm suggests that plating in the laboratory gave a somewhat porous deposit. (This was expected, since the method of plating was chosen primarily for convenience.) On the other hand, the commercially obtained material was plated very unevenly and the zinc, which was about 30 μm thick, gave little protection against corrosion under the condition of test.

Conclusions

The present work shows that, in the laboratory, one-coat white and pastel-tinted glossy finishes can be applied to a metallic substrate by electrodeposition. On zinc-plated steel the colour of the deposited films is comparable with that obtained in sprayed coatings; likewise the visual gloss, provided that an after-treatment with gaseous ammonia is used to achieve proper dispersion in the paint film.

The fact that these results can be achieved with commercially available materials suggests that the possibility of applying glossy whites and pastel tints in a production line would be worth investigating. When a suitable medium is chosen there is no special problem in using rutile pigments. It is particularly beneficial to employ the recently developed superopaque pigments if the highest gloss is required.

For the best results, the metallic substrate itself should have an adequately smooth surface. The successful use of electrodeposition for one-coat finishes will therefore depend upon whether the economic benefits of the method outweigh the extra cost of using a polished substrate.

Acknowledgments

The authors wish to express their thanks to the Directors of British Titan Products Co. Ltd. for permission to publish this paper. They also thank their colleagues in the Technical Service and Development Departments of BTP who carried out experimental work or who gave helpful advice.

[Received 11 August 1965]

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Appendix 1

Formulation of white paint

Milling was carried out over a period of 18 hours by trundling in 1,200 ml jars with 700 gm of 8 mm glass ballotini.

Mill base :

Rutile titanium dioxide	300 g
Synocryl 840S (reduced to 20 per cent non-volatile with de-ionised water)	150 g

Further resin and water were added as resin solution whilst stirring with a high speed stirrer, to obtain the required solids content and pigment/binder quotient. This method of addition eliminated the need for a separate stabilisation stage.

Formulation of tinter

Milling was carried out over a period of 18 hours by trundling in 225 ml jars with 150 g of 8 mm glass ballotini.

Mill base :

Coloured pigment	20 g
Synocryl 840S (reduced to 15 per cent non-volatile with de-ionised water)	35 g

Stabilisation :

Synocryl 840S (reduced to 25 per cent non-volatile with de-ionised water)	45 g
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The tinter was added to the finished white paint and incorporated with a high speed stirrer.

Appendix 2

The zinc plating of steel

The steel was first degreased in the mixed vapours of ethanol and trichloroethylene.

Chemical polishing was then carried out at a temperature of 35°C in the following solution :

80 ml	distilled water
28 ml	oxalic acid (100 g/l)
4 ml	hydrogen peroxide (30 per cent)

The steel was then zinc plated at a current density of 0.029 amp/cm² with a solution of the following composition :

300 g	zinc sulphate (ZnSO ₄ .7H ₂ O)
12.5 g	sodium chloride
19 g	boric acid
19 g	aluminium sulphate (Al ₂ (SO ₄) ₃)
1,000 g	distilled water

Microelectrophoresis of pigment particles

By S. Wilska

Vuorikemia Oy, Pori, Finland

Summary

A method is described for the measurement of the electrophoretic velocity of particles in a few drops of a suspension in which no special instruments are required. Only an ordinary microscope, a dry cell and two small platinum electrodes are needed for the determination of the sign of the charge on the particle. The apparatus can be improved by adding a thermocouple for temperature measurement and a camera for microphotography. Two photographic techniques have been developed for recording the direction and velocity of migration of the particles. A further improvement is the use of a heating or cooling stage to regulate the temperature of the sample.

Microélectrophorèse des particules de pigment

Résumé

Cet article décrit une méthode—n'ayant pas besoin d'appareil spécial—pour déterminer la vitesse électrophorétique des particules dans quelques gouttes d'une suspension. Afin de déterminer le signe de la charge de la particule, on a besoin d'un microscope normal, une pile sèche et deux petites électrodes en platine. L'appareil peut se perfectionner par l'introduction d'une thermocouple pour mesurer la température et aussi d'une chambre pour faire des microphotographies. Deux techniques photographiques furent mises au point pour enregistrer la direction et la vitesse de migration des particules. Une autre perfectionnement peut s'obtenir par l'emploi d'une platine de chauffage ou de refroidissement pour contrôler la température de l'échantillon.

Mikroelektrophorese von Pigmentpartikeln

Zusammenfassung

Es wird eine Messmethode beschrieben, mit Hilfe deren die elektrophoretische Geschwindigkeit der Teilchen in nur wenigen Tropfen einer Suspension bestimmt wird, für die keinerlei spezielle Instrumente benötigt werden. Um das Vorzeichen der Aufladung der Partikel zu bestimmen, braucht man lediglich ein gewöhnliches Mikroskop, eine Trockenzelle und zwei kleine Platinelektroden. Man kann die Apparaturen noch dadurch verbessern, dass man zusätzlich ein Thermoelement zur Messung der Temperatur und eine für Mikrophotographie geeignete Kamera anwendet. Um die Richtung und Geschwindigkeit der Teilchenwanderung zu registrieren, wurden zwei photographische Techniken entwickelt. Eine weitere Verbesserung besteht in dem Einsatz einer Plattform zur Erhitzung und Abkühlung, um die Temperatur der Probe regulieren zu können.

Микроэлектрофореза грунтовых красочных частиц.

Резюме

Описывается метод для измерения электрофоретической скорости частиц в нескольких каплях суспензии, который не требует специальных инструментов. Употребляется только обыкновенный микроскоп, сухой элемент и два маленьких электрода изготовленных из платины для определения знака заряда на частице. Этот аппарат можно улучшить посредством прибавления термопары для измерений температур и фотографического аппарата для микрофотографических снимков. Излагаются два фотографических способа для записи направления и скорости миграции частиц. Дальнейшее усовершенствование состоит из применения подогревательного или охлаждающего этапов для контроля температуры образца.

Introduction

The surface charges of small particles are known to influence greatly the phenomena that occur in suspensions of these particles. Recent developments in electrophoretic painting techniques have greatly increased the need to know how different paint ingredients in different media behave when exposed to electrostatic and magnetic fields. The selection of the best deflocculants for the effective dispersion of pigments in water-based paints and the control of flocculation and deflocculation in the manufacture of pigments require detailed knowledge of the signs and magnitudes of the surface charges on the pigment particles in water at various pH values and in the presence of cationic, anionic or non-ionic surface-active agents and other additives.

A close relationship exists between the charges of small particles and their electrokinetic properties. As in electrophoresis, an external electric field causes charged particles to migrate; the moving particles, in turn, produce an opposing migration potential. Other electrokinetic phenomena are electro-osmosis, the movement of a liquid past a solid in an electric field, and the opposing force, the streaming potential. All these phenomena have been studied to determine electrical interfacial properties; one of which is the *zeta* potential, the electrical potential in the plane between the fixed and flowing layers of liquid around a particle.¹

Electrophoresis is the method most commonly used to measure this potential, the magnitude and sign of which are decisive in most problems relating to dispersions. The expression for the electrophoretic velocity v_E derived by Hückel²,

$$v_E = \frac{\epsilon \zeta E}{6 \pi \eta} \quad (1)$$

where ζ is the *zeta* potential, η the viscosity of the medium, ϵ the dielectric constant of the medium and E the strength of the electric field, is now commonly accepted. From this expression it is seen that the *zeta* potential is directly proportional to the electrophoretic velocity of the particles in the medium.

The purpose of electrophoretic analytical techniques is, first to determine the velocity of the particles in a known electric field and the sign of the charge. As both the dielectric constant and the viscosity of the medium vary with temperature, it is necessary to measure and control the temperature in order to attain reliable results. Although the results of electrophoretic experiments may be misinterpreted, equation (1) is valid, especially when the electrophoretic velocity is low and the double layer around the particles is thin as it is, in general, in suspensions.

Instrumentation

The most common methods of studying electrophoretic behaviour are: (i) the moving boundary method, in which measurement of the displacement of the boundary between a colloidal solution and the colloid-free dispersion medium is used; (ii) a modification of the moving boundary method, known as the Tiselius method, and; (iii) microscopic and ultramicroscopic observation.

In the microscopic methods the movement of the individual particles is observed in a suspension or colloidal solution contained in a rectangular or circular cell. Because of secondary electro-osmotic phenomena at the walls of the cell, the matter is complicated and the quantity measured is actually the

sum of the two movements, electrophoresis of the particles and electro-osmosis of the liquid. It is, however, possible to determine where the velocity of the liquid is zero and the true electrophoretic velocity can hence be measured.

Electrophoretic measurements are carried out in a number of different ways using cells of different types. Apart from standard cells and ordinary microscopes, special instruments developed for this purpose are commercially available. As the use of ordinary cells and a microscope is too complicated and slow for the purposes here described, and commercial instruments are unnecessarily expensive, a new method has been developed which is described in this paper. Briefly, the method is as follows, a few drops of suspension are applied to a microscope slide, the objective of the microscope is immersed in the suspension together with two small platinum electrodes and a miniature thermocouple, and the movement of the particles observed visually or recorded by photography. When two photographs are exposed in succession, the whole procedure takes less than one minute and a new sample can be introduced in a few seconds. The optical and electrical arrangement is shown schematically in Fig. 1.

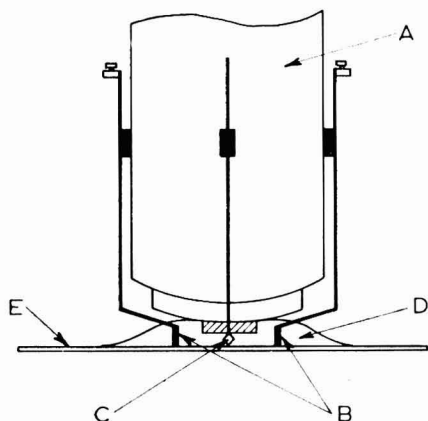


Fig. 1. 'Measuring' principle

- A = microscope objective body with an immersion lens
- B = platinum plate electrodes
- C = thermocouple
- D = sample
- E = microscopic slide

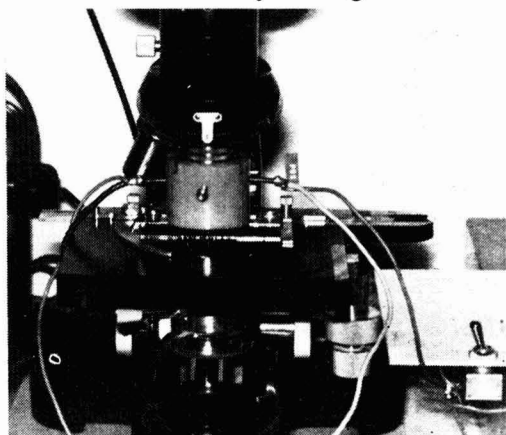


Fig. 2. Research microscope adapted for electrophoretic measurements. Electrodes and thermocouple are attached to the body of the objective by a plastic holder around it

The electrophoretic equipment in use in the laboratory is described in the following.

Electrophoretic equipment

Optical

The microscope used is an Ortholux Research Microscope, manufactured by Ernst Leitz GmbH, West Germany. It is equipped with a quadruple nosepiece, a binocular eyepiece and an attachment for a Leica camera for microphotography, and the sample can be illuminated by transmitted light. The most effective objective-eyepiece combination permits a linear magnification of up to $ca \times 1200$. The sample can be illuminated either by a standard microscope lamp or by an electronic flash.

Electrical

As shown in Fig. 1 (in principle only), two platinum electrodes (one cm² each, from a broken conductivity cell) and a miniature thermocouple are attached to the body of the objective by a plastic holder (Fig. 2). This arrangement permits the immersion of the lower edges of the electrodes and the thermocouple simultaneously with the objective into the sample drop resting on the slide. It is also possible to use an objective of non-immersion type to observe the particles through the air-liquid interface. The distance between the electrodes is kept constant at 20 mm. The electrodes are connected to a rectifier unit which in turn is fed through a Powerstat-type transformer, by means of which the DC potential between the electrodes can be regulated to an appropriate value. A polarity reversing switch facilitates the operation. Ordinary dry cells work as well, of course.

The thermocouple is made of very thin chromel-alumel wires, and the joints are insulated by a thin layer of acrylic resin. The reference junction is kept in crushed ice in a Dewar flask and the thermoelectric potential is measured with a sensitive galvanometer. The accuracy of temperature measurement was found to be $\pm 0.1^{\circ}\text{C}$ (at a level of 20°C) and there was practically no lag.

The measurements were carried out at "room" temperature, but if desired the slide can be placed on a standard microscope heating or cooling stage and measurements can be made at higher or lower temperatures. A thermostat was not considered necessary for routine measurements as the actual temperature of the sample suspension between the electrodes can be read accurately when

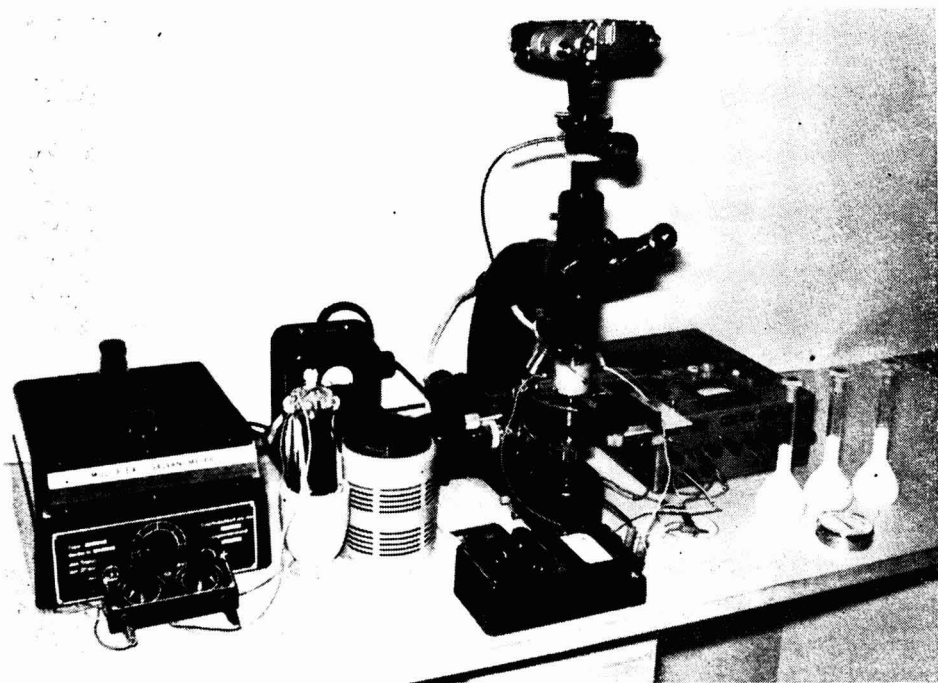


Fig. 3. Arrangement of the electrophoretic equipment, (from left to right) galvanometer, Dewar flask, lamp transformer, Powerstat-transformer, voltmeter, microscope with its accessories, and rectifier unit

the other measurements are being made. The arrangement of the micro-electrophoretic equipment is shown in Fig. 3.

Procedure

The suspension to be examined is suitably diluted (to give a solids content of one g/litre in the case of titanium dioxide pigments) and 3 to 5 drops of the diluted suspension are transferred to the microscope slide. The objective is accurately focused, whereupon, the attached electrodes and thermocouple will be ready for operation. A vigorous Brownian motion is discerned in the microscope field with the particles dark against a light background. When a potential is applied between the electrodes, the electrophoretic behaviour of the particles can be easily observed and the rate of migration can be measured visually with the aid of a scale calibrated in microns in the eyepiece or recorded by a special photographic technique.

Two methods have been developed for photographing the tracks of the migrating particles, a double electronic flash and a continuous exposure technique.

In the double electronic flash technique, the sample is illuminated by a transmitted flash twice in succession after switching on the current, the time between the flashes being recorded by means of a stop watch. As seen in Fig. 4,

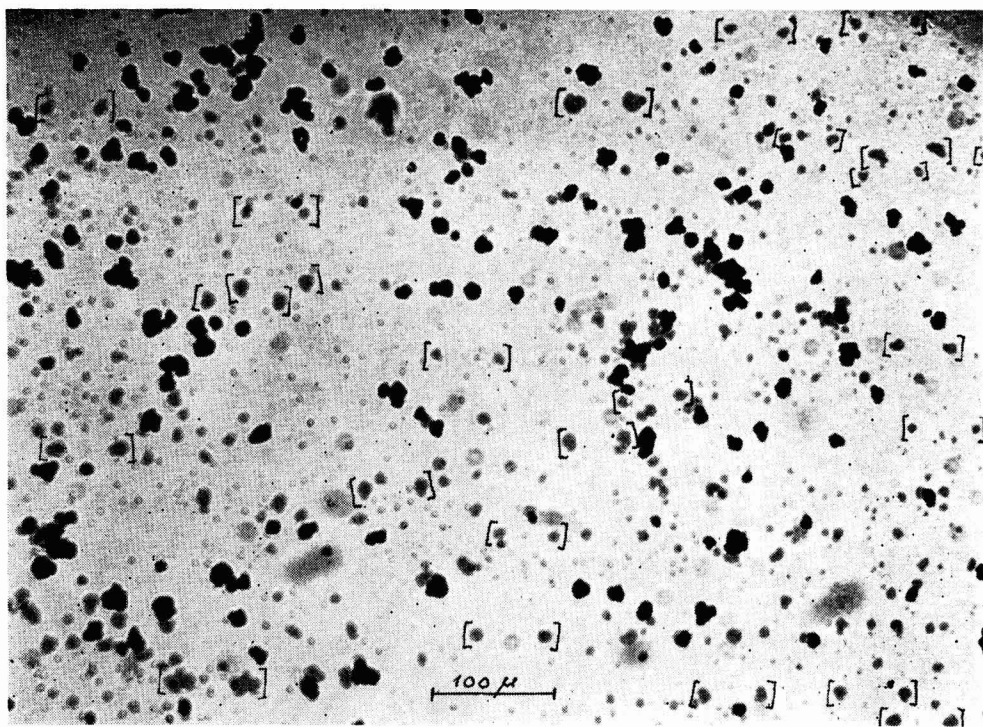


Fig. 4. Microphotograph taken by the double flash exposure technique. Particle migration time (time between two successive flashes) 14 sec., potential 3 volts, electrode distance 2.0 cm. Original magnification $\times 120$. Some typical image pairs have been marked with brackets. Pigment particles appearing on the photographs are in fact aggregates consisting of a number of primary particles

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the particles that have moved during the interval between the exposures give rise to two spots on the photograph, whereas stationary particles give rise to single darker spots owing to the double exposure. The direction of migration, i.e. the sign of the particle charge, must be recorded visually ; it is not possible to distinguish on the photograph between particles moving in opposite directions (which actually is a rather rare occurrence). The electrophoretic velocity of the particles can be calculated from the distance between the particles recorded as image-pairs on the photograph by taking into account the magnification and the time between the exposures.

The continuous exposure technique makes use of the fact that a moving particle gives a readily visible track on the photographic film when the exposure is prolonged. The shutter of the camera is thus kept open for a suitable length of time while the particles are moving in the electric field. In order to obtain tracks with sharp end points and to record the initial and final positions of the particles, the shutter is opened for a few seconds before and after the current is switched on. Fig. 5 shows tracks of moving particles photographed using



Fig. 5. Continuous exposure technique. Initial exposure 2 sec., migration time 5 sec., and final exposure 1 sec. Potential 6.5 volts, electrode distance 2.0 cm. Original magnification $\times 120$. The direction of all moving particles has been from left to right (towards cathode); in addition, some stationary particles can be observed. The Brownian motion causes a diffuse circle around stationary particles

initial and final exposure periods of 2 and 1 second respectively, and a continuous period of exposure of 5 seconds. It is easily seen that the particles in the picture migrated from left to right because the images on the left are darker owing to the longer period of exposure. The images are not as distinct and sharp as those in Fig. 4 which were recorded by the double flash technique ; this is due to the Brownian motion of the particles. Particles migrating to opposite directions can be seen in Fig. 6.

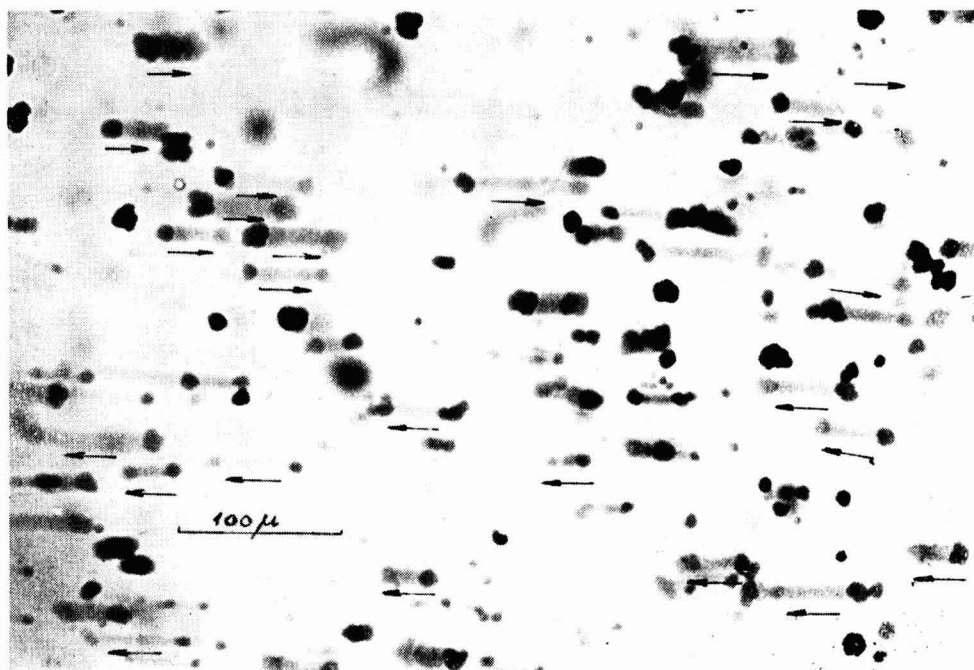


Fig. 6. Same technique as in Fig. 5. In this case particles of opposite charges (the direction of migration marked with arrows) appear, also a particle moving stepwise (upper left corner)

After the electrophoretic velocity has been determined visually or by either of the described techniques, the *zeta* potential can be calculated from (1) as the values of ϵ , η and E are known. The *zeta* potential has the same sign as the charge on the particle.

As mentioned, electro-osmotic phenomena at the walls of the tube in a conventional microelectrophoretic apparatus can be serious sources of error and may lead to uncertainty in the interpretation of the results. Whether electro-osmosis influences the results obtained with the method is a matter for further study. In any case one would expect that these secondary phenomena are of less significance in this method. Optical corrections that are necessary because of the curvature of the tube wall that are needed in some systems of measurement are not, of course, required in this method.

Acknowledgment

The author gratefully acknowledges the practical assistance of Mr. V. Sarkkinen, Research Chemist, Vuorikemia Oy, and expresses his gratitude to the management of Vuorikemia Oy for permission to publish this paper.

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1. Overbeek, J. Th. G. in : Kruyt, H. R. Colloid Science I. Elsevier Publishing Company, Amsterdam, 1963, pp. 194-244.
2. Hückel, E. *Physik Z.* (1924), **25**, 204 ; ref. Overbeek loc. cit.

Reviews

PRINCIPLES OF SURFACE COATING TECHNOLOGY

By D. H. PARKER. Interscience Publishers Inc., New York, 1965. 8 vo. Pp. 817.

This volume is based on the lecture notes used at Wayne County University for their course on surface coating technology. It is aimed at students with limited experience in this field, and provides a broad general picture of the technological side of the United States paint industry.

In 48 chapters the book covers the physical and chemical properties of pigments, solvents, resins and additives ; the application and testing of paints, the principles of formulation and the detailed formulation of the more important types of coatings used in America.

To keep the book within reasonable bounds, it has been necessary to limit the details of the raw materials to essentials only, chemical constitution, properties and important applications ; however, nothing has been lost by this and the information is adequate. The inclusion of a chapter headed newer types of resins is possibly an error, since all the products mentioned are well established, e.g. sulphonamide/formaldehyde condensates, chlorinated biphenyls and polysulphide polymers.

Resin manufacture is dealt with in some detail, and a number of formulations for alkyd and phenolic resins is given.

The chapters dealing with the surface preparation of substrates and the application and drying of paints are well written and comprehensive, as is the chapter on testing, which, not surprisingly, refers strongly to the ASTM methods.

The later chapters in which specific formulations are discussed will probably prove most interesting to the United Kingdom technologist, since they cover the whole gamut of surface coatings from exterior finishes for wood and metal to furniture and marine systems, whilst powder coatings and water thinnable paints have chapters to themselves.

A comprehensive list of further reading matter concludes the book.

It can be fairly stated that this book :

1. Complies with the terms of reference given in the preface and is well balanced.
2. Gives an interesting and detailed view of the other man's technology.
3. Might well have been improved by more references in the text.
4. Is well worth reading.

D. S. NEWTON.

SAFETY AND ACCIDENT PREVENTION IN CHEMICAL OPERATIONS

Edited by E. H. FAWCETT AND W. S. WOOD. John Wiley and Sons 1965. Pp. 617. Price £7 10s.

This is no midget book that can be slipped into the pocket of one's laboratory overall and carried about. It is a weighty volume, well illustrated and referenced, which deals more thoroughly with safety problems than any other work known to the reviewer.

The main object of the book, as stated on the loose cover, is to serve as an authoritative and practical guide to personal safety, industrial health and loss prevention wherever chemicals are handled or used. The problems are approached mainly from the management point of view and the authors appear to be well qualified for the task.

In their foreword the editors draw a distinction between accidents which are regarded as "Acts of God" and those which a little forethought or common sense would have enabled them to be avoided. The former are few and far between and are no concern of this book.

The preface is followed by 31 chapters or sections written by 22 experts, in addition to Fawcett and Wood, dealing with various aspects of the subject of which they have special cognisance. These include, amongst others, as space permits us to mention only a few—4, Chemical Hazards and Worker Safety ; 7 and 8, Hazards of Chemical Reactions and Operations ; 11, Design and Inspection of Pressure Vessels ; 15, A Nurse's Role in Chemical Plant Safety ; 16, Effect of Toxic Agents ; 21, Flammable Materials ; 22, Radiation ; 23, Respiratory Hazards and Protection ; 24, Eye Safety in Chemical Processing ; 28, Fire Extinguishing Agents.

A very wide field is thus seen to be covered. Section 22 contains much information on radiation not readily accessible, and illustrates the Curie plea that

Nothing in life is to be feared ;
It is only to be understood.

Section 23 is particularly well illustrated with excellent photographs of various types of gas masks and respirators.

All firms that manufacture chemicals or have occasion to use them in bulk would be well advised to have a copy of this book readily accessible on their shelves.

J. NEWTON FRIEND.

Information Received

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

Triganox X37 is a liquid organic peroxide developed by NOVADEL LTD. for the curing of unsaturated polyester resins at temperatures between 80°C and 130°C.

BADISCHE ANALIN- & SODA- FABRIK AG are now marketing, through ALLIED COLLOIDS LTD., the following products: Sudan Blue liquid, the first of range of liquid dyes for colouring oil products; Euviprint pigment dispersions for the production of gravure inks and lacquers for package printing; Victoria Pure Blue FGA for flexographic and moisture set inks.

A new £150,000 plant is being built by VINYL PRODUCTS LTD. at Carshalton to meet the increased demand for Vinamal and Vinacryl synthetic resin emulsions.

L. A. Mitchell Ltd. is transferring the activities of its fluid mixer division to Mitchell Craig Pumps Ltd. from 1 February 1966.

Those companies who wish to evaluate their products under conditions of high solar radiation coupled with long hours of sunshine and high relative humidity may wish to take advantage of the service offered by the Paint Exposure Laboratory, Cascais, Portugal. Observations are under the control of an experienced paint technologist and results are submitted monthly.

Publications Received

Publications received at the Association's offices include *Tin and Its Uses*, the quarterly publication of the Tin Research Institute; *Chemical Comments*, a Union Carbide publication; a new brochure on triple roll mills from the Pascall Engineering Co. Ltd.; *Compass*, the quarterly magazine of Scott Bader & Co. Ltd., a booklet entitled "Paint and Varnish Manufacturers: Easy to Handle, Certain to Operate Fire Extinguishers" from Nu-Swift International Ltd. and Snowcal Whiting Manual Natural calcium carbonate fillers and their industrial application, published by the Whiting Division: The Cement Marketing Co. Ltd.

Section Proceedings

Bristol

Coloured pigmentation for electrodeposition

At the Ordinary Meeting of the Bristol Section held at the Royal Station Hotel, Bristol, on 7 January 1966, a paper was presented by two members of the Section—Mr. L. Tasker and Mr. J. R. Taylor—entitled “Coloured Pigmentation for Electrodeposition.”

Earlier work on the effect of various pigments, particle size and degree of dispersion on film resistance was reviewed. Additional results obtained by variation of conditions in an electrodeposition tank, i.e. temperature, pH value and solids content, were presented.

Photographs comparing the dispersion of titanium dioxide and phthalocyanine blue in films deposited by electrodeposition and by spraying were shown.

Further microscopic examination of such films was illustrated by slides showing shadowed film surface replicates and Talysurf traces.

A lively discussion followed the paper, which was curtailed only by time.

The 35 members and visitors present responded to the vote of thanks by Mr. R. Coomber.

L. T.

Irish

Interesting aspects in emulsion paints

“Interesting aspects in emulsion paints” by Dr. P. M. Troll, was the title of a lecture given to the Irish Branch of the Bristol Section (now the Irish Section) on 29 October 1965.

The meeting was well attended, 31 members and 16 visitors being present for a most interesting lecture well supported by demonstration panels of newly developed pva compositions.

After being welcomed by the chairman, Dr. Troll gave his lecture on the uses of pva in the paint industry. Many panels, including weathered panels were exhibited to the audience showing various effects and finishes that could be achieved with pva.

General purpose emulsion paints were first discussed and it was pointed out that with their good brushability and fair wash resistance they were almost too good for interior purposes, but not quite good enough for exterior use as well. The lecturer then demonstrated that by varying the characteristics of the paint by different application techniques, or by the introduction of different pigments, interesting decorative effects could be obtained.

In the protective rather than the decorative field the use of fibre filled paints was described particularly in relation to application on cracking substrate. Fire retardant paints having foam forming properties were demonstrated.

So far as gloss paints are concerned Dr. Troll stated that these were still typical emulsion paints and could not be compared with alkyds in every respect.

In exterior paints pva emulsions prevented water penetration to the substrate whilst retaining water permeability and hence they reduced the tendency to blister. Formulation of these paints depended not only on selection of a suitable emulsion but also on the choice of fillers, pigments and other ingredients and weathering tests were particularly important. Dr. Troll then described the type of coatings needed for wood and porous surfaces, including the use of sand filled paints.

The subject of lime blooming on alkaline substrates was then discussed and panels were demonstrated showing that excellent resistance could be obtained, even on very fresh asbestos cement, using special emulsions.

In summing up, Dr. Troll emphasised that exterior emulsion paints for wood must be flexible to allow for natural movement of the timber. Suitable homopolymer, copolymer and acrylic emulsions can be used. On masonry harder emulsions were used. Much emphasis was placed on the correct priming of the surface.

In the ensuing discussion a considerable number of questions were asked and the Chairman was reluctantly compelled to close the meeting although it was obvious that members had many other questions for the speaker. A vote of thanks was proposed by Mr. J. Kirwan and seconded by Mr. D. Godham.

Printability of paper and board

The November meeting of the Irish Branch/Bristol Section (now the Irish Section) was held at the Dolphin Hotel, Dublin, on 26 November. Miss E. J. Pritchard gave a lecture entitled "Printability of paper and board" to an audience of 36 members and visitors.

Miss Pritchard described in her lecture the various properties required for printing paper, and how absorbency, smoothness and reflectance are all inter-related with one another. The necessity of a smooth surface for quality printing by the gravure process and the methods for assessing this were fully described.

The many test procedures that were available did not fully cover the problems involved and did not correspond directly with gravure printability. It was therefore necessary, the lecturer added, to devise a means which would not only be quick but also accurate thereby allowing one to determine the printability of paper for gravure without printing on a press. To do this she had used a slightly modified Chapman tester so that now a measurement not only of the number but also the size of pit that existed in any given paper could be made.

The method devised by Miss Pritchard still had the great disadvantage of being somewhat slow but further work was being done, she said, to overcome this. Instead of counting and measuring the pits on the prints from the modified Chapman it was intended to do the test by optical scanning. The lecture aroused great interest and in particular our members associated with the printing industry asked a large number of questions. A vote of thanks was proposed by Mr. D. Campion and seconded by Mrs. H. Stokes.

M. O'C.

London

Surface properties of titanium dioxide pigments

The fifth technical meeting of the session was held on 10 January 1966 at 14 Belgrave Square, S.W.1. This was a joint meeting with the Colloid and Surface Chemistry Group of the Society of Chemical Industry. Mr. C. R. Pye took the chair, and Mrs. S. M. Rybicka and Mr. A. F. Sherwood, of the Paint Research Station, gave a paper on "Surface Properties of Titanium Dioxide Pigments."

Mr. Sherwood said that a general investigation into pigment/medium relationships was in progress at the Paint Research Station, and as part of this programme a study was being made of the interactions between titanium dioxide pigments and simplified systems representing the functional groupings of an alkyd resin. These interactions were followed by a combination of adsorption techniques and infrared spectroscopic examination of the pigment/adsorbate complex.

The effects of water in the adsorption processes were examined at three levels of moisture conditioning of the pigments. It was shown that an increase in the quantity of stearic acid adsorbed by a complex surface-treated rutile pigment resulted from an increased water content of the pigment, whereas the reverse occurred with the anatase materials.

The adsorption data indicated that stearic acid was chemisorbed by the complex pigments, but only held physically by the simple types. This was confirmed by the spectroscopic evidence that stearic acid was present on a complex rutile pigment as the carboxylate ion, whereas with the simple types of pigment the acid was predominantly hydrogen bonded to the substrate.

Appreciably less oleic acid was adsorbed by all of the pigments than stearic acid. Water did not enhance the adsorption of this acid by a complex rutile pigment. Evidence for the ionic adsorption mechanism on a complex rutile pigment and a hydrogen bonding mechanism on the simple types was again found. Frequency shifts were observed for the $-\text{CH}_2-$ and $-\text{CH}=\text{CH}-$ regions, and in several instances disappearance of the $\text{CH}=\text{CH}$ spectral band indicating that chain/chain or chain/substrate interaction existed. No similar effects were observed for stearic acid.

The adsorption isotherms for stearyl alcohol were "S" shaped and were shifted towards a higher concentration by the presence of water on the pigments. The spectra showed the alcohol to be hydrogen bonded on to all of the types of pigment and also revealed a chain interaction of a different type from that found for oleic acid.

The predominant group of an alkyd resin, ester, was not observed to interact with any of the pigments studied. Mr. Sherwood concluded by reviewing some of the implications of this fundamental study in the complex relations occurring between the pigment and the medium in practical dispersion processes.

In the extensive discussion which followed the paper, questions were asked concerning the surface modification of titanium dioxide pigments, and the effects of the Al/Si ratio, the degree of completeness of the coating process, the degree of retention of the adsorbates, and the different types of adsorption isotherms obtained with different adsorbates. Those taking part in the discussion included: Mr. G. C. Hurst, Dr. J. A. Kitchener, Sir Eric Rideal, Mr. C. I. Snow, Dr. S. H. Bell, Mr. K. W. Green, Mr. K. Goldsbrough and Mr. M. C. Grint.

In proposing a vote of thanks, Mr. R. C. Tarring, Hon. Treasurer of the Colloid and Surface Chemistry Group, expressed the appreciation of the Group at the opportunity for collaboration with the London Section, and thanked Mr. Sherwood and Mrs. Rybicka for a lecture which had been both absorbing and adsorbing, and of considerable interest to all those present.

V. T. C.

Southern Branch

Decorating plastics

The fourth meeting of the session was held jointly with the Southern Section of the Plastics Institute on 9 December 1965 at Southampton University, when Mr. S. E. Francis gave a lecture on "Decorating Plastics."

Mr. Francis said plastic products were painted to gain advantages not provided by the moulding process; these advantages could be either functional or decorative.

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He described some of the requirements of paints for use on plastics such as the importance of right solvent to obtain good adhesion and the need for close colour control especially when different types of plastic are used in a single assembly.

The properties of paints for plastics vary with the end use and some typical properties for the car and appliance industries were described. Various methods of application, including spray painting, dip coating and roller coating as well as screen process printing, were also covered.

Metallising of plastics was described in some detail and examples were shown of car and other components finished in this way.

Finally, Mr. Francis discussed some of the more commonly available plastics, the uses to which they were put and some of the problems involved in coating them. In this survey he covered ABS plastics (macromolecules formed from acrylonitrile, butadiene and styrene monomers) and used for car dash panels, covers for machines, etc.; acrylics widely used for signs, street lighting, architectural application and car parts; cellulose for packaging and toys; polystyrene for packaging components, toys, signs, radio cabinets and so on. The list of plastics seemed to be ever increasing and he referred to some of the newer materials, such as polycarbonates, which were of considerable interest.

After a discussion and demonstrations of some of the exhibits, Mr. Carey thanked the speaker for an interesting and instructive evening, and said he hoped it would be possible to arrange more joint meetings with the Plastics Institute.

Corrosion mechanisms and prevention

The fifth meeting of the session of the Southern Branch was held on 10 January 1966 at the Queens' Hotel, Southsea, when Dr. G. Butler gave a paper on "Corrosion Mechanisms and Prevention."

Dr. Butler said that although about 80 per cent of corrosion problems were atmospheric, his paper concerned mainly immersed corrosion. Under immersed conditions, corrosion was due to electrochemical action. The forms this action could take were various and included the corrosive effect caused by sulphate reducing bacteria. Corrosion could occur as the result of the current which flowed between dissimilar metals in an electrolyte such as sea water, or between different parts of the same metals. Other factors such as concentration of the electrolyte, surface oxide layers, metal inclusions all played a part in the type and extent of corrosion.

Dr. Butler then dealt with polarisation and showed how corrosion could be controlled by anodic control, cathodic control or mixed control. The relative areas of anode and cathode were important in controlling corrosion especially in high conductivity water.

Dealing with the effect of the composition of the electrolyte, Dr. Butler said the noble metals were resistant to acid and alkali conditions, while aluminium and zinc were attacked in both. Nickel, copper and cobalt were attacked in acid conditions only, but iron was complex in its behaviour.

Dissolved gases such as oxygen and carbon dioxide often played an important part in immersed corrosion, while in atmospheric corrosion particle contaminants containing carbon, sulphur dioxide and moisture all increased corrosion.

Methods of preventing corrosion included alloying with other metals, such as copper. Modification of environment could prevent atmospheric corrosion by controlling the humidity in packaging by the use of vapour phase inhibitors. Similarly water could be treated with such substances as lime or chromates to reduce corrosion. For immersed conditions the cathodic protection technique using either sacrificial anodes of magnesium or zinc or controlled impressed current was widely used for

structures and ships. Possibly the commonest form of corrosion protection was by means of metal or organic coatings. Dr. Butler said only zinc and cadmium metals were effective on iron. Paints and plastics protected by stopping either the anodic reaction, the cathodic reaction or by providing a resistant barrier. Most paints acted both as barrier and corrosion inhibitors. He concluded by stressing the importance of design in structure and equipment to avoid conditions which encouraged corrosion.

The paper was illustrated by many excellent slides and, following the usual break for coffee and biscuits, Dr. Butler answered questions from Mr. Kingcome, Mr. Targett, Mr. Chapman, Mr. Burke and Mr. Bargrove.

In his own inimitable manner, Mr. Targett thanked the lecturer for his excellent discourse on corrosion.

J. C. K.

Thames Valley Branch

Aerosols

The fifth technical meeting of the 1965-66 session was held at the Royal White Hart Hotel, Beaconsfield, on Thursday 9 December 1965, when Mr. W. H. Brown gave a talk entitled "Aerosols".

Mr. Brown said that he had decided to talk about the subject generally. An aerosol is a suspension of a liquid or a solid in a gas, but in common usage, the term has come to be applied to a container from which material is dispensed by a propellant gas.

The first patent, describing a bottle with a valve which dispersed an aerated liquid was taken out in 1862. In 1930, patents were taken out describing the use of fluorocarbons which had been developed for the refrigeration industry. In the 1940's, the spread of diseases by insects among American troops in the jungle led the US Dept. of Agriculture to produce a portable aerosol dispenser. Thousands of these were produced, and at the end of the war it was realised that they were an effective means of dispensing, but the heavy steel container was inconvenient. In 1945-46, modern aerosols appeared on the US market.

Modern aerosols can be divided into two classes, those operating by compressed gas, e.g. carbon dioxide, nitrous oxide, and those operating by liquefied gas. The first type gives a very good spray initially, but its performance drops off in use. By varying the propellant, pressures of 12-70 p.s.i. can be obtained.

Liquefied gas systems may again be subdivided into two-phase systems, when the product and liquid propellant are capable of forming a homogeneous liquid, and three-phase systems, when the product and propellant form separate phases. These systems give a constant pressure during their life, the propellant reverting to the gas phase. Solvent-borne products are generally packaged as two-phase systems, with fluorocarbon as propellants, whereas water-borne products are three phase, with hydrocarbons as propellants.

The containers can be filled by cooling the product and propellant to -5°F ., filling the can, then crimping on the valve, or by filling at room temperature, purging the can of air by a shot of propellant vapour, crimping the valve, then filling the propellant under pressure. Two-phase systems are generally cold-filled, three-phase pressure-filled.

Containers may be of tin-plate, aluminium, glass (which can be plastic coated for safety) and plastic (polythene, polypropylene, nylon, Delrin). Plastic containers may be permeable and need very extensive testing.

Before a product is packed, extensive laboratory testing is necessary for compatibility, pressure developed, can corrosion and stability, and no weight loss should occur on storage. Every aerosol leaving the factory is tested for leakage through a hot water bath system.

Aerosols are easy to use, efficient, not always economical, quite often the only practical way in which a product can be dispensed. They are believed to have a very promising future.

A discussion followed in which Messrs. Pilkington, Riley, Pearson, Simpson, Horne, Inshaw and Drs. Henderson and Long took part. A vote of thanks was proposed by Mr. Jacob.

W. S.

Midlands

Thermosetting acrylic resins

At a meeting of the Section on 21 January, at Birmingham Chamber of Commerce, Messrs. J. R. Taylor and T. I. Price gave papers on "Thermosetting Acrylic Resins."

Mr. Price began by saying that his paper would concern general aspects of the chemistry of thermosetting acrylic resins and that Mr. Taylor would concentrate on their performance in paint. The co-monomers from which thermosetting acrylic resins were produced could be divided into hardeners, plasticisers and monomers having functional groups to permit cross linking. The incorporation of styrene or vinyl-toluene gave hardness and chemical resistance but reduced outdoor durability and affected compatibility with amino resins. The optimum level could be demonstrated with the aid of triangular graphs and for the system vinyl-toluene, methylol acrylamide, ethyl acrylate the optimum level was about 20 per cent vinyl toluene. With systems containing hydroxyalkyl methacrylates in place of acrylamide he had not found such well defined peaks but this might be due to use of a different melamine resin. He had also observed differences between accelerated weathering and exposure in Florida and between paints incorporating metallics and titanium dioxide.

He had been unable to substantiate a claim that hydroxypropyl acrylate, with its secondary hydroxyl groups gave better stability and gloss than hydroxyethyl acrylate but he thought that a claim, that hydroxybutyl acrylate gave improved can stability, might be justified.

Instead of using hydroxyalkyl acrylates it was possible to incorporate acids and react the copolymer with alkylene oxides. This route appeared cheaper but undesirable side products could not be removed. Resins having a carbon to carbon backbone and pendant hydroxyl groups could also be obtained by reaction of acids with a glycidyl fatty acid or by reaction of alkylene oxides with copolymers from styrene and maleic anhydride.

For retouch paints which were to be stoved below 120°C curing of hydroxyalkyl acrylate resins with melamine could be catalysed with acids, for example acid butyl maleate. The use of amino resins, specially developed for low temperature curing, was also advantageous.

Mr. Taylor then took the floor and said that he had examined the effect on acrylic paints when some of the resin was replaced by alkyds of the Cardura type and by epoxide resins. With a resin incorporating methylol acrylamide the optimum replacement by alkyd was about 30 per cent. This gave improved short-term gloss and colour retention on overstoving. Provided that the system was fully cured fat and stain resistance was good. There was some reduction of alkali and detergent resistance but only to a level which was acceptable for most purposes. On weathering, the presence of Cardura resin improved gloss retention whilst the presence of epoxide resin promoted chalking.

In systems suitable for car production and based on a coconut/TMP alkyd, an acrylic and an amino resin, the presence of alkyd permitted lower stoving temperatures.

Weathering compared favourably with systems used at present but the amino-resin had to be selected with care. Butylated melamine resins gave best gloss retention whilst benzoguanamine resins caused chalking.

Surfactants had been examined to assess their effect on uniformity and gloss in pigmented systems. The optimum hydrophilic-lyophilic balance was found to be ten and different chemical types having HLB of ten were then examined. Different types of titanium dioxide were also evaluated, type CR3 giving very good results.

During the ensuing discussion period questions were put and answered concerning HLB calculations, reducing stoving schedules, an apparent anomaly in weathering curves, the behaviour of benzoguanamine resins, the design of the coconut TMP alkyd used, polishability and solid contents of acrylic resins.

Mr. P. Daley proposed a vote of thanks to the speakers which produced a cordial response from those present.

L. R. S.

Trent Valley Branch

Impact of plastics on the paint industry

A technical meeting of the Trent Valley Branch of the Midlands Section was held on 13 January at British Railways School of Transport, Derby. Twenty-five members listened to a talk by Dr. Keenan entitled "The Impact of Plastics on the Paint Industry."

Dr. Keenan spoke of several developments in the Plastics Industry gradually replacing items used in the building industry, which had previously required paint to protect them. In particular, he mentioned rainwater pipes and gutters, window fittings, exterior cladding and fascias and also—more recently—attempts to market completely prefabricated plastic buildings.

Much of the talk was about developments in jointless plastic flooring, especially the lecturer's personal experience with polyester compositions. He said that a normal system comprised a coat of primer, a second coat, which was normally pigmented and filled with sand, and a third or skim coat, pigmented but without sand. Various effects could be obtained by simple modifications of the final coat, e.g. a speckled effect by inclusion of pvc chips. Mention was also made of use of moisture cured polyurethanes for floor lacquers.

Finally, Dr. Keenan mentioned the use of glass reinforced plastics in the vehicle building industry, but felt that paint would not be replaced in this field, owing to the difficulty of precise colour matching with plastic mouldings.

The talk was well-illustrated with numerous slides, and provided all present with a very pleasant evening.

J. R. B.

Scottish

Micronised pigments

The January meeting of the Scottish Section was held in More's Hotel, Glasgow, on Thursday 13 January when Mr. W. G. Wade delivered a lecture on "Micronised Pigments" to a large number of members.

After describing the principle and operation of the Microniser, Mr. Wade pointed out that, although the equipment gave a product of remarkable consistency of fineness, control could only be exercised over the top size of the pigment, varying from two to

ten micron, and no control was possible with regard to particle size distribution. The top size of particles varied according to the pigment. The advantages of micronised pigments were the enormously increased rate of paint production possible, especially in the new types of paint mill as well as in some of the older types ; complete absence of mill wear, this being especially true of the single roll type ; elimination of waste of pigments and paint ; greater opacity, which is normal for almost all micronised pigments by a margin frequently exceeding 10 per cent ; and improved gloss.

One of the widest fields of application of micronised pigments was in processing with high speed impellor equipment, when high output of quality decorative paints was possible. On the other hand, it did not seem likely that a first grade industrial finish, such as a car enamel, could be obtained in this way without some after-treatment. The combination of micronised pigments with the single roll mill offered great possibilities, particularly to the small manufacturer, who could often produce tremendous quantities of high grade paint with only a light premix. Using standard practice in heavy ball mills, micronised pigments had not been successful, often leading to overgrinding. On the other hand, it had been found possible to produce excellent results, using $1\frac{1}{2}$ cm. balls, with a resin solids of 20 per cent, grinds of 7+ on a 7-8 Hegman gauge being possible. Thus, it was feasible to reduce the normal 10-16 hour overnight grind to something in the region of 1 to $2\frac{1}{2}$ hours.

In some respects, the physical characteristics of micronised pigments were apparently anomalous. For example, contrary to expectation, the surface area of many micronised pigments was less than that of the untreated ones and it was assumed that this was due either to the loss of superfines or to the rounder, smoother contours of the micronised pigments, or to both. Again, although one would expect the reverse, the oil absorption of micronised pigments was very close to that of ordinary ones.

Micronised pigments exhibited higher tinting strength, not so much because of any intrinsic increase in this property, but simply because their ready dispersibility rendered their full strength immediately available. Opacity, gloss and resistance to settlement were also improved.

Micronised pigments, and particularly red oxides, found ready application in the electrodeposition process. Apart from advantages in manufacture, the fineness of the pigment reduced settlement in the tank and also gave a higher resistance in the deposited film.

A discussion ensued at the end of the lecture, in which several members participated. A vote of thanks was proposed by Mr. J. F. McVey.

A. MCL.

Student Group

December meeting

The second film show of the session took place in December. Three films were shown. The first, "To Dream of Yachts," illustrated the pleasures of yachting. The second, "Black Monday," was in sharp contrast, pointing out the hazards of working in a laboratory and showing how easily accidents could happen. The last film, "Point of New Departure," showed the development of nylon from very modest beginnings to what is now one of the most important industries in the world.

In proposing a vote of thanks, Mr. Birrell warmly thanked Mr. Smith, the projectionist, for giving up some of his holiday in order that the films could be shown.

R. F. H.

West Riding

Metal powders in emulsion paints

On 11 January the Section was addressed by Dr. Troll on the subject of the use of metal powders in emulsion paints. Dr. Troll stated that whereas solvent paints containing metal powders were well known, it had not previously been possible to make emulsion equivalents because of gassing and poor storage stability. It had now been found that certain types of polyvinyl acetate homo- and copolymers could be formulated into stable systems by (1) choice of an electrolytically stable emulsion (2) suitable buffering of the system to prevent formation of acetic acid which would otherwise attack the metal and (3) choice of a suitable grade of metal powder.

Dr. Troll then proceeded by describing various types of paint which were shown on demonstration panels. Incorporation of a leafing grade of aluminium gave paints not only with good insulation properties but which were ideal for sealing bituminous surfaces. Decorative effects were produced from paints having inherently bad flow and two-colour effects were possible using a brush, roller or similar implement. The use of aluminium flitter in place of powder gave a finish termed "liquid wallpaper" which was particularly suitable for a quick cheap job. A series of hammer finish effects was shown using aluminium and gold/bronze powders in conjunction with additions of a suitable silicone oil and organic pigments. The method of variation of cell size was discussed and also the production of multi-coloured effects by the so-called "wet-in-wet" technique. This effect could also be used to decorate glass panels where the coating is applied to the underside of the glass and viewed through the glass. It was emphasised that it was necessary to have a substrate of uniform absorbency when applying finishes and hence it might be desirable in many cases to use a primer or base coat to achieve a successful result.

Solvent solutions of polyvinyl acetate were particularly suitable especially on very porous or crumbly surfaces. Lastly, it was stressed that there was an inherent danger in applying metal-containing paints to alkaline substrates particularly in the presence of humidity since chemical reaction might result. Thus if it was not possible to wait until the alkali on the surface had reacted with the atmospheric carbon dioxide suitable chemical neutralisation should be adopted; for example, by treatment with magnesium silico fluoride.

In summing up, Dr. Troll stressed the importance of surface preparation and mentioned that the outdoor resistance of these types of paints is less satisfactory than might have been hoped.

J. N. MCK.

OCCA 18

108 Stands and 25,000 sq. ft. of stand area at the largest Association Technical Exhibition

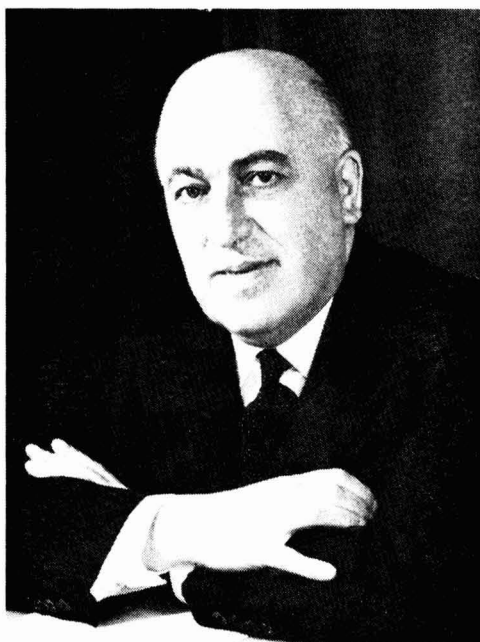
The largest OCCA Exhibition held will take place this year at Alexandra Palace, London, N.22. The Exhibition, which provides a focus for the display of technical advances in raw materials, equipment and technology by the suppliers to the industries covered by the Oil and Colour Chemists' Association will be open on the following days and at the following times :

Monday		
14 March	..	3 p.m. to 6.30 p.m.
Tuesday		
15 March	..	10 a.m. to 6 p.m.
Wednesday		
16 March	..	10 a.m. to 6 p.m.
Thursday		
17 March	..	10 a.m. to 6 p.m.
Friday		
18 March	..	10 a.m. to 4 p.m.

The Exhibition will again be open for five days and the 108 stands will cover approximately 25,000 sq ft, with the added advantage that the Exhibition will be housed under one roof. A layout of the Exhibition can be found on page 245 but it should be noted that after this plan and the *Official Guide* were prepared Gebruder Netzsch has been allocated space (stand 107); details of this stand will be found under "Latest Information." A numerical list of Exhibitors is also given.

A plan showing how to reach Alexandra Palace is displayed on page 244; ample free car parking is available for those travelling by road and for those using the Piccadilly

Line of the London Transport Underground system a free bus service will be operated from Wood Green station to the Exhibition and return.



The Rt Hon. the Earl of Kilmuir,
P.C., G.C.V.O.

The Exhibition Luncheon will be held at the Savoy Hotel, at 12.45 p.m., on Monday 14 March when the Rt. Hon. the Earl of Kilmuir, P.C., G.C.V.O., President of the British Standards Institution and Chairman of The Plessey Co. Ltd., will reply to the Address of Welcome by the President, Dr. S. H. Bell. The opening ceremony will take place at 3 p.m. at the Main entrance to

the Great Hall immediately prior to the admission of visitors.

In addition to the *Official Guide* sent to all members of the Association and also to individual chemists and technologists on the Continent many hundreds of invitation cards printed in four languages have been despatched to paint and printing ink firms. Non-members may obtain copies of the *Official Guide*, free of charge, from the Association's offices.

The Association's Information Bureau will be situated on stand 55 (Tel. TUDor 0932) ; this telephone number is available only during the period of the Exhibition, as are all the numbers shown in the *Official Guide*.

Invitations have again been extended to schools to send parties of sixth-form students to visit the Exhibition and so schools in the Greater London Area have arranged to send parties on the mornings of 15, 16, 17 March, when they will be given a short introductory talk by a member of the Association before touring the Exhibition Hall. The Technical Education Stand will be arranged on the theme *The Historical and Technological Development of the Paint Industry*. The Stand will be manned by representatives of Technical Colleges, together with representatives from industry.

The fact that the Exhibition has outgrown four venues is indicative of the importance of this event to the surface coatings industry. The number of overseas visitors is growing each year and in order to assist both these visitors—in 1965 from 27 countries—and exhibitors, interpreters will be present on either Stand 69 or at the OCCA Information Bureau. In case of accidents British Red Cross Society personnel will be in attendance on Stand 27.

There are two restaurants with full dining facilities—the Edinburgh Room, where tables can be reserved, gives a table d'hôte menu with a wide choice of dishes and the Alexandra Room, in which tables cannot be reserved. In addition there are two buffets and several

bars which will be open throughout the period of the Exhibition.

As a service to exhibitors and visitors the London Telecommunications Region of the GPO will be installing a complete telecommunications centre with inland and overseas telegraph, telephone and telex services. The telex number of the Stand is 21567 and the telephone number TUDor 7764. A GPO postal unit will also be available and postcards and letters will be collected several times each day.

A full, illustrated report of the Exhibition will appear in the **May** issue of the *Journal* and will contain extracts from the speeches given at the Luncheon and a review of the outstanding exhibits. Any inquiries concerning the Exhibition should be addressed to the General Secretary, Mr. R. H. Hamblin, at the Association's offices, Wax Chandlers' Hall, Gresham Street, London, E.C.2.

Latest Information

Since the *Official Guide* was printed the following further information has been supplied.

Stand 107—Gebruder Netzsch.

The Netzsch-Molinex Mill conforms in principle to continuous working mills of the attrition type in which the grinding media is agitated by a vertical stirrer. But by a very special and patented stirrer construction developed and proven by intensive research during a period of four years before being offered to industry, phenomenal results on particle size reduction are obtainable. The grinding media is not restricted to a particular size or material. Special glass beads, steatite balls, aluminium oxide balls and also hardened steel balls are used, being selected to suit the raw material being ground and dispersed and the end product required.

The working principle is as follows : a pre-mixed slurry is fed, by a variable delivery pump to the lower end of the vertical working chamber which has been charged with the selected grinding media. Whilst rising through the chamber



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BUFFING COMPOSITIONS

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at a rate determined by the setting of the feed pump, the material is subjected to intensive attrition and particle size reduction due to the violent but controlled cyclic movement and pulsation of the grinding media caused by the unique design of the stirrer.

The particle size reduction obtainable is a function of the duration of passage of the material through the container from bottom to top, this being controlled by the delivery rate of the feed pump. Particle size reduction to less than one micron is practical with this Mill.

After passing completely through the Mill the material emerges through a sieve of special design, which retains the grinding media but allows free passage of the finished product.

Whilst the change-over from one product to another is normally rapid, the containers are removable and interchangeable together with the grinding media and stirrer. Being provided with means for thermal control, highly volatile substances as well as those of a waxy nature, can both be economically and efficiently processed.

Naturally, all parts of the machine in contact with the product are of special abrasive and corrosion resisting material.

Outside the paint and lacquer industry the machine has a wide field of application wherever rapid and uniform particle size reduction is required.

Produced in sizes with hourly outputs of seven to 300 gallons all normal requirements of industry have been catered for.

Stand 97—Dunlop Chemical Products Division.

Water-based printing inks will be shown. These have the advantage of being water reducible to any level without harmful effect and have been found to be suitable for printing paper bags and sacks.

Stand 42—Kunstharsfabriek Synthese N.V.

The new *Setal* resins mentioned in the *Official Guide* have been designated

Setal 160XX-60, *Setal 161 XX-60* and *Setal 162 XX-60*.

Stand 41—Johns-Manville Co. Ltd.

For those finishes requiring "enamel-like" film smoothness, the company has just introduced *Celite 499*. It is said to give fast easy dispersion without sacrificing gloss or sheen control and to improve stain removal and burnish resistance.

Stand 47—Victor Blagden and Co. Ltd.

Details of the use of moisture cure polyurethanes in seamless flooring will be shown. The method is a relatively new concept of applying monolithic flooring and employs moisture cure polyurethanes with decorative chips or granules giving a terrazzo-like appearance.

Stand 36—William Boulton Ltd.

A machine designed for the high speed production of all types of dispersions and solutions will be exhibited for the first time. A 50 gallon model of this machine, the *Dynaflow*, is specifically intended for emulsion paints and incorporates a Boulton fibreglass fabricated tank.

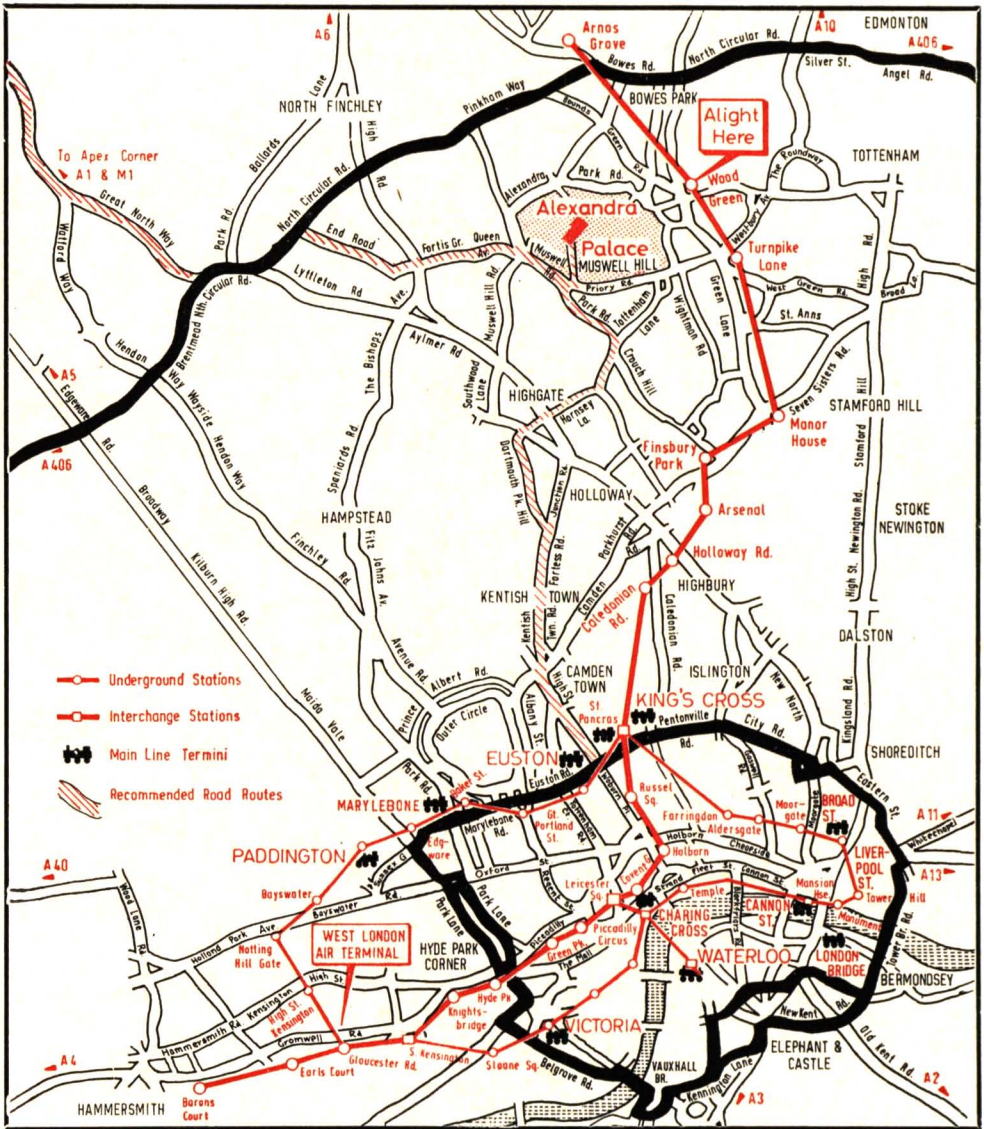
Stand 73—Imperial Chemical Industries Ltd.

A new organic yellow—Monolite Fast Yellow 6 GS—will be shown and its fastness properties demonstrated. The Dyestuffs Division will show Bedacryl 12 W, a vinylated alkyd resin mainly used as an industrial finishing on large vehicles; nine panels will demonstrate durability and drying properties. Another product to be featured will be Proxel AB paste, a low-toxicity preservative for emulsion paints and other aqueous compositions. The Division will also be showing a technical exhibit illustrating the use of toluene di-isocyanate in the manufacture of urethanes.

Stand—38 Allied Colloids Ltd.

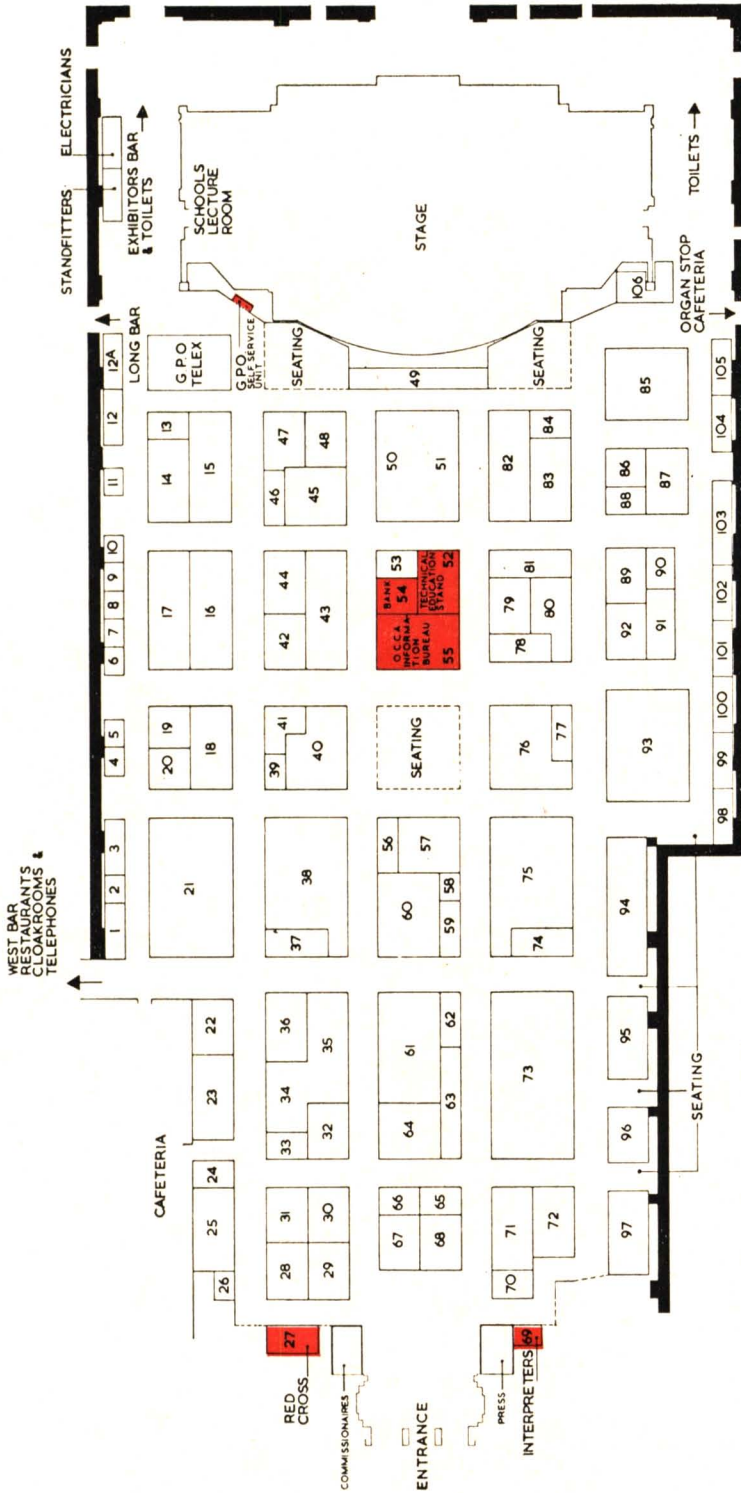
A further development is a very versatile emulsion for surface coatings. Applications will be demonstrated for the crosslinking of Acronal emulsions and solutions in pressure-sensitive adhesives, sealing compounds and flooring adhesives.

HOW TO REACH ALEXANDRA PALACE



1. The free bus shuttle service will operate between Alexandra Palace and Wood Green Station on the Piccadilly Line (Underground), which is denoted by the thick red line.
2. Those travelling by road will find ample free car parking facilities at Alexandra Palace.
3. Visitors arriving at West London Air Terminal may board the Piccadilly Line trains at Gloucester Road Station.
4. The map also shows the position of the main line stations in relation to the Piccadilly Line.

Plan of Exhibition



Numerical List of Exhibitors

Stand

1. Rhone-Poulenc, Societe des Usines Chimiques
2. Torsion Balance Co. (Great Britain) Ltd.
3. Lancashire Tar Distillers Ltd.
4. Research Equipment (London) Ltd.
5. Churchill Instrument Co. Ltd.
6. Beckman Instruments Ltd.
7. Society of Dyers & Colourists
8. Hilger & Watts Ltd.
9. Reeves & Sons Ltd.
10. Coulter Electronics Ltd.
11. Joyce, Loebel, & Co. Ltd.
12. Sangamo Controls Ltd.
- 12A. Little, J. H., & Co. Ltd.
13. Grampian Press Ltd. (*Paint Manufacture*)
14. Plastanol Ltd.
15. Laporte Industries Ltd.
16. CIBA Clayton Ltd.
17. CIBA (A.R.L.) Ltd.
18. Crosfield, Joseph, & Sons Ltd.
19. British Celanese Ltd.
20. Bakelite Ltd.
21. Farbenfabriken Bayer A.G.
22. Svenska Oljeslageri Aktiebolaget
23. Pfizer Ltd.
24. Amoco International S.A. Geneva
25. Maschinenfabrik Heidenau Veb.
26. Marchant Bros. Ltd.
27. British Red Cross Society
28. Styrene Co-Polymers Ltd.
29. Draiswerke GmbH
30. Novadel Ltd.
31. Premier Colloid Mills Ltd.
32. Steele & Cowlshaw Ltd.
33. Baird & Tatlock Group of Companies
34. Kingsley & Keith (Chemicals) Ltd.
35. Allied Chemical Corporation
36. Boulton, Wm., Ltd.
37. Ferranti Ltd.
38. Allied Colloids Ltd.
38. Badische Anilin & Soda Fabrik A.G.
39. Elcometer Instruments Ltd.
40. S.C.C. Colours Ltd.

Stand

41. Johns-Manville Co. Ltd.
42. Kunsttharsfabriek Synthese N.V.
43. Berk Limited.
44. Sturge, John & E., Ltd.
45. Resinous Chemicals Ltd.
46. Sawell Publications Ltd. (*Paint Technology*)
47. Blagden, Victor, & Co. Ltd.
48. Scott Bader & Co. Ltd.
49. British Oxygen Chemicals Ltd.
50. British Titan Products Co. Ltd.
51. Titanium Intermediates Ltd.
52. Technical Education Stand
53. Paint Research Station
54. National Provincial Bank Ltd.
55. Oil & Colour Chemists' Association Information Bureau
56. Campbell, Rex, & Co. Ltd.
56. Chemical Supply Co. Ltd.
57. Scado-Archer-Daniels N.V.
58. Byk Gulden Lomberg GmbH
59. Chemische Werke Huels A.G.
60. Shell Chemical Co. Ltd.
61. Kronos Titanium Pigments Ltd.
62. *Paint, Oil & Colour Journal*
63. Silverson Machines Ltd.
64. B.I.P. Chemicals Ltd.
65. Pure Chemicals Ltd.
66. Imperial Smelting Corporation Ltd.
67. Hercules Powder Co. Ltd.
68. Torrance & Sons Ltd.
68. Holmes Bros. Paint Machinery Ltd.
69. Interpreters
70. N.V. Titaandioxydefabriek Tiofine
71. Bush Beach & Segner Bayley Ltd.
71. Degussa
72. Mitchell, L. A., Group of Companies
73. Imperial Chemical Industries Ltd.
74. Sheen Instruments (Sales) Ltd.
75. British Resin Products Ltd.
75. Distillers Co. Ltd., The, Industrial Solvents Division
76. Price's (Bromborough) Ltd.
77. Meijer's, Rudolph, Inc.
78. Durham Raw Materials Ltd.

Numerical List of Exhibitors—*continued**Stand*

79. Albright & Wilson (Mfg) Ltd.
 80. Dow Chemical Company (U.K.) Ltd.
 81. Surface Coating Synthetics Ltd.
 82. Geigy (U.K.) Ltd.
 83. Lennig Chemicals Ltd.
 84. Gardner Laboratory Inc.
 85. Vinyl Products Ltd.
 86. Spelthorne Metals Ltd.
 87. Anchor Chemical Co. Ltd.
 88. Livingston Electronics Ltd.
 89. Boehm, Fredk., Ltd.
 90. Vuorikemia OY
 91. Cornelius Chemical Co. Ltd.
 92. Universal Oil Co. Ltd., The
 92. Seaton, John L., & Co. Ltd.

Stand

92. Premier Oil & Cake Mills Ltd.
 93. Hoechst (U.K.) Ltd.
 94. D.H. Industries Ltd.
 95. Beck, Koller & Co. (England) Ltd.
 96. English Clays Lovering Pochin & Co. Ltd.
 97. Dunlop Chemical Products Division
 98. Winkworth Machinery Ltd.
 99. Cornbrook Resin Co. Ltd.
 100. Carless, Capel & Leonard Ltd.
 101. Shawinigan Ltd.
 102. Elga Products Ltd.
 103. Armour Hess Chemicals Ltd.
 104. Croxton & Garry Ltd.
 105. Pyrene Co. Ltd., The
 106. Molteni Off. Mecc.
 107. Gebruder Netzsch

In addition to the exhibitors listed above, reference is also made in the *Official Guide* to the following companies.

<i>Stand</i>	<i>Official Guide</i> <i>Page No.</i>	<i>Stand</i>	<i>Official Guide</i> <i>Page No.</i>
34 Amoco Chemical Corporation	113	87 Marbon Chemical Division of Borg-Warner Corporation	54
94 AMF Corporation	86	91 Mearl Corporation	80
64 Barter Trading Corporation Ltd.	62	75 Methylating Co. Ltd., The	86
33 Brookfield Engineering Laboratories	56	47 Mulhouse, Societe de Produits Chimiques et Matieres Colorantes de,	63
47 Cargill Incorporated	63	30 Noury and van der Lande N.V.	125
91 Cray Valley Products Ltd.	80	78 Nuodex Ltd.	89
89 Chemische Werke Albert	64	33 Optica (UK) Ltd.	56
47 Claremont Polychemical Corporation	63	94 Pamasol	86
15 Fullers Earth Union Ltd.	116	104 Pluess-Stauber AG	84
33 Gebruder Haake K.G.	56	104 RBH Interchemical Corporation	85
43 Georgia Kaolin Co. Ltd.	61	91 Rohm and Haas Ltd.	80
12a Henkel International	118	34 Sherwin Williams Co.	112
15 Howards of Ilford Ltd.	116	91 Sinclair Petrochemicals Incorporated	80
77 IGT Graphic Arts Research Institute	122	34 Stapern Ltd.	112
91 Industrial Colours Ltd.	80	94 Sussmeyer, Ateliers. SPRL	85
94 Jenag Equipment Ltd.	86	34 Trojan Powder Co.	110
15 Laporte Titanium Ltd.	116	76 Unilever Emery N.V.	137
15 Laporte Chemicals Ltd.	116	94 Vree, J de, & Co. Ltd.	86
36 Lehmann, F. B., GmbH	65	71 Wacker Chemie GmbH	70

Exhibitor	Stand No.	Resins	Pigments	Extenders	Additives	Solvents	Oils	Machinery	Lab. Machinery	Instruments	Misc.
Elga Products Ltd. ..	102										De-ionised water equipment
English Clays Lovering Pochin & Co. Ltd. ..	96			•							
Farbenfabriken Bayer AG	21	•	•								
Ferranti Ltd.	37									•	
Gardner Laboratories Inc.	84									•	
Gebruder Netzsch ..	107							•			
Geigy (UK) Ltd. ..	82		•								
Hercules Powder Co. Ltd.	67	•			•						
Hilger and Watts Ltd. ..	8									•	
Hoechst (UK) Ltd. ..	93	•	•								
Imperial Chemical Industries Ltd.	73	•	•								Trichloroethylene based paints
Imperial Smelting Corporation Ltd.	66		•								
Johns-Manville Ltd. ..	41			•							
Joyce Loebel & Co. Ltd.	11									•	
Kingsley & Keith (Chemicals) Ltd.	34	•	•								
Kronos Titanium Pigments Ltd.	61		•								
Kunstharsfabriek Synthese NV	42	•									
Lancashire Tar Distillers Ltd.	3	•				•					
Laporte Industries Ltd. ..	15		•	•		•					
Lennig Chemicals Ltd. ..	83	•									
J. H. Little & Co. Ltd. ..	12a	•			•	•					

A large, stylized graphic of the letter 'G' is the central focus. The 'G' is white with a thick red outline and is set against a solid red background. The 'G' is positioned in the upper half of the page, with its bottom curve extending towards the bottom of the page. The overall design is minimalist and high-contrast.

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S. Pedro do Estoril, Portugal.**

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Savoy Dinner Dance · 15 April 1966

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for further details contact the General Secretary
at the address on the front cover

Exhibitor	Stand No.	Resins	Pigments	Extenders	Additives	Solvents	Oils	Machinery	Lab. Machinery	Instruments	Misc.
S. C. C. Colours Ltd. . .	40		•								
Scott Bader & Co. Ltd. . .	48	•									
Shawinigan Ltd.	101	•									Raw materials
Sheen Instruments (Sales) Ltd.	74									•	
Shell Chemical Co. Ltd.	60	•			•	•					
Silverson Machines Ltd.	63							•	•		
Spelthorne Metals Ltd. . .	86		•								
Steele & Cowlshaw Ltd.	32							•			
John & E. Sturge Ltd. . .	44		•	•	•						
Styrene Co-Polymers Ltd.	28	•									
Surface Coatings Synthetics Ltd.	81	•									
Svenska Oljeslageri Aktiebolaget	22	•									
NV Titaandioxydfabrik . .	70		•								
Titanium Intermediates Ltd.	51				•						
Torrance & Sons Ltd. . .	68							•	•		
The Torsion Balance Co. (Great Britain) Ltd. . .	2									•	
The Universal Oil Co. Ltd.	92						•				
Vinyl Products Ltd. . .	85	•									
Vourikemia OY	90		•								
Winkworth Machinery Ltd.	98							•	•		

The Foundation Lecture

(Instituted in 1963 in memory of the late H. A. Carwood Esq.)

As announced in the January issue, the next occasion on which the lecture will be delivered will be following the Association's Annual General Meeting on 30 June 1966 at the Painter Stainers' Hall, Little Trinity Lane, London, E.C.4.

The lecture, entitled "Craft Against Craft, Makes No Living," will be given on this occasion by Dr. N. F. Astbury, Director, British Ceramic Research Association, and Chairman of the Committee

of Directors of Research Association. The lecture will commence at 7 p.m. and be followed by a reception and dinner. Members only may attend and the inclusive charge for the dinner together with wines and drinks at the reception will be £2 10s. per head.

Full details have been circulated to all members attached to the United Kingdom and General Overseas Sections, and the latest date for the receipt of applications is 16 June 1966.

Irish Section

Dinner and Dance 1965

The last social function of the Bristol Section's Irish Branch (since 1 January 1966, the Irish Section) took place at the South County Hotel, Stillorgan, Dublin, on Thursday 9 December 1965 and a large number of members, their wives, and guests enjoyed a very pleasant evening.

The total number present was 94 and this was a considerable increase on the

previous year's figures. The Chairman, Mr. J. H. Quick, announced the receipt of a telegram from London advising that Section status had been approved as from 1 January 1966. A large number of spot prizes were distributed during the evening and the Chairman expressed his thanks to the donors who had responded so well in making available many excellent gifts.

M. O'C.

Midlands Section

Ladies' Night

The annual Ladies' Night of the Midlands Section was held on 6 January at the George Hotel, Solihull. The guests were received by Mr. R. D. Calvert (Chairman, Midlands Section), Mrs. Calvert and Dr. S. H. Bell (President). Mr. D. J. Silsby, the Hon. Secretary of the Section, acted as toastmaster.

After members and guests had enjoyed

an excellent dinner the President proposed a toast to the Section. He said that the Association would soon be celebrating its fiftieth anniversary and gave an impressive list of outstanding contributions made by members of the Section to the growth of the Association. It was not too early to consider the role of the Association in the next 50 years and he was confident that members of Midlands Section would continue to play an



The President (right) with the Chairman (Mr. R. D. Calvert) and Mrs. Calvert at the Reception

important part. He then invested the Chairman with the Chairman's Insignia.

Mr. R. D. Calvert replied to the President's speech and went on to propose a toast to the Ladies and Guests. The principal guests were the President, Dr. J. Newton Friend (Senior Past President) and Mrs. Friend, Mr. S. A. Ray (President of Birmingham Paint Varnish & Lacquer Club) and Mrs. Ray, Mr. H. F. Clay (Chairman, Manchester Section) and Mrs. Clay, Mr. N. Cochrane (Chairman, West Riding Section) and Mrs. Cochrane, Mr. R. J. Woodbridge (Chairman, Bristol Section) and Mrs. Woodbridge and Mr. R. H. Hamblin (General Secretary). As it was 6 January, the Chairman quoted some pertinent remarks about ladies by Shakespeare in "Twelfth Night."

The response to the toast was made by Mr. N. Cochrane who also expressed thanks on behalf of the ladies for the present which each had received. After a short interval, whilst the tables were cleared, dancing to the Mary Arden Orchestra continued until 1.00 a.m.

L. R. S.

Scottish Section

Annual Dinner-Dance

The Annual Dinner-Dance of the Scottish Section was held in the Central Hotel, Glasgow, on Friday 14 January. Guests were received by Dr. Atherton and Mrs. Atherton and Dr. Bell.

Proposing what he said would be a brief and lighthearted toast to the Scottish Section, Dr. Bell immediately laid claim to a large measure of Highland ancestry, being, as he put it, "more than half Scotch." Moreover, he was, in the event, delighted to leave the currently frozen south for the more temperate climate of the north. He reminded the company that the Association would achieve its jubilee shortly, in 1968. This was a great milestone and an occasion truly worthy of celebration.

Within this context, the Scottish Section had a considerable record of work, that amongst the Junior Members being particularly excellent. He noted, with satisfaction, that Dr. Atherton was looking resplendent in his new badge of office.

In reply, Dr. Atherton thanked Dr. Bell for his gracious words to the Section. He wished to take the opportunity of expressing his thanks to the Section Committee as a whole, to Mr. Horsburgh and Mr. Hutchison, in particular, for their organisation of the dinner-dance, to Mr. Birrell for his excellent work with the Student Group, and to the Chairman and Committee of the Eastern Branch for their assistance throughout the year. He took special pride in

congratulating Mr. R. Hill, Chairman of the Student Group, who had not only won the Scottish Section Essay Competition with a paper on "Phthalocyanine Pigments," but had had the paper published in the Association's *Journal* and had also won with it a first prize in the *Paint Technology* Essay Competition. Dr. Atherton was glad to have amongst the guests Dr. Bell (President of the Association), Mr. Clay (Chairman of the Manchester Section) and Mrs. Clay, Mr. Holmes (Chairman of the Scottish Section, Eastern Branch) and Mrs. Holmes, Mr. Hamblin (the General Secretary), and Mr. Rudram (Hon. Research Liaison Officer of the Association) and Mrs. Rudram. He extended a very warm welcome to all old friends and especially to the ladies.

Replying on behalf of the guests, Mr. Clay commented that it was quite remarkable how, over the years, a succession of guests managed to lay claim to Scottish ancestry! For his own part, he was afraid that this was something he was unable to do. He was most grateful to all who had organised the function and, on behalf of everyone, expressed his thanks to the Scottish Section.

Thereafter the evening was devoted to dancing, which continued until 1 a.m. The entire function was, as ever, a most enjoyable one, and the fact that the demand for tickets greatly exceeded the supply is indicative of the degree to which the Scottish Section Dinner-Dance goes from strength to strength.

A. MCL.

West Riding Section

Dinner and Dance

The Seventh Annual Dinner and Dance of the Section was held at the Granby Hotel, Harrogate, the principal guests being the President, Dr. S. H. Bell, and the Rt. Hon. James Ramsden, MP, Harrogate Division. Despite heavy snow there was an excellent attendance to enjoy the Christmas fare, and the dancing until 2 a.m.

Speechmaking was kept to a minimum

and a highlight was the presentation to the President by the Section Chairman (Mr. N. Cochrane) of a shield bearing a "West Riding Section Coat-of-Arms" in recognition of the President's interest in heraldry.

During the speeches the Chairman read out a cable of greetings from the Section's Vice-Chairman, Mr. L. Silver, who was in the USA on business.

J. N. MCK.

Joint Polymer Conference

The next Joint Polymer Conference sponsored by the Society of Chemical Industry, the Plastics Institute and the Institution of the Rubber Industry will take place in London on 20, 21, 22 September 1966. The Association and the Society of Dyers and Colourists have been invited to appoint representatives on the Organising Committee and the Honorary Research and Development Officer (Mr. A. T. S. Rudram) and the General Secretary (Mr. R. H. Hamblin)

serve as the Association's representatives. The Conference will be divided into four sessions, all of which will take place at the Institution of Electrical Engineers. Provisionally, fees for the Conference will be £4 each to members of the five societies, £6 each for others and £2 each for bona fide full-time students. Full details will be available in a Conference programme and application form from the Society of Chemical Industry, 14 Belgrave Square, London, S.W.1.

Fatipec Congress 1966

A supply of copies of the provisional programme and enrolment form for the 8th FATIPEC Congress, which will take place in Holland, 6-10 June, 1966, have been received at the Association's offices and these are available to members upon application.

The subject chosen for the Congress is "Scientific Research on the Protection of Substrates by Paints" and the Secretariat of the Congress is situated at 14, Burgemeester de Monchylein, La Haye, Pays-Bas.

Symposium on Colour

A symposium on colour, organised by the Society of Cosmetic Chemists of Great Britain, will be held at the Grand Hotel, Eastbourne, Sussex, on 26 and 27 April, 1966.

The text of the 13 lectures will be circulated beforehand to registered participants and the symposium periods will be devoted to discussion on the topics.

The registration fee is 5 gns. for members of societies affiliated to the International Federation of Societies of Cosmetic Chemists and 8 gns. for non-members. Registration forms and full details are available from Mrs. D. Mott, 18 Warner Close, Harlington, Middlesex.

News of members

Mr. R. F. Hill, the Secretary of the Scottish Section Student Grouping, has been awarded 1st prize in the "Paint Technology" annual award scheme for his paper on "Phthalocyanine Pigments and their Applications" (*JOCCA* 48, 603). We congratulate Mr. Hill on this achievement.

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

- ANDOCZY, BELA, 50 Minnamurra Road, Northbridge, New South Wales, Australia.
(New South Wales)
- ANWAR, MUHAMMAD, B.SC., 22 Grantham Place, Bradford, 7.
(Midlands)
- BIEGEL, JAMES A., B.SC., M.SC., Box 106, Portland, Maine, USA
(Overseas)
- BISHOP, NIGEL MINARDS, 11 Sheepcotes Road, Chadwell Heath, Romford, Essex.
(London)
- BUTLER, MICHAEL JOHN, Adshead, Ratcliffe & Co., Belper, Derbyshire.
(Midlands—Trent Valley Branch)
- ENTWHISTLE, OLIVER ALMOND, PO Box 704, Alrode, Transvaal, South Africa.
(South African)
- FROHLICH, PETER, c/o Ciba Co., Orion Road, Lane Cove, New South Wales, Australia.
(New South Wales)
- GWATKIN, EDWARD ALAN DELAFIELD, B.SC., A.S.T.C., 45 Roseview Avenue, Punchbowl, New South Wales, Australia.
(New South Wales)
- HURST, ALBERT ROBERT, 82 Tara Street, Sylvania Heights, New South Wales, Australia.
(New South Wales)
- JOHANNSEN, RALF, Plascon Paints, PO Box 10, Luipaardsvlei, Transvaal, South Africa.
(South African)
- LONG, JAMES SCOTT, M.SC., PH.D., F.R.S.A., F.A.I.C., 1109 Windsor Drive, Hattiesburg, Mississippi, USA.
(Overseas)
- MUEEZ, MOHAMMAD ANWAR, M.SC., D.I.C., The Paint Research Station, Waldegrave Road, Teddington, Middlesex.
(London)

- OSBORN, CLIVE ALEXANDER, Warden & Hotchkiss Ltd., PO Box 399, Durban, South Africa. *(South African)*
- ROCHE, HOWARD OWEN, c/o The Standard Manufacturing Co. Ltd., Water Street East, St. John's, Newfoundland, Canada. *(Overseas)*
- RYAN, GREGORY JOSEPH, John Lysaght (Australia) Ltd., CRM Works, Box 77, PO, Port Kembla, New South Wales, Australia. *(New South Wales)*
- SMITH, JOHN PROTHEROE, B.SC., Laporte Titanium Ltd., New Bond Street House, 1-5 New Bond Street, London, W.1. *(London)*
- SORENSEN, BRIAN HENRY, B.SC., A.R.A.C.I., 253 Northan Avenue, Bankstown, New South Wales, Australia. *(New South Wales)*
- SUSSMAN, JOHN, B.SC., 10-16 Glen Street, Paddington, New South Wales, Australia. *(New South Wales)*
- WATSON, MALCOLM PATRICK, B.SC., c/o Buffalo Paints, PO Box 2390, Durban, South Africa. *(South African)*
- WATSON, ROBERT, B.SC., 309 Strand Lane, Radcliffe, Manchester. *(Manchester)*
- WATSON, ROY GRANT, ICI (SA) Ltd., Box 1519, Cape Town, South Africa. *(South African)*
- WHITE, PHILIP STANWAY, Laporte Titanium Ltd. (Australia), Bunbury, Western Australia. *(Western Australian)*

Associate Members

- BACKHOUSE, DONALD, 94 MacKenzie Street, Revesby, New South Wales, Australia. *(New South Wales)*
- CAMERON, MICHAEL PHILIP, 52 Carlton Gore Road, Grafton, Auckland, New Zealand. *(Auckland)*
- COURTNEY, ALBERT CHARLES, 71 Monteith Street, Warrawee, New South Wales, Australia. *(New South Wales)*
- DEVENS, HORST P. K., c/o Hoechst SA (Pty.) Ltd., PO Box 8692, Johannesburg, South Africa. *(South African)*
- MORAN, JAMES STANLEY, BASF (Australia) Ltd., 1 Price Street, Subiaco, Western Australia. *(Western Australian)*
- SCHROETER, HORST MAX, c/o Hoechst SA (Pty.) Ltd., PO Box 8692, Johannesburg, Transvaal, South Africa. *(South African)*
- SEYD, ROLF DIETER, c/o Hoechst SA (Pty.) Ltd., PO Box 8692, Johannesburg, South Africa. *(South African)*
- SUTHERLAND, ALEXANDER REGINALD, 55 Northwood Road, Northwood, New South Wales, Australia. *(New South Wales)*

Junior Members

- CHELL, NEIL, 32 Coronation Avenue, Belper, Derbyshire. *(Midlands—Trent Valley Branch)*
- CHURNSIDE, MICHAEL JOHN, British Paints Ltd., Portland Road, Newcastle upon Tyne, 2. *(Newcastle)*
- DAVISON, JOHN, 32 Ladysmith Terrace, Craghead, Stanley, Co. Durham. *(Newcastle)*
- DUNN, DOUGLAS ROLAND, 30 Windermere Terrace, North Shields, Northumberland. *(Newcastle)*
- FIELD, ALLAN JOHN, Smith & Walton (SA) Pty. Ltd., PO Box 46, Jacobs, Natal, South Africa. *(South African)*
- LEWIN, DAVID, Dulux Ltd., PO Box 704, Alrode, Transvaal, South Africa. *(South African)*
- WROE-STREET, RONALD HUGH, Dulux Ltd., PO Box 704, Alrode, Transvaal, South Africa. *(South African)*

Amendment to January Journal

- LEWIS HUW HENRY, 6 Cromwell Meadow, Litchfield, Staffs. *(Manchester)*

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 after publication.

Thursday 3 March

Newcastle Section. "To Automate? . . . To Speculate?," by Mr. H. R. Touchin, to be held at Royal Turks Head Hotel, Grey Street, Newcastle, at 6.30 p.m.

Monday 7 March

Bristol Section. Annual Dance to be held at Hawthorns Hotel, Bristol.

Hull Section. "The Evaluation of the Optical Properties of Titanium Dioxide Pigments," by Mr. F. D. Robinson, to be held at Royal Station Hotel, Hull, at 7 p.m.

Tuesday 8 March

West Riding Section. "The Exploitation of New Methods of Paint Application and the Examination of their Potential Advantages and the Development of Paints Designed to Exploit Them," by Mr. F. G. Dunkley, to be held at Great Northern Hotel, Leeds, at 7.30 p.m.

Wednesday 9 March

London Section—Southern Branch. "Colour Photography," by Mr. R. G. Barber, at the Polygon Hotel, Southampton, at 7.30 p.m.

Thursday 10 March

Scottish Section. "The Manufacture of Carbon Black Pigment and Dispersions," by Mr. L. Venuto, to be held at Mores Hotel, India Street, Glasgow, at 6.30 p.m.

Friday 11 March

Manchester Section. "Modern Developments in Industrial Surface Coatings," by Mr. F. Armitage, at The Strand Hotel, Brunswick Street, Liverpool.

Saturday 12 March

Scottish Section—Junior Group. "Paint Testing," by Mr. R. Blackie (British Titan Products Ltd.), to be held at Mores Hotel, India Street, Glasgow, C.2, at 10.30 a.m.

Monday 14 March to Friday 18 March

OCCA Exhibition. 18th Technical Exhibition (for full details, see p. 241).

Wednesday 23 March

Scottish Section—Eastern Branch. Annual General Meeting, at 7 p.m., followed by lecture "Protection of Structures," by Mr. F. G. Dunkley, to be held at North British Hotel, Princes Street, Edinburgh.

Thursday 24 March

London Section. "Adhesion as a Factor in the Performance of Solvent-free Coatings," by Messrs. E. W. Garnish and P. T. Brown. Refreshments will be available from 5.45 p.m. Lecture will commence at 6.30 p.m., at Small Physics Lecture Theatre, Imperial College of Science and Technology, South Kensington, S.W.7.

Friday 25 March

Bristol Section. "Outdoor Exposure Results with Titanium Pigments," by Dr. Epple and Dr. Wagener, to be held at Royal Hotel, St. Mary Street, Cardiff.

Irish Section. Annual General Meeting, at 7.30 p.m., followed by lecture "Design and Decoration in Modern Architecture," by Mr. P. N. Linahan, at 8 p.m. Venue to be announced.

Midlands Section. "Robert Boyle and his Experiments Touching Colour," by

Mr. G. A. Campbell (Newton Friend Lecture), to be held at Chamber of Commerce House, 75 Harborne Road, Birmingham 15, at 6.30 p.m.

Newcastle Section. Ladies' Night, to be held at Beaumont Hotel, Hexham.

Monday 18 April to Tuesday 19 April

Manchester Section. A Symposium on Emulsion Paints, to be held at Manchester College of Science and Technology, The Large Lecture Theatre, Renold Building, Monday 18, 2 to 5 p.m., Tuesday 19, 9.30 to 4.30 p.m.

Wednesday 30 March

London Section—Thames Valley Branch. "Vivid Lightfast Pigments," by a speaker from Harmon Division of Allied Chemicals Corporation, to be held at Royal White Hart Hotel, Beaconsfield, Buckinghamshire, at 7 p.m.

Wednesday 20 April

Scottish Section—Eastern Branch. "Resins from Cyclic Ketones," by Mr. V. F. Jenkins (Howards of Ilford), to be held at North British Hotel, Princes Street, Edinburgh, at 7.30 p.m.

Friday 1 April

Scottish Section. Annual General Meeting, at St. Enochs Hotel, at 6 p.m., followed by the Annual Smoking Concert, at the Eglington Arms Hotel, Eaglesham, at 7.30 p.m.

Thursday 21 April

London Section. Annual General Meeting, to be held at the Criterion-in-Piccadilly, London, W.1.

New South Wales Section. "Automotive Finishing," by Mr. P. Fairbain, to be held at the Theatre, MLC Building, Miller Street, North Sydney, at 8 p.m.

Monday 4 April

London Section—Southern Branch. Annual General Meeting.

Friday 22 April

Manchester Section. Annual General Meeting, to be held at Waldorf Hotel, Copper Street, Manchester, 2, at 6.30 p.m.

Midlands Section. Annual General Meeting, venue to be arranged.

Thursday 7 April

Midlands Section—Trent Valley Branch. Annual General Meeting. Venue to be arranged.

Newcastle Section. Annual General Meeting.

Friday 29 April

Bristol Section. Annual General Meeting. Followed by "Colour Photography as a Recording Medium" by Kodak Limited, to be held at Royal Hotel, Bristol, at 7.15 p.m.

Tuesday 12 April

West Riding Section. Annual General Meeting.

Thursday 14 April

London Section—Thames Valley Branch. Annual General Meeting, to be held at the Royal White Hart Hotel, Beaconsfield, Buckinghamshire, at 7 p.m.

Thursday 19 May

New South Wales Section. "A lecture on Costing and Forecasting in the Paint Industry."

Friday 15 April

OCCA BIENNIAL DINNER-DANCE to be held at Savoy Hotel, London.

Oil and Colour Chemists' Association

President: S. H. BELL, PH.D., D.I.C., A.R.C.S., F.R.I.C.

The Oil and Colour Chemists' Association was formed in 1918, to cover paint, printing inks, pigments, varnishes, drying and essential oils, resins, lacquers, soaps, linoleum and treated fabrics, and the plant, apparatus and raw materials useful in their manufacture. In 1923 it absorbed the Paint and Varnish Society. The stated purpose of the Association is to promote by discussion and scientific investigation the technology of the industries concerned with the above-mentioned products, and to afford members opportunity for the interchange of ideas. This is achieved by the regular holding of ordinary meetings at which papers are presented, and the organisation of annual technical exhibitions, biennial conferences, educational activities and practical co-operative experimental work. Details of these activities are given in the *Journal of the Oil and Colour Chemists' Association*, which is published monthly, and whose pages are open to receive communications and other pronouncements on scientific and technical matters affecting the members of the Association and the industries concerned. The Association's meetings also afford opportunities for members to meet informally and socially.

There are Sections of OCCA in Auckland, Bristol (with an Irish Branch), Hull, London (with Southern and Thames Valley Branches), Manchester, the Midlands (with a Trent Valley Branch), Newcastle upon Tyne, New South Wales, Queensland, Scotland (with an Eastern Branch, South Africa (with Branches in the Cape, Transvaal and Natal), South Australia, Victoria, Wellington, West Australia and the West Riding, and these are responsible for the conduct of their own local affairs. There is also a General Overseas Section. There is also a close alliance between the Association, the Federation of Societies for Paint Technology in the United States, and the Fédération d'Associations des Techniciens de l'Industrie des Peintures, Vernis, Emaux et Encres d'Imprimerie de l'Europe Continentale (FATIPEC). The Association also maintains cordial relations with the Scandinavian Federation of Paint and Varnish Technicians (SLF).

Ordinary Membership is granted to scientifically trained persons, and Associate Membership to others interested in the industries covered. Junior Membership, which is intended primarily for students, is open without restriction to persons under the age of 21 and to those up to 25 who are following a course of technical study. The annual subscription in each case is three guineas, except for Junior Members whose subscription is 10s. 6d. An entrance fee of 10s. is payable by all members. Applications for membership are invited from suitably qualified persons who are engaged or otherwise interested in the industries noted above. Applications, which should be supported by two members of the Association (one of whom must be an Ordinary Member) should be forwarded to the General Secretary at the address given below. Application forms and full details of membership may be obtained from the offices of the Association.

PUBLICATIONS

Journal of the Oil and Colour Chemists' Association, Published monthly. Subscription rate to non-members in UK and abroad, £7 10s. p.a. post free; payable in advance.

An Introduction to Paint Technology (Second Edition). Pp. 187, illustrated, with index, 15s. (including postage).

Paint Technology Manuals

Part 1 : "Non-convertible Coatings," Pp. 326, 35s.

Part 2 : "Solvents, Oils, Resins and Driers," Pp. 239, 35s.

Part 3 : "Convertible Coatings," Pp. 318, 35s.

Part 4 : "The Application of Surface Coatings," Pp. 345, 35s.

Part 5 : "The Testing of Paints," Pp. 196, 35s.

General Secretary : R. H. Hamblin, M.A., F.C.I.S., F.C.C.S., Wax Chandler's Hall, Gresham Street, London, E.C.2.

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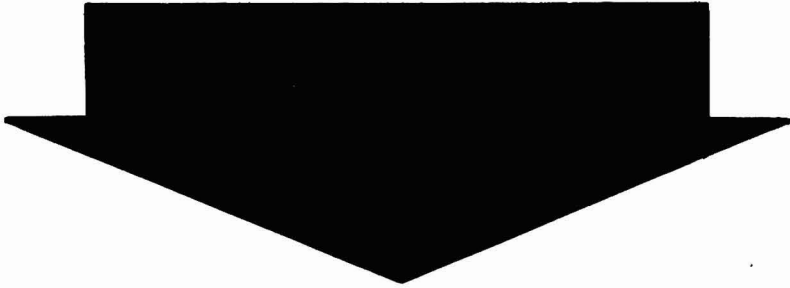
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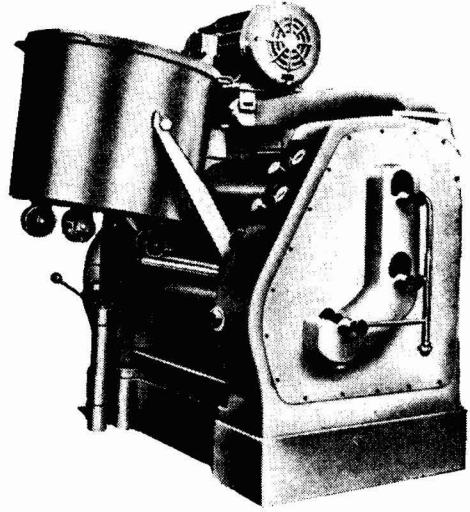
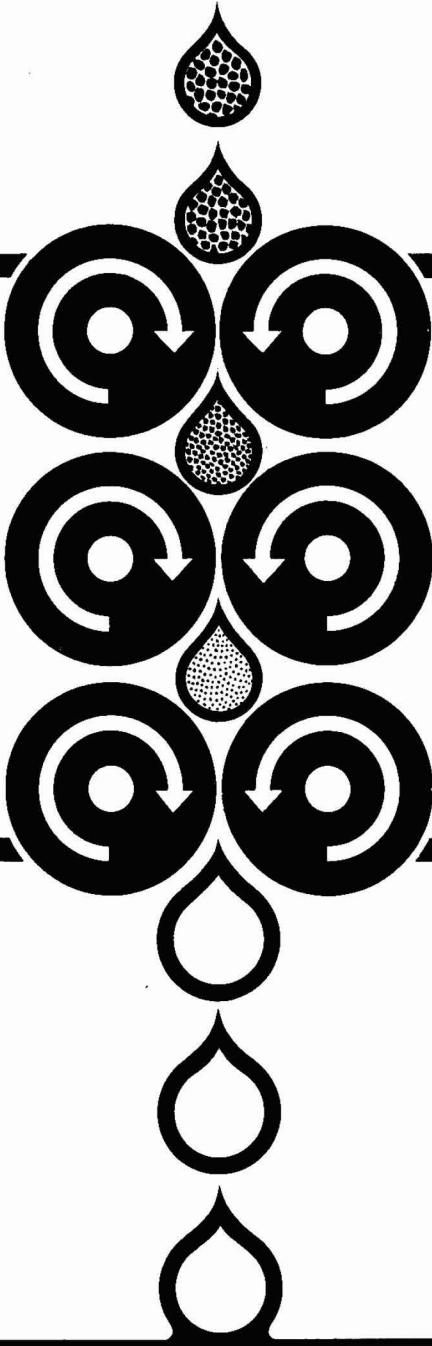
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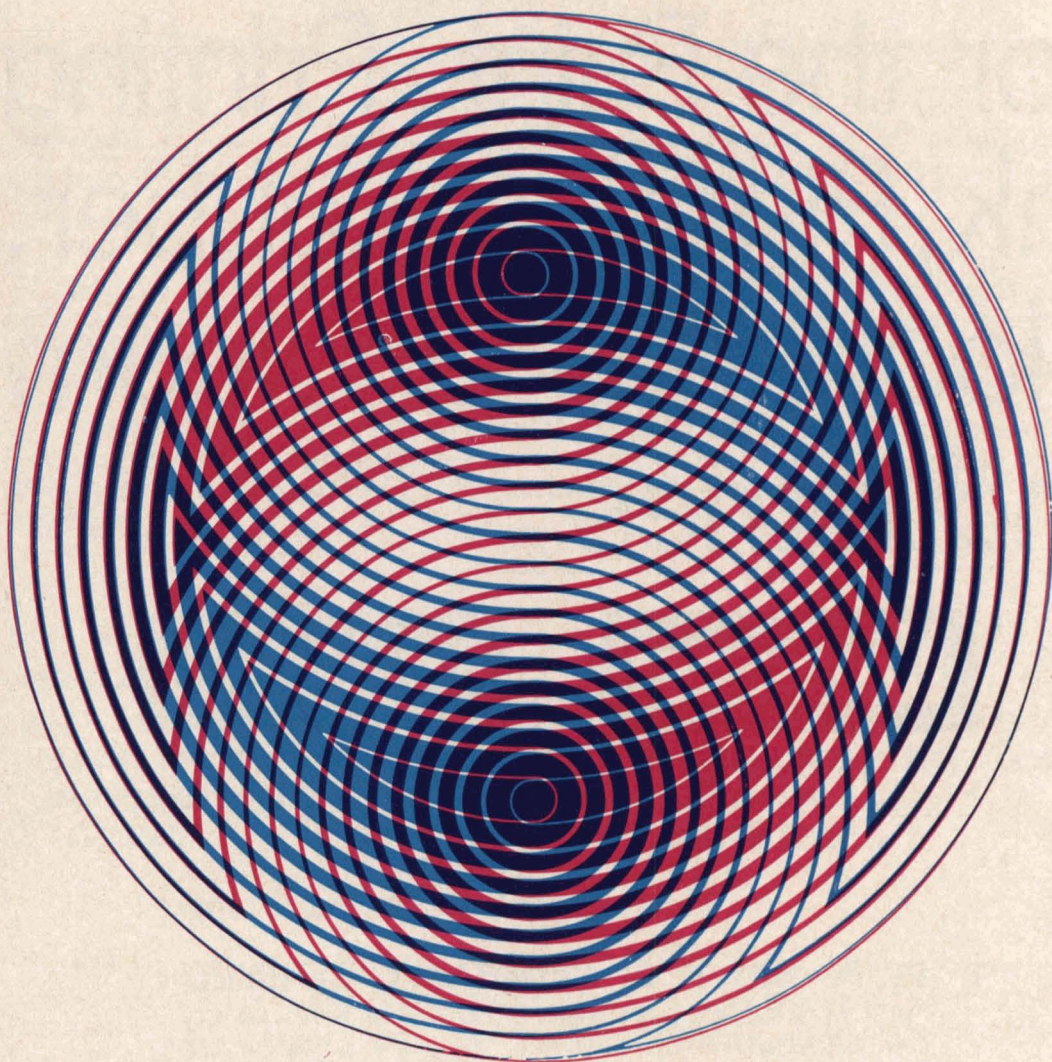
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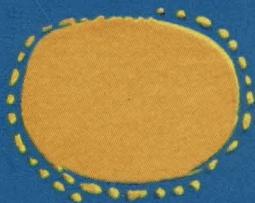
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